

Evaluation of Solidification History Dependent Crystal Defects Using X-ray Diffraction

A dissertation submitted by

Colby Azersky

IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF

DOCTOR OF PHILOSOPHY

IN

MECHANICAL ENGINEERING

Tufts University

August 2024

Advisor: Dr. Douglas Matson

Abstract

This dissertation investigates the practical implications of the solidification process for metals. The density of atomic defects resulting from solidification is quantified, as this value influences mechanical properties on a macro scale. The study examines three metal alloy powders: Inconel 718, Aluminum Alloy 2024, and a binary Fe-Al alloy. This work addresses a gap in the literature, as most existing research on dislocation density pertains to bulk samples rather than metal powders with relatively higher cooling rates. With the rise of additive manufacturing, understanding these properties in metal powders is essential.

The samples were isolated into distinct particle size ranges to analyze the effects of varying solidification histories. The size range isolation was verified using scanning electron microscopy (SEM). Thermodynamic equations and metallographic analysis were used to correlate the particle size ranges with their cooling rates during solidification. The research employs line profile analysis (LPA) of X-ray diffraction (XRD) data to determine dislocation density using the Modified Williamson-Hall (MWH) and Modified Warren-Averbach methods (MWA). This work culminated in synchrotron XRD using a particle accelerator. Electron backscatter diffraction (EBSD) was used to approximate dislocation density trends using average misorientation angle to validate results.

The findings indicate a correlation between solidification history and dislocation density, with smaller particles exhibiting higher dislocation densities due to faster cooling rates. In addition to advancing the fundamental understanding of solidification and dislocation formation in metal powders, this work provides practical insights for industry applications. Through an improved understanding of the relationship between solidification and resulting microstructure, manufacturing processes can be optimized to improve quality and reliability.

Signature Page

Author

Colby Azersky



Advisor & Committee Chair

Dr. Douglas M. Matson, Professor
Department of Mechanical Engineering
Tufts University

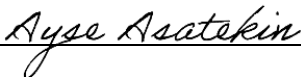


Committee

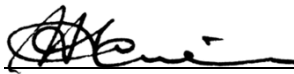
Dr. Jannatun Nower, Research Assistant Professor
Department of Mechanical Engineering
Tufts University



Dr. Ayse Asatekin, Associate Professor
Department of Chemical and Biological Engineering
Tufts University



Dr. Hani Henein, Professor
Department of Chemical and Materials Engineering
University of Alberta



Acknowledgments

I would like to express my sincere gratitude to my advisor, Dr. Douglas M. Matson, for his guidance, support, and mentorship in both my education and career. My life would not be the same if I did not join your research group.

I would also like to thank dissertation committee members Dr. Jannatun Nawer, Dr. Ayse Asatekin, and Dr. Hani Henein for their valuable suggestions and support. I am also thankful to everyone who has helped me with technical work along the way: Dr. Sangho Jeon for teaching me dislocation density analysis methods, Dr. Charles Settens for teaching me how to perform XRD experiments at MIT, and Dr. Matthias Kolbe for teaching me about EBSD evaluations. Additionally, I would like to thank Dr. Peggy Cebe for support at the Tufts XRD facility, Dr. Andrew Chuang for support at APS, and Dr. Evan Baker for support with proposals. Finally, I would also like to thank Mr. Michael Sansoucie, Dr. Caitlin Walde, and Dr. Abdoul-Aziz Bogno for assistance with the Inconel 718, Al2024, and Fe-Al samples.

This research was supported by NASA grants NNX17AH41G, 80NSSC19K0256, and 80NSSC23K0573, and MIT XRD facility grants DMR-1419807, DMR-0602473, DMR-0100646, and NAG8-1167. I am grateful to the staff of the APS synchrotron at Argonne National Laboratory and the DESY synchrotron for granting me beam time and technical support during experiments.

Most importantly, I recognize my mom, dad, family, and friends for their unwavering support throughout this process. I could not have done it without you all in my corner.

Thank you all for your contributions and support.

Colby J. Azersky

August 2024

Table of Contents

Abstract	ii
Acknowledgments.....	iv
Table of Contents	v
List of Figures	viii
List of Tables.....	xii
List of Appendices	xiii
1 Introduction.....	1
2 Background.....	6
2.1 Materials	6
2.1.1 Inconel 718.....	6
2.1.2 Aluminum Alloy 2024	7
2.1.3 Iron-Aluminum Alloy	7
2.1.4 Atomization Processes	8
2.2 Solidification.....	9
2.2.1 Solidification Theory	10
2.2.2 Heat Balance	11
2.2.3 Microstructural Evolution.....	15
2.3 Crystallography.....	16
2.3.1 Crystal Structures.....	16
2.3.2 Crystal Defects.....	18
2.3.3 Heat Treatment.....	18
2.4 X-Ray Diffraction	19
2.4.1 Bragg's Law	20
2.4.2 Bragg Peaks	21
2.4.3 Scattering Vector	22
2.5 Scanning Electron Microscopy	23
2.5.1 Secondary Electron Imaging.....	23
2.5.2 Energy Dispersive X-ray Spectroscopy	23
2.5.3 Electron Backscatter Diffraction.....	24

3	Experimental Methods	26
3.1	Sample Preparation Procedure	26
3.1.1	Materials	26
3.1.2	Particle Size Isolation	29
3.1.3	Particle Size Validation	30
3.1.4	Heat Treatment.....	32
3.2	X-Ray Diffraction	35
3.2.1	X-Ray Diffraction Calibration	35
3.2.2	X-Ray Diffraction Facilities and Test Sequence	35
3.2.3	X-Ray Diffraction Sample Preparation.....	40
3.3	Electron Backscatter Diffraction.....	41
4	Data Analysis Methods	42
4.1	X-Ray Diffraction Line Profile Analysis	42
4.1.1	Transformation to Reciprocal Space.....	44
4.1.2	Curve Fitting Technique.....	44
4.1.3	Peak Location Calibration.....	49
4.1.4	Peak Broadening Calibration	51
4.1.5	Modified Williamson-Hall Method.....	53
4.1.6	Modified Warren-Averbach Method	58
4.2	Electron Back Scatter Diffraction Analysis	62
4.2.1	Misorientation Angle	62
4.2.2	Dislocation Density Approximation	65
4.3	Cooling Rate Approximation	66
4.3.1	Cooling Rate Determination using Particle Size	67
4.3.2	Cooling Rate Determination using Cell Size	73
4.3.3	Cell Identification Methods	74
5	Results and Discussion	78
5.1	Inconel 718 Results.....	78
5.1.1	X-Ray Diffraction	78
5.1.2	Electron Back Scatter Diffraction.....	85
5.1.3	Cooling Rate Approximation	86

5.2	Al2024 Results.....	89
5.2.1	X-Ray Diffraction	89
5.2.2	Electron Back Scatter Diffraction	94
5.2.3	Cooling Rate Approximation	97
5.3	Fe-Al Results	101
5.3.1	X-Ray Diffraction	101
5.3.2	Cooling Rate Approximation	106
5.4	Comparative Analysis	107
5.5	Literature Comparison	109
5.6	Error Sources	110
6	Conclusions and Future Work.....	114
6.1	Conclusions.....	114
6.1.1	Inconel 718.....	115
6.1.2	Al2024.....	116
6.1.3	Fe-Al	116
6.1.4	Application to Industry	117
6.2	Future Work	117
	References.....	119
	Appendix.....	129

List of Figures

Figure 2-1: Iron-aluminum binary phase diagram with 92% Fe 8% Al composition highlighted.	8
Figure 2-2: Schematic of gas and impulse atomization processes used to produce the samples.	9
Figure 2-3: Equipment used to conduct containerless processing research.	11
Figure 2-4: Heat transfer mechanisms in a falling particle.	14
Figure 2-5: Microstructural development during homogenous nucleation.	15
Figure 2-6: Simplified classification of crystallinity.	16
Figure 2-7: Simple cubic, body-centered cubic, and face-centered cubic atomic arrangements.	17
Figure 2-8: Slip planes of body-centered cubic and face-centered cubic atomic arrangements.	17
Figure 2-9: Edge and screw dislocations, demonstrating types of crystal defects.	18
Figure 2-10: Simplified X-ray diffraction experimental configuration.	19
Figure 2-11: Visualization of Bragg's Law, showing how X-rays interact with atomic planes.	20
Figure 2-12: X-ray diffraction data collection and transformation into a diffractogram.	21
Figure 2-13: Diagram showing the representation of a scattering vector.	22
Figure 2-14: Schematic relating crystallographic orientation to EBSD Kikuchi patterns.	24
Figure 2-15: Electron backscatter diffraction Kikuchi patterns.	25
Figure 2-16: EBSD inverse pole figure for Al2024 particle size 53-63 μm sample.	25
Figure 3-1: Fe-Al samples with distinct particle size ranges between 180 μm and 1180 μm	29
Figure 3-2: Sieve shaker with associated sieve sizes and respective isolated samples.	30
Figure 3-3: Inconel 718 SEM particle analysis for 45-53 μm sample.	31
Figure 3-4: Al2024 SEM micrographs of the particle size 20-25 μm	31
Figure 3-5: EBSD micrograph Al2024 polished 20-25 μm particle.	32
Figure 3-6: Tube furnace heat treatment setup for Inconel 718.	33
Figure 3-7: Time-Temperature-Transformation diagram for Inconel 718.	34
Figure 3-8: Time-Temperature-Transformation diagram for Al2024.	34

Figure 3-9: Phillips PW 3020 X’Pert diffractometer located at Tufts University.	37
Figure 3-10: PANalytical diffractometer located at Massachusetts Institute of Technology.	38
Figure 3-11: Synchrotron XRD located at the Argonne National Laboratory.....	39
Figure 3-12: Stoe STADI P transmission powder diffractometer located at IFW Dresden.	39
Figure 3-13: Various samples prepared for X-ray diffraction.	41
Figure 4-1: Schematic of logical flow of data analysis methods used in the study.....	42
Figure 4-2: Flow chart of the X-ray diffraction line profile analysis method.....	43
Figure 4-3: Calibration scans for various radiation energies plotted versus theta and wave vector.	44
Figure 4-4: Voigt, Gaussian, and Lorentzian curve fit applied to a Bragg peak.	46
Figure 4-5: Schematic illustrating full-width-half-maximum determination from Bragg peak.....	47
Figure 4-6: Voigt fit to Bragg peak of Fe-Al 200 μm diameter sample.	48
Figure 4-7: Calibration full-range diffractogram of lanthanum hexaboride sample.	49
Figure 4-8: Peak position calibrations for three experiments using the PANalytical diffractometer.	50
Figure 4-9: Cu-K α doublet example using the PANalytical diffractometer.	51
Figure 4-10: Peak breadth calibrations for three experiments using the PANalytical diffractometer.	53
Figure 4-11: Conventional Williamson-Hall plot for Inconel 718 particle size 20-25 μm sample.....	56
Figure 4-12: Modified Williamson-Hall plot for Inconel 718 particle size 20-25 μm sample.	58
Figure 4-13: Continuous peak functions for Inconel 718 particle size 20-25 μm sample.	59
Figure 4-14: Fourier transformation and quadratic fit for Inconel 718 particle size 20-25 μm sample.....	60
Figure 4-15: Dislocation density determination for Inconel 718 particle size 20-25 μm sample.	61
Figure 4-16: Local misorientation angle mapping for Al2024 particle size 53-63 μm sample.	62
Figure 4-17: Schematic illustrating misorientation angle calculation.....	63
Figure 4-18: Relative frequency distribution for local misorientation angle example.....	64
Figure 4-19: Lognormal fit to misorientation angle data example.....	64
Figure 4-20: Simplified illustration of GNDs preserving continuity throughout the lattice.	66
Figure 4-21: Estimated cooling rates for Inconel 718, Al2024, and Fe-Al displayed on a log-log plot.	72

Figure 4-22: Metallography polishing and etching process.	75
Figure 4-23: SEM cell size determination for Al2024 particle size 38-45 μm sample.	76
Figure 4-24: Cell characterization for Al2024 particle 20-25 μm size sample.	77
Figure 4-25: EBSD IPF (Y0 orientation) of Al2024 particle 53-63 μm size sample.	77
Figure 5-1: Inconel 718 particle 53-63 μm size repeated low-resolution diffractograms.	79
Figure 5-2: Inconel 718 45-53 μm particle size high-resolution diffractogram and peak fit.	80
Figure 5-3: Inconel 718 low-resolution diffraction scan for as-atomized and annealed samples.	81
Figure 5-4: Inconel 718 high-resolution diffraction scan for as-atomized and annealed samples.	81
Figure 5-5: Comparison of PANalytical and Phillips diffractometers using calibration scan.	82
Figure 5-6: Inconel 718 as-atomized dislocation density results using two different diffractometers.	84
Figure 5-7: Inconel 718 powder 20-25 μm and 53-63 μm particle size EBSD local misorientation.	85
Figure 5-8: Inconel 718 model-based cooling rate prediction for all particle sizes.	87
Figure 5-9: Inconel 718 predicted cooling rate results using cell size for all particle sizes.	87
Figure 5-10: Inconel 718 dislocation density as a function of predicted cooling rate.	88
Figure 5-11: Al2024 low-resolution diffraction scan for as-atomized and annealed samples.	89
Figure 5-12: Al2024 powder synchrotron diffraction results showing full-range diffractograms.	90
Figure 5-13: Al2024 powder 20-25 μm particle size Voigt curve fit to Bragg peak.	91
Figure 5-14: Al2024 powder 20-25 μm particle size Modified Williamson-Hall plot.	91
Figure 5-15: Al2024 powder 20-25 μm particle size Modified Warren-Averbach plot.	92
Figure 5-16: Al2024 as-atomized and annealed powder dislocation density results for all particle sizes. .	93
Figure 5-17: Al2024 particle size 20-25 μm , 38-45 μm and 53-63 μm EBSD and local misorientations. .	94
Figure 5-18: Al2024 annealed particle size and local misorientation results.	95
Figure 5-19: Al2024 EBSD local misorientation angle results with varying Kernal radius.	96
Figure 5-20: Al2024 model-based cooling rate prediction for all particle sizes.	98
Figure 5-21: Al2024 cooling rate predictions using published empirical relationships.	98
Figure 5-22: Al2024 cooling rate prediction compared to best-fit literature empirical relationship.	99

Figure 5-23: Al2024 dislocation density vs. cooling rate displayed on a semi-log plot.	100
Figure 5-24: Fe-Al powder 200 μm particle size low-resolution diffractogram.	102
Figure 5-25: Fe-Al and Al2024 Bragg peak width comparison for all particle sizes.	102
Figure 5-26: Simplified schematic of artificial broadening due to particle size.	103
Figure 5-27: Speculative relationship between variables affecting artificial peak broadening.	104
Figure 5-28: Fe-Al dislocation density results for all particle sizes.	105
Figure 5-29: Al2024 and Inconel 718 as-atomized samples dislocation density comparison.	107
Figure 5-30: Al2024 and Inconel 718 comparison of normalized misorientation angles.	107
Figure 5-31: Dislocation density results for all materials displayed on a log-log plot.	108
Figure 5-32: Comparison of observed dislocation density trends to AlCrFe ₂ Ni ₂ alloy.	110
Figure 5-33: SEM micrograph at 1000X magnification showing satellite formation.	111
Figure 5-34: The powder sieving process showing trapped powder.	111
Figure 5-35: EDX spectroscopy micrograph for an annealed Al2024 with Al ₂ CuMg precipitates.	112

List of Tables

TABLE I: Inconel 718 Chemical Composition.....	27
TABLE II: Al2024 Chemical Composition.....	28
TABLE III: Al2024 Powder Size Distribution.....	28
TABLE IV: X-Ray Diffraction Test Sequence.....	36
TABLE V: Comparison of Curve Fitting Functions	46
TABLE VI: Voigt Fit Parameters for X-Ray Diffraction Data.....	48
TABLE VII: Dimensionless Quantities Utilized in Cooling Rate Calculation	68
TABLE VIII: Published Material Properties Utilized in Cooling Rate Calculation	71
TABLE IX: Published Gas Properties Utilized in Cooling Rate Calculation	72
TABLE X: Published Relationships for Cooling Rate using Dendrite Arm Spacing	74
TABLE XI: Inconel 718 Expected Diffraction Angle Calculations.....	79
TABLE XII: Inconel 718 Observed Diffraction Angles	79
TABLE XIII: Comparison of Diffractometer Performance Metrics	83
TABLE XIV: Inconel 718 Dislocation Density Results.....	83
TABLE XV: Inconel 718 EBSD Misorientation Angle Results.....	85
TABLE XVI: Inconel 718 Calculated Cooling Rate and Apparent Cell Size.....	86
TABLE XVII: Al2024 Dislocation Density Results	92
TABLE XVIII: Al2024 EBSD Misorientation Angle Results	94
TABLE XIX: Annealed Al2024 EBSD Misorientation Angle Result.....	95
TABLE XX: Al2024 EBSD GND Density Approximation	96
TABLE XXI: Al2024 Calculated Cooling Rate and Apparent Cell Size	97
TABLE XXII: Fe-Al Calculated Cooling Rate	106
TABLE XXIII: Dislocation Density Literature Review	109

List of Appendices

A. Material and Equipment Specifications	129
B. Publications	149
C. Conferences.....	150
D. Software Code.....	151
E. Dislocation Density Analysis Guide	153

1 Introduction

In a world where metal parts are all around us, studying metal solidification is an inherent necessity: it affects the buildings we live in, the cars we drive, and the spacecraft we launch. Understanding solidification is imperative to guarantee the quality, performance, and safety of metal components across diverse applications, ranging from aerospace and automotive to energy sectors and more. Continued investigation into solidification will improve current manufacturing and fabrication processes, including casting, welding, and additive manufacturing. Armed with a better understanding of how solidification influences microstructure formation, industrial processes can be tuned to manufacture improved parts with predetermined material properties.

The microstructure of the material, representing its physical structure at a microscopic size scale, forms and transforms during the solidification process. Understanding this microstructural evolution is crucial to controlling microscale structures that dictate material properties at the macroscale [1]. Therefore, advancements in computational modeling for metal solidification have the potential to yield significant savings in both time and resources across various industries [2]. This study aims to further identify and quantify the relationship between material solidification history and dislocation density, a crucial microstructural feature controlling macroscale properties such as strength, ductility, and toughness.

There is an extensive amount of existing research on dislocation density in metals. However, nearly all studies have been predominantly conducted on bulk samples [3]–[7], such as tensile specimens, pucks, and sheets rather than metal powders. Existing studies primarily focus on larger test specimens with slower cooling rates during solidification, leaving metallic powders with relatively higher cooling rates largely uninvestigated. With the rapid emergence of additive manufacturing (AM), an industrial manufacturing technique that has seen a tripling in market share

over the last five years [8] and shows no signs of slowing, understanding the solidification of metal powders used in AM has become a new niche of the solidification field. This gap in the literature regarding the understanding of dislocation density in powders subjected to high solidification rates highlights the need for further investigation in this area.

While the trends investigated in this study can be broadly applied to a larger field of materials, three representative materials were selected: Inconel 718 (UNS N07718), Aluminum Alloy 2024 (UNS A92024), and a binary alloy Fe-Al (92% iron and 8% aluminum, by weight). It should be noted that the first two alloys can be identified by their UNS number (Unified Numbering System for Metals and Alloys). In contrast, the third alloy was produced by our academic collaborators at the Department of Chemical and Materials Engineering at the University of Alberta. Inconel 718, a nickel-chromium-based superalloy, is one of the most commonly employed alloys for metal additive manufacturing and has a wide range of high-temperature applications in the aerospace and energy industries [9]. Al2024, an alloy composed of primarily aluminum and copper, has been used for several decades in the aerospace and automotive industries due to its relatively high strength and low weight [10]. The Fe-Al alloy has no distinct commercial application; it was used as an easily reproducible and simple model material in this study.

The typical industrial manufacturing process of metallic powders used in additive manufacturing produces a powder in which the diameters of individual particles vary among the entire population. According to the governing thermodynamics, variations in particle size result in differences in cooling rates and solidification histories due to differences in surface-to-volume ratios. Smaller particles have a comparatively higher surface area to dissipate heat relative to their thermal mass retaining that heat energy, meaning that smaller particles will have a higher cooling rate than larger particles. While the variation in solidification histories of the samples may appear

to pose a challenge to this study correlating solidification history with dislocation density, this variability was strategically utilized. A spectrum of solidification history subgroups within each sample material was analyzed by separating the entire sample into specific particle size ranges. This technique allowed for identifying trends between dislocation density and differing solidification history. The experimental samples were annealed to determine that differences in dislocation density should be attributed to microstructural variations rather than differences in particle size.

This study employed X-ray diffraction (XRD) and electron backscatter diffraction (EBSD) to analyze the dislocation densities of the three model materials experimentally. Dislocations of atoms in the otherwise ordered crystal lattice structure create microstrain due to the manifestation of electromagnetic forces acting upon the surrounding atoms [11]. The dislocation density can be determined by the measurement of diffracted X-rays interacting with this periodic arrangement of atoms in the crystalline sample. Initial experimentation began utilizing standard Cu-K α radiation X-ray diffraction instruments and eventually evolved to utilizing synchrotron X-ray diffraction, which utilizes a particle accelerator to generate radiation. In addition to XRD measurements, EBSD techniques were used to approximate dislocation density trends. The average misorientation angle between neighboring areas of the surface of the particle can be determined through EBSD, which is related to dislocation density. Results from the XRD analysis will be compared to the approximations from the EBSD analysis.

This investigation into the relationship between dislocation density and solidification history within metals addresses current gaps in published literature and provides valuable data trends to industry. Subsequent chapters of this dissertation explain the experimental methodologies, empirical data analysis methods, and overarching conclusions from this study.

This dissertation consists of the following chapters:

- **Chapter 1, Introduction:** This Chapter identifies the unmet needs and industrial application motivating this research, establishes a gap in published knowledge in this field, and provides background information guiding the experimental flow of this study.
- **Chapter 2, Background:** This Chapter provides essential context and foundational knowledge of the theories and principles relevant to the experiments.
- **Chapter 3, Experimental Methods:** This Chapter documents the sample preparation and experimental methods utilized to obtain data.
- **Chapter 4, Data Analysis Methods:** This Chapter describes the data analysis techniques used to analyze the collected data.
- **Chapter 5, Results and Discussion:** This Chapter presents the results of the data analysis and provides further interpretation and discussion. It compares the results of the two main analysis techniques, XRD and EBSD. Additionally, error analysis, assumptions, and limitations are discussed.
- **Chapter 6, Conclusions and Future Work:** This Chapter summarizes the key conclusions identified by this series of experiments and proposes possibilities for future work.

Aims

This research addresses the following key scientific questions:

1. How does solidification history influence microstructural evolution?
2. Can a relationship between cooling rate and dislocation density be established in the high cooling rate regime of metal powders?
3. If such a relationship exists, is it consistent across different materials?

Objectives

This work is structured to achieve the following objectives:

1. Analyze dislocation densities in Inconel 718, Aluminum Alloy 2024, and Fe-Al alloy powders across various solidification histories.
2. Validate resulting dislocation density trends using the average local misorientation angle.
3. Correlate sample particle size with cooling rates using a thermodynamic model anchored with metallographic observations.

Approach

To meet these objectives, the study includes the following tasks:

1. Isolate particle size ranges for metallic powders using sieving techniques and validate the method using scanning electron microscopy.
2. Apply a thermodynamic model to predict cooling rates based on particle size and validate the model using cell size observations.
3. Conduct X-ray diffraction experiments and validate trends using EBSD analysis.

Significance

Achieving these aims will result in:

1. Advanced understanding of how solidification history affects dislocation density.
2. A comprehensive methodology for using XRD and EBSD in dislocation density analysis.
3. Insights for industrial applications, enhancing the quality and reliability of manufactured parts by tailoring solidification conditions to achieve desired mechanical properties.

2 Background

This chapter provides essential background information to understand the materials, experiments, and results of this study. The industrial significance and production method of each material used in this study are described. The solidification theory of covering the thermodynamics governing solidification and resulting microstructures is explained. Crystallography is then addressed to detail the crystal formations and defects. The chapter also outlines the framework for X-ray diffraction and scanning electron microscopy techniques.

2.1 Materials

This study utilizes Inconel 718, aluminum alloy 2024, and a binary iron-aluminum alloy. Inconel 718 and aluminum alloy 2024 are produced using conventional gas atomization techniques, while the binary iron-aluminum alloy is manufactured using impulse atomization techniques.

2.1.1 Inconel 718

Inconel 718 is one of the top-performing alloys in the Inconel family, developed by the International Nickel Company, excelling in high-temperature, high-stress environments. The alloy is composed of primarily nickel, iron, and chromium (nearly 90%, by weight, similar to austenitic steels); additions of niobium, titanium, and aluminum strengthen the primary nickel-based γ phase through the formation of precipitates ($\text{Ni}_3(\text{Al,Ti})$) γ' phase and Ni_3Nb γ'' phases) [12][15][13]. In recent years, the popularity of Inconel 718 has continued to rise due to the ability to be additively manufactured through processes such as powder bed fusion (PBF), selective laser melting (SLM), and directed energy deposition (DED) [14]. The combination of excellent material properties and ability to be additively manufactured is particularly attractive to leading aerospace facilities, such

as NASA Marshall Space Flight Center (MSFC) [15], where high-strength parts with complex geometry must be manufactured at a low production scale. Further investigation of the relationship between solidification history and resulting material properties using additive manufacturing powders will enable industries using Inconel 718 to improve the quality of manufactured parts.

2.1.2 Aluminum Alloy 2024

Aluminum 2024 (Al2024) excels in applications requiring a high strength-to-weight ratio, offering a high strength and significantly lower density than alloys like Inconel 718. Aluminum 2024 is a member of the 2000 series of aluminum alloys, which are characterized by the alloying element copper. This alloy is composed of primarily aluminum (90-95%, by weight), additions of copper (4.5%) and magnesium (1.6%) strengthen the aluminum-based α phase through the formation of precipitates (Al_2Cu θ phase and Al_2CuMg S phase) [15][16]. With the rise of additive manufacturing, Aluminum 2024 has also gained prominence, similar to Inconel 718, due to its suitability for high-performance applications.

2.1.3 Iron-Aluminum Alloy

The binary iron-aluminum (Fe-Al) alloy used in this series of experiments differs from the previous two alloys in that it currently lacks industrial applications and was specifically produced in the lab using impulse atomization techniques for experimental purposes. While the class of ferroaluminum alloys does exist, they typically contain an even mix of iron and aluminum by weight, whereas this alloy contains 92% iron by weight. Evaluating the phase diagram [17] in Figure 2-1, this alloy is expected to form a purely iron-based α phase during solidification. While this alloy does not offer significant industrial applications due to the lack of additional alloying elements, it does serve as an ideal model material due to its simplicity.

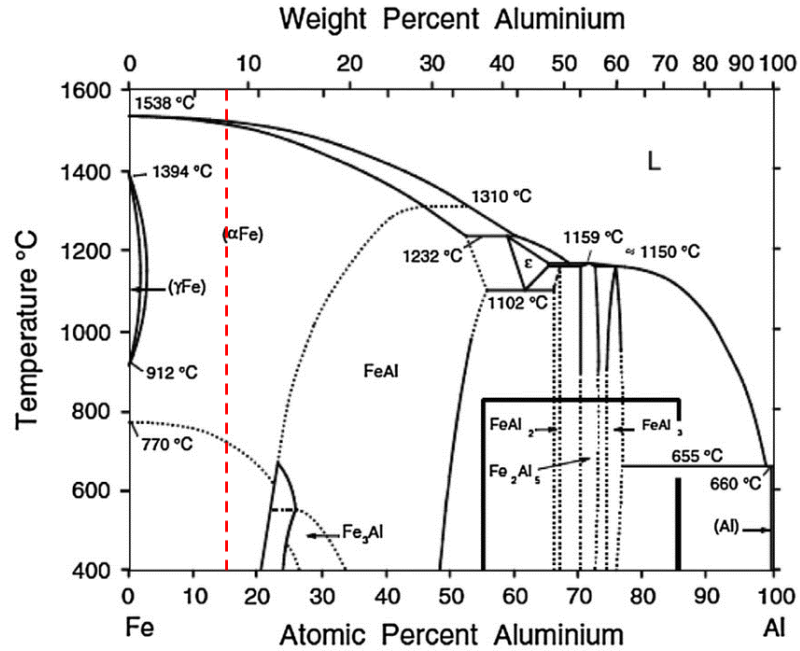


Figure 2-1: Iron-aluminum binary phase diagram with 92% Fe 8% Al composition highlighted.

This study seeks to establish a general relationship between solidification history and resulting microstructure that can be broadly applied to various solidification processes beyond the three model materials examined.

2.1.4 Atomization Processes

The samples investigated in this study were produced through atomization processes, wherein a molten metal stream is split into tiny droplets that rapidly solidify. Gas atomization is a commonly used industrial process for producing metallic powders, especially for additive manufacturing. This method involves a high-velocity inert gas breaking up a continuous stream of molten metal. In contrast, impulse atomization [18] is not an industrial technique but rather an experimental apparatus used to study solidification phenomena. This technique utilizes an oscillating impulse applicator to expel discrete molten metal streams through a nozzle plate with known-size orifices. These small streams then break down into droplets through Rayleigh

instability [18]. Impulse atomization offers superior control over powder particle size and the corresponding cooling rate compared to spray forming techniques. Schematics of both atomization processes are provided in Figure 2-2. The Inconel 718 and Al2024 samples used in this experiment were produced using gas atomization, while the Fe-Al samples were made using impulse atomization.

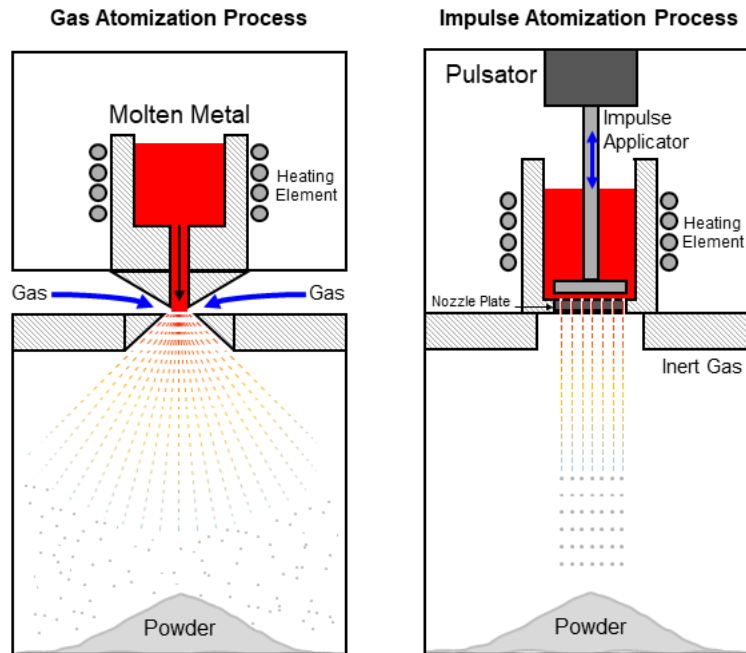


Figure 2-2: Schematic of gas and impulse atomization processes used to produce the samples.

2.2 Solidification

This section will describe the solidification theory governing the atomization process used to produce the samples in this study. The transition from liquid to solid will be analyzed within the context of solidification theory, including the derivation of an energy balance to predict cooling rates and the resulting microstructure of the solidified particles.

2.2.1 Solidification Theory

Put simply, solidification is the phase change from liquid to solid. This change is characterized by the formulation of the solid phase. In metal alloys (aside from the eutectic composition), a gap exists between the solid and liquid phases, known as the "mushy zone." This gap is defined by the liquidus temperature, T_L , above which the material is entirely liquid, and the solidus temperature, T_S , below which the material is completely solid. As a liquid is cooled below the liquidus temperature, energy begins to build, and solidification begins through a nucleation process defined by the classical nucleation theory (CNT) [19].

Nucleation can occur through one of two mechanisms: heterogeneous nucleation or homogenous nucleation. Heterogeneous nucleation is a lower energy pathway that occurs when a foreign particle (impurity, casting wall, etc.) allows the solidification to occur. Meanwhile, homogeneous nucleation is a higher energy pathway where no specific site for solidification exists. In this case, the random motion of atoms will generate a higher-density zone that provides a simultaneous site for nucleation [20]. Homogenous nucleation is significantly more challenging to study because the liquid cannot be in contact with anything around it - this method of study is known as "containerless processing." Methods such as electrostatic levitation (ESL) and electromagnetic levitation (EML) are among the techniques being used to study homogeneous nucleation. Ground-based ESL work is being conducted at facilities such as NASA Marshall Space Flight Center [21], and space-based EML work is being conducted by astronauts aboard the International Space Station [22], as shown in Figure 2-3. Homogeneous nucleation also applies to atomization processes that manufacture additive manufacturing powders since the particles are solidified while in flight.

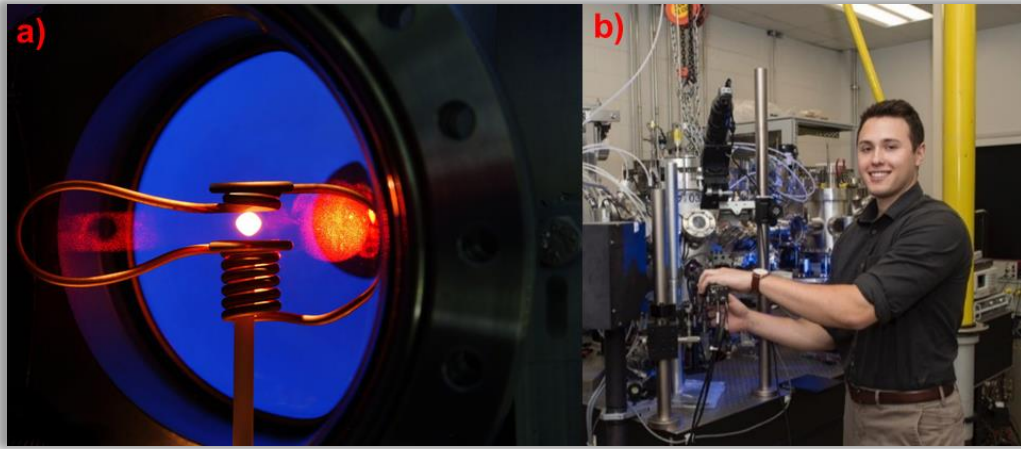


Figure 2-3: Equipment used to conduct containerless processing research.

- a) space-based electromagnetic levitation aboard the ISS,
- b) ground-based electrostatic levitation at NASA MSFC.

The cooling rate is a crucial element in the solidification process, as it significantly impacts the resulting microstructure. This parameter can be controlled during production, making it a key factor in material design. The cooling rate can be accurately defined by applying the laws of thermodynamics. Understanding the heat energy balance in this context is essential for predicting and managing the solidification process.

2.2.2 Heat Balance

This section will define a general form of the heat transfer equation for a spherical particle representing a single particle of the additive manufacturing powder used in this series of experiments.

The law of conservation of energy states that energy cannot be created or destroyed, only transferred from one form to another. The first law of thermodynamics describes this general relationship for a system:

$$\Delta U = Q - W \quad (2.1)$$

where ΔU is the change in the internal energy of a system, Q is the heat **added** to a system, and W is the work done by the system.

The work term is negligible in this scenario, so this relationship becomes:

$$\Delta U = Q \quad (2.2)$$

The internal heat energy of a particle is related to temperature:

$$\Delta U = mC_p\Delta T \quad (2.3)$$

where m is the mass of the particle, C_p is the specific heat capacity, and ΔT is the change in temperature.

To express this equation in terms of particle size, the mass of the particle can be related to volume and density:

$$m = \rho V \quad (2.4)$$

where ρ is the density of the particle and V is the volume.

$$\Delta U = \rho V C_p \Delta T \quad (2.5)$$

Assuming the particle is spherical:

$$V = \frac{\pi D^3}{6} \quad (2.6)$$

where D is the diameter of the particle.

$$\Delta U = \frac{\rho \pi D^3 C_p}{6} \Delta T \quad (2.7)$$

Applying the first law of thermodynamics (law of conservation of energy), energy is conserved throughout the solidification process. Therefore, the rate of this energy leaving and entering the system is equal at any point in time. The rate of change is equivalent to taking the derivative with respect to time:

$$\frac{dU}{dt} = - \frac{\rho \pi D^3 C_p}{6} \frac{dT}{dt} \quad (2.8)$$

where $\frac{dT}{dt}$ is the rate of change of the temperature; to adhere to the proper convention, this term would be negative for “cooling” rate.

Equating this term to the rate of heat transfer yields:

$$-\frac{\rho\pi D^3 C_p}{6} \frac{dT}{dt} = \frac{dQ}{dt} \quad (2.9)$$

The heat transfer can be expressed as a sum of the three mechanisms of heat exchange: conduction, convection, and radiation.

$$-\frac{\rho\pi D^3 C_p}{6} \frac{dT}{dt} = \frac{dQ_{conduction}}{dt} + \frac{dQ_{convection}}{dt} + \frac{dQ_{radiation}}{dt} \quad (2.10)$$

Fourier's law describes heat transfer through conduction:

$$\frac{dQ_{conduction}}{dt} = kA \frac{dT}{dr} \quad (2.11)$$

where k is the thermal conductivity of the particle, A is the surface area of the particle, dT/dr is the temperature gradient (evaluated at the surface, $r = R$).

Newton's law of cooling describes heat transfer through convection:

$$\frac{dQ_{convection}}{dt} = hA(T - T_{\infty}) \quad (2.12)$$

where h is the convective heat transfer coefficient, A is the surface area of the particle, and T_{∞} is the ambient temperature.

Heat transfer through radiation is described by the Stefan-Boltzmann law:

$$\frac{dQ_{radiation}}{dt} = \sigma \varepsilon A (T^4 - T_{\infty}^4) \quad (2.13)$$

where σ is the Stefan-Boltzmann constant ($\sigma = 5.67037442 \times 10^{-8} \text{ kg s}^{-3} \text{ K}^{-4}$) and ε is the emissivity.

Assuming the particle is spherical:

$$A = \pi D^2 \quad (2.14)$$

$$-\frac{\rho\pi D^3 C_p}{6} \frac{dT}{dt} = k\pi D^2 \frac{dT}{dr} + h\pi D^2 (T - T_{\infty}) + \sigma \varepsilon \pi D^2 (T^4 - T_{\infty}^4) \quad (2.15)$$

A schematic for this example is shown in Figure 2-4.

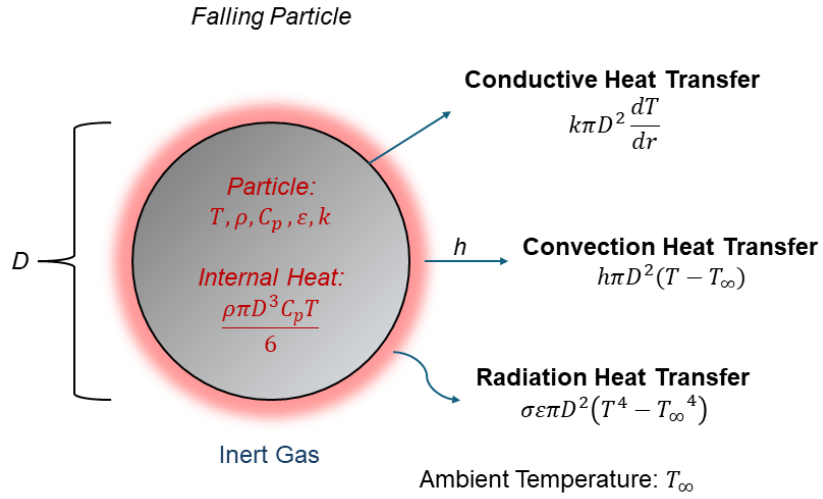


Figure 2-4: Heat transfer mechanisms in a falling particle.

Simplifying and solving for cooling rate as a function of particle diameter:

$$\frac{dT}{dt}(D) = -\frac{6}{\rho D C_p} \left(k \frac{dT}{dr} + h(T - T_\infty) + \sigma\epsilon(T^4 - T_\infty^4) \right) \quad (2.16)$$

While it may appear that we are now well-equipped to calculate cooling rate as a function of particle diameter at this point, that is unfortunately *not* the case. Specifically, the coefficients used in Equation (2.16) are more complex functions dependent upon solidification conditions. Additionally, this model is also dependent on the regime of solidification for which the material constants are evaluated. In this work, material constants are evaluated in the transition solidification regime rather than the liquid or solid phases. The evaluation of Equation (2.16) for conditions representative of the samples used in this work is provided in Section 4.3.1.

2.2.3 Microstructural Evolution

The structures formed during solidification at a microscopic scale (microstructures) are influenced by solidification conditions. Once solidification begins at a nucleation site, dendrite structures form outwards. These structures are tree-like in form, almost resembling the growth of a snowflake (which is a crystal of ice), as shown in Figure 2-5. The dendrites continue to grow in an energetically favorable manner wherein the primary arms form branching secondary arm offshoots. These dendrites grow large enough to impinge upon each other and eventually merge into cells/grains. Wherein the meeting of two dendritic structures forms the boundary between the cells. The microstructure developed during solidification influences the final mechanical properties of the solidified component [23]. In rapid solidification processes, such as those used in additive manufacturing, the cells tend to be smaller and more equiaxed due to the high nucleation rate and the limited growth time. This fine-celled structure can significantly enhance the strength and hardness of the material. Through the evaluation of secondary dendrite arm spacing, the cooling rate during solidification can be approximated using empirical relationships [24]. For rapidly solidified alloys with limited growth time, the cell size can be used in place of secondary dendrite arm spacing [25][24].

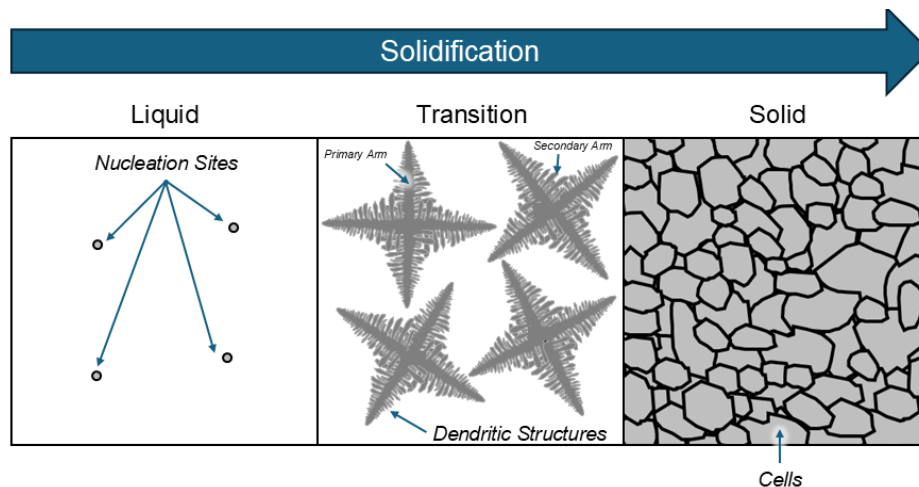


Figure 2-5: Microstructural development during homogenous nucleation.

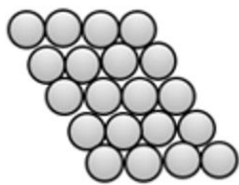
2.3 Crystallography

The cells formed during the microstructural evolution of solidification are composed of an orderly arrangement of atoms, creating a crystal structure. Crystallography is the study that defines the orientation and structure of these crystals. These crystals may include defects where atoms deviate from their expected positions. These defects can be healed by adding energy in the form of heat. In this process, recrystallization will occur where atoms reconfigure into the ordered arrangement.

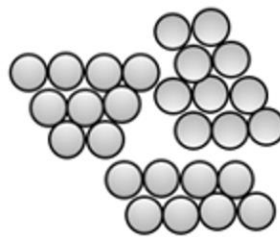
2.3.1 Crystal Structures

All solid materials can be classified by their order of crystallinity. Monocrystalline materials contain a single, perfectly ordered crystal, polycrystalline materials contain many adjoining crystals of different orientations, and amorphous materials have no discernable crystal structure, as shown in Figure 2-6. To relate crystallinity to standard materials, most gemstones are monocrystalline, most metals are polycrystalline, and most glasses are amorphous.

Monocrystalline



Polycrystalline



Amorphous

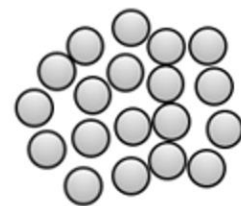


Figure 2-6: Simplified classification of crystallinity.

The arrangement of atoms defines these crystal structures, specifically by their smallest repeating unit, known as a unit cell. Examples of a visualization of a single unit cell of this repeating structure are provided in Figure 2-7. In a simple cubic arrangement, atoms are located in

each corner of the unit cell. In a body-centered cubic (BCC) arrangement, atoms are located at each corner as well as in the center (with the body-centered atom depicted in green in Figure 2-7). In a face-centered cubic (FCC) arrangement, atoms are located at each corner as well as each of the six faces (with the face-centered atoms depicted in red in Figure 2-7). Since these units are repeating, atoms on the faces and corners of the structure are shared with neighboring unit cells.

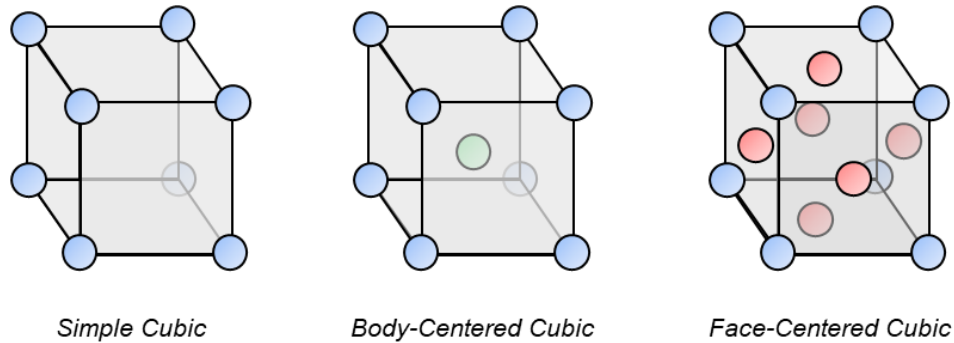


Figure 2-7: Simple cubic, body-centered cubic, and face-centered cubic atomic arrangements.

The Miller Index notation system is utilized to classify a specific plane of atoms within a crystal. They are denoted as (hkl) and represent the reciprocal of the intercepts that a plane makes with the crystallographic axes. The family of all similar planes is denoted using braces as $\{hkl\}$. The plane containing the highest atomic density is known as the slip plane because this plane requires the least amount of energy for atoms to move past each other during plastic deformation; the slip planes for BCC and FCC atomic arrangements are shown in Figure 2-8.

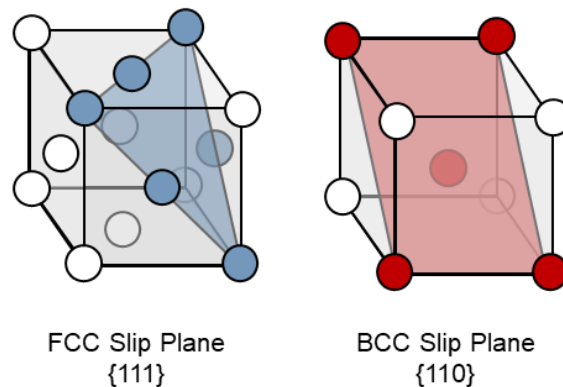


Figure 2-8: Slip planes of body-centered cubic and face-centered cubic atomic arrangements.

2.3.2 Crystal Defects

A defect may break the repeating pattern within otherwise regularly ordered crystal structures. These defects are known as atomic dislocations where an atom is dislocated from the intended position in the lattice. The two fundamental categories of line dislocations are edge dislocations and screw dislocations. Edge dislocations can be visualized as an additional half-plane of atoms wedged into the existing structure. Screw dislocations can be visualized as a helical plane shift resulting from a shearing deformation. In reality, most dislocations are a combination of both types and characterized by their edge vs. screw character. The two dislocation modes are demonstrated in Figure 2-9 with a visualization of Burgers vector (b), the “added step” caused by this location. The presence of dislocations impedes shearing deformation, which will affect the overall mechanical properties at a macroscale [26]. These defects can be observed through differences in interatomic planar spacing using X-ray diffractometry.

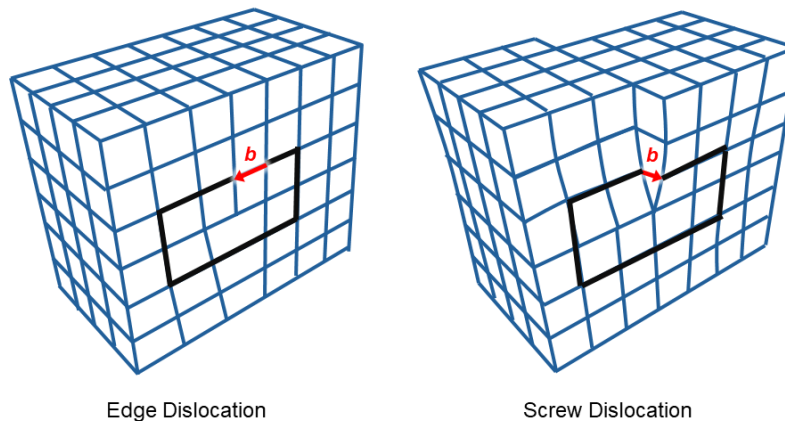


Figure 2-9: Edge and screw dislocations, demonstrating types of crystal defects.

2.3.3 Heat Treatment

Dislocations within the microstructure can be healed using heat treatment. This process involves heating and holding the material isothermally for a specific duration. The added energy

allows for atomic structure rearrangement, enabling dislocation annihilation and cell growth [27]. Heat-treated samples are expected to exhibit significantly lower dislocation density, irrespective of particle size since sufficient time is provided for dislocations to heal in all sample sizes.

2.4 X-Ray Diffraction

X-ray diffraction is a non-destructive evaluation technique utilized to characterize the crystallographic structure of materials. The governing principle of this analysis is that X-ray radiation incident to the sample will coherently scatter from planes of atoms in accordance with Bragg's Law. An example of a simplified X-ray diffraction configuration is provided in Figure 2-10. This study utilizes this technique to examine differences in interplanar spacing caused by dislocations.

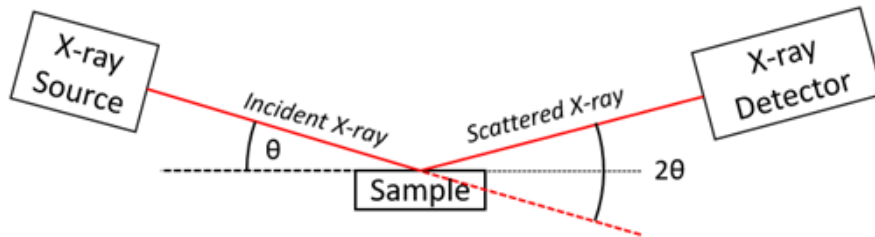


Figure 2-10: Simplified X-ray diffraction experimental configuration.

The X-ray source and detector (or the sample stage in some types of diffractometers) rotate through a range of angles. The intensity of diffracted X-rays is obtained as a function of the incident angle, θ . According to the convention, the angle measurements are recorded as 2θ , the sum of the incidence angle and scattering angle relative to the horizontal plane of the sample. Line profile analysis (LPA) of this data can be used to evaluate dislocations within the microstructure of a material [28].

2.4.1 Bragg's Law

When an X-ray strikes the atom of the sample material, it is reflected at the same incident angle and energy in a process called elastic scattering [29]. Individual crystallites in polycrystalline metals provide coherent, ordered domains for X-ray diffraction to occur [30][31]. Incident X-rays penetrate the surface of the sample material and strike atoms on many atomic planes at various depths within the crystallite.

As an X-ray propagates through space, it will complete one periodic cycle in the distance of its wavelength. Only X-rays that have traveled an integer multiple of their wavelength remain in phase with each other. Out-of-phase X-rays will destructively interfere and generate no net signal. However, at incident angles, known as Bragg angles, the total path length of X-rays striking different atomic planes is some integer of the wavelength. Therefore, all X-rays are in-phase with each other, and generate constructive interference as shown in Figure 2-11. The relationship that describes the occurrence of constructive X-ray interference in relation to interplanar atomic spacings is Bragg's Law [32]:

$$n\lambda = 2d\sin(\theta) \quad (2.17)$$

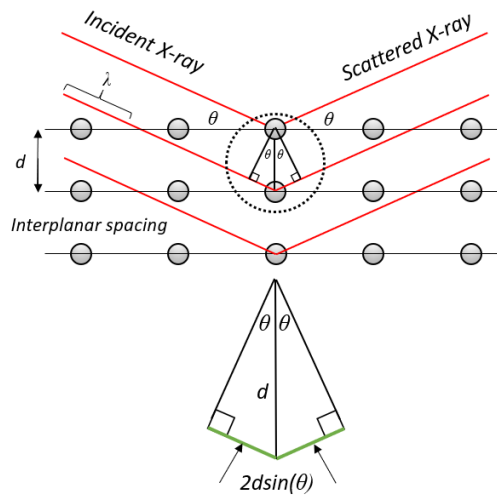


Figure 2-11: Visualization of Bragg's Law, showing how X-rays interact with atomic planes.

2.4.2 Bragg Peaks

Constructive interference of the X-rays at Bragg angles will appear as a peak in the XRD data known as a Bragg peak or diffraction peak. The transformation from the physical space in the diffractometer to the diffractogram datasets is shown in Figure 2-12.

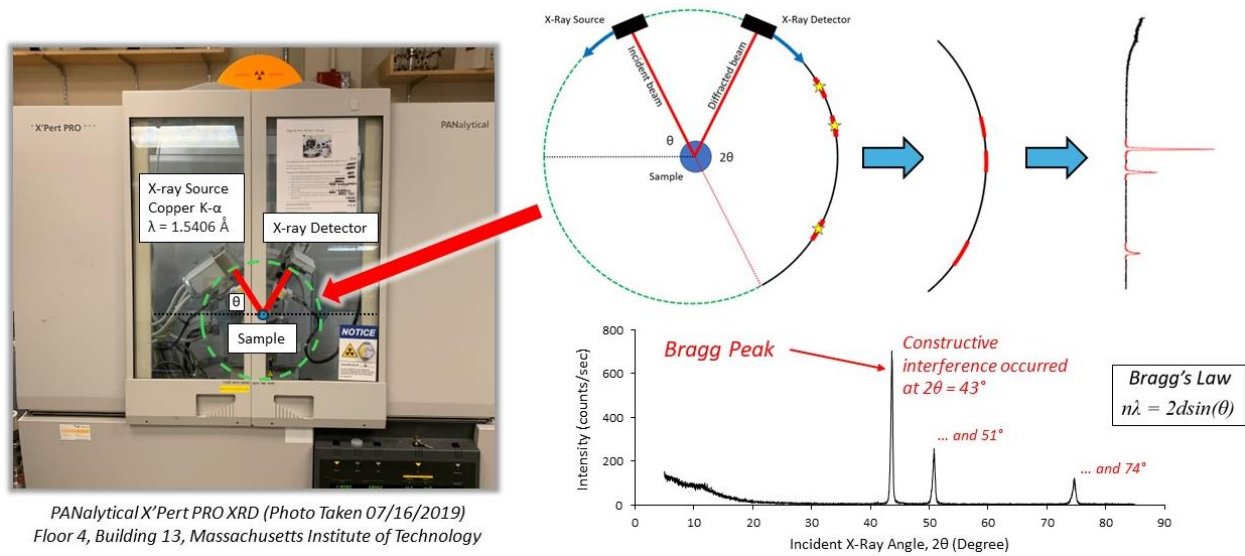


Figure 2-12: X-ray diffraction data collection and transformation into a diffractogram.

In a perfectly ordered crystal with infinite crystallite size and a perfect diffractometer, this peak would be infinitely thin, only occurring at the exact Bragg angle. However, in actual samples with definite crystallite size and atomic imperfections, a small angle range near the Bragg angle will also generate a signal. This peak will appear as some distribution around the expected Bragg angle. This study uses Bragg peak broadening and other diffraction characteristics to quantify the density of dislocations displacing atomic planes. In this study, the modified Williamson-Hall and Warren-Averbach methods [30], [33]–[38] are used to quantify line-broadening effects due to only dislocation density. While other methods, such as using the Scherrer Equation, the Size-Strain Plot Method, or Rietveld Refinement, can be utilized, the modified Williamson-Hall and Warren-Averbach methods are the most comprehensive because they allow for the separation of the

contributions to peak broadening due to crystallite size and microstrain and account for anisotropic strain effects. The specific theory governing this method is described in Section 4.1, and a comprehensive guide to conducting this analysis is provided in Appendix Section A.

2.4.3 Scattering Vector

While Bragg's Law is useful in determining interplanar spacing within a material, the incident angle for diffraction to occur, θ , is tied to the specific wavelength of radiation, λ , utilized by the diffractometer. This problem makes comparing results less intuitive between different experimental setups that use different radiation energies (and, therefore, different wavelengths). A method of wavelength-normalization can be utilized by representing angles in terms of momentum transfer of the X-ray. The scattering/wave vector, often denoted as $\vec{q}$, describes the change in momentum that an incident X-ray experiences when it is scattered by an atom, as shown in Figure 2-13 and described by Equation (2.18).

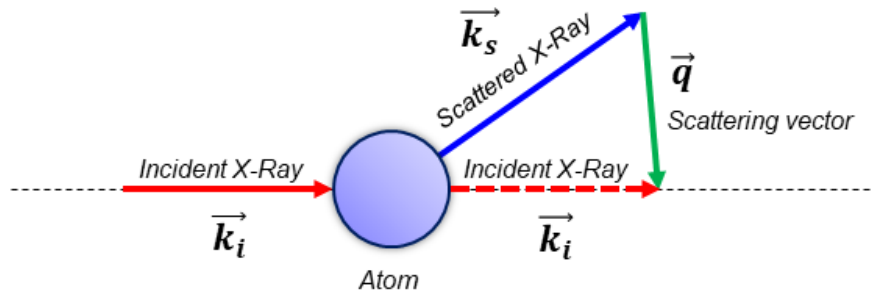


Figure 2-13: Diagram showing the representation of a scattering vector.

$$\vec{q} = \vec{k}_s - \vec{k}_i \quad (2.18)$$

When working in reciprocal/q-space, comparisons are based on intrinsic structural features of the material rather than instrument-dependent variables (such as radiation energy/wavelength). The magnitude of the scattering vector can be determined using the incident angle and X-ray radiation, as shown in Equation (2.19).

$$q = \frac{4\pi \sin(\theta)}{\lambda} \quad (2.19)$$

where q is the scattering vector, θ is the incident angle, and λ is the wavelength of radiation.

It should be noted that after applying this transformation, the associated diffractograms are now plotted as intensity vs. q (with reciprocal length units, typically nm^{-1} or $\text{\AA}^{-1}$) rather than intensity vs. incident angle (degrees).

2.5 Scanning Electron Microscopy

Scanning electron microscopy is a technique used to analyze the surface morphology, composition, and crystallographic information of materials at high resolution. SEM provides detailed images and data through various detection methods using a focused electron beam to interact with the sample. This section covers the primary SEM techniques used in this study: Secondary Electron Imaging (SEI), Energy-Dispersive X-ray Spectroscopy (EDS), and Electron Backscatter Diffraction (EBSD).

2.5.1 Secondary Electron Imaging

SEI detects secondary electrons emitted from the sample surface due to primary electron beam interaction. This technique provides a detailed visualization of surface features. This technique was utilized to analyze the particle and cell morphology of the powder samples used in this study.

2.5.2 Energy Dispersive X-ray Spectroscopy

Some scanning electron microscopes are equipped with instruments capable of Energy-Dispersive X-ray spectroscopy (EDX), a method used to determine elemental analysis and chemical characterization of materials. The physical principle governing EDX analysis is that the electron beam directed at the sample excites atoms which emit X-rays at different energy levels;

these energy levels are characteristic of the material [39]. This data was utilized to verify the composition of some samples as well as phase identification for aggregates and precipitates produced during heat treatment processes.

2.5.3 Electron Backscatter Diffraction

When an electron beam is coherently diffracted using electron backscatter diffraction, the characteristic lines observed can be utilized to determine information about the orientation of crystal planes. First observed by Seishi Kikuchi in 1928 [40], these patterns were used in this study to map the misorientation angle of crystallographic planes within samples. A schematic demonstrating the correlation between plane orientation and the subsequent Kikuchi pattern is shown in Figure 2-14.

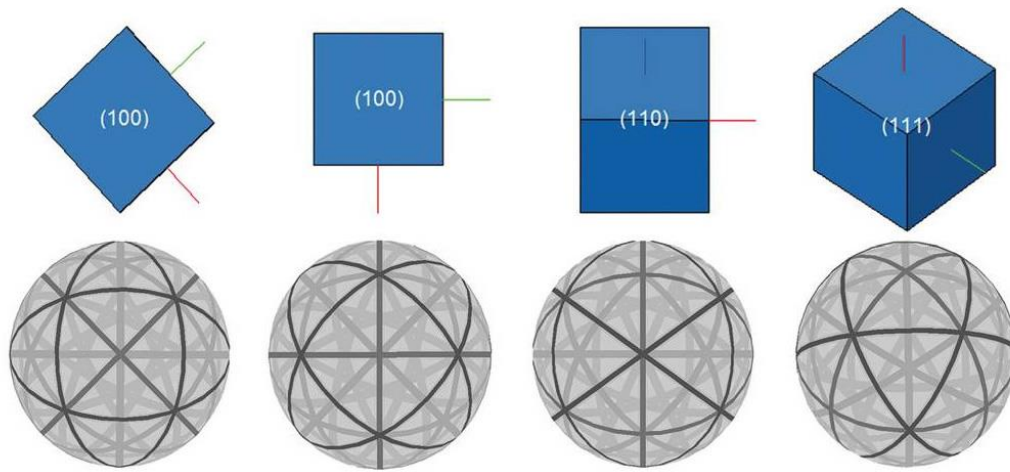


Figure 2-14: Schematic relating crystallographic orientation to EBSD Kikuchi patterns.
Image reproduced from source [41].

As the device scans across the sample surface, the detector captures Kikuchi patterns, and software is used to identify the Kikuchi bands through analysis of the intensity variations using a transformation. These bands are then correlated to corresponding crystal planes to determine the crystallographic orientation [41]. As the instrument performs a raster scan across the surface, each pixel of the resulting image is characterized based on orientation derived from the Kikuchi pattern.

An example of the raw Kikuchi pattern data for the Inconel 718 and Al2024 samples used in this study is shown in Figure 2-15.

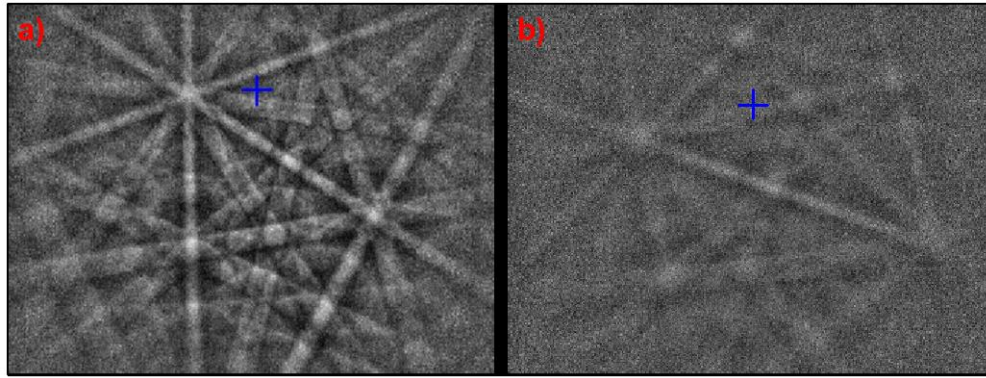


Figure 2-15: Electron backscatter diffraction Kikuchi patterns.
a) Inconel 718 and b) Al2024.

This data can be used to produce an inverse pole figure, which is a representation of the orientation of cells relative to a sample reference frame. Software maps the distribution of these orientations by coherently diffracting cells using a coloring scheme, as shown in Figure 2-16. Alternatively, at a smaller size scale, this data can be used to map the misorientation angle between neighboring pixels within a cell. Local misorientations are accommodated by geometrically necessary atomic dislocations [42]; this concept and associated analysis method is further discussed in Section 4.2.

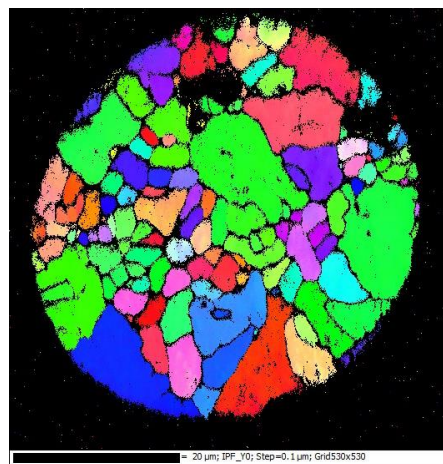


Figure 2-16: EBSD inverse pole figure for Al2024 particle size 53-63 μm sample.

3 Experimental Methods

This chapter outlines the experimental methods used to collect and analyze data on the dislocation density and solidification history of three metal powders: Inconel 718, Aluminum Alloy 2024, and the binary Fe-Al alloy. The chapter details the sample preparation procedures, including sieving and validation of particle sizes, and describes the heat treatments applied to each material. Sample preparation and operating parameters are also discussed for X-ray diffraction and electron backscatter diffraction evaluations.

3.1 Sample Preparation Procedure

The gas-atomized Inconel 718 and Al2024 material and the impulse-atomized Fe-Al material were first prepared for X-ray diffraction experiments by isolating distinct particle size ranges. A sieving technique was developed and verified for Inconel 718 additive manufacturing powder, then applied to Al2024 powder. Due to the nature of impulse atomization, the Fe-Al samples arrived at our facility already isolated by particle size. The isolated size ranges for particles of all materials were validated using image analysis and computer vision. In addition to the experimental samples, Lanthanum Hexaboride, LaB_6 , powder provided by the National Institute of Standards and Technology (NIST) was used to calibrate the X-ray diffraction facilities. Further information and material specification sheets are available in Appendix Section A.

3.1.1 Materials

The Inconel 718 additive manufacturing powder was provided by our collaborators at the additive manufacturing facility of the Materials and Processes Laboratory of the EM42 Advanced Manufacturing Branch at the National Aeronautics and Space Administration (NASA) Marshall Space Flight Center (MSFC) in Huntsville, Alabama, USA. The powder was initially purchased

from Carpenter Powder Products Inc. under the product name CarTech® 718 Alloy (UNS N07718) with applicable specifications: AMS 5662, AMS 5663, API 6A718, and NACE MR0175. The powder was estimated to have particle diameters within the range of 10 μm to 100 μm . The chemical composition is documented in TABLE I.

TABLE I: Inconel 718 Chemical Composition

Element	Weight Percentage	Element	Weight Percentage
Carbon (Maximum)	0.08 %	Manganese (Maximum)	0.35 %
Phosphorus (Maximum)	0.015 %	Sulfur (Maximum)	0.015 %
Silicon (Maximum)	0.35 %	Chromium	17.00 to 21.00 %
Molybdenum	2.80 to 3.30 %	Copper (Maximum)	0.15 %
Cobalt (Maximum)	1.00 %	Titanium	0.65 to 1.15 %
Aluminum	0.35 to 0.80 %	Columbium + Tantalum	4.75 to 5.50 %
Boron	0.001 to 0.006 %	Nickel + Cobalt	50.00 to 55.00 %
Iron	<i>Balance</i>		

The Al2024 additive manufacturing powder was provided by our industrial collaborators at Solvus Global: Technology Solutions in Worcester, Massachusetts, USA. The powder was described as a high-quality, aerospace-grade Al2024 alloy with lot number SG02.AL2024.04A.0000. Trace metal analysis was completed by Luvak Inc. to verify the composition documented in TABLE II. The material is classified as a spherical geometry powder with a compaction efficiency of 55.58% and a Hausner ratio (flowability) of 1.282. The size distribution of the powders is documented in TABLE III.

TABLE II: Al2024 Chemical Composition

Element	Weight Percentage	Element	Weight Percentage
Copper	4.19 %	Magnesium	1.59 %
Manganese	0.61 %	Oxygen	0.026 %
Silicon	0.003 %	Aluminum	<i>Balance</i>

TABLE III: Al2024 Powder Size Distribution

Cumulative Frequency	Particle Size (μm)
D10	19.30
D25	23.63
D50	31.22
D75	42.50
D90	49.04

The Fe-Al impulse atomized particles were provided by our academic collaborators at the Advanced Materials Processing Laboratory of the Chemical and Materials Engineering at the University of Alberta using their impulse atomization process [43] to produce these samples. The aluminum was sourced from CKOE materials and had a purity of at least 99.99%. The iron was sourced from ALFA materials and had a purity of at least 99.97%. The alloy was produced in an induction furnace at 10 ppm O₂ in He gas at small positive pressure.

The atomization temperature for this alloy was 1570°C. A 100-micron nozzle size was used to generate particles that fell through the helium environment into an oil quench. A pulsating impulse applicator was used to produce distinct particle sizes with diameters between 180 μm and 1180 μm , as shown in Figure 3-1.



Figure 3-1: Fe-Al samples with distinct particle size ranges between 180 μm and 1180 μm .

3.1.2 Particle Size Isolation

Inconel 718 and Al2024 powders were separated by particle size using a series of sieves with varying mesh sizes, and the fractions retained on each sieve were collected. A DUAL Manufacturing Co. Inc. Model # D-4326 sieve shaker was used with DUAL Manufacturing Co. Inc. A.S.T.M. E-11 specification sieves for roughly 15 minutes to isolate individual particle diameter ranges. The sieve shaker and respective sieve sizes are shown in Figure 3-2. Further information on the sieve ASTM E11 specification is available in Appendix Section A.

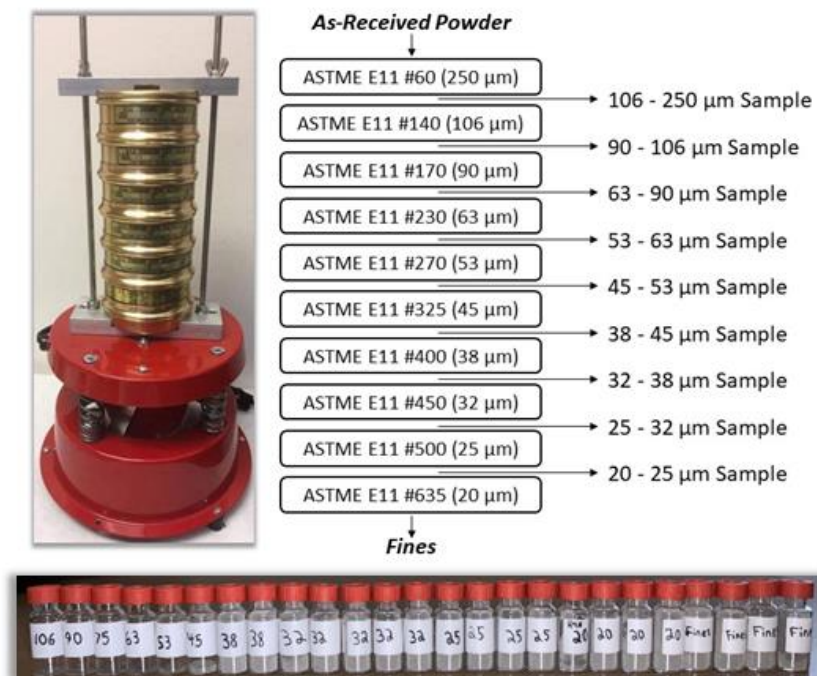


Figure 3-2: Sieve shaker with associated sieve sizes and respective isolated samples.

3.1.3 Particle Size Validation

The particle isolation process was validated for the Inconel 718 powder using scanning electron microscopy (SEM). A Phenom World Pro (PW-100-012) model 800-07334 scanning electron microscope was utilized to obtain the micrographs. Individual particles of Inconel 718 powder sample size ranges 45-53 μm and 90-106 μm were quantified and analyzed using NI Vision Assistant Software. Powder samples were sprinkled onto double-stick carbon tape, and an eye dropper was used to blow the surface of the powder to remove any un-adhered/stacked particles. Images used for analysis were captured at 140x magnification at 5kV, as shown in Figure 3-3. The analyzed particle diameter averages and standard deviations were within the expected ranges for the respective sieve sizes.

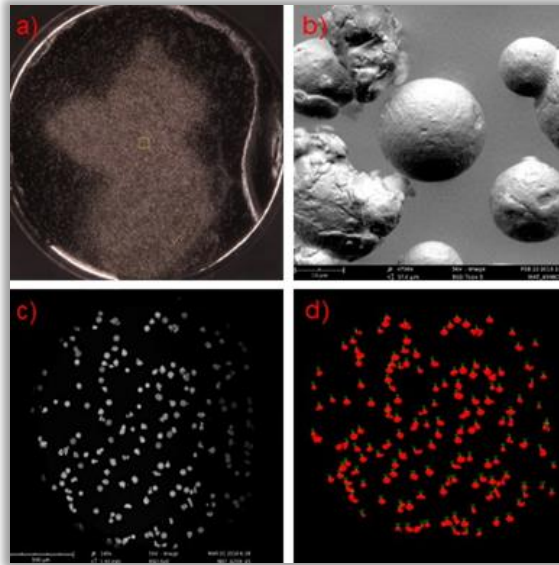


Figure 3-3: Inconel 718 SEM particle analysis for 45-53 μm sample.
a) unmagnified view, b) micrograph at 4700X magnification,
c) micrograph at 140X magnification, d) particle analysis using machine vision.

The particle isolation process was also validated for Al2024 powder using scanning electron microscopy (SEM). A JEOL Model JSM-7600F Field emission scanning electron microscope (FESEM) was utilized to obtain the micrographs, as shown in Figure 3-4. Al2024 powder particle diameter ranges of 20-25 μm , 25-32 μm , 32-38 μm , 38-45 μm , 45-53 μm , and 54-63 μm were measured. The micrographs were analyzed using ImageJ analysis software. The results for particle diameter averages and standard deviation were within the expected ranges for the respective sieve sizes.

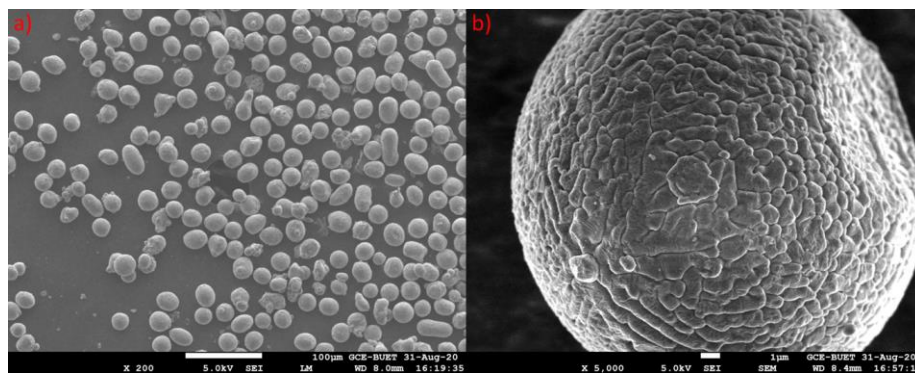


Figure 3-4: Al2024 SEM micrographs of the particle size 20-25 μm .
a) micrograph at 200X magnification, b) micrograph at 5000X magnification.

Images produced using electron backscatter diffraction also provided information regarding the particle diameters of Al2024 powder particles. These samples were prepared using a polishing process using increasingly fine abrasives to grind the away material exposing the interior of the sample. If the sample were polished to the center (leaving a half-sphere) the observed diameter would be representative of the particle. At any other polishing depth, the observed two-dimensional cross section would appear smaller than the actual particle diameter prior to polishing. Regardless, the images, with an example shown in Figure 3-5, were analyzed using ImageJ software to verify that the particles were within the expected approximate range.

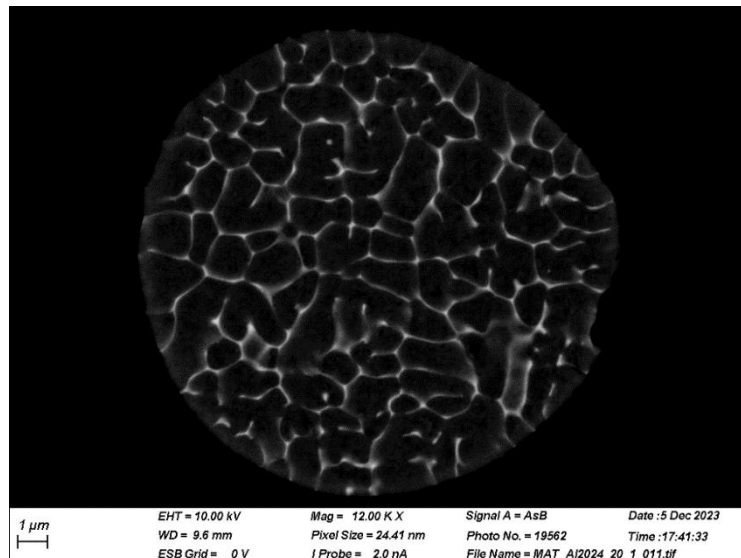


Figure 3-5: EBSD micrograph Al2024 polished 20-25 μm particle.

3.1.4 Heat Treatment

Portions of each isolated particle size range for each material were subjected to heat treatment to heal microstructural defects and provide a baseline for the dislocation density results for the as-atomized samples.

For the Inconel 718 material, the heat treatment was performed at Tufts University by loading the powder into alumina crucibles placed in a Model STF55433PC-1 Lindberg/Blue M

Furnace equipped with a Model CN310-K-C Omega Engineering Inc. Digital Temperature Controller. The heat treatment was performed in an argon environment with two different approaches: 1 hour at 1100°C and 1 hour at 1250°C, both followed by passive furnace cooling to ambient temperature. Portions of this setup are provided in Figure 3-6.

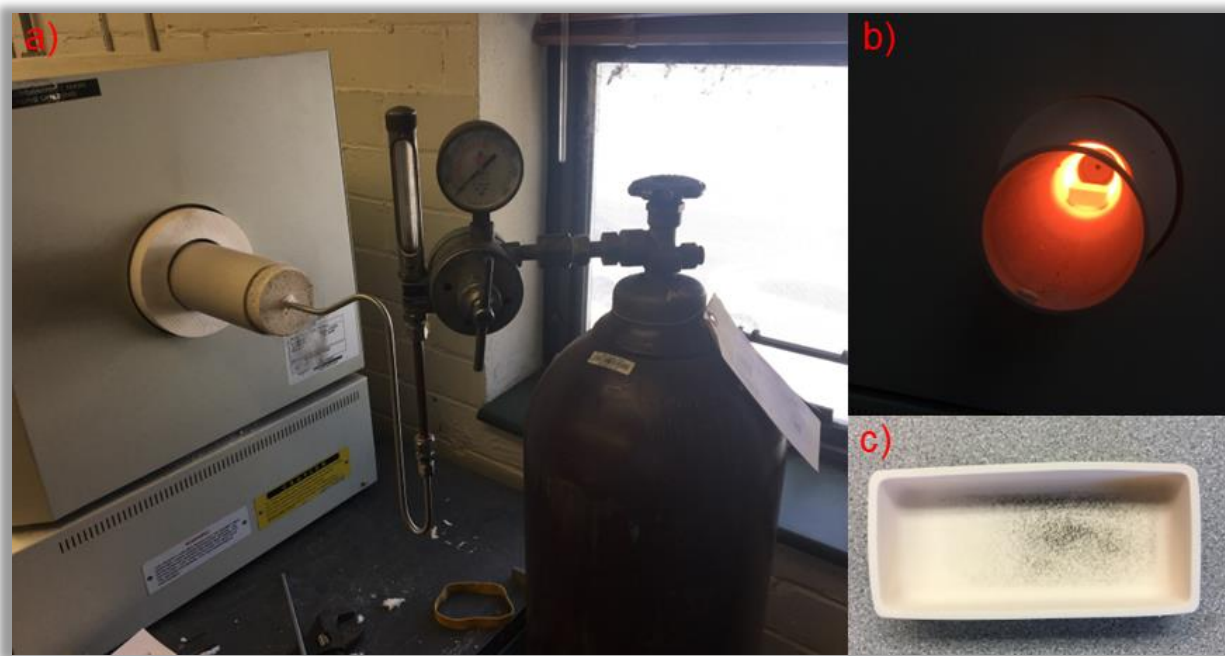


Figure 3-6: Tube furnace heat treatment setup for Inconel 718.

a) inert gas connection to the furnace, b) furnace interior with the empty crucible, c) crucible loaded with powder.

The undesired effect of significant precipitation formation occurred, as shown by the time-temperature-transformation diagram for Inconel 718 [44]. The overlaid purple line in Figure 3-7 for the 1,100 °C 1-hour heat treatment, highlights the regions where precipitates formed. This precipitate formation also occurred for the samples subjected to the 1250 °C 1-hour heat treatment. Additionally, while positive pressure was obtained for the inert gas, the presence of oxygen was not measured. These samples were likely oxidized due to imperfect sealing of the furnace environment. The ramifications of this heat treatment are discussed further in the data analysis and error sources sections.

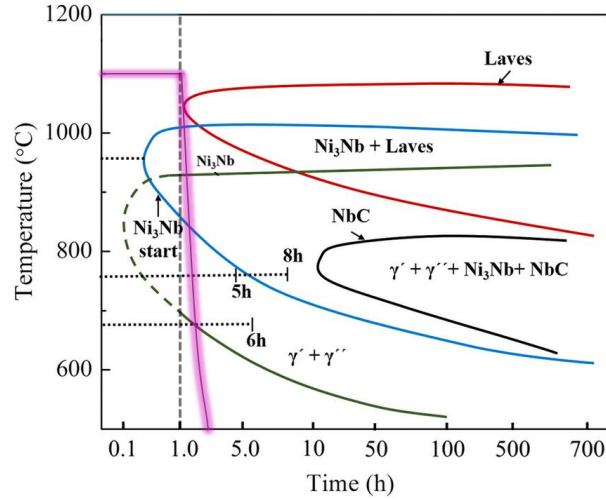


Figure 3-7: Time-Temperature-Transformation diagram for Inconel 718.

For the Al2024 material, the heat treatment was performed by our industrial collaborators at Solvus Global using the proper facilities for this work. A separate portion of this sample was annealed using an isothermal hold at 490°C for 6 hours in an argon environment. The associated time-temperature-transformation diagram for Al2024 [45]. As shown by the overlaid purple line (for the 490 °C 6-hour heat treatment) in Figure 3-8, some precipitates were formed. Despite the formation of precipitates, the Bragg peaks of the primary matrix could still be identified. The ramifications of this heat treatment are discussed further in the data analysis and error sources sections.

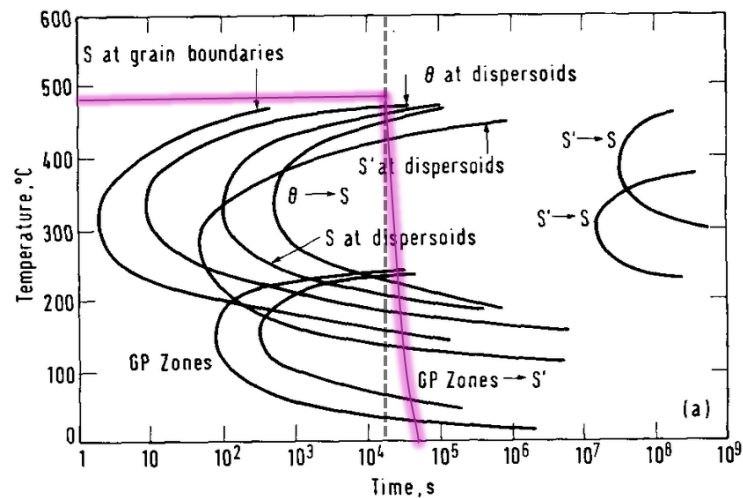


Figure 3-8: Time-Temperature-Transformation diagram for Al2024.

Our academic collaborators at the University of Alberta in Edmonton performed heat treatment for the Fe-Al material. A separate portion of this sample was heat treated using an isothermal hold at 875°C for 1 hour in a nitrogen environment. There is no published time-temperature-transformation diagram for this specific alloy, but it was not expected to form precipitants. This temperature was chosen because it is similar to heat treatments used for steel alloys, and this Fe-Al alloy is 92% iron by weight.

3.2 X-Ray Diffraction

Several X-ray diffraction facilities were used within the scope of this research, each necessitating distinct sample preparation, instrument calibration, and operational parameters.

3.2.1 X-Ray Diffraction Calibration

Before and after data collection for the test samples, lanthanum hexaboride powder was used as standard reference material to correct line position and shape alterations caused by instrumental aberrations. Specific instrumental correction and calibration data analysis methods are available in the Data Analysis Methods Sections 0 and 4.1.4.

3.2.2 X-Ray Diffraction Facilities and Test Sequence

This series of experiments occurred over many years at many different experimental facilities, as shown in TABLE IV. Initial experiments were conducted to develop a hypothesis, identify potential trends, and enhance the quality of future experiments. This preliminary work was necessary to produce proposals for subsequent studies to access facilities with superior equipment with more selective availability.

TABLE IV: X-Ray Diffraction Test Sequence

Test ID	Date	Facility	Material	Particle Diameter (μm)	Heat Treatment
1	Spring 2018	Tufts	Inconel 718	45-53	None
2	Spring 2019	Tufts	Inconel 718	45-53	None
3	Spring 2019	Tufts	Inconel 718	45-53	Annealed (1,100°C) Annealed (1,250°C)
4	Spring 2019	Tufts	Inconel 718	20-25, 38-45	None
5	Fall 2019	MIT	Inconel 718	20-25, 38-45	None
6	Spring 2020	Tufts	LaB ₆	N/A	N/A
7	Spring 2020	MIT	Al2024	20-25	None and Annealed (490°C)
8	Winter 2020	APS	Al2024	20-25, 25-32, 32-38 38-45, 45-53, 54-63	None
9	Winter 2020	APS	Al2024	20-25, 25-32, 32-38 38-45, 45-53, 54-63	Annealed (490°C)
10	Spring 2022	Tufts	Sample Holders	N/A	N/A
11*	Spring 2022	APS	Fe-Al	180-212, 425-500, 600-710	None
12*	Winter 2022	APS	Fe-Al	180-212, 355-425 500-600, 850-1000	None and Annealed (875°C)
13*	Winter 2022	BNL	Fe-Al	180-212, 355-425 500-600, 850-1000	None and Annealed (875°C)
14*	Summer 2023	DESY	Fe-Al	180-212, 355-425 500-600, 850-1000	None and Annealed (875°C)
15	Winter 2023	IFW Dresden	Fe-Al	180-212, 355-425 500-600, 850-1000	None
16	Summer 2024	DESY	Fe-Al	180-212, 355-425 500-600, 850-1000	None and Annealed (875°C)

* - Due to COVID-19, many experiment proposals were either postponed or rejected, leading to a backlog that has made the proposal process significantly more competitive.

Initial data collection began at Tufts University using the wide-angle X-ray scattering (WAXS) facility at the Cebe Research Lab, managed by Dr. Peggy Cebe, in the Tufts Collaborative Learning and Innovation Complex (CLIC) located in Medford, MA, USA. In this facility, a Phillips PW 3020 X'Pert diffractometer system was used containing PANalytical Long Focus Copper XRD X-Ray Tube Type PW2273/20 using Cu- $K\alpha$ radiation ($E = 8.04 \text{ keV}$, $\lambda = 1.5406 \text{ nm}$) and Ludlum Measurements, Inc. Model 3 General Purpose Survey Meter Part Number 48-1605, as shown in

Figure 3-9. X-ray diffraction data were obtained within the angular range (2θ) from 20° to 90° with a step size of 0.01° .

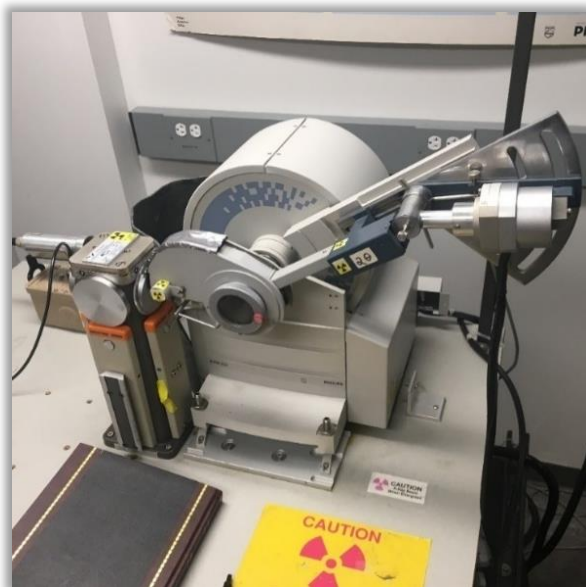


Figure 3-9: Phillips PW 3020 X'Pert diffractometer located at Tufts University.

After these initial experiments at Tufts University, another facility was identified at the Massachusetts Institute of Technology Center for Materials Science and Engineering (CMSE) Materials Research Science and Engineering Center (MRSEC), managed by Dr. Charlie Settens and located in Cambridge, MA, USA. This facility would provide data with quality sufficient to perform the advanced line profile analysis to identify dislocation density. A PANalytical X'Pert Pro MPD X-ray diffractometer using Cu- $K\alpha$ radiation ($E = 8.04 \text{ keV}$, $\lambda = 1.5406 \text{ nm}$) with an ultrafast X'Celerator RTMS detector and high-speed Bragg–Brentano optics was used, as shown in Figure 3-10. A 0.02 rad Soller slit, a divergent slit of $1/4^\circ$, an anti-scatter slit of $1/2^\circ$, and a 10 mm mask were used in the incident beam path. For the diffracted-beam side, a 0.02 rad Soller slit, a Ni filter, and an anti-scatter slit of $1/4^\circ$ were used. The X-ray generator tension and current were set at 45 kV and 40 mA , respectively. X-ray diffraction data was obtained within the angular range (2θ) from 30° to 120° with a step size of 0.008° using the X'Pert Data Collector program.



Figure 3-10: PANalytical diffractometer located at Massachusetts Institute of Technology.

Experimental data obtained from the Massachusetts Institute of Technology facility was used to generate a proposal and secure beam time at the Advanced Photon Source (APS) synchrotron X-ray facility of Argonne National Laboratory located in Lemont, IL, USA. Some Experiments for Al₂O₃ were performed at beamline 11-ID-C of the Advanced Photon Source (APS) at Argonne National Laboratory using high-energy radiation ($E = 105.7 \text{ keV}$, $\lambda = 0.01173 \text{ nm}$), as shown in Figure 3-11. The 11-ID-C beamline specifications are as follows: SCU1 source, Laue Si (311) monochromator, 5×10^{-3} resolution ($\Delta E/E$), 1×10^{11} photons/second flux, with an unfocused beam size of $0.5 \text{ mm} \times 0.5 \text{ mm}$ which can be focused to $300 \text{ }\mu\text{m} \times 2.5 \text{ }\mu\text{m}$. Two-dimensional (2D) diffraction patterns were recorded with an exposure time of 1 second in transmission mode using a Perkin-Elmer amorphous silicon-made 2D detector. The recorded diffraction patterns were azimuthally integrated using the FIT2D software.



Figure 3-11: Synchrotron XRD located at the Argonne National Laboratory.
Image reproduced from source: [46].

Verification experiments before future synchrotron X-ray diffraction work were conducted at the Leibniz Institute for Solid State and Materials Research (Leibniz-Institut für Festkörper- und Werkstoffforschung), often shortened to IFW Dresden, located in Dresden, Germany. The Stoe STADI P Transmission Powder Diffractometer, as shown in Figure 3-12, utilizes Mo- $K\alpha_1$ radiation ($E = 17.45 \text{ keV}$, $\lambda = 0.071 \text{ nm}$).



Figure 3-12: Stoe STADI P transmission powder diffractometer located at IFW Dresden.
Image reproduced from source [47].

Synchrotron X-ray diffraction experiments will also be conducted at Deutsches Elektronen-Synchrotron (DESY), a Research Centre of the Helmholtz Association located in Hamburg, Germany. Experiments will be performed using the P02.1 Powder Diffraction and Total Scattering Beamline of the PETRA III storage ring light source. This beamline uses high energy radiation ($E = 60\text{keV}$, $\lambda = 0.0207\text{ nm}$) with an unfocused beam spot size of $1.0 \times 1.0\text{ mm}^2$, which can be adjusted to $\sim 0.2 \times 0.2\text{ mm}^2$. This facility uses a Perkin Elmer XRD1621 detector with a sample-to-detector-distance (SDD) of about 2.7 m.

3.2.3 X-Ray Diffraction Sample Preparation

Each of these five X-ray diffraction facilities required the experimental samples to be prepared using different methods. For the facility at Tufts University, powder samples were mounted to acrylic slides using Kapton tape. For the Massachusetts Institute of Technology facility, powder samples were packed into the bore feature of the powder specimen holders. For the facility at Argonne National Labs, the powder samples were loaded into sealed, 1 mm thick quartz capillary sample holders. For the facility at IFW Dresden, the impulse atomized samples were loaded into powder specimen holders. For the facility at DESY, the samples were loaded into sealed quartz capillaries. The various sample preparations are shown in Figure 3-13.

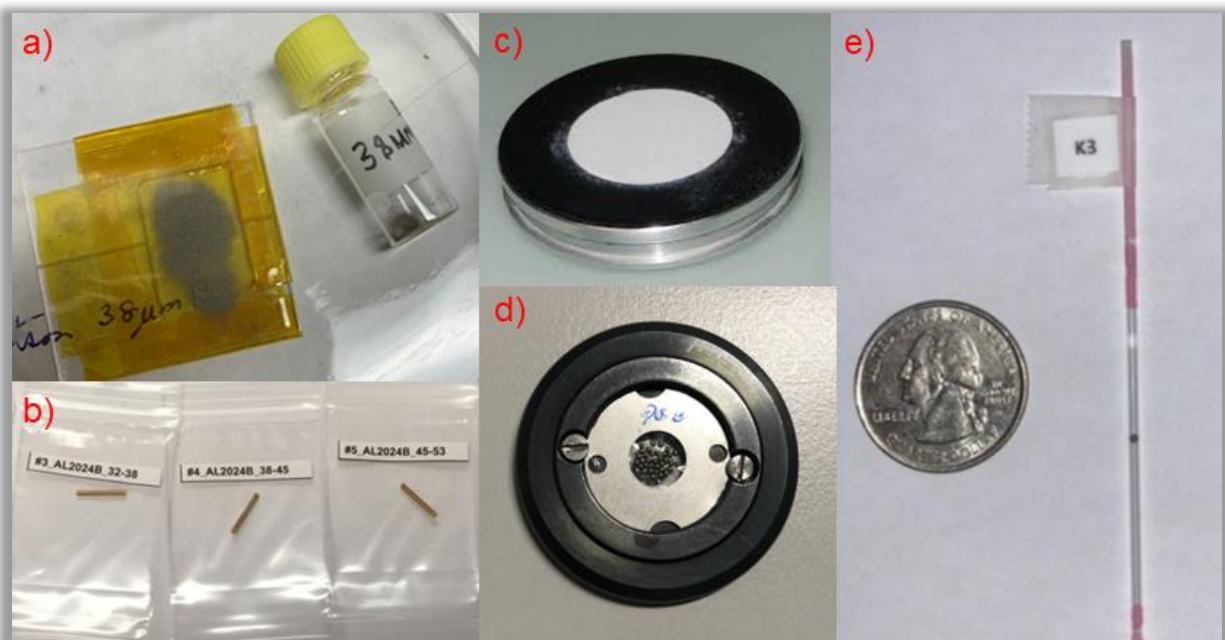


Figure 3-13: Various samples prepared for X-ray diffraction.

- a) Inconel 718 for the Phillips diffractometer, b) Al2024 powder for the APS synchrotron,
- c) Al2024 powder for the PANalytical diffractometer,
- d) Fe-Al particles for the STADI P diffractometer, e) Fe-Al particle for DESY synchrotron.

3.3 Electron Backscatter Diffraction

Electron backscatter diffraction experiments were conducted at the Space Material Science Institute of the German Aerospace Center (Institut für Materialphysik im Weltraum, Deutsches Zentrum für Luft- und Raumfahrt), often shortened to DLR, in Cologne, Germany. The EBSD samples were embedded in an electrically conductive material (Polyfast by Struers) at a temperature of 450 K and pressure of 25 kN. These specimens were gently ground with SiC paper and polished with a chemically neutral suspension of alumina particles followed by a colloidal silica suspension (diameter = 100 nm) with a pH = 9.8. Channel V software was used to visualize the local misorientation; further specifications of this method are available in Appendix Section A.

4 Data Analysis Methods

This chapter defines the data analysis methods utilized in this study, including X-ray diffraction Line Profile Analysis (XRD LPA), Electron Backscatter Diffraction (EBSD) analysis, and Dendrite Arm Spacing (DAS) analysis. XRD LPA is the conventional method for quantifying dislocation density, while EBSD analysis offers an alternative approach for estimation and comparison purposes. Both techniques are employed to ascertain dislocation density, whereas cell size analysis is utilized to experimentally estimate cooling rate. The relationship between all analysis methods used in this study is outlined in Figure 4-1. Detailed explanations of these methods will be provided in their respective sections.

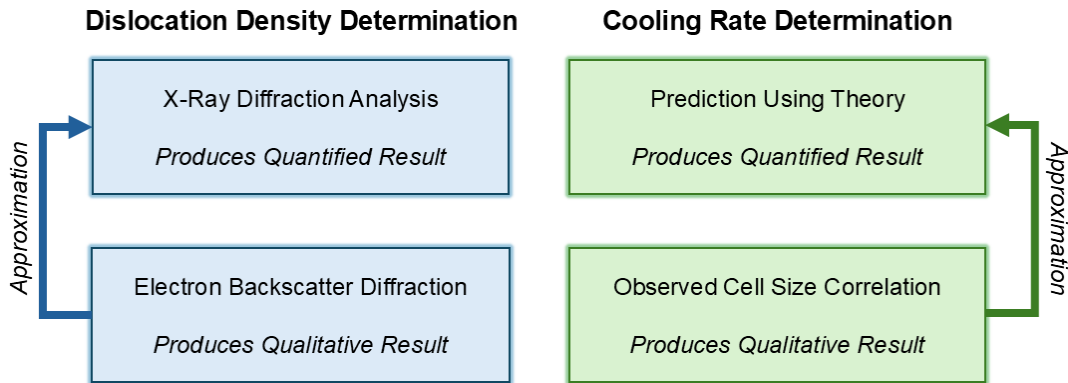


Figure 4-1: Schematic of logical flow of data analysis methods used in the study.

4.1 X-Ray Diffraction Line Profile Analysis

The rigorous data analysis approach utilized in this work is a mathematical approach to quantifying dislocation density using X-ray diffraction data. While the primary motivation is determining dislocation density, this method also yields additional information, including instrument precision, instrument bias, and dislocation character. A visual representation of this approach is shown in Figure 4-2.

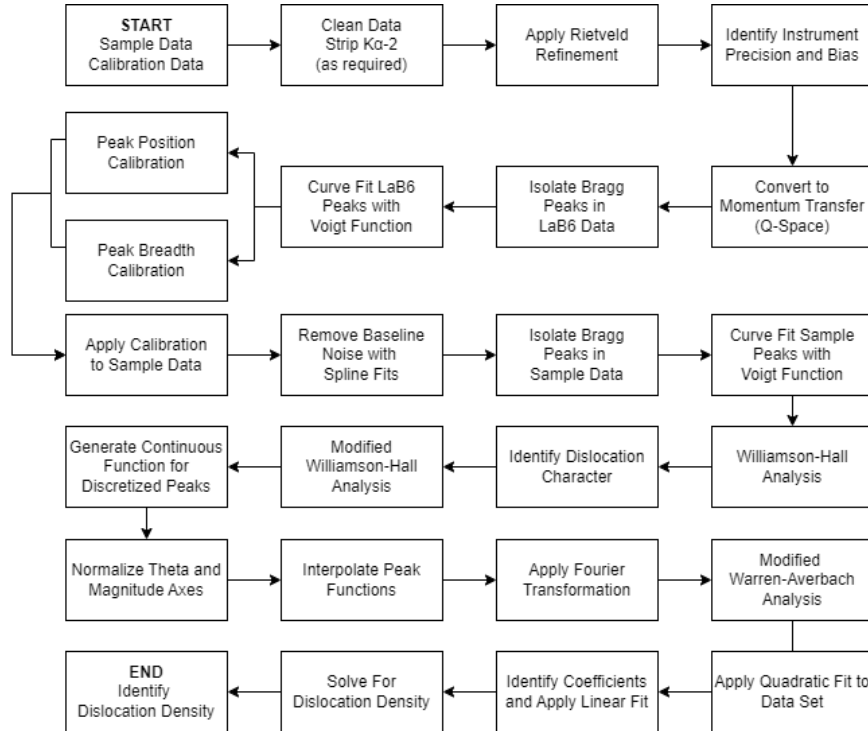


Figure 4-2: Flow chart of the X-ray diffraction line profile analysis method.

First, the instrumental profile of the diffractometer was assessed using the measured X-ray diffraction data from the Lanthanum Hexaboride calibration sample. The Bragg peaks for the calibration and experimental data were fit using a Voigt profile. The centroid shift and broadening can be identified by comparing the observed Bragg peaks for the calibration sample. This correction was then applied to the diffraction data for the material sample. Additionally, background noise was modeled with best-fit splines and subtracted from the data.

Subsequently, the modified Williamson-Hall (MWH) analysis was applied to the data. Plots generated using this technique were used to determine dislocation character, a representation of the proportion of edge and screw dislocations. Next, parameters from the curve-fitting of each Bragg peak were used to transform the data, quantized per each angular step of the diffractor, into a continuous function utilized in the next step. After, the modified Warren-Averbach (MWA) analysis was applied to the continuous functions representing each Bragg Peak. A Fourier

transformation was applied to these functions, and the resulting data was used to determine dislocation density. Each portion of this comprehensive analysis is explained in detail in its respective section.

4.1.1 Transformation to Reciprocal Space

As described in Section 2.4.3, the X-ray diffraction data is converted to q-space to enable comparisons between different diffractometers. This transformation is used in several places within the subsequent analysis, including converting the NIST-reported LaB6 calibration peak positions from Cu-K α radiation and the modified Williamson-Hall and Warren-Averbach equations. An example of the utility of the conversion is shown via a transformation for lanthanum hexaboride calibration data obtained using three different radiation energies in Figure 4-3.

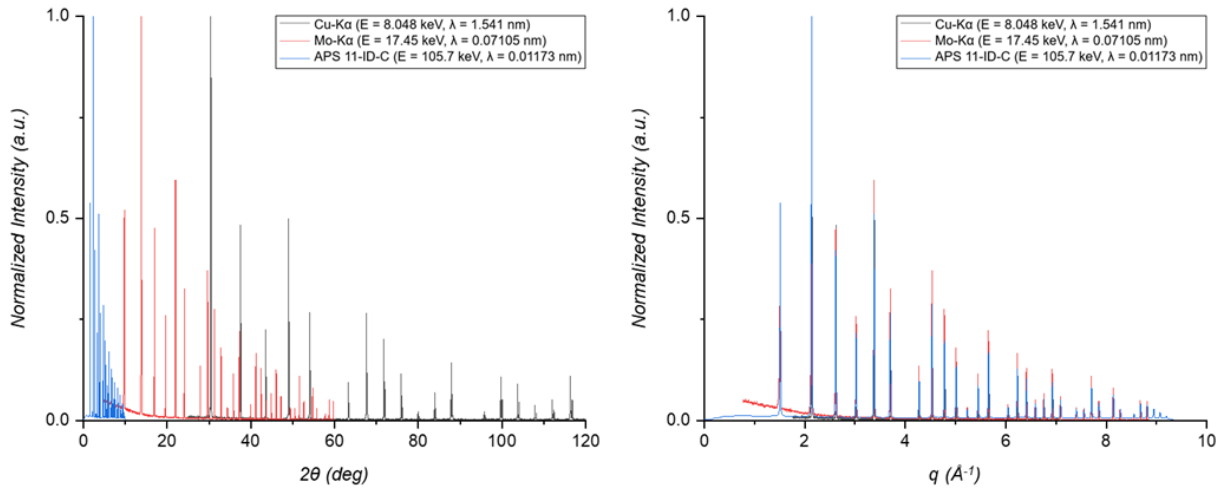


Figure 4-3: Calibration scans for various radiation energies plotted versus theta and wave vector.

4.1.2 Curve Fitting Technique

Employing the appropriate curve fitting method to analyze the Bragg peaks in X-ray diffraction data is essential for obtaining accurate results. Research published by NIST [48] suggests that the conventional methods of using only a Gaussian function (normal distribution), Equation (4.1):

$$I(\theta) = I_B + \frac{Ae^{\frac{-4\ln(2)(\theta-\theta_c)^2}{w^2}}}{w\sqrt{\frac{(\pi)}{4\ln(2)}}} \quad (4.1)$$

or a Lorentzian function (Cauchy distribution), Equation (4.2):

$$I(\theta) = I_B + \frac{2Aw}{4\pi(\theta - \theta_c)^2 + w^2} \quad (4.2)$$

is not adequate to accurately represent X-ray diffraction line profiles. Instead, a convolution of both a Gaussian and Lorentzian function, known as a Voigt function, is required. Within the analysis software used for this data analysis, the Pseudo-Voigt function [49] was utilized, a weighted sum of the Gaussian and Lorentzian functions which provides a close approximation to the Voigt profile, Equation (4.3):

$$I(\theta) = I_B + A \left[m_u \frac{2}{\pi} \frac{w}{4\pi(\theta - \theta_c)^2 + w^2} + (1 - m_u) \frac{\sqrt{4\ln(2)}}{\sqrt{\pi}w} e^{\frac{-4\ln(2)(\theta-\theta_c)^2}{w^2}} \right] \quad (4.3)$$

where $I(\theta)$ is the intensity of the diffracted X-rays as a function of incident angle, I_B is the intensity of the background noise, A is the amplitude of the curve, m_u is the linear weighting factor of the Lorentzian/Gaussian functions, w is the peak width, and θ_c is the centroid of the peak.

The OriginPro 2019b software used in this analysis utilizes a Levenberg–Marquardt optimization algorithm [50] to apply curve fits to data points. To demonstrate the importance of using a Voigt profile, all three of the curve-fits as mentioned earlier to a Bragg peak of an experimental data set are shown in Figure 4-4.

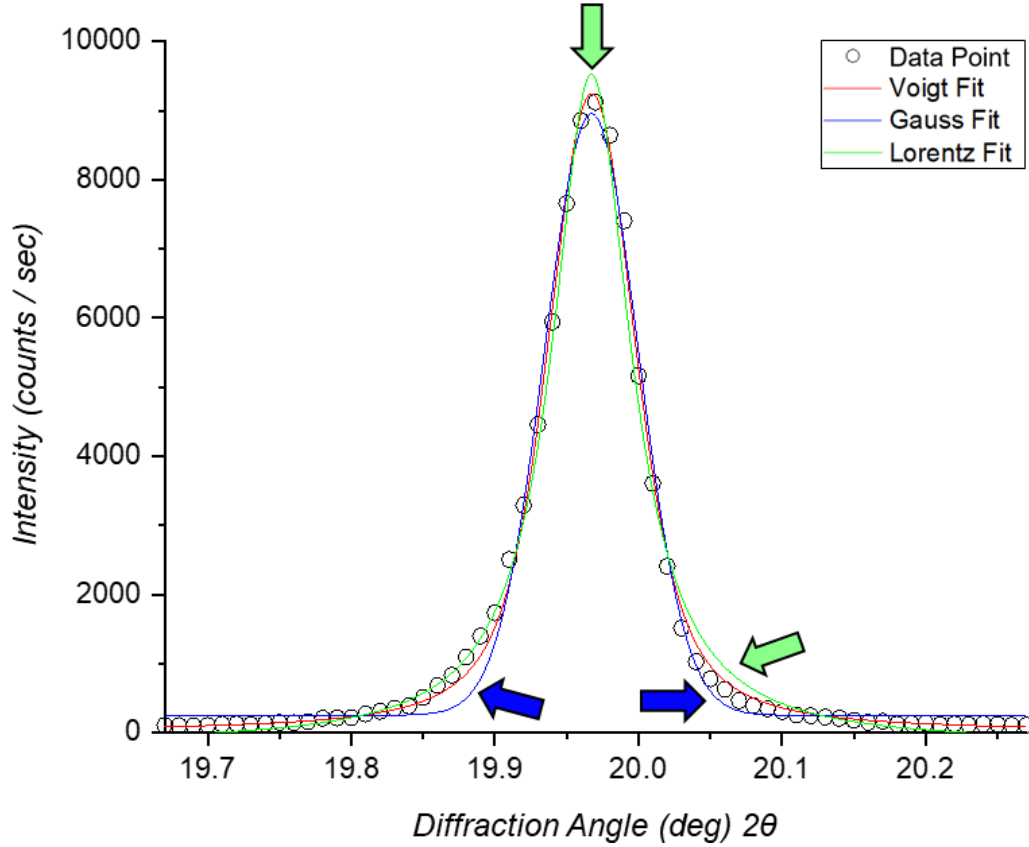


Figure 4-4: Voigt, Gaussian, and Lorentzian curve fit applied to a Bragg peak.

TABLE V: Comparison of Curve Fitting Functions

Fitting Function	R-Square Value
Voigt	0.9973
Gaussian	0.9917
Lorentzian	0.9818

As shown in TABLE V, the Lorentzian function has the lowest R^2 value (0.9818) and overestimates peak intensity, as shown by the green arrows in Figure 4-4. While the Gaussian function has a relatively higher R^2 value (0.9913), it underestimates peak breadth, as indicated by the blue arrows in Figure 4-4. “Fittingly”, the linear combination of both functions, known as the Voigt function, provided the best representation of a diffraction peak with the highest R^2 value (0.9971). It is well known that adding more parameters to a curve fitting technique will provide better results. However, there is a theoretical backbone to this approach because the observed

signal is a contribution of physical phenomena represented by Gaussian and Lorentzian distributions [51].

The peak position and breadth are the key curve parameters utilized in the following analysis. The geometric centroid of the peak characterizes the peak position parameter. The peak breadth is characterized by the full width at half maximum (FWHM). Both parameters are shown in Figure 4-5. The width of the curve at half of the maximum intensity value is utilized because it provides a normalization of the intensity. Depending on the X-ray diffractometer setup and scanning parameters, the intensity of a Bragg peak may change considerably for the same experimental sample. Therefore, the simple width of a curve is not sufficient to properly characterize peak broadening; instead, a determination of the FWHM is required.

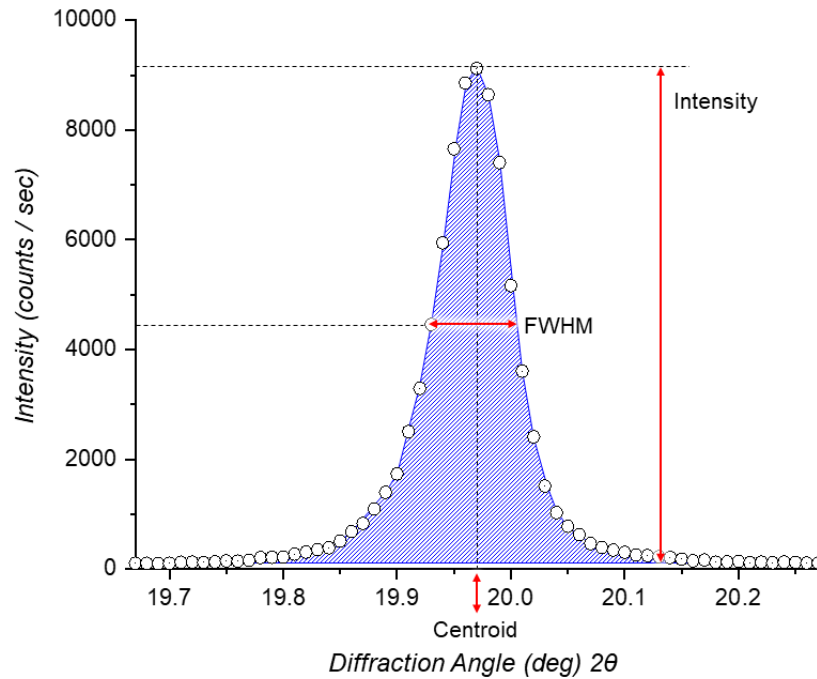


Figure 4-5: Schematic illustrating full-width-half-maximum determination from Bragg peak.

An example Voigt fit was applied to experimental data, as shown in Figure 4-6 with the resulting parameters provided in TABLE VI.

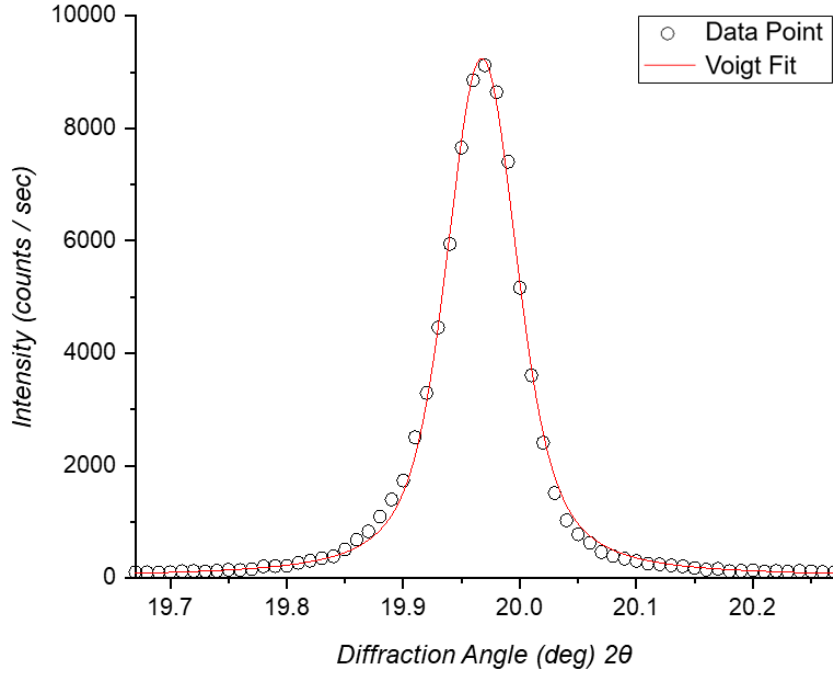


Figure 4-6: Voigt fit to Bragg peak of Fe-Al 200 μm diameter sample.

TABLE VI: Voigt Fit Parameters for X-Ray Diffraction Data

Parameter	Value
y_0	28.1332 ± 30.55658
x_c	19.9674 ± 0.000288984
A	888.68084 ± 19.09164
w_G (Gaussian FWHM)	0.04847 ± 0.00302
w_L (Lorentzian FWHM)	0.03940 ± 0.00338
R^2	0.99705
FWHM	0.07289 ± 0.00519

While the analysis software does not explicitly calculate the FWHM, it can be determined through an expression shown in Equation (4.4):

$$FWHM(w_G, w_L) = 0.5346w_L + \sqrt{0.2166w_L^2 + w_G^2} \quad (4.4)$$

Finally, all Bragg peak curve-fits in the scope of this study are evaluated at 95% confidence interval.

4.1.3 Peak Location Calibration

Apparent shifts in the centroid of Bragg peaks within X-ray diffraction data occur due to aberrations of the beam path of the X-ray diffractometer. Correcting these peak shifts is crucial for accurate data interpretation. To correct for instrumental peak shifts, the diffractometer is first calibrated using the lanthanum hexaboride standard reference material, which has well-known and accurately determined peak positions [52]. This provides a reference for identifying and correcting peak positions observed in the data obtained from the experimental samples. An example diffractogram for a lanthanum hexaboride calibration sample is shown in Figure 4-7.

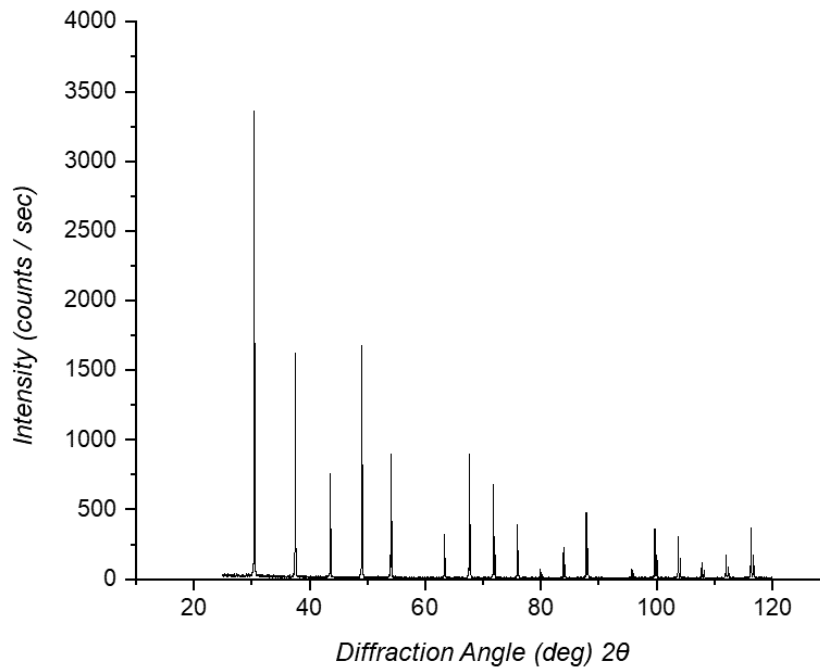


Figure 4-7: Calibration full-range diffractogram of lanthanum hexaboride sample.

All Bragg peaks from calibration datasets were curve-fit to identify peak centroids. These values were compared to the reported values located in Appendix A Material and Equipment Specifications. The difference between the observed values and expected values was then plotted as a function of incident angle and modeled using the Wilson formula [53], Equation (4.5):

$$\Delta_{2\theta}(\theta) = a \tan^{-1}(\theta) + bx + cx \tan(\theta) + dx \tan^2(\theta) + ex \tan^3(\theta) \quad (4.5)$$

where $\Delta_{2\theta}$ is the change in peak centroid position from the expected value, θ is the 2-theta angle of X-ray incidence, and a, b, c, d, e are fitting parameters.

Three example peak shift calibration curves from separate experiments conducted using the MIT diffractometer are shown modeled with the Wilson formula are provided in Figure 4-8.

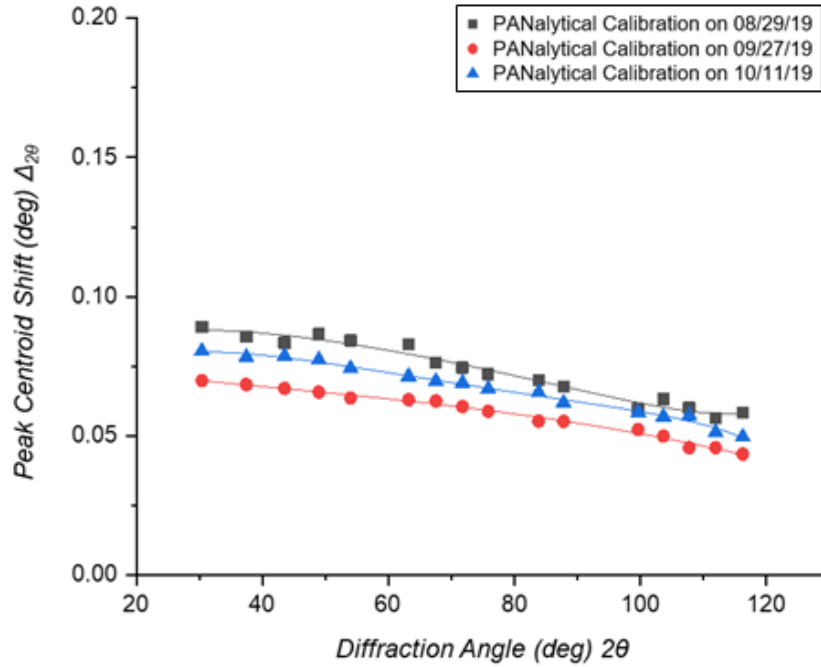


Figure 4-8: Peak position calibrations for three experiments using the PANalytical diffractometer.

The derived Wilson formulae were applied to each experimental data set. It should be noted that experiments conducted using Cu-K α radiation X-ray diffraction instruments required additional treatment if the Cu-K α “doublet”, shown in Figure 4-9 could not be removed from the dataset before analysis. This “doublet” is an artifact of Cu-K α radiation because the L shell electron can have two slightly different energy levels, resulting in two distinct wavelengths of radiation that

diffract at two different angles. To accurately quantify the centroid location of these “doublets,” a multi-peak Voigt fit is utilized. Only the centroid position of the $\text{Cu-K}\alpha_1$ is used.

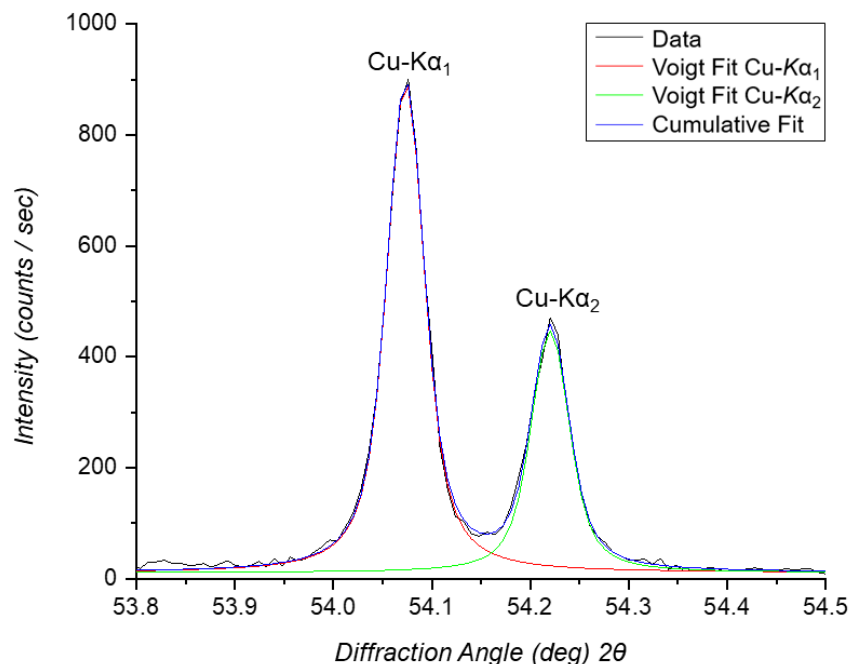


Figure 4-9: Cu-K α doublet example using the PANalytical diffractometer.

Lanthanum hexaboride calibration material was not available at the Tufts facility until after the data was collected. Once available, the standard material was eventually measured at the Tufts facility. However, due to the sensitivity of the diffractometer, the $K\alpha$ doublet components of the doublet could not be reliably separated in nearly all calibration peaks. For these reasons, the position was not calibrated for data at the Tufts facility. This artifact was either removed or easily identifiable and removable for all other facilities used in this experiment.

4.1.4 Peak Broadening Calibration

In addition to the peak shifting effect, the peak broadening effect of the diffractometer must also be identified and removed from the experimental data sets using a calibration sample. While peak broadening is attributed to both the physical sample and the imperfections of the

diffractometer, the instrument effect must be identified and removed from the data sets. Limitations of the diffractometer which contribute to artificial peak broadening include finite slit sizes, detector resolution, and monochromator effects. Proper calibration and correction are necessary to account for these effects.

Peak broadening due to the diffractometer, $\beta_{\text{instrument}}$, was identified using the lanthanum hexaboride calibration samples. Once the peak shift effect was removed from the calibration dataset, all Bragg peaks were curve-fit again to identify peak broadening using the FWHM. Peak breadths were then plotted as a function of incident angle and modeled using the Caglioti formula [54], Equation (4.6):

$$FWHM(\theta) = [W + V\tan(\theta) + U\tan^2(\theta)]^{1/2} \quad (4.6)$$

where w , v , and u are fitting parameters, and θ is the incident angle.

Three example peak broadening calibration curves from separate experiments conducted using the MIT diffractometer are shown modeled with the Caglioti formula provided in Figure 4-10.

Since the following analysis depends on accurate peak broadening statistics, the differences in the calibration curves illustrate the need for calibration before each data set is collected. The state of the diffractometer varies over time, as do lab conditions such as temperature and humidity. Therefore, it is imperative to calibrate the datasets, especially when comparing results from many diffractometers across the world.

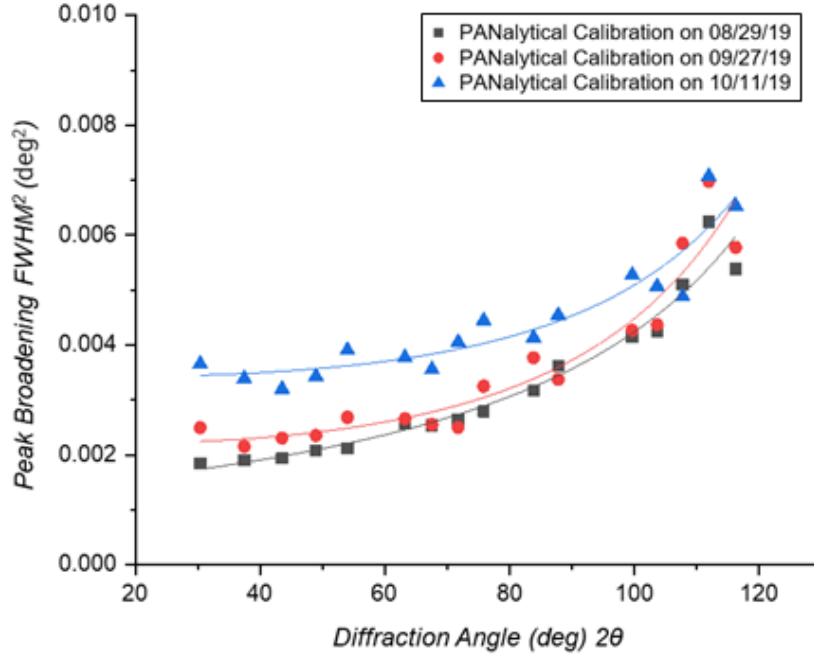


Figure 4-10: Peak breadth calibrations for three experiments using the PANalytical diffractometer.

Once the FWHM of the Bragg peaks for the experimental data has been obtained, the instrumental broadening effect may be removed. The curves observed within the experimental data are convolutions of both the instrumental broadening effects and the broadening due to the physical characteristics of the sample itself [48][55][56], as shown in Equation (4.7).

$$\beta_{measured}^2 = \beta_{sample}\beta_{measured} + \beta_{instrument}^2 \quad (4.7)$$

The correction to remove the instrumental broadening, as a function of incident angle, can be applied to obtain the broadening only due to the sample, as shown in Equation (4.8).

$$\beta_{sample} = \frac{\beta_{instrument}^2 \cdot \beta_{measured}^2}{\beta_{measured}} \quad (4.8)$$

4.1.5 Modified Williamson-Hall Method

The Williamson-Hall (WH) equation [57], proposed in 1953, is utilized to interpret Bragg peak broadening within X-ray diffraction data. The analysis method distinguishes between the

size-induced broadening and strain-induced broadening of diffraction peaks by considering the total broadening as a combination of both effects [33] as shown in Equation (4.9).

$$\beta_{sample} = \beta_{size} + \beta_{strain} \quad (4.9)$$

The Williamson-Hall equation utilizes the original work of the Scherrer equation [58], proposed in 1918 to describe the size-induced broadening of X-ray diffraction profiles. The original Scherrer equation was utilized to identify crystallite size in a material using the observed broadening of the Bragg peak:

$$D = \frac{K\lambda}{\beta_{size}\cos(\theta)} \quad (4.10)$$

where β_{size} is peak broadening, K is the Scherrer constant (equal to 0.9), λ is the radiation wavelength, D is the average crystallite size, and $\cos(\theta)$ is the Bragg angle.

For this analysis, we are primarily interested in peak broadening, so this equation can be rearranged to:

$$\beta_{size} = \frac{K\lambda}{D\cos(\theta)} \quad (4.11)$$

Equation (4.12) is generated with substitution of Equation (4.11) into Equation (4.9)

$$\beta_{observed} = \frac{0.9\lambda}{D\cos(\theta)} + \beta_{strain} \quad (4.12)$$

Next, the broadening due to strain is incorporated into Equation (4.12):

$$\beta_{observed} = \frac{0.9\lambda}{D\cos(\theta)} + 4\epsilon\tan(\theta) \quad (4.13)$$

Rearranging Equation (4.13) yields:

$$\beta_{observed}\cos(\theta) = \frac{0.9\lambda}{D} + 4\epsilon\sin(\theta) \quad (4.14)$$

Using this linear function, $\beta_{observed} \cos(\theta)$ may be plotted versus $4\sin(\theta)$ to solve for the slope, ε , which is the microstrain of the sample.

As mentioned in Section 4.1.1, a transformation to reciprocal space provides a more useful representation of this data, as it is normalized by wavelength, meaning results from different diffractometers can be compared easily. It is implied that the analysis of these peaks occurs at Bragg angles, as defined by Equation (2.17), where constructive interference between X-rays diffracting from atomic planes with interplanar spacing, d . A variable, K , is introduced to represent this interplanar spacing in reciprocal space, which is the magnitude of the diffraction vector, as shown in Equation (4.15).

$$K = \frac{1}{d} \quad (4.15)$$

When substituted into Equation (2.17), (for simplicity, consider $n = 1$):

$$\lambda = \frac{2 \sin(\theta)}{K} \rightarrow K = \frac{2 \sin(\theta)}{\lambda} \quad (4.16)$$

K will be utilized in the subsequent analysis to define peak position, and, by extension, ΔK will define peak broadening. To derive ΔK , Equation (4.16) is differentiated with respect to θ :

$$\Delta K = \frac{2 \cos(\theta) \Delta \theta}{\lambda} \quad (4.17)$$

Now, the Williamson-Hall equation can be represented in reciprocal space by substituting K and ΔK and simplifying:

$$\Delta K = \frac{0.9}{D} + 4K\varepsilon \quad (4.18)$$

The “conventional” Williamson-Hall plot can be generated by plotting K versus ΔK and solving for the slope, a value that represents microstrain, as shown in Figure 4-11.

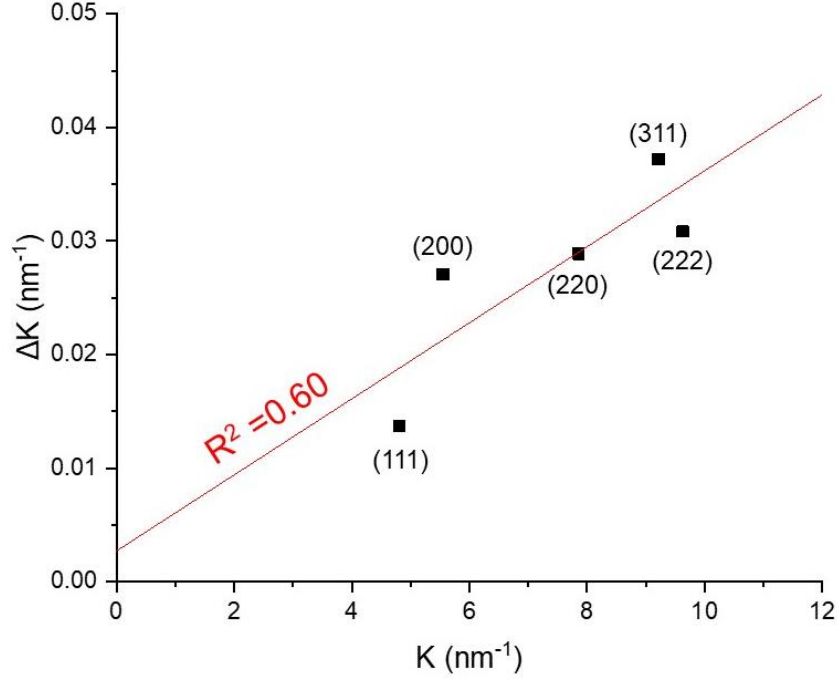


Figure 4-11: Conventional Williamson-Hall plot for Inconel 718 particle size 20-25 μm sample.

The isotropic strain due to dislocations in the conventional Williamson-Hall Model can be defined in Equation (4.19).

$$\Delta K_{strain} = A(\rho^*)^{1/2} + A'(Q^*)^{1/2} \quad (4.19)$$

where A and A' are parameters determined by the effective outer cutoff radius of dislocations, ρ^* is the “formal” dislocation density (defined later), and Q^* is related to the two-particle correlations in the dislocation ensemble [33].

However, this conventional model does not take anisotropic microstrain into account. A modification proposed by Ungár in 1996 [33] to provide a dislocation contrast factor, $\bar{C}$, to the conventional Williamson-Hall model, as shown in Equation (4.20).

$$\Delta K = \frac{0.9}{D} + \left(\frac{\pi A b^2}{2} \right)^{1/2} \rho^{1/2} (K \bar{C}^{1/2}) + \left(\frac{\pi A' b^2}{2} \right) (Q)^{1/2} (K^2 \bar{C}) \quad (4.20)$$

where b is Burgers vector.

In the cubic crystal system, the average contrast factors are a linear function of the fourth-order invariant of the hkl indices of the different reflections [38], as shown in Equation (4.21).

$$\bar{C} = A - \frac{B(h^2k^2 + h^2l^2 + k^2l^2)}{(h^2 + k^2 + l^2)^2} \quad (4.21)$$

where A and B are constants derived from the elastic constants of the material, and hkl are the Miller indices of the atomic planes.

Equation (4.21) can be rewritten as Equation (4.22).

$$\bar{C} = \bar{C}_{h00}(1 - qH^2) \quad (4.22)$$

where q is the quotient of the B and A constants from Equation (4.21), and H^2 is the orientation parameter derived from Equation (4.21). $\bar{C}_{h00}$ is the average contrast factor for the h_{00} reflections, determined using the elastic constants of the material [56].

$$\bar{C}_{h00} = a_c \left[1 - e^{\frac{-A_i}{b_c}} \right] + c_c A_i + d_c \quad (4.23)$$

where A_i is the elastic anisotropy defined as $A_i = 2c_{44}/(c_{11} - c_{22})$ and a , b , c , and d are parameters depending on the dislocation character, the crystal structure, and the ratio of c_{12}/c_{44} .

The q value of Equation (4.22) can be calculated from Equation (4.20). With the assumption that the third-order term $O(K^2\bar{C})$ is negligibly small [56], Equation (4.24) can be derived.

$$\frac{\Delta K^2 - \left(\frac{0.9}{D}\right)^2}{K^2} \approx -qH^2 \frac{\pi M^2 b^2 \rho}{2} \bar{C}_{h00} + \frac{\pi M^2 b^2 \rho}{2} \bar{C}_{h00} \quad (4.24)$$

To simplify Equation (4.24), $\alpha = \left(\frac{0.9}{D}\right)^2$ and $\beta = \frac{\pi M^2 b^2 \rho}{2}$ are introduced:

$$\frac{\Delta K^2 - \alpha}{K^2} \approx -qH^2 \beta \bar{C}_{h00} + \beta \bar{C}_{h00} \quad (4.25)$$

From Equation (4.25), the value of α can be determined by optimizing the linear relationship between $\frac{\Delta K^2 - \alpha}{K^2}$ and H^2 . The value of α is related to the Scherrer Equation (4.10), and thus can be used to determine crystallite size. The slope of this plot can be utilized to determine q .

Equation (4.22) can then be utilized to determine $\bar{C}$ which is used in Equation (4.20) to generate a “Modified” Williamson-Hall plot. The key difference to the previous Williamson-Hall plot is that the broadening is plotted versus $K\bar{C}^{1/2}$ rather than K . This model provides an improved scaling factor for the peak breadths, as shown in Figure 4-12.

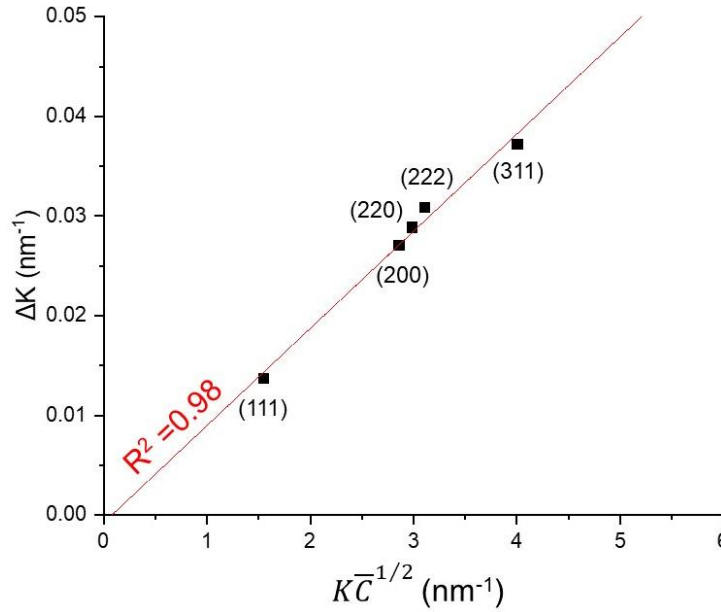


Figure 4-12: Modified Williamson-Hall plot for Inconel 718 particle size 20-25 μm sample.

4.1.6 Modified Warren-Averbach Method

The Warren-Averbach method [59], proposed in 1959, uses Fourier transformations to separate the effects of crystallite size and microstrain on X-ray diffraction peak broadening. Fourier transformations decompose the peak profiles into their fundamental components, making it easier to distinguish between the broadening caused by small crystallite sizes and the broadening caused by internal strains [36]. Similar to the “conventional” Williamson-Hall method, the “conventional” Warren-Averbach method begins with the extensive derivation [59] to define an equation relating observed broadening as the sum of the contributions from both size and strain:

$$\ln A(L) = \ln A_{size}(L) + \ln A_{strain}(L) \quad (4.26)$$

where $A(L)$ is the associated the Fourier transformation of the XRD profile and L is the associated “Fourier Length” defined as:

$$L = \frac{n\lambda}{2(\sin \theta_2 - \sin \theta_1)} \quad (4.27)$$

where n is a series of integers (0, 1, 2, 3 ...), θ_1 and θ_2 are the start and end of the angular range of the XRD profile.

The same modification proposed by Ungár in 1996 [57] to account for anisotropic microstrain is then applied to the conventional Warren-Averbach method to generate Equation (4.28).

$$\ln A(L) = Q \left(K^4 \bar{C}^2 \right) - \frac{\rho \pi b^2 L^2}{2} \ln \left(\frac{R_e}{L} \right) (K^2 \bar{C}) + \ln A_{size}(L) \quad (4.28)$$

where R_e is the effective outer cut-off radius of dislocations.

Before performing the subsequent Fourier transformation, the X-ray diffraction data, quantized by theta step, must be converted to a continuous function, as shown in Figure 4-13. These functions are generated using the results from the prior curve fits and normalized, which does not affect broadening character.

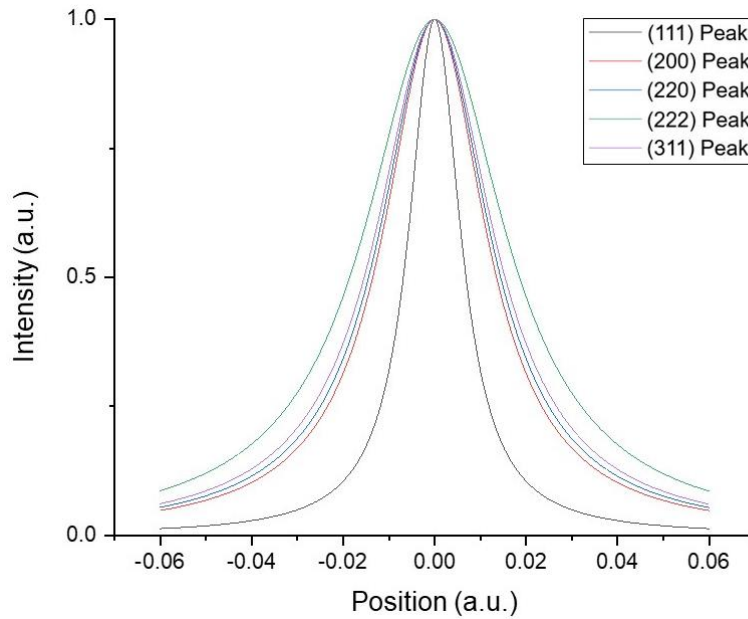


Figure 4-13: Continuous peak functions for Inconel 718 particle size 20-25 μm sample.

A Fourier transformation is then applied to these peaks, the results of which are evaluated at multiple Fourier Lengths, as shown in Figure 4-14. These results are plotted and fit with a quadratic function to determine the coefficients for $Q \left(K^4 \overline{C}^2 \right)$ of Equation (4.28).

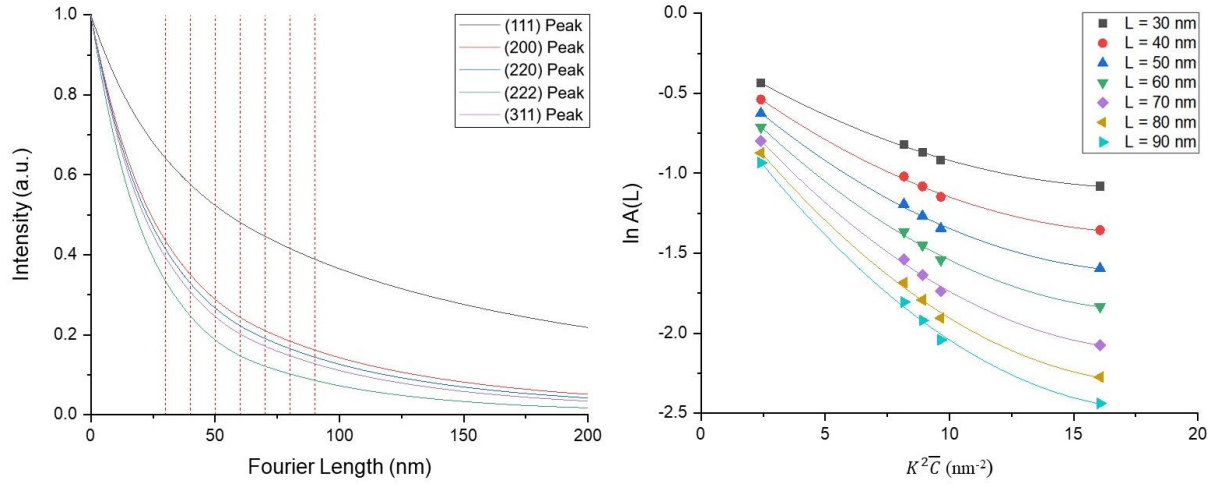


Figure 4-14: Fourier transformation and quadratic fit for Inconel 718 particle size 20-25 μm sample.

Once the quadratic coefficients have been determined, Equation (4.28) can be manipulated:

$$\frac{Q \left(K^4 \overline{C}^2 \right)}{(K^2 \overline{C})} = \frac{\rho \pi b^2 L^2}{2} \ln \left(\frac{R_e}{L} \right) \quad (4.29)$$

The logarithm of the Equation (4.29) can be expanded:

$$Q = -\frac{\rho \pi b^2 L^2}{2} \ln(L) + \frac{\rho \pi b^2 L^2}{2} \ln(R_e) \quad (4.30)$$

Equation (4.30) can be simplified:

$$\frac{Q}{L^2} = -\frac{\rho \pi b^2}{2} \ln(L) + \frac{\rho \pi b^2}{2} \ln(R_e) \quad (4.31)$$

The linear Equation (4.31) can be plotted as $\frac{Q}{L^2}$ versus $\ln(L)$, as shown in Figure 4-15, to determine the slope (m) $-\frac{\rho\pi b^2}{2}$.

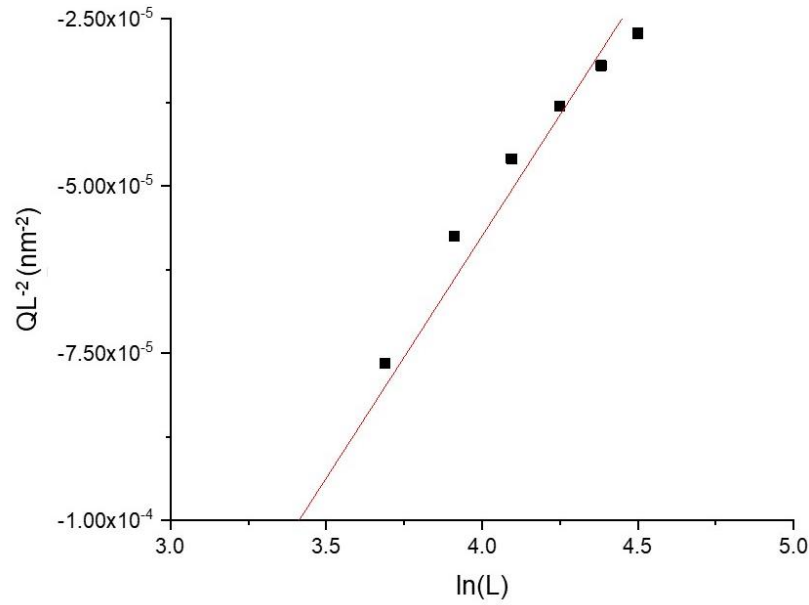


Figure 4-15: Dislocation density determination for Inconel 718 particle size 20-25 μm sample.

The dislocation density can then be determined using the slope.

$$\rho = \frac{-2m}{\pi b^2} \quad (4.32)$$

4.2 Electron Back Scatter Diffraction Analysis

While X-ray diffraction line profile analysis served as the primary technique to determine dislocation density trends within the samples, scanning electron microscopy (SEM) techniques were also utilized to approximate dislocation density trends to provide additional verification through estimation. SEM analysis techniques were used to determine phase composition, cell size, crystallographic plane orientation, and misorientation angle, which provided a basis for approximating dislocation density.

4.2.1 Misorientation Angle

While inverse pole figures are beneficial for providing information on whole cells and analyzing texture, identifying local misorientations is crucial for approximating dislocation density. Instead of aggregating similarly oriented pixels into cells, this approach highlights the minute differences between individual pixels and their neighboring pixels within a cell. An example of a misorientation angle distribution map is shown in Figure 4-16.

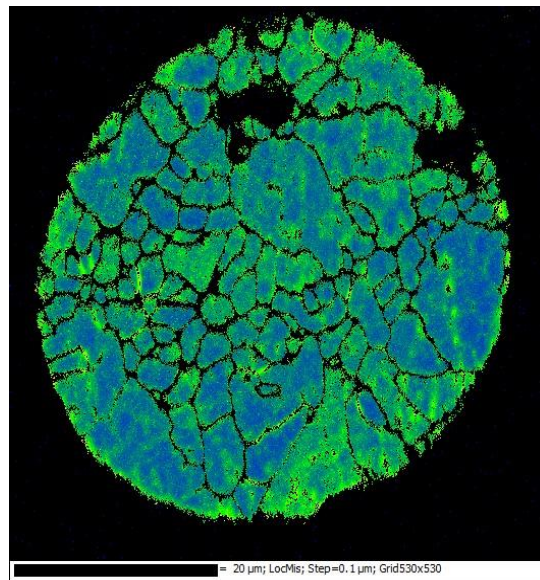


Figure 4-16: Local misorientation angle mapping for Al2024 particle size 53-63 μm sample. The color scale varies from blue (roughly 0 degrees) to green (roughly 2 degrees).

The software uses a kernel average misorientation (KAM) calculation, which determines the local misorientation value of a pixel by calculating misorientations between the center pixel of the kernel and all surrounding pixels [60]. In this analysis, the KAM calculation used a 7x7 pixel grid surrounding the center pixel to determine local misorientation, as shown in Equation (4.33). A simplified example calculation is shown in Figure 4-17.

$$\Delta\theta_{x,y} = \left| \theta_{x,y} - \frac{1}{n^2} \sum_{i=\lfloor x-n/2 \rfloor}^{\lfloor x+n/2 \rfloor} \sum_{j=\lfloor y-n/2 \rfloor}^{\lfloor y+n/2 \rfloor} \theta_{i,j} \right| \quad (4.33)$$

where $\Delta\theta_{x,y}$ is the local misorientation angle of the pixel located at the (x,y) coordinate, $\theta_{x,y}$ is the orientation angle of the pixel located at the (x,y) coordinate, and n is the length of the square pixel grid (7 in this case).

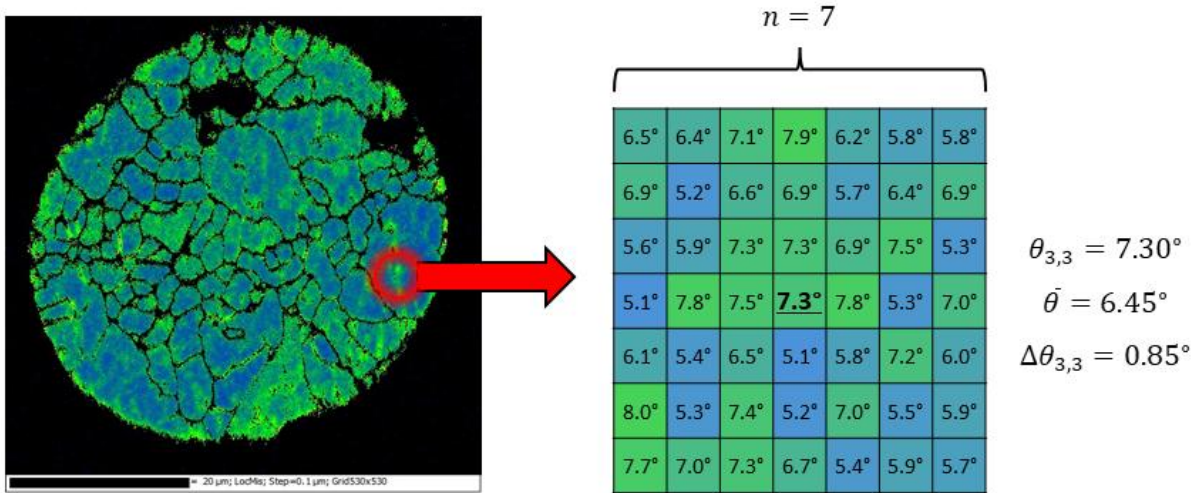


Figure 4-17: Schematic illustrating misorientation angle calculation.

The calculated misorientation angles are then grouped by 0.1 degree increments in this example and plotted as a function of relative frequency. An example of this is shown in Figure 4-18.

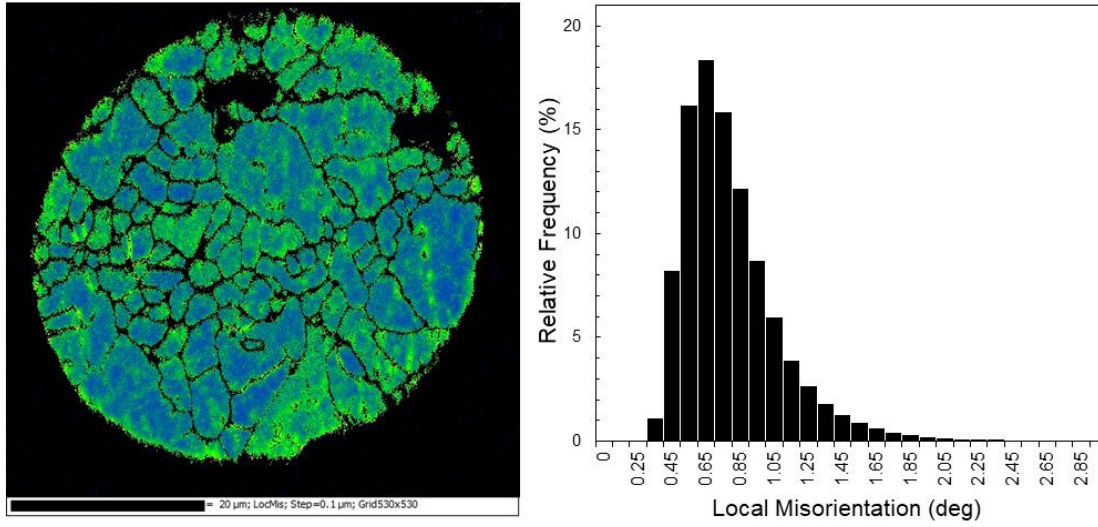


Figure 4-18: Relative frequency distribution for local misorientation angle example.

A lognormal fit was utilized to characterize the distribution of misorientation angles and determine a mean from the local misorientation mapping [61], as shown in Figure 4-19.

$$\Delta\theta = \frac{1}{\Delta\theta\sigma^*\sqrt{2\pi}} e^{-\frac{1}{2}\left(\frac{\ln(\Delta\theta)-\mu^*}{\sigma^*}\right)^2} \quad (4.34)$$

where $\Delta\theta$ is the misorientation angle, σ^* is the standard deviation (log), μ^* is the mean (log).

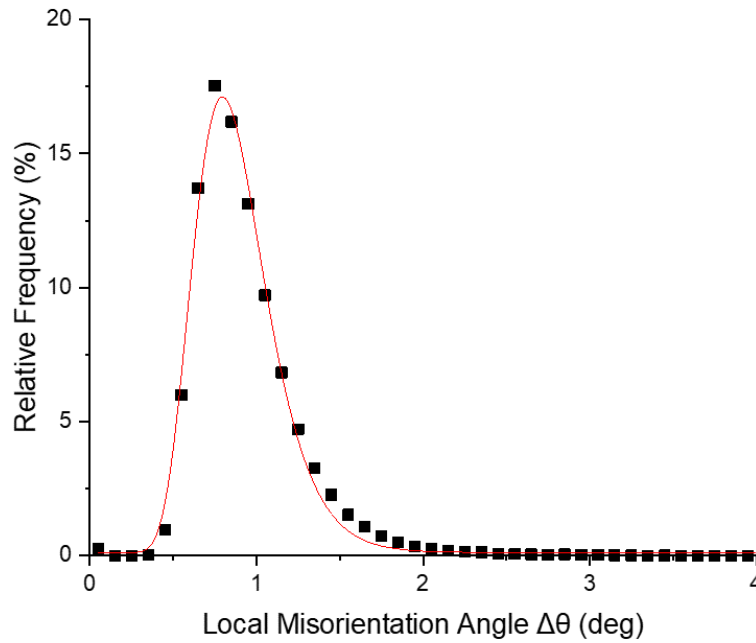


Figure 4-19: Lognormal fit to misorientation angle data example.

This fit was generated by calculating μ^* and σ^* from the dataset and substituting into Equation (4.34). There is a distinction made between μ^* and μ (and similarly for σ^* and σ) where μ represents the mean of the fit while μ^* represents the mean of the logarithm of the data, the same distinction also holds for the standard deviation. As a result of this distinction, uneven $\pm$ error bars are generated when the exponential of the result is calculated. The μ^* parameter was determined by calculating the sum of the products where the logarithm of the misorientation angle was multiplied by the relative frequency. The σ^* parameter was found by calculating the root of the squared difference between the product and the mean divided by the number of data points. The mean is calculated from the dataset using Equation (4.35).

$$\mu = e^{\mu^* + \frac{\sigma^{*2}}{2}} \quad (4.35)$$

Using this method, the asymmetric uncertainty could be carried through, as shown in Equation (4.36).

$$\mu \pm \sigma = e^{(\mu^* \pm \sigma^*)} \quad (4.36)$$

4.2.2 Dislocation Density Approximation

Dislocations distributed throughout the microstructure generate microstrain in the lattice, which causes local misorientations. Initially proposed by Nye in 1953 [62], the relative curvature of the lattice could be related to dislocations. Coined geometrically necessary dislocations (GNDs), these dislocations were theoretically *required* to account for non-uniform plastic deformation. The geometrically necessary dislocations are needed to preserve the continuity of the lattice [42][61]. A simplified illustration showing the necessary dislocations is shown in Figure 4-20.

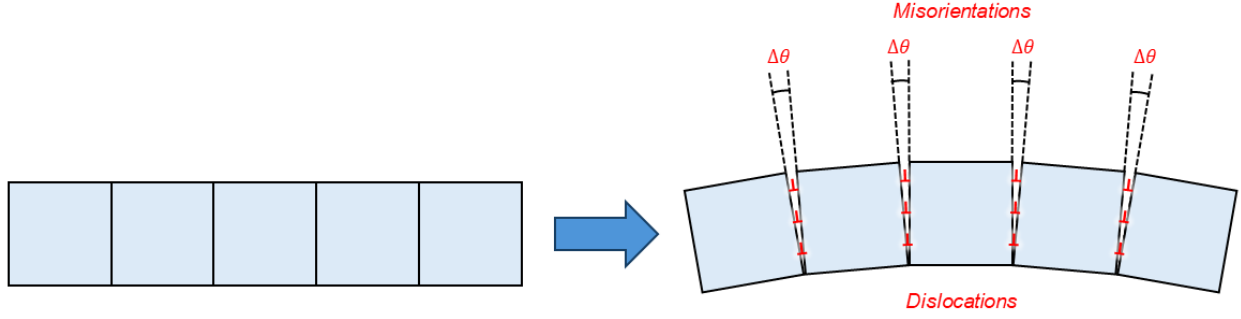


Figure 4-20: Simplified illustration of GNDs preserving continuity throughout the lattice.

The average density [42] of GNDs can be determined using Equation (2.1). Note that this can only be determined using the change of average misorientation angle and the change of Kernel radius. However, there are limitations to this method [63].

$$\rho_{GND} = \frac{k\Delta\bar{\theta}}{b\Delta d} \quad (4.37)$$

where k is numerical factor, b is the Burgers vector, and d is the Kernel radius.

If the ratio of GNDs to all dislocations is assumed to be constant, then the dislocation density is proportional to the average misorientation angle. This relation provided in Equation (4.38) enables the approximation of dislocation density trends using misorientation angle from EBSD.

$$\rho \propto \theta \quad (4.38)$$

4.3 Cooling Rate Approximation

The previous section established a relationship between dislocation density and particle size. The results of this relationship can be extended beyond just the samples in this experiment by correlating particle size to general solidification history, such as cooling rate. It is well understood that the microstructure is closely related to the cooling rate [64]. Using a thermodynamic heat balance, cooling rates can be approximated on a particle-size basis using

material properties and atomizer operating parameters. However, the accuracy of this approximation is dependent on accurate solidification conditions, which may not always be available or correct. Alternatively, evidence of cooling rate is embedded in the microstructure of each particle as cell size or dendrite arm spacing. Cooling rates can also be approximated on a cell-size-basis using experimentally established relationships. These two methods of approximating cooling rates will be evaluated in this section. Microstructural evolution can be predicted across a broader span of solidification processes by relating dislocation density to cooling rate.

4.3.1 Cooling Rate Determination using Particle Size

In addition to the above heat transfer terms mentioned in Section 2.2.2, there is also heat loss due to internal conduction. A dimensionless parameter, the Biot number, is frequently used to prove this term is negligible if $Bi < 0.1$.

$$Bi = \frac{hL}{k} \quad (4.39)$$

where Bi is the Biot number and L is the characteristic length (volume/area).

The thermal conductivity of all the metals in these experiments was above $10^1 \text{ Wm}^{-1}\text{K}^{-1}$. Thermophysical properties for the gases, nitrogen and argon, used in atomization for these experiments are well below $10^{-1} \text{ Wm}^{-1}\text{K}^{-1}$ for the expected temperature range [65]-[66]. Boettinger [64] provides an approximation of h :

$$h \approx \frac{2k_{gas}}{D} \quad (4.40)$$

Applying this approximation and generous bounds for the conductivities yields:

$$Bi = \frac{2k_{gas}}{k_{metal}} \approx \frac{10^{-1}}{10^1}; Bi \approx 0.01; Bi \ll 0.1 \quad (4.41)$$

Therefore, this condition will be satisfied, so the internal convection term is negligible.

Returning to the heat balance equation, a simplification of Equation (2.16) was proposed by Wiskel et al. [67] to describe the heat energy lost at the droplet surface by combining the conduction, convection, and radiation terms:

$$q = h_{eff} A (T_m - T_\infty) \quad (4.42)$$

where h_{eff} is the effective heat transfer coefficient and consists of the additive contribution of convection, conduction and radiation heat transfer mechanisms, A is the surface area of the droplet, T_m is the droplet surface temperature and T_∞ is the free stream gas temperature.

$$\frac{dT}{dt} = -\frac{6}{\rho D C_p} \left(k \frac{dT}{dr} + h(T - T_\infty) + \sigma \varepsilon (T^4 - T_\infty^4) \right) \quad (4.43)$$

This modifies the equation to:

$$\frac{dT}{dt} = -\frac{6h_{eff}}{\rho D C_p} (T_m - T_\infty) \quad (4.44)$$

Eloquently phrased by Wiskel et al., the simplicity of this equation belies the complexity of determining the effective heat transfer coefficient. The conductive component of the effective heat transfer coefficient is typically quantified through the use of semi-empirical equations involving the following dimensionless numbers, as provided in TABLE VII: Nusselt number (Nu), Reynolds number (Re), and the Prandtl number (Pr).

TABLE VII: Dimensionless Quantities Utilized in Cooling Rate Calculation

Dimensionless Quantity	Equation	Explanation of Use Case
Nusselt Number	$Nu = \frac{h_c D}{k}$	The ratio of the total heat transfer between droplet and gas to the heat transfer by conduction.
Reynolds Number	$Re = \frac{\rho_g v D}{\mu_g}$	The ratio of inertial forces to viscous forces in fluid flow, indicating whether the flow is laminar or turbulent.
Prandtl Number	$Pr = \frac{C_{p_g} \mu_g}{k_g}$	Ratio of momentum diffusivity to thermal diffusivity, indicating the relative thickness of velocity and thermal boundary layers.

where h_c is the convective component of h_{eff} , D is the droplet diameter, k is the conductivity, ρ_g is the density, v is the flow speed, μ_g is the viscosity, and C_{p_g} is the heat capacity of the gas.

The empirical relationship between these dimensionless numbers is fit to the following equation:

$$Nu = A + B \cdot Pr^m \cdot Re^n \quad (4.45)$$

where A , B , m , and n are experimentally determined constants.

Ranz-Marshall [68] proposed:

$$Nu = 2 + 0.6Re^{1/2}Pr^{1/3} \quad (4.46)$$

Whitaker [69] proposed:

$$Nu = 2 + (0.4Re^{1/2} + 0.06Re^{2/3})Pr^{0.4} \left(\frac{\mu_\infty}{\mu_s} \right)^{1/4} \quad (4.47)$$

where μ_∞ and μ_s are the viscosities at ambient temperature and at a surface temperature of the droplet.

These equations fail to account for the sizeable initial temperature gradient between the particle and the surrounding gas, where thermophysical properties vary dramatically. Additionally, in the case of small droplets produced during atomization processes, modeling efforts have determined that the Reynolds number is relatively low for the majority of the flight distance [70]. Therefore, the value of the constant in the equation becomes increasingly important as Reynolds number approaches zero. To address these issues, Wiskel [67] proposes a modification to the first term of the Ranz-Marshall and Whitaker equations:

Modified Ranz-Marshall Equation:

$$Nu = 2 \cdot \frac{B}{k_s \cdot (m + 1)} \cdot \frac{(T_s^{m+1} - T_\infty^{m+1})}{T_s - T_\infty} + 0.6Re^{1/2}Pr^{1/3} \quad (4.48)$$

Modified Whitaker Equation:

$$Nu = 2 \cdot \frac{B}{k_s \cdot (m + 1)} \cdot \frac{(T_s^{m+1} - T_\infty^{m+1})}{T_s - T_\infty} + 0.6Re^{1/2}Pr^{1/3} \quad (4.49)$$

A Kirchoff Transformation [71] is applied to Equation (4.50) to remove the radial dependence of the gas thermal conductivity term [67]:

$$\frac{d}{dr} \left(r^2 k_g \frac{dT}{dr} \right) = 0 \quad (4.50)$$

For cases where Reynolds number is approximated to be zero, the Nusselt number is now corrected to:

$$Nu = 2 \cdot \frac{B}{k_s \cdot (m + 1)} \cdot \frac{(T_s^{m+1} - T_\infty^{m+1})}{T_s - T_\infty} \quad (4.51)$$

The combined conduction/convection heat transfer coefficient can be described as a function of Nu :

$$h_c = \frac{Nu \cdot k_s}{D} \quad (4.52)$$

$$h_c = \frac{2B(T_s^{m+1} - T_\infty^{m+1})}{(m + 1)(T_s - T_\infty)D} \quad (4.53)$$

While the radiation term can be linearly approximated Taylor series expansion and retain only the first-order term:

$$h_{rad}(T) = \sigma \varepsilon (T_s^3 - T_\infty^3) \quad (4.54)$$

Collecting these terms provides an effective heat transfer coefficient:

$$h_{eff} = \frac{2B(T_s^{m+1} - T_\infty^{m+1})}{(m + 1)(T_s - T_\infty)D} + \sigma \varepsilon (T_s^3 - T_\infty^3) \quad (4.55)$$

Now, the cooling rate can be presented as:

$$\frac{dT}{dt}(T) = - \frac{6 \left(\frac{2B(T_s^{m+1} - T_\infty^{m+1})}{(m + 1)D} + \sigma \varepsilon (T_s^4 - T_\infty^4) \right)}{\rho D C_p} \quad (4.56)$$

Equation (4.56) describes cooling rate as a function of temperature. For this approximation, the goal is to approximate each particle size to a single cooling rate. Therefore, an “instantaneous” cooling rate must be determined since the cooling rate is a function of temperature. Other published work [25][72] uses the atomization temperature. However, this fails to capture the cooling rate around the time when cells are formed. In this work, the temperature utilized is the liquidus-solids midpoint temperature, T_{mid} , the average of $T_{solidus}$ and $T_{liquidus}$; this method is consistent with techniques from other published work [24]. However, if the $T_{solidus}$ and $T_{liquidus}$ temperatures are utilized instead of T_{mid} , the maximum variation in resulting cooling rate model for the applicable particle sizes are as follows: Inconel 718 $\pm 4.5\%$, Al2024 $\pm 15\%$, and Fe-Al $\pm 0.1\%$. The relative variations of the cooling rate results for different materials are tied to the difference between solidus and liquidus temperatures reported in TABLE VIII. The material and gas properties used to perform this calculation are provided in TABLE VIII and TABLE IX, respectively. In this remainder of this study, the cooling rates are evaluated at the midpoint between the liquidous and solidus temperatures of the alloy.

$$\frac{dT}{dt}(T = T_{mid}) = - \frac{6 \left(\frac{2B(T_{mid}^{m+1} - T_{\infty}^{m+1})}{(m+1)D} + \sigma \epsilon (T_{mid}^4 - T_{\infty}^4) \right)}{\rho D C_p} \quad (4.57)$$

TABLE VIII: Published Material Properties Utilized in Cooling Rate Calculation

Material	ρ (kgm^{-3})	C_p ($Jkg^{-1}K^{-1}$)	$T_{solidus}$ (K)	$T_{liquidus}$ (K)	Emissivity (ϵ)
Inconel 718	8221	720 [73]	1533 [74]	1609 [74]	0.385 [75]
Al2024	2270 [76]	1140 [76]	775 [77]	911 [77]	0.1 [78]
Fe-Al	7460 [79][80]	922 [81][82]	1811 [83]	1813 [83]	0.39 [84]

In TABLE VIII, the density of Inconel 718 was obtained from the material specification sheet provided with the samples and listed in the Appendix Material and Equipment Specifications section. The emissivity value used for Inconel 718 is for an unoxidized powder. While the emissivity of oxidized Inconel 718 is much higher, it is assumed there is no oxidation because of the inert gas environment and aluminum and chromium in the alloy. The density of the Fe-Al alloy is approximated using a linear combination of both elements by weight proportion. The solidus and liquidus temperatures for the Fe-Al alloy were estimated from the phase diagram. The emissivity value for the Fe-Al alloy was approximated as the emissivity for iron.

TABLE IX: Published Gas Properties Utilized in Cooling Rate Calculation

Gas	ρ (kgm^{-3})	C_p ($Jkg^{-1}K^{-1}$)	k ($Wm^{-1}K^{-1}$) $\times 10^4$	η ($Pa \cdot s$) $\times 10^5$
Argon [67]	$539.23 \cdot T^{-1.0205}$	520	$1.86 \cdot T^{0.7915}$	$0.0238 \cdot T^{0.7913}$
Nitrogen [85]	$335.55 \cdot T^{-0.9971}$	$0.182 \cdot T + 978$	$3.44 \cdot T^{0.7609}$	$3.79 \cdot T^{0.6766}$
Helium [85]	$37.303 \cdot T^{-0.9559}$	5197	$27.78 \cdot T^{0.7025}$	$4.3679 \cdot T^{0.6702}$

The results from the model are provided in Figure 4-21.

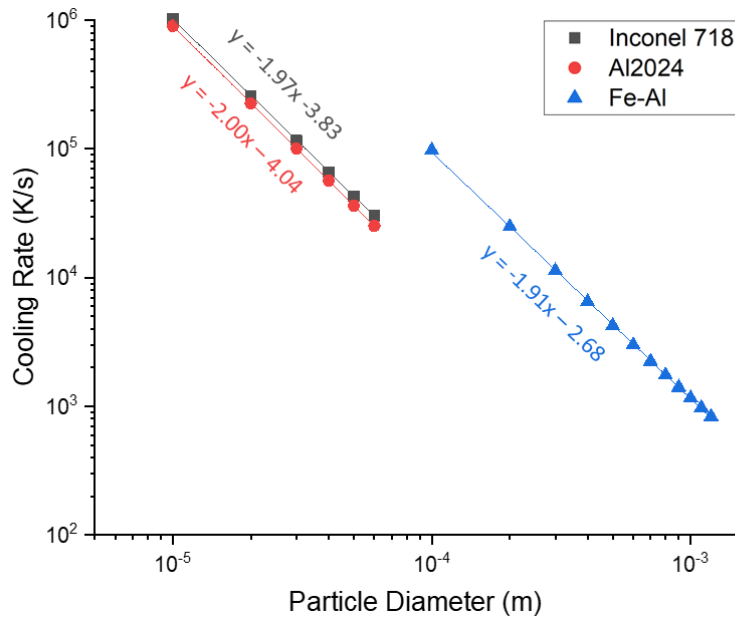


Figure 4-21: Estimated cooling rates for Inconel 718, Al2024, and Fe-Al displayed on a log-log plot.

4.3.2 Cooling Rate Determination using Cell Size

If an experimental approach is taken rather than a model-based approach, the cooling rate can also be approximated using the secondary dendrite arm spacing. Specifically, the relationship between secondary dendrite arm spacing and cooling rate is inversely proportional with additional factors depending on the material type and solidification conditions. The established, general relationship between secondary dendrite arm spacing and cooling rate is provided in Equation (4.58).

$$\lambda_2 = \lambda_0 \left(\frac{dT}{dt} \right)^{-n} \quad (4.58)$$

Solving for cooling rate yields Equation (4.59).

$$\frac{dT}{dt} = \left(\frac{\lambda_0}{\lambda_2} \right)^{1/n} \quad (4.59)$$

where $\frac{dT}{dt}$ is the cooling rate, λ_2 is the dendrite arm spacing, λ_0 is a constant dependent on material and solidification conditions, and n is a geometric constant based on a spherical morphology.

The dendritic structures of cast samples are more pronounced than the microstructure of rapidly solidified particles. Therefore, in this study, secondary arm spacing is approximated by measuring the apparent cell size of the particles. This method aligns with the methodology of other studies conducted on rapidly solidified samples [25][24].

Most published literature in metallurgy relating cell size to cooling rate utilizes experimental data from castings, which may not apply to the rapid cooling rates experienced by these sub-millimeter-sized particles [25]. However, several studies have provided correlations relevant to this regime, as provided in TABLE X. Significantly more published work offers a relationship for Al2024 than Inconel 718 or Fe-Al alloys.

TABLE X: Published Relationships for Cooling Rate using Dendrite Arm Spacing

Material	λ_0 [10^{-6} m]	n	Reference
Inconel 718	63	0.4	Bassini et al. [86]
Inconel 718	$4.38 \cdot 10^5$	0.75	Kumar et al. [87]
Al2024	54.5	0.328	Kasperovich et al. [88]
Al2024	102.33	0.33	Bäckerud et al. [89]
Al2024	43.4	0.32	Mullis et al. [24]
Al2024	86.74	0.33	Sousa et al. [76]
Al2024	45	0.39	Flemings et al. [90]
Fe-Al	80	0.33	Katayama et al. [91]

In TABLE X, the values for Inconel 718 provided by Kumar et al. [86] were obtained from Equation 8 of their work and adapted to SI units. These values are unexpectedly high but correlate with the data presented in Figure 10 of their work. The values for Al2024 provided by Flemings et al. [90] are widely cited for this material but not directly applicable to the cooling ranges within the scope of this experimentation [24]. There is no published work on this relationship for this exact Fe-Al alloy studied (92% Fe 8% Al, by weight), so a correlation for stainless steel provided by Katayama et al. [91] was utilized as an approximation.

4.3.3 Cell Identification Methods

Image analysis software was utilized on micrographs to determine the cell morphology of the samples. The first attempt to identify and quantify cells was conducted on Inconel 718 at Tufts University. The powder samples were mounted in an epoxy resin mold, polished using a rotating abrasive pad, and etched using Kaling's #2 reagent [92]. These samples were then observed using optical microscopy and uEye Cockpit software to record micrographs, as shown in Figure 4-22 (b). Eventually, this technique was improved for obtaining micrographs of Al2024 samples, as

shown in Figure 4-22 (c). However, these methods were insufficient to determine cell size accurately, and SEM was utilized as this research matured.

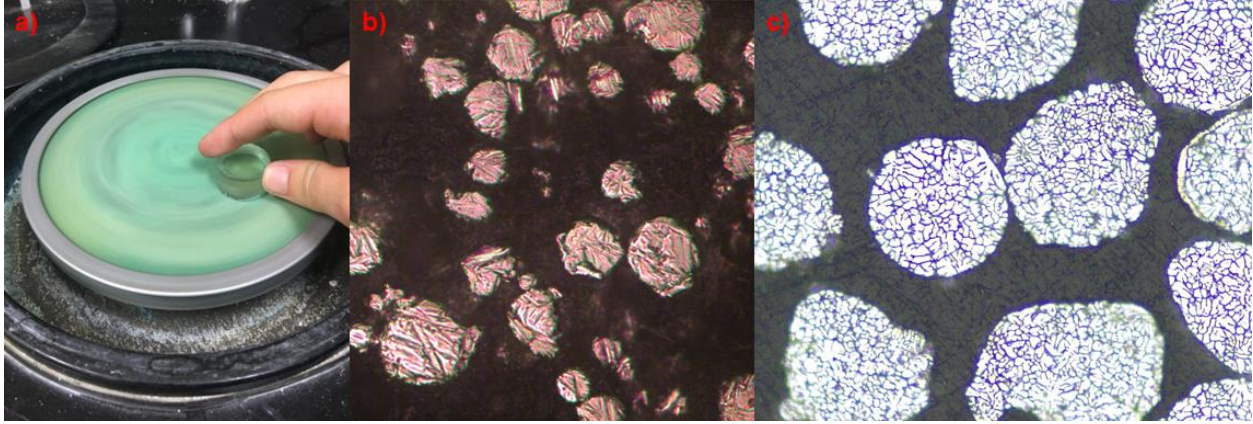


Figure 4-22: Metallography polishing and etching process.

- a) polishing of mounted powder, b) optical microscopy of Inconel 718 particles,
- c) improved optical microscopy of Al2024 samples.

Although the initial effort to quantify cell size using optical microscopy was unsuccessful, it indicated that cell size variation from the center to the outer surface of the particles was not significant. Subsequently, the outer surfaces of samples were analyzed using Field Emission Scanning Electron Microscopy (FE-SEM). Examining the outer surface of the particle was acceptable but not optimal for determining cell size, as cell size is influenced by the cooling rate. During solidification, a cooling rate gradient exists between the center of the particle and the outer surface. However, given the small size of these particles, solidification occurs rapidly, resulting in a less pronounced difference in cooling rates and cell size variation compared to larger metal parts such as castings. FE-SEM was used to image surface cells for all particle sizes of Inconel 718 and Al2024 samples; an example of the cell size analysis and image processing is shown in as shown in Figure 4-23. This automated cell processing method was first verified manually by determining the average cell diameter by measuring five equally spaced lines passing through the center of the cell and the linear intercept method [93].

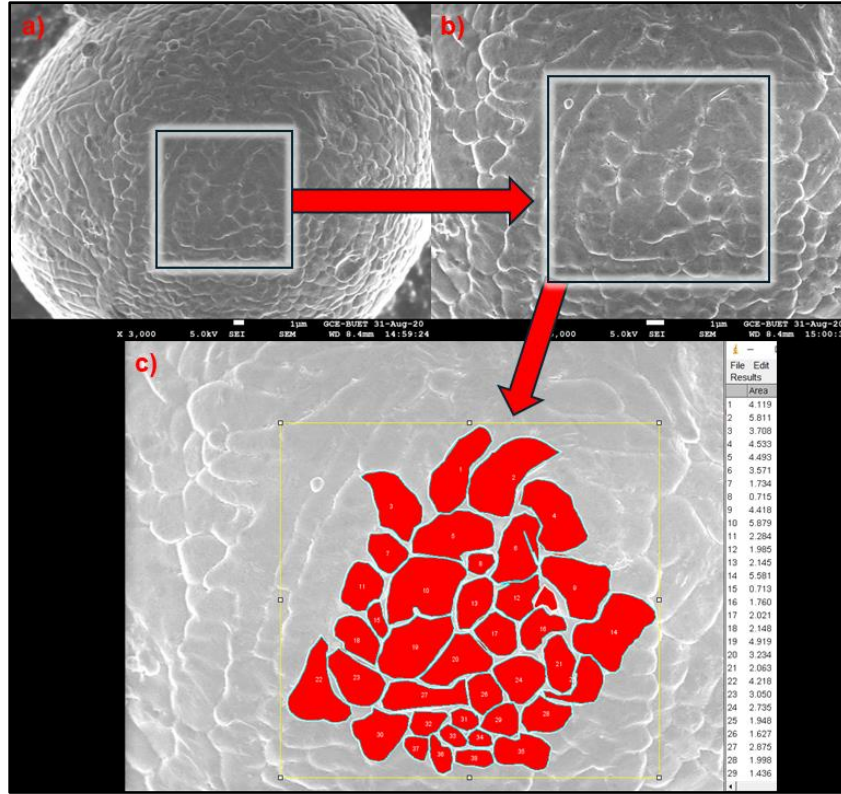


Figure 4-23: SEM cell size determination for Al2024 particle size 38-45 μm sample.
a) micrograph at 3,000X magnification, b) micrograph at 5,000X magnification,
c) isolation of apparent cells.

While the FE-SEM method of determining cell size was an improvement to the optical microscopy method, another etching method and EBSD analysis was also used to characterize cell morphology within the interior of the samples. Individual cells can be identified within micrographs because cell boundaries appear as brighter contours due to differences in back electron scatter intensity [94]. Al2024 particles were mounted, polished, and etched to reveal the microstructure of an interior cross section. These images were processed using image analysis software to characterize average cell size, as shown in Figure 4-24.

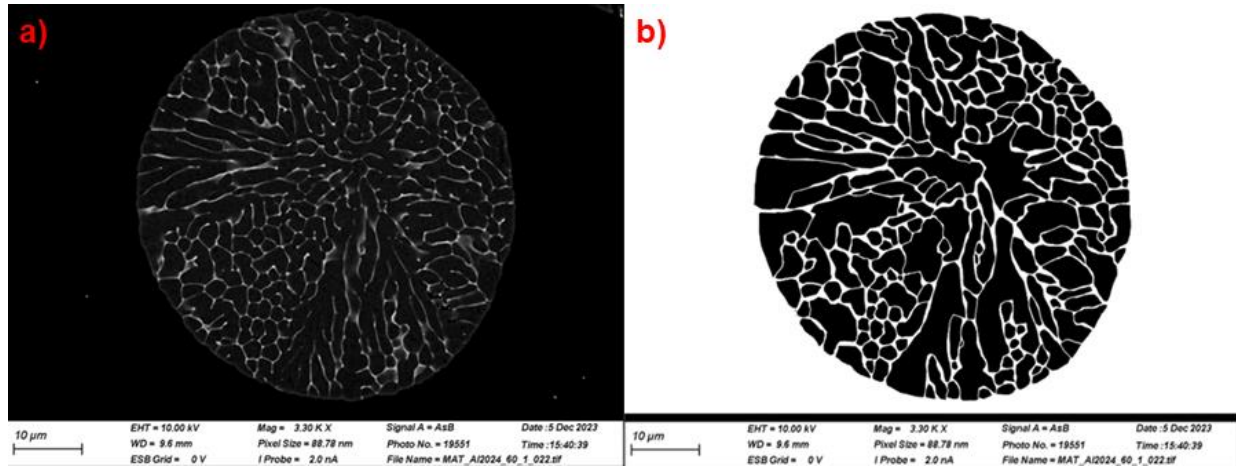


Figure 4-24: Cell characterization for Al2024 particle 20-25 μm size sample.
a) EBSD micrograph cross-section, b) isolation of apparent cells.

Additionally, attempts to quantify cell size using the inverse pole figures (IPFs) generated based on crystallographic plane orientation. However, this method was not as successful due to portions of the image where the crystallographic orientation could not be resolved. To attempt to resolve this issue, a smoothing filter was applied to the original micrograph before automated image analysis, as shown in Figure 4-25. This method provided a rough estimation for verification but was not used.

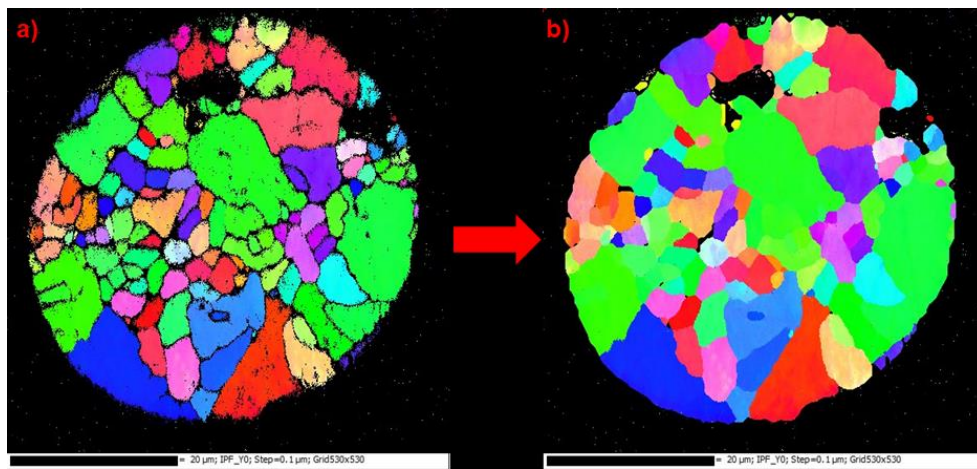


Figure 4-25: EBSD IPF (Y0 orientation) of Al2024 particle 53-63 μm size sample.
a) unprocessed IPF, b) IPF after smoothing filter is applied.

5 Results and Discussion

This chapter presents the findings from the experiments processed using the data analysis methods described in Chapter 4: Data Analysis Methods. The primary focus is the relationship between solidification history and dislocation density in the three selected materials: Inconel 718, Aluminum Alloy 2024, and the Fe-Al binary alloy. The results from the X-ray diffraction are presented and compared to the approximated values obtained using electron backscatter diffraction. The solidification history of these alloys is estimated using observed cell size and compared to model-based cooling rate approximations. The dislocation density of each alloy is then correlated with their respective solidification history.

5.1 Inconel 718 Results

5.1.1 X-Ray Diffraction

As the initial step in this research, the original experiment conducted in 2018 [95] utilized X-ray diffraction of Inconel 718 powder to achieve several objectives:

1. Confirm the Bragg peaks were at the anticipated Bragg angles.
2. Confirm that two separate samples of the same particle size produce similar diffractograms.
3. Identify the Bragg peak broadening from the diffraction data.
4. Assess whether annealing healed dislocations and reduced Bragg peak broadening.

The first and second objectives were accomplished using the data from Figure 5-1 and calculations from TABLE XI and TABLE XII. These experiments correspond to Test IDs 1 and 2 of TABLE IV. The observed Bragg diffraction peak angles and the expected values were less than 0.2% from each other.

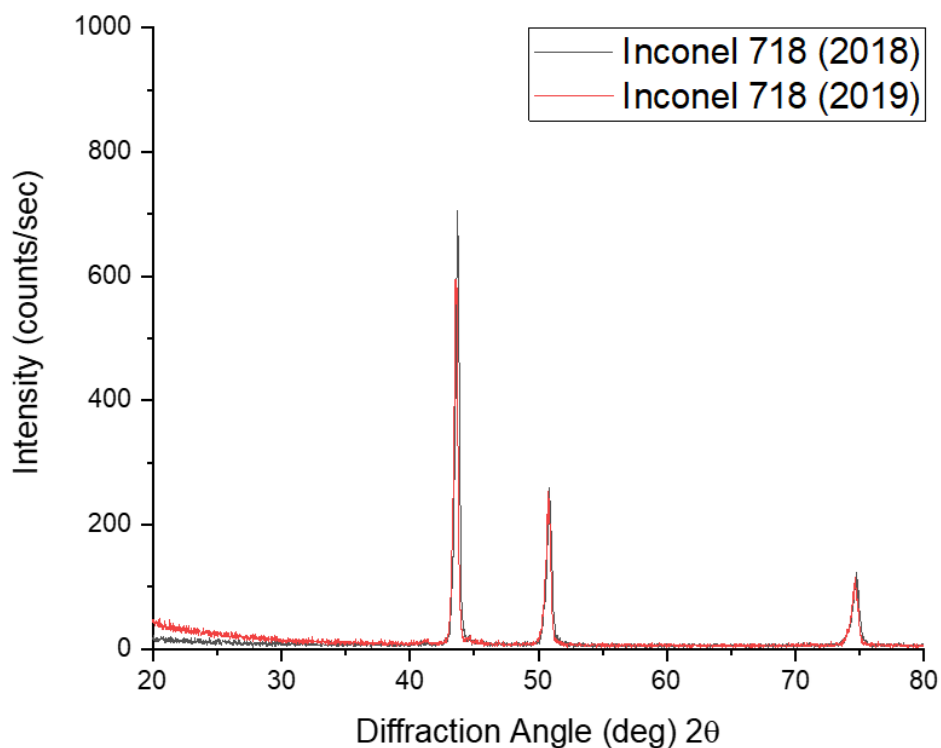


Figure 5-1: Inconel 718 particle 53-63 μm size repeated low-resolution diffractograms.

TABLE XI: Inconel 718 Expected Diffraction Angle Calculations

Lattice Parameter (Å)	Miller Indices	Interplanar atomic spacing (Å)	X-Ray Wavelength (Å)	Order of Diffraction	Expected Diffraction Angle (rad)	Expected Diffraction Angle (deg)
a	h l k	d	λ	n	2θ	2θ
3.591	1 1 1	2.0733	1.5406	1	0.761	43.621
3.591	2 0 0	1.7955	1.5406	1	0.887	50.810
3.591	2 2 0	1.2696	1.5406	1	1.304	74.706

TABLE XII: Inconel 718 Observed Diffraction Angles

Sample	Peak	Expected Angle (deg)	Observed Angle (deg)	Difference (%)
Inconel 718 (2018)	(111)	43.621	43.70	0.18
Inconel 718 (2018)	(200)	50.810	50.86	0.10
Inconel 718 (2018)	(220)	74.706	74.72	0.20
Inconel 718 (2019)	(111)	43.621	43.56	0.14
Inconel 718 (2019)	(200)	50.810	50.74	0.14
Inconel 718 (2019)	(220)	74.706	74.76	0.07

Once the Bragg peak location was confirmed, subsequent scans were completed at a higher resolution for a single peak to obtain high-quality data used to determine peak width and satisfy the third objective of this portion of the research. In the scope of this study, the (220) peak was analyzed over a smaller angle range around the expected Bragg angle of 74.7 degrees. The experimental data and associated curve fit is shown in Figure 5-2.

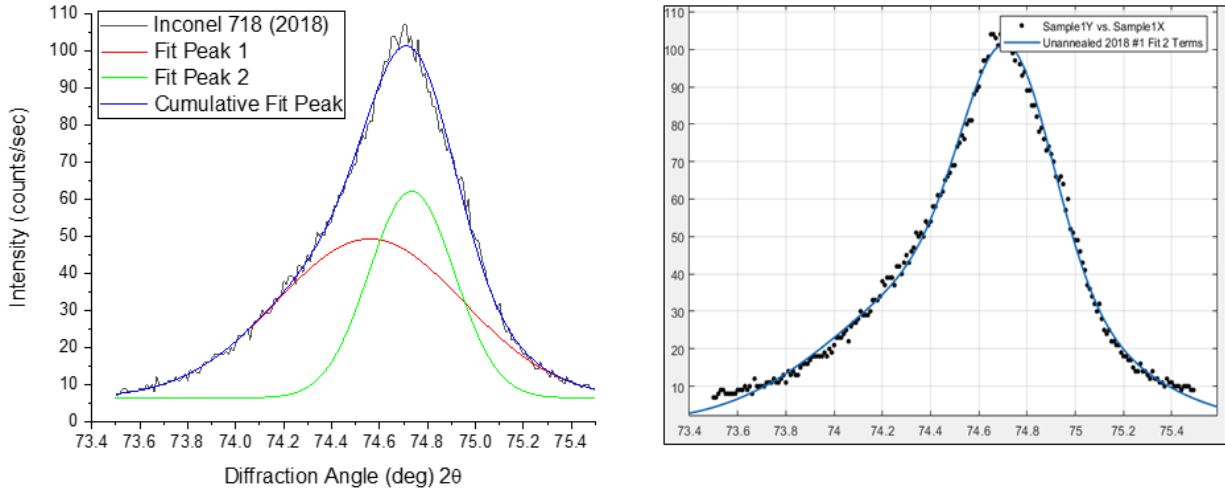


Figure 5-2: Inconel 718 45-53 μm particle size high-resolution diffractogram and peak fit.

The peak is evidently skewed, with the higher-angle side of the dataset exhibiting a steeper slope than the lower-angle side. A single Gaussian curve could not accurately represent this dataset. MATLAB software was employed to determine the best fit using a convolution of two Gaussian peaks. It was later discovered [96] the peak “doublet” was likely due to the $\text{Cu-K}\alpha 1$ and $\text{Cu-K}\alpha 2$ contributions, and this two-term Gaussian MATLAB fit was insufficient to capture peak width.

The Inconel 718 powder was subsequently annealed for one hour at 1,100°C and 1,200°C to accomplish the fourth objective of this portion of the research. The results compared to the baseline, as-atomized powder for 45-53 μm particle size is shown for low-resolution and high-resolution scans in Figure 5-3 and Figure 5-4, respectively. These experiments correspond to Test IDs 2-4 of TABLE IV.

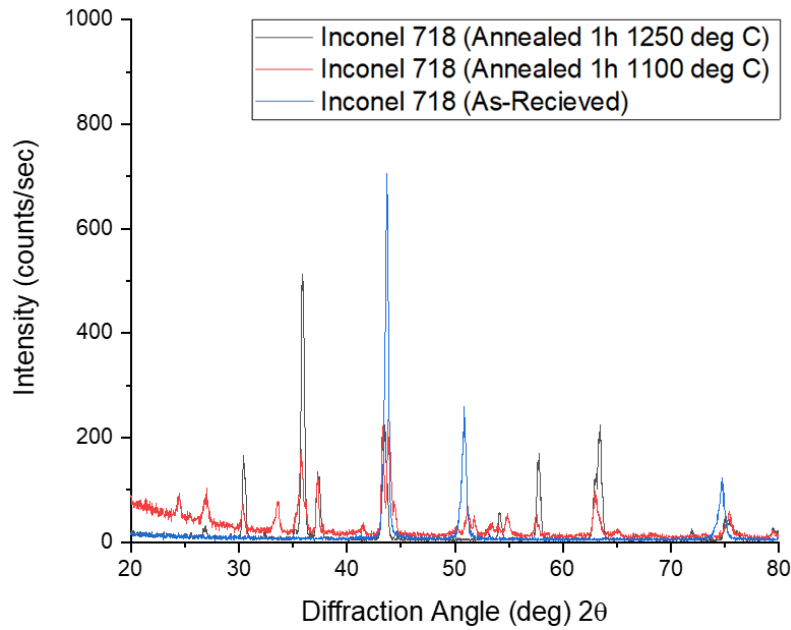


Figure 5-3: Inconel 718 low-resolution diffraction scan for as-atomized and annealed samples.

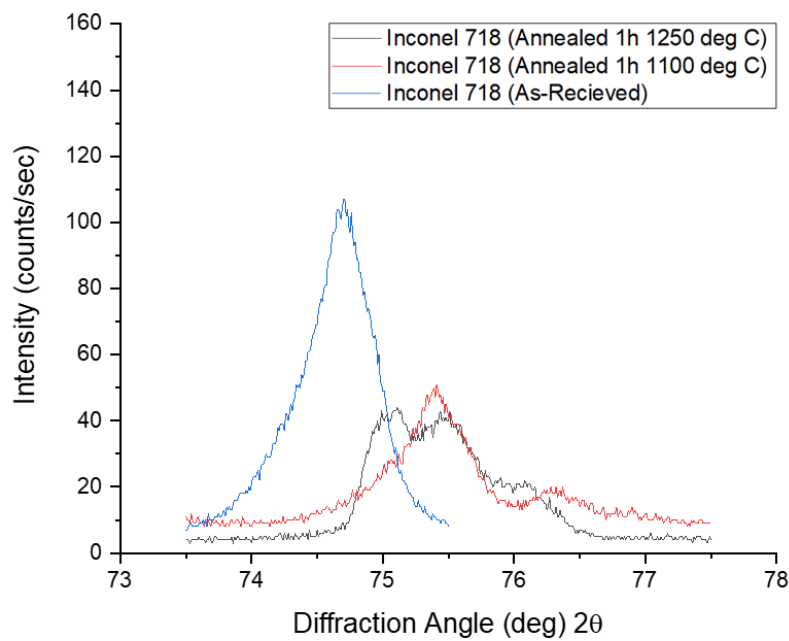


Figure 5-4: Inconel 718 high-resolution diffraction scan for as-atomized and annealed samples.

The applied heat treatment did not have the intended effect, which, at the time, was hypothesized to be a broadening of the Bragg peak observed at a similar angle. It was concluded [95] that improper heat treatment formed precipitates, and no further analysis was completed.

The next step in this research [96], utilized improved instrumentation [97] and additional Inconel 718 powder particle sizes to achieve several objectives:

1. Utilize calibration methods to remove instrumental broadening effects.
2. Identify dislocation density using line profile analysis of X-ray diffraction data.
3. Observe the trend in dislocation density between larger and smaller particles.
4. Compare the variation in outcomes obtained using different diffractometers.

The first objective was accomplished by comparing new data obtained using a PANalytical X'Pert Pro MPD X-ray diffractometer system at the Massachusetts Institute of Technology with data obtained using a Phillips PW 3020 X'Pert diffractometer system at Tufts University. Lanthanum hexaboride calibration samples were analyzed to remove instrument effects and provide a means of comparing the two systems, as shown in Figure 5-5.

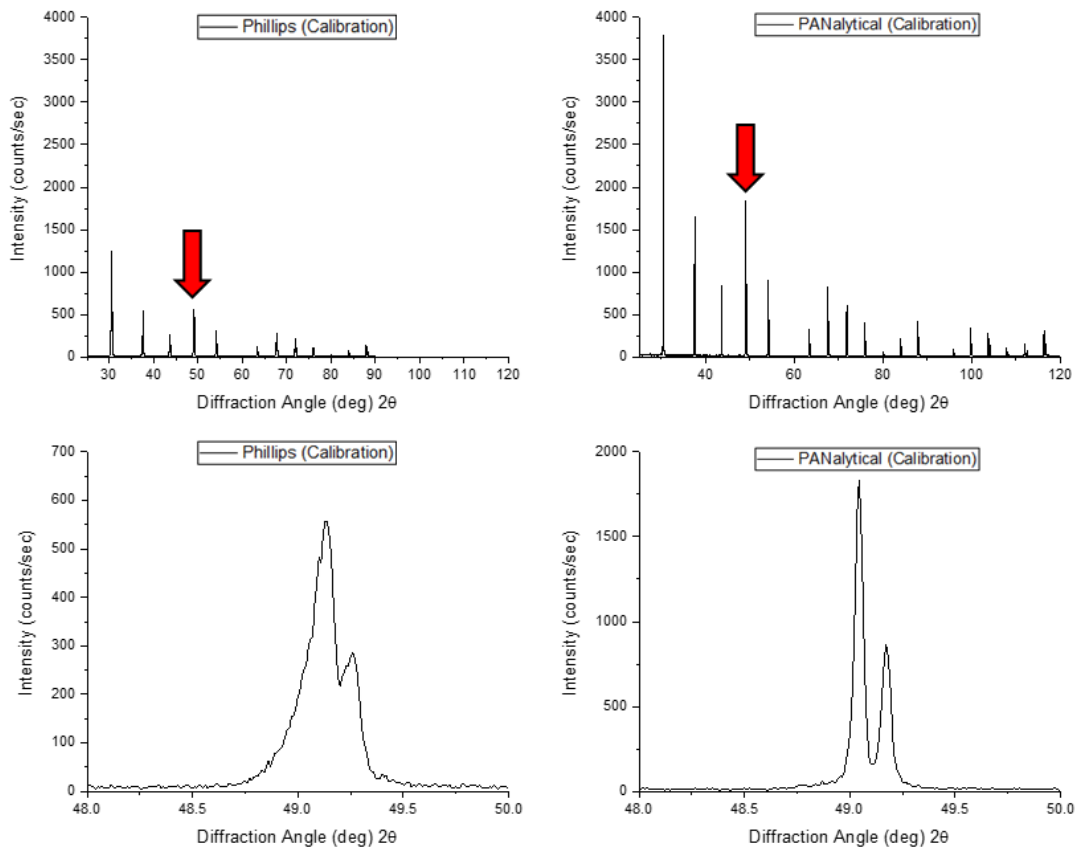


Figure 5-5: Comparison of PANalytical and Phillips diffractometers using calibration scan.

The primary advantages of the PANalytical system were a superior signal with improved resolution over a greater angle range. The increased angle range provides access to additional high-angle Bragg peaks, as shown in Figure 5-6. The improved resolution shown in Figure 5-7 is exemplified with a more apparent distinction of the individual Cu-K α contributions, indicating improved data quality. This improvement in instrumentation was quantified using Rietveld refinement techniques to determine measurement bias and precision [97], as shown in TABLE XIII.

TABLE XIII: Comparison of Diffractometer Performance Metrics

Facility	Average Lattice Parameter (Å)	Diffractometer Measurement Bias (Å)	Standard Deviation [Precision] (Å)
NIST Standard	4.1568	N/A	N/A
Phillips	4.1566	$-1.5490 \cdot 10^{-3}$	$6.1000 \cdot 10^{-5}$
PANalytical	4.1553	$-1.7633 \cdot 10^{-4}$	$7.6917 \cdot 10^{-5}$

Experiments were conducted at both facilities using two different particle size ranges with diameters of 20-25 μm and 38-45 μm ; these experiments correspond to Test IDs 4-6 of TABLE IV. The dislocation density results are presented in TABLE XIV and Figure 5-6. A Gaussian distribution was assumed between neighboring sieve sizes to calculate the error for particle size. This method is used for the error distribution of particle size for all materials.

TABLE XIV: Inconel 718 Dislocation Density Results

Facility	Particle Diameter D (m) $\times 10^{-6}$	Dislocation Density ρ (m ⁻²) $\times 10^{14}$
Phillips	22.5 ± 1.4	3.0 ± 0.3
Phillips	41.5 ± 2.0	1.4 ± 0.1
PANalytical	22.5 ± 1.4	4.7 ± 0.4
PANalytical	41.5 ± 2.0	4.0 ± 0.3

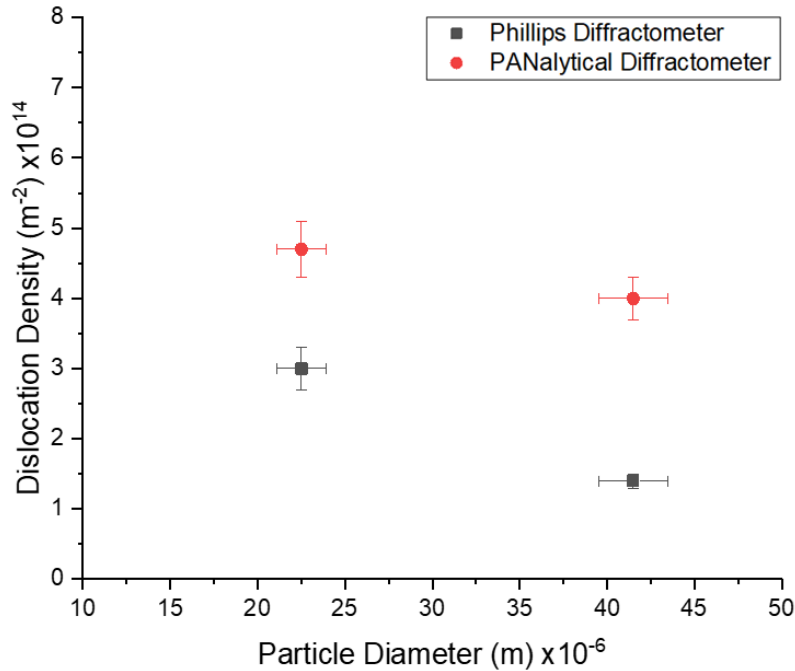


Figure 5-6: Inconel 718 as-atomized dislocation density results using two different diffractometers.

The dislocation density results for this research segment address objectives two through four. Improved peak analysis techniques were utilized to accomplish the line profile analysis of the data, as described in Section 4.1. Results from both facilities indicate a trend of decreasing dislocation density with increasing particle size. Additionally, the results were within the same order of magnitude as the published literature for similarly sized particles [56], albeit for a different material. The smaller reported error range of the Phillips diffractometer results is unexpected since it has been claimed to be a lower quality diffractometer; however, this can be attributed to the fact that a smaller number of Bragg peaks were accessible for this facility. Within the angular ranges of the diffractometers, the Phillips facility could access only three Bragg peaks, while the PANalytical facility could access five peaks. Less data leads to an artificially “better” fit. Furthermore, a sensitivity study of the PANalytical facility only using three peaks was reported [97].

5.1.2 Electron Back Scatter Diffraction

EBSD analysis was completed for two as-atomized particle size ranges of 20-25 μm and 53-63 μm ; the results are presented in Figure 5-7 and TABLE XV. The misorientation angle was fit using a log-normal distribution described in Section 4.2.1.

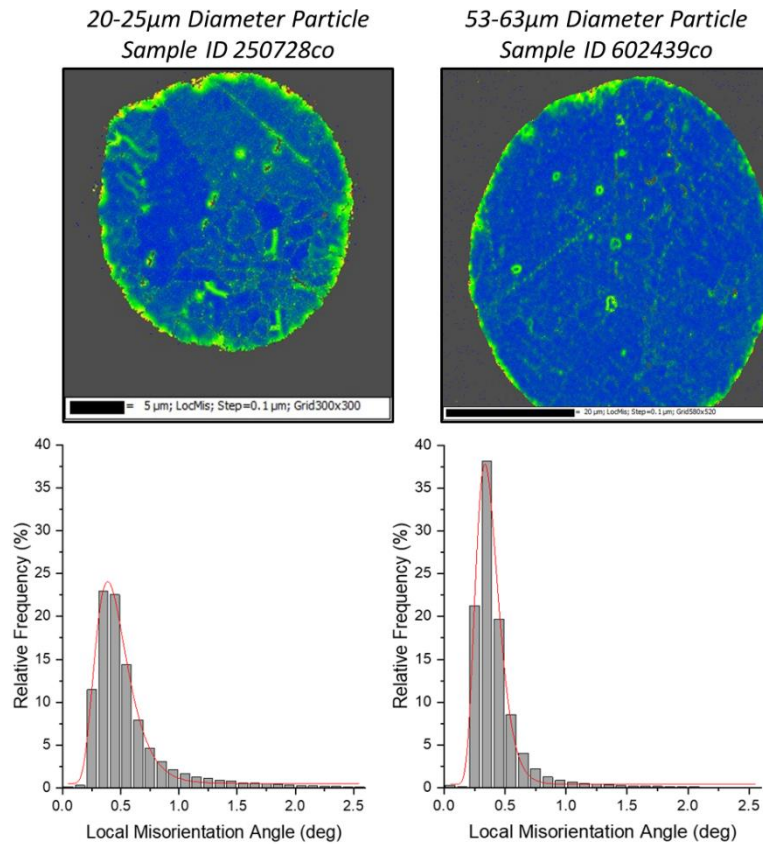


Figure 5-7: Inconel 718 powder 20-25 μm and 53-63 μm particle size EBSD local misorientation.

TABLE XV: Inconel 718 EBSD Misorientation Angle Results

Scan ID	Particle Diameter (m) $\times 10^{-6}$	Step Size (m) $\times 10^{-6}$	Grid Size (pixels)	Kernal Radius (m) $\times 10^{-6}$	Average Misorientation (deg)
250728co	22.5 ± 1.4	0.1	7x7	0.35	$0.503^{+0.340}_{-0.203}$
602439co	58.0 ± 2.9	0.1	7x7	0.35	$0.396^{+0.245}_{-0.152}$

Only observations using the same Kernel radius can be compared to examine trends in dislocation density, due to the limitation noted in Section 4.2.2. Comparing scans for the smaller

particle range (ID 250728co) and the large particle size range (ID 602439co), a trend of decreasing misorientation angle with increasing particle size is observed. Since dislocation density is proportional to misorientation angle, a qualitative observation of decreasing dislocation density with increasing particle size can be made again, similar to the observation identified from the XRD data. There is a significant error range in the EBSD calculation, so this is only valid as an approximation of the any dislocation density trend.

5.1.3 Cooling Rate Approximation

The model-based cooling rate equation determined in Section 4.3.1 was evaluated for Inconel 718 to derive Equation (5.1). The equation was evaluated for the various particle sizes in TABLE XVI and presented in Figure 5-8. Secondary dendrite arm spacing was determined using surface cell morphology from SEM micrographs. Cooling rates were calculated using the equation defined in Section 4.3.2 using coefficients from published work, as shown in Figure 5-9. The model-based cooling rate determined from particle size is then compared to the cooling rate determined from cell size.

$$\frac{dT}{dt} = (1.30615 * 10^{-4}) \cdot D^{-1.9792} \quad (5.1)$$

TABLE XVI: Inconel 718 Calculated Cooling Rate and Apparent Cell Size

Particle Diameter $D \text{ (m)} \times 10^{-6}$	Cooling Rate $\frac{dT}{dt} \text{ (Ks}^{-1}\text{)} \times 10^4$	Apparent Cell Size $d \text{ (m)} \times 10^{-6}$
22.5 ± 1.4	20.7 $^{+2.8}_{-2.3}$	1.0 ± 0.3
28.5 ± 2.0	13.0 $^{+2.0}_{-1.6}$	1.3 ± 0.4
35.0 ± 1.7	8.7 $^{+0.9}_{-0.8}$	1.8 ± 0.7
41.5 ± 2.0	6.2 $^{+0.6}_{-0.5}$	1.6 ± 0.4
49.0 ± 2.3	4.5 $^{+0.4}_{-0.4}$	2.0 ± 0.6
58.0 ± 2.9	3.2 $^{+0.3}_{-0.3}$	3.0 ± 1.1

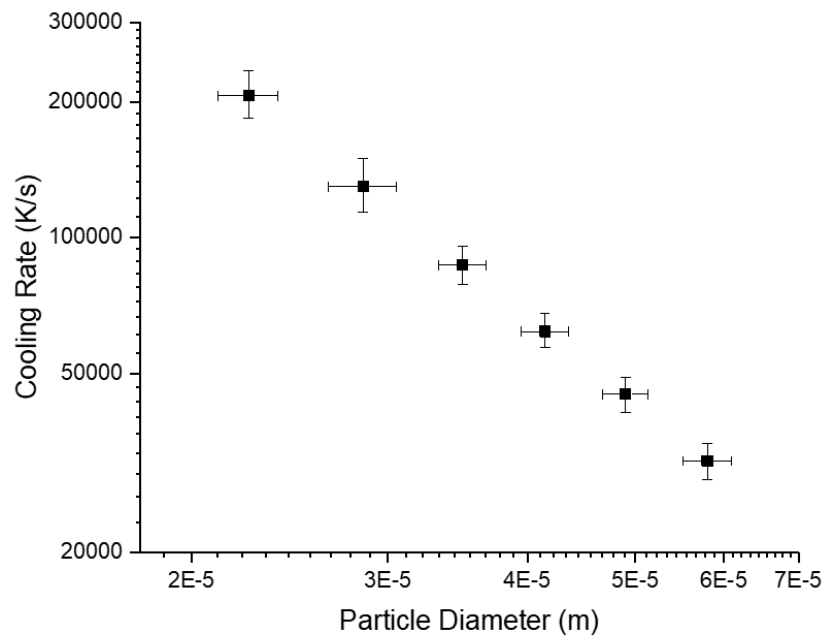


Figure 5-8: Inconel 718 model-based cooling rate prediction for all particle sizes.

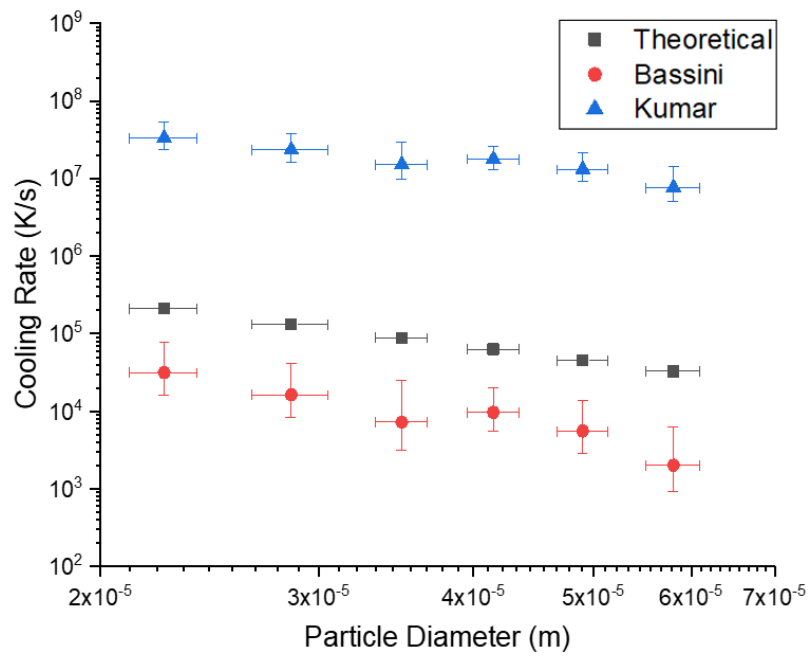


Figure 5-9: Inconel 718 predicted cooling rate results using cell size for all particle sizes.

There is significant variation in the reported cooling rate values for Inconel 718 estimated using cell size based on the small amount of published literature on the topic. While these approximations vary significantly, this finding does provide a small amount of reassurance given the model-based approach is enveloped by the results from published literature. It should be noted that these correlations depend on solidification conditions and may vary significantly depending on the powder production method. These factors and other sources of error are discussed in Section 5.6.

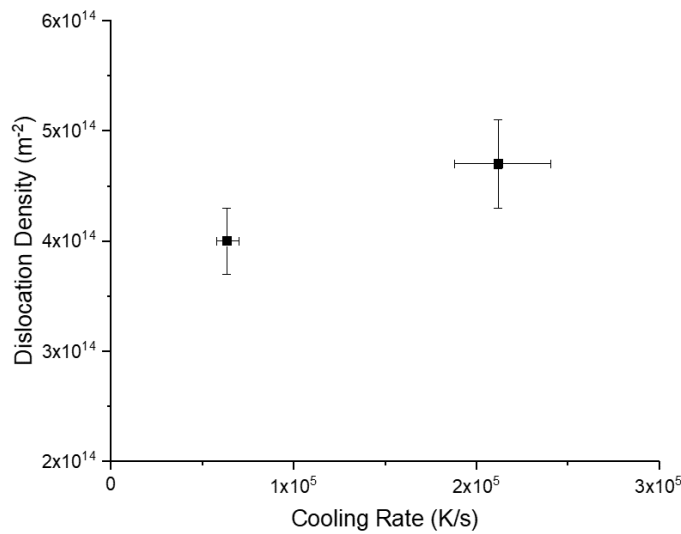


Figure 5-10: Inconel 718 dislocation density as a function of predicted cooling rate.

There are insufficient results for Inconel 718, as shown in Figure 5-10, to provide a quantitative correlation between dislocation density and solidification history. However, the cooling rate for smaller particle sizes is higher than for larger particle sizes due to the inverse relationship between particle diameter and cooling rate. A tentative qualitative conclusion based on these results alone is that dislocation density increases as the cooling rate increases.

5.2 Al2024 Results

The observed correlation between dislocation density and solidification history in Inconel 718 prompted the extension of research to another model material: Al2024. To strengthen the conclusions of this experimental effort, a broader range of particle sizes was used, and the material was obtained in both as-quenched and annealed states.

5.2.1 X-Ray Diffraction

Before further experimentation, particles with a diameter range of 20-25 μm were analyzed using the PANalytical diffractometer, as shown in Figure 5-11. The objective of this experiment, corresponding to Test ID 7 of TABLE IV, was to confirm that the Bragg peaks appeared at the expected positions and that the diffraction data for the annealed samples was as anticipated. Preliminary qualitative analysis indicated that the peaks of the annealed samples were less broad than those of the as-quenched samples, suggesting a decrease in dislocation density.

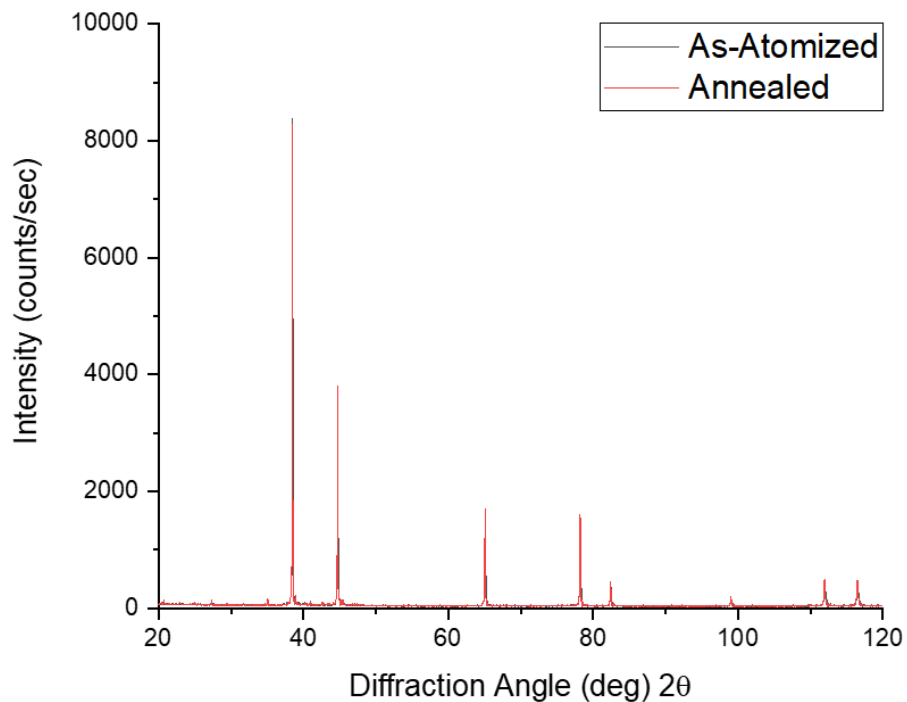


Figure 5-11: Al2024 low-resolution diffraction scan for as-atomized and annealed samples.

As a further improvement to this experiment, high-energy synchrotron X-ray diffraction was utilized to obtain even more Bragg peaks than either of the previous facilities, as shown in Figure 5-12. The proposal to the Advanced Photon Source of Argonne National Laboratory was accepted, and the corresponding experiments are identified as Test IDs 8-9 of TABLE IV.

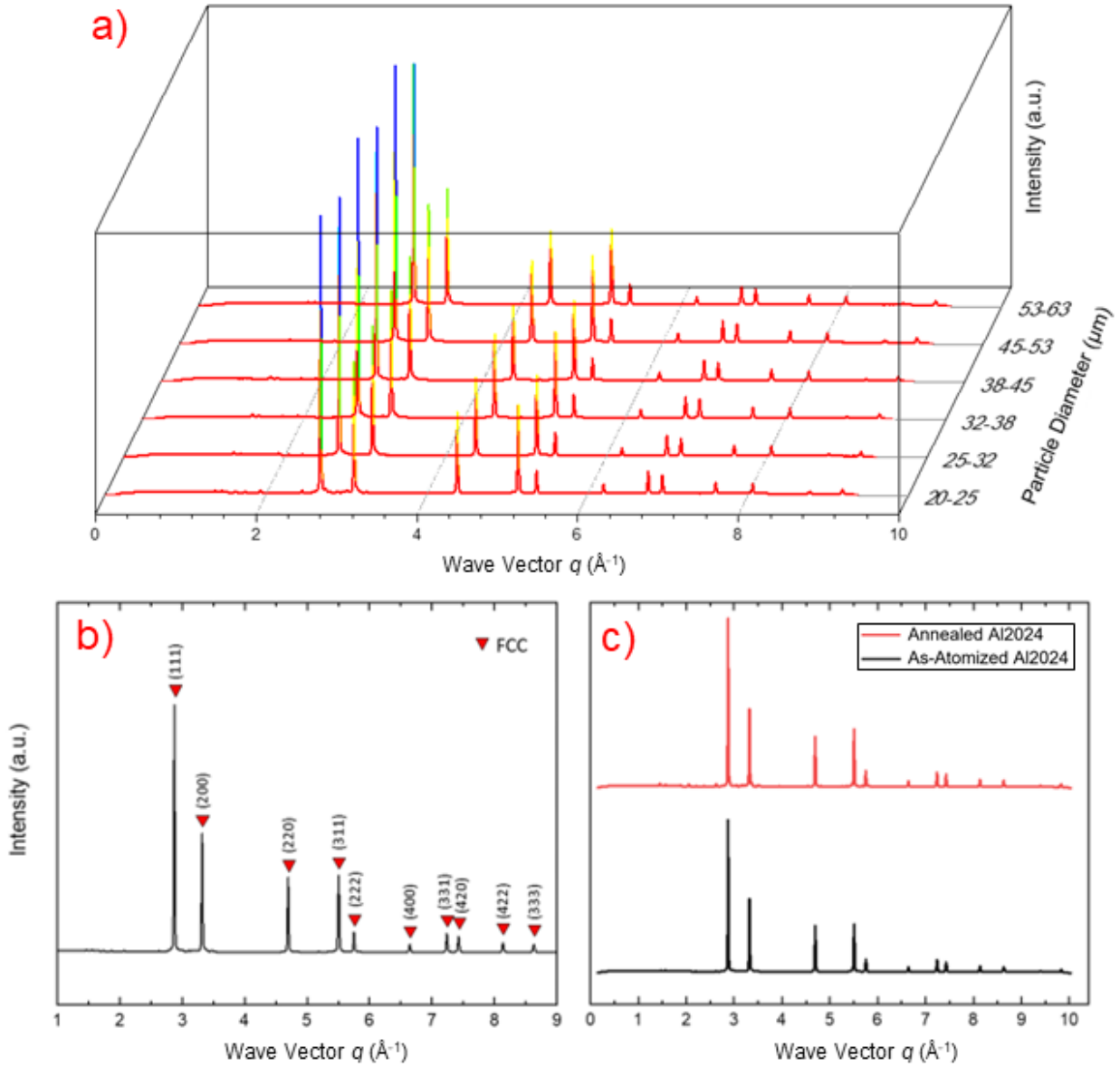


Figure 5-12: Al₂O₃ powder synchrotron diffraction results showing full-range diffractograms. a) as-atomized particles of all size ranges, b) as-atomized 20-25 μm particles, c) as-atomized and annealed 20-25 μm particles.

A curve-fit to a Bragg peak using the Voigt function is provided in Figure 5-13.

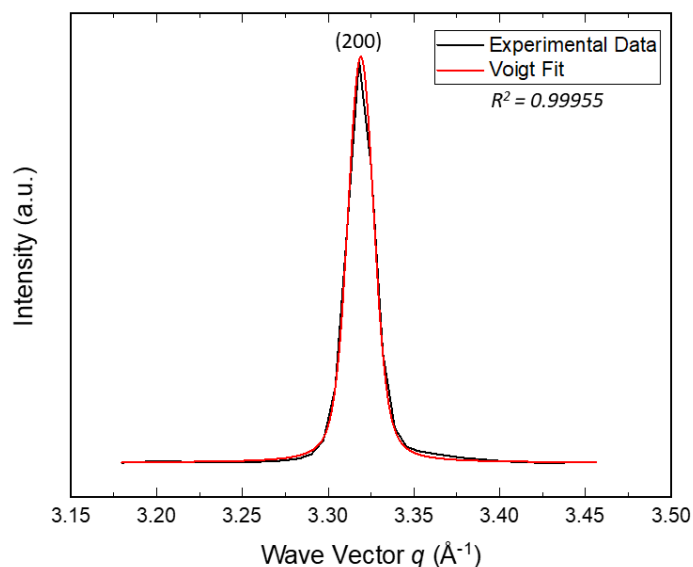


Figure 5-13: Al2024 powder 20-25 μm particle size Voigt curve fit to Bragg peak.

A Modified Williamson-Hall fit is provided in Figure 5-14. This plot highlights the difference in obtained peaks for different facilities: The Phillips diffractometer reaches the first three (111) (200) (220), the PANalytical diffractometer reaches the following two (222) (311), and high-energy synchrotron diffraction reaches the following five (400) (331) (420) (422) (333).

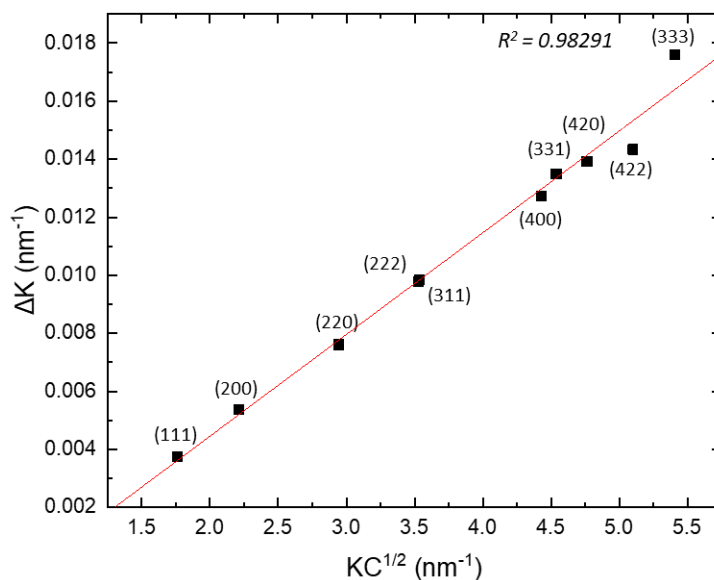


Figure 5-14: Al2024 powder 20-25 μm particle size Modified Williamson-Hall plot.

A Modified Warren-Averbach fit is provided in Figure 5-15.

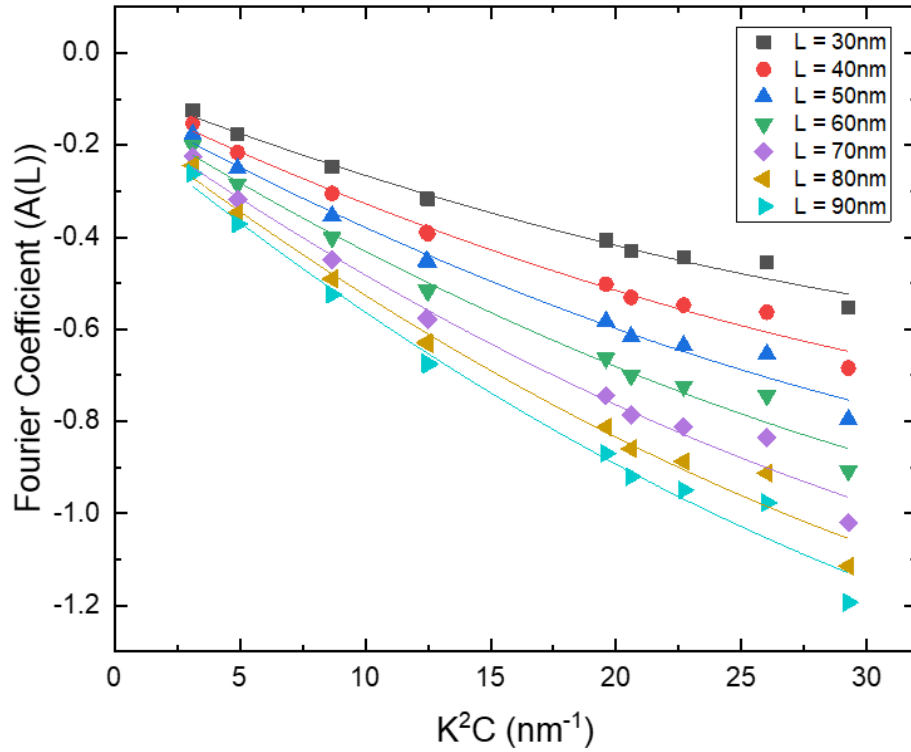


Figure 5-15: Al2024 powder 20-25 μm particle size Modified Warren-Averbach plot.

The obtained dislocation density results are provided in TABLE XVII and Figure 5-16.

TABLE XVII: Al2024 Dislocation Density Results

Particle Diameter $D \text{ (m)} \times 10^{-6}$	As-Atomized Sample Dislocation Density $\rho \text{ (m}^{-2}\text{)} \times 10^{14}$	Annealed Sample Dislocation Density $\rho \text{ (m}^{-2}\text{)} \times 10^{14}$
22.5 ± 1.4	7.4 ± 1.3	2.3 ± 0.4
28.5 ± 2.0	6.7 ± 1.2	2.2 ± 0.4
35.0 ± 1.7	5.3 ± 1.0	1.3 ± 0.2
41.5 ± 2.0	6.1 ± 1.1	4.0 ± 0.7
49.0 ± 2.3	5.0 ± 0.9	3.1 ± 0.6
58.0 ± 2.9	3.1 ± 0.6	2.0 ± 0.4

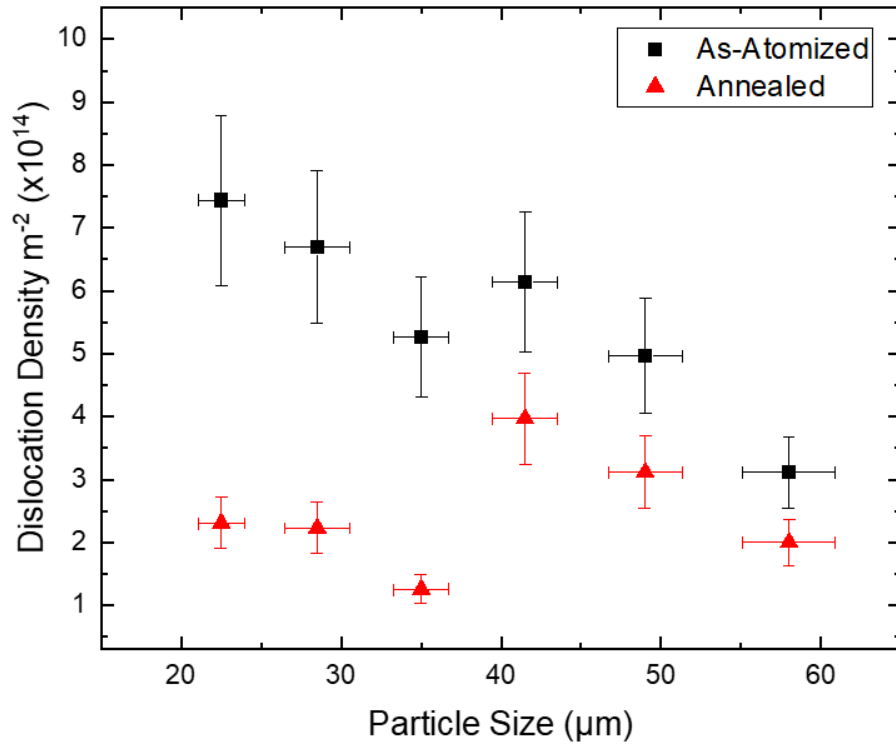


Figure 5-16: Al2024 as-atomized and annealed powder dislocation density results for all particle sizes.

The heat treatment of Al2024 powder samples allowed dislocations in the as-atomized microstructure to move and annihilate, reducing dislocation density. Figure 5-16 shows that the initial dislocation density varied with particle diameter due to different solidification histories. Annealing removed this trend by healing the dislocations. As-atomized Al2024 samples showed a more convincing trend of decreasing dislocation density with increasing particle size compared to Inconel 718 results. Additionally, the annealed Al2024 samples show little to no trend of dislocation density change across the span of particle sizes, despite their varying initial solidification histories during atomization.

5.2.2 Electron Back Scatter Diffraction

EBSD analysis was completed for three as-atomized particle size ranges 20-25 μm , 38-45 μm and 53-63 μm ; the results are presented in Figure 5-17 and TABLE XVIII. The misorientation angle was fit using a log-normal distribution described in Section 4.2.1. Additionally, a single annealed sample of particle size ranges 20-25 μm was analyzed as shown in Figure 5-18 and TABLE XIX.

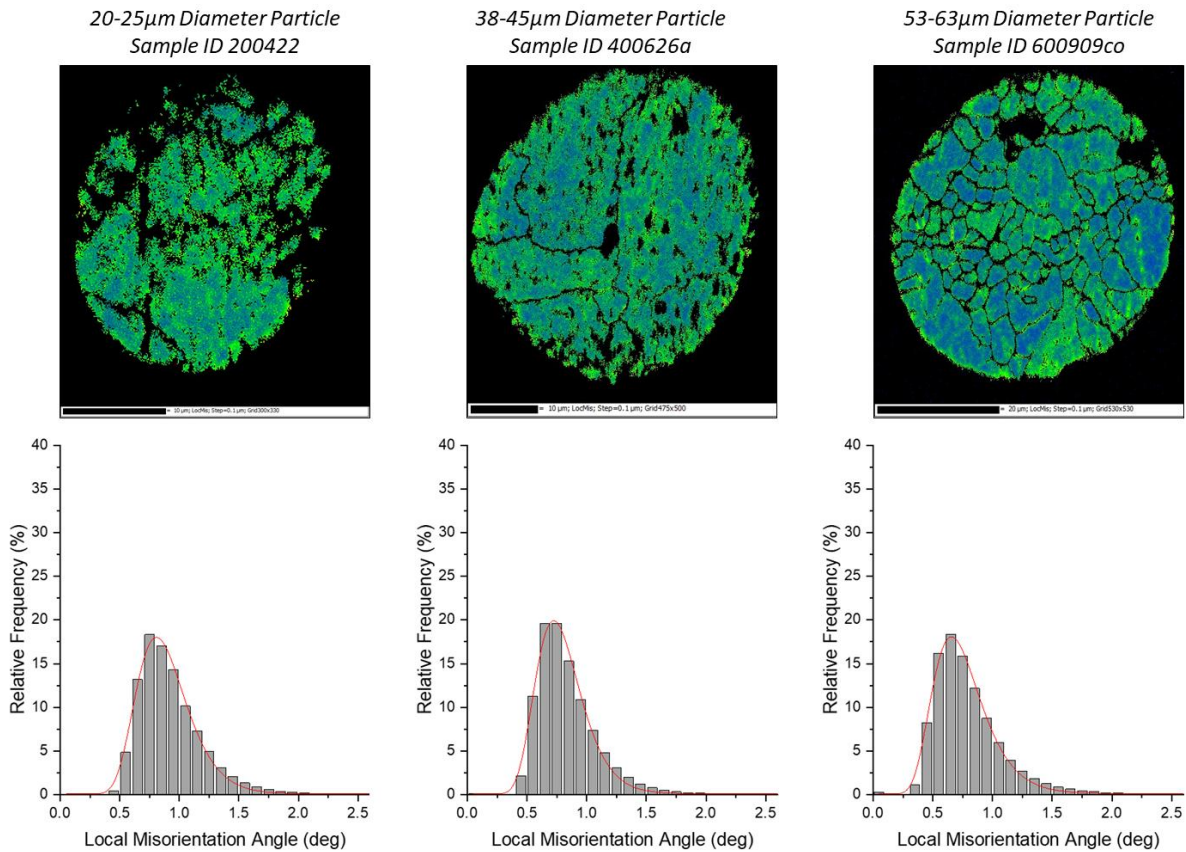


Figure 5-17: Al2024 particle size 20-25 μm , 38-45 μm and 53-63 μm EBSD and local misorientations.

TABLE XVIII: Al2024 EBSD Misorientation Angle Results

Scan ID	Particle Diameter $\text{m} \times 10^{-6}$	Step Size $\text{m} \times 10^{-6}$	Grid Size (pixels)	Kernal Radius $\text{m} \times 10^{-6}$	Average Misorientation (deg)
200422	22.5 ± 1.4	0.10	7x7	0.35	$0.897^{+0.356}_{-0.255}$
400626a	41.5 ± 2.0	0.10	7x7	0.35	$0.806^{+0.290}_{-0.213}$
600909co	58.0 ± 2.9	0.10	7x7	0.35	$0.750^{+0.338}_{-0.233}$

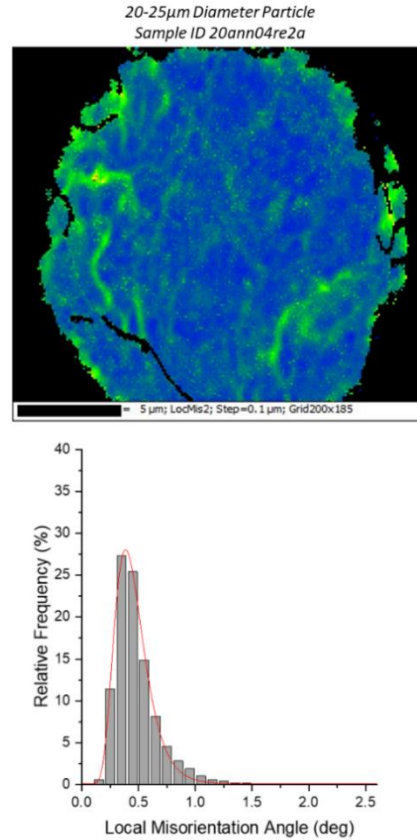


Figure 5-18: Al2024 annealed particle size and local misorientation results.

TABLE XIX: Annealed Al2024 EBSD Misorientation Angle Result

Scan ID	Particle Diameter $\text{m} \times 10^{-6}$	Step Size $\text{m} \times 10^{-6}$	Grid Size (pixels)	Kernal Radius $\text{m} \times 10^{-6}$	Average Misorientation (deg)
20ann04re2a	22.5 ± 1.4	0.10	5x5	0.25	$0.451^{+0.319}_{-0.187}$

The density of GNDs was calculated for the largest particle size range 53-63 μm (IDs 600606co-a and 600606co-b) using a Burgers vector for Al2024 of $2.86 \times 10^{-10} \text{ m}$, as provided in TABLE XX. The grid sampling grid size was changed from 3x3 to 7x7 pixels for this observation which enabled this calculation. The difference between observed misorientation angle using a different grid size is evident in Figure 5-19 as a change in color from blue to green/yellow.

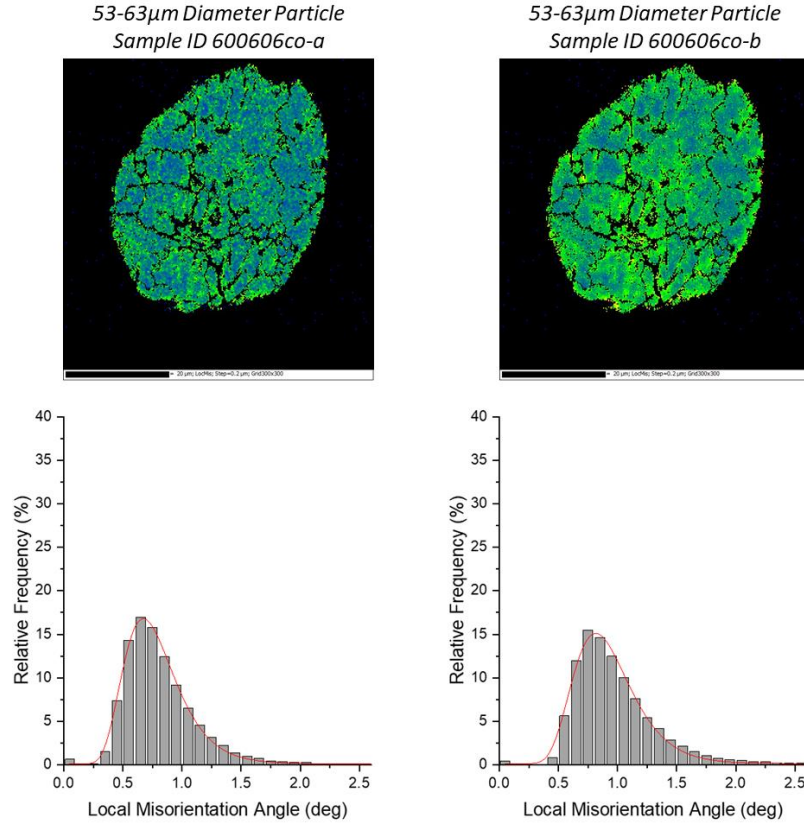


Figure 5-19: Al2024 EBSD local misorientation angle results with varying Kernel radius.

TABLE XX: Al2024 EBSD GND Density Approximation

Scan ID	Particle Diameter (m) x 10 ⁻⁶	Step Size (m) x 10 ⁻⁶	Grid Size (pixels)	Kernal Radius (m) x 10 ⁻⁶	Average Misorientation (deg)	ρ_{GND} (m ⁻²) x 10 ¹⁴
600606co-a	58.0 ± 2.9	0.20	3x3	0.35	0.761 ^{+0.392} _{-0.259}	0.25
600606co-b	58.0 ± 2.9	0.20	7x7	0.70	0.926 ^{+0.509} _{-0.329}	

The result of $\rho_{GND} = 0.25 * 10^{14} \text{ m}^{-2}$ is expectedly lower than the associated $\rho_{total} = 3.1 * 10^{14} \text{ m}^{-2}$ result obtained using synchrotron X-ray diffraction. EBSD evaluations of dislocation density do not incorporate statistically stored dislocations nor higher dislocation density regions near large-angle cell boundaries.

5.2.3 Cooling Rate Approximation

The model-based cooling rate equation determined in Section 4.3.1 was evaluated for Al2024 to derive Equation (5.2). The equation was evaluated for the various particle sizes in TABLE XXI and presented in Figure 5-20. Secondary dendrite arm spacing was determined using EBSD micrographs of polished and etched samples (see Figure 4-24 for reference). Cooling rates were calculated using the equation determined in Section 4.3.2 using coefficients from published work. The model-based cooling rate determined from particle size is then compared to the cooling rate determined from cell size.

$$\frac{dT}{dt} = (0.916349 * 10^{-4}) \cdot D^{-1.9991} \quad (5.2)$$

TABLE XXI: Al2024 Calculated Cooling Rate and Apparent Cell Size

Particle Diameter D (m) $\times 10^{-6}$	Cooling Rate $\frac{dT}{dt}$ (Ks⁻¹) $\times 10^4$	Apparent Cell Size d (m) $\times 10^{-6}$
22.5 $\pm$ 1.4	17.9 $^{+2.5}_{-2.0}$	1.6 $\pm$ 0.6
28.5 $\pm$ 2.0	11.2 $^{+1.7}_{-1.4}$	1.7 $\pm$ 0.7
35.0 $\pm$ 1.7	7.4 $^{+0.8}_{-0.7}$	1.9 $\pm$ 0.8
41.5 $\pm$ 2.0	5.3 $^{+0.5}_{-0.5}$	1.8 $\pm$ 0.7
49.0 $\pm$ 2.3	3.8 $^{+0.4}_{-0.3}$	1.8 $\pm$ 0.7
58.0 $\pm$ 2.9	2.7 $^{+0.3}_{-0.3}$	3.2 $\pm$ 1.3

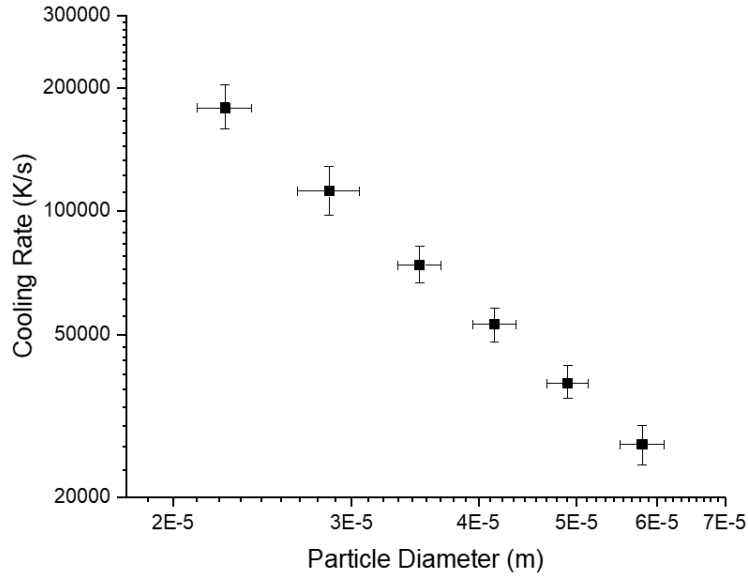


Figure 5-20: Al2024 model-based cooling rate prediction for all particle sizes.

The model-based cooling rate prediction is compared to published literature applicable to similar cooling rates, as provided in Section 4.3.2. The comparison was produced with linear fits for ease of viewing, as shown in Figure 5-21. However, the linear fits do not capture the non-linear cell size dataset or the non-linear error bars associated with the correlation.

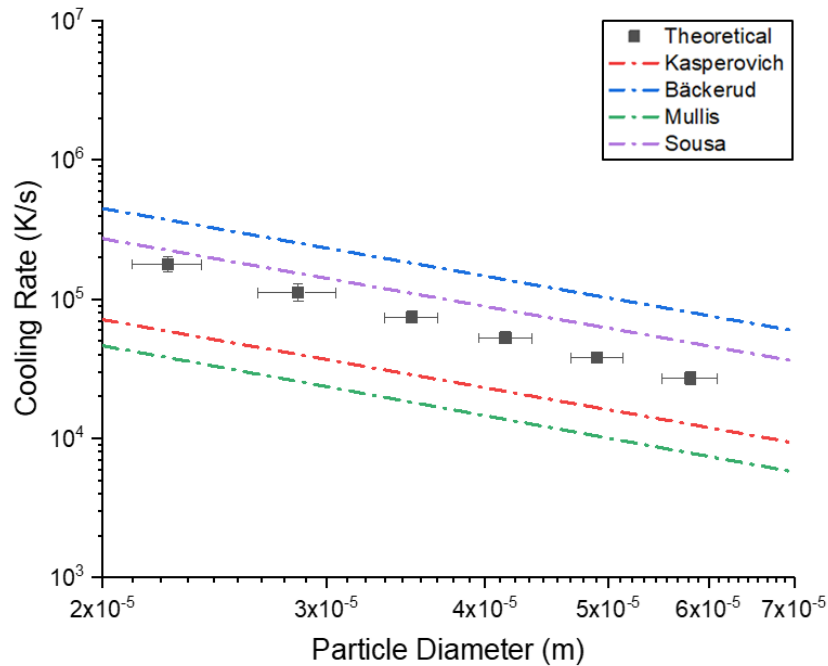


Figure 5-21: Al2024 cooling rate predictions using published empirical relationships.

When considering all the published correlations between secondary dendrite arm spacing and cooling rate, it is clear there is a significant variance. The correlation depends on solidification conditions, which likely vary widely between experiments. However, the model predictions appear well-enveloped between conclusions from other published work, specifically the parameters published by Sousa et al. [76], appear to correlate the most closely with the data, as shown in Figure 5-22. Since the Al2024 samples used in this experiment were manufactured in the same facility that published this research, the solidification conditions were likely remarkably similar. However, this result does imply that the cell size estimation was accurate and that the model-based cooling rate approach is valid since it was anchored using physical observations.

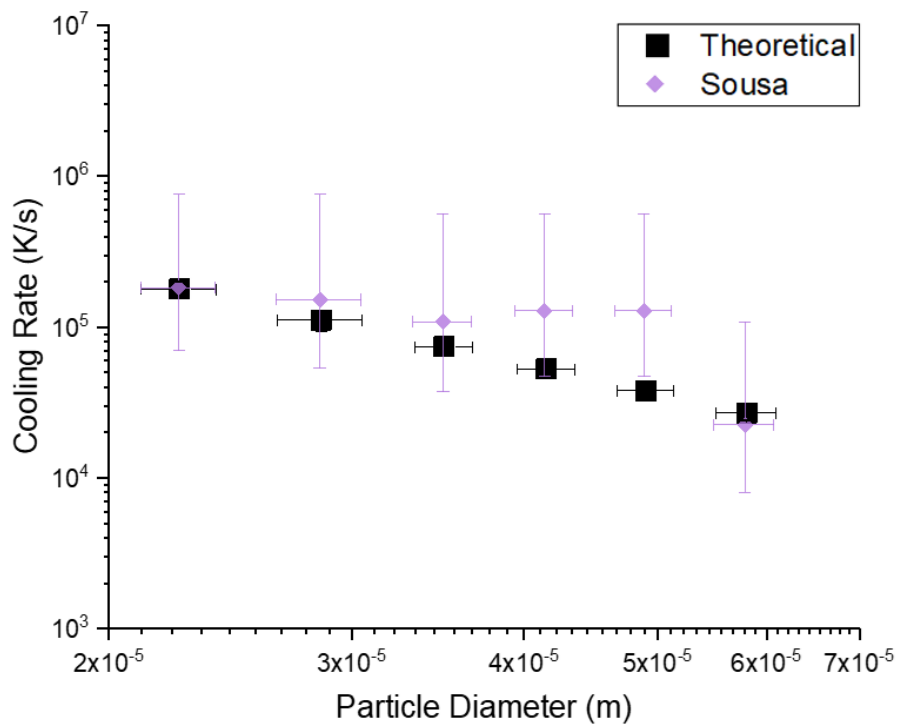


Figure 5-22: Al2024 cooling rate prediction compared to best-fit literature empirical relationship.

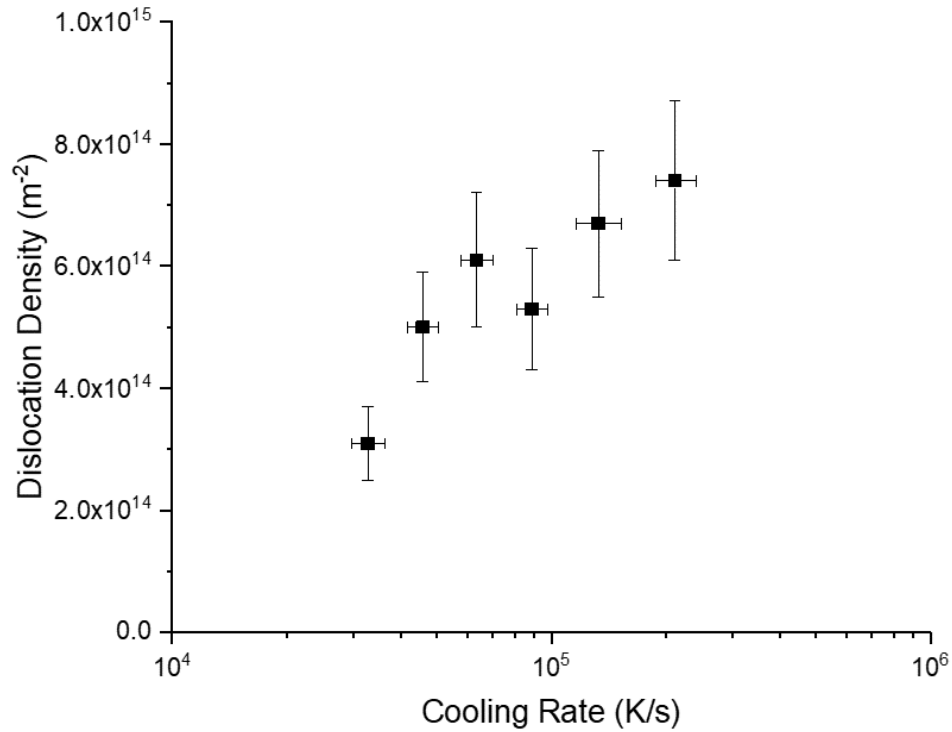


Figure 5-23: Al2024 dislocation density vs. cooling rate displayed on a semi-log plot.

The dislocation density versus cooling rate of the Al2024 particles is presented in a semi-log plot shown in Figure 5-23. This form was chosen to reflect the exponential nature of Newton's Law of Cooling. The plot demonstrates that as the cooling rate increases, the dislocation density also increases. This trend can be attributed to higher cooling rates, resulting in higher thermal gradients and increased thermal stress in the material during solidification. Increased stress promotes the formation of more dislocations in the crystal lattice of the material as a higher number of atoms do not find their place in the lattice during rapid solidification. Understanding this relationship is crucial for industry to predict material properties from solidification history, especially for rapid solidification use cases such as additive manufacturing or welding.

5.3 Fe-Al Results

The observed correlation between dislocation density and solidification history in Al2024 prompted the extension of this research to another method of sample production. Instead of gas atomization, where thermal conditions are poorly controlled, impulse atomization methods of sample production were used. The model material Fe-Al was selected to continue this research and was obtained in as-atomized and annealed states.

5.3.1 X-Ray Diffraction

Before any X-ray diffraction data was collected for the Fe-Al samples, the sample holder capillaries and sealant material were verified to have an amorphous structure, which would not affect the results of the experiment; this corresponded to Test ID 10 of TABLE IV. Then, several attempts to measure these samples were made unsuccessfully, corresponding to Test IDs 11-14 of TABLE IV. Preliminary X-ray diffraction data was collected at the Leibniz Institute for Solid State and Materials Research (IFW Dresden) for as-atomized sample particle diameters of 200 μm , 300 μm , 500 μm , 800 μm , and 1,100 μm , corresponding to Test ID 15 of TABLE IV. This work was completed to verify the expected diffraction peak locations before future synchrotron X-ray diffraction measurements. The results for the 200 μm particle diameter are provided in Figure 5-24.

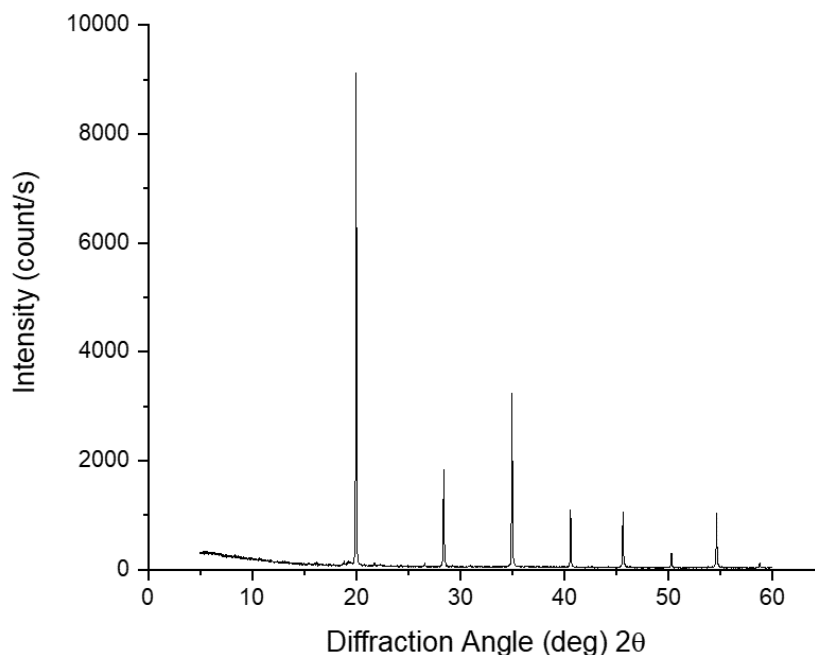


Figure 5-24: Fe-Al powder 200 μm particle size low-resolution diffractogram.

The observed diffraction peaks align with the expected diffraction angles for the BCC α -Fe phase. However, an initial observation of diffraction peak width yielded unexpected results. A comparison of peak width as a function of particle size for Fe-Al using the standard X-ray diffractometer and Al2024 using synchrotron diffraction is presented in Figure 5-25.

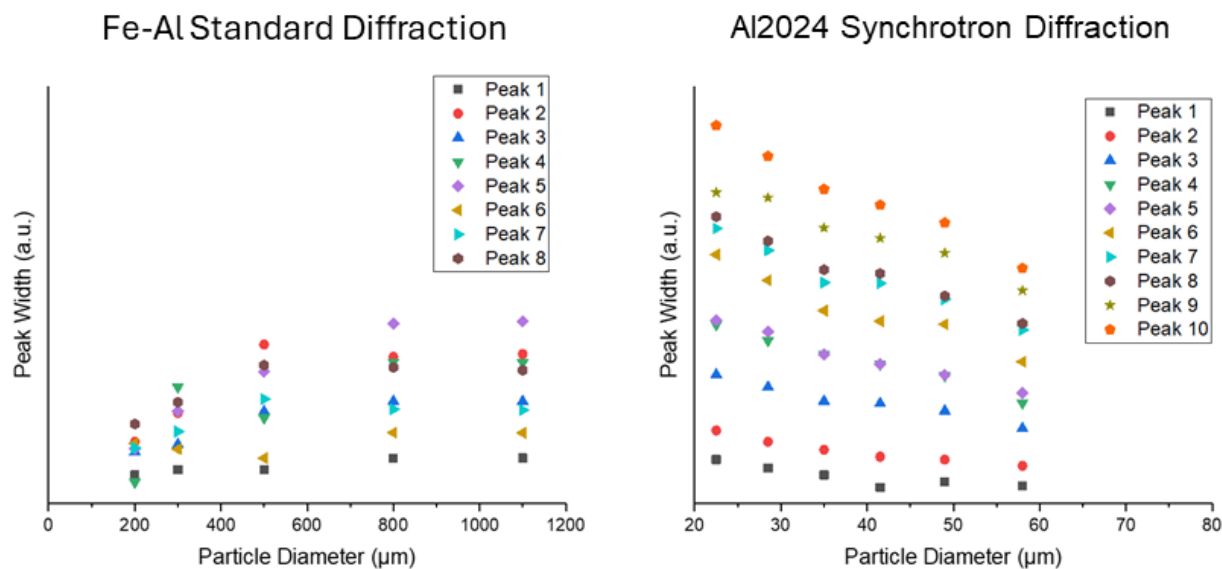


Figure 5-25: Fe-Al and Al2024 Bragg peak width comparison for all particle sizes.

Several new variables introduced in this experiment may have influenced the observed peak width trends. The most significant variable is the sample preparation for measurement. These particles are an order of magnitude larger than the powders used in previous experiments. Only a single layer of particles will fit when loaded into the sample holder, unlike bulk powder, which is consistently packed to the same thickness. This variation means that the distance some X-rays travel before being diffracted depends on the particle size. According to Bragg's law, this results in artificial broadening as a function of particle size. A geometric schematic illustrating this effect is shown in Figure 5-26 (note that this is not dependent on beam spot size).

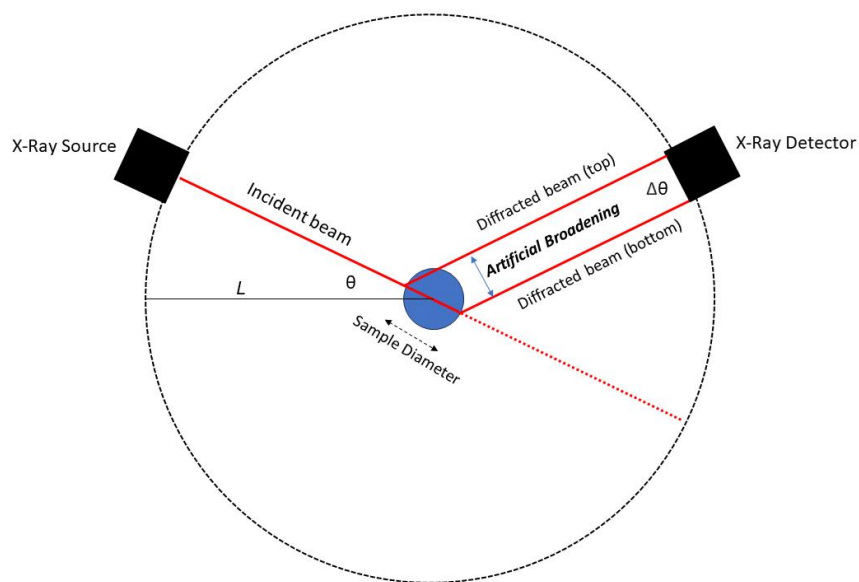


Figure 5-26: Simplified schematic of artificial broadening due to particle size.

While this may appear to be a quantifiable geometry problem, this schematic oversimplifies the process to explain why artificial broadening may happen. The actual interaction and geometry are significantly more complex. For example, the beamline is not infinitely thin; the spot size is focused on the sample center after the beam passes through several filters (Soller slits, divergence slits, detector slits, etc.). The broadening above the noise baseline is < 0.01 degrees. At best, even armed with precise measurements of every aspect of the experiment setup, it is unlikely that this

artificial broadening could be mathematically quantified through geometry alone. However, a qualitative relationship between particle diameter, incident angle, and associated artificial peak broadening can be hypothesized.

$$\Delta\theta \approx \tan^{-1} \left(\frac{D}{L} * \tan(\theta) \right) \quad (5.3)$$

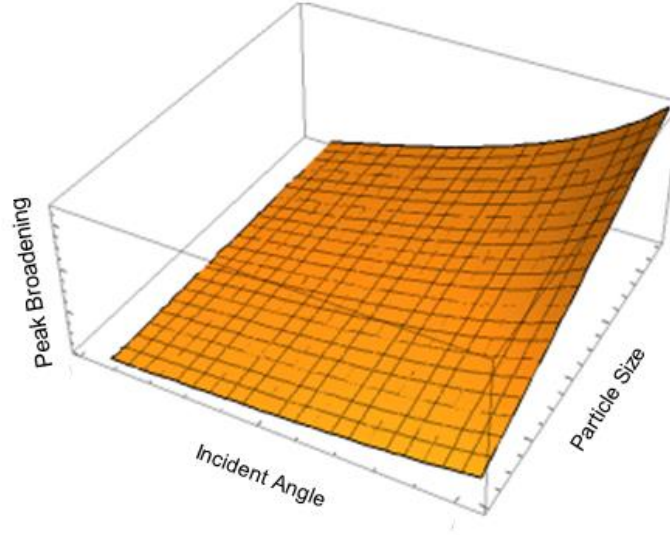


Figure 5-27: Speculative relationship between variables affecting artificial peak broadening.

Through inspection of the application of this qualitative relationship to the observed data, the results may form into an expected trend, but again, it is not possible to feasibly calculate a quantitative relationship for this type of laboratory diffractometer.

Next, following the hypothesis proposed in this work, these larger particles are expected to have a lower cooling rate and, therefore, a lower dislocation density. Relating this back to diffractometry, this would mean less broad diffraction peaks. Thinner diffraction peaks would require even more accurate measurements since the measurements would be close to the noise floor calculated from the calibration sample. Comparing the Al2024 and Fe-Al datasets, the peak widths of the Fe-Al are close to the corresponding LaB₆ noise floor. If the trend shown in Figure 5-27 was utilized to remove the artificial geometric broadening effect, the data may look similar

to the other results. However, guessing at a quantitative relationship would not provide meaningful results. Regardless of these newly introduced error sources, a dislocation density was still calculated for the samples, as shown in Figure 5-28.

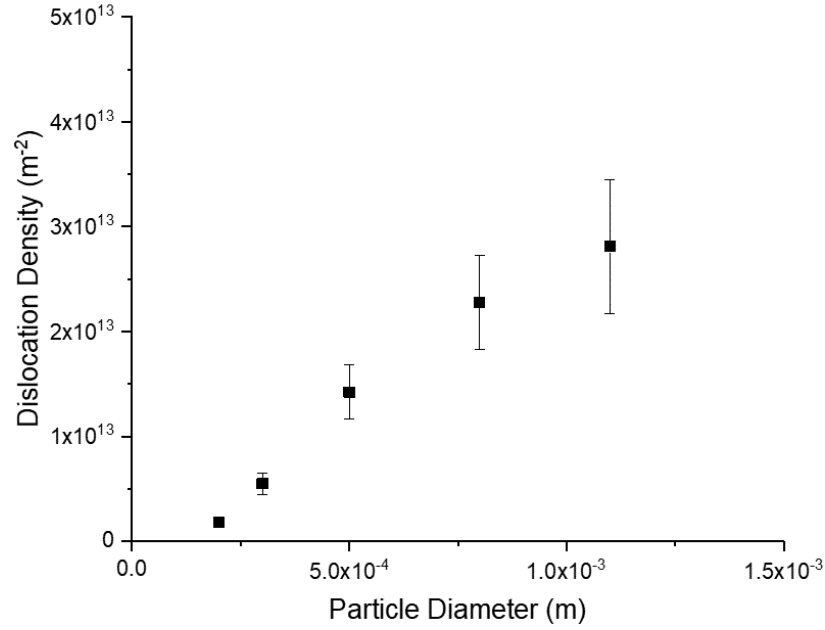


Figure 5-28: Fe-Al dislocation density results for all particle sizes.

The magnitude of observed dislocation densities is significantly lower than the results of the other smaller samples, which was expected. While the trend of dislocation density and particle size was initially unexpected, it is likely explained due to the particle size and geometrical limitations of the instrument used. Future experiments using synchrotron diffraction with a larger sample-detector distance are planned to address accuracy limitations and artificial broadening, corresponding to Test ID 15 of TABLE IV.

Due to the precise nature of impulse atomization, an assumption is made that the proposed particle diameter is correct, so there is no error range associated with the particle diameter. Future quantification of the accuracy of impulse atomization is planned but not within the scope of this work.

5.3.2 Cooling Rate Approximation

The model-based cooling rate equation determined in Section 4.3.1 was evaluated for Fe-Al samples to derive Equation (5.4). The equation was evaluated for the various particle sizes in TABLE XXII. Plans to perform metallography on the Fe-Al samples to approximate the cooling rate using dendrite arm spacing are in place, but this is not included in this scope of work.

$$\frac{dT}{dt} = (1.40283 \times 10^{-3}) \cdot D^{-1.96084} \quad (5.4)$$

TABLE XXII: Fe-Al Calculated Cooling Rate

Particle Diameter D (m) $\times 10^{-6}$	Cooling Rate $\frac{dT}{dt}$ (Ks^{-1}) $\times 10^4$
200	2.50
300	1.13
500	0.424
800	0.175
1100	0.0978

The predicted cooling rate of the Fe-Al samples is significantly lower than the associated cooling rates of the other samples of this study. This is expected primarily due to their significantly larger size and secondarily because they are cooling in helium, which has a higher thermal conductivity.

5.4 Comparative Analysis

The as-atomized samples of both Al2024 and Inconel 718 show similar trends in dislocation density as a function of particle size and cooling rate, as shown in Figure 5-29. Only the dislocation density results for Inconel 718 using the higher-quality diffractometer were compared. While merely an approximation of dislocation density trends, the misorientation angle can also be compared between materials if the angle is normalized by the Burgers vector.

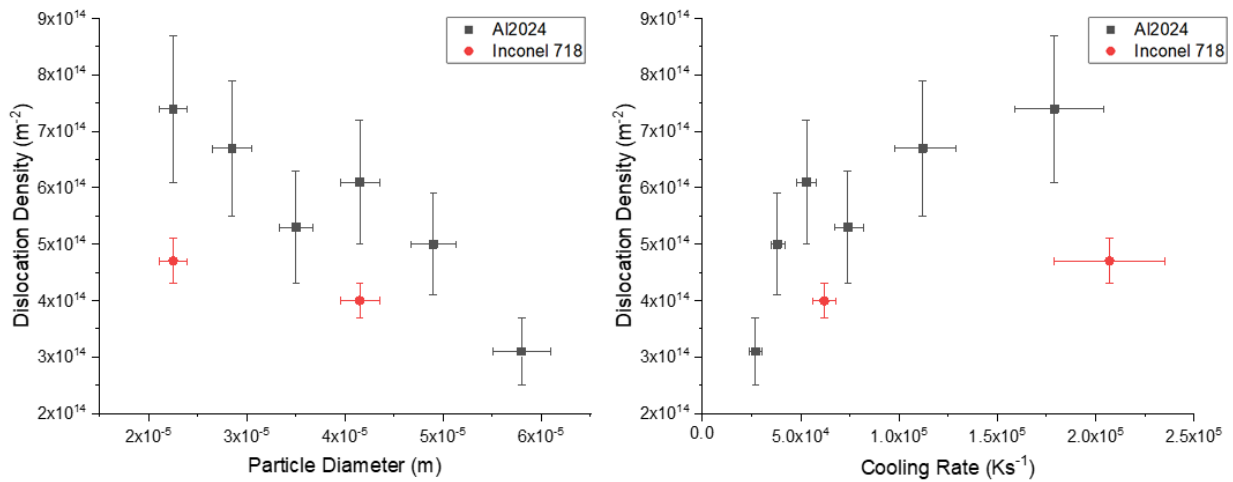


Figure 5-29: Al2024 and Inconel 718 as-atomized samples dislocation density comparison.

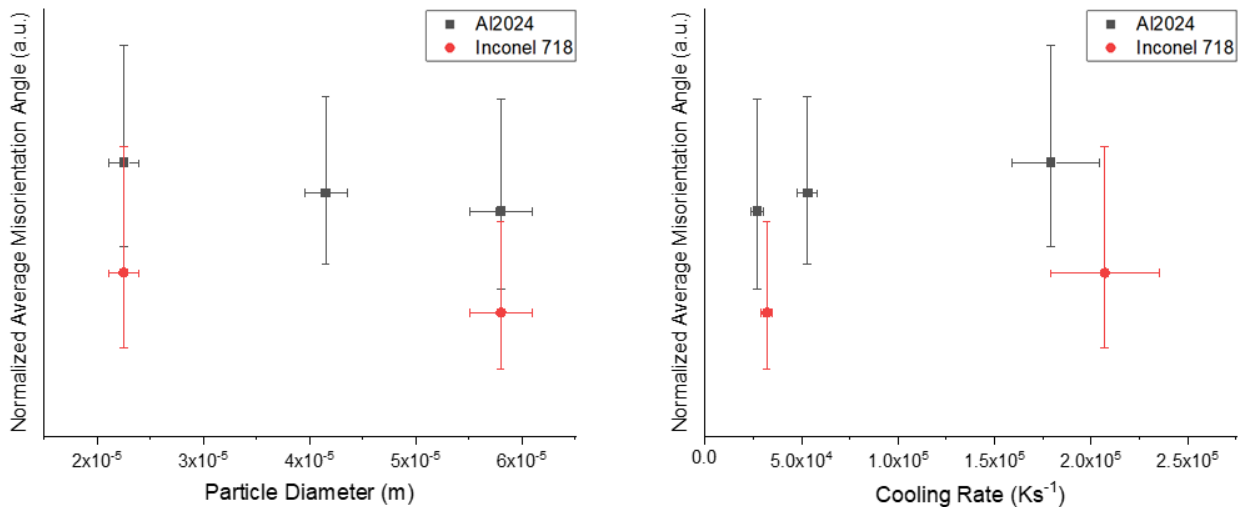


Figure 5-30: Al2024 and Inconel 718 comparison of normalized misorientation angles.

In both Al2024 and Inconel 718, a trend of increasing dislocation density is observed with decreasing particle diameter and increasing cooling rate, as shown in Figure 5-29. Using approximation methods, a similar trend (with expectedly large error bars) is observed with misorientation angle through EBSD, as shown in Figure 5-30. The misorientation angle is proportional to dislocation density, so the trend is in accordance with the results obtained through X-ray diffraction.

While the dislocation density trends for Fe-Al, as shown in Figure 5-31, are skewed due to the artificial broadening effect discussed earlier, the order of magnitude of the results can still be compared to those from Al2024 and Inconel 718.

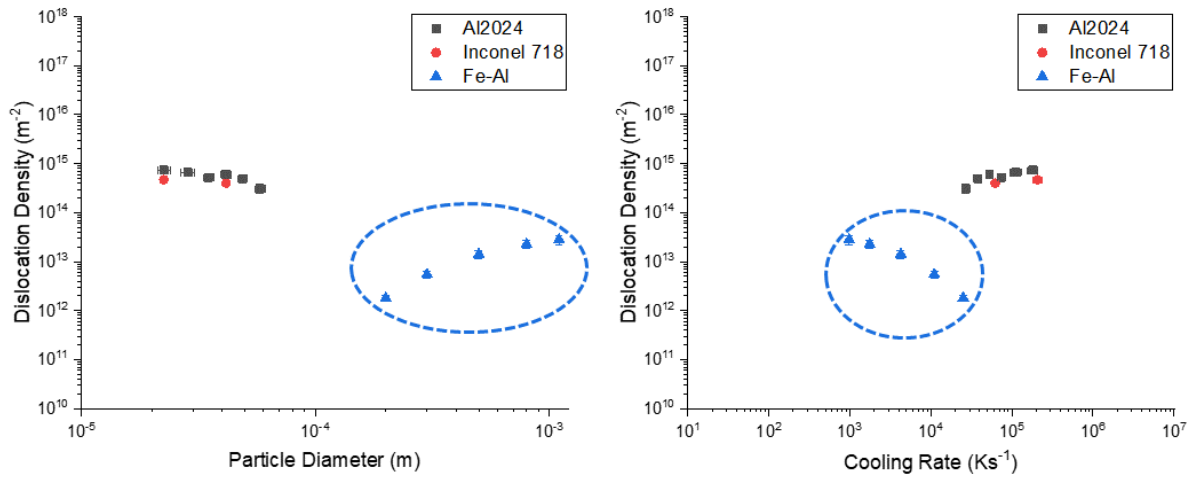


Figure 5-31: Dislocation density results for all materials displayed on a log-log plot.

The order of magnitude for the dislocation density results of Fe-Al samples ($\rho = 10^{12} - 10^{14}$ m⁻²) was consistent with the trends observed in the other materials. While there are difficulties in analyzing this new regime of particle size and cooling rate, future studies that will remove the artificial broadening effect appear promising. This effect may also be related to the possibility of multiple nucleation sites occurring on this relatively larger sample size. This is an opportunity for future work using X-ray tomography.

5.5 Literature Comparison

Most published research on dislocation density for samples within this size regime are additively manufactured specimens made from metallic powder. While not identical to measuring the as-atomized powders, this literature does provide a fair estimate of the order of magnitude for dislocation density. Several studies using the same XRD LPA method to determine dislocation density are provided in TABLE XXIII.

TABLE XXIII: Dislocation Density Literature Review

Material	Size (m) x 10⁻⁶	Dislocation Density (m⁻²) x 10¹⁴	Comments	Reference
AlCrFe ₂ Ni ₂ (Atomized)	20 - 100	6 - 8	Powder	Jeon et al. [56]
AlCrFe ₂ Ni ₂ (Annealed)	20 - 100	3 - 5	Powder	Jeon et al. [56]
Fe powder Al powder (40% Al at)	60 - 80 120 - 80	9	Mechanically Alloyed Ball Milled	Mhadhbi et al. [98]
Inconel 718	10 - 45	18.5	Additively Manufactured	Prasad et al. [4]
Inconel 718	10 - 45	4*	Additively Manufactured Determined Using TEM	Kouraytem et al. [99]
Inconel 718	20 - 60	4 - 5	<i>This Work</i>	N/A
Al2024	15 - 45	5.7	Additively Manufactured Resonance Fatigue	Zhan et al. [100]
Al2024	Sheet specimen	9	Cold rolled	Mirzaei et al. [7]
Al2024	20 - 60	3 - 9	<i>This work</i>	N/A
Type 304 Steel	Plate specimen	3.3	Cold rolled	Shintani et al. [101]
Fe-Al	200 - 1100	~0.02 – 0.30	<i>This work</i>	N/A

Dislocation density results for Al2024 and Inconel 718 obtained in this study are consistent with the published results for similar, but not identical, experiments. The dislocation density results for Fe-Al samples are significantly lower than these values, but there is currently no supporting literature for this cooling rate regime. The only published comparison of dislocation density trend

as a function of particle size [56] for samples similar to this set of experiments is consistent with the results of this study, as shown in Figure 5-32.

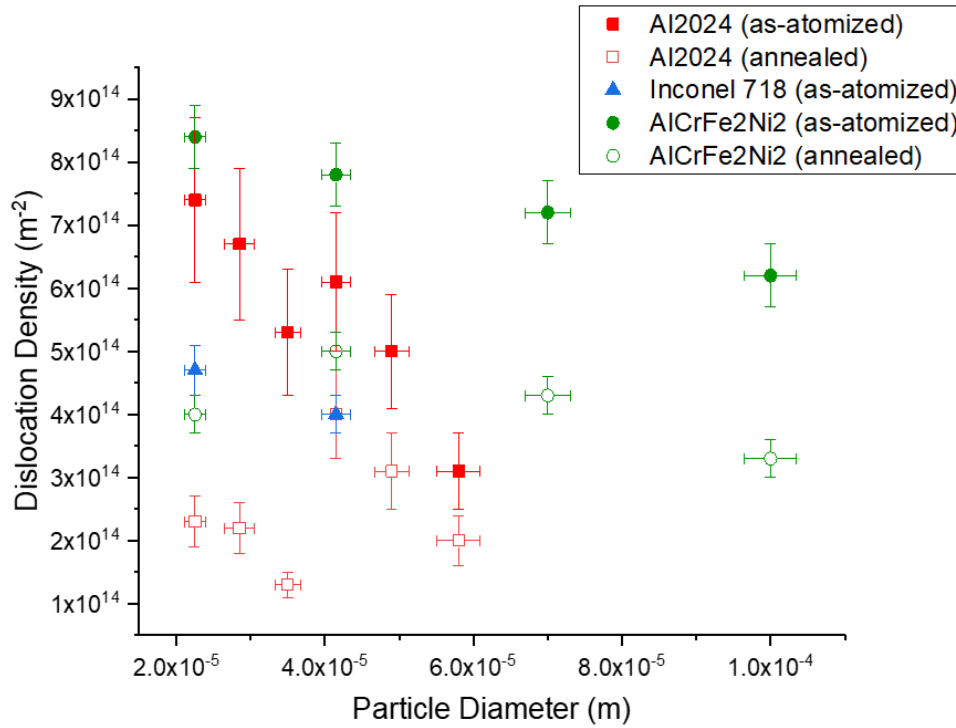


Figure 5-32: Comparison of observed dislocation density trends to AlCrFe₂Ni₂ alloy.

5.6 Error Sources

Various sources of error are introduced, from the initial production of the powders to the final calculation. This section will identify several sources of errors and discuss their potential impact on results.

During the gas atomization process used to make metallic powders, smaller, solidified particles may impact larger particles that are not yet solidified. The two particles will fuse into one single particle with an appendage called a satellite, as shown in Figure 5-33 for an as-atomized Inconel 718 powder sample with 45-53 μm particle size. Other published literature has also reported this using gas-atomized samples [102]. The smaller satellite particle may not potentially remelt, which would generate artificially higher dislocation density. Additionally, this appendage

may prevent the fused particle from being sieved adequately due to its irregular shape. Regardless, this effect is expected to be minimal and not significantly impact the outcome of this experiment.

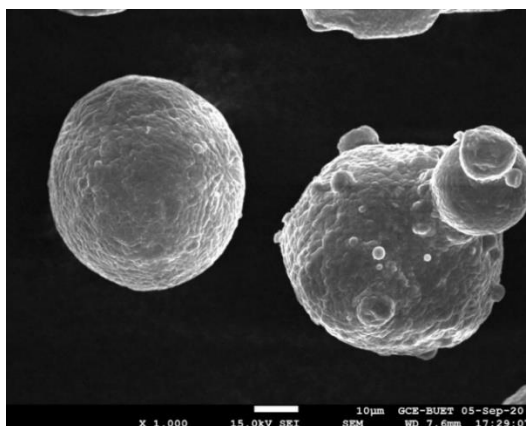


Figure 5-33: SEM micrograph at 1000X magnification showing satellite formation.

During the sieving process to isolate powders into distinct particle size ranges, particles may be trapped within the mesh wire screen of the sieve, as shown in Figure 5-34. This may not allow smaller particles to filter through the sieve properly and may result in smaller particles being introduced into large particle size ranges. The effect of this error would be a reduction in the observed dislocation density trend as a function of particle size. Additionally, this may result in cross-sample contamination if the sieve is not thoroughly cleaned between different materials. This error is not expected to impact the outcome of the experiment significantly.

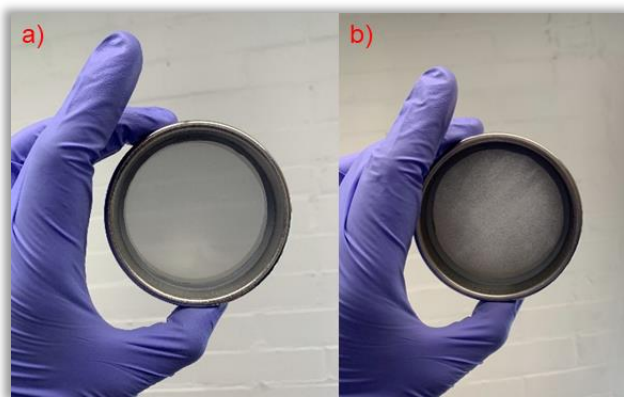


Figure 5-34: The powder sieving process showing trapped powder.
a) a new sieve, b) a used sieve with trapped powder.

The annealed Al2024 samples were found to contain Al₂CuMg aggregates as well as smaller precipitates at cell boundaries, as shown in Figure 5-35 for a powder sample of particle size 20-25 μm . These Al₂CuMg precipitates observed through EDX analysis may have influenced the XRD LPA. Specifically, the orthorhombic crystal structure of the precipitate has a similar diffraction peak to the FCC α phase for orientations (220) and (111), respectively. However, as shown by the full-range diffractogram for the annealed Al2024, this contribution is insignificant.

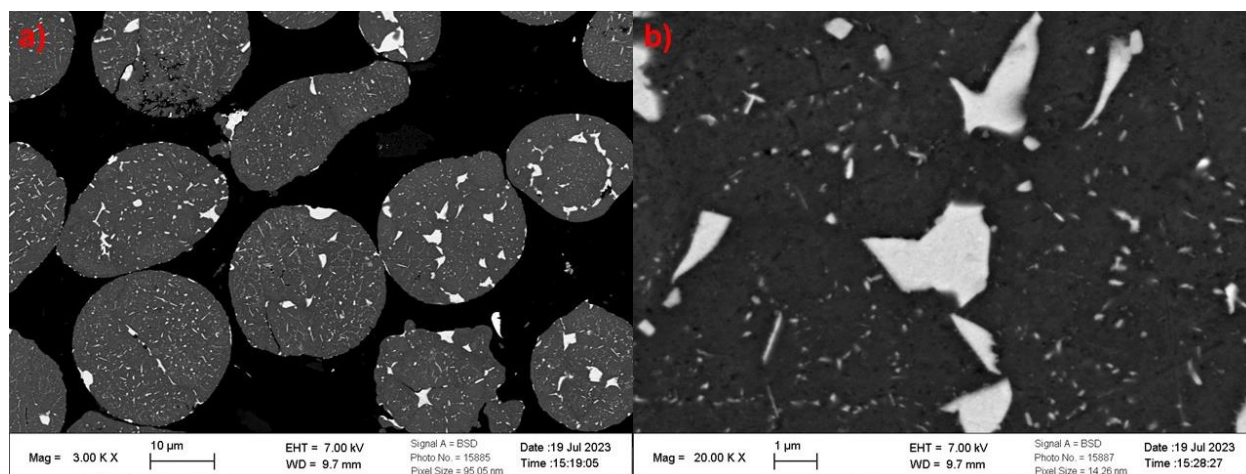


Figure 5-35: EDX spectroscopy micrograph for an annealed Al2024 with Al₂CuMg precipitates.
a) SEM micrograph at 3,000X magnification, b) SEM micrograph at 20,000X magnification.

Within XRD LPA, some variability exists during the curve fitting process. For example, identifying the best-fit spline to characterize background noise and choosing the angle rate at which the automated curve fitting is applied may change the observed Bragg peak locations and widths but are not expected to be a significant source of error.

During the XRD data collection, there is some variability in how the samples are loaded into their corresponding container, as shown in Figure 3-13. Accurate characterization of Bragg peaks is significantly dependent on sample position. Aside from the Fe-Al experiments, this is not expected to be a significant source of error. The sample size/position for Fe-Al XRD was a considerable source of error, which positively skewed the dislocation density results for larger

particles. Thicker samples are also more likely to exhibit multiple scattering events, which would generate more broad peaks, but this effect specifically is not expected to be significant.

There are several sources of error associated with the cooling rate correlations. The velocity is assumed to be zero for the gas-atomized samples, resulting in a lower Nusselt number and a lower cooling rate. This is not expected to be a significant source of error. An instantaneous cooling rate was determined for each particle size, which is not representative of the entire solidification process. Secondary dendrite arm spacing is approximated as cell size, consistent with other studies on rapidly solidified samples. Oxidation may occur during solidification, dramatically increasing emissivity, specifically for Inconel 718. Many material properties were estimated to perform model-based cooling rate prediction calculations. The error associated with the cooling rate calculations is not significant because this calculation is only an approximation to identify general trends.

The misorientation angle calculation using EBSD contains several additional sources of error. Preparation of the samples for this analysis occasionally generated scratches or pores that may have artificially increased the local density of dislocations and associated average local misorientation angle. EBSD is only performed at one cross-sectional slice, which may not represent the entire sample. These errors are not expected to significantly affect the outcome of this experiment because the misorientation angle was only used as an approximation to identify dislocation density trends.

6 Conclusions and Future Work

The relationship between solidification history and dislocation density has been investigated using metallic powders of Inconel 718, Al2024, and Fe-Al to access the regime of rapid solidification. Data was collected using X-ray diffraction, electron backscatter diffraction, and metallographic techniques. This section synthesizes the key findings of this research, discusses implications for industrial applications, and proposes directions for future research.

6.1 Conclusions

The as-atomized powders were successfully isolated into distinct particle size ranges representative of unique solidification histories. The cooling rate for each particle size range was predicted using theory and verified with experimental observation of apparent cell size. The dislocation density of each sample was successfully analyzed using line profile analysis of X-ray diffraction data. A trend of increasing dislocation density with increased cooling rate was observed in both Inconel 718 and Al2024 samples. The dislocation density of annealed Al2024 samples showed no trend of dislocation density as a function of particle size. These results indicate that the trend observed in the as-atomized samples was due to solidification history rather than particle size. Observed trends in dislocation density were substantiated using approximations of dislocation density trends using misorientation angle found through EBSD analysis. Fe-Al experimentation was limited due to the diffractometer, but future synchrotron diffraction is planned outside of the scope of this work to address this limitation. A summary of each portion of experimentation is provided below.

6.1.1 Inconel 718

- Initial XRD experiments confirmed that Bragg peaks appeared at the expected angles.
- Dislocation density analysis using data from the Phillips diffractometer was successful but limited by the instrument capability.
- A comparison study of the Phillips and PANalytical diffractometers demonstrated the superior capability of the PANalytical instrument.
- Dislocation density analysis with the PANalytical diffractometer was successful, though it could benefit from improved instrumentation, such as synchrotron XRD.
- Heat treatment successfully healed dislocations, but quantification of efficacy is complicated by significant coarsening of precipitates.
- Cooling rate calculations were based on governing theory but could not be validated using cell size approximations due to wide variations in the published literature.
- However, the observed cell size was comparable to Al₂O₃ particles with similar cooling rates.
- EBSD measurements of local misorientation angles were successful but limited by the amount of data collected.
- Without heat-treated samples, the dislocation density results for the as-atomized samples lacked a baseline for comparison.
- Dislocation density results for the as-atomized samples showed an increasing trend with decreasing particle size and increasing cooling rate, though more data would have strengthened this observation.
- Trends in local misorientation angles supported the observed trends in dislocation density.

- Results for dislocation density magnitude and trend aligned with expected values from the limited published literature.

6.1.2 Al₂O₃

- Cooling rate predictions based on governing theory were validated using observed apparent cell size to predict cooling rate from relationships in published literature.
- Heat treatment was successful in healing dislocations.
- Preliminary X-ray diffraction experiments successfully verified the Bragg peak location for as-atomized and heat-treated samples.
- Subsequent experiments using synchrotron X-ray diffraction were successful.
- Dislocation density results for the as-atomized samples showed an increasing trend with decreasing particle size and increasing cooling rate.
- Dislocation density results for the heat-treated samples showed little to no trend with particle size and cooling rate.
- Trends in local misorientation angles supported the observed trends in dislocation density.
- Results for dislocation density magnitude and trend aligned with expected values from the limited published literature.

6.1.3 Fe-Al

- Cooling rate predictions based on governing theory were predicted but have not been validated with observed cell size within the scope of this work.
- Heat treatment was performed but has not been verified to be successful within the scope of this work.

- Preliminary X-ray diffraction experiments to verify Bragg peak location were successful.
- Dislocation density results for the as-atomized samples were unsuccessful due to limitations associated with the laboratory diffractometer.
- Future synchrotron X-ray diffraction experiments are planned to address these limitations, but they are outside the scope of this work.
- The observed dislocation density results are comparable in magnitude to other materials studied in this work but expectedly lower.

6.1.4 Application to Industry

The relationship between solidification history and dislocation density proposed in this study applies to industrial solidification processes, especially processes involving rapid solidification such as welding or additive manufacturing. The findings from this study provide data trends that can be used to improve the quality of metal parts produced through casting, welding, and other fabrication techniques. Practical applications of this research may include setting limitations on acceptable particle sizes for additive manufacturing powders, adjusting heating parameters during additive manufacturing or welding, and improving the predictability of material properties of additively manufactured parts based on powder particle size and required post-production heat treatments. These insights demonstrate the practical applications of this research, contributing to advancements in manufacturing technology and material science.

6.2 Future Work

Several additional experiments are proposed to further enhance the understanding of the relationship between solidification history and defect structures. Experiments with additional materials would increase the breadth of this study. Specifically, the Ti-6Al-4V alloy would be a

promising candidate for future research due to its status as the most widely used titanium alloy [103], and an increasing amount of literature is being published on additively manufactured parts made from this material. Further experimentation on the Fe-Al alloy using synchrotron diffraction will address the limitations identified in this study. A proposal to conduct the Fe-Al experiments has been accepted at the DESY synchrotron and will be completed in the near future. Additional EBSD evaluations should be performed to reduce measurement noise and provide a more accurate baseline estimate for GND density. Specifically, three-dimensional EBSD [94] techniques may yield even better results. Transmission electron microscopy [104] techniques may offer improved evaluations of dislocation density but would require a more complex sample preparation. In-situ XRD measurements during solidification [46] might offer enhanced insights into microstructural evolution. However, this could be challenging due to rapid solidification, necessitating larger samples. Additionally, computational solidification modeling [105] could improve predictions of microstructural evolution. Exploring additional atomization techniques, such as water atomization [106], may enable the evaluation of new cooling rate regimes. Techniques using the Read-Shockley model [107] could further relate the energy associated with dislocations to solidification phenomena like undercooling. Evaluating the constitutional undercooling using the growth-restriction parameter [107] may provide additional insight into energy balance. These proposed experiments and analyses could offer deeper insights into the relationship between solidification history and defect structures, ultimately contributing to optimized manufacturing processes.

References

- [1] P. Thirathipviwat *et al.*, “Interaction of local solidification and remelting during dendrite coarsening - Modeling and comparison with experiments,” *Sci. Rep.*, vol. 7, no. 1, pp. 1–13, 2017, doi: 10.1038/s41598-017-17857-2.
- [2] D. M. Herlach, “Non-equilibrium solidification of undercooled metallic melts,” *Metals*. 2014. doi: 10.3390/met4020196.
- [3] S. Zhang *et al.*, “Strengthening mechanisms in selective laser-melted Inconel718 superalloy,” *Mater. Sci. Eng. A*, vol. 812, no. March, p. 141145, 2021, doi: 10.1016/j.msea.2021.141145.
- [4] K. Prasad, M. Obana, A. Ito, and S. Torizuka, “Synchrotron diffraction characterization of dislocation density in additively manufactured IN 718 superalloy,” *Mater. Charact.*, vol. 179, no. July, p. 111379, 2021, doi: 10.1016/j.matchar.2021.111379.
- [5] A. Arcari, D. Horton, M. J. Chen, and M. A. Zikry, “Precipitate and dislocation-density interactions affecting strength and ductility in inconel alloys,” *J. Mater. Sci.*, vol. 59, no. 12, pp. 4965–4977, 2024, doi: 10.1007/s10853-023-08822-8.
- [6] Y. Chen, Y. Tang, H. Zhang, N. Hu, N. Gao, and M. J. Starink, “Microstructures and hardness prediction of an ultrafine-grained al-2024 alloy,” *Metals (Basel)*., vol. 9, no. 11, pp. 1–10, 2019, doi: 10.3390/met9111182.
- [7] M. Mirzaei, M. R. Roshan, and S. A. Jenabali Jahromi, “Microstructure and mechanical properties relation in cold rolled Al 2024 alloy determined by X-ray line profile analysis,” *Mater. Sci. Eng. A*, vol. 620, no. 2015, pp. 44–49, 2015, doi: 10.1016/j.msea.2014.09.110.
- [8] R. Jemghili, A. Ait Taleb, and K. Mansouri, “Additive manufacturing progress as a new industrial revolution,” *2020 IEEE 2nd Int. Conf. Electron. Control. Optim. Comput. Sci. ICECOCS 2020*, no. February, 2020, doi: 10.1109/ICECOCS50124.2020.9314623.
- [9] E. Hosseini and V. A. Popovich, “A review of mechanical properties of additively manufactured Inconel 718,” *Addit. Manuf.*, vol. 30, no. September, p. 100877, 2019, doi: 10.1016/j.addma.2019.100877.
- [10] O. A. Ruano, A. Orozco-Caballero, M. Álvarez-Leal, and F. Carreño, “Influence of Solid Solutions on the Al2024 High-Temperature Deformation Behavior,” *Materials (Basel)*., vol. 16, no. 18, 2023, doi: 10.3390/ma16186251.
- [11] Z. Zhang *et al.*, “Dislocations in Grain Boundary Regions: The Origin of Heterogeneous Microstrains in Nanocrystalline Materials,” *Metall. Mater. Trans. A Phys. Metall. Mater. Sci.*, vol. 51, no. 1, pp. 513–530, 2020, doi: 10.1007/s11661-019-05492-7.
- [12] M. Shahwaz, P. Nath, and I. Sen, “A critical review on the microstructure and mechanical properties correlation of additively manufactured nickel-based superalloys,” *J. Alloys*

- Compd.*, vol. 907, p. 164530, 2022, doi: 10.1016/j.jallcom.2022.164530.
- [13] C. Kumara, *Microstructure Modelling of Additive Manufacturing of Alloy 718*, no. 23. 2020.
 - [14] M. Godec *et al.*, “Hybrid additive manufacturing of Inconel 718 for future space applications,” *Mater. Charact.*, vol. 172, 2021, doi: 10.1016/j.matchar.2020.110842.
 - [15] G. Mrówka and J. Sieniawski, “Analysis of Intermetallic Phases in 2024 Aluminium Alloy,” *Solid State Phenom.*, vol. 197, pp. 238–243, 2013, doi: 10.4028/www.scientific.net/SSP.197.238.
 - [16] A. Staszczyk, J. Sawicki, and B. Adamczyk-Cieslak, “A study of second-phase precipitates and dispersoid particles in 2024 aluminum alloy after different aging treatments,” *Materials (Basel)*, vol. 12, no. 24, 2019, doi: 10.3390/ma1224168.
 - [17] S. Zandsalimi, A. Heidarzadeh, and T. Saeid, “Dissimilar friction-stir welding of 430 stainless steel and 6061 aluminum alloy: Microstructure and mechanical properties of the joints,” *Proc. Inst. Mech. Eng. Part L J. Mater. Des. Appl.*, vol. 233, no. 9, pp. 1791–1801, 2019, doi: 10.1177/1464420718789447.
 - [18] H. Henein, “Single fluid atomization through the application of impulses to a melt,” *Mater. Sci. Eng. A*, vol. 326, no. 1, pp. 92–100, 2002, doi: 10.1016/S0921-5093(01)01429-0.
 - [19] K. F. Kelton and A. L. Greer, “Test of classical nucleation theory in a condensed system,” *Phys. Rev. B*, vol. 38, no. 14, pp. 10089–10092, Nov. 1988, doi: 10.1103/PhysRevB.38.10089.
 - [20] U. K. Mohanty and H. Sarangi, “Solidification of Metals and Alloys,” in *Casting Processes and Modelling of Metallic Materials*, Z. Abdallah and N. Aldoumani, Eds. Rijeka: IntechOpen, 2020. doi: 10.5772/intechopen.94393.
 - [21] S. Jeon, M. P. Sansoucie, and D. M. Matson, “Hyper-cooling limit, heat of fusion, and temperature-dependent specific heat of Fe-Cr-Ni melts,” *J. Chem. Thermodyn.*, vol. 138, pp. 51–58, 2019, doi: 10.1016/j.jct.2019.06.001.
 - [22] D. M. Matson *et al.*, “Electromagnetic levitation containerless processing of metallic materials in microgravity: rapid solidification,” *npj Microgravity*, vol. 9, no. 1, 2023, doi: 10.1038/s41526-023-00310-2.
 - [23] Y. Wang *et al.*, “Revealing the Diversity of Dendritic Morphology Evolution During Solidification of Magnesium Alloys using Synchrotron X-ray Imaging: A Review,” *Acta Metall. Sin. (English Lett.)*, vol. 35, no. 2, pp. 177–200, 2022, doi: 10.1007/s40195-021-01319-w.
 - [24] A. M. Mullis, L. Farrell, R. F. Cochrane, and N. J. Adkins, “Estimation of cooling rates during close-coupled gas atomization using secondary dendrite arm spacing

- measurement,” *Metall. Mater. Trans. B Process Metall. Mater. Process. Sci.*, vol. 44, no. 4, pp. 992–999, 2013, doi: 10.1007/s11663-013-9856-2.
- [25] S. Bryer C, C. Victor K, N. Aaron T, and C. Danielle L, “Experimental Substantaton of Computatonal Thermodynamic, Kinetc, and Solidificaton Modeling for Cold Sprayable, Gas-atomized, Al 6061 Powder,” *Int. J. Metall. Met. Phys.*, vol. 5, no. 4, 2020, doi: 10.35840/2631-5076/9261.
- [26] T. Hochrainer, L. Laurson, S. Papanikolaou, G. Po, and R. B. Sills, “Characterization of dislocation ensembles: measures and complexity,” *Mater. Theory*, vol. 6, no. 1, 2022, doi: 10.1186/s41313-022-00045-9.
- [27] A. H. Baghdadi, A. Rajabi, N. F. M. Selamat, Z. Sajuri, and M. Z. Omar, “Effect of post-weld heat treatment on the mechanical behavior and dislocation density of friction stir welded Al6061,” *Mater. Sci. Eng. A*, vol. 754, no. March, pp. 728–734, 2019, doi: 10.1016/j.msea.2019.03.017.
- [28] L. Rebuffi, M. S. Del Río, E. Busetto, and P. Scardi, “Understanding the instrumental profile of synchrotron radiation X-ray powder diffraction beamlines,” *J. Synchrotron Radiat.*, vol. 24, no. 3, pp. 622–635, 2017, doi: 10.1107/S1600577517005434.
- [29] B. E. Warren, *X-ray diffraction*, Dover ed. New York SE - vii, 381 p. : il. ; 22 cm: Dover Publications, 1990. doi: LK - <https://worldcat.org/title/970413955>.
- [30] T. Ungár, G. Tichy, J. Gubicza, and R. J. Hellmig, “Correlation between subgrains and coherently scattering domains,” *Powder Diffr.*, vol. 20, no. 4, pp. 366–375, 2005, doi: 10.1154/1.2135313.
- [31] J. I. Langford, D. Louër, and P. Scardi, “Effect of a crystallite size distribution on X-ray diffraction line profiles and whole-powder-pattern fitting,” *J. Appl. Crystallogr.*, vol. 33, no. 3 Part 2, pp. 964–974, Jun. 2000, doi: 10.1107/S002188980000460X.
- [32] L. R. B. Elton and D. F. Jackson, “X-Ray Diffraction and the Bragg Law,” *Am. J. Phys.*, 1966, doi: 10.1119/1.1972439.
- [33] T. Ungár and A. Borbély, “The effect of dislocation contrast on x-ray line broadening: A new approach to line profile analysis,” *Appl. Phys. Lett.*, vol. 69, no. 21, pp. 3173–3175, 1996, doi: 10.1063/1.117951.
- [34] T. Ungár, J. Gubicza, G. Ribárik, and A. Borbély, “Crystallite size distribution and dislocation structure determined by diffraction profile analysis: principles and practical application to cubic and hexagonal crystals,” *J. Appl. Crystallogr.*, vol. 34, no. 3, pp. 298–310, Jun. 2001, doi: 10.1107/S0021889801003715.
- [35] T. Ungár, I. Dragomir, Á. Révész, and A. Borbély, “The contrast factors of dislocations in cubic crystals: The dislocation model of strain anisotropy in practice,” *J. Appl. Crystallogr.*, vol. 32, no. 5, pp. 992–1002, Oct. 1999, doi: 10.1107/S0021889899009334.

- [36] T. Ungár, I. Groma, and M. Wilkens, “Asymmetric X-ray line broadening of plastically deformed crystals. II. Evaluation procedure and application to [001]-Cu crystals,” *J. Appl. Crystallogr.*, vol. 22, no. 1, pp. 26–34, Feb. 1989, doi: 10.1107/S0021889888009720.
- [37] T. Ungár, I. Dragomir, Á. Révész, and A. Borbély, “The contrast factors of dislocations in cubic crystals: the dislocation model of strain anisotropy in practice,” *J. Appl. Crystallogr.*, vol. 32, no. 5, pp. 992–1002, Oct. 1999, doi: 10.1107/S0021889899009334.
- [38] T. Ungár and G. Tichy, “The Effect of Dislocation Contrast on X-Ray Line Profiles in Untextured Polycrystals,” *Phys. status solidi*, vol. 171, no. 2, pp. 425–434, Feb. 1999, doi: 10.1002/(SICI)1521-396X(199902)171:2<425::AID-PSSA425>3.0.CO;2-W.
- [39] J. I. Goldstein, D. E. Newbury, J. R. Michael, N. W. M. Ritchie, J. H. J. Scott, and D. C. Joy, “Energy Dispersive X-ray Spectrometry: Physical Principles and User-Selected Parameters,” in *Scanning Electron Microscopy and X-Ray Microanalysis*, New York, NY: Springer New York, 2018, pp. 209–234. doi: 10.1007/978-1-4939-6676-9_16.
- [40] S. NISHIKAWA and S. KIKUCHI, “The Diffraction of Cathode Rays by Calcite,” *Proc. Imp. Acad.*, vol. 4, no. 8, pp. 475–477, 1928, doi: 10.2183/pjab1912.4.475.
- [41] Oxford-Instruments, “An Introduction to EBSD,” *Oxford Instruments NanoAnalysis*, pp. 1–10, 2015.
- [42] Q. Li, “Geometrically Necessary Dislocation Analysis of Deformation Mechanism for Magnesium under Fatigue Loading at 0 °C,” *Crystals*, vol. 13, no. 3, 2023, doi: 10.3390/cryst13030490.
- [43] H. Henein, V. Uhlenwinkel, and U. Fritsching, *Metal sprays and spray deposition*. 2017. doi: 10.1007/978-3-319-52689-8.
- [44] A. K. Sahu and S. Bag, “Design of a double aging treatment for the improvement of mechanical and microstructural properties of pulse micro-plasma arc welded alloy 718,” *J. Mater. Sci.*, vol. 56, no. 23, pp. 13400–13415, 2021, doi: 10.1007/s10853-021-06121-8.
- [45] J. T. Staley and M. Tiryakioğlu, “The use of TTP curves and quench factor analysis for property prediction in aluminum alloys,” *Adv. Metall. Alum. Alloy.*, no. December, pp. 6–15, 2001.
- [46] E. B. Baker, “Mechanisms for metastable phase transformations in Co-Si and Fe-Co alloys,” p. 24, 2021, [Online]. Available: <https://www.proquest.com/openview/bb592d4e05e4f67bca54a75247522c5b/1?pq-origsite=gscholar&cbl=18750&diss=y>
- [47] “Stoe STADI P Transmission Powder Diffractometer.” [Online]. Available: <https://www.kofo.mpg.de/603842/stoe-stadi-p>
- [48] D. Balzar, “Modeling of X-Ray Diffraction Line Broadening With the Voigt Function :,” 1993, [Online]. Available:

<https://www.osti.gov/biblio/6953055%0Ahttps://www.osti.gov/servlets/purl/6953055>

- [49] D. Mani, A. Kupsch, B. R. Müller, and G. Bruno, “Diffraction Enhanced Imaging Analysis with Pseudo-Voigt Fit Function,” *J. Imaging*, vol. 8, no. 8, 2022, doi: 10.3390/jimaging8080206.
- [50] A. Ranganathan, “The Levenberg-Marquardt Algorithm 3 LM as a blend of Gradient descent and Gauss-Newton itera,” *Internet httpexcelsior cs ucsb educoursescs290ipdfL MA pdf*, vol. 142, no. June, pp. 1–5, 2004, [Online]. Available: <http://twiki.cis.rit.edu/twiki/pub/Main/AdvancedDipTeamB/the-levenberg-marquardt-algorithm.pdf>
- [51] D. Balzar, “Voigt-function model in diffraction line-broadening analysis,” *Defect Microstruct. Anal. By Diffraction*, p. 44, 1999, [Online]. Available: <http://mysite.du.edu/~balzar/Documents/IUCRbook.pdf>
- [52] D. R. Black, M. H. Mendenhall, C. M. Brown, A. Henins, J. Filliben, and J. P. Cline, “Certification of Standard Reference Material 660c for powder diffraction,” *Powder Diffraction*, vol. 35, no. 1, pp. 17–22, 2020, doi: 10.1017/S0885715620000068.
- [53] A. J. C. Wilson, “Mathematical Theory of X-ray Powder Diffractometry,” *Philips’ Tech. Libr.*, p. 435, 1963.
- [54] G. Caglioti, A. Paoletti, and F. P. Ricci, “Choice of collimators for a crystal spectrometer for neutron diffraction,” *Nucl. Instruments*, vol. 3, no. 4, pp. 223–228, 1958, doi: 10.1016/0369-643X(58)90029-X.
- [55] N. Sallel *et al.*, “In situ characterization of microstructural instabilities: Recovery, recrystallization and abnormal growth in nanoreinforced steel powder,” *Acta Mater.*, vol. 87, pp. 377–389, 2015, doi: 10.1016/j.actamat.2014.11.051.
- [56] S. Jeon *et al.*, “Particle size effects on dislocation density, microstructure, and phase transformation for high-entropy alloy powders,” *Materialia*, vol. 18, no. December, p. 101161, 2021, doi: 10.1016/j.mtla.2021.101161.
- [57] G. K. Williamson and W. H. Hall, “X-ray line broadening from filed aluminium and wolfram,” *Acta Metall.*, vol. 1, no. 1, pp. 22–31, 1953, doi: 10.1016/0001-6160(53)90006-6.
- [58] P. Scherrer, “Estimation of the Size and Internal Structure of Colloidal Particles,” *Nachrichten von der Gesellschaft der Wissenschaften zu Göttingen*, vol. 2, pp. 96–100, 1918, doi: 10.1007/978-3-662-11836-8_26.
- [59] B. E. Warren, “X-ray studies of deformed metals,” *Prog. Met. Phys.*, vol. 8, no. C, pp. 147–202, 1959, doi: 10.1016/0502-8205(59)90015-2.
- [60] H. Liu, S. Zhu, T. B. Abbott, Z. Zhen, and J. F. Nie, “Quasi-in-situ EBSD Study of the Microstructure and Texture Evolution During Static Recrystallization in an Extruded Mg-

- Mn-Ce Alloy,” *Jom*, vol. 74, no. 7, pp. 2592–2608, 2022, doi: 10.1007/s11837-022-05321-0.
- [61] O. Muránsky, L. Balogh, M. Tran, C. J. Hamelin, J. S. Park, and M. R. Daymond, “On the measurement of dislocations and dislocation substructures using EBSD and HRSD techniques,” *Acta Mater.*, vol. 175, pp. 297–313, 2019, doi: 10.1016/j.actamat.2019.05.036.
 - [62] J. F. Nye, “Some geometrical relations in dislocated crystals,” *Acta Metall.*, vol. 1, no. 2, pp. 153–162, 1953, doi: 10.1016/0001-6160(53)90054-6.
 - [63] L. P. Kubin and A. Mortensen, “Geometrically necessary dislocations and strain-gradient plasticity: A few critical issues,” *Scr. Mater.*, vol. 48, no. 2, pp. 119–125, 2003, doi: 10.1016/S1359-6462(02)00335-4.
 - [64] W. J. Boettinger, L. Bendersky, and J. G. Early, “ANALYSIS OF THE MICROSTRUCTURE OF RAPIDLY SOLIDIFIED Al-8 Wt Pct Fe POWDER,” *Metall. Trans. A, Phys. Metall. Mater. Sci.*, vol. 17, no. 5, pp. 781–790, 1986, doi: 10.1007/BF02643853.
 - [65] C. E. W. and R. E. W. J.R. Welty, *Fundamentals of Momentum, Heat and Mass Transfer*, 3rd ed. John Wiley and Sons, 1984. doi: 10.1201/9781315273259.
 - [66] V. A. Rabinovich, A. A. Vasserman, V. I. Nedostup, and L. S. Veksler, “Thermophysical Properties of Neon, Argon, Krypton, and Xenon,” 1987. [Online]. Available: <https://api.semanticscholar.org/CorpusID:93621938>
 - [67] J. B. Wiskel, H. Henein, and E. Maire, “Solidification study of aluminum alloys using impulse atomization: Part I - Heat transfer analysis of an atomized droplet,” *Can. Metall. Q.*, vol. 41, no. 1, pp. 97–110, 2002, doi: 10.1179/cmqr.2002.41.1.97.
 - [68] W. E. Ranz and W. R. Marshall, “Evaporation from drops - Part 1,” *Chemical Engineering Progress*, vol. 48, pp. 141–148, 1952.
 - [69] S. Whitaker, “Forced convection heat transfer correlations for flow in pipes, past flat plates, single cylinders, single spheres, and for flow in packed beds and tube bundles,” *AIChE J.*, vol. 18, no. 2, pp. 361–371, 1972, doi: 10.1002/aic.690180219.
 - [70] E. S. Lee and S. Ahn, “Solidification progress and heat transfer analysis of gas-atomized alloy droplets during spray forming,” *Acta Metall. Mater.*, vol. 42, no. 9, pp. 3231–3243, 1994, doi: 10.1016/0956-7151(94)90421-9.
 - [71] M. El-Bealy, “Monotonic and fluctuated cooling approaches in secondary cooling zones during continuous casting,” *Can. Metall. Q.*, vol. 36, no. 1, pp. 49–56, 1997, doi: 10.1016/s0008-4433(96)00034-1.
 - [72] He Shiwen, Liu Yong, and Guo Sheng, “Cooling rate calculation of non-equilibrium aluminum alloy powders prepared by gas atomization,” *Rare Met. Mater. Eng.*, vol. 38,

- no. S1, pp. 353–356, 2009.
- [73] S. Gowri and F. H. Samuel, “Effect of cooling rate on the solidification,” *Metall. Trans. A*, vol. 23, no. 1 Supplement, pp. 3369–3376, 1992, doi: 10.1007/BF03024544.
 - [74] MATWEB, “Special Metals INCONEL® Alloy 718.”
 - [75] C. Cagran, H. Reschab, R. Tanzer, W. Schützenhöfer, A. Graf, and G. Pottlacher, “Normal spectral emissivity of the industrially used alloys NiCr20TiAl, inconel 718, X2CrNiMo18-14-3, and another austenitic steel at 684.5 nm,” *Int. J. Thermophys.*, vol. 30, no. 4, pp. 1300–1309, 2009, doi: 10.1007/s10765-009-0604-4.
 - [76] B. C. Sousa, C. Walde, V. K. Champagne, A. T. Nardi, R. D. Sisson, and D. L. Cote, “Rapidly solidified gas-atomized aluminum alloys compared with conventionally cast counterparts: Implications for cold spray materials consolidation,” *Coatings*, vol. 10, no. 11, pp. 1–28, 2020, doi: 10.3390/coatings10111035.
 - [77] L. L. C. MatWeb, “ASM Material Data Sheet: Aluminum Alloy 2024.” 2024. [Online]. Available: <https://www.matweb.com/search/datasheet.aspx?matguid=642e240585794f0ab91428aa78c27b4e>
 - [78] C. Da Wen and I. Mudawar, “Emissivity characteristics of roughened aluminum alloy surfaces and assessment of multispectral radiation thermometry (MRT) emissivity models,” *Int. J. Heat Mass Transf.*, vol. 47, no. 17–18, pp. 3591–3605, 2004, doi: 10.1016/j.ijheatmasstransfer.2004.04.025.
 - [79] J. R. Davis, Ed., “Metals Handbook Desk Edition.” ASM International, Dec. 01, 1998. doi: 10.31399/asm.hb.mhde2.9781627081993.
 - [80] D. S. Mackenzie, *Handbook of Aluminum*. 2003. doi: 10.1201/9780429223259.
 - [81] A. I. Savvatimskiy and S. V. Onufriev, “Specific Heat of Liquid Iron from the Melting Point to the Boiling Point,” *High Temp.*, vol. 56, no. 6, pp. 933–935, 2018, doi: 10.1134/S0018151X18060202.
 - [82] M. Leitner, T. Leitner, A. Schmon, K. Aziz, and G. Pottlacher, “Thermophysical Properties of Liquid Aluminum,” *Metall. Mater. Trans. A Phys. Metall. Mater. Sci.*, vol. 48, no. 6, pp. 3036–3045, 2017, doi: 10.1007/s11661-017-4053-6.
 - [83] U. R. Kattner and B. P. Burton, “Phase Diagrams of Binary Iron Alloys, Al-Fe (Aluminum-Iron),” no. July, 2019.
 - [84] H. Kobatake, H. Khosroabadi, and H. Fukuyama, “Normal spectral emissivity measurement of liquid iron and nickel using electromagnetic levitation in direct current magnetic field,” *Metall. Mater. Trans. A Phys. Metall. Mater. Sci.*, vol. 43, no. 7, pp. 2466–2472, 2012, doi: 10.1007/s11661-012-1101-0.

- [85] H. Henein *et al.*, “Droplet solidification of impulse atomized Al-0.61Fe and Al-1.9Fe,” *Can. Metall. Q.*, vol. 49, no. 3, pp. 275–292, 2010, doi: 10.1179/cm.2010.49.3.275.
- [86] E. Bassini *et al.*, “Effect of the particle size distribution on physical properties, composition, and quality of gas atomized Astroloy powders for HIP application,” *J. Alloys Compd.*, vol. 890, p. 161631, 2022, doi: 10.1016/j.jallcom.2021.161631.
- [87] G. S. Kumar *et al.*, “Particle size dependent microstructure evolution of inert gas atomized nickel base superalloy powders,” *J. Alloys Compd.*, vol. 909, p. 164772, 2022, doi: 10.1016/j.jallcom.2022.164772.
- [88] G. Kasperovich, T. Volkman, L. Ratke, and D. Herlach, “Microsegregation during solidification of an Al-Cu binary alloy at largely different cooling rates (0.01 to 20,000 K/s): Modeling and experimental study,” *Metall. Mater. Trans. A Phys. Metall. Mater. Sci.*, vol. 39 A, no. 5, pp. 1183–1191, 2008, doi: 10.1007/s11661-008-9505-6.
- [89] L. Bäckerud, E. Król, and J. Tamminen, *Solidification characteristics of aluminium alloys. Vol. 1 Wrought alloys*. Oslo SE - 156 Seiten: Skanaluminium, 1986. doi: LK - <https://worldcat.org/title/257711474>.
- [90] M. C. Flemings, T. Z. Kattamis, and B. P. Bardes, “Dendrite arm spacing in aluminum alloys,” *AFS Trans.*, vol. 99, pp. 501–506, 1991.
- [91] S. Katayama and A. Matsunawa, *Solidification microstructure of laser welded stainless steels*. 1984. doi: 10.2351/1.5057623.
- [92] G. F. Vander Voort and E. P. Manilova, “Metallographic techniques for superalloys,” *Microsc. Microanal.*, vol. 10, no. SUPPL. 2, pp. 690–691, 2004, doi: 10.1017/S1431927604883442.
- [93] E. Heyn, “The Metallographist,” *Metallogr.*, vol. 5, pp. 3–64, 1903.
- [94] B. K. Kim and J. A. Szpunar, *Collection, Processing, and Analysis of Three-Dimensional EBSD Data Sets*. 2009. doi: 10.1007/978-0-387-88136-2_27.
- [95] X. Diffractometry and C. Azersky, “Characterizing the Microstructure of Inconel 718 Powder Using Submitted By,” 2019.
- [96] C. Azersky, “Evaluation of Particle Size Dependent Crystal Defects in Inconel 718 by Line Profile Analysis of X-ray Diffraction Submitted By,” 2020.
- [97] C. Azersky, S. Jeon, C. Settens, M. Fakhrul, and D. Matson, “Quantifying X-ray diffractometer precision for measurement of dislocation density and the evolution of defect structures during solidification of additive manufacturing powders,” *IOP Conf. Ser. Mater. Sci. Eng.*, vol. 1281, no. 1, p. 012033, 2023, doi: 10.1088/1757-899x/1281/1/012033.
- [98] M. Khitouni, M. Mhadhbi, L. Escoda, J. J. Suñol, and M. Dammak, “Characterization of

- mechanically alloyed nanocrystalline Fe(Al): Crystallite size and dislocation density,” *J. Nanomater.*, vol. 2010, 2010, doi: 10.1155/2010/712407.
- [99] N. Kouraytem *et al.*, “A recrystallization heat-treatment to reduce deformation anisotropy of additively manufactured Inconel 718,” *Mater. Des.*, vol. 198, 2021, doi: 10.1016/j.matdes.2020.109228.
- [100] J. Z. Zhan, G. Zhang, J. F. Ma, Z. Liu, and J. Song, “Determination of the Resonance Fatigue Dislocation Density of a 2024 Aluminum Alloy by X-Ray Diffraction,” *Strength Mater.*, vol. 53, no. 4, pp. 610–618, 2021, doi: 10.1007/s11223-021-00323-w.
- [101] T. Shintani and Y. Murata, “Evaluation of the dislocation density and dislocation character in cold rolled Type 304 steel determined by profile analysis of X-ray diffraction,” *Acta Mater.*, vol. 59, no. 11, pp. 4314–4322, 2011, doi: 10.1016/j.actamat.2011.03.055.
- [102] Y. Sakaguchi, T. Harada, and T. Kuji, “Microstructural studies of NdFeB powders produced by gas atomization,” *Mater. Sci. Eng. A*, vol. 181–182, no. C, pp. 1232–1236, 1994, doi: 10.1016/0921-5093(94)90837-0.
- [103] S. Liu and Y. C. Shin, “Additive manufacturing of Ti6Al4V alloy: A review,” *Mater. Des.*, vol. 164, p. 107552, 2019, doi: 10.1016/j.matdes.2018.107552.
- [104] D. K. Q. Mu *et al.*, “Microstructural evolution, precipitation and mechanical properties of ultrafine-grained and coarse-grained 2024 aluminum alloys fabricated by powder metallurgy,” *J. Mater. Res.*, vol. 37, no. 3, pp. 692–704, 2022, doi: 10.1557/s43578-022-00490-9.
- [105] M. F. Zhu, W. Cao, S. L. Chen, C. P. Hong, and Y. A. Chang, “Modeling of microstructure and microsegregation in solidification of multi-component Alloys,” *J. Phase Equilibria Diffus.*, vol. 28, no. 1, pp. 130–138, 2007, doi: 10.1007/s11669-006-9011-8.
- [106] A. Asgarian *et al.*, “Experimental and computational analysis of a water spray; application to molten metal atomization,” *Adv. Powder Metall. Part. Mater. - 2018 Proc. 2018 Int. Conf. Powder Metall. Part. Mater. POWDERMET 2018*, vol. 2018-June, no. June, pp. 126–137, 2018.
- [107] W. T. T. Read and W. Shockley, “Dislocation models of crystal grain boundaries,” *Phys. Rev.*, vol. 78, no. 3, pp. 275–289, 1950, doi: 10.1103/PhysRev.78.275.
- [108] X. Xiao, R. W. Hyers, R. K. Wunderlich, H. J. Fecht, and D. M. Matson, “Deformation induced frequency shifts of oscillating droplets during molten metal surface tension measurement,” *Appl. Phys. Lett.*, vol. 113, no. 1, 2018, doi: 10.1063/1.5039336.
- [109] D. M. Matson and X. Xiao, “Identifying metastable interface potency limits during steel alloy transformations,” *Mater. Lett.*, vol. 212, pp. 256–258, 2018, doi: 10.1016/j.matlet.2017.10.098.

- [110] D. M. Matson, “Retained free energy as a driving force for phase transformation during rapid solidification of stainless steel alloys in microgravity,” *npj Microgravity*, vol. 4, no. 1, 2018, doi: 10.1038/s41526-018-0056-x.
- [111] J. Nower, M. Pleskus, C. Azersky, X. Xiao, M. P. Sansoucie, and D. M. Matson, “Measurement of Mass Evaporation and Density of Commercial Super Alloys by Electro Static Levitation (ESL) Jannatun Nower,” no. November 2018, 2020, doi: 10.13140/RG.2.2.35949.74726.

Appendix

A. Material and Equipment Specifications



CarTech® 718 Alloy

Identification

UNS Number

• N07718

DIN Number

• 2.4668

Type Analysis

Single figures are nominal except where noted.

Carbon (Maximum)	0.08 %	Manganese (Maximum)	0.35 %
Phosphorus (Maximum)	0.015 %	Sulfur (Maximum)	0.015 %
Silicon (Maximum)	0.35 %	Chromium	17.00 to 21.00 %
Molybdenum	2.80 to 3.30 %	Copper (Maximum)	0.15 %
Cobalt (Maximum)	1.00 %	Titanium	0.65 to 1.15 %
Aluminum	0.35 to 0.80 %	Columbium + Tantalum	4.75 to 5.50 %
Boron	0.001 to 0.006 %	Nickel + Cobalt	50.00 to 55.00 %
Iron	Balance		

The Cobalt content is on an "If determined" basis.

General Information

Description

CarTech 718 alloy is a precipitation hardenable nickel-base alloy designed to display exceptionally high yield, tensile and creep-rupture properties at temperatures up to 1300°F (704°C). The sluggish age-hardening response of CarTech 718 permits annealing and welding without spontaneous hardening during heating and cooling. This alloy has excellent weldability when compared to the nickel-base superalloys hardened by aluminum and titanium.

Applications

This alloy has been used for jet engine and high-speed airframe parts such as wheels, buckets, spacers, and high temperature bolts and fasteners. It has also been used extensively in oil and gas drilling and production due to its high strength and resistance to chlorides, stress corrosion and sulfide stress cracking. Within the oil and gas industry, this alloy has been used in valves, tubing hangers, MWD/LWD parts, fishing tools, pump shafts and wellhead components.

Corrosion Resistance

Important Note: The following 4-level rating scale is intended for comparative purposes only. Corrosion testing is recommended; factors which affect corrosion resistance include temperature, concentration, pH, impurities, aeration, velocity, crevices, deposits, metallurgical condition, stress, surface finish and dissimilar metal contact.

Nitric Acid	Good	Sulfuric Acid	Moderate
Phosphoric Acid	Moderate	Acetic Acid	Good
Sodium Hydroxide	Good	Salt Spray (NaCl)	Excellent
Sea Water	Moderate	Sour Oil/Gas	Good
Humidity	Excellent		

Properties

Physical Properties

Density 0.2970 lb/in³

CarTech® 718 Alloy

Mean CTE

77 to 200°F	7.10 x 10 ⁻⁶ in/in/°F
77 to 400°F	7.50 x 10 ⁻⁶ in/in/°F
77 to 600°F	7.70 x 10 ⁻⁶ in/in/°F
77 to 800°F	7.90 x 10 ⁻⁶ in/in/°F
77 to 1000°F	8.00 x 10 ⁻⁶ in/in/°F
77 to 1200°F	8.40 x 10 ⁻⁶ in/in/°F
77 to 1400°F	8.90 x 10 ⁻⁶ in/in/°F

Coefficient of Thermal Expansion

Temperature Range		10 ⁻⁶ /°F	10 ⁻⁶ /°C
77°F to	25°C to		
200	93	7.1	12.8
400	204	7.5	13.5
600	316	7.7	13.9
800	427	7.9	14.2
1000	538	8.0	14.4
1200	649	8.4	15.1
1400	760	8.9	16.0

Modulus of Elasticity (E)

70°F	29.6 x 10 ³ ksi
200°F	29.2 x 10 ³ ksi
400°F	28.8 x 10 ³ ksi
600°F	27.6 x 10 ³ ksi
800°F	26.5 x 10 ³ ksi
1000°F	25.5 x 10 ³ ksi
1200°F	24.5 x 10 ³ ksi
1400°F	23.1 x 10 ³ ksi
1600°F	18.1 x 10 ³ ksi
1750°F	11.1 x 10 ³ ksi

Modulus of Elasticity (E)

Temperature		psi x 10 ⁶	MPa x 10 ³
°F	°C		
70	21	29.6	204
200	93	29.2	201
400	204	28.8	199
600	316	27.6	190
800	427	26.5	183
1000	538	25.5	176
1200	649	24.5	169
1400	760	23.1	159
1600	871	18.1	125
1750	954	11.1	76.5

Electrical Resistivity (70°F)

728.0 ohm-cir-mil/ft

Melting Range

2200 to 2450 °F

CarTech® 718 Alloy

Typical Mechanical Properties

Effect of Solution Treating Temperature on Transverse Room Temperature Properties – Pyromet Alloy 718

Tensile properties of a 4" (101.6 mm) square billet

Solution Treating Temperature		0.2% Yield Strength		Tensile Strength		% Elongation in 2" (50.8 mm)	% Reduction of Area	Rockwell C Hardness	ASTM Grain Size
°F	°C	ksi	MPa	ksi	MPa				
1750	954	160.1	1104	183.7	1267	8.1	13.6	42	7
1900	1038	163.7	1129	183.7	1267	12.8	20.3	42	5/6
1950	1066	158.2	1091	180.9	1247	14.0	19.6	42	4/5
2000	1093	154.6	1066	178.0	1227	17.3	24.5	40	4/4.5
2050	1121	149.9	1033	171.1	1180	17.5	27.9	39	3.5/4

Note: All samples solution treated 2 hours at the temperature indicated and air cooled. Aging consisted of 8 hours at 1325°F (718°C), cooled 100°F/hr to 1150°F (56°C/hr to 621°C), held 8 hours at 1150°F (621°C), and air cooled.

Effect of Solution Treating Temperature on the Transverse Stress Rupture Properties – Pyromet Alloy 718

4" (101.6 mm) square billet

Solution Treating Temperature		Test Temperature		Stress		Rupture Life Hours	% Elongation in 2" (50.8 mm)	% Reduction of Area
°F	°C	°F	°C	ksi	MPa			
1750	954	1300	704	75	517	64.8	17.4	25.3
1900	1038	1300	704	75	517	132.1	9.4	17.2
1950	1066	1300	704	75	517	95.2	7.9	10.8
2000	1093	1300	704	75	517	141.0	2.5	6.8

Notes: All samples solution treated 2 hours at the temperatures indicated and air cooled. Aging consisted of 8 hours at 1325°F (718°C), cooled 100°F/hr to 1150°F (56°C/hr to 621°C), held 8 hours at 1150°F (621°C), and air cooled.

All specimens were combination smooth-notch bars with 0.178" (4.52 mm) gage diameter, 0.712" (18.1 mm) gage length and $K_t = 3.8$.

Stress rupture notch sensitivity may result from 1900°F (1038°C) solution treatment. This is due to reduction of precipitate at grain boundaries with this treatment.

Elevated Temperature Tensile Tests – Pyromet Alloy 718

½" (12.7 mm) bar stock

Test Temperature		0.2% Yield Strength		Tensile Strength		% Elongation 2" (50.8 mm)
°F	°C	ksi	MPa	ksi	MPa	
70	21	175	1207	210	1448	22.0
200	93	170	1172	204	1407	21.0
400	204	183	1124	198	1365	20.0
600	316	159	1096	195	1344	20.0
800	427	156	1076	191	1317	19.0
1000	538	155	1069	185	1276	18.0
1200	649	149	1027	168	1158	19.0
1400	760	110	758	110	758	27.0

Heat Treatment: 1 hr—1800°F (982°C), air cool + 8 hr—1325°F (718°C), cool 100°F/hr to 1150°F (56°C/hr to 621°C), hold 8 hr, air cool.

CarTech® 718 Alloy

Low Temperature Tensile Data – Pyromet Alloy 718

Transverse tests—4" (101.6 mm) square billet

Test No.	Test Temperature		0.2% Yield Strength		Tensile Strength		% Elongation In 2" (50.8 mm)	% Reduction of Area
	°F	°C	ksi	MPa	ksi	MPa		
1	Room	Room	157	1082	183.8	1267	18.0	21.6
2	Room	Room	156	1076	181.3	1250	16.5	23.3
3	Room	Room	152.5	1051	179.5	1238	19.0	29.1
4	-423	-253	196.2	1353	250.4	1726	14.0	16.2
5	-423	-253	200	1379	244.2	1684	13.0	17.5
6	-423	-253	194.2	1339	241.9	1668	12.0	16.7

Heat Treatment: 2 hr—1950°F (1066°C), air cool + 8 hr—1325°F (718°C), cool 100°F/hr to 1150°F (56°C/hr to 621°C), hold 8 hr, air cool.

Stress Rupture Data – Pyromet Alloy 718

Test Temperature		Stress for Rupture							
		100 Hours				1000 Hours			
		Smooth		Notch		Smooth		Notch	
°F	°C	ksi	MPa	ksi	MPa	ksi	MPa	ksi	MPa
1100	593	170	1172	220	1517	130	896	205	1413
1200	649	110	758	195	1344	85	586	170	1172
1300	704	75	517	130	896	55	379	80	552
1400	760	44	303	63	434	25	172	35	241

Heat Treatment: 1 hr—1800°F (982°C), air cool + 8 hr—1325°F (718°C), furnace cool 100°F/hr to 1150°F (56°C/hr to 621°C), hold 8 hr, air cool.

Heat Treatment

The following heat treatment should be used to obtain the best combination of tensile properties and stress rupture properties:

1 hr 1750°F to 1800°F (954°C to 982°C) air cool + 8 hr 1325°F (718°C) cool 100°F/hr to 1150°F (56°C/hr to 621°C), hold 8 hr and air cool.

To obtain the best room temperature and cryogenic tensile properties, the following heat treatment should be used:

1 to 2 hr 1950°F (1066°C), air cool + 8 hr 1325°F (718°C) cool 100°F/hr to 1150°F (56°C/hr to 621 °C), hold 8 hr and air cool.

Workability

Hot Working

Hot working is carried out using a 2050°F (1121°C) maximum furnace temperature. Hot-cold working in the range 1700/1850°F (927/1010°C) will improve the strength of the forging if the service temperature is below about 1100°F (593°C). Prolonged soaking at the forging temperature is not desirable. The material should be given uniform reductions to avoid the formation of duplex grain structures.

Machinability

The alloy can be readily machined in either the annealed or the age-hardened condition. The age-hardened condition gives better chip action on chip breaker tools and produces a better finish. The annealed condition will give a slightly longer tool life. Tooling and procedures are similar to those used for Carpenter Pyromet alloy X-750.

Weldability

Pyromet alloy 718 can be welded in either the annealed or the aged condition. Welding in the aged condition will cause the formation of a softened heat-affected zone.

CarTech® 718 Alloy

Other Information

Applicable Specifications

- | | |
|-------------|---------------|
| • AMS 5662 | • AMS 5663 |
| • API 6A718 | • NACE MR0175 |

Forms Manufactured

- | | |
|--------------|--------------|
| • Bar-Rounds | • Bar-Shapes |
| • Billet | • Strip |
| • Wire | • Wire-Rod |

Technical Articles

- [A Designer's Manual On Specialty Alloys For Critical Automotive Components](#)
- [A Guide to Etching Specialty Alloys for Microstructural Evaluation](#)
- [Alloy Selection for Cold Forming \(Part I\)](#)
- [Alloy Selection for Cold Forming \(Part II\)](#)
- [Carpenter 286-LNi Alloy - A Lower Cost Option for High Temperature Auto and Truck Fasteners](#)
- [Forging Difficult Alloys: How to Get Better Results, Consistently](#)
- [How to Select the Right Stainless Steel or High Temperature Alloy for Heading](#)
- [New Requirements for Ferrous-Base Aerospace Alloys](#)
- [Selecting High Temperature Alloys for Fasteners in Automotive Exhaust Systems](#)
- [Trends in High Temperature Alloys](#)

Disclaimer:

The information and data presented herein are typical or average values and are not a guarantee of maximum or minimum values. Applications specifically suggested for material described herein are made solely for the purpose of illustration to enable the reader to make his/her own evaluation and are not intended as warranties, either express or implied, of fitness for these or other purposes. There is no representation that the recipient of this literature will receive updated editions as they become available.

Unless otherwise specified, registered trademarks are property of
CRS Holdings Inc., a subsidiary of [Carpenter Technology Corporation](#)
Copyright © 2020 CRS Holdings Inc. All rights reserved.

Visit us on the web at www.cartech.com

Edition Date: 07/15/11



MATERIAL CERTIFICATE

AL2024 - Aerospace Grade Aluminum**Process Specifications**

Powder Description	High quality, aerospace grade AL2024 alloy.
Specification	-
Manufacturing Method	Proprietary
Solvus Global Lot Number	SG02.AL2024.04A.0000

Material Specifications

Powder Size	-
Hausner Ratio	1.282
Compaction Efficiency	55.58%
Geometry	Spherical
Carney Flow (g/s)	

Size Distribution

D10	19.30 μm
D25	23.63 μm
D50	31.22 μm
D75	42.50 μm
D90	49.04 μm

The test results above are certified to be true and correct.

	CAMERON MAXWELL	Process Technician	14FEB2020
Signature	Print	Title	Date

Composition, wt%

Al	Cu	Mg	Si	Mn
BAL	3.8-4.9	1.2-1.8	0.50 MAX	0.3-0.9

Solvus Global, LLC
104 Prescott St
Worcester, MA 01605
United States of America

1-717-602-511
inquiries@solvusglobal.cc
www.solvusglobal.cc

SAFETY DATA SHEET



104 Prescott St.
Worcester, MA 01605
www.solvusglobal.com
508-733-1808

In Case of Emergency:
Call representative of ChemTel Inc.
Emergency Response
1-800-255-3924
Contract Number: MIS7687904

SECTION 1: IDENTIFICATION OF SUBSTANCE/MIXTURE AND COMPANY/UNDERTAKING

1.1 Product Identifiers

Product Name: SAAM-AL2024-G1H2 SAAM-AL6061-G1H1
SAAM-AL7050-G1H3 SAAM-AL7075-G1H1
SAAM-AL5056-G1HX

Synonyms: Aluminum alloy, 2024, 5056, 6061, 7050, 7075

1.2 Product Use

Product Use: Cold spray, additive manufacturing, repair

Restrictions: Industrial use or research purposes only

1.3 Manufacturer/Supplier

Solvus Global, LLC
104 Prescott St.
Worcester, MA 01605
www.solvusglobal.com
1 – (508) – 733 – 1808

2 Emergency Telephone Numbers

1 – 800 – 255 – 3924
Contract Number: MIS7687904

SECTION 2: HAZARDS IDENTIFICATION

2.1 Hazard Classification

If suspended in air (dust cloud) fine grades can be ignited in the presence of an ignition source and could pose an explosion risk in a confined environment.

Prolonged contact with water may result in a reaction releasing hydrogen – ignition risk.

Will react with oxidizing agents or acids and alkalis, causing heating and hydrogen release – explosion risk.

Can violently react with halogenated hydrocarbons.

May cause sensitization by inhalation and skin contact.

May cause allergy or asthma symptoms or breathing difficulties if inhaled.

May cause damage to organs through prolonged or repeated exposure.



2.2 Statements

Hazard Statements

H260: In contact with water releases flammable gases which may ignite

H317: May cause an allergic skin reaction.

H334: May cause allergy or asthma symptoms or breathing difficulties if inhaled

H373: May cause damage to organs through prolonged or repeated exposure

SAAM-Aluminum-Processed

Issue Date: 10JAN2019

1

Precautionary Statements

P201: Obtain special instructions before use.
P202: Do not handle until all safety precautions have been read and understood.
P210: Keep away from heat/sparks/open flames/hot surfaces. No smoking.
P240: Ground/bond container and receiving equipment.
P241: Use explosion-proof electrical/ventilating/lighting equipment.
P261: Avoid breathing dust/ fume/ gas/ mist/ vapors/ spray.
P272: Contaminated work clothing should not be allowed out of the workplace.
P280: Wear protective gloves/protective clothing/eye protection/face protection.
P302+P352: IF ON SKIN: Wash with plenty of soap and water.
P308+P313: IF exposed or concerned: Get medical advice/ attention.
P321: Specific treatment (see supplemental first aid instructions on this label).
P333+P313: If skin irritation or rash occurs: Get medical advice/ attention.
P363: Wash contaminated clothing before reuse.
P370: Use for extinction: Special powder for metal fires.
P378: Use for extinction: Dry sand.
P405: Store locked up.
P501: Dispose of contents/ container to an approved waste disposal plant.

SECTION 3: COMPOSITION/INFORMATION ON INGREDIENTS

3.1 Composition

Ingredient	CAS Number	Weight (%)	Synonym	CLP Hazard	H Statement
Al	7429-90-5	Bal	Aluminum	Water-react. 2, Flam. Sol. 1	H261, H228
Cr	7440-47-3	0 – 0.5	Chromium	Eye Irrit. 2, Resp. Sens. 1	H319, H334
Cu	7440-50-8	0 – 5	Copper	Aquatic Chron 1, Aquatic Acute 1	H410, H400
Mg	7439-95-4	0 – 6	Magnesium	Aquatic Chron 3, Flam. Sol. 1	H412, H228
Zn	7440-66-6	0 – 6.1	Zinc	Aquatic Chron 1, Aquatic Acute 1	H410, H400
Si	7440-21-3	0 – 13	Silicon	Flam. Sol. 2, Skin Irrit. 2, Eye Irrit. 2	H228, H315, H319

SECTION 4: FIRST AID MEASURES

4.1 Eyes

Flush eyes with plenty of water, lifting the upper and lower eyelids occasionally. Get medical attention if irritation develops.

4.2 Skin

Wash skin using soap or a mild detergent and warm water.

4.3 Inhalation

Move exposed individuals to fresh air at once. If breathing has stopped, perform artificial respiration. Call a physician.

4.4 Ingestion

If swallowed, call a physician immediately. Rinse mouth with water. Do not induce vomiting unless directed by medical personnel.

SECTION 5: FIRE FIGHTING MEASURES**5.1 Extinguishing Media**

Dry powder, do not use water. Gently smother burning material with dry sand or other inert substance. Special Powder (Class D – dry powder) extinguishers with spin applicators for smoother effect application may be used carefully.

Not to be used: water, carbon dioxide, foam, ABC powder

5.2 Unusual Fire and Explosion Hazards

Dust can combine with air to form an explosive mixture.

Contact with water releases flammable gases (hydrogen)

Decomposition of this product may yield metallic oxides

5.3 Fire Fighting Equipment

Wear self-contained breathing apparatus for firefighting if necessary and protective clothing.

Avoid creation of dusts.

Prevent firefighting water entering watercourses or groundwater.

5.4 Precautions

Keep away from ignition sources.

SECTION 6: ACCIDENTAL RELEASE MEASURES

1. Restrict area to those wearing appropriate respiratory and personal protective equipment
2. Ventilate area
3. Collect powder to minimize spread to environment and dust generation
4. Keep out of waterways and sewers
5. Dispose of as appropriate per regional, local, state, and federal regulations (see section 13)
6. Avoid using a vacuum unless certified to handle Class 4.1 PG II metal powders such as aluminum and titanium. May use water to wash spill area. Collect wash water for approved disposal. Avoid allowing water to enter drain.

SECTION 7: HANDLING AND STORAGE**7.1 Handling**

Provide appropriate exhaust ventilation where dust may form

Keep away from sources of ignition

Avoid breathing dust

Use non-sparking tools when opening or closing containers. Avoid sources of spark and employ electrostatic discharge clothing and equipment when possible.

7.2 Storage

Storage: Store at ambient temperatures in tightly sealed container. The area should be suitably marked to indicate the presence of ignitable dust. No-smoking warning should be present. Do not store with reactive materials. Ground storage.

Shelf Life: Unlimited shelf life if kept dry and inert packaged

SECTION 8: EXPOSURE CONTROLS & PERSONAL PROTECTION

8.1 Ventilation Requirements

Keep dust and fume levels below occupational exposure limits. Provide adequate local exhaust ventilation at the work area where spraying and fusing are being done.

8.2 Personal Protective Equipment

Eyes: Wear dust-proof safety goggles. Contact lenses are not recommended.

SKIN: Protective gloves and clothing are recommended.

INHALATION: Do not breathe dust or fume. Use with adequate ventilation. Where appropriate, use of a respirator type N100 (US) or type P3 (EN 143) is recommended. Dust masks alone are insufficient as particles smaller than typical safety standards may be present in small quantities in the powder. Generally, proper engineering controls should be implemented to reduce the chances of dust inhalation as the greater danger is dust combustion under source of ignition.

8.3 Occupational Exposure Limits

Component	CAS-No	Limited Value - 8 hours	Basis	Category
Aluminum	7429-90-5	15 mg/m ³	USA - OSHA	Total Dust
	7429-90-5	5 mg/m ³	USA - OSHA	Respirable Dust
Copper	7440-50-8	0.2 mg/m ³	United Kingdom	-
	7440-50-8	0.01 mg/m ³	Germany	Respirable Fraction
Zinc	7440-66-6	0.1 mg/m ³	Germany	Respirable aerosol
	7440-66-6	0.1 mg/m ³	Switzerland	Respirable aerosol
Chromium	7440-47-3	0.05 mg/m ³	Finland	-
	7440-47-3	0.5 mg/m ³	Japan - JSOH	-
Magnesium	7440-47-3	15 mg/m ³	USA - OSHA	Total Dust
	7440-47-3	4 mg/m ³	Germany	Inhalable aerosol
Manganese	7439-96-5	4 mg/m ³	United Kingdom - HSE	Respirable dust
Silicon	7440-21-3	5 mg/m ³	USA-OSHA	Respirable Fraction

SECTION 9: CHEMICAL AND PHYSICAL PROPERTIES

Property	Descriptor
Appearance and Odor	Grey or silver, odorless
Flammability	-
Autoignition Temperature	Product is not self-igniting
pH	Not applicable
Vapor Pressure	1mm Hg @ 1628°C
Vapor Density	Not determined
Melting Point	660°C
Boiling Point	2460°C
Solubility in Water	Not soluble
Relative Density	2.7 g/cm ³

SECTION 10: STABILITY AND REACTIVITY**10.1 Stability**

Stable under normal conditions.

10.2 Incompatible Materials

Acids, alkalines, oxidizers, bases, halogens.

Some sensitivity to moisture and oxygen, particularly at elevated temperatures

Strong oxidizing agents

10.3 Hazardous Decomposition

Will react exothermically if mixed with a strong oxidizing substance and ignited. Contact with water releases flammable gases.

10.4 Hazardous Polymerization

Will not occur.

10.5 Reactivity

Stable under recommended storage and handling conditions. Will react exothermically if mixed with a strong oxidizing substance and ignited.

10.6 Conditions to Avoid

Formation of dust cloud, high temperatures, contact with water.

SECTION 11: TOXICOLOGICAL INFORMATION**11.1 Potential Health Effects**

Inhalation of metal fumes may cause metal fume fever, a flu-like illness generally lasting 24 hours or less.

11.2 Substance Analysis

Aluminum (7429-90-3)

Oral LD50 Rat: 15900 mg/kg

Magnesium (7439-95-4)

Oral LD50 Rat: >2000 mg/kg

Copper (7440-50-8)

Oral LD50 Rat: 300-500 mg/kg

Inhalation (D50 < 10 microns)

LD50 Rat 1-4 g/m³ air

Chromium (7440-47-3)

Oral LD50 Rat: >3400 mg/kg

Silicon (7440-21-3)

Oral LD50 Rat: >3160 mg/Kg

Manganese (7439-95-5)

Oral LD50 Rat: >3478 mg/Kg

Zinc (7440-66-6)

Oral LD50 Rat: >2000 mg/kg

Iron (7439-89-6)

Oral LD50 Rat: > 30000 mg/Kg

11.3 Information On Toxicological Effects

Routes of Exposure: Eye Contact, Ingestion, Inhalation, Skin Contact, Injection

Carcinogenicity: None known

IARC: No component of this product present at levels greater than or equal to 0.1% is identified as probable, possible or confirmed human carcinogen by IARC.

NTP: No component of this product present at levels greater than or equal to 0.1% is identified as a known or anticipated carcinogen by NTP.

SAFETY DATA SHEET



OSHA: No component of this product present at levels greater than or equal to 0.1% is identified as a carcinogen or potential carcinogen by OSHA.

SECTION 12: ECOLOGICAL INFORMATION

12.1 Ecological Toxicity	12.2 Persistence and Degradability	12.3 Bioaccumulate Potential
No Data Available	No Data Available	No further relevant information
12.4 Mobility in Soil	12.5 Results of PBT and Vpvp Assessment	12.6 Other Adverse Effects
No Data Available	Water Reactive	No Data Available

SECTION 13: DISPOSAL CONSIDERATIONS

Disposal must be in accordance with applicable local, state, and federal regulations. Do not dump into sewers, on the ground, or into any body of water.

SECTION 14: TRANSPORTATION INFORMATION

	14.1 DOT	14.2 ADR/RID	14.3 IMO/IMDG	14.4 ICAO/IATA
UN Number	UN1309	UN1309	UN1309	UN1309
Shipping Name	Aluminum powder, coated	Aluminum powder, coated	Aluminum powder, coated	Aluminum powder, coated
Class	4.1	4.1	4.1	4.1
Packing Group	II	II	II	II

SECTION 15: HAZARDOUS MATERIAL IDENTIFICATION SYSTEM/REFULATORY INFORMATION

Electrical equipment must be suitable for use in hazardous atmospheres involving Group E combustible dusts in accordance with 29CFR1910.307. Refer to the National Electrical Code (NFPA 70) for guidance with determining the type and design of equipment and installation which meets this requirement.

SARA Title III

Section 311/312: Reactive (Sudden Release of Pressure)

Section 313 Toxic Chemicals: Aluminum (Fume or Dust)
Chromium (1.0% de minimus if present)
Copper (1.0% de minimus if present)
Silver (1.0% de minimus if present)
Zinc (Fume or Dust) (1.0% de minimus if present)

SECTION 16: OTHER INFORMATION

Prepared on 10JAN2020 with resources we believe to be reliable and up to date. This SDS is correct to the best of our knowledge. Although reasonable care has been taken in the preparation of this document to assess and summarize the hazard properties of this product, the user must satisfy him/herself that the information contained herein is pertinent to permit the safe handling under the use conditions since the supplier cannot foresee all conditions of use.



National Institute of Standards & Technology

Certificate

Standard Reference Material[®] 660b

Line Position and Line Shape Standard for Powder Diffraction

This Standard Reference Material (SRM) is intended for use in calibration of diffraction line positions and line shapes determined through powder diffractometry. A unit of SRM 660b consists of approximately 6 g of lanthanum hexaboride, LaB₆, powder bottled under argon.

Material Description: The SRM was prepared in a dedicated processing run using the ¹¹B isotope enriched to a nominal 99 % concentration. This isotopic enrichment renders this SRM relevant to the neutron diffraction community. The resulting powder was then annealed and bottled under argon. An analysis of X-ray powder diffraction data indicated that the SRM material is homogeneous with respect to diffraction properties.

Certified Value: The certified lattice parameter for a temperature of 22.5 °C is

$$0.415\,689\,\text{nm} \pm 0.000\,008\,\text{nm}$$

The interval defined by this value and its expanded uncertainty ($k = 2$) is dominated by a Type B uncertainty estimated from a technical understanding of the measurement data and its distribution [1]. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or taken into account. A certified value is the present best estimate of the true value based on the results of analyses performed at NIST.

Information Values: The fundamental parameters analyses included refinement of the full-width half-maximum (FWHM) of a Lorentzian profile to account for sample-induced broadening. The angular dependence of the FWHM term varying as $1/\cos \theta$ is interpreted as size-induced broadening. The value obtained was consistent with a domain size of approximately 0.7 μm . The term varying as $\tan \theta$, interpreted as microstrain, refined to zero. The information values for computed peak positions are given in Table 1. The typical particle size distribution as determined by laser scattering is given in Figure 1. An information value is considered to be a value that will be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value.

Expiration of Certification: The certification of SRM 660b is valid indefinitely, within the uncertainty specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Use"). The certification is nullified if the SRM is damaged, contaminated or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

The overall coordination of the preparation and technical direction of the certification were performed by J.P. Cline, D. Black, D. Windover, and A. Henins of the NIST Ceramics Division.

The collection of the laser scattering particle size data for informational value was performed by M. Peltz of the NIST Materials and Construction Research Division.

Contributions towards the optimization of the annealing schedule, and therefore, the microstructure were provided by B. Toby, Argonne National Laboratory, Argonne, IL USA, A. Fitch, European Synchrotron Research Facility, Grenoble, France, and W.I.F. David, Rutherford Appleton Laboratory, Didcot, UK. J. Evans of Durham University, Durham, UK developed a template for the input files used in data analysis.

Debra L. Kaiser, Chief
Ceramics Division

Gaithersburg, MD 20899
Certificate Issue Date: 29 April 2010

Robert L. Watters, Jr., Chief
Measurement Services Division

SRM 660b

Page 1 of 5

Statistical analysis was by provided J.J. Filliben of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

INSTRUCTIONS FOR USE

Storage: SRM 660b was bottled under argon to protect against humidity. When not in use, store the unused portion of this powder tightly capped in the original bottle or in a manner with similar or greater protection against humidity.

SOURCE, PREPARATION, AND ANALYSIS¹

Source of Material: The ¹¹B-enriched boron carbide precursor was obtained from Ceradyne Boron Products LLC, Quapaw, OK, USA. The lanthanum hexaboride was synthesized by H.C Starck GmbH, Goslar, Germany. The annealing was performed by Cerac Inc., Milwaukee, WI, USA.

Certification Method: Certification was performed using data from a NIST-built diffractometer that includes several advanced design features. The optical layout is that of a conventional divergent-beam diffractometer of Bragg-Brentano geometry. Rigorous analyses of data from said geometry requires knowledge of both the diffraction angle and the effective source-sample-detector distance. Two additional models must therefore be included in the data analyses to account for the factors that affect the distances critical in the use of this geometry. The inclusion of these models places additional uncertainties on the certified lattice parameters relative to those determined from a parallel beam instrument. Linkage to the International System of Units (SI) is established via the emission spectrum of Cu K α radiation employed as the basis for constructing the diffraction profiles via the fundamental parameters approach (FPA) [2] method of data analyses. Data were analyzed in the context of both Type A uncertainties, assigned by statistical analysis, and Type B uncertainties, based on knowledge of the nature of errors in the measurements, to result in the establishment of robust uncertainties for the certified values.

The aforementioned NIST-built diffractometer is of θ - 2θ geometry and is of a conventional optical layout, though it is built with several features atypical of conventional equipment of this nature. The θ and 2θ motions of the goniometer assembly are provided by Huber 420 rotation stages that are actuated via a worm gear driving a ring gear. These are mounted concentrically with the rotation axes horizontal, allowing for utilization of an automatic sample changer/spinner. The alignment specifications realized for the goniometer assembly matched those cited by the manufacturer for the individual stages: an eccentricity (concentricity) of less than 3 μ m, and a wobble (parallelism) of less than 0.0008° (3 arc-seconds). Both stages incorporate Heidenhain optical encoders mounted so as to measure the angle of the ring gear. The encoders with the associated Heidenhain IK220 interpolation electronics provide ± 1 arc-second accuracy, and approximately 0.035 arc-second precision. The optics, X-ray generator, tube shield, and sample spinner of the machine are conventional in nature; they were originally components of a Siemens D5000 diffractometer, ca. 1992.

Certification Procedure: The 2.2 kW sealed copper tube of long fine-focus geometry was operated at a power of 1.8 kW during certification measurements. The source size was approximately 12 mm $\times$ 0.04 mm and the variable divergence slit was set nominally to 0.8°. Axial divergence of the incident beam was limited by a 2.2° Soller slit. The goniometer radius was 217.5 mm. A 2 mm anti-scatter slit was placed approximately 113 mm in front of the 0.2 mm (0.05°) receiving slit. Scattered X-rays were filtered with a graphite post-sample monochromator, and detected with a scintillation detector. Samples were spun at 0.5 Hz during data collection. The machine was located within a temperature-controlled laboratory space where the nominal short-range control of temperature was ± 0.1 K. The instrument was controlled via LabVIEW software. Data were recorded in true x-y format. The source was allowed to equilibrate at operating conditions for at least an hour prior to recording any certification data. The performance of the machine was qualified with the use of NIST SRM 660a Lanthanum Hexaboride Powder Line Position and Line Shape Standard for Powder Diffraction [3] and SRM 676a Alumina Powder for Quantitative Analysis by X-Ray Diffraction [4] using procedures discussed by Cline [5].

Ten units of SRM 660b were selected in a stratified random manner from the population of units during the bottling operation. Certification data were recorded from 2 samples prepared from each of 10 bottles, for a total of 20 samples. Data were collected from 24 selected regions of the diffraction pattern, each region including one of the

¹Certain commercial equipment, instruments, or materials are identified in this in order to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

reflections accessible within the 2θ range of 20° to 150° . The angular widths of the scan ranges were 20 to 30 times the observed FWHM values of the profiles and were chosen to provide at least 0.3° 2θ of apparent background straddling each peak. The step width was chosen to include at least eight data points above the FWHM. The count time spent on each profile was inversely proportional to the observed diffraction intensity so as to realize constant counting statistics amongst the profiles. The total collection time for each sample was about 24 hours.

Data Analysis: The certification data were analyzed using the FPA method with a Rietveld [6-8] refinement as implemented in TOPAS [9]. The analysis used the Cu $K\alpha_1/K\alpha_2$ emission spectrum, including a satellite component, as characterized by G. Hölzer *et al.* [10,11]. The Lorentzian breadths of the Cu emission spectrum were refined with constraints to preserve asymmetric profile shape as modeled by Hölzer. The refined parameters included the scale factors, second-order Chebyshev polynomial terms for modeling of the background, the lattice parameters, the intensities and position of the $K\alpha_2$ and satellite components of the Cu $K\alpha$ emission spectrum, terms indicating the position and intensity of the “tube tails” [12], a Soller slit value in the “full” axial divergence model [13], specimen displacement, an attenuation term, structural parameters, and a size-broadening term of a Lorentzian profile.

Examination of the fit to the individual profiles revealed a discrepancy between the model and the observations in the low-angle region. It is well known that low-angle profiles are more prone to error than high-angle lines as the optical aberrations affecting their position are more complex. Also, the reported lattice parameter is more strongly affected by angular errors in the low-angle region. The 100 and 110 lines were, therefore, not used in obtaining the certified parameters. The thermal expansion of lanthanum hexaboride as reported by Sirota *et al.* [14] was used to adjust the lattice parameter to 22.5°C . A statistical analysis of the data indicated that the mean of the measurements was $0.415\,688\,46\text{ nm}$ with a $k=2$ Type A expanded uncertainty of $0.000\,000\,25\text{ nm}$. However, a Type B uncertainty due to systematic error must be incorporated into the uncertainty bounds of the certified lattice parameter. Consideration of trends in the data used in the certification leads to an assignment of a Type B uncertainty and value as stated on page 1.

Table 1. Information Values for Peak Positions Computed for SRM 660b Using Cu $K\alpha$ Radiation,
 $\lambda = 0.154\,059\,29\text{ nm}$

h	k	l	2θ , degrees
1	0	0	21.358
1	1	0	30.385
1	1	1	37.442
2	0	0	43.507
2	1	0	48.958
2	1	1	53.989
2	2	0	63.219
3	0	0	67.548
3	1	0	71.746
3	1	1	75.844
2	2	2	79.870
3	2	0	83.846
3	2	1	87.792
4	0	0	95.672
4	1	0	99.643
3	3	0	103.661
3	3	1	107.749
4	2	0	111.934
4	2	1	116.245
3	3	2	120.723
4	2	2	130.409
5	0	0	135.800
5	1	0	141.775
5	1	1	148.679

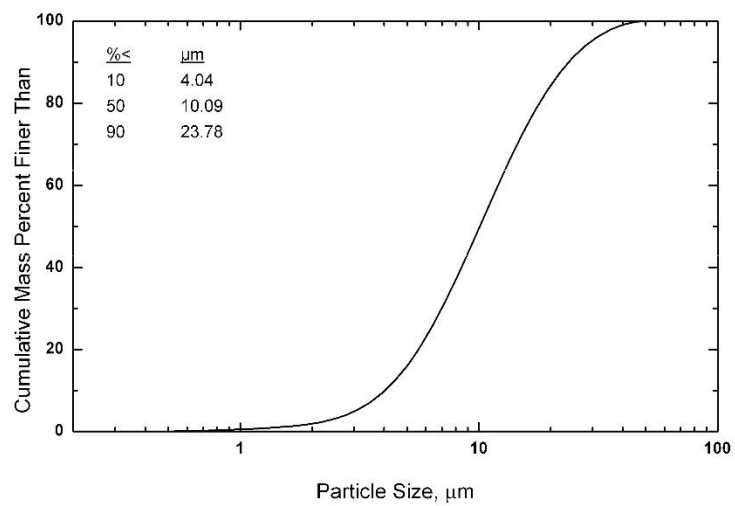


Figure 1. Typical Particle Size Distribution as Determined by Laser Scattering

REFERENCES

- [1] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (ISO GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (2008); available at http://www.bipm.org/utls/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Apr 2010); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297; U.S. Government Printing Office: Washington, DC (1994); available at <http://www.nist.gov/physlab/pubs/index.cfm> (accessed Apr 2010).
- [2] Cheary, R.W.; Coelho, A.A.; *A Fundamental Parameters Approach to X-ray Line-Profile Fitting*; J. Appl. Cryst., Vol. 25, pp. 109-121 (1992).
- [3] SRM 660a; *Lanthanum Hexaboride Powder Line Position and Line Shape Standard for Powder Diffraction*; National Institute of Standards and Technology; U.S. Department of Commerce: Gaithersburg, MD (13 September 2000).
- [4] SRM 676a; *Alumina Internal Standard for Quantitative Analysis by X-ray Powder Diffraction*; National Institute of Standards and Technology; U.S. Department of Commerce: Gaithersburg, MD (28 January 2008).
- [5] Cline, J.P.; *Use of NIST Standard Reference Materials for Characterization of Instrument Performance*; Chapter in *Industrial Applications of X-ray Diffraction*, ed. by F.H. Chung and D.K. Smith, pub. by Marcel Dekker, Inc, pp 903-917 (2000).
- [6] Rietveld, H.M.; *Line Profiles of Neutron Powder Diffraction Peaks for Structure Refinement*, Acta Crystallogr., Vol. 22, pp. 151-152 (1967); Rietveld, H.M., *A Profile Refinement Method for Nuclear and Magnetic Structures*; J. Appl. Cryst., Vol. 2, pp. 65-71 (1969).
- [7] Young, R.A.; *The Rietveld Method*; Oxford University Press: New York (1993).
- [8] Bish, D.L.; Post, J.E., Eds.; *Modern Powder Diffraction*; Rev. Mineral., Vol. 20, p. 369 (1989).
- [9] TOPAS, *General Profile and Structure Analysis Software for Powder Diffraction Data*; V4.2, Bruker AXS GmbH, Karlsruhe, Germany.
- [10] Hölzer, G.; Fritsch, M.; Deutsch, M.; Härtwig, J.; Förster, E.; *K $\alpha_{1,2}$ and K $\beta_{1,3}$ X-Ray Emission Lines of the 3d Transition Metals*; Phys. Rev. A, Vol. 56, Issue (6), pp. 4554-4568 (1997).
- [11] Maskil, M.; Deutsch, M.; *X-Ray K α Satellites of Copper*; Phys. Rev. A, Vol. 38, pp. 3467-3472 (1988).
- [12] Bergmann, J.; Kleeberg, R.; Haase, A.; Breidenstein, B.; *Advanced Fundamental Parameters Model for Improved Profile Analysis*; In *Proceedings of the 5th European Conference on Residual Stresses*, Delft-Noordwijkerhout, The Netherlands, September 29-30, 1999, Editors: A.J. Böttger, R. Delhez, and E.J. Mittemeijer, Trans Tech Publications, 347-349, pp. 303-308 (2000).
- [13] Cheary, R.W.; Coelho, A.A.; *Axial Divergence in a Conventional X-Ray Powder Diffractometer I. Theoretical Foundations*, J. Appl. Cryst., Vol. 31, pp. 851-861 (1998), and Cheary, R.W.; Coelho, A.A.; *Axial Divergence in a Conventional X-Ray Powder Diffractometer II, Implementation and Comparison with Experiment*, J. Appl. Cryst., Vol. 31, pp. 862-868 (1998).
- [14] Sirota, N.N.; Novikov, V.V.; Vinokrov, V.A.; Paderno, B.Yu.; *Temperature Dependence of Heat Capacity and Lattice Constant of Lanthanum and Samarium Hexaborides*; Phys. Solid State, Vol. 40 (11), pp. 1856-1858 (1998).

Users of this SRM should ensure that the Certificate in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at <http://www.nist.gov/srm>.

GILSON COMPANY, INC.
COMPLIANCE TEST SIEVE
CERTIFICATION

P.O. BOX 200, LEWIS CENTER, OHIO 43035-0200 USA
Phone: 800-444-1508
E-mail: customerservice@gilsonco.com

This certifies that this compliance test sieve has been produced using manufacturing methods and quality control procedures to assure conformity to the standards listed below (latest revisions) at time of shipment. If found to be outside of tolerances prior to use, this test sieve will be replaced free of charge. Contact Gilson for optional Verification of this sieve to Inspection or Calibration Grade.

ASTM E11

ISO 565
ISO 3310-1

BS 410

Serial Number: 151934449



STANDARD SPECIFICATION FOR THE WOVEN WIRE TEST SIEVE CLOTH AND TEST SIEVES



Nominal dimensions and permissible variations for sieve cloth (mesh) for Compliance, Inspection and Calibration grade test sieves

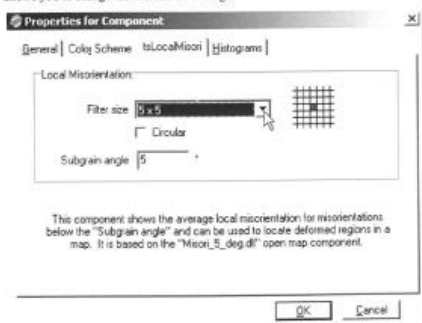
Sieve Designation		Nominal Sieve Opening	± Y Variation for Average Opening	+ X Maximum Variation for Opening	Resulting Maximum Individual Opening	Typical Wire Diameter	Permissible Average Wire Diameter	
U.S. Alternative	Standard						Min	Max
		Millimeters	inches	Millimeters	Millimeters	Millimeters		
5"	125	5.00	3.300	4.06	129.06	8.00	6.8	9.2
4.24"	106	4.24	2.800	3.59	109.59	6.30	5.4	7.2
4"	100	4.00	2.650	3.44	103.44	6.30	5.4	7.2
3-1/2"	90	3.50	2.390	3.18	93.18	6.30	5.4	7.2
3"	75	3.00	2.000	2.78	77.78	6.30	5.4	7.2
2-1/2"	63	2.50	1.690	2.44	65.44	5.60	4.8	6.4
2.12"	53	2.12	1.420	2.15	55.15	5.00	4.3	5.8
2"	50	2.00	1.340	2.06	52.06	5.00	4.3	5.8
1-3/4"	45	1.75	1.210	1.91	46.91	4.50	3.8	5.2
1-1/2"	37.5	1.50	1.010	1.67	39.17	4.50	3.8	5.2
1-1/4"	31.5	1.25	0.855	1.47	32.97	4.00	3.4	4.6
1.06"	26.5	1.06	0.722	1.29	27.79	3.55	3.0	4.1
1.00"	25	1.00	0.682	1.24	26.24	3.55	3.0	4.1
7/8"	22.4	0.875	0.613	1.14	23.54	3.55	3.0	4.1
3/4"	19	0.750	0.522	1.01	20.01	3.15	2.7	3.6
5/8"	16	0.625	0.441	0.89	16.89	3.15	2.7	3.6
0.530"	13.2	0.530	0.365	0.78	13.98	2.80	2.4	3.2
1/2"	12.5	0.500	0.346	0.75	13.25	2.50	2.1	2.9
7/16"	11.2	0.438	0.311	0.69	11.89	2.50	2.1	2.9
3/8"	9.5	0.375	0.265	0.61	10.11	2.24	1.9	2.6
5/16"	8	0.312	0.224	0.54	8.54	2.00	1.7	2.3
0.265"	6.7	0.265	0.189	0.48	7.18	1.80	1.5	2.1
1/4"	6.3	0.250	0.178	0.46	6.76	1.80	1.5	2.1
#3.5	5.6	0.223	0.159	0.42	6.02	1.60	1.3	1.9
#4	4.75	0.187	0.135	0.37	5.12	1.60	1.3	1.9
#5	4	0.157	0.114	0.33	4.33	1.40	1.2	1.7
#6	3.35	0.132	0.096	0.29	3.64	1.25	1.06	1.50
#7	2.8	0.110	0.081	0.26	3.06	1.12	0.95	1.30
#8	2.36	0.0937	0.069	0.23	2.59	1.00	0.85	1.15
#10	2	0.0787	0.059	0.20	2.20	0.90	0.77	1.04
#12	1.7	0.0661	0.050	0.18	1.88	0.80	0.68	0.92
#14	1.4	0.0555	0.042	0.16	1.56	0.71	0.60	0.82
#16	1.18	0.0469	0.036	0.140	1.320	0.63	0.54	0.72
#18	1	0.0394	0.030	0.130	1.130	0.560	0.480	0.64
		Micrometers	inches	Micrometers	Micrometers	Micrometers		
#20	850	0.0331	26.2	114	964	0.500	0.43	0.58
#25	710	0.0278	22.2	101	811	0.450	0.38	0.52
#30	600	0.0234	19.0	91	691	0.400	0.34	0.46
#35	500	0.0197	16.2	80	580	0.315	0.27	0.36
#40	425	0.0165	14.0	73	498	0.280	0.24	0.32
#45	355	0.0139	12.0	65	420	0.224	0.19	0.26
#50	300	0.0117	10.4	58	358	0.200	0.17	0.23
#60	250	0.0098	8.9	52	302	0.160	0.13	0.19
#70	212	0.0083	7.8	47	259	0.140	0.12	0.17
#80	180	0.0070	6.8	43	223	0.125	0.106	0.150
#100	150	0.0059	6.0	38	188	0.100	0.085	0.115
#120	125	0.0049	5.2	34	159	0.090	0.077	0.104
#140	106	0.0041	4.7	31	137	0.071	0.060	0.082
#170	90	0.0035	4.2	29	119	0.063	0.054	0.072
#200	75	0.0029	3.7	26	101	0.050	0.043	0.058
#230	63	0.0025	3.4	24	87	0.045	0.038	0.052
#270	53	0.0021	3.1	21	74	0.036	0.031	0.041
#325	45	0.0017	2.8	20	65	0.032	0.027	0.037
#400	38	0.0015	2.6	18	56	0.030	0.024	0.035
#450	32	0.0012	2.4	17	49	0.028	0.023	0.033
#500	25	0.0010	2.2	15	40	0.025	0.021	0.029
#635	20	0.0008	2.1	13	33	0.020	0.017	0.023

Electron Backscatter Diffraction Local Misorientation Manual:

Local Misorientation Component

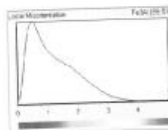
This component is for displaying small orientation changes on the map, highlighting regions of higher deformation. The component calculates the average misorientation between every pixel and its surrounding pixels, and assigns the mean value to that pixel. Misorientations over a certain value are discarded, so that the misorientations associated with discrete subgrain and grain boundaries are excluded.

To display the local misorientation, drag the component from the component library to the relevant phase in the map (you may have to add it to your component library from the component gallery). Double clicking on the component allows you to edit the settings - choosing the tsLocalMisori tab allows you to change the calculation settings:



Increasing the filter size will produce a smoother misorientation map, decreasing it will highlight more local variations.

Select the Color Scheme tab to edit the display settings for the component. The rainbow colour scheme generally highlights the misorientations very well.

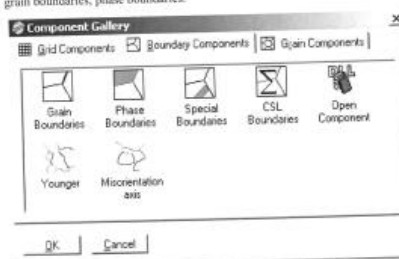


Misorientation map of a deformed intermetallic using a 5x5 filter size, with the legend shown to the left.

Note: This component can take a long time to calculate for large maps

Boundary components

Boundary components correspond to measurements between grid points, e.g. grain boundaries, phase boundaries.



B. Publications

- Paper, [*in process*] Primary Author: *Influence of Solidification History and Particle Size on Defect Evolution in Impulse Atomized Fe-Al Alloy*
- Paper, [*in review*] Primary Author (Journal of Applied Crystallography): *Influence of Solidification History and Particle Size on Defect Evolution in Al2024 Alloy Additive Manufacturing Powder*
- Paper, Primary Author (IOP Science): *Quantifying X-Ray Diffractometer Precision for the Measurement of Dislocation Density and the Evolution of Defect Structures During Solidification of Additive Manufacturing Powders* [97]
- Paper, Coauthor (Materialia): *Particle Size Effects On Dislocation Density, Microstructure, And Phase Transformation For High-entropy Alloy Powders* [56]
- Master's Thesis (ProQuest): *Evaluation of Particle Size Dependent Crystal Defects in Inconel 718 by Line Profile Analysis of X-Ray Diffraction* [96]
- Undergraduate Thesis (Tufts Archive): *Characterizing the Microstructure of Inconel 718 Powder Using X-Ray Diffractometry* [95]
- Contributor: *Deformation induced frequency shifts of oscillating droplets during molten metal surface tension measurement* [108]
- Contributor: *Identifying metastable interface potency limits during steel alloy transformation* [109]
- Contributor: *Retained free energy as a driving force for phase transformation during rapid solidification of stainless steel alloys in microgravity* [110]
- Presentation, Coauthor (ASGSR): *Measurement Of Mass Evaporation And Density Of Commercial Superalloys By Electrostatic Levitation (ESL)* [111]
- Presentation (Tufts University Senior Honors Thesis Poster Session): *Quantifying the Influence of Powder Size and Processing History on Inconel 718 Solidification Structures Using Diffractometry and Scanning Electron Microscopy*
- Conference Presentation (MS&T 21): *An Analysis Of The Dislocation Density Of Inconel 718 Additive Manufacturing Powder*

C. Conferences

1 Materials Science & Technology, October 21st, 2021, Columbus, Ohio, USA.

**AN ANALYSIS OF THE DISLOCATION
DENSITY OF INCONEL 718
ADDITIVE MANUFACTURING POWDER**

Colby Azersky¹
Sangho Jeon², Peggy Cebe², and Douglas Matson²

NASA Goddard Space Flight Center¹ and Tufts University²

MS&T21 MATERIALS SCIENCE & TECHNOLOGY
Technical Meeting and Exhibition

October 17–21, 2021 | Columbus, Ohio

1

2 Modelling of Casting, Welding and Advanced Solidification Processes, June 23rd, 2023, Banff, Alberta, Canada

MCWASPXVI
MODELLING OF CASTING, WELDING AND ADVANCED SOLIDIFICATION PROCESSES

**Quantifying X-Ray Diffractometer Precision for the Measurement
of Dislocation Density and the Evolution of Defect Structures
During Solidification of Additive Manufacturing Powders**

Colby Azersky¹
Sangho Jeon², Charlie Settens³, Muneeba Fakhru⁴, and Douglas Matson⁴

NASA Goddard Space Flight Center¹, Greenbelt, Maryland, MD 20771, USA
Deutsches Elektronen-Synchrotron (DESY)², Notkestraße 85 D-22607 Hamburg, Germany
Massachusetts Institute of Technology (MIT)³, X-ray Diffraction Shared Experimental Facility, Cambridge, MA 02139, USA
Tufts University⁴, Department of Mechanical Engineering, Medford, MA 02155, USA

D. Software Code

Automated Williamson-Hall Solver coded in MATLAB.

```
% Colby Azersky
% Automated Williamson Hall
% 11/05/2021
clc
clear all
close all

% Associated step from analysis guide:
% plot I vs. J: J = 100000*((col(E)^2-col(Q))/col(C)^2)

% User input
I = [0.33333,0,0.25];
C = [4.79961,5.54241,7.84915];
E = [0.02669,0.03272,0.03794];

% Initialization
Amatrix = linspace(1E-5,1E-3,1000);
errormatrix=NaN(length(Amatrix),1);

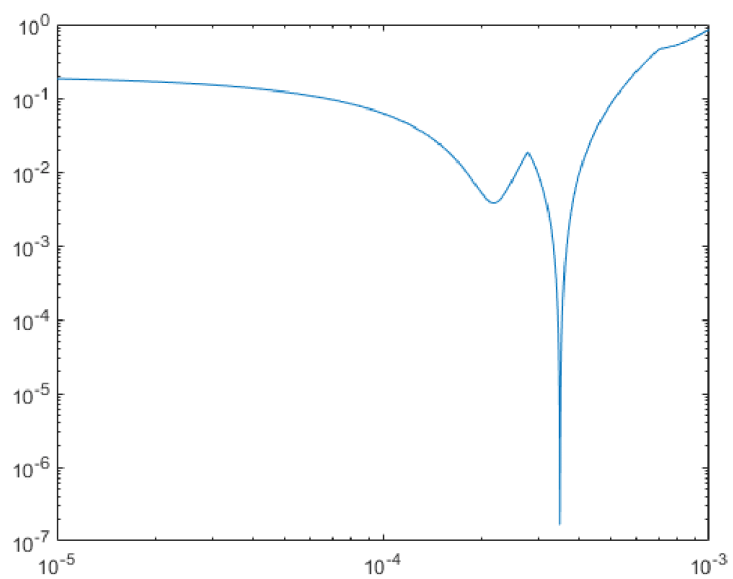
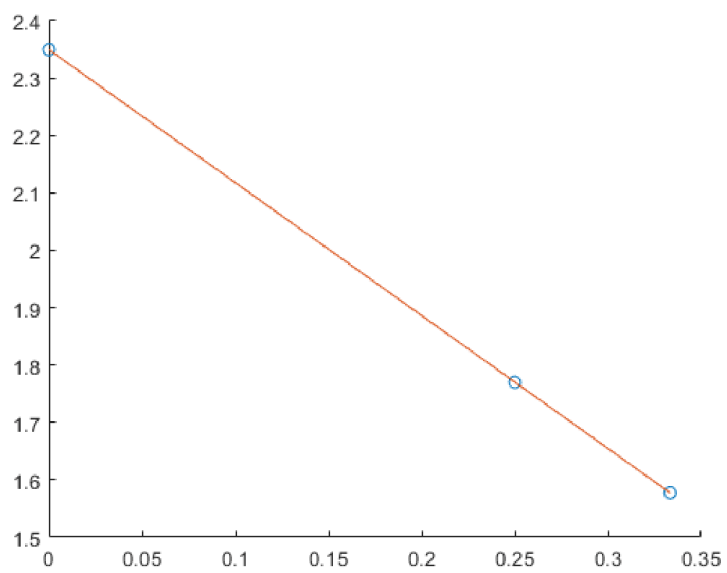
% Iteration
for i=1:length(Amatrix)
    BestFit=Amatrix(i);
    J = 100000*((E.^2-BestFit)./C.^2);
    P = polyfit(I,J,1);
    x=sort(I);
    y=x*P(1)+P(2);
    J=sort(J);
    y=sort(y);
    error = sum((J-y).^2);
    errormatrix(i)=error;
end
[M,I3] = min(errormatrix);

% Result
BestFit=Amatrix(I3)

% Plots
J = 100000*((E.^2-BestFit)./C.^2);
P = polyfit(I,J,1);
x=linspace(min(I),max(I),length(I));
y=x*P(1)+P(2);
scatter(I,J)
hold on
plot(x,y)
figure
loglog(Amatrix,errormatrix)

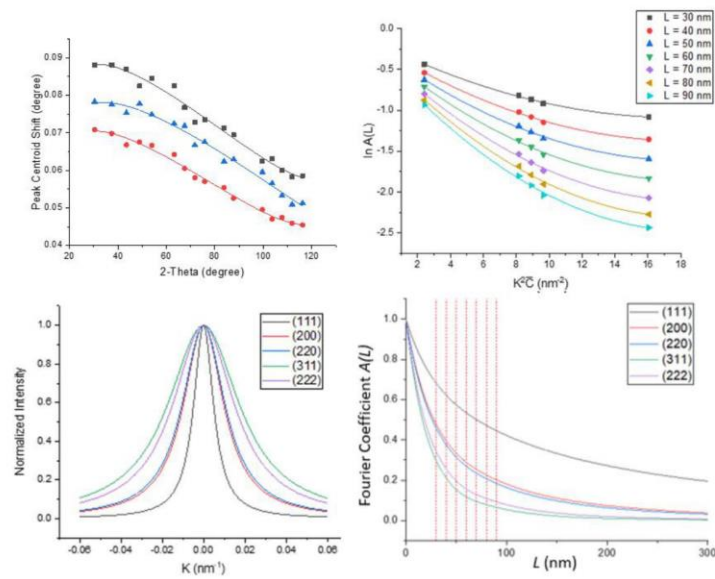
BestFit =

    3.4892e-04
```



E. Dislocation Density Analysis Guide

Advanced Dislocation Density Analysis Walkthrough



Written by Colby Azersky
Support from: Sangho Jeon and Dr. Douglas Matson
2019 - 2020

TABLE OF CONTENTS

MOTIVATION	1
SOFTWARE REQUIREMENTS	2
FILE PREPARATION	3
PEAK LOCATION CALIBRATION	6
PEAK BREADTH CALIBRATION	25
NOISE BASELINE CALIBRATION	34
PEAK CURVE FITTING	36
WILLIAMSON-HALL ANALYSIS	40
CURVE SIMULATION	46
FOURIER TRANSFORMATON	52
WARREN-AVERBACH ANALYSIS	56

MOTIVATION

This walkthrough is intended to take the user through the advanced dislocation density analysis procedure in short and simple steps. Without any prior experience, one should be able to follow this guide from start to finish to evaluate the dislocation density of a material using x-ray diffraction data. The research datasets analyzed in this walkthrough consist of high-resolution XRD scans of individual diffraction peaks. The calibration dataset analyzed in this walkthrough is a large angle range XRD scan of a standard reference material, lanthanum hexaboride.

This procedure uses the Wilson formula and the Caglioti formula to calibrate the peak centroid and peak breadth of the x-ray diffraction data. Then, the Modified Williamson-Hall and Warren-Averbach plots are used to derive dislocation density:

Wilson Formula:

$$\Delta_{2\theta}(\theta) = ax \tan^{-1} \theta + bx + cx \tan \theta + dx \tan^2 \theta + ex \tan^3 \theta$$

Caglioti Formula:

$$\text{FWHM}(\theta) = [W + V \tan \theta + U \tan^2 \theta]^{1/2}$$

Modified Williamson-Hall Equation:

$$\Delta K \cong \frac{0.9}{D} + \left(\frac{\pi M^2 b^2}{2} \right)^{1/2} \rho^{1/2} K \bar{C}^{1/2} + O(K^2 \bar{C})$$

Modified Warren-Averbach Equation:

$$\ln A(L) \cong \ln A^S(L) - \rho \frac{\pi b^2}{2} L^2 \ln \left(\frac{R_c}{L} \right) (K^2 \bar{C}) + Q(K^4 \bar{C}^2)$$

First, the x-ray diffraction data will be calibrated using a LaB₆ (lanthanum hexaboride) standard. The calibration will correct both line position and instrumental broadening effects. Next, the background noise will be removed, and the datasets will be fitted with Lorentzian curves. After, analysis using the Williamson-Hall equation will be used to deduce the dislocation character of the sample. Then, each diffraction peak will be isolated from the raw data, normalized, and subjected to a Fourier Transformation. The resulting transformation will be used in conjunction with the Warren-Averbach equation to derive the dislocation density of the sample.

SOFTWARE REQUIREMENTS

In this walkthrough, the following versions of the following programs were used:

1. Origin 2018
2. HighScore Plus Version 4.7

This analysis is possible with different software, but this guide covers the steps through these specific versions of these programs. Keyboard shortcuts are shown for Windows machines (for example, CTRL+C to copy) use the equivalent shortcut if you are not running Windows.

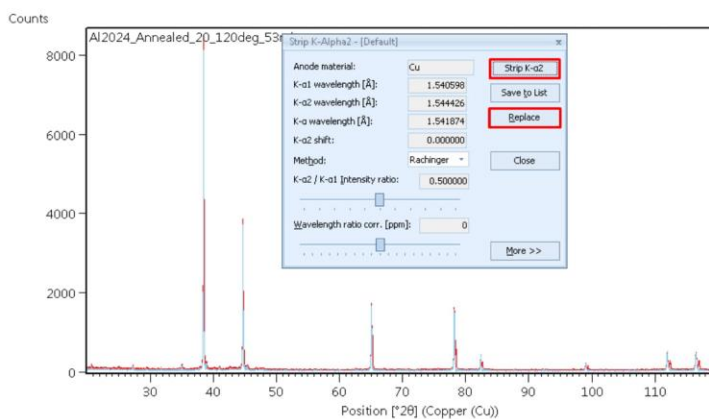
FILE PREPARATION

For all calibration data files:

1. Open HighScore Plus
2. Within the upper menu bar, select "File" > "Open..." then select an XRD data file
3. Within the upper menu bar, select "File" > "Save As..."
4. Within the "Save as type:" dropdown menu, select "General ASCII scan (*.ASC)"
5. Select "Save"
6. Repeat this process for all calibration data files

For all research material data files:

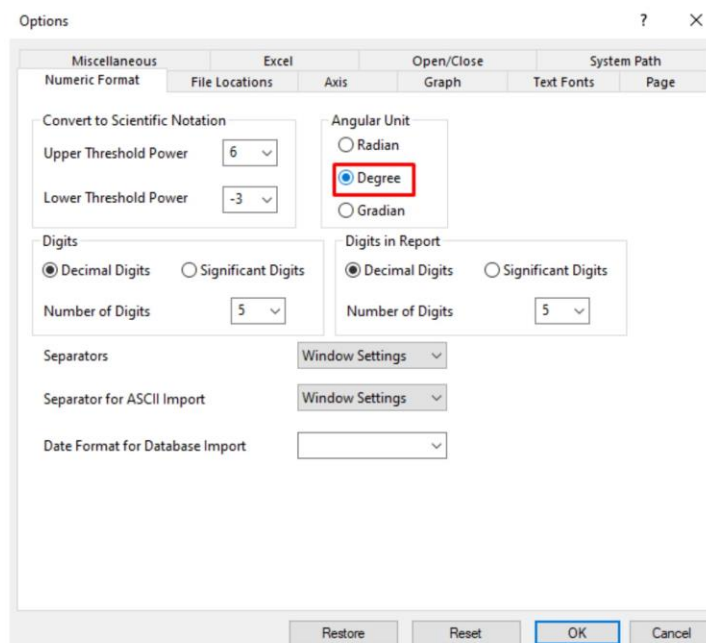
7. Open HighScore Plus
8. Within the upper menu bar, select "File" > "Open..." then select an XRD data file
9. Within the upper menu bar, select "Treatment" > "Strip K-Alpha2..."
10. Select "Strip K-a2"
11. Select "Replace"



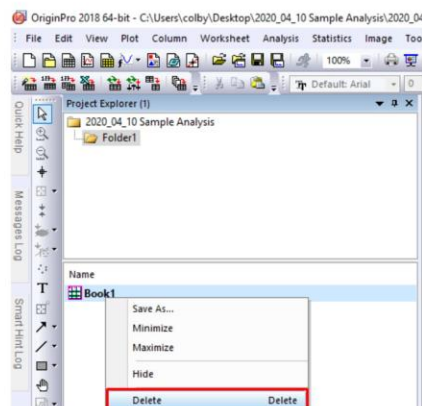
12. Within the upper menu bar, select "File" > "Save As..."
13. Within the "Save as type:" dropdown menu, select "General ASCII scan (*.ASC)"
14. Select "Save"
15. Repeat this process for all research data files

16. Open Origin

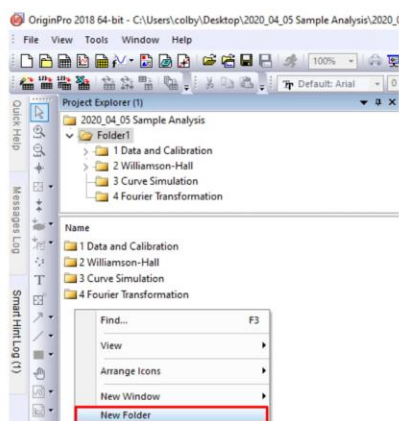
17. Within the upper menu bar, select "Tools" > "Options..." and adjust the "Angular Unit" to "Degree"



18. Right click on "Book1" in the "Project Explorer" section and select "Delete"

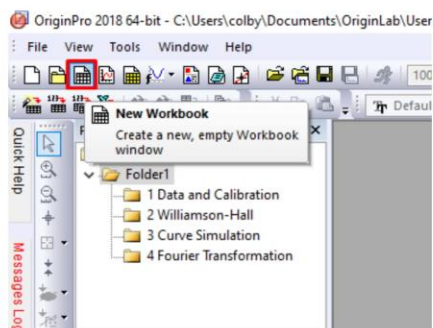


19. Right click in the "Project Explorer" section and create 4 new folders. Name them:
- 1 Data and Calibration
 - 2 Williamson-Hall
 - 3 Curve Simulation
 - 4 Fourier Transformation

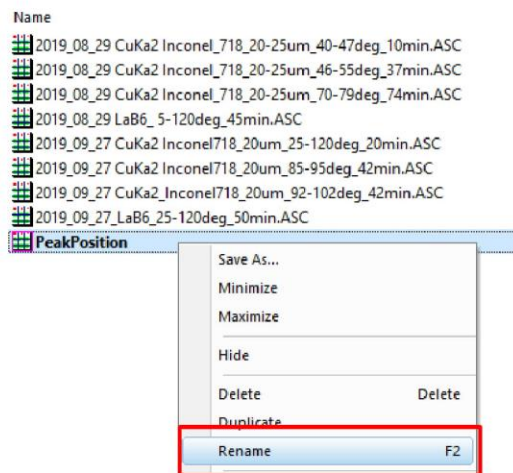


PEAK LOCATION CALIBRATION

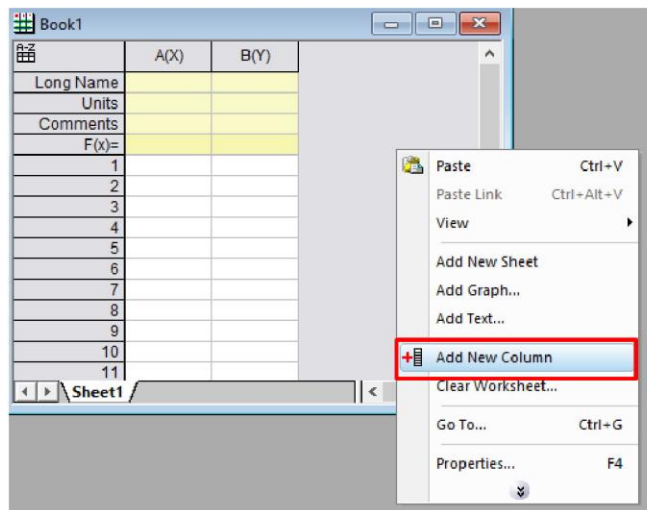
1. Drag and drop your data files into the "1 Data and Calibration" folder
2. Open the "1 Data and Calibration" folder
3. Select the "New Workbook" icon



4. Rename this workbook "PeakPosition"



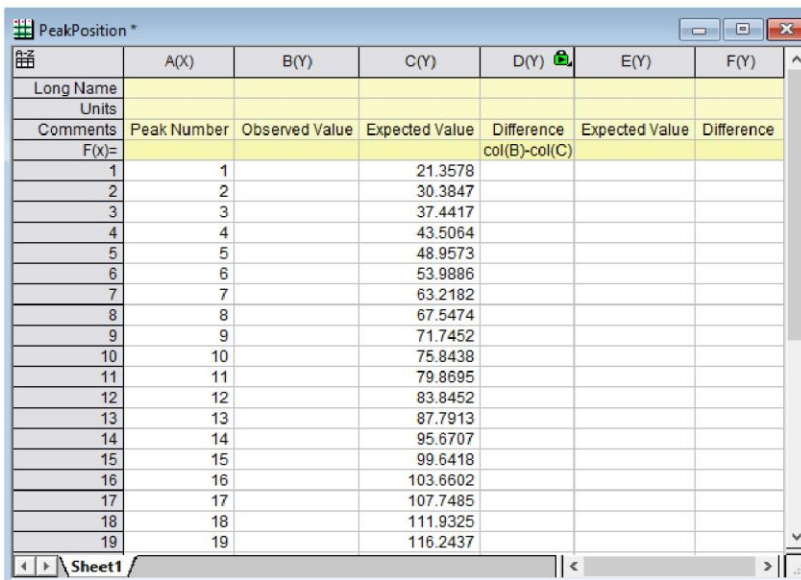
5. Right click on the workbook and select "Add New Column"



6. Add 4 columns (6 total in the workbook)
7. In the "Comments" row:
- Name column A: "Peak Number"
 - Name column B: "Observed Value"
 - Name column C: "Expected Value"
 - Name column D: "Difference"
 - Name column E: "Expected Value"
 - Name column F: "Difference"
8. In the "F(x)=" row of column D, input: " col(B)-col(C) "

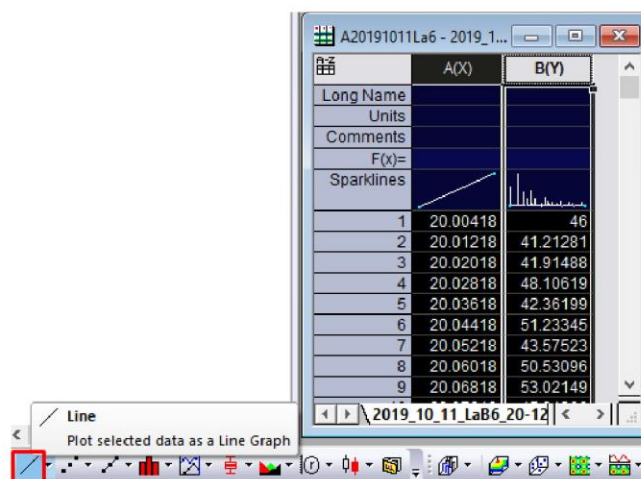
9. Starting at row 1 of the workbook, input:
- In column A, the numbers 1 through 19
 - In column C, the peak position values from the NIST Specification for Standard Reference Material 660a Lanthanum Hexaboride Powder (link below):
https://www.aps.anl.gov/files/APS-Uploads/DET/Detector-Pool/Beamline-Components/NIST_Standard_Reference/660a_Certificate.pdf

The workbook should look like this:



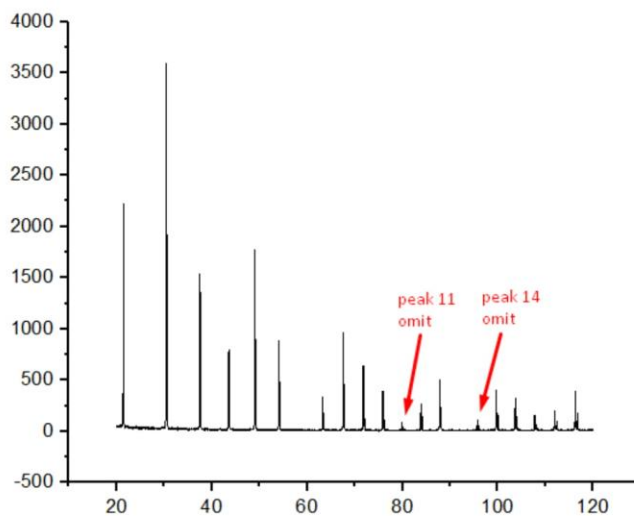
	A(X)	B(Y)	C(Y)	D(Y)	E(Y)	F(Y)
Long Name						
Units						
Comments	Peak Number	Observed Value	Expected Value	Difference	Expected Value	Difference
F(x)=				col(B)-col(C)		
1	1		21.3578			
2	2		30.3847			
3	3		37.4417			
4	4		43.5064			
5	5		48.9573			
6	6		53.9886			
7	7		63.2182			
8	8		67.5474			
9	9		71.7452			
10	10		75.8438			
11	11		79.8695			
12	12		83.8452			
13	13		87.7913			
14	14		95.6707			
15	15		99.6418			
16	16		103.6602			
17	17		107.7485			
18	18		111.9325			
19	19		116.2437			

10. Open the calibration data worksheet
11. Select both columns (*2-theta as X and intensity as Y*)
12. Create a line graph



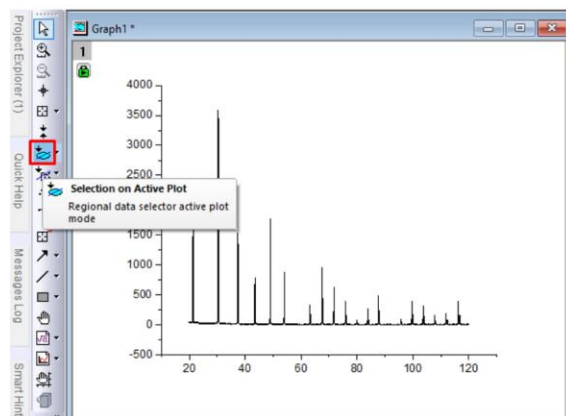
13. Notice the relative intensities of each peak. If there are any peaks with exceptionally small intensities, omit them by inserting "omit" in column B, "Observed Value", in the "PeakPosition" workbook for the corresponding peak row

Sample data shown below:

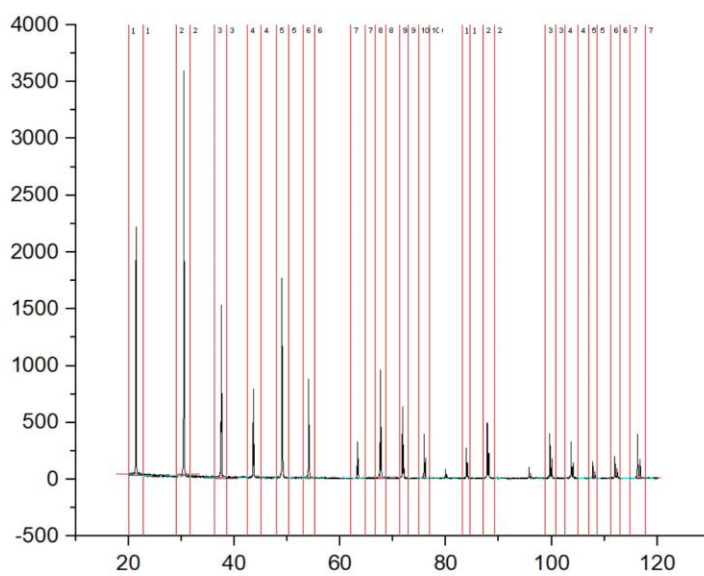


PeakPosition *						
Long Name	A(X)	B(Y)	C(Y)	D(Y)	E(Y)	F(Y)
Units						
Comments	Peak Number	Observed Value	Expected Value	Difference	Expected Value	Difference
F(x)=				col(B)-col(C)		
1	1	--	21.3578	--		
2	2	--	30.3847	--		
3	3	--	37.4417	--		
4	4	--	43.5064	--		
5	5	--	48.9573	--		
6	6	--	53.9886	--		
7	7	--	63.2182	--		
8	8	--	67.5474	--		
9	9	--	71.7452	--		
10	10	--	75.8438	--		
11	11	omit	79.8695	--		
12	12	--	83.8452	--		
13	13	--	87.7913	--		
14	14	omit	95.6707	--		
15	15	--	99.6418	--		
16	16	--	103.6602	--		
17	17	--	107.7485	--		
18	18	--	111.9325	--		
19	19	--	116.2437	--		
20						

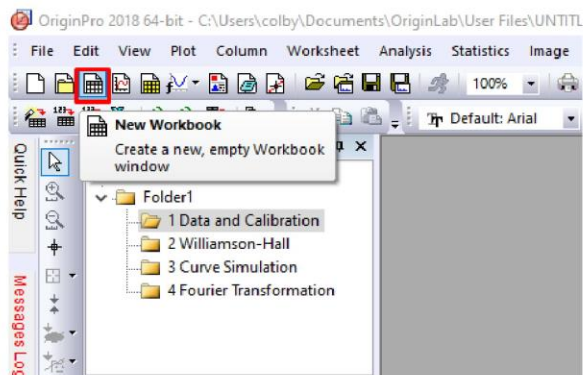
14. Select the "Selection on Active Plot" icon



15. Draw a rectangle around every peak that will be used

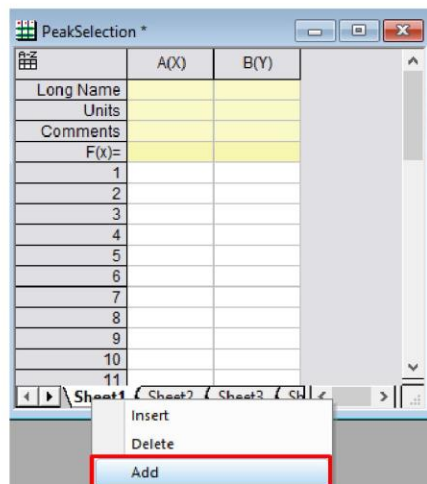


16. Create a new workbook

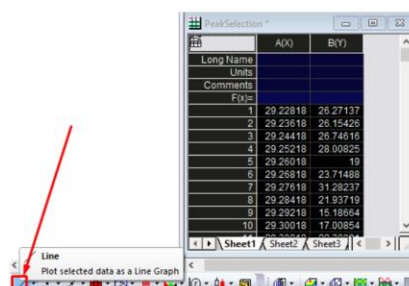


17. Name the new workbook: "PeakSelection"

18. Right click the "Sheet1" icon and select "Add"

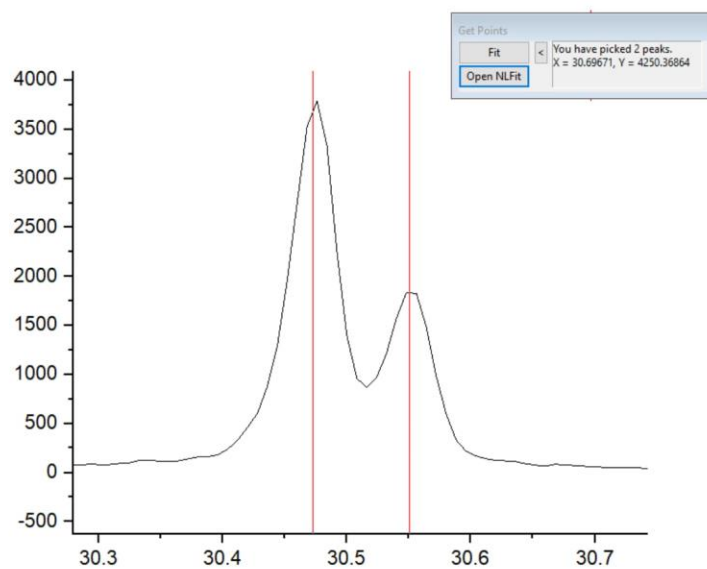
19. Repeat this process until the number of sheets is equal to the number of peaks that will be analyzed (*#total peaks - #omitted peaks, 17 total sheets in this example*)

20. Return to the graph and select the first peak by left clicking on the vertical red lines (*arrows should appear on the top and bottom of the lines if done correctly*)
21. Copy this data selection (CTRL+C on Windows)
22. Return to the "PeakSelection" workbook and paste (CTRL+V on Windows) into "Sheet1"
23. Select both columns
24. Create a line graph
25. Repeat this process for each peak



26. For each graph that has been created: open the graph, within the upper menu bar, select "Analysis" > "Peaks and Baseline" > "Multiple Peak Fit" > "Open Dialog"
27. Within the "Peak Function" drop-down menu, select "Lorentz" then "OK"
28. Click and drag to align the cursor with the left peak center, then double click
29. Click and drag to align the cursor with the right peak center, then double click (*it is very important that this fit is accurate, so using the zoom in tool is helpful*)

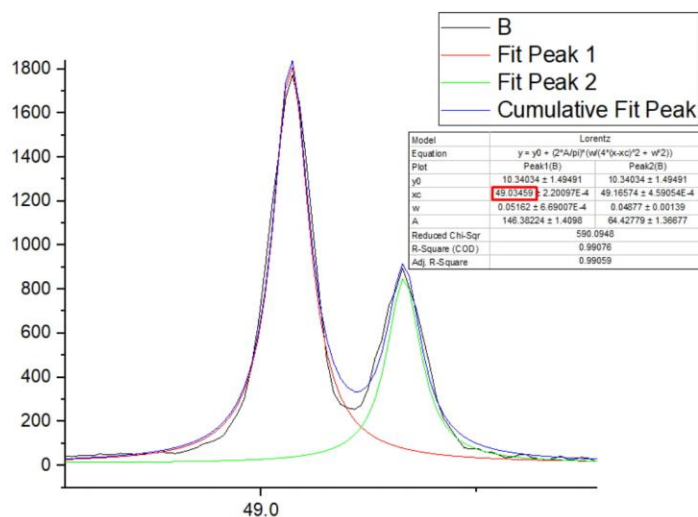
An example is shown below:



30. When finished, select "Fit"

31. Find the “xc” value of the leftmost peak, “Fit Peak 1” and record this value in Column B (Observed Value) of the “PeakPosition” workbook under the row corresponding to the peak number. Omit the +/- value (*Double clicking on this value opens a pop-up that is much larger and easier to read*).

An example is shown below:



Note: for both peak position and peak breadth calibrations, it is important that the Cu-Kα1 radiation (left peak) and Cu-Kα2 radiation (right peak) are properly isolated. The relative intensity of Cu-Kα1 to Cu-Kα2 is roughly 2:1. As a rule of thumb for this analysis, I threw out any data with a relative intensity greater than 2.5:1. At lower angles, the peaks are stacked onto of each other and the curve-fitting software may not be able to properly separate them. In my experience, this typically results in an overestimation of the Cu-Kα1 peak and an underestimation of the Cu-Kα2.

32. Repeat this process for all peaks to fill the "PeakPosition" workbook. (If any value in Column D, "Difference" is particularly large/small compared to the other values, check the curve-fit on the corresponding graph. The curve-fitting process may need to be re-done if the fit is not accurate)
33. Copy the values from the non-omitted peaks from columns C and D and paste them into columns E and F

A completed workbook is shown below:

PeakPosition *						
	A(X)	B(Y)	C(Y)	D(Y)	E(Y)	F(Y)
Long Name						
Units						
Comments	Peak Number	Observed Value	Expected Value	Difference	Expected Value	Difference
F(x)=				col(B)-col(C)		
1	1	21.43722	21.3578	0.07942	21.3578	0.07942
2	2	30.46299	30.3847	0.07829	30.3847	0.07829
3	3	37.51926	37.4417	0.07756	37.4417	0.07756
4	4	43.58176	43.5064	0.07536	43.5064	0.07536
5	5	49.03509	48.9573	0.07779	48.9573	0.07779
6	6	54.06349	53.9886	0.07489	53.9886	0.07489
7	7	63.29068	63.2182	0.07248	63.2182	0.07248
8	8	67.61927	67.5474	0.07187	67.5474	0.07187
9	9	71.81194	71.7452	0.06674	71.7452	0.06674
10	10	75.91139	75.8438	0.06759	75.8438	0.06759
11	11	omit	79.8695	--	83.8452	0.06235
12	12	83.90755	83.8452	0.06235	87.7913	0.06294
13	13	87.85424	87.7913	0.06294	99.6418	0.05956
14	14	omit	95.6707	--	103.6602	0.05653
15	15	99.70136	99.6418	0.05956	107.7485	0.05326
16	16	103.71673	103.6602	0.05653	111.9325	0.0509
17	17	107.80176	107.7485	0.05326	116.2437	0.05127
18	18	111.9834	111.9325	0.0509		
19	19	116.29497	116.2437	0.05127		
20						

34. Right click column A > "Set As" > "Y"

	A(X)	B(Y)	C(Y)	D(Y)	E(Y)	F(Y)	G(Y)
Long Name	Plot						
Units	Copy						
Comments	Copy Columns to...				Expected Value	Difference	
F(x)=	Delete				30.3847	0.08811	
1	Set As	X				0.08809	
2	Set As Categorical	Y				0.08701	
3	Set Column Values... Ctrl+Q	Z				0.08243	
4						0.08459	
5						0.08249	
6						0.07384	
7							

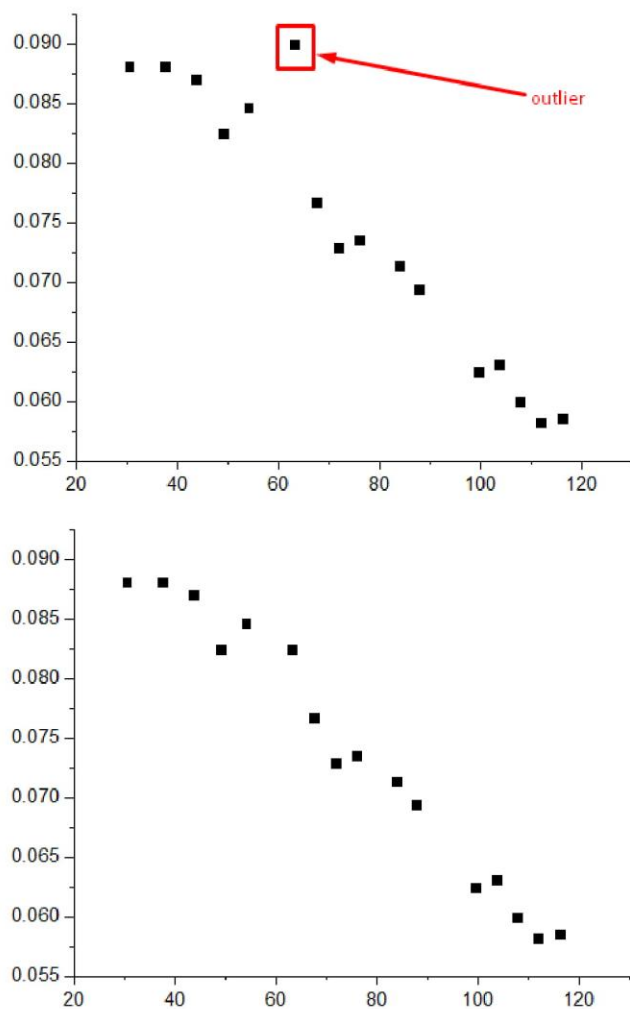
35. Right click column E > "Set As" > "X"

36. Select columns E and F

37. Create a scatter graph

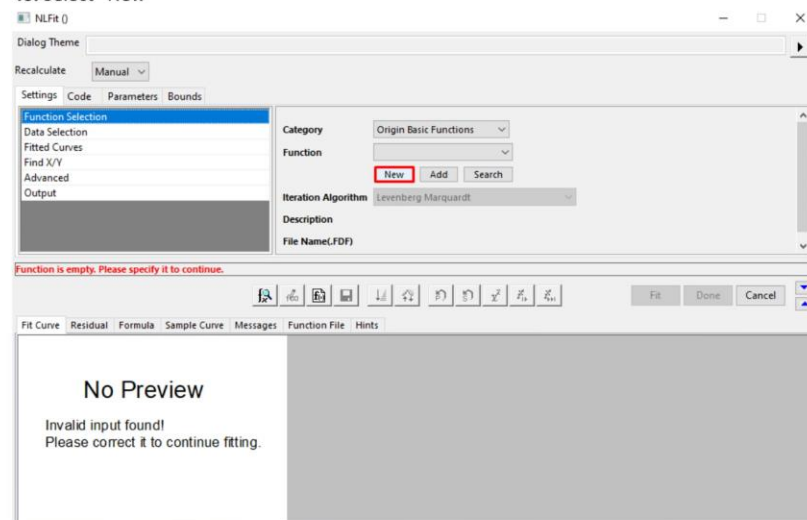
38. Rename this graph "LocationFit" (Ensure this scatter graph has no obvious outliers before moving on)

	A(Y)	B(Y)	C(Y)	D(Y)	E(X)	F(Y)
Long Name	Peak Number	Observed Value	Expected Value	Difference	Expected Value	Difference
Units				col(B)-col(C)		
Comments						
F(x)=						
1	1	21.43722	21.3578	0.07942	21.3578	0.07942
2	2	30.46299	30.3847	0.07829	30.3847	0.07829
3	3	37.51926	37.4417	0.07756	37.4417	0.07756
4	4	43.58176	43.5064	0.07536	43.5064	0.07536
5	5	49.03509	48.9573	0.07779	48.9573	0.07779
6	6	54.06349	53.9886	0.07489	53.9886	0.07489
7	7	63.29068	63.2182	0.07248	63.2182	0.07248
8	8	67.61927	67.5474	0.07187	67.5474	0.07187
9	9	71.81194	71.7452	0.06674	71.7452	0.06674
10	10	75.91139	75.8438	0.06759	75.8438	0.06759
11	omit		79.8695	—	83.8452	0.06235
12	12	83.90755	83.8452	0.06235	87.7913	0.06294
13	13	87.85424	87.7913	0.06294	99.6418	0.05956
14	omit		95.6707	—	103.6602	0.05653
15	15	99.70136	99.6418	0.05956	107.7485	0.05326
16	16	103.71673	103.6602	0.05653	111.9325	0.0509
17	17	107.80176	107.7485	0.05326	116.2437	0.05127
18	18	111.9834	111.9325	0.0509		
19	19	116.29497	116.2437	0.05127		
20						

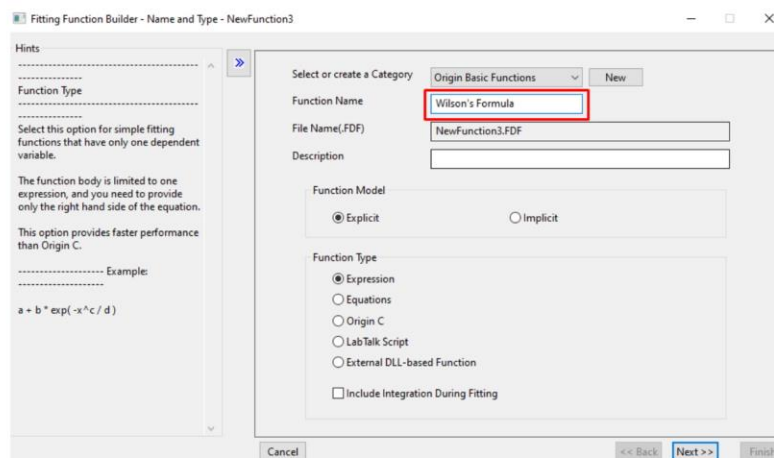


39. Within the upper menu bar, select "Analysis" > "Fitting" > "Nonlinear Curve Fit" > "Open Dialog..."

40. Select "New"



41. Within the "Function Name", type "Wilson's Formula", then select "Next >"



42. Enter the following parameters:
- a. Independent Variables: x
 - b. Dependent Variables: y
 - c. Parameters: a,b,c,d,e

Independent Variables: x

Dependent Variables: y

Parameters: a,b,c,d,e

Derived Parameters:

Constants:

☐ Peak Function

Cancel << Back Next >> Finish

43. Select "Next >"

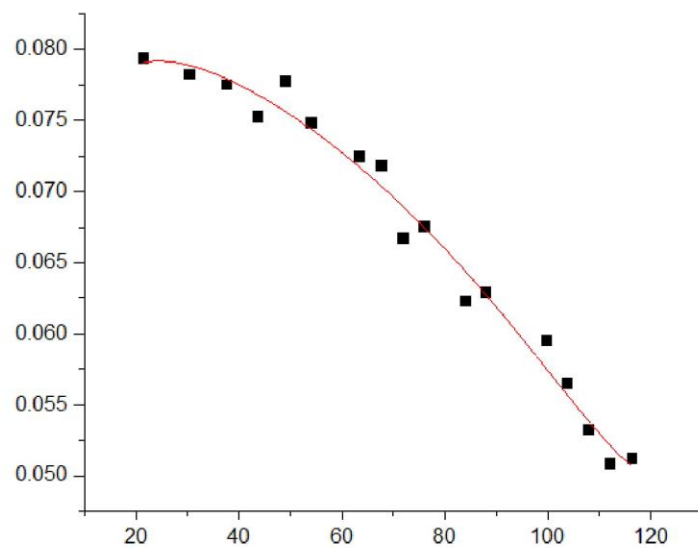
44. Within the "Function Body" box, type:

$$a/\tan(x/2)+b+c*\tan(x/2)+d*\tan(x/2)^2+e*\tan(x/2)^3$$

45. Select "Finish"

46. Select "Fit"

The result should be something like this:

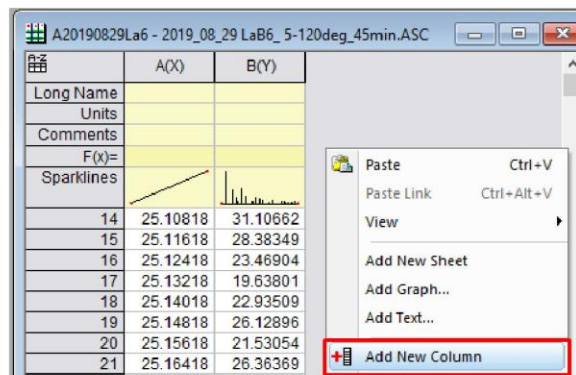


47. Create a new workbook
48. Rename this workbook "WilsonsParams"
49. Transfer the "a,b,c,d,e" parameters to this new workbook

	A(X)	B(Y)
Long Name		
Units		
Comments		
F(x)=		
1 a	-0.00117	
2 b	0.08953	
3 c	-0.02055	
4 d	-0.01254	
5 e	0.00658	
6		
7		
8		
9		
10		
11		

Update Table	
A	B1
1 Model	WilsonsFormula (User)
2 Equation	$a/\tan(x/2)+b+c*\tan(x/2)+d*\tan(x/2)^2+e*\tan(x/2)^3$
3 Plot	F
4 a	-0.00117 ± 0.00191
5 b	0.08953 ± 0.01503
6 c	-0.02055 ± 0.03693
7 d	-0.01254 ± 0.03474
8 e	0.00658 ± 0.01089
9 Reduced Chi-Sqr	2.23258E-6
10 R-Square (COD)	0.9832
11 Adj. R-Square	0.9776

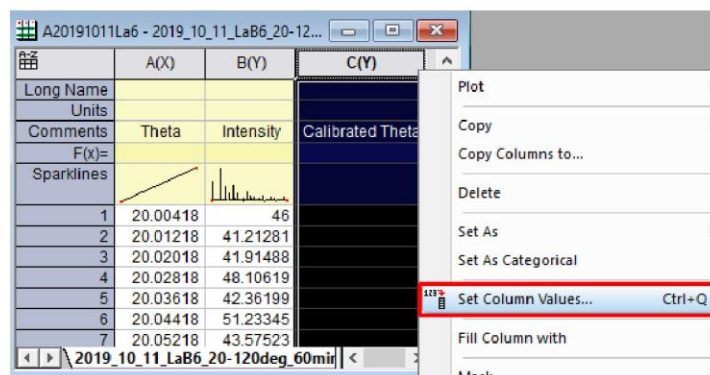
50. Return to the original calibration file and add a new column



51. In the comments row:

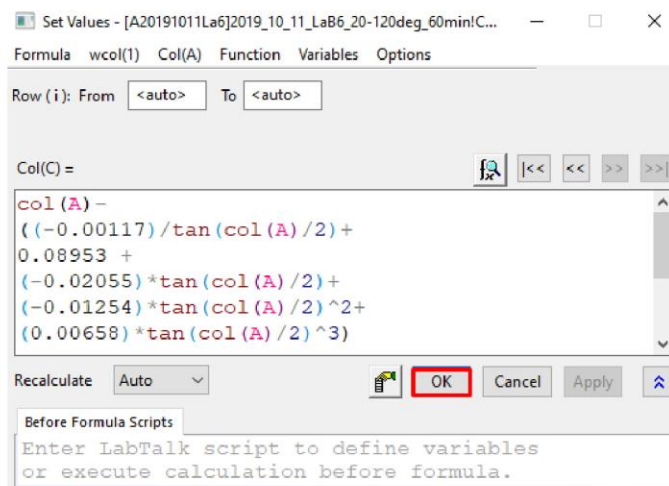
- Name column A: "Theta"
- Name column B: "Intensity"
- Name column C: "Calibrated Theta"

52. Right click column C > select "Set Column Values..."



53. Input the following formula (use the values from "WilsonsParams" for the a,b,c,d,e parameters):

```
col(A)-
(("a" parameter)/tan(col(A)/2)+
"b" parameter +
("c" parameter)*tan(col(A)/2)+
("d" parameter)*tan(col(A)/2)^2+
("e" parameter)*tan(col(A)/2)^3)
```



54. Select "OK"

55. Right click column A > "Set As ..." > "Y"

56. Right click column C > "Set As ..." > "X"

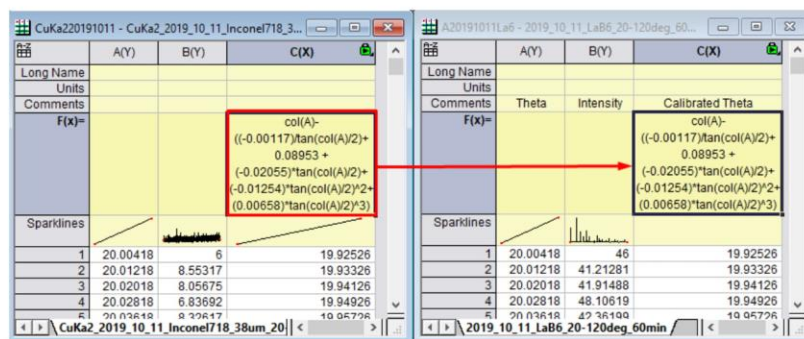
57. Now select columns B and C

58. Create a line graph

59. Rename this graph "LaB6Calibrated" (this is now a peak-position calibrated graph)

60. Repeat the following process for all datasets:

- Open the dataset workbook
- Create a new column and rename it "Calibrated Theta"
- Copy the "F(x)=" formula from the calibrated dataset
- Paste the formula into the "Calibrated Theta" column of the dataset
- Right click column A > "Set as" > "Y"
- Right click column C > "Set as" > "X"

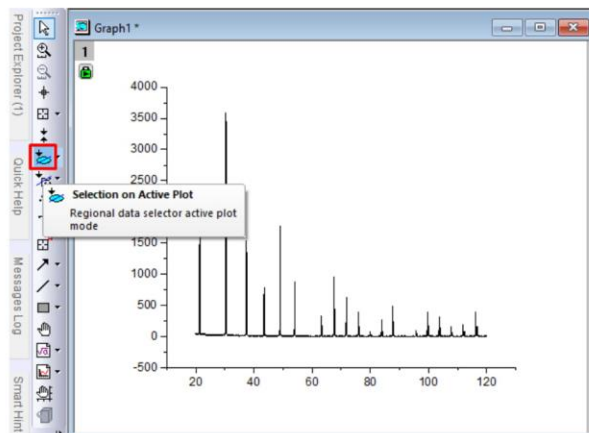


PEAK BREADTH CALIBRATION

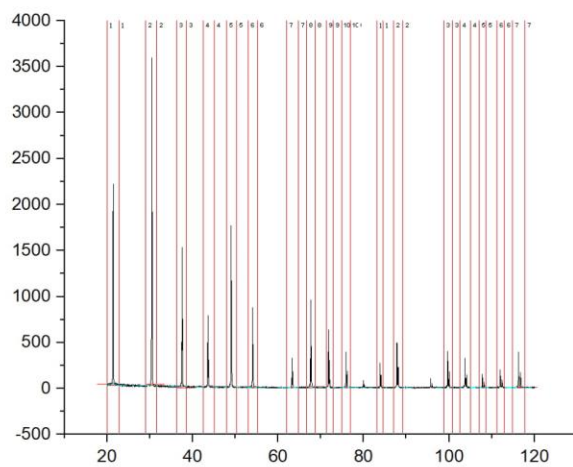
1. Create a new workbook
2. Name the new workbook: "PeakBreadth"
3. Create two new columns
4. In the "Comments" row:
 - a. Name column A: "Peak number"
 - b. Name column B: "Expected value"
 - c. Name column C: "FWHM"
 - d. Name column D: "FWHM^2"
5. In the "F(x)=" row of column D, insert " col(C)^2 "
6. Copy and paste columns A and B from the "PeakPosition" workbook
7. Remove the rows of the peaks which have been omitted

	A(X)	B(Y)	C(Y)	D(Y)
Long Name				
Units				
Comments	Peak Number	Observed Value	FWHM	FWHM^2
F(x)=				col(C)^2
1	1	21.43754	--	--
2	2	30.46491	--	--
3	3	37.51976	--	--
4	4	43.58447	--	--
5	5	49.03459	--	--
6	6	54.06477	--	--
7	7	63.29209	--	--
8	8	67.61889	--	--
9	9	71.81361	--	--
10	10	75.91125	--	--
11	12	83.90708		
12	13	87.85278		
13	15	99.70167		
14	16	103.71641		
15	17	107.80223		
16	18	111.98375		
17	19	116.29505		

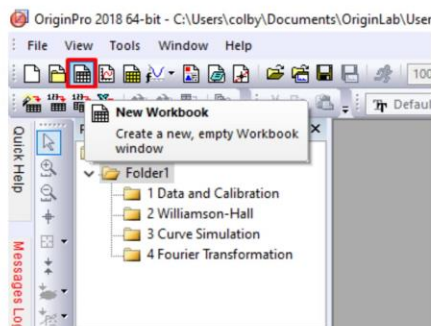
8. Open the "LaB6Calibrated" graph
9. Select the "Selection on Active Plot" icon



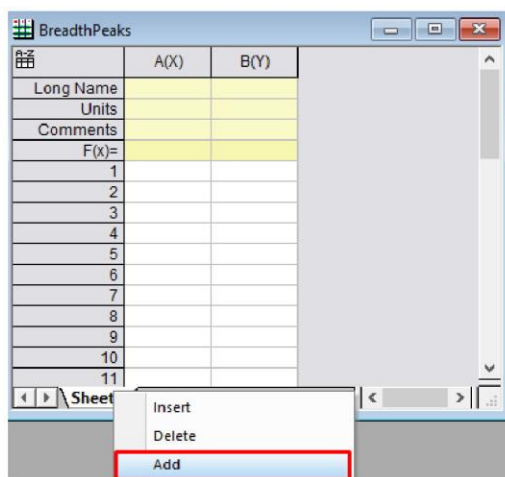
10. Draw a rectangle around every peak that will be used



11. Create a new workbook
12. Name the new workbook: "BreadthPeaks"

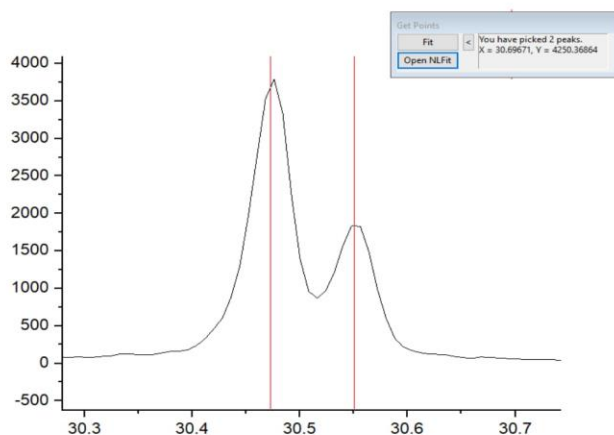


13. Right click the "Sheet1" icon and select "Add"
14. Repeat this process until the number of sheets is equal to the number of peaks that will be analyzed ($\#total\ peaks - \#omitted\ peaks$, 17 total sheets in this example)



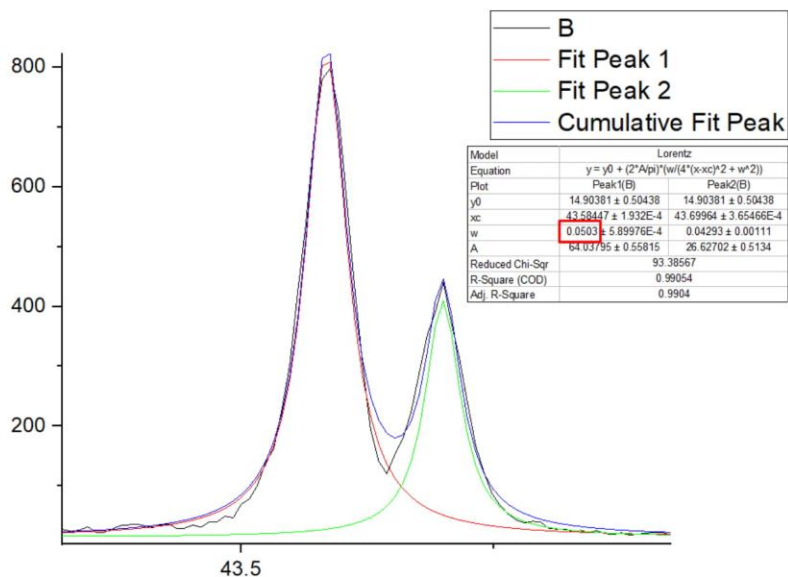
15. Return to the graph and select the first peak by left clicking between the vertical red lines (arrows should appear on the top and bottom of the lines if done correctly)
16. Copy this data selection

17. Return to the "PeakSelection" workbook and paste into "Sheet1"
18. Select both columns (CTRL+A on Windows)
19. Create a line graph
20. Repeat this process for each peak
21. For each graph that has been created: open the graph, within the upper menu bar, select "Analysis" > "Peaks and Baseline" > "Multiple Peak Fit" > "Open Dialog"
22. Within the "Peak Function" drop-down menu, select "Lorentz" then "OK"
23. Click and drag to align the cursor with the left peak center, then double click
24. Click and drag to align the cursor with the right peak center, then double click (*it is very important that this fit is accurate, so using the zoom in tool is helpful*)



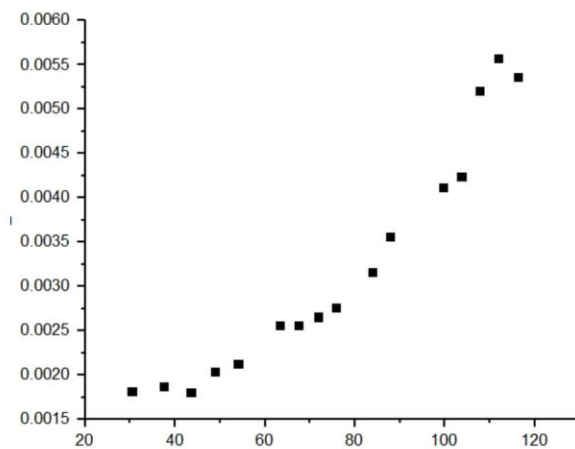
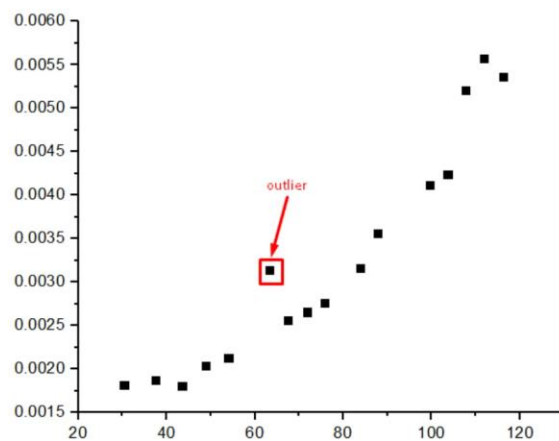
25. When finished, select "Fit"

26. Find the “w” value of the leftmost peak, “Fit Peak 1” and record this value in column C (FWHM) of the “PeakBreadth” workbook under the row corresponding to the peak number. Omit the +/- value (Double clicking on this value opens a pop-up that is much larger and easier to read)

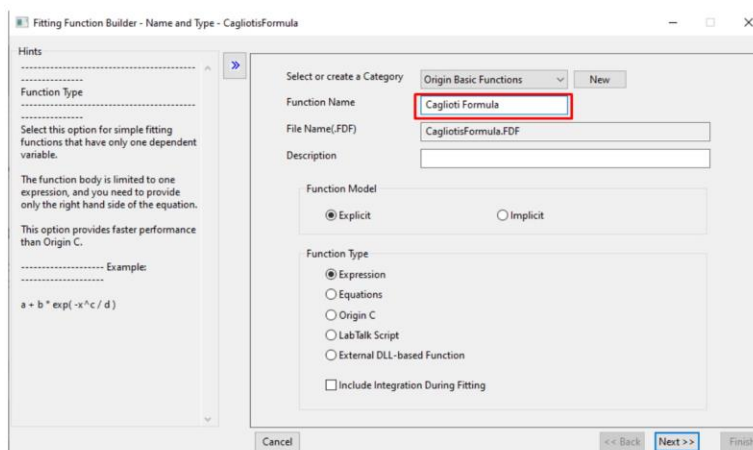


Note: for both peak position and peak breadth calibrations, it is important that the Cu-Kα1 radiation (left peak) and Cu-Kα2 radiation (right peak) are properly isolated. The relative intensity of Cu-Kα1 to Cu-Kα2 is roughly 2:1. As a rule of thumb for this analysis, I threw out any data with a relative intensity greater than 2.5:1. At lower angles, the peaks are stacked onto of each other and the curve-fitting software may not be able to properly separate them. In my experience, this typically results in an overestimation of the Cu-Kα1 peak and an underestimation of the Cu-Kα2.

27. Repeat this process for all peaks to fill the "PeakBreadth" workbook
28. Right click column A > "Set As" > "Y"
29. Right click column B > "Set As" > "X"
30. Select columns B and D
31. Create a scatter graph
32. Rename this graph "BreadthFit" (*Ensure this scatter graph has no obvious outliers before moving on*)



33. Within the upper menu bar, select "Analysis" > "Fitting" > "Nonlinear Curve Fit" > "Open Dialog..."
34. Select "New"
35. Within the "Function Name", type "Caglioti Formula", then select "Next >"

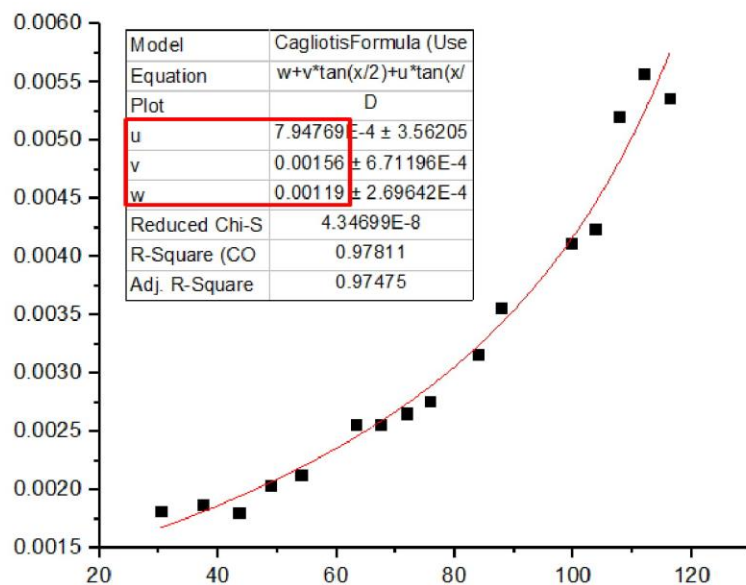


36. Enter the following parameters:
 - a. Independent Variables: x
 - b. Dependent Variables y
 - c. Parameters u, v, w
37. Select "Next >"
38. Within the function body enter:

$$w+v*\tan(x/2)+u*\tan(x/2)^2$$

39. Select "Finish"
40. Select "Fit"

The result should be similar to the graph below:



41. Create a new workbook
42. Name this workbook: "CagliotiParam"
43. Transfer the "u,v,w" parameters to this new workbook

The screenshot shows a software window titled "CagliotiParam *". It contains two main tables. The left table is a parameter list with columns for a row number, a parameter name, and its value. The right table is a model summary with columns for a property and its value.

	A(X)	B(Y)
Long Name		
Units		
Comments		
F(x)=		
1 u	7.94769E-4	
2 v	0.00156	
3 w	0.00119	
4		
5		
6		

Model	CagliotiFormula (User)
Equation	$w+v*\tan(x/2)+u*\tan(x/2)^2$
Plot	D
u	$7.94769E-4 \pm 3.56205E-4$
v	$0.00156 \pm 6.71196E-4$
w	$0.00119 \pm 2.69642E-4$
Reduced Chi-Sqr	4.34699E-8
R-Square (COD)	0.97811
Adj. R-Square	0.97475

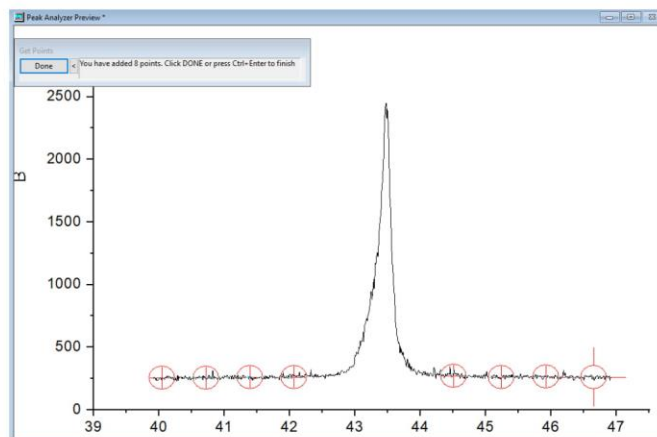
44. Return to the original calibration file and create a new column
45. In the "Comments" row, name this column "Instrumental FWHM"
46. Right click on Column D > "Set column values..."

$((\text{"u parameter"}) * \tan(\text{col(C)}/2) * \tan(\text{col(C)}/2) +$
 $(\text{"v parameter"}) * \tan(\text{col(C)}/2)$
 $+ (\text{"w parameter"})^{\wedge}.5$

(This column is the instrumental peak breadth correction factor as a function of theta)

NOISE BASELINE CALIBRATION

1. Open the angle-calibrated workbook for a single peak
2. Select column C, "Calibrated Theta", and column B, "Intensity"
3. Create a line graph
4. Within the upper menu bar, select "Analysis" > "Peaks and Baseline" > "Peak Analyzer" > "Open Dialog..."
5. Under the Goal section, select "Subtract Baseline"
6. Select "Next"
7. Change the "Baseline Mode" to "User Defined"
8. Disable "Enable Auto Find" by clicking the checkbox
9. Select "Clear All"
10. Select "Add"
11. Select 8 points on the baseline, do not select points close to the peak



12. Select "Done"
13. Select "Next"
14. Under Interpolation Method, click "BSpline"
15. Select "Next"

16. Under "Subtracted Data" select "<new>: New XY"

Subtract

Subtracted Data (<new>, <new>)

Baseline ☒

<input>: XY = input XY
<new>: New XY
<new>: !<new>, <new>

17. Uncheck the box next to "Baseline"

18. Select "Finish"

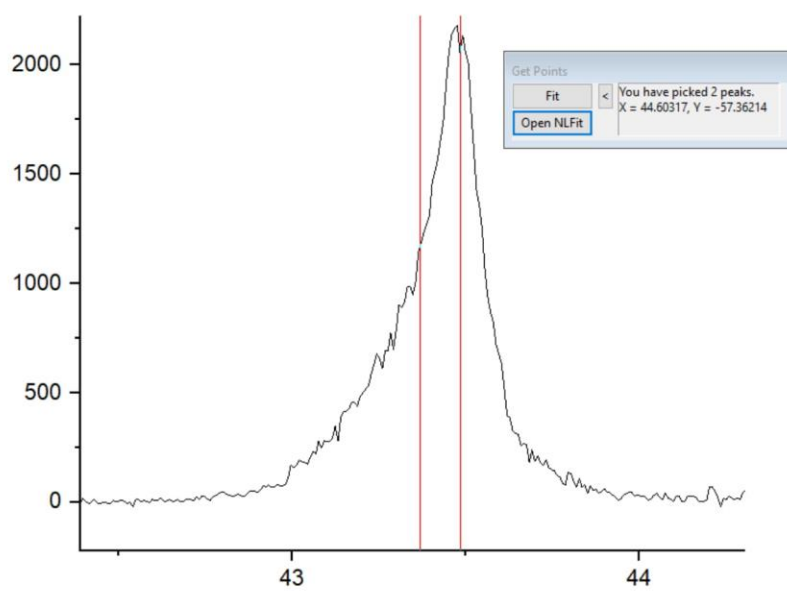
19. Repeat this process for all peaks

PEAK CURVE FITTING

1. Navigate to the "2 Williamson-Hall" folder
2. Create a new workbook
3. Rename the notebook: "CurveFits"
4. Create 5 new columns
5. In the "comments" row:
 - a. Name column A: "Peak Number"
 - b. Name column B: "Peak Height"
 - c. Name column C: "Peak Center"
 - d. Name column D: "Peak Width"
 - e. Name column E: "Peak Amplitude"
 - f. Name column F: "Instrumental Width"
 - g. Name column G "Corrected Width"
6. Under the "F(x)=" row for Column G, insert " $(\text{col(D)}^2 - \text{col(F)}^2) / \text{col(D)}$ "
7. Fill in column A with the number of peaks in the dataset (*In this example data set there are five peaks, so column A is "1,2,3,4,5"*)
8. Open the dataset workbook for a single peak from the "1 Data and Calibration" folder
9. Select the baseline-subtracted data (columns D and E)

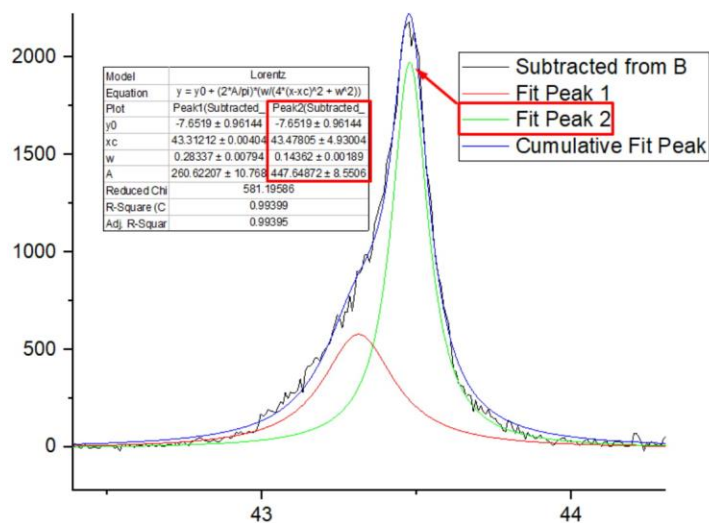
	A(Y)	B(Y)	C(X1)	D(X2)	E(Y2)
Long Name				Subtracted	Subtracted
Units					
Comments	Theta	Intensity	Calibrated Theta		Subtracted from B
F(x)=					
Sparklines					
1	70.00418	1570	69.92761	69.92761	0.48934
2	70.01218	1561.70221	69.93561	69.93561	-8.03687

10. Create a line graph
11. Select "Analysis" > "Peaks and Baseline" > "Multiple Peak Fit" > "Open Dialog..."
12. Under the drop-down menu "Peak Function", select "Lorentz" > "OK"
13. If there is asymmetry in the peaks, select two data points as shown below:



14. Select "Fit"

15. The major peak should be utilized from this curve fitting (in this case, "Fit Peak 2" is the major peak)
16. Copy the "y0", "xc", "w", and "A" values into the "CurveFits" workbook into columns B, C, D, and E respectively (double clicking these values pops out another window which can be read in both folders)



17. Repeat this process for all peaks

An example workbook is shown below:

CurveFits *							
	A(X)	B(Y)	C(Y)	D(Y)	E(Y)	F(Y)	G(Y)
Long Name							
Units							
Comments	Peak Number	Peak Height	Peak Center	Peak Width	Peak Amplitude	Instrumental Width	Corrected Width
F(x)=							(col(D))^2-col(F)^2)/col(D)
1	1	-7.65591	43.49092	0.13585	199.8089		--
2	2	1.5702	50.6435	0.24546	382.20539		--
3	3	-6.24032	74.51602	0.29187	406.53775		--
4	4	-7.23658	90.43471	0.53379	317.85103		--
5	5	2.2001	95.77343	0.48499	109.90497		--
6							

-
- The screenshot shows the Origin 8.5 software interface. The main data table has columns labeled F(Y), G(Y), A(Y), B(Y), C(X), and D(Y). The 'Instrumental Width' column is highlighted with a red box. The 'D(Y)' column contains a formula: $769E-4) \cdot \tan(\text{col}(C)/2) \cdot \tan(\text{col}(C)/2) \cdot (0.00156) \cdot \tan(\text{col}(C)/2) + (0.00119))^5$. A red arrow points from the 'Instrumental Width' column to the 'D(Y)' column.

WILLIAMSON-HALL ANALYSIS

1. Create a new workbook
2. Rename the workbook: "WH Analysis"
3. Add 16 new columns
4. In the "Comments" row,
 - a. Name column A: "Peak Number"
 - b. Name column B: "Peak Position"
 - c. Name column C: "K"
 - d. Name column D: "FWHM"
 - e. Name column E: " ΔK "
 - f. Name column F: "h"
 - g. Name column G: "l"
 - h. Name column H: "k"
 - i. Name column I: " H^2 "
 - j. Name column J: " $(\Delta K^2 - \alpha) / K^2$ "
 - k. Name column K: "q"
 - l. Name column L: " $C(H00)$ "
 - m. Name column M: "C"
 - n. Name column N: " $KC^{1/2}$ "
 - o. Name column O: " ΔK "
 - p. Name column P: " λ "
 - q. Name column Q: " α "
 - r. Name column R: "D"

5. In the formula, "F(x)=" row,
 - a. In column C, insert: " $2 \cdot \sin(\text{col(B)}/2)/\text{col(P)}$ "
 - b. In column E, insert: " $2 \cdot \cos(\text{col(B)}/2) \cdot \text{radians}(\text{col(D)}/2)/\text{col(P)}$ "
 - c. In column I, insert:

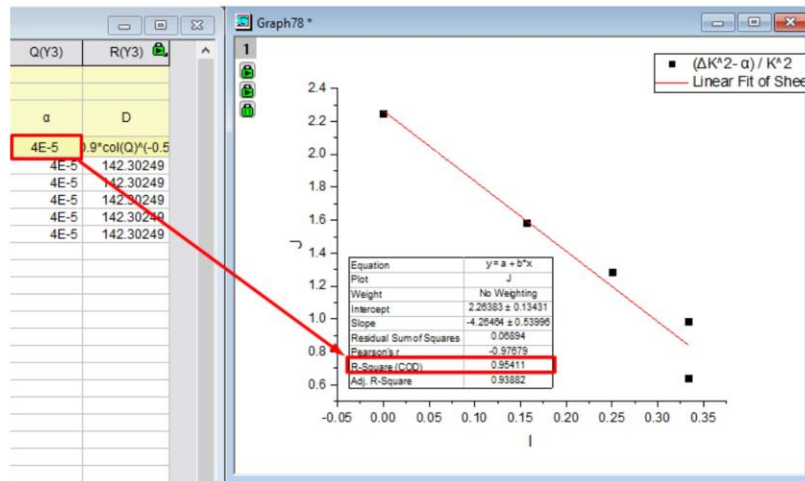
$$\frac{(\text{col(F)}^2 \cdot \text{col(G)}^2 + \text{col(F)}^2 \cdot \text{col(H)}^2 + \text{col(G)}^2 \cdot \text{col(H)}^2)}{(\text{col(F)}^2 + \text{col(G)}^2 + \text{col(H)}^2)^2}$$
 - d. In column J, insert: " $100000 \cdot ((\text{col(E)}^2 - \text{col(Q)})/\text{col(C)}^2)$ "
 - e. In column L, insert C_{H00} , a constant related to the material's elastic constraints
(0.265 for Inconel718 in this example)
 - f. In column M, insert: " $\text{col(L)} \cdot (1 - \text{col(K)} \cdot \text{col(I)})$ "
 - g. In column N, insert: " $\text{col(C)} \cdot \text{col(M)}^{0.5}$ "
 - h. In column O, insert: " $2 \cdot \cos(\text{col(B)}/2) \cdot \text{radians}(\text{col(D)}/2)/\text{col(P)}$ "
 - i. In column P, insert: λ which is the wavelength of diffraction x-ray in nm *(0.15406 for CuKa2 in this example)*
 - j. In column Q, insert: " $4E-5$ "
 - k. In column R, insert: " $0.9 \cdot \text{col(Q)}^{(-0.5)}$ "
6. In row 1,
 - a. In column A, insert: "1"
 - b. In column B, insert: the "Peak Center" value of the first peak in "CurveFits"
 - c. In column D, insert: the "Corrected Width" value of the first peak in "CurveFits"
 - d. In column F, insert: the "h" Miller index of refraction for the first peak
 - e. In column G, insert: the "k" Miller index of refraction for the first peak
 - f. In column H, insert: the "l" Miller index of refraction for the first peak
7. In row 2,
 - a. In column A, insert: "2"
 - b. In column B, insert: the "Peak Center" value of the second peak in "CurveFits"
 - c. In column D, insert: the "Corrected Width" value of the second peak in "CurveFits"
 - d. In column F, insert: the "h" Miller index of refraction for the second peak
 - e. In column G, insert: the "k" Miller index of refraction for the second peak
 - f. In column H, insert: the "l" Miller index of refraction for the second peak
8. Continue to fill in every row for each peak that is being analyzed
9. Right click column A > "Set As" > Y
10. Right click column C > "Set As" > X
11. Right click column I > "Set As" > X
12. Right click column N > "Set As" > X

13. The completed workbook should look like this: (split in two pictures to fit on page)

WHAnalysis											
	A(Y)	B(Y)	C(X1)	D(Y1)	E(Y1)	F(Y1)	G(Y1)	H(Y1)	I(X2)	J(Y2)	
Long Name											
Units											
Comments	Peak Number	Peak Position	K	FWHM	ΔK	h	k	l	H ²	$(\Delta K^2 - a) / K^2$	
F(x)=			$\sqrt{\text{col(B)/2}}/c$		$\gamma^{\circ} \text{radians}(\text{col(B)/2})$				$\text{col(G)}^2 \cdot c^2 \cdot \text{col(I)}^2 \cdot c^2 \cdot \text{col(J)}^2$	$(\text{col(E)}^2 - c^2 \cdot \text{col(J)}) / (c^2)$	
1	1	43.41212	4.80131	0.13012	0.0137	1	1	1	0.33333	0.64022	
2	2	50.63537	5.55156	0.26432	0.02707	2	0	0	0	2.24756	
3	3	74.48148	7.85623	0.32028	0.02889	2	2	0	0.25	1.28708	
4	4	90.4047	9.21199	0.4059	0.03719	3	1	1	0.15702	1.58267	
5	5	95.71995	9.62621	0.40563	0.03083	2	2	2	0.33333	0.9827	

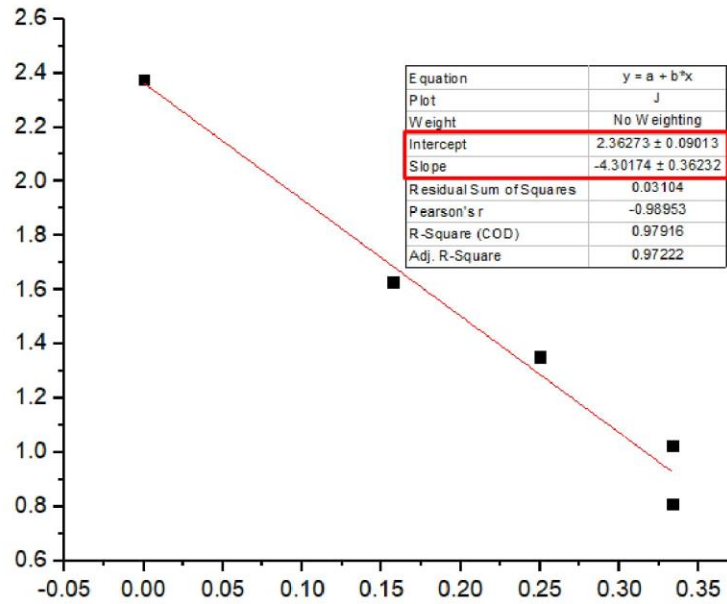
K(Y2)	L(Y2)	M(Y2)	N(X3)	O(Y3)	P(Y3)	Q(Y3)	R(Y3)
q	C(H00)	C	KC^1/2	ΔK	λ	α	D
	0.265	$y*(1-\text{col}(K)*\text{col}(C))*\text{col}(M)^2*\text{radians}(c$		0.15406		4E-5	$0.9*\text{col}(Q)^*(-0.5$
--	0.265	--	--	0.0137	0.15406	4E-5	142.30249
--	0.265	--	--	0.02707	0.15406	4E-5	142.30249
--	0.265	--	--	0.02889	0.15406	4E-5	142.30249
	0.265	--	--	0.03719	0.15406	4E-5	142.30249
	0.265	--	--	0.03083	0.15406	4E-5	142.30249

14. Select columns I and J
15. Create a scatter graph
16. Within the upper menu bar, select "Analysis" > "Fitting" > "Linear Fit" > "Open Dialog" > "OK"



17. Iteratively adjust the alpha value until the R-Square value of the fit is maximized.

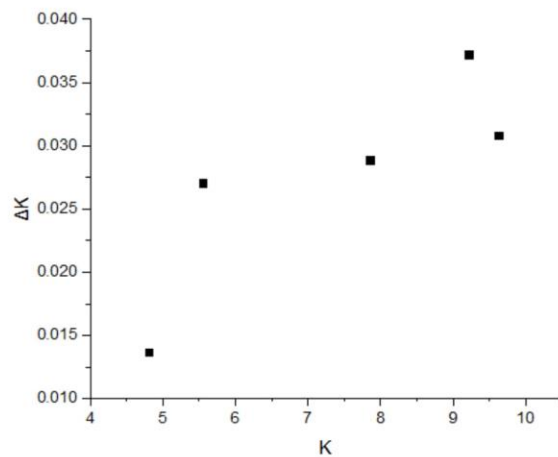
18. Once the trendline has been optimized to minimize the R-Square (COD) value, divide the slope by the **opposite** of the intercept to find the dislocation character, q
 (for this example: $q = -b/a = -4.30174 / -2.36273 = 1.82066454$)



19. Insert this value into the "F(x)=" row of column K of the "WHAnalysis" workbook

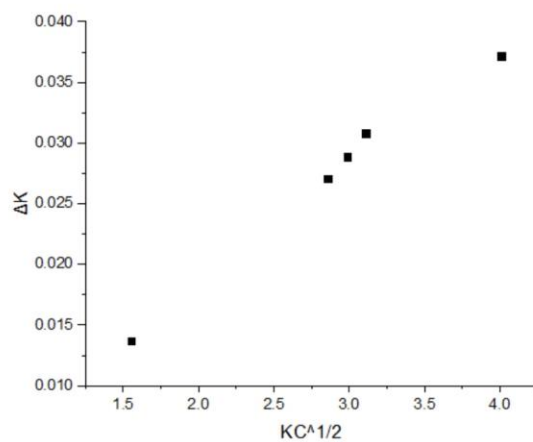
20. Select columns C and E in the "WHAnalysis" workbook

21. Create a scatter graph (*this is the classical Williamson-Hall graph*)



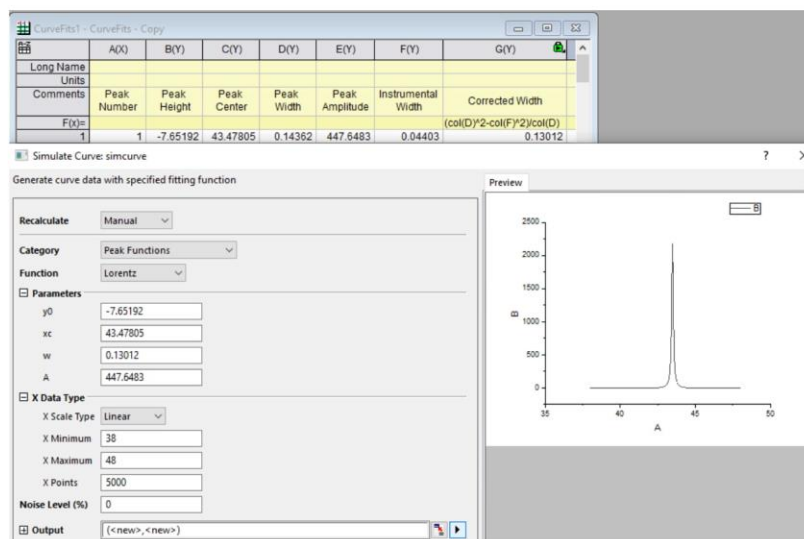
22. Select columns N and O in the "WHAnalysis" workbook

23. Create a scatter graph (*this is the modified Williamson-Hall graph*)



CURVE SIMULATION

1. Right click the "CurveFits" workbook > "Duplicate"
2. Drag and drop the duplicated workbook into the "3 Curve Simulation" folder
3. Navigate to the "3 Curve Simulation" folder
4. Open the "CurveFits - Copy" workbook
5. Within the upper menu bar, select "Analysis" > "Fitting" > "Simulate Curve..."
6. Under the "Recalculate" dropdown menu, select "Manual"
7. Under the "Category" dropdown menu select "Peak Functions"
8. Under the "Function" dropdown menu select "Lorentz"
9. Under the "Parameters" section, add the parameters from the "CurveFits" workbook:
 - a. In the "y0" section, add the "Peak Height" value for the first peak
 - b. In the "xc" section, add the "Peak Center" value for the first peak
 - c. In the "w" section, add the "Corrected Width" value for the first peak
 - d. In the "A" section, add the "Peak Amplitude" value for the first peak
10. Under the "X Data Type" section, add the following parameters:
 - a. In the "X Minimum" section, round the "Peak Center Value" and subtract 5
 - b. In the "X Maximum" section, round the "Peak Center Value" and add 5
 - c. In the "X Points" section, change the value to "5000"

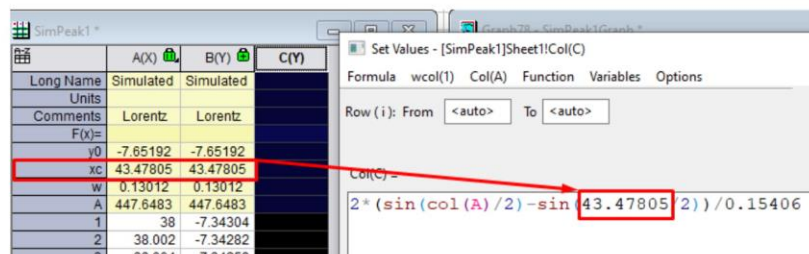


11. Select "OK" (this will create a new workbook and a new graph)

12. Rename the workbook to "SimPeak1"
13. Open "SimPeak1" workbook add another column
14. Right click this column and select "Set Column Values..."
15. Within the formula box, insert:

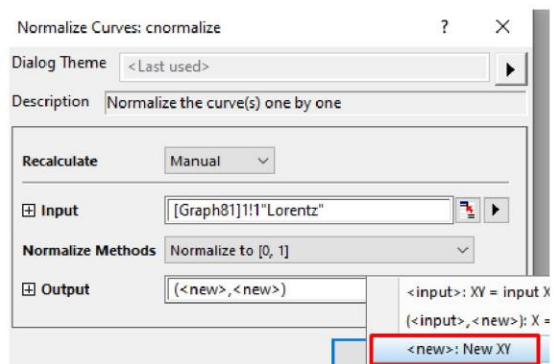
$$2 * (\sin(\text{col(A)}/2) - \sin(\text{"YOUR XC VALUE HERE"}/2)) / 0.15406$$

(where "YOUR XC VALUE HERE" is the corresponding peak center from column C of "CurveFits")



16. Select "OK"
17. Right click column A > Set As > "Y"
18. Right click column C > Set As > "X"
19. Select columns B and C
20. Create a line graph (this is the x-axis normalized, simulated peak)
21. Within the upper menu bar, select "Analysis" > "Mathematics" > "Normalize Curves" > "Open Dialog..."

22. Under Output, select "<new>: New XY"

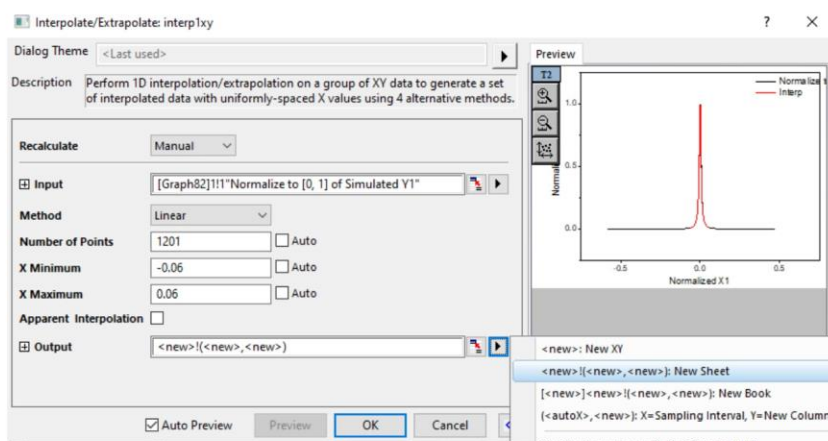


23. Select "OK"

24. Select the new columns D and E

25. Create a line graph

26. Within the upper menu bar, select "Analysis" > "Mathematics" > "Interpolate/Extrapolate" > "Open Dialog..."
27. Under "Number of Points" uncheck the "Auto" box and enter "1201"
28. Under "X Minimum" uncheck the "Auto" box and enter "-0.06"
29. Under "X Maximum" uncheck the "Auto" box and enter "0.06"
30. Under "Output", select "<new>!(<new>,<new>): New Sheet"



31. Select "OK"
32. Repeat this process for all peaks

33. Create a new workbook
34. Name the new workbook: "SimPeaks"
35. Continue to add columns until there is two columns for every peak (for example 5 peaks require 10 total columns in the workbook)
36. Right click every odd numbered column (1,3,5...) > "Set as" > X
37. In the "Comments" row, label each peak
38. The setup should look like this (example for 5 peaks):

The screenshot shows the 'SimPeaks' workbook with columns A(X1) through J(Y5). The 'Comments' row is labeled with 'Peak 1' through 'Peak 5'. The 'F(x)=', '1', '2', and '3' rows are empty.

	A(X1)	B(Y1)	C(X2)	D(Y2)	E(X3)	F(Y3)	G(X4)	H(Y4)	I(X5)	J(Y5)
Long Name										
Units										
Comments	Peak 1	Peak 1	Peak 2	Peak 2	Peak 3	Peak 3	Peak 4	Peak 4	Peak 5	Peak 5
F(x)=										
1										
2										
3										

39. For each peak, copy the columns in "Sheet2" of the "SimPeakx" workbook and paste them into the "SimPeaks" workbook

The screenshot shows two workbooks. The top workbook, 'SimPeak1', has columns A(X) and B(Y). The bottom workbook, 'SimPeaks', has columns A(X1) and B(Y1). Red arrows indicate data being copied from 'SimPeak1' to 'SimPeaks'.

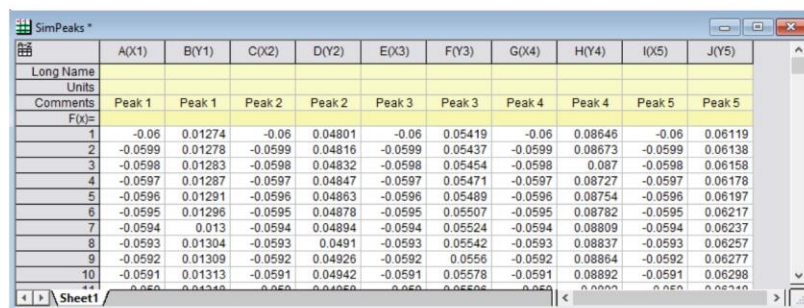
SimPeak1 (Sheet2) Data:

	A(X)	B(Y)
Long Name	Interpolate	Interpolate
Units		
Comments		Linear interp of Normalized Y1*
F(x)=		
1	-0.06	0.01274
2	-0.0599	0.01278
3	-0.0598	0.01283
4	-0.0597	0.01287
5	-0.0596	0.01291
6	-0.0595	0.01296
7	-0.0594	0.013

SimPeaks (Sheet1) Data:

	A(X1)	B(Y1)
Long Name		
Units		
Comments	Peak 1	Peak 1
F(x)=		
1		
2		
3		
4		
5		
6		

The result should look like this:

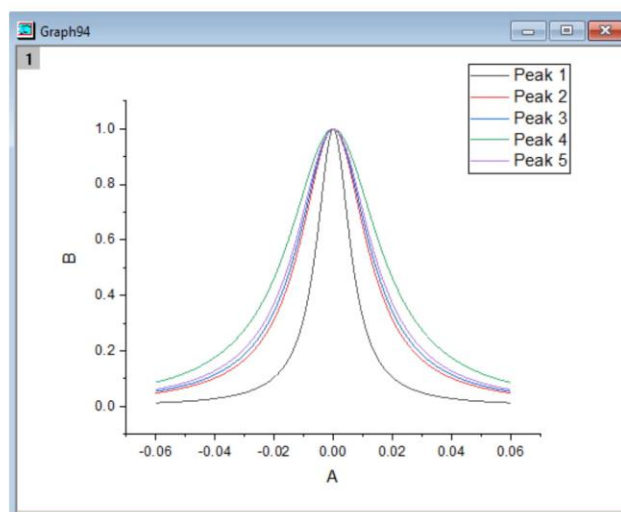


	A(X1)	B(Y1)	C(X2)	D(Y2)	E(X3)	F(Y3)	G(X4)	H(Y4)	I(X5)	J(Y5)
Long Name										
Units										
Comments	Peak 1	Peak 1	Peak 2	Peak 2	Peak 3	Peak 3	Peak 4	Peak 4	Peak 5	Peak 5
F(x)=										
1	-0.06	0.01274	-0.06	0.04801	-0.06	0.05419	-0.06	0.08646	-0.06	0.06119
2	-0.0599	0.01278	-0.0599	0.04816	-0.0599	0.05437	-0.0599	0.08673	-0.0599	0.06138
3	-0.0598	0.01283	-0.0598	0.04832	-0.0598	0.05454	-0.0598	0.087	-0.0598	0.06158
4	-0.0597	0.01287	-0.0597	0.04847	-0.0597	0.05471	-0.0597	0.08727	-0.0597	0.06178
5	-0.0596	0.01291	-0.0596	0.04863	-0.0596	0.05489	-0.0596	0.08754	-0.0596	0.06197
6	-0.0595	0.01296	-0.0595	0.04878	-0.0595	0.05507	-0.0595	0.08782	-0.0595	0.06217
7	-0.0594	0.013	-0.0594	0.04894	-0.0594	0.05524	-0.0594	0.08809	-0.0594	0.06237
8	-0.0593	0.01304	-0.0593	0.0491	-0.0593	0.05542	-0.0593	0.08837	-0.0593	0.06257
9	-0.0592	0.01309	-0.0592	0.04926	-0.0592	0.0556	-0.0592	0.08864	-0.0592	0.06277
10	-0.0591	0.01313	-0.0591	0.04942	-0.0591	0.05578	-0.0591	0.08892	-0.0591	0.06298

40. Select all columns (CTRL+A)

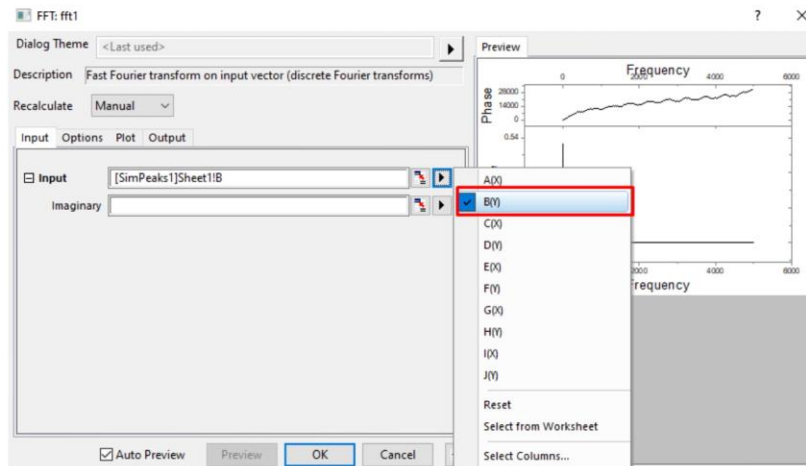
41. Create a line graph

The result should be similar to the graph below:

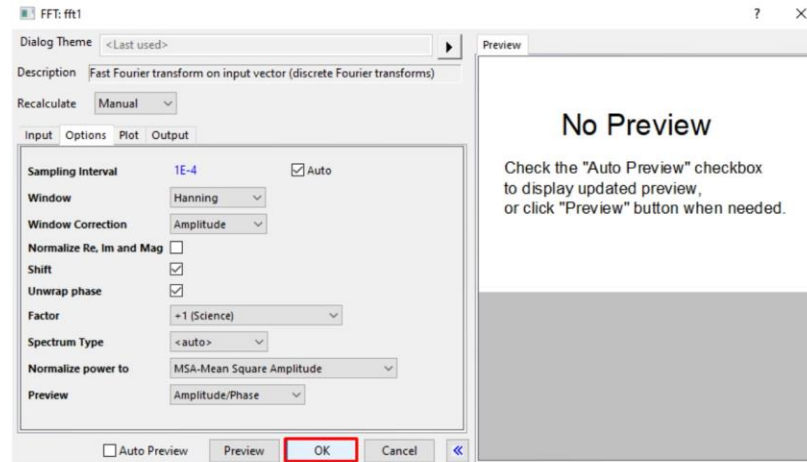


FOURIER TRANSFORMATON

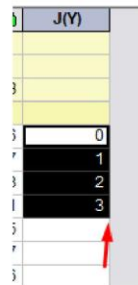
1. Right click the "SimPeaks" workbook > "Duplicate"
2. Drag and drop the duplicated workbook into the "4 Fourier Transformation" folder
3. Navigate to the "4 Fourier Transformation" folder
4. Open the "SimPeaks - Copy" workbook
5. Within the upper menu bar, select "Analysis" > "Signal Processing" > "FFT" > "FFT" > "Open Dialog..."
6. Under the "Input" tab menu, select column B (peak 1)



7. Under the "Options" tab menu,
 - a. Under "Window" select "Hanning"
 - b. Under "Factor" select "+1 (Science)"
8. Select "OK"



9. Add a new column to this sheet
10. In this new column, add the numbers 0, 1, 2, 3. Select these rows and autofill the rest of the column by double-clicking the box in the bottom right corner as shown below:

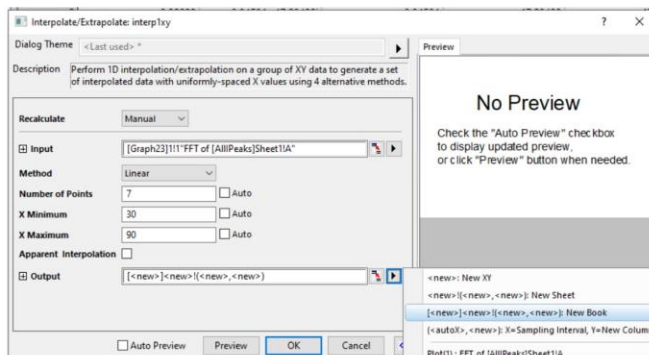


11. Add a new column to this sheet
12. In the "F(x)=" row of this new column, type: "col(A)*col(J)"

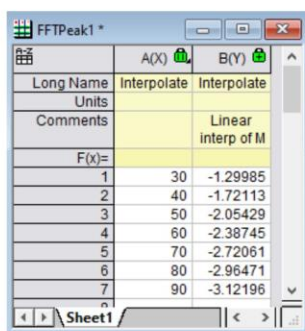
13. Right click Column E > Normalize > OK
14. Add a new column to this workbook
15. In the "F(x)=" row of this new column, type "ln(col(L))" as shown below:

J(Y)	K(Y)	L(Y)	M(Y)
		Normalized	
		Normalize t	
	col(A)*col(J)		ln(col(L))
0	0	1	0
1	8.32639	0.79909	-0.22428
2	33.30558	0.46135	-0.77361
3	74.93755	0.25249	-1.3764
4	#####	0.13958	-1.96911
5	#####	0.07674	-2.56728

16. Right click Column A > Set As > Y
17. Right click Column K > Set As > X
18. Select columns K and M
19. Create a line graph
20. Within the upper menu bar, select Analysis > Mathematics > Interpolate/Extrapolate > Open Dialog...
 - a. Under "Number of Points" unclick "Auto" and input "7"
 - b. Under "X Minimum" unclick "auto" and input "30"
 - c. Under "X Maximum" unclick "auto" and input "90"
 - d. Under "Output", select "New Book"



21. Select "OK" (this will create a new workbook)



	A(X)	B(Y)
Long Name	Interpolate	Interpolate
Units		
Comments		Linear interp of M
F(x)=		
1	30	-1.29985
2	40	-1.72113
3	50	-2.05429
4	60	-2.38745
5	70	-2.72061
6	80	-2.96471
7	90	-3.12196

22. Rename this new workbook "FFTPeak1"

23. Repeat this process for all peaks

WARREN-AVERBACH ANALYSIS

1. Create a new workbook
2. Name the new workbook: "WAnalysis"
3. Add 8 new columns
4. Right click Column A > Set As > Y
5. Right click Column C > Set As > X
6. In the "F(x)=" row of Column C, insert: " $\text{col(A)} * \text{col(B)}^2$ "
7. Refer to the "WAnalysis" workbook in the "2 Williamson-Hall" folder to find and copy the "C" and "K" values for each peak
8. Paste "C" and "K" values for each peak into columns A and B
9. In the "Comments" row,
 - a. Name column A: "C"
 - b. Name column B: "K"
 - c. Name column C: " $C * K^2$ "
 - d. Name column D: "30"
 - e. Name column E: "40"
 - f. Name column F: "50"
 - g. Name column G: "60"
 - h. Name column H: "70"
 - i. Name column I: "80"
 - j. Name column J: "90"

The result should look like this:

	A(Y)	B(Y)	C(X)	D(Y)	E(Y)	F(Y)	G(Y)	H(Y)	I(Y)	J(Y)
Long Name										
Units										
Comments	C	K	$C * K^2$	30	40	50	60	70	80	90
F(x)=			$\text{col(A)} * \text{col(B)}^2$							
1	0.06609	4.80352	1.525							
2	0.265	5.54587	8.15052							
3	0.11582	7.85015	7.13734							
4	0.1713	9.20584	14.51721							
5	0.06609	9.62266	6.11986							

10. Copy the data of the "FFTPeak" workbooks from the last section to the "WAAAnalysis" workbook

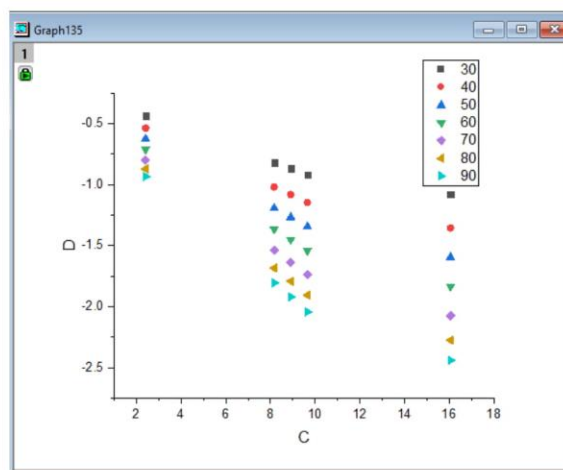
The result should look like this:

	A(Y)	B(Y)	C(X)	D(Y)	E(Y)	F(Y)	G(Y)	H(Y)	I(Y)	J(Y)
Long Name										
Units										
Comments	C	K	C^*K^2	30	40	50	60	70	80	90
F(x)=			$ol(A)*col(B)^x$							
1	0.10417	4.80825	2.40844	-0.43594	-0.53835	-0.62502	-0.7117	-0.79837	-0.8722	-0.9335
2	0.265	5.55156	8.16726	-0.8188	-1.01992	-1.19243	-1.36494	-1.53745	-1.68367	-1.80425
3	0.14438	7.85623	8.91124	-0.86722	-1.08163	-1.26596	-1.45028	-1.6346	-1.79067	-1.91921
4	0.18924	9.21199	16.05899	-1.08019	-1.35531	-1.59431	-1.83331	-2.0723	-2.27347	-2.43774
5	0.10417	9.62621	9.65324	-0.91816	-1.14679	-1.34381	-1.54084	-1.73786	-1.9045	-2.04151

11. Select columns C, D, E, F, G, H

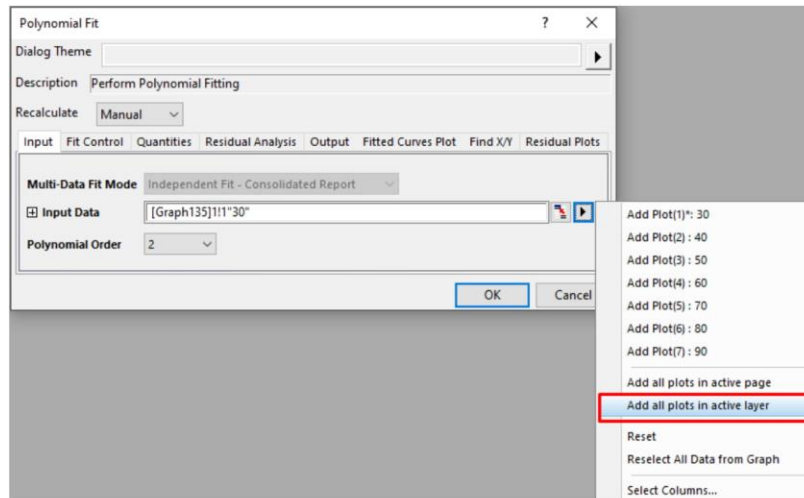
12. Create a scatter graph

The result should look like this:



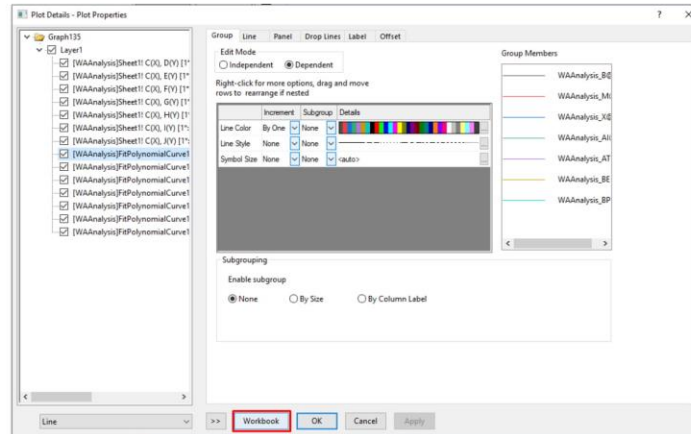
13. Within the upper menu bar, select "Analysis" > "Fitting" > "Polynomial Fit" > "Open Dialog..."

14. Under "Input Data", select "Add all plots in active layer"



15. Select "OK"

16. Double click any polynomial fit line on the graph
 17. Select "Workbook"



18. Select the "FitPolynomial1" tab

WAAAnalysis				
Long Name	A(X1)	B(Y1)	C(X2)	D(Y2)
Units	Independent Variable	Polynomial Fit of Sheet1 D	Independent Variable	Regular Residual of Sheet1 D
Comments	Fit Curve of D	Fit Curve of D	Fit Curve of D	Fit Curve of D
Parameters	Fitted Curves Plot			
F(x)=				
2	2.42211	-0.43554	8.16726	0.0123
3	2.43577	-0.43669	8.91124	0.00196
4	2.44943	-0.43784	16.05899	0.00131
5	2.4631	-0.43899	9.65324	-0.014
6	2.47676	-0.44015		
7	2.49043	-0.4413		
8	2.50409	-0.44244		
9	2.51776	-0.44359		
10	2.53142	-0.44474		
11	2.54508	-0.44588		
12	2.55875	-0.44703		
13	2.57241	-0.44817		
14	2.58608	-0.44932		
15	2.59974	-0.45046		
16	2.6134	-0.4516		
17	2.62707	-0.45274		

19. Scroll down to the "Summary" section
20. Copy the values in the "B1" column

WAAAnalysis

Standard Error was scaled with square root of reduced Chi-Sqr.

Statistics

	D	E	F	G	H	I	J
Number of Points	5	5	5	5	5	5	5
Degrees of Freedom	2	2	2	2	2	2	2
Residual Sum of Squares	3.55255E-4	6.03406E-4	8.88468E-4	0.00123	0.00162	0.00199	0.00238
R-Square (COD)	0.99843	0.99834	0.99826	0.9982	0.99816	0.99813	0.9981
Adj. R-Square	0.99687	0.99668	0.99652	0.9964	0.99631	0.99626	0.9962

Summary

	Intercept		B1		B2		Statistics
	Value	Standard Error	Value	Standard Error	Value	Standard Error	Adj. R-Square
D	-0.21493	0.02261	-0.09768	0.00511	0.00272	2.63973E-4	0.99687
E	-0.26095	0.02947	-0.12246	0.00666	0.00338	3.44029E-4	0.99668
F	-0.29882	0.03576	-0.14388	0.00808	0.00393	4.17456E-4	0.99652
G	-0.33668	0.04205	-0.16529	0.0095	0.00448	4.90884E-4	0.9964
H	-0.37454	0.04834	-0.18671	0.01092	0.00503	5.64311E-4	0.99631
I	-0.40707	0.05353	-0.20485	0.0121	0.00551	6.24809E-4	0.99626
J	-0.43438	0.05763	-0.21981	0.01302	0.00591	6.72696E-4	0.99624

ANOVA

	DF	Sum of Squares	Mean Square	F Value	Prob>F
FitPolynomial1	1	0.000355255	0.000355255	1.00000	0.33333
FitPolynomialCurve1	2	0.000603406	0.000301703	0.88889	0.44444

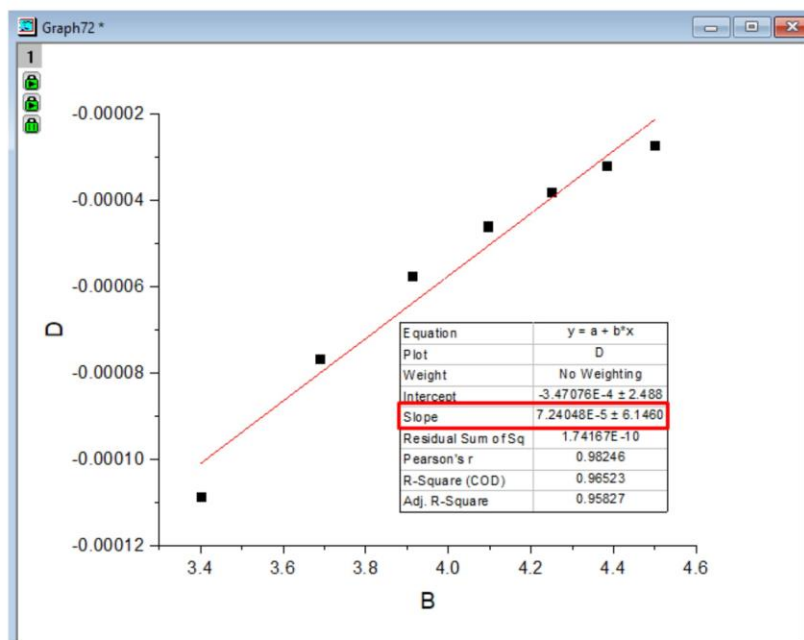
Sheet1 | FitPolynomial1 | FitPolynomialCurve1

21. Create a new workbook
22. Add 2 new columns
 - a. Set the column A values as: 30,40,50,60,70,80,90
 - b. Set the column B formula as: " $\ln(\text{col}(A))$ "
 - c. Paste the "B1" copied values into column C
 - d. Set the column D formula as: " $\text{col}(C)/\text{col}(A)^2$ "

	A(Y)	B(X)	C(Y)	D(Y)
Long Name				
Units				
Comments				
F(x)=		$\ln(\text{col}(A))$		$\text{col}(C)/\text{col}(A)^2$
1	30	3.4012	-0.09768	-1.08528E-4
2	40	3.68888	-0.12246	-7.65375E-5
3	50	3.91202	-0.14388	-5.75504E-5
4	60	4.09434	-0.16529	-4.59145E-5
5	70	4.2485	-0.18671	-3.81038E-5
6	80	4.38203	-0.20485	-3.20083E-5
7	90	4.49981	-0.21981	-2.71366E-5
8				

23. Right click column A > "Set As" > Y
24. Right click column B > "Set As" > X
25. Select columns B and D
26. Create a scatter graph

27. Within the upper menu bar, select "Analysis" > "Fitting" > "Linear Fit" > "Open Dialog..."
28. Select "OK"
29. Copy the "Slope" value from this linear fit



30. Create a new workbook
31. Name the new workbook: "SlopeAnalysis"
32. Add 7 new columns
33. In the "Comments" row,
 - a. Name column A: "Peak Position"
 - b. Name column B: " λ "
 - c. Name column C: "Lattice Constant"
 - d. Name column D: "h"
 - e. Name column E: "k"
 - f. Name column F: "l"
 - g. Name column G: " b^2 "
 - h. Name column H: "Slope"
 - i. Name column I: "Dislocation Density"
34. Insert the slope value into the first row of column H
35. Insert the X-ray wavelength (in nm) for the dataset into the first row of column B (*in the example, it is 0.15406*)
36. Identify the major slip plane for the crystal structure of the sample (BCC is 110, FCC is 111)
37. Insert these values into columns D, E, and F
38. Navigate to the "2 Williamson-Hall" folder
39. Open the "WHAnalysis" workbook
40. Copy the value in column C (Peak Position) for the major slip plane for the crystal structure of the sample (*in this example it is the first peak*)
41. Navigate to the "4 Fourier Transformation" folder
42. Open the "SlopeAnalysis" workbook
43. Paste the peak position value in the first row of column A
44. In the "F(x)=" row,
 - a. In column C, insert: " $(\text{col(B)}/(2*\sin(\text{col(A)}/2)))*\sqrt{\text{col(D)}^2+\text{col(E)}^2+\text{col(F)}^2}$ "
 - b. In column G, insert: " $\text{col(C)}^2 / 4*(\text{col(D)}^2+\text{col(E)}^2+\text{col(F)}^2)$ "
 - c. In column I, insert: " $2*\text{col(H)}/(\pi*\text{col(G)})$ "

The resulting workbook should look like this:

	A(X)	B(Y)	C(Y)	D(Y)	E(Y)	F(Y)	G(Y)	H(Y)	I(Y)
Long Name									
Units									
Comments	Peak Position	λ	Lattice Constant	h	k	l	b^2	Slope	Dislocation Density
F(x)=		$\frac{\pi \lambda^2}{4 \sqrt{h^2 + k^2 + l^2}}$					$\frac{2}{4 \sqrt{h^2 + k^2 + l^2}}$		$\frac{2 \sqrt{h^2 + k^2 + l^2}}{\pi \lambda^2}$
1	43.47805	0.15406	0.36022	1	1	1	0.09732	7.24048E-5	4.7363E-4
2									
3									
4									

You are finished!

The value in row one of column H is the dislocation density of the sample in nm^{-2}