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Effect of Acidic pH on Surface Roughness of Current Esthetic Dental Materials

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ABSTRACT

Aim and Hypothesis

The aim of this study was to compare the surface roughness changes of different CAD/CAM restorative dental materials after exposure to acidic pH by using a 3-D optical interferometer.

After exposure to hydrochloric acid solution for 45 hours and 91 hours, it was hypothesized that Vita Enamic® and IPS Empress® CAD would have less surface roughness changes than VITABLOCS® Mark II CAD. On the other hand, they would have more surface roughness changes than IPS e.max® CAD and BruxZir® Solid Zirconia.

Materials and Methods

Five groups of esthetic CAD/CAM block materials were selected: Leucite glass ceramic 'IPS Empress® CAD'; zirconia 'BruxZir® Solid Zirconia'; hybrid ceramic 'VITA Enamic®'; lithium disilicate glass ceramic'IPS e.max® CAD'; and feldspathic porcelain 'VITABLOCS® Mark II CAD'. Eighteen specimens were sectioned from CAD/CAM blocks into rectangular plates (2 mm thick) for each group. All specimens from each group were immersed in a petri dish filled with 20 ml of 5% hydrochloric acid (HCl) with a pH of two and placed into an incubator at 37 °C for 45 hours and 91 hours. The surface roughness average (Ra) of the specimens was measured using a 3-D optical interferometer before the storage period (baseline), after 45 hours, and after 91 hours of exposure to HCl.

Results

Regarding the comparison of surface roughness changes at different periods of evaluation (baseline, 45 hours, 91 hours), there were no statistically significant differences for IPS e.max® CAD (P = 0.063) or BruxZir® Solid Zirconia (P = 0.513).

On the other hand, IPS Empress® CAD, VITABLOCS® Mark II CAD, and VITA Enamic® demonstrated statistically significant differences ($P < 0.001$). IPS Empress® CAD demonstrated statistically significant differences between all time points. VITABLOCS® Mark II CAD and VITA Enamic® showed statistically significant differences between baseline and 45 hours and between baseline and 91 hours, but not between 45 and 91 hours. For all tests that were statistically significant, there was greater surface roughness at the time point with longer duration of HCl exposure (e.g., for IPS Empress® CAD, there was greater surface roughness at 91 hours than at 45 hours, and greater surface roughness at 45 hours than at baseline).

Regarding the comparison of materials in terms of change in surface roughness between baseline – 45 hours and baseline – 91 hours, the Kruskal-Wallis test indicated a statistically significant difference between the materials in both cases ($P < 0.001$). IPS e.max® CAD and BruxZir® Solid Zirconia exhibited the least surface roughness change among the five materials. For both baseline – 45 hours and baseline – 91 hours, the results of the post-hoc tests were the same: there were statistically significant differences between IPS e.max® CAD and all other material groups except BruxZir® Solid Zirconia, and there were statistically significant differences between BruxZir® Solid Zirconia and all other material groups except IPS e.max® CAD. No other post-hoc comparisons were statistically significant.

Conclusion

IPS Empress® CAD, VITABLOCS® Mark II CAD, and VITA Enamic® showed significant increases in surface roughness when they were exposed to simulated gastric acid for 45 and 91 hours. IPS e.max® CAD and BruxZir® Solid Zirconia showed no significant change in surface roughness after exposure to HCl for

45 hours and 91 hours.

DEDICATION

I dedicate my master thesis work to my mother for her boundless love, guidance, support, and encouragement.

To my father for his believing and faith on me.

To my wife, Batool, for her endless sacrifice, support, and encouragement during my educational journey.

To my daughter, Reem, for bringing the happiness to my life.

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LIST OF ABBREVIATIONS

CAD/CAM: Computer-aided design and computer-aided manufacturing.

TEGDMA: Triethylene glycol dimethacrylate.

UDMA: Urethane dimethacrylate.

LIST OF SYMBOLS

Al_2O_3: Aluminium oxide.

B_2O_3: Boron oxide.

CaO: Calcium oxide.

Fe_2O_3: ferric oxide.

HfO_2: Hafnium oxide.

K_2O: Potassium oxide.

Li_2O: Lithium oxide.

MgO: Magnesium oxide.

Na_2O: Sodium oxide.

P_2O_5: Phosphorus oxide.

SiO_2: Silicon dioxide.

TiO_2: Titanium oxide

Y_2O_3: Yttrium oxide.

ZnO: Zinc oxide.

ZrO_2: Zirconium oxide.

SD: Standard deviation.

IQR: Interquartile range.

°C: Celsius.

μm: Micrometer.

Vol%: Volume percent.

Wt%: Weight percent.

EFFECT OF ACIDIC pH ON SURFACE ROUGHNESS OF CURRENT ESTHETIC DENTAL MATERIALS

INTRODUCTION

The development of all ceramic restorative materials has undergone many important changes, and it is expected that the future will bring more brilliant ideas and will improve contemporary ceramics and existing techniques. [1] On account of their excellent physical properties, ceramics are restorative materials that can imitate natural teeth in many aspects like esthetics, biocompatibility, thermal conductibility, and wear resistance. These features allow dental ceramics to be widely utilized in restorative procedures including inlays, onlays, crowns, and veneers. [2]

A new ceramic restorative materials classification was developed by Gracis et al. to classify the materials into three categories based on the presence of specific attributes in their formulation. The first type is glass-matrix ceramics, which is defined as nonmetallic inorganic ceramic materials that contain a glass phase. Second, polycrystalline ceramics can be defined as nonmetallic inorganic ceramic materials that do not have any glass phase. The third type, which is a new type, is resin-matrix ceramics. It is polymer-matrices that contain predominantly inorganic refractory compounds including glasses, ceramics, and glass ceramics. [3]

In glass-matrix ceramics, filler particles are added to the glass composition in order to improve the physical and mechanical properties. These fillers are usually crystalline but can also be particles of a higher melting glass.[4]

Polycrystalline ceramics do not contain a glassy phase. They are tougher and stronger than glass matrix ceramics, but they are more opaque compared with other ceramics. [4] There are many types of polycrystalline ceramics available in dentistry, such as zirconia and alumina. Zirconia ceramics have better mechanical properties compared to other ceramic materials because of the transformation toughening mechanisms in their microstructure.[5]

Resin-matrix ceramics have the advantages of both ceramics and composites. They consist of a hybrid structure with two interpenetrating networks of ceramics and polymers called a double network hybrid. These materials have high flexural strength and elasticity close to dentin because they have a fine structure of feldspathic ceramic and the acrylate polymer network. Vickers hardness of the material, which is a resistance of indentation test, is between dentin and enamel. Wear properties are comparable to common dental ceramics; however, opposing tooth wear is less compared to all ceramic materials. It is also more conservative than conventional ceramic materials as it can be made thinner compared to all ceramic restorations, resulting in less invasive removal of the tooth structure. [6] Hybrid ceramics are presented as highly durable and esthetic materials and are comparable to lithium disilicate and leucite-reinforced ceramics, which can cause less wear to opposing enamel. [7]

Surface roughness of ceramic restorations has an important effect on the strength of restoration and results in a non-uniform stress distribution. [1,8] A rough surface also causes biofilm bacterial accumulation that leads to gingival diseases. [9] The color of porcelain is also affected by the surface roughness because a rough surface reflects less light than a smooth surface.[10]

In the oral environment, dynamic changes occur constantly. The oral environment affects the mechanical properties of ceramic restorations. These effects come from water that come from saliva, the luting cement, and the dentin tubules. Occlusal forces and stresses that are associated with differences in the coefficient of thermal expansion of the restoration components and changes in temperature and pH of the oral environment have negative effects on the mechanical properties of ceramic restorations as well. In terms of pH variations, porcelain restorations are immersed in

saliva in the oral cavity. Although the normal pH of saliva ranges from 6.8 to 7.2, having carbohydrates in the mouth causes the dental plaque to produce organic acids, resulting in lower pH (around 4.5). Food can have a direct effect on decreasing the oral pH that results in acidic pH without the involvement of microorganisms.[11]

Teeth exposure to gastric acid that is regurgitated into the oral cavity causes demineralization of enamel and dentin especially in palatal surfaces of maxillary anterior teeth. This acid exposure often happens in patients with bulimia nervosa and gastro-esophageal reflux disease (GERD). [12] In an acidic environment, the aqueous corrosion of ceramic glasses happens because of selective leaching of alkali ions. Under static or slow flow conditions, the pH value at the ceramic surface usually increases, which will increase the amount of silicon loss from the glass network.[13]

Polymer-based esthetic materials are relatively new in dentistry. Therefore, there is relatively little scientific data found regarding behavior of the material in the oral environment, especially in investigations about the effects of acidic pH changes in the oral cavity.

Ceramic Restorative Materials Classification

A new dental ceramic materials classification was proposed by Gracis et al. to classify ceramic restorative materials into three types based on the content of specific materials in their formulation.

1. Glass-matrix ceramic

There are three types of glass-matrix ceramics:

A. Feldspathic

Feldspathic ceramic consists of clay/kaolin (hydrated aluminosilicate), silicon dioxide (quartz) and a mixture of potassium and sodium aluminosilicates (feldspars). [3] It is formed by ceraming, which is a conversion process from a glass to a partially

crystalline glass. It is a multiphase solid containing a residual glass phase and evenly distributed tiny crystalline phase that results from the controlled crystallization of the glass. [14] It is usually utilized to veneer metal alloy and ceramic substructure.[3]

B. Synthetic

This includes leucite-based, lithium disilicate, and fluorapatite and is composed of silicon dioxide, potassium oxide, sodium oxide, and aluminum oxide. [3] In this type, the fillers are incorporated mechanically during manufacturing by special nucleation and growth-heating treatments to increase strength and thermal expansion of the ceramic materials.[4]

The main component of leucite-based ceramics is feldspathic porcelain. A large amount of leucite crystals are added during ceraming to make 35% by volume. [14] In lithium disilicate-based materials, particles of high-melting glass are added to improve the mechanical properties of ceramic materials. The crystalline phase formed in the crystallization process is lithium disilicate (70% by volume).[14]

C. Glass-Infiltrated

The first glass-infiltrated material introduced was VITA In-Ceram® ALUMINA (VITA Zahnfabrik, Bad Säckingen, Germany) in 1989 using the slip-casting technique. Aluminum oxide (Al_2O_3) is sintered to form a porous skeleton of alumina particles. Then, lanthanum glass is infiltrated in a second firing. VITA In-Ceram® SPINELL (VITA Zahnfabrik, Bad Säckingen, Germany) has a similar mechanism of fabrication, but the skeleton is a porous magnesium Aluminate core. VITA In-Ceram® ZIRCONIA (VITA Zahnfabrik, Bad Säckingen, Germany) is fabricated in the same way as In-Ceram alumina fabrication. However, the slip composition of the Al_2O_3 has stabilized zirconia oxide added to make the ceramic stronger.[3]

2. Polycrystalline ceramics

A. Alumina

This contains a high percentage of Al_2O_3 (to 99.5%). It has been used as a core material since it was introduced in 1990. [3]

B. Zirconia

Pure zirconia has three crystallographic forms. It is monoclinic from room temperature to 1,170°C. Then, it transforms to tetragonal from 1,170°C to 2,370°C, and then cubic when the temperature is higher than 2,370°C. The tetragonal to monoclinic transformation results in a 4% volume increase that can close cracks and increase fracture toughness of the material. [3,15] Adding oxides, such as yttrium, magnesium, calcium, and cerium, to pure zirconia stabilize the tetragonal or cubic phases at room temperature to control the volume expansion. [3,15] This process is called transformation-toughening phenomenon. [3,15] The stabilization of those phases can be full or partial stabilization. [3] Partially stabilized zirconia (PSZ) is formed at room temperature when stabilizing oxides like ceria, magnesia, or yttria are added. [3] Most of the zirconia used in dentistry is yttrium oxide tetragonal zirconia polycrystals because this type of zirconia possesses the highest strength and fracture toughness. [3] Nowadays, zirconia is not only used as a substructure material but also used as monolithic restorations.[3]

3. Resin-matrix ceramics

This type of ceramic is composed of organic matrix filled with ceramic particles. These materials were developed to simulate the modulus of elasticity of dentin, make milling and adjustment easier compared to other types of ceramics, and make a repairable material using composite resin. Based on their inorganic composition, the resin-matrix ceramic materials are divided into different types.[3]

A. Resin nanoceramic

This is composed of a highly cured resin matrix reinforced with nanoceramic particles (approximately 80% by weight). Lava™ Ultimate (3M ESPE, St. Paul, MN, USA) is an example of resin nanoceramics.[3]

B. Glass ceramic in a resin-interpenetrating matrix

This consists of a hybrid structure with two interpenetrating networks of feldspathic ceramic (86% by weight and 75% by volume) and polymer (14% by weight and 25% by volume). That is called double network hybrid. VITA Enamic® (VITA Zahnfabrik, Bad Säckingen, Germany) is an example of this type.[3]

C. Zirconia-silica ceramic in a resin-interpenetrating matrix

Different organic matrices as well as different ceramic weight percentages are used that contain more than 60% of inorganic content by weight.[3]

Chemical Durability

Mechanical forces and chemical attack cause the degradation of dental ceramics. [16-18] Chemical durability is defined as the ability of glass material to resist water and aqueous solutions attack. [16-18] In the past, it was proposed that the glass corrosion caused by the ion exchange process between the glass (alkali ions) and the water (hydrogen ions). [16-18] However, Ernsberger suggested that chemical corrosion occurs by the diffusion of water molecules to form hydroxyl ions by reacting with non-bridging oxygen atoms in glass. Hydroxyl ions diffuse out with the alkali ions to maintain electrical neutrality.[16,17,19]

In the oral environment, where ceramic materials are exposed to an aqueous environment with unstable pH and temperature as well as masticatory forces, surface changes are enhanced. [13] These changes are induced by a corrosion process. [13] In an acidic environment, the corrosion of alkali-silicate glasses occurs as a result of

selective leaching of alkali ions.[13]

Ceramic materials have good chemical durability. However, this chemical durability may be affected by the composition of the ceramic material and the pH, the exposure time, and the temperature of the corrosive medium.[20] Consequently, the corrosion of ceramic materials can occur in different levels.[20] The initial superficial reaction is an acid-base reaction where H+ ions replace alkali ions, resulting in an alkali-ion-depleted leach layer.[20] Beneath this layer, the corrosion process will produce a silica-rich layer that will protect the bulk material from corrosion.[20] However, a partial breakdown of the silicate structure at the surface may occur depending on composition and microstructure of the ceramic material and corrosive medium.[20]

Differences in the microstructure and composition of ceramic materials available in the market will strongly affect the corrosion process qualitatively and quantitatively when ceramic materials are exposed to corrosive media.[13,20] For example, ceramic materials containing zirconia and alumina show less surface degradation compared to leucite containing ceramics.[13,20]

Dental Erosion

Dental erosion is a common cause of tooth damage in children and adults. The extent of destruction caused by dental erosion may range from a superficial loss of enamel surface to complete exposure of dentine.[21] Dental erosion is known as loss of tooth substances due to a nonbacterial chemical process.[21] This acid attack process results in tooth surface demineralization. Consequently, the tooth surface will become more susceptible to erosive wear.[21]

The origin of erosive acid can be intrinsic or extrinsic.[21] Intrinsic origin, such as gastric acid exposure, happens because of vomiting, regurgitation, or GERD.[21]

Extrinsic origin comes when a patient consumes an acidic diet or certain medications. [21] Due to the lower pH of the gastric acid, exposure to this acid causes more corrosion to the tooth structure than an acidic diet. [21,22] GERD can be defined as chronic involuntary movement of gastric acid into the oral cavity. [22] Bulimia nervosa is defined as a life-threatening eating disorder characterized as recurrent cycles of uncontrolled eating accompanied by compensatory self-induced vomiting to avoid weight gain. [23]

Surface Roughness

Surface roughness is known as the irregularities of the surface texture that result from the manufacturing process or material condition. [24]

Surface Roughness Parameters

In engineering, there are many surface roughness parameters used to measure surface roughness. Those parameters give values by recoding the highest peaks and lowest valleys of surface profile. [25] In dentistry, roughness average (Ra) is the most used surface roughness parameter in dental studies. [25,26] Ra is defined as the arithmetic average of all deviations of the roughness profile from the central line. [25,27] There are many other surface roughness parameters used in dentistry like Rq, Rp, Rv, and Rmax (Figure 1). [25] Rq (rms) is the root mean square of all deviations of the roughness profile from the central line. [25,28] Rp is the height of the highest point above the central line within the length of the profile, while Rv is the depth of the lowest point below the central line within the length of the profile. [25] Rmax is the maximum peak to valley roughness.[25,28]

Surface Roughness Measurement

Currently, surface roughness can be measured by several methods, including contact stylus tracing, non-contact laser stylus metrology, scanning electron

microscopy (SEM), atomic force microscopy (AFM), and 3-D Optical Profilometry (white light interferometry). The most common type is the contact stylus tracing. [29] This type of tracing provides a quantitative measurement of surface roughness. Although the contact profilometer measures a large surface area, it can damage a specimen by creating scratches on the surface. [25,30] Furthermore, the stylus radius should be smaller than the concavities on the rough surface. [25,29] Otherwise, the profilometer will not be able to record the concavities and give less accurate reading. [25] 3-D optical interferometer obtains both quantitative and qualitative descriptions of surface topography. Studies have found that the contact profilometer provides more accurate results in measuring changes in surface height like assessment of marginal discrepancies of restorations. [31] However, the laser profilometer is more accurate in surface texture measurements.[31]

3-D Optical Profilometer

3-D optical profilometer, or white light interferometer, is an optical imaging technique utilized for surface roughness measurements. [32] The technique is based on low-coherence interferometry as the light reflects from the specimen surface and without penetrating it because low wavelength light is used. [32] It is designed to measure the 3-D profile of a relatively wide surface area.[32]

Surface Roughness and Plaque Formation

Quirynen et al. proved that there is a clear association between plaque accumulation and surface roughness. [33] The result of this study clearly illustrates that the bacterial colonization rate is directly proportional to surface roughness. [33] According to this study, there are two explanations for this phenomenon. [33] First, the microorganisms imbedded in irregularities of a rough surface are protected against mechanical removal of those microorganisms during mastication. [33] The second

explanation is that the surface area increases by two or three times in rough surfaces compared to smooth surfaces. [33] This increase in surface roughness enhances bacterial adhesion and colonization.[33]

Surface Roughness and Restoration Color

A rough surface will change and affect the restoration color and appearance by reflecting an irregular and diffuse pattern of light. Besides the surface roughness, surface gloss and fluorescence affect the restoration color and appearance. [10] A smooth surface will reflect more light compared to a rough surface, and this is important for the color and appearance of the restoration.[10]

Surface Roughness and Flexural Strength

During function, ceramic restorations with rough surfaces will concentrate an applied stress and lead to a non-uniform stress distribution resulting in a lower flexural strength.[34]

Surface Roughness and Gastric Acid

Exposures of porcelain restorations to gastric acid result in more severe erosion compared to the acidic diet. [22] The reason is that the gastric acid has a lower pH than that of the acidic diet. [22] Porcelain restorations get exposed to gastric acid through vomiting or regurgitation. [22] GERD also results in gastric acid movement from the stomach into the oral cavity. [22] Furthermore, some eating disorders such as anorexia and bulimia nervosa are common causes of erosion. [22] While the critical pH of enamel is 5.5, gastric acid has an extremely acidic pH that is less than two. [22] This means that the amount of erosive degradation caused by gastric acid exposure is huge. [22] The buffering capacity of saliva protects teeth by reducing the effects of erosive destruction from an acid attack. [22] However, in patients suffering from GERD, the saliva does not have enough buffering time to prevent erosion.[22]

No clear agreement was found in the literature regarding the method of gastric acid simulation in lab studies to replicate an in-vivo model. The ISO standard 6872 solubility tests for dental ceramics suggests using 4% acetic acid and an exposure time of 16 hours at 80°C, which is equivalent to approximately two years of a normal clinical situation. [22,35] Concentration of the corrosive acid, the exposure time, and the temperature affect the in-vitro simulation of the effect of acid on the surface roughness of ceramic restorations. [22] Harryparsad et al. have found that the average exposure time of teeth to gastric acid in a bulimic patient is 15 minutes daily. [12] This means that immersion of a sample in HCl (pH = 2) for 45 hours and 91 hours represents gastric acid exposure in a bulimic patient for 6 months and 12 months, respectively.[12]

Tooth structure that is affected by dental erosion can be restored with either direct or indirect restorations. Few studies in the literature have reviewed the effects of acidic and erosive conditions on those restorations.[22]

SPECIFIC AIM

The aim of this study was to compare the surface roughness changes of different CAD/CAM restorative dental materials after exposure to acidic pH by using a 3-D optical interferometer.

HYPOTHESIS

After exposure to HCl acid solution for 45 hours and 91 hours, Vita Enamic® and IPS Empress® CAD have less surface roughness changes than VITABLOCS® Mark II CAD. On the other hand, they have more surface roughness changes than IPS e.max® CAD and BruxZir Solid Zirconia.

CLINICAL SIGNIFICANCE OF THE STUDY

Results of this study will give clinicians an idea about the performance of resin-matrix ceramics as compared to other CAD/CAM esthetic materials in terms of surface roughness after HCl acid exposure that often happens in patients with bulimia nervosa and GERD.

MATERIALS AND METHODS

1. Specimen preparation

Five groups were fabricated with five esthetic CAD/CAM block materials (Table 1): high leucite glass ceramic 'IPS Empress® CAD' (Ivoclar Vivadent AG - Schaan, Liechtenstein); partially stabilized zirconia 'BruxZir® Solid Zirconia' (Glidewell Laboratories, Irvine, USA); hybrid ceramic 'VITA Enamic®' (VITA Zahnfabrik, Bad Säckingen, Germany); lithium disilicate porcelain 'IPS e.max® CAD' (Ivoclar Vivadent AG - Schaan, Liechtenstein); and feldspathic porcelain 'VITABLOCS® Mark II CAD' (VITA Zahnfabrik, Bad Säckingen, Germany).

Eighteen specimens were sectioned from CAD/CAM blocks into rectangular plates (2 mm thick) for each group with a sectioning saw (Isomet 1000; Buehler, Lake Bluff, IL) (Figures 1, 2). The BruxZir® zirconia and IPS e.max® CAD blocks were cut in the green stage and then sintered according to the manufacturers' instructions. Each specimen was attached to a sample holder using sticky wax (Figure 3). To standardize the polishing procedure before the surface roughness test, the test surfaces of all specimens were flattened and polished under water-cooling in an automatic polishing machine (Ecomet 250; Buehler, Lake Bluff, IL) with 120, 240, 320, and 600 grit silicon carbide grinding papers (Buehler, Lake Bluff, IL) at 350 rpm for 120 seconds with an applied force of 13.34 Newton (Figure 4). The final thickness of each specimen was measured using a digital caliper (Dentaguage 1, erskineDental, Macksville, Australia; Figure 5). All of the specimens were then ultrasonically cleaned in distilled water (Quantrex LR 606, Maplewood, NJ) for 10 minutes and air dried and kept at room temperature before testing.

2. Storage

All specimens from each material group were immersed in a petri dish filled

with 20 ml of 5% hydrochloric acid HCl with pH of 2 (DR. CLARK DIGESTIVE POWER; Self Health Resource Center, Chula Vista, CA) and placed into an incubator (Thermo Fisher Scientific, Waltham, MA) at 37°C for 45 hours and 91 hours; this equals six months and one year of exposure to HCl in a bulimic patient, respectively (Figure 6). [12] After each step, the specimens were rinsed with deionized water and air-dried to be ready for quantitative and qualitative analysis. Each specimen was tested before exposure (baseline), after 45 hours of exposure to HCl, and after 91 hours of exposure to HCl (Figure 7).

3. Surface roughness measurement

For the quantitative analysis, a profilometric evaluation was performed. The surface roughness average (Ra) of the specimens was measured using a 3-D optical interferometer (Zygo NewView 600, Zygo corporation, Middlefield, CT) before the storage period (baseline), after 45 hours of exposure to HCl, and after 91 hours of exposure to HCl (Figure 8). Silicon mold (Genie Putty; Sultan Healthcare, York, PA) was made for each material group with a 5-mm hole created in the center of the silicon mold using disposable punch (Premier, Plymouth Meeting, PA) to standardize the test area and facilitate analyzing the same test area before and after exposure to the acidic solution (Figure 9). Light and focus were adjusted automatically. A field of view 526 x 702 μm^2 was acquired using an objective with 1x zoom and 10x of magnification to cover area of 0.37 mm^2. The measurement control panel was adjusted to make a bipolar scan mode for 10 seconds and 40 μm for vertical scan length. Images were captured with 640 x 480 high-resolution and they were analyzed with Metro Pro software. [36] The analysis control panel was adjusted to remove 2.50 μm spikes and to fill missing data points using adjacent valid data points. The surface roughness values were recorded in micrometers (μm) and 3-D images were displayed.

For the qualitative characterization of wear patterns, the specimens' test areas were visually examined by a 3-D image of surface profile using the 3-D optical interferometer. Both quantitative and qualitative tests were performed in Mechanical Engineering lab, School of Engineering, Tufts University, Medford, MA.

STATISTICAL ANALYSIS

1. Sample Size Calculation

Version 7.0 of the statistical software package nQuery Advisor was used to perform a power calculation. Anticipated means of the IPS e.max® CAD group, IPS Empress® CAD group and VITABOCS® Mark II CAD, as well as the anticipated within-group standard deviation, were based on the results of Harryparsad et al. [12] The calculation further assumed that the IPS e.max® CAD group and the BruxZir® Solid Zirconia group would have the same mean, and that the IPS Empress® CAD group and VITA Enamic® group would have the same mean. Based on these assumptions, a sample size of n = 18 per group was adequate to obtain a Type I error rate of 5% and a power greater than 99% for the comparison of materials.

2. Data Analysis

To compare the surface roughness between the materials at each time of evaluation (baseline, 45 hours, and 91 hours), descriptive statistics (means, medians, standard deviations, and inter-quartile ranges) were calculated. Because the data were not normally distributed, statistical significance for each time of evaluation was assessed via the Kruskal-Wallis test with Dunn's test and Bonferroni correction used for post-hoc tests.

In order to test the difference in surface roughness between the periods of evaluation for each material, the Friedman test was used. The Wilcoxon signed-rank test with Bonferroni correction was used for post-hoc comparisons.

The change in surface roughness between baseline and 45 hours ($\Delta_{45h\text{-}baseline}$ = surface roughness at 45 hours – surface roughness at baseline) and the change between baseline and 91 hours ($\Delta_{91h\text{-}baseline}$ = surface roughness at 91 hours – surface roughness at baseline) were calculated. The $\Delta_{45h\text{-}baseline}$ and $\Delta_{91h\text{-}baseline}$ values were

compared between the material groups using the Kruskal-Wallis test, with post-hoc tests again conducted via Dunn's test and Bonferroni correction. SPSS Version 22 was used in the analysis.

RESULTS

1. Surface Roughness Measurements

The surface roughness results (mean, median, standard deviation, and inter-quartile range) for each material at each period of evaluation (baseline, 45 hours, 91 hours) are shown in Table 2 (Figures 10, 11, 12). At baseline evaluation, IPS e.max® CAD and VITA Enamic® demonstrated the lowest surface roughness results compared to the other groups. The Kruskal-Wallis test showed that there was a statistically significant difference between the materials ($P < 0.001$). In post-hoc testing, there was no statistically significant difference between VITA Enamic® and IPS e.max® CAD ($P = 0.446$). There was also no significant difference between VITA Enamic® and VITABLOCS® Mark II CAD when the Bonferroni correction was used ($P = 0.032$). There was no statistically significant difference, using the Bonferroni correction, between BruxZir® Solid Zirconia and either IPS Empress® CAD ($P = 0.042$) or VITABLOCS® Mark II CAD ($P = 0.331$). All other post-hoc tests were statistically significant.

At 45 hours, the Kruskal-Wallis test showed a statistically significant difference between materials in terms of surface roughness ($P < 0.001$). In post-hoc testing, there was no statistically significant difference when the Bonferroni correction was used between VITA Enamic® and either IPS e.max® CAD ($P = 0.022$) or VITABLOCS® Mark II CAD ($P = 0.043$). There was no statistically significant difference between BruxZir® Solid Zirconia and either VITA Enamic® ($P = 0.237$) or VITABLOCS® Mark II CAD ($P = 0.400$). The difference between VITABLOCS® Mark II CAD and IPS Empress® CAD was not statistically significant when the Bonferroni correction was used ($P = 0.019$). All other post-hoc tests were statistically significant.

At 91 hours, the Kruskal-Wallis test indicated a statistically significant difference between materials in terms of surface roughness ($P < 0.001$). In post-hoc testing, there was no statistically significant difference between VITA Enamic® and either BruxZir® Solid Zirconia ($P = 0.901$) or VITABLOCS® Mark II CAD ($P = 0.082$). There was no statistically significant difference between VITABLOCS® Mark II CAD and either BruxZir® Solid Zirconia ($P = 0.106$) or IPS Empress® CAD ($P = 0.018$) when the Bonferroni correction was used. All other post-hoc tests were statistically significant.

2. Surface Roughness Changes

Regarding the comparison of the periods of evaluation for each material, the Friedman test (Figure 13) showed that there were no statistically significant differences for IPS e.max® CAD ($P = 0.063$) or BruxZir® Solid Zirconia ($P = 0.513$). On the other hand, IPS Empress® CAD, VITABLOCS® Mark II CAD, and VITA Enamic® demonstrated statistically significant differences ($P < 0.001$). IPS Empress® CAD demonstrated statistically significant differences between all time points ($P = 0.001$ for baseline – 45 hours, $P < 0.001$ for baseline – 91 hours, $P = 0.001$ for 45 hours – 91 hours). VITABLOCS® Mark II CAD and VITA Enamic® showed statistically significant differences between baseline and 45 hours ($P < 0.001$ for VITABLOCS® Mark II CAD, $P = 0.001$ for VITA Enamic®) and between baseline and 91 hours ($P < 0.001$ for VITABLOCS® Mark II CAD, $P < 0.001$ for VITA Enamic®), but not between 45 and 91 hours when using the Bonferroni correction ($P = 0.048$ for VITABLOCS® Mark II CAD, $P = 0.048$ for VITA Enamic®). For all tests that were statistically significant, there was greater surface roughness at the time point with longer duration of HCl exposure (e.g., for IPS Empress® CAD, there was greater

surface roughness at 91 hours than at 45 hours, and greater surface roughness at 45 hours than at baseline).

Results regarding the amount of change in surface roughness $\Delta_{45h\text{-}baseline}$ and $\Delta_{91h\text{-}baseline}$ (mean, median, standard deviation, and inter-quartile range) are shown in Table 3 (Figures 14, 15). In both cases, the Kruskal-Wallis test indicated a statistically significant difference between the materials ($P < 0.001$), with IPS e.max® CAD and BruxZir® Solid Zirconia exhibiting the least surface roughness change among the five materials. Furthermore, in both cases, the results of the post-hoc tests were the same: there were statistically significant differences between IPS e.max® CAD and all other material groups except BruxZir® Solid Zirconia, and there were statistically significant differences between BruxZir® Solid Zirconia and all other material groups except IPS e.max® CAD. No other post-hoc comparisons were statistically significant.

3. Surface Topography

The 3-D models and surface profile charts for each material group at each test time (baseline, 45 hours, 91 hours) were created using the 3-D optical interferometer (Figures 16, 17, 18, 19, 20). IPS Empress® CAD, VITABLOCS® Mark II CAD, and VITA Enamic® showed obvious changes in surface topography by increase in the depth of pores and valleys after exposure to HCl. On the other hand, IPS e.max® CAD and BruxZir® Solid Zirconia showed almost no change in surface topography after immersion in HCl for 45 hours or 91 hours compared to the baseline 3-D model.

DISCUSSION

The objective of this in-vitro study was to compare the surface roughness changes of different CAD/CAM restorative dental materials after exposure to gastric acid. Increasing in surface roughness of a dental material affects physical and mechanical properties of the material negatively. It was found in previous works that changes in surface roughness stimulate plaque accumulation and color change and decrease flexural strength of the restoration. [10,33,34] The present study showed that some materials are affected more in terms of surface roughness than other materials.

Surface roughness can be measured by several methods including contact stylus tracing, non-contact laser stylus metrology, scanning electron microscopy, atomic force microscopy and 3-D Optical profilometry. One study compared contact and non-contact profilometry and found that both methods provided a reliable measurement of corrosion. [37] Although contact profilometers measure large surface areas, they create scratches on the surface that cause damage to the test materials. [25,30] Furthermore, if the stylus radius was larger than the concavities on the rough surface, then the profilometer will not be able to record the concavities and will give a less accurate reading. [25] Those limitations can be avoided by using non-contact profilometer. In the present study, 3-D optical interferometer was used.

Roughness average (Ra), which is the most used surface roughness parameter in dental studies, was used in the present study to compare the surface roughness of the different materials. [25,26] At baseline, IPS e.max® CAD and VITA Enamic® demonstrated the lowest surface roughness, while IPS Empress® CAD showed the highest surface roughness which was not significantly different from either BruxZir® Solid Zirconia or VITABLOCS® Mark II CAD. Baseline surface roughness was measured to record the initial surface roughness for each group. Harryparsad et al.

supported the finding of the present study that IPS e.max® CAD has less baseline surface roughness than IPS Empress® CAD and VITABLOCS® Mark II CAD. [12] However, another study found that there is no significant difference in baseline surface roughness between IPS e.max® CAD and either high leucite ceramic or Feldspathic ceramic. [38] The result of this study is different from the present study because the samples were glazed after polishing, which is not the case for our study.

There is no clear agreement in the literature in regards to the method of gastric acid simulation in lab studies to replicate an in-vivo model. Backer et al. used gastric juice (pH = 1.2) for six hours and 18 hours that represent two and eight years of exposure to vomiting, respectively. [23] Sulaiman et al. used HCl (pH = 1.2) for 96 hours at 37°C to simulate over 10 years of clinical exposure. [22] Another study exposed test materials to acidic solution (pH = 3.8) for 24 hours. [39] The bulimic scenario used for the present study was designed according Harryparsad et al. who found exposure to HCl (pH = 2) for 45 hours and 91 hours represents gastric acid exposure in a bulimic patient for six months and 12 months, respectively.[12]

After exposure to HCl for 45 hours and 91 hours, IPS e.max® CAD and BruxZir® Solid Zirconia were not significantly affected by the HCl immersion in terms of surface roughness change. IPS Empress® CAD, VITABLOCS® Mark II CAD, and VITA Enamic® showed a significant increase in surface roughness after exposure to gastric acid compared to the baseline surface roughness.

Regarding the amount of change in surface roughness ($\Delta_{45h\text{-}baseline}$ and $\Delta_{91h\text{-}baseline}$), IPS e.max® CAD and BruxZir® Solid Zirconia were resistant to acid corrosion and demonstrated almost no change in terms of $\Delta_{45h\text{-}baseline}$ and $\Delta_{91h\text{-}baseline.}$ IPS Empress® CAD, VITABLOCS® Mark II CAD, and VITA Enamic® displayed significant increase in surface roughness in $\Delta_{45h\text{-}baseline}$ and $\Delta_{91h\text{-}baseline.}$

Few studies tested the effect of gastric acid on the surface roughness on esthetic dental materials. Backer et al. found that gastric acid has a negative effect on surface roughness of two brands of CAD/CAM resin composite restorations. [23] Another study found the surface roughness and micro hardness of direct composite restorations were compromised when it is exposed to gastric acid for 24 hours. [40] On the other hand, one study found that exposure to HCl has no significant effects on the surface change on different glass ceramic materials. [39] The exposure time for this study was relatively short (24 hours) and pH of the corrosive solution (pH = 3.8) used was higher compared to the present study.[39]

The local composition and microstructure of the restorative material are the major reasons of the surface roughness changes that occur after exposure to an acidic solution. [13] VITA Enamic® consists of a hybrid structure with two interpenetrating networks of feldspathic ceramic and composite resin. [3] Increases in surface roughness of VITA Enamic® after exposure to HCl acid can be explained by hydrolysis of methacrylate ester bonds within the resin matrix of polymer-based materials when they are exposed to acidic solution. [23,41] Furthermore, the main reason for surface roughness changes in this material is the detachment of inorganic fillers that are attached to the resin matrix from the resin composite's surface as coupling agents connect the organic and inorganic components together. [23,42] According to Harryparsad et al., the size of glass particles lost in glass matrix ceramic materials during acid exposure is directly proportional to the amount surface roughness change. [12] Thus, because the grain size of IPS Empress® CAD (1-5 μm) and VITABLOCS® Mark II CAD (4 μm) is larger than that of IPS e.max® CAD (0.2–1.0 μm), they show more surface roughness change than IPS e.max® CAD. [12,43-45]

Another study found that IPS e.max® CAD showed significant increase in

surface roughness compared to different types of partially and fully stabilized zirconia when they were exposed to HCl for 96 hours. [22] However, the present study found that both IPS e.max® CAD and BruxZir® Solid Zirconia showed no significant change in surface roughness after exposure to HCl for 45 hours and 91 hours. The pH of HCl used in the Sulaiman et al. study was more acidic (pH =1.2) and exposure time was longer (96 hours).[22]

There are some limitations in the present study that need to be addressed in future experiments. It is claimed that the color and transparency of the tested material can affect the accuracy of the optical profilometer results. [25] It is better to combine more than one surface roughness measurement technique to provide more accurate results. [25] Furthermore, it is suggested to include more than one surface roughness parameter beside Ra, [46] which is a two-dimensional value and provides no information about surface profile characteristics.[25,38]

In the present study, the role of saliva in oral cavity was not considered. Saliva plays a major role in buffering the acidic diet and solutions. [38] Also, pH and temperature in oral cavity are not stable and are dynamically changed. [38] This fluctuation scenario is difficult to be applied in in-vitro studies. Another limitation of the present study is lack of ion leaching measurements that provide further information about the materials' behavior in a corrosive medium. [22] Samples used in this study were polished but not glazed before surface roughness measurement. Glazing of test materials before exposure to the corrosive solution will mimic the real situation and will give more accurate results. However, previous studies found that there is no significant difference between polished and glazed porcelain surfaces in terms of surface smoothness and characteristics. [47-49]

CONCLUSION

Within the limitations of the present study and based on the study findings, IPS Empress® CAD, VITABLOCS® Mark II CAD, and VITA Enamic® showed significant increase in surface roughness when they were exposed to simulated gastric acid for 45 and 91 hours. IPS e.max® CAD and BruxZir® Solid Zirconia showed no significant change in surface roughness after exposure to HCl for 45 hours and 91 hours. Thus, full zirconia and lithium disilicate porcelain materials are more resistant to HCl exposure than other glass matrix and resin matrix porcelain materials.

REFERENCES

1. Rashid H. The effect of surface roughness on ceramics used in dentistry: A review of literature. *Eur J Dent*. 2014;8(4):571-579.

2. Camacho GB, Vinha D, Panzeri H, Nonaka T, Goncalves M. Surface roughness of a dental ceramic after polishing with different vehicles and diamond pastes. *Braz Dent J*. 2006;17(3):191-194.

3. Gracis S, Thompson VP, Ferencz JL, Silva NR, Bonfante EA. A new classification system for all-ceramic and ceramic-like restorative materials. *Int J Prosthodont*. 2015;28(3):227-235.

4. Kelly JR, Benetti P. Ceramic materials in dentistry: Historical evolution and current practice. *Aust Dent J*. 2011;56 Suppl 1:84-96.

5. Piconi C, Maccauro G. Zirconia as a ceramic biomaterial. *Biomaterials*. 1999;20(1):1-25.

6. Dirxen C, Blunck U, Preissner S. Clinical performance of a new biomimetic double network material. *Open Dent J*. 2013;7:118-122.

7. VITA ENAMIC® Technical and scientific documentation. . 2011.

8. Giordano R, Cima M, Pober R. Effect of surface finish on the flexural strength of feldspathic and aluminous dental ceramics. *Int J Prosthodont*. 1995;8(4):311-319.

9. Kawai K, Urano M, Ebisu S. Effect of surface roughness of porcelain on adhesion of bacteria and their synthesizing glucans. *J Prosthet Dent*. 2000;83(6):664-667.

10. Sarac D, Sarac YS, Yuzbasioglu E, Bal S. The effects of porcelain polishing systems on the color and surface texture of feldspathic porcelain. *J Prosthet Dent*. 2006;96(2):122-128.

11. Pinto MM, Cesar PF, Rosa V, Yoshimura HN. Influence of pH on slow crack growth of dental porcelains. *Dent Mater*. 2008;24(6):814-823.

12. Harryparsad A, Dullabh H, Sykes L, Herbst D. The effects of hydrochloric acid on all-ceramic restorative materials: An in-vitro study. *SADJ*. 2014;69(3):106-111.

13. Milleding P, Wennerberg A, Alaeddin S, Karlsson S, Simon E. Surface corrosion of dental ceramics in vitro. *Biomaterials*. 1999;20(8):733-746.

14. Shenoy A, Shenoy N. Dental ceramics: An update. *J Conserv Dent*. 2010;13(4):195-203.

15. Conrad HJ, Seong WJ, Pesun IJ. Current ceramic materials and systems with clinical recommendations: A systematic review. *J Prosthet Dent*. 2007;98(5):389-404.

16. Anusavice KJ. Degradability of dental ceramics. *Adv Dent Res*. 1992;6:82-89.

17. Anusavice KJ, Zhang NZ. Chemical durability of dicor and lithia-based glass-ceramics. *Dent Mater*. 1997;13(1):13-19.

18. Newton RG. The durability of glass: A review. *Glass technology* . In: Vol 26. ; 1985:21-38.

19. Ernsberger FM. The role of molecular water in the diffusive transport of protons in glasses. *Physics chem glasses* . In: Vol 21. ; 1980:146-149.

20. Milleding P, Haraldsson C, Karlsson S. Ion leaching from dental ceramics during static in vitro corrosion testing. *J Biomed Mater Res*. 2002;61(4):541-550.

21. Pace F, Pallotta S, Tonini M, Vakil N, Bianchi Porro G. Systematic review: Gastro-oesophageal reflux disease and dental lesions. *Aliment Pharmacol Ther*. 2008;27(12):1179-1186.

22. Sulaiman TA, Abdulmajeed AA, Shahramian K, et al. Impact of gastric acidic challenge on surface topography and optical properties of monolithic zirconia. *Dent Mater*. 2015;31(12):1445-1452.

23. Backer AD, Munchow EA, Eckert GJ, Hara AT, Platt JA, Bottino MC. Effects of simulated gastric juice on CAD/CAM resin composites-morphological and mechanical evaluations. *J Prosthodont*. 2015.

24. Paravina RD PJ. *Esthetic color training in dentistry*. Saint Louis: Mosby; 2004.

25. Field J, Waterhouse P, German M. Quantifying and qualifying surface changes on dental hard tissues in vitro. *J Dent*. 2010;38(3):182-190.

26. Al-Shammery HA, Bubb NL, Youngson CC, Fasbinder DJ, Wood DJ. The use of confocal microscopy to assess surface roughness of two milled CAD-CAM ceramics following two polishing techniques. *Dent Mater*. 2007;23(6):736-741.

27. Misumi I, Naoi K, Sugawara K, Gonda S. Profile surface roughness measurement using metrological atomic force microscope and uncertainty evaluation. . 2015;73:295-303.

28. Zygo® surface texture parameters. . April 2013.

29. Whitehead SA, Shearer AC, Watts DC, Wilson NHF. Comparison of two stylus methods for measuring surface texture. *Dental Materials*. 1999;15:79-86.

30. Heurich E, Beyer M, Jandt KD, et al. Quantification of dental erosion--a comparison of stylus profilometry and confocal laser scanning microscopy (CLSM). *Dent Mater*. 2010;26(4):326-336.

31. Whitehead SA, Shearer AC, Watts DC, Wilson NH. Comparison of two stylus methods for measuring surface texture. *Dent Mater*. 1999;15(2):79-86.

32. Larimer C, Suter JD, Bonheyo G, Addleman RS. In situ non-destructive measurement of biofilm thickness and topology in an interferometric optical microscope. *J Biophotonics*. 2016;9(6):656-666.

33. Quirynen M, Marechal M, Busscher HJ, Weerkamp AH, Darius PL, van Steenberghe D. The influence of surface free energy and surface roughness on early plaque formation. an in vivo study in man. *J Clin Periodontol*. 1990;17(3):138-144.

34. de Jager N, Feilzer AJ, Davidson CL. The influence of surface roughness on porcelain strength. *Dent Mater*. 2000;16(6):381-388.

35. International standards for dental ceramics. ISO 6872. . 1995.

36. Khayat W, Chebib N, Ali A, Finkelman M, Khayat S. *The effect of grinding and polishing on roughness of zirconia and its consequent effect on mechanical behavior*. Tufts University School of Dental Medicine; 2015.

37. Paepegaey AM, Barker ML, Bartlett DW, et al. Measuring enamel erosion: A comparative study of contact profilometry, non-contact profilometry and confocal laser scanning microscopy. *Dent Mater*. 2013;29(12):1265-1272.

38. Kukiattrakoon B, Hengtrakool C, Kedjarune-Leggat U. Effect of acidic agents on surface roughness of dental ceramics. *Dent Res J (Isfahan)*. 2011;8(1):6-15.

39. Matsou E, Vouroutzis N, Kontonasaki E, Paraskevopoulos KM, Koidis P. Investigation of the influence of gastric acid on the surface roughness of ceramic materials of metal-ceramic restorations. an in vitro study. *Int J Prosthodont*. 2011;24(1):26-29.

40. Cengiz S, Sarac S, Ozcan M. Effects of simulated gastric juice on color stability, surface roughness and microhardness of laboratory-processed composites. *Dent Mater J*. 2014;33(3):343-348.

41. Turssi CP, Hara AT, de Magalhaes CS, Serra MC, Rodrigues AL,Jr. Influence of storage regime prior to abrasion on surface topography of restorative materials. *J Biomed Mater Res B Appl Biomater*. 2003;65(2):227-232.

42. Karabela MM, Sideridou ID. Synthesis and study of properties of dental resin composites with different nanosilica particles size. *Dent Mater*. 2011;27(8):825-835.

43. VITABLOCS® Working instructions. . February 2016.

44. Scientific Documentation IPS e.max® CAD. . March 2011.

45. Scientific documentation IPS empress CAD®. . October 2011.

46. Whitehead SA, Shearer AC, Watts DC, Wilson NH. Comparison of methods for measuring surface roughness of ceramic. *J Oral Rehabil*. 1995;22(6):421-427.

47. Hmaidouch R, Muller WD, Lauer HC, Weigl P. Surface roughness of zirconia for full-contour crowns after clinically simulated grinding and polishing. *Int J Oral Sci*. 2014;6(4):241-246.

48. Sulik WD, Plekavich EJ. Surface finishing of dental porcelain. *J Prosthet Dent*. 1981;46(2):217-221.

49. Brewer JD, Garlapo DA, Chipps EA, Tedesco LA. Clinical discrimination between autoglazed and polished porcelain surfaces. *J Prosthet Dent*. 1990;64(6):631-634.

APPENDICES

Appendix A: Tables

Appendix B: Figures

APPENDIX A: TABLES

Table 1: CAD/CAM materials used in the study

Brand Name	Manufacturer	Composition
IPS Empress® CAD	Ivoclar Vivadent AG - Schaan, Liechtenstein	SiO_2, Al_2O_3, K2O, Na_2O, Other oxides, Pigments[45]
BruxZir® Solid Zirconia	Glidewell Laboratories, Irvine, USA	Y_2O_3, HfO_2, Al_2O_3, SiO_2, Fe_2O_3, Na_2O, and balance ZrO_2[22]
VITA Enamic®	VITA Zahnfabrik, Bad Säckingen, Germany	Composition of the ceramic part (86 wt% / 75 vol%) SiO_2, Al_2O_3, Na_2O, K_2O, B_2O_3, ZrO_2, CaO Composition of the polymer part (14 wt% / 25 vol%) UDMA, TEGDMA[7]
IPS e.max® CAD	Ivoclar Vivadent AG - Schaan, Liechtenstein	SiO_2, Li_2O, K_2O, P_2O_5, ZrO2, ZnO, Al_2O_3, MgO, Colouring oxides[44]
VITABLOCS® Mark II CAD	VITA Zahnfabrik, Bad Säckingen, Germany	Al_2O_3, SiO_2, Na_2O, K_2O, CaO, TiO_2[43]

Table 2: Surface roughness results for each material at each period of evaluation (baseline, 45 hours, 91 hours)

Group	Baseline				45 hours				91 hours				**P value
	Mean	SD	Median	IQR	Mean	SD	Median	IQR	Mean	SD	Median	IQR	
IPS Empress® CAD	1.23	0.66	1.26	1.11	1.65	0.60	1.57	1.08	1.95	0.64	1.98	1.13	< 0.001
BruxZir® Solid Zirconia	0.54	0.23	0.59	0.34	0.51	0.20	0.50	0.24	0.52	0.19	0.55	0.28	0.513
VITA Enamic®	0.18	0.22	0.11	0.12	0.43	0.32	0.31	0.30	0.58	0.43	0.46	0.36	< 0.001
IPS e.max® CAD	0.13	0.14	0.08	0.19	0.16	0.20	0.07	0.27	0.13	0.15	0.07	0.22	0.063
VITABLOCS® Mark II CAD	0.61	0.60	0.43	0.97	1.08	0.89	0.75	1.75	1.15	0.86	0.93	1.68	< 0.001
*P value	< 0.001				< 0.001				< 0.001				

*P values comparing materials at each time point (Kruskal-Wallis test)
**P values comparing time points for each material (Friedman test)

Table 3: Results regarding the amount of change in surface roughness $\Delta_{45h\text{-}baseline}$ and $\Delta_{91h\text{-}baseline}$

Group	$\Delta_{45h\text{-}baseline}$				$\Delta_{91h\text{-}baseline}$			
	Mean	SD	Median	IQR	Mean	SD	Median	IQR
IPS Empress® CAD	0.42	0.36	0.39	0.38	0.72	0.37	0.64	0.46
BruxZir® Solid Zirconia	-0.03	0.15	0.01	0.15	-0.02	0.14	0.01	0.11
VITA Enamic®	0.25	0.21	0.21	0.23	0.40	0.27	0.36	0.33
IPS e.max® CAD	0.03	0.12	0.01	0.08	0.01	0.05	0.01	0.06
VITABLOCS® Mark II CAD	0.47	0.42	0.28	0.53	0.54	0.42	0.43	0.42
P value	< 0.001				< 0.001			

P values compare the materials in terms of Δ45h-baseline and Δ91h-baseline

(Kruskal-Wallis test)

APPENDIX B: FIGURES

Figure 1: Cutting machine used to section CAD/CAM blocks

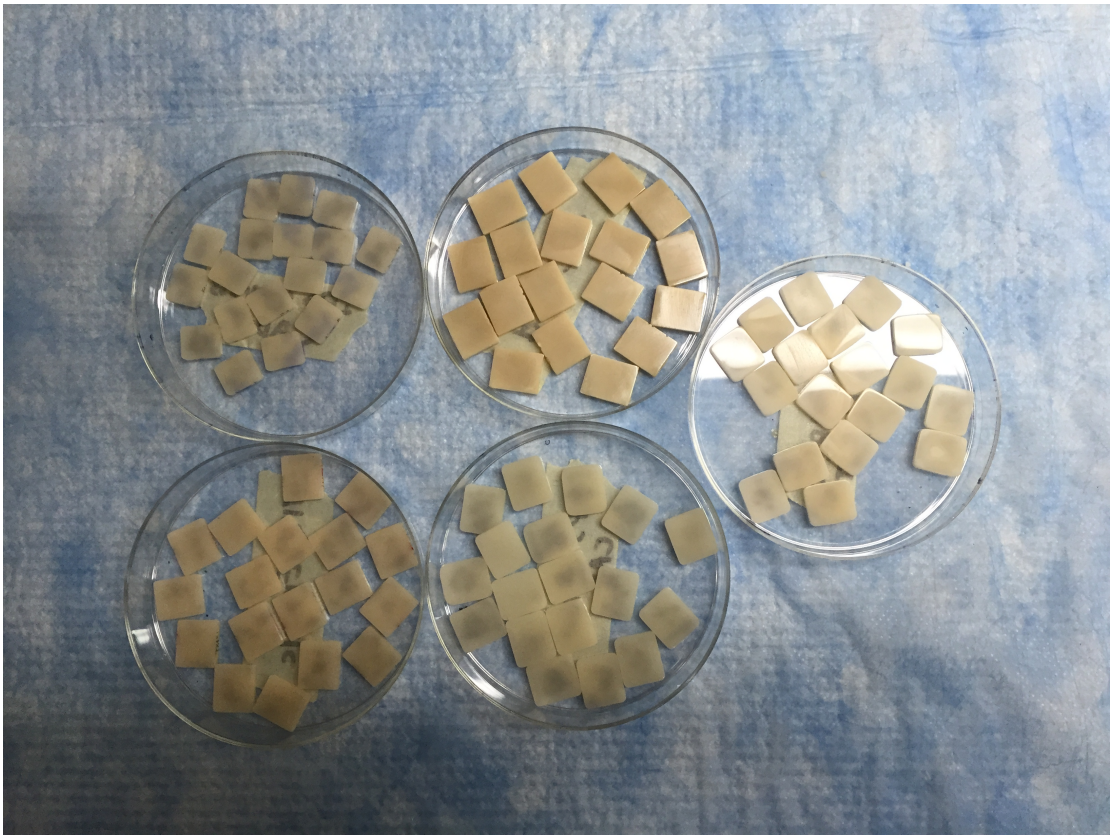


Figure 2: Rectangular plates (2 mm thick) of the five material groups

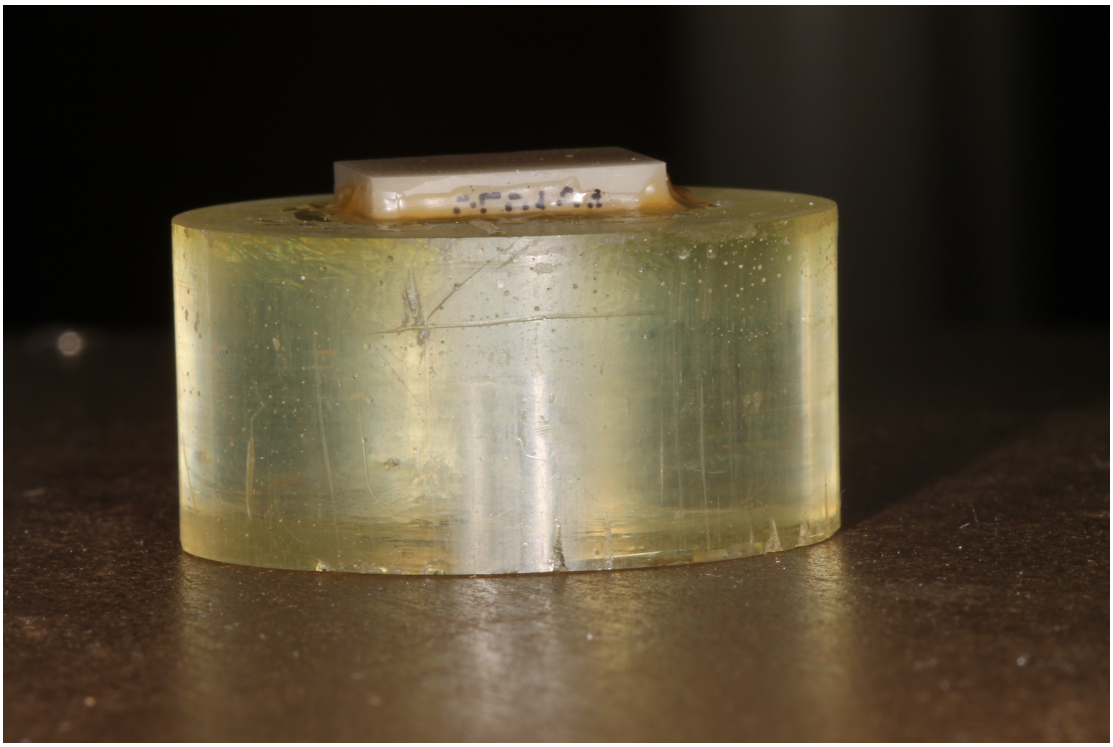
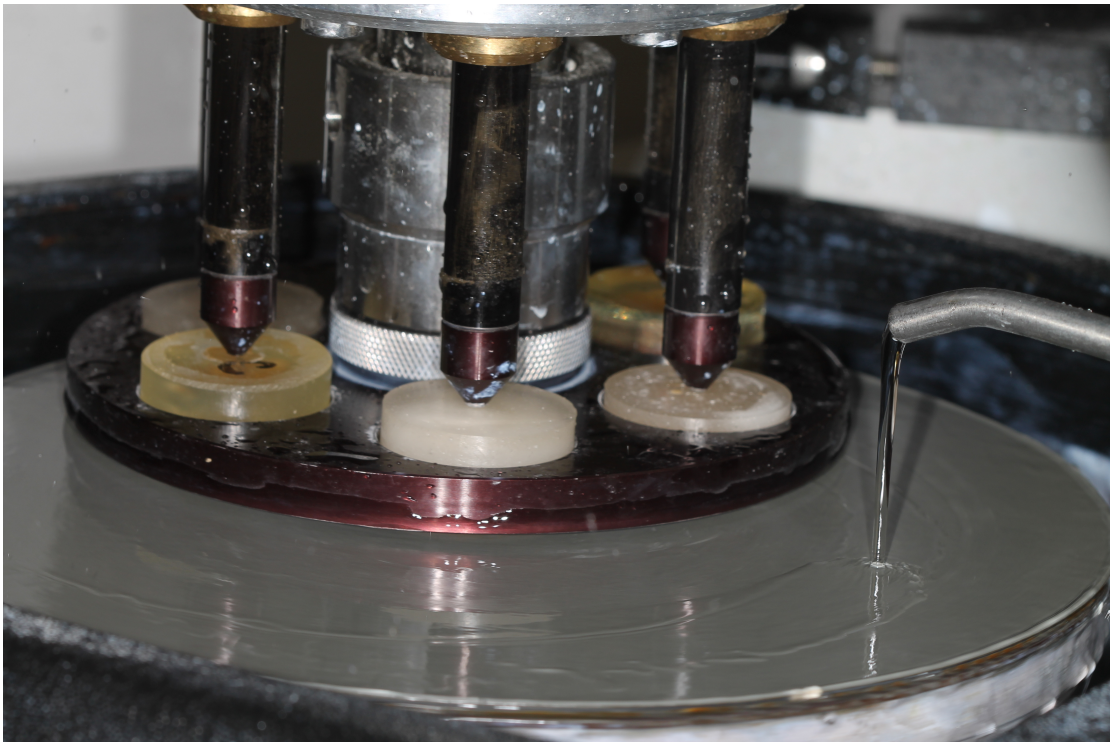


Figure 3: A specimen attached to sample holder using sticky wax before polishing

(a)



(b)

Figure 4: (a, b) Grinding machine used for polishing specimens

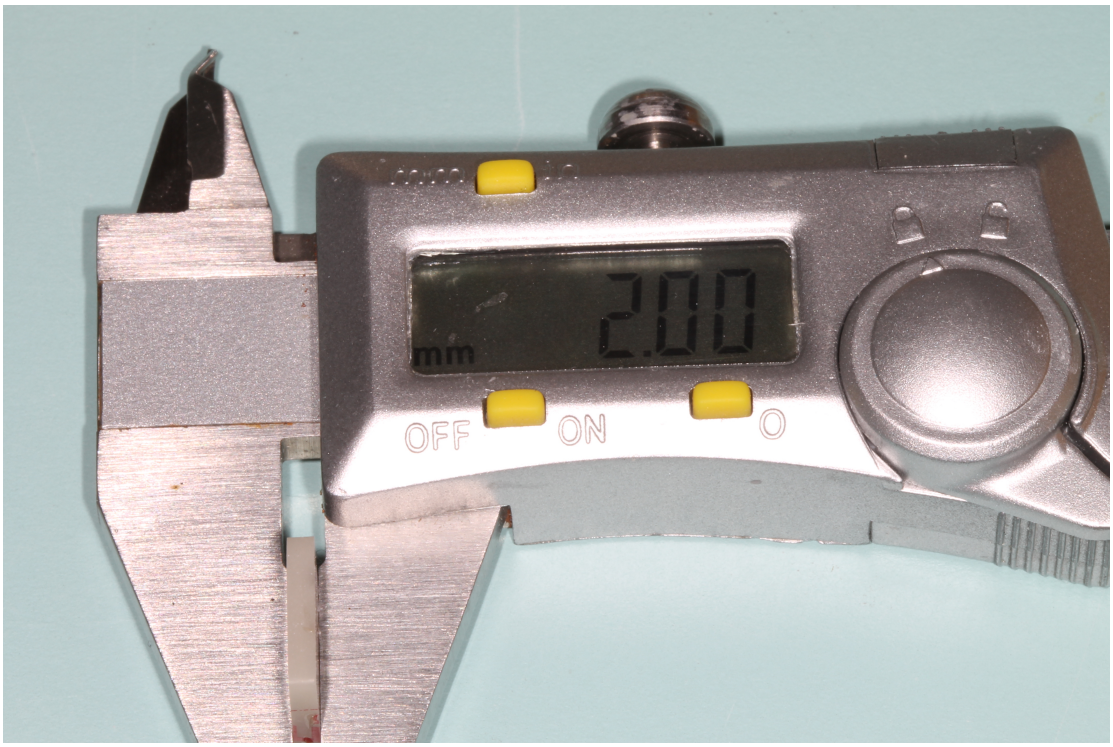


Figure 5: Digital caliper used to verify the thickness of specimens

Figure 6: Specimens immersed in petri dishes filled with 20 ml of 5% hydrochloric acid HCl and placed into incubator at 37°C.

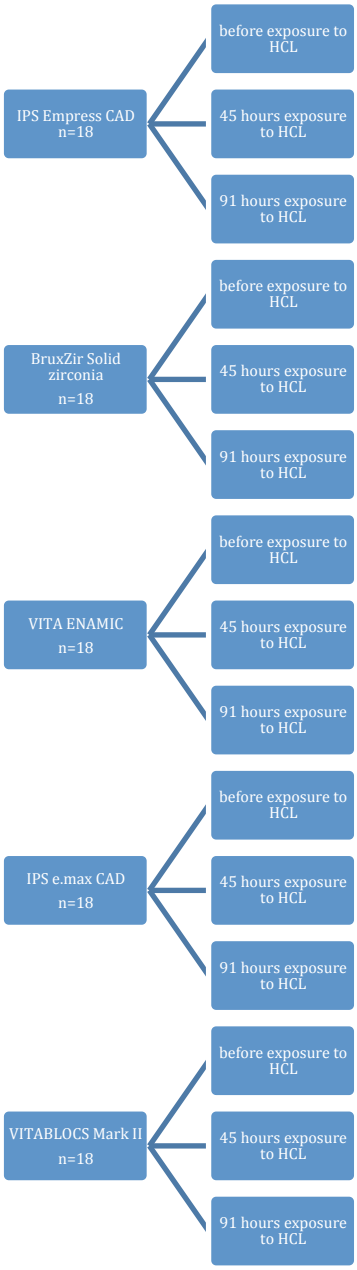


Figure 7: Study groups.

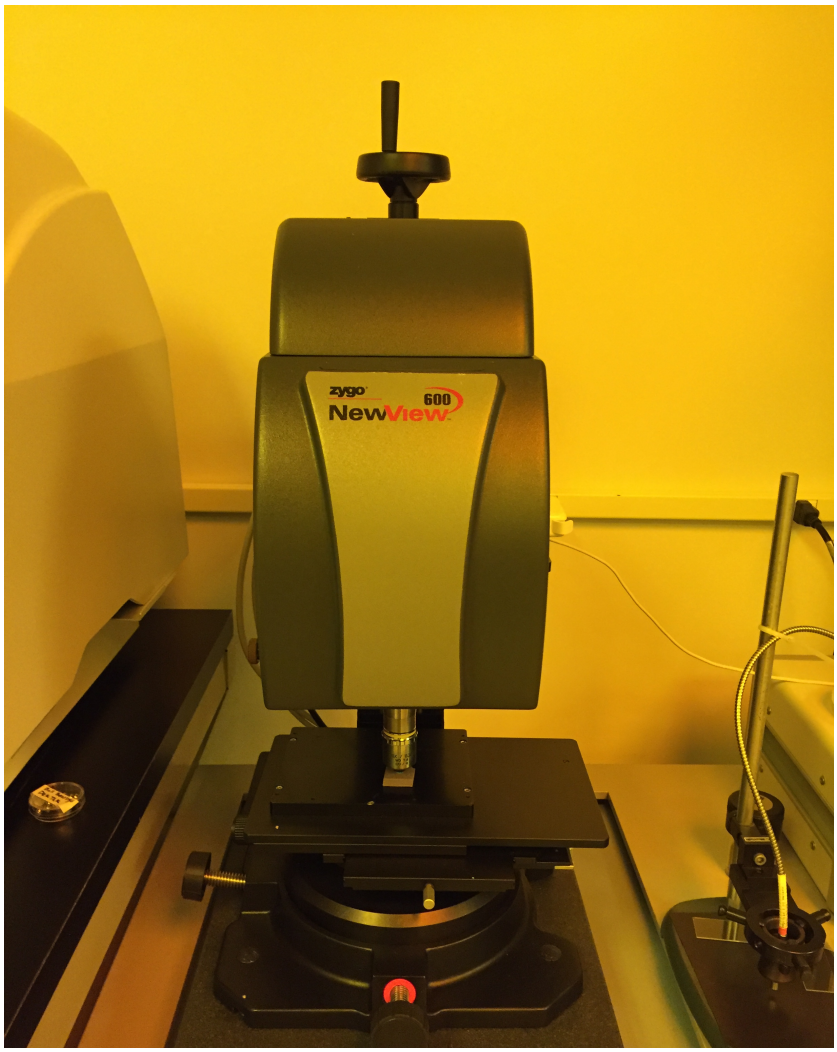
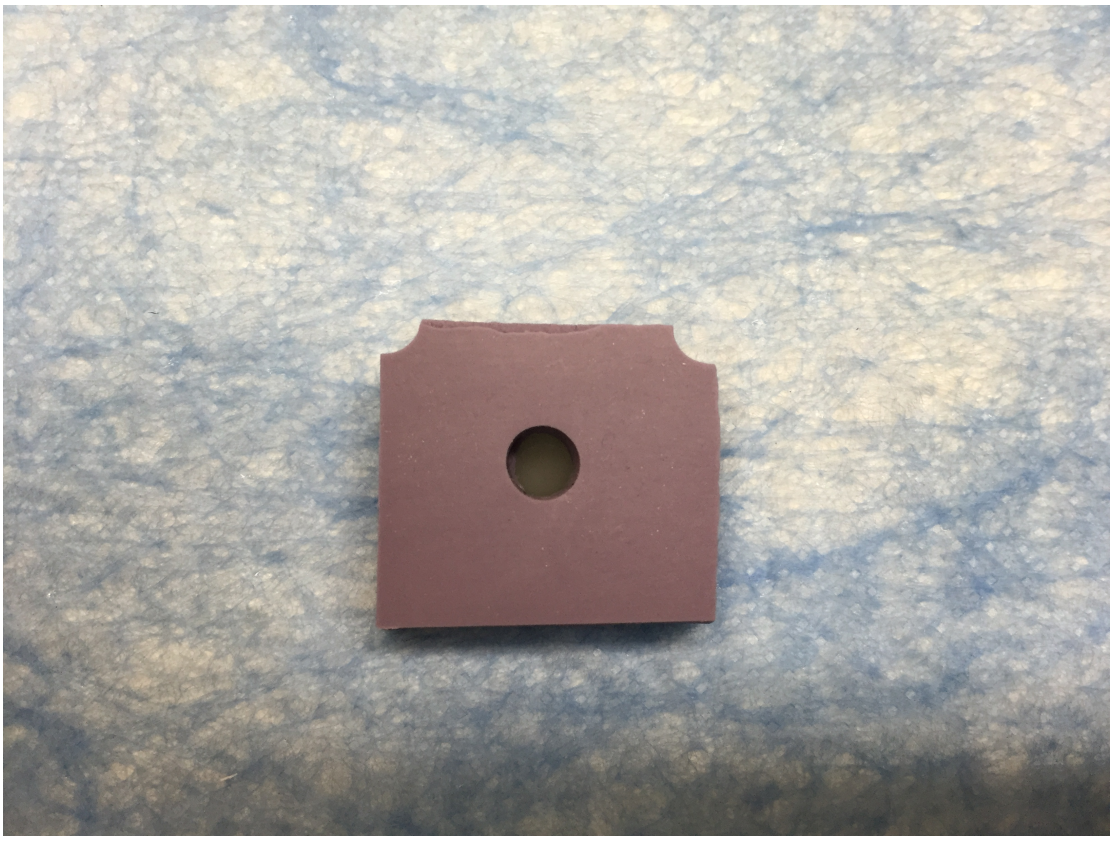
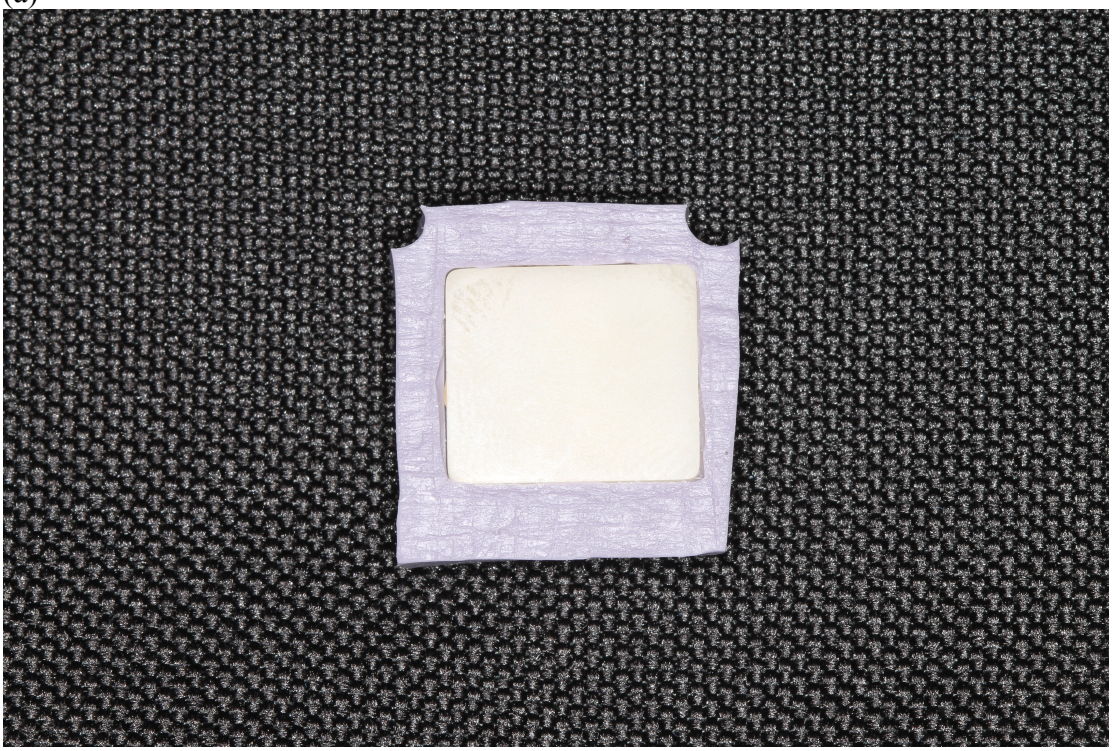


Figure 8: 3-D Optical interferometer used to measure surface roughness of specimens.



(a)



(b)

Figure 9: (a, b) Silicon mold was made for each material group with 5 mm hole created in the center of the silicon mold

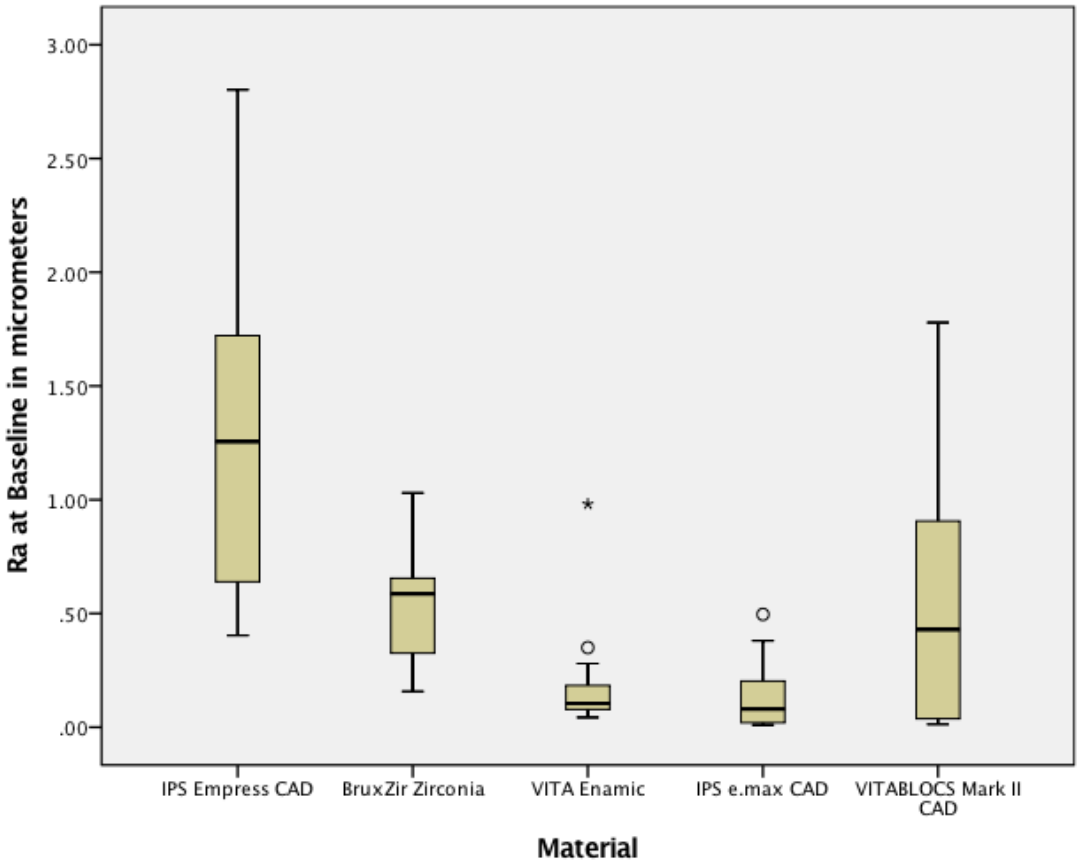


Figure 10: Boxplots of surface roughness (Ra) for each material group at baseline in µm.

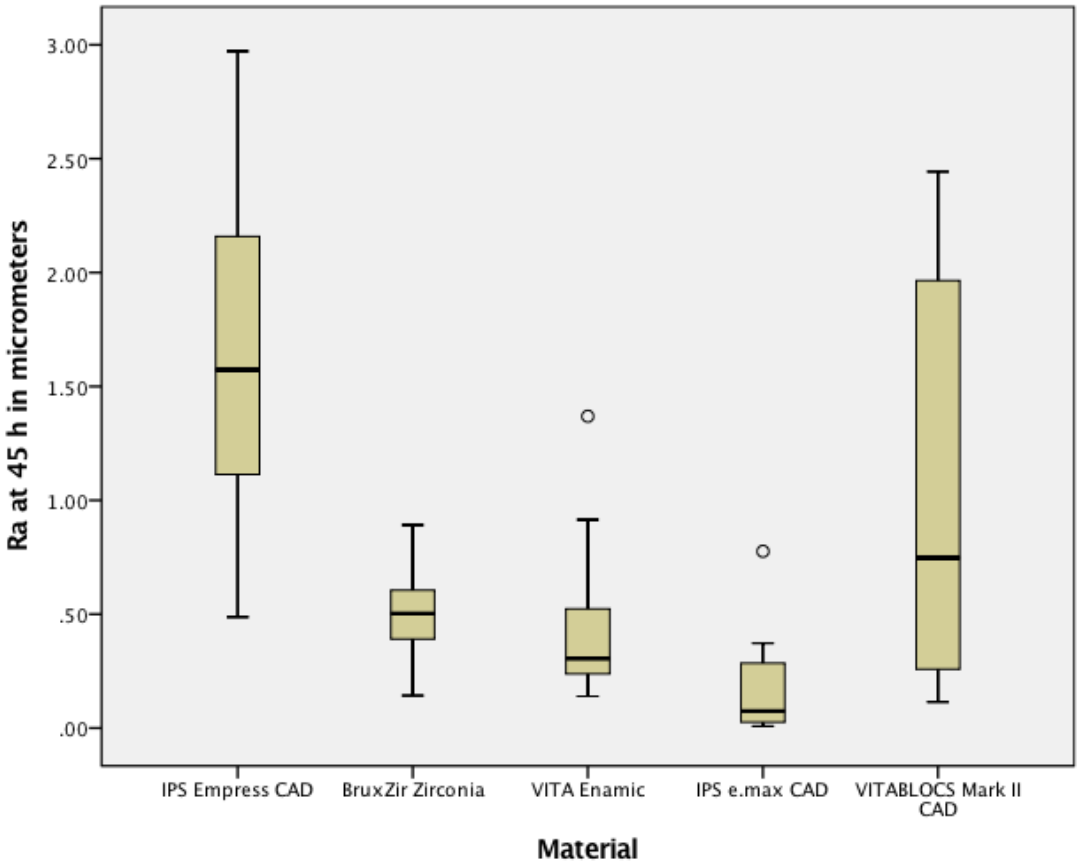


Figure 11: Boxplots of surface roughness (Ra) for each material group at 45 hours in μm.

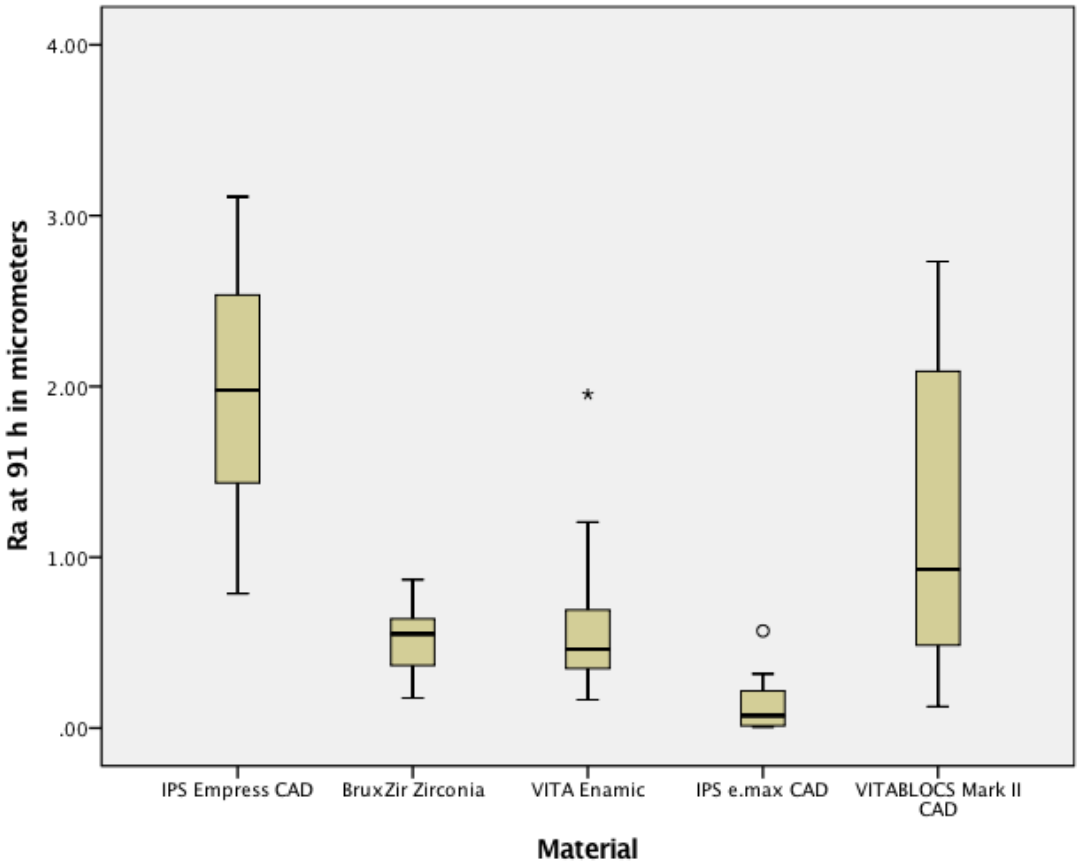


Figure 12: Boxplots of surface roughness (Ra) for each material group at 91 hours in μm.

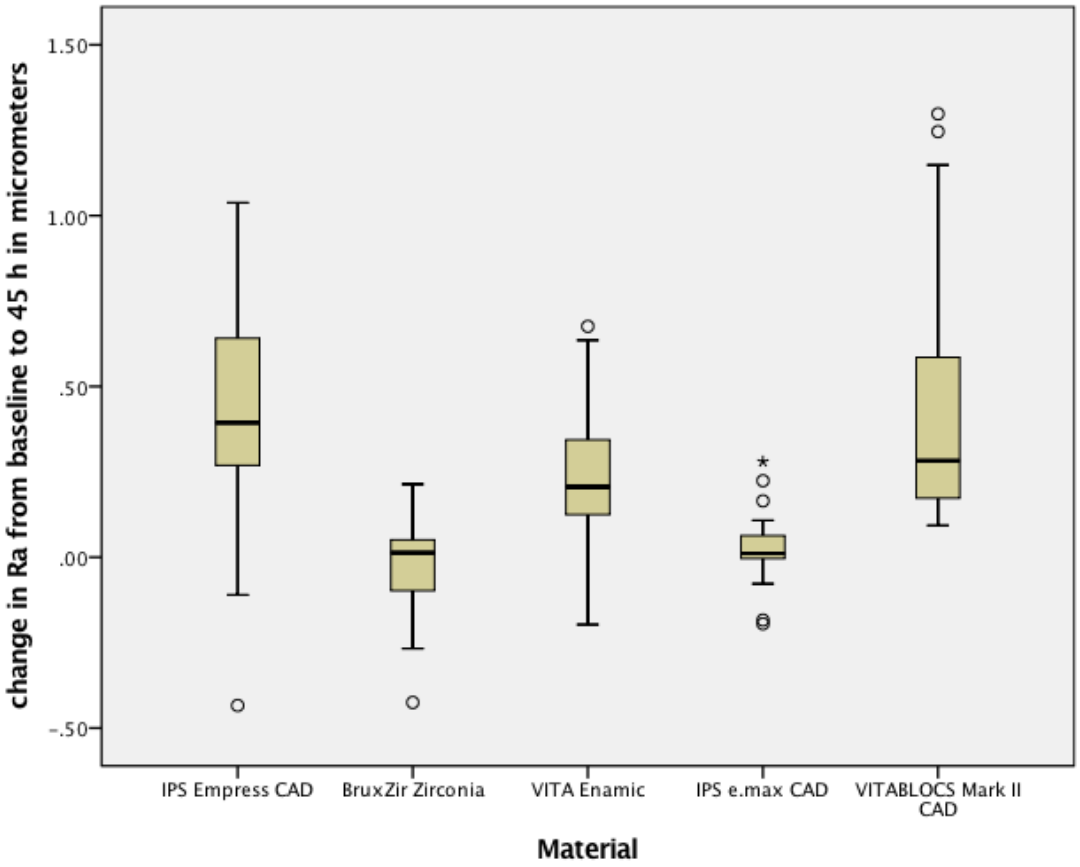


Figure 13: Boxplots of surface roughness change for each material group between baseline and 45 hours in µm.

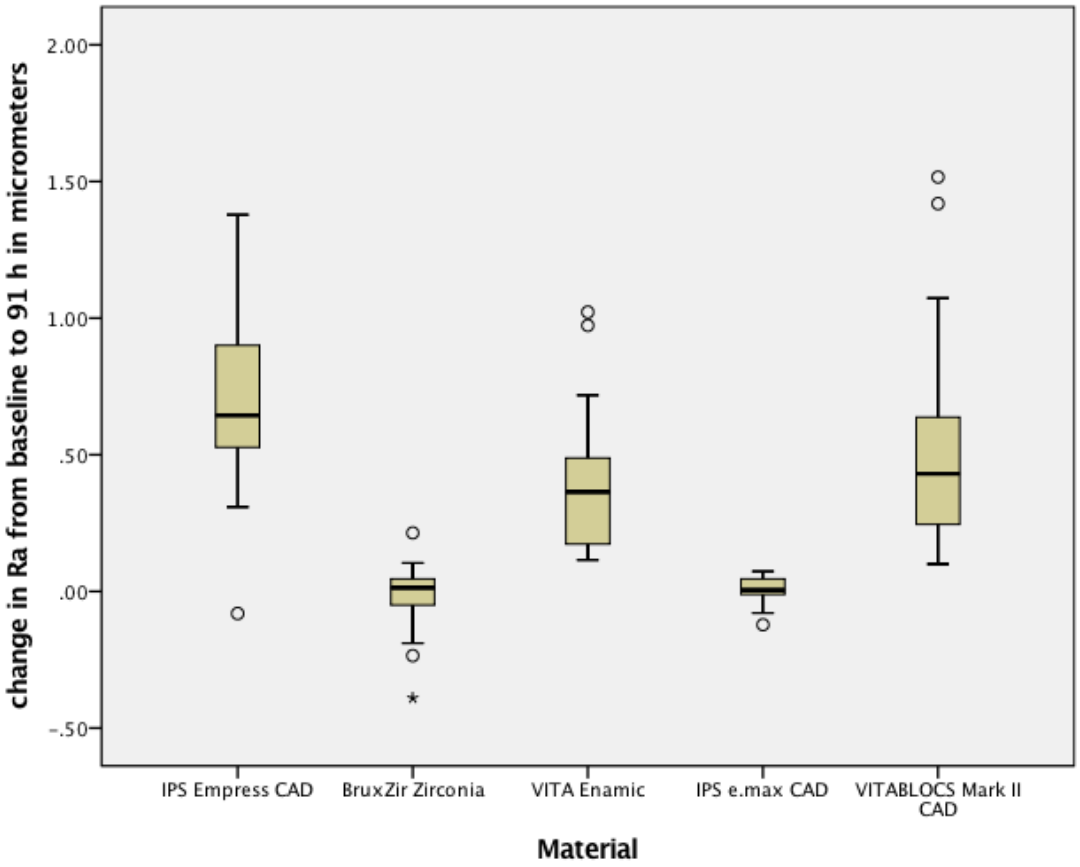
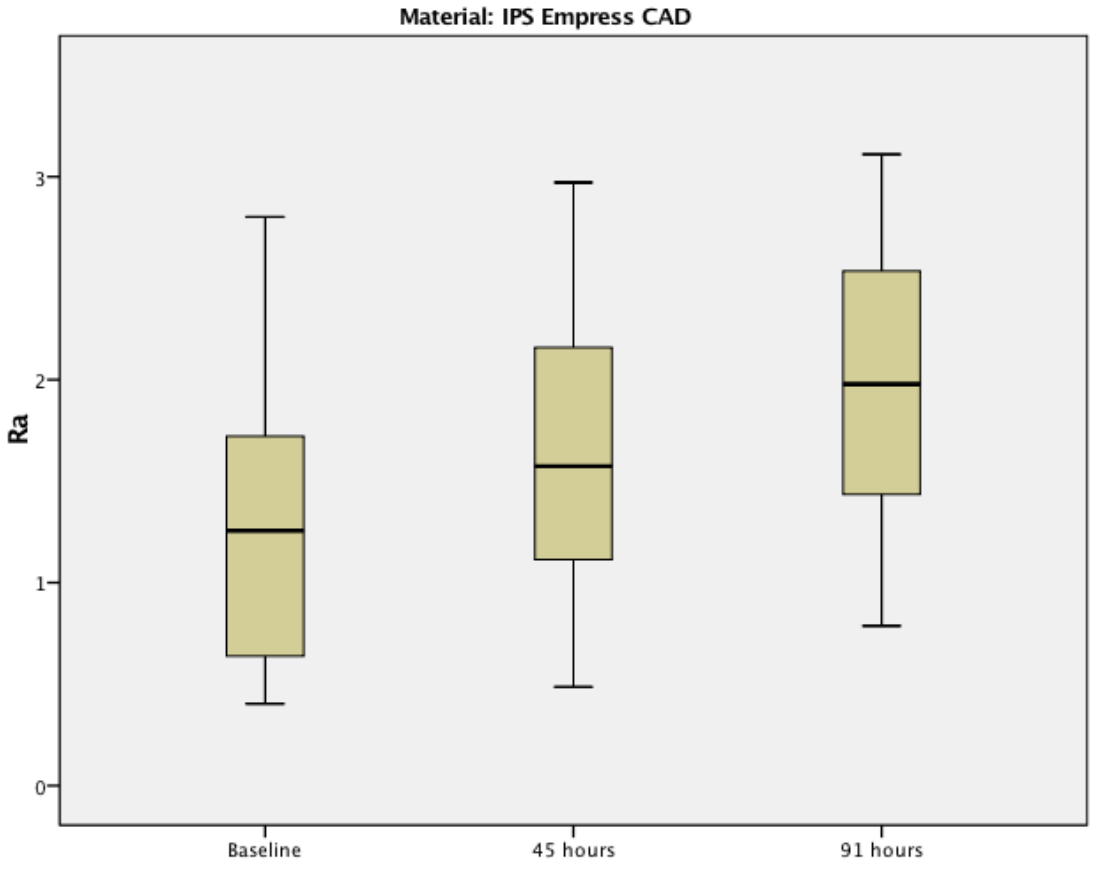
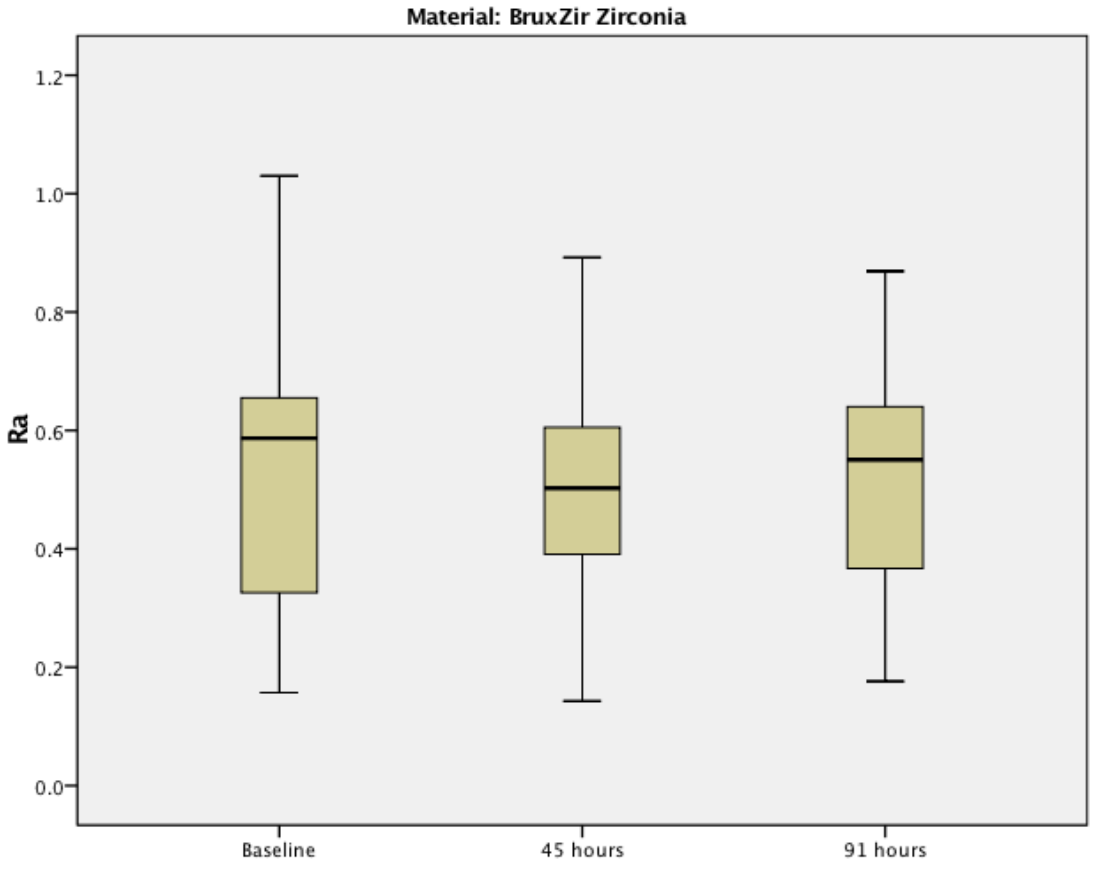


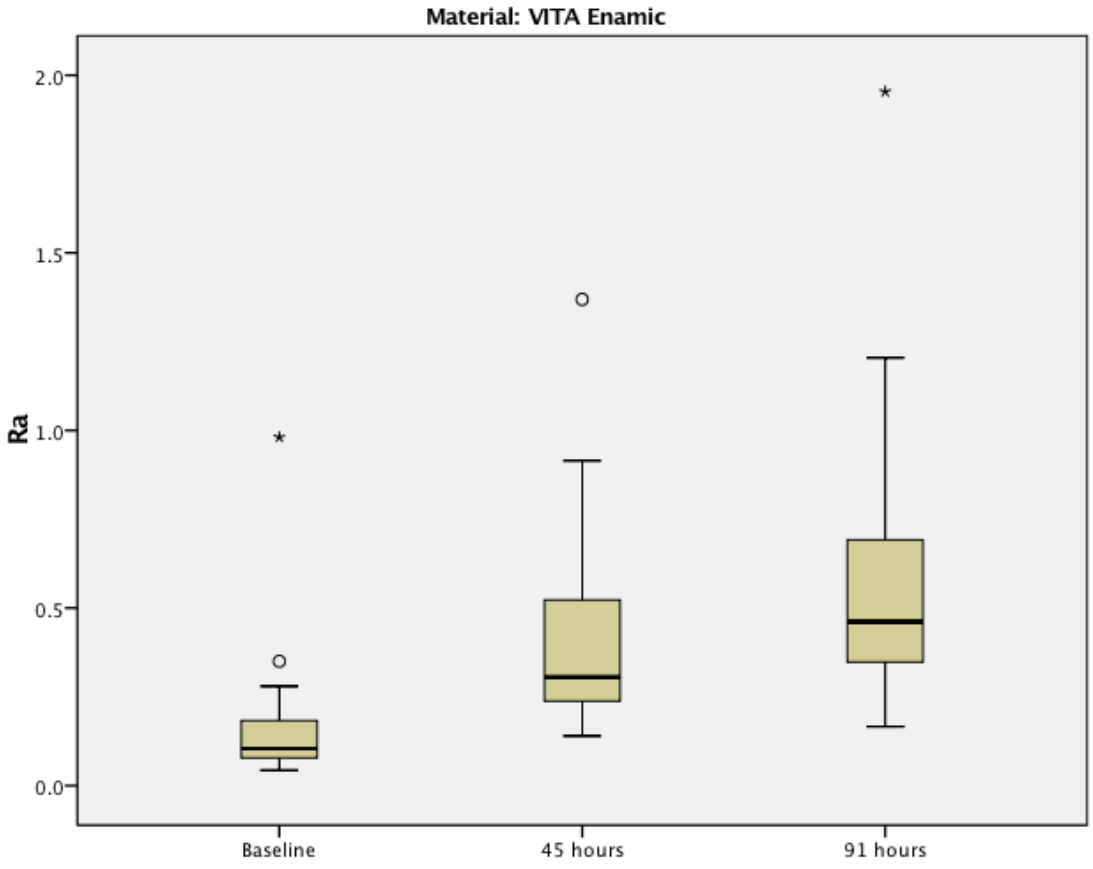
Figure 14: Boxplots of surface roughness change for each material group between baseline and 91 hours in µm.



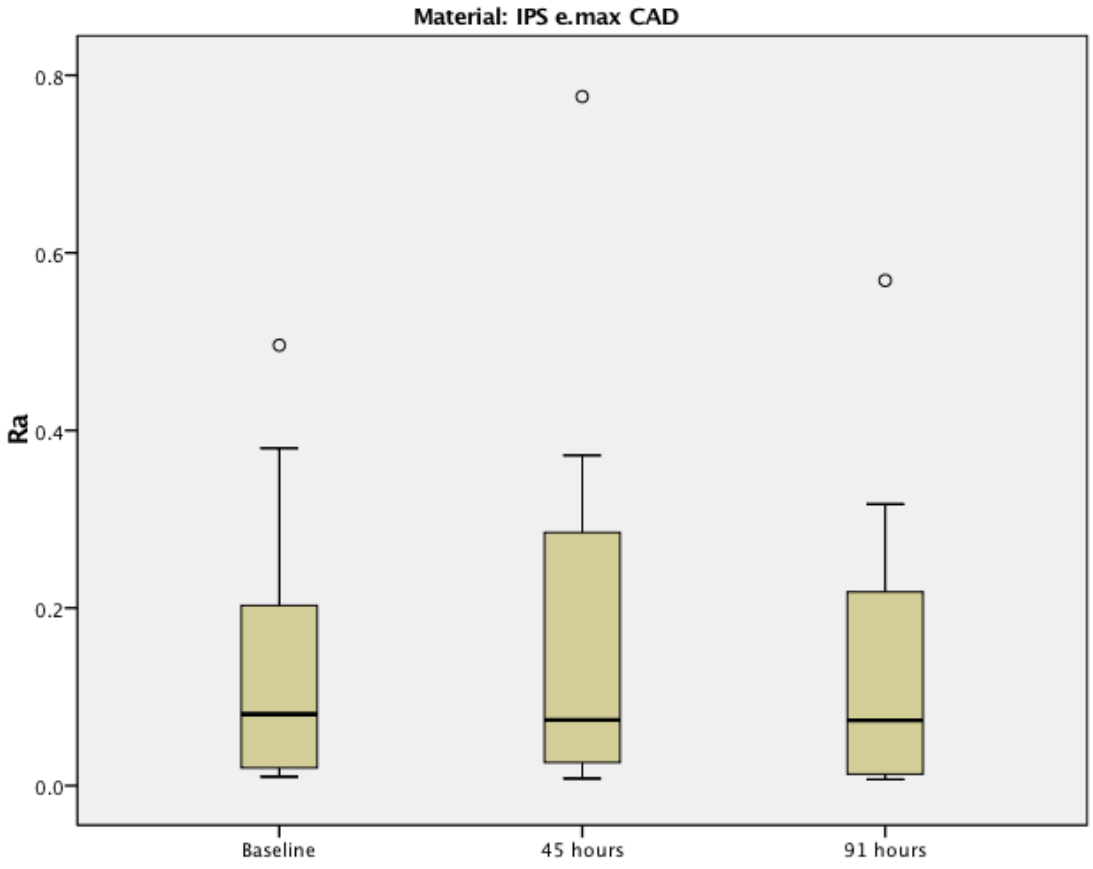
(a)


Material: BruxZir Zirconia
Ra
1.2
1.0
0.8
0.6
0.4
0.2
0.0
Baseline
45 hours
91 hours

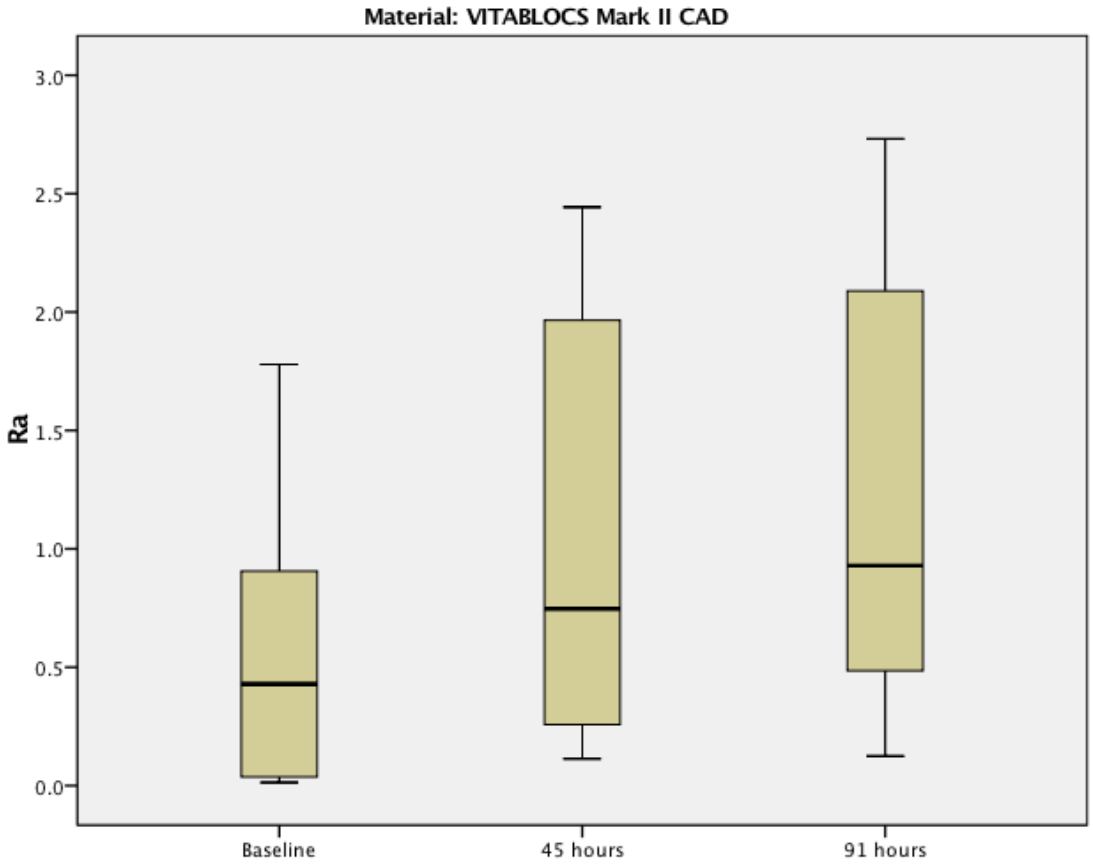
(b)


Material: VITA Enamic
Ra
2.0
1.5
1.0
0.5
0.0
Baseline
45 hours
91 hours

(c)

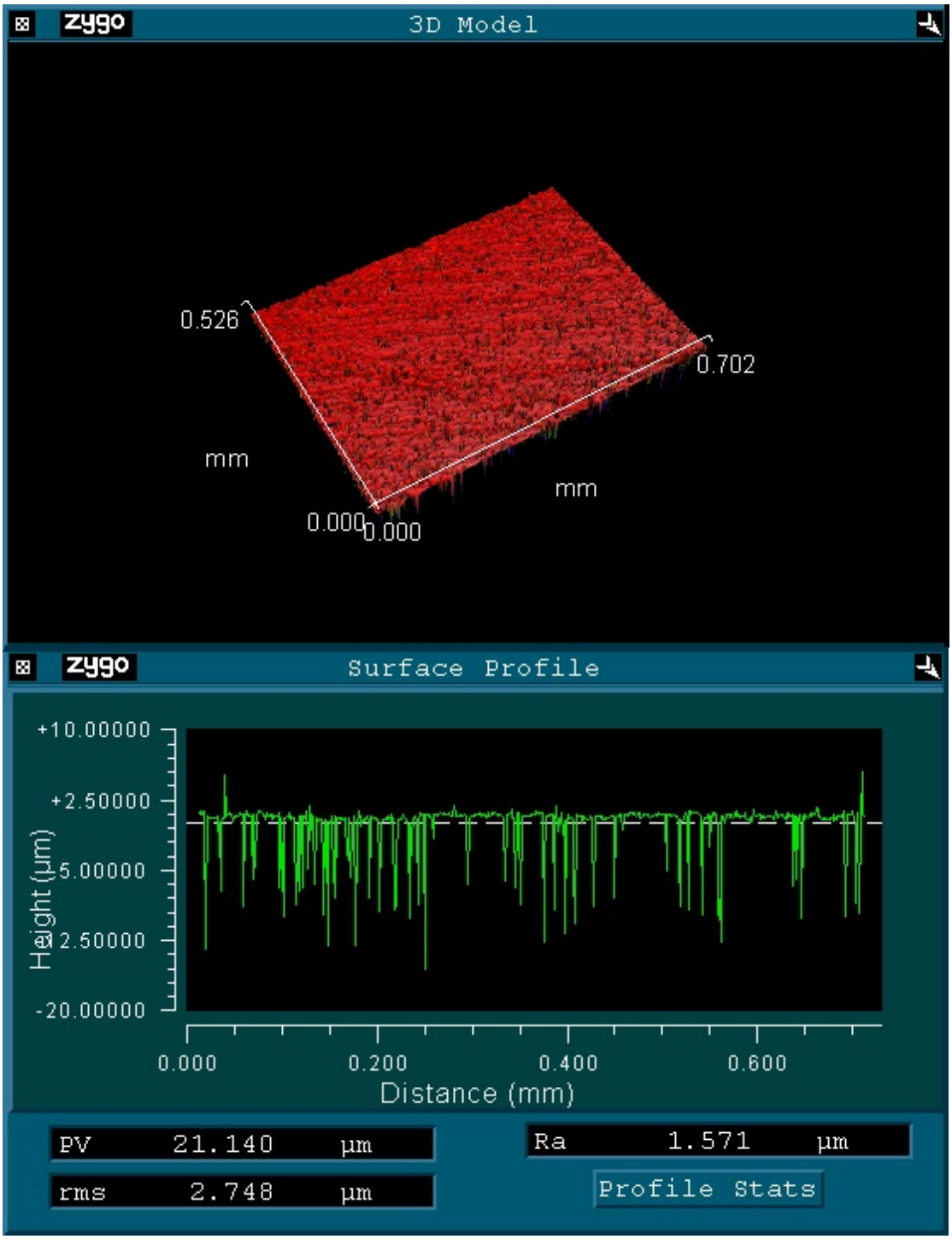

Material: IPS e.max CAD
Ra
0.8
0.6
0.4
0.2
0.0
Baseline
45 hours
91 hours

(d)

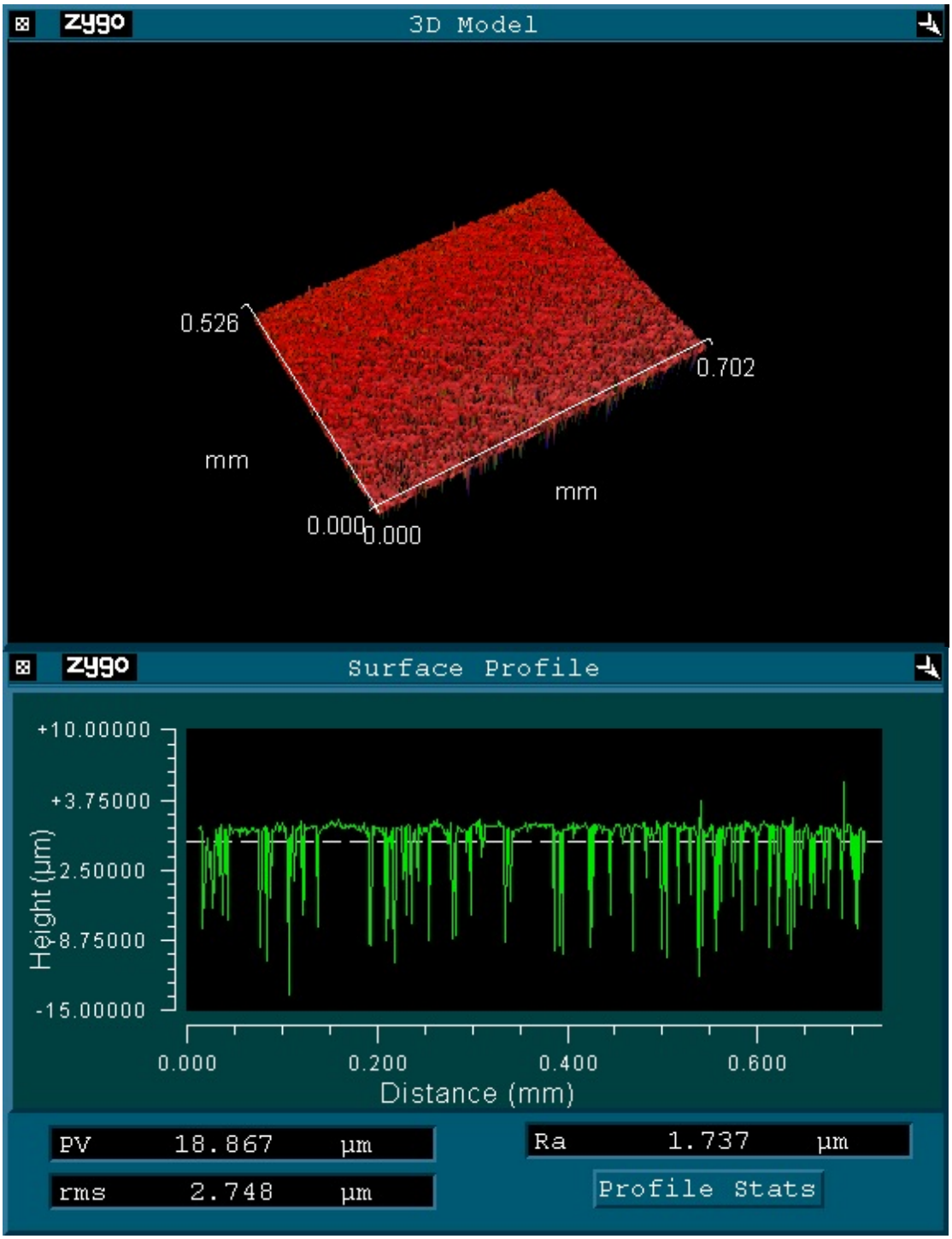


(e)

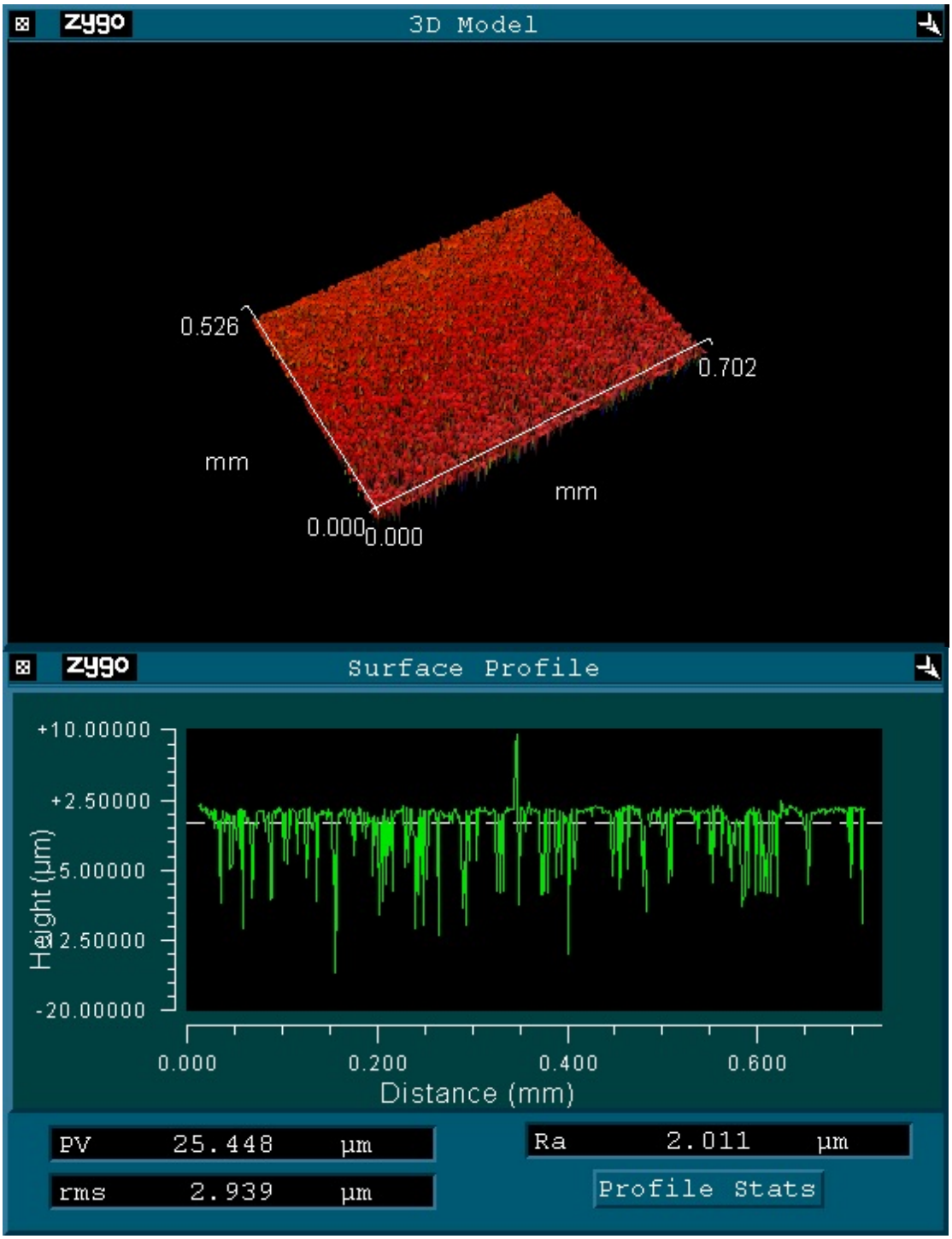
Figure 15: Boxplots of surface roughness (Ra) at baseline, 45 hours, 91 hours in µm (a) IPS Empress CAD (b) BruxZir Solid zirconia (c) VITA ENAMIC (d) IPS e.max CAD (e) VITABLOCS Mark II.


zygo
3D Model
0.526
0.702
mm
mm
0.000
0.000
zygo
Surface Profile
+10.00000
+2.50000
-5.00000
-12.50000
-20.00000
Height (µm)
0.000
0.200
0.400
0.600
Distance (mm)
PV 21.140 µm
rms 2.748 µm
Ra 1.571 µm
Profile Stats

(a)

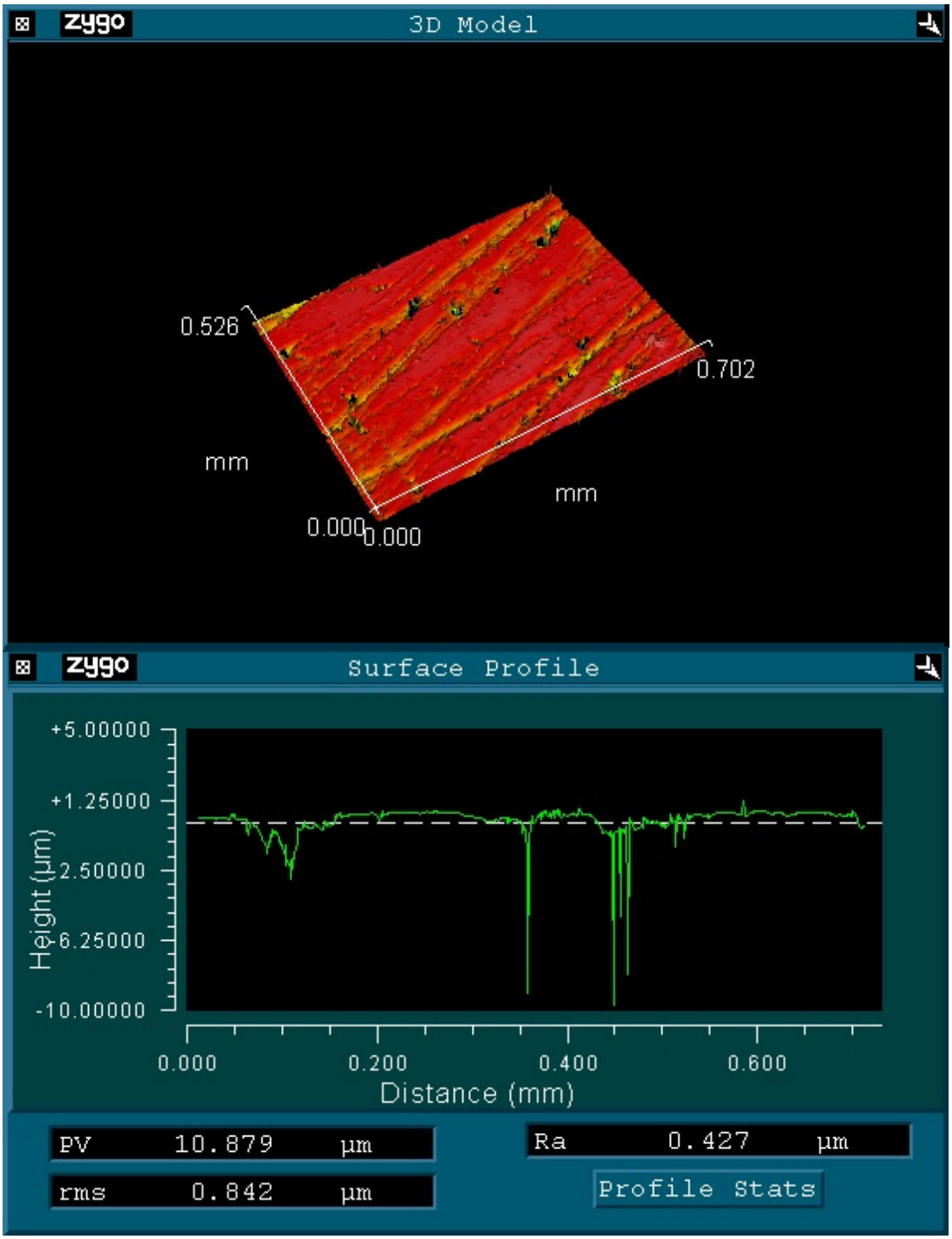

zygo
3D Model
0.526
mm
0.000
0.000
mm
0.702
zygo
Surface Profile
+10.00000
+3.75000
-2.50000
-8.75000
-15.00000
Height (µm)
0.000
0.200
0.400
0.600
Distance (mm)
PV 18.867 µm
rms 2.748 µm
Ra 1.737 µm
Profile Stats

(b)

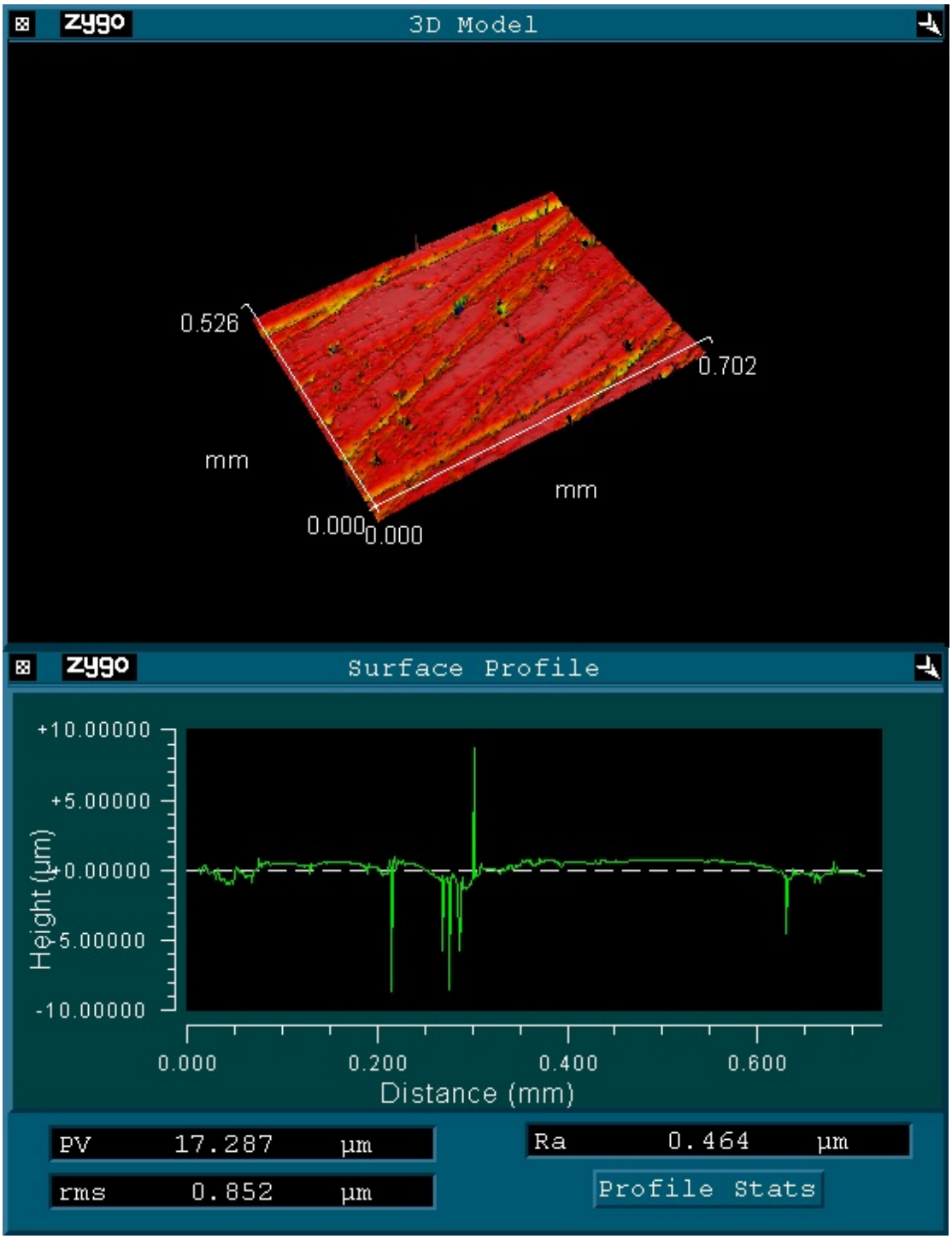


(c)

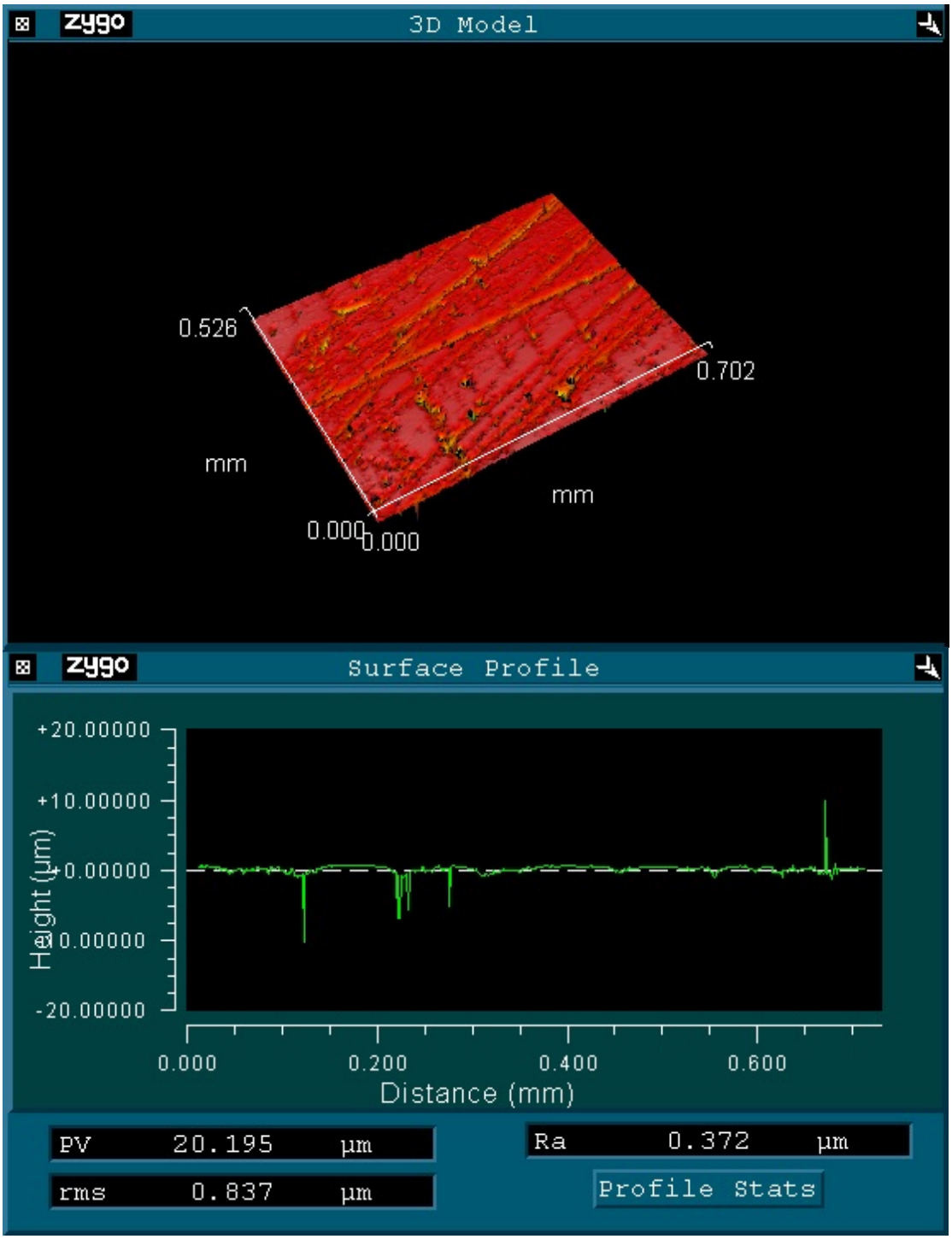
Figure 16: 3-D models and surface profiles of IPS EMPRESS CAD at baseline (a), 45 hours (b), and 91 hours (c).


zygo
3D Model
0.526
0.702
mm
mm
0.000
0.000
zygo
Surface Profile
+5.00000
+1.25000
-2.50000
-6.25000
-10.00000
Height (µm)
0.000
0.200
0.400
0.600
Distance (mm)
PV 10.879 µm
rms 0.842 µm
Ra 0.427 µm
Profile Stats

(a)

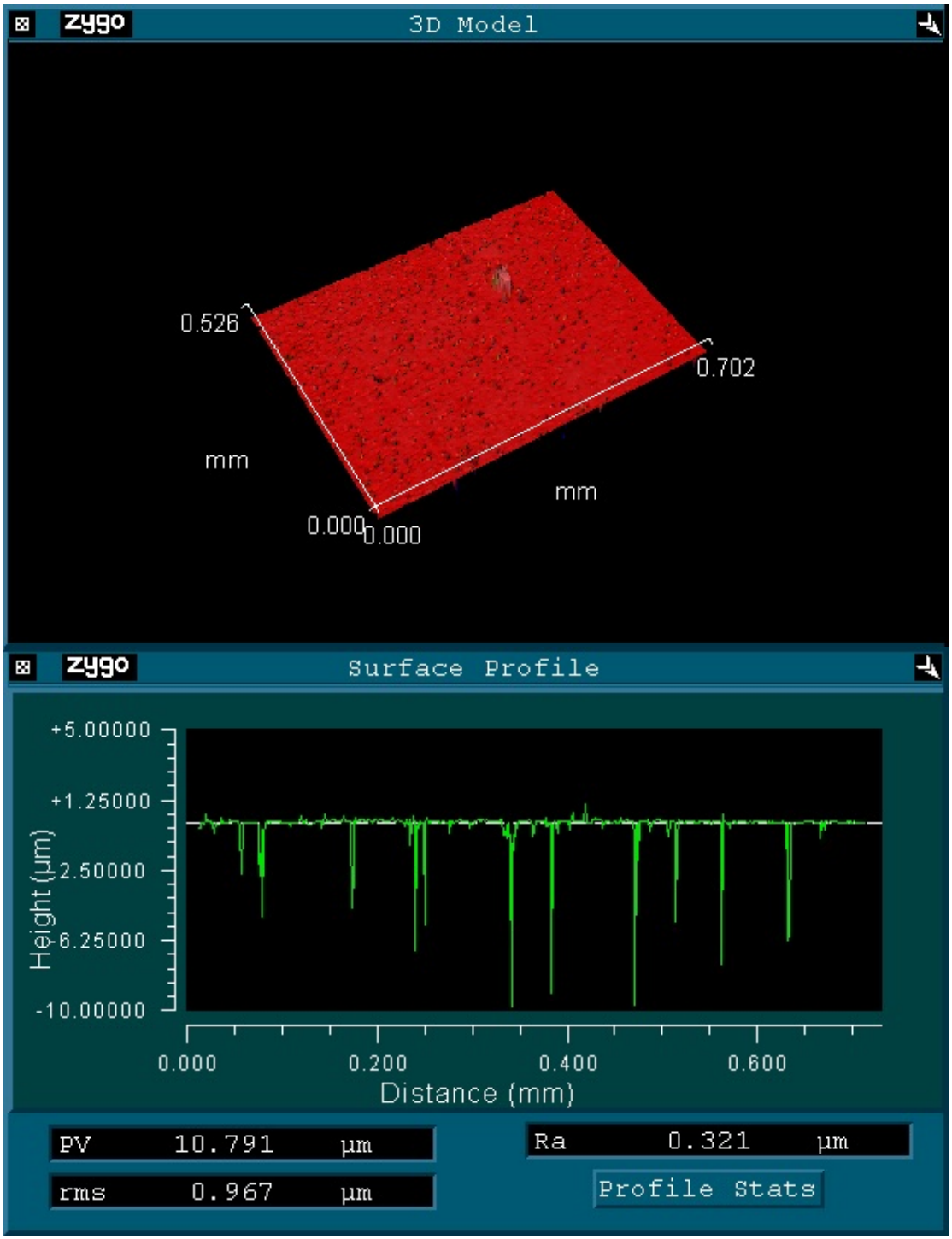

zygo
3D Model
0.526
mm
0.000
0.000
mm
0.702
zygo
Surface Profile
+10.00000
+5.00000
+0.00000
-5.00000
-10.00000
Height (µm)
0.000
0.200
0.400
0.600
Distance (mm)
PV 17.287 µm
rms 0.852 µm
Ra 0.464 µm
Profile Stats

(b)

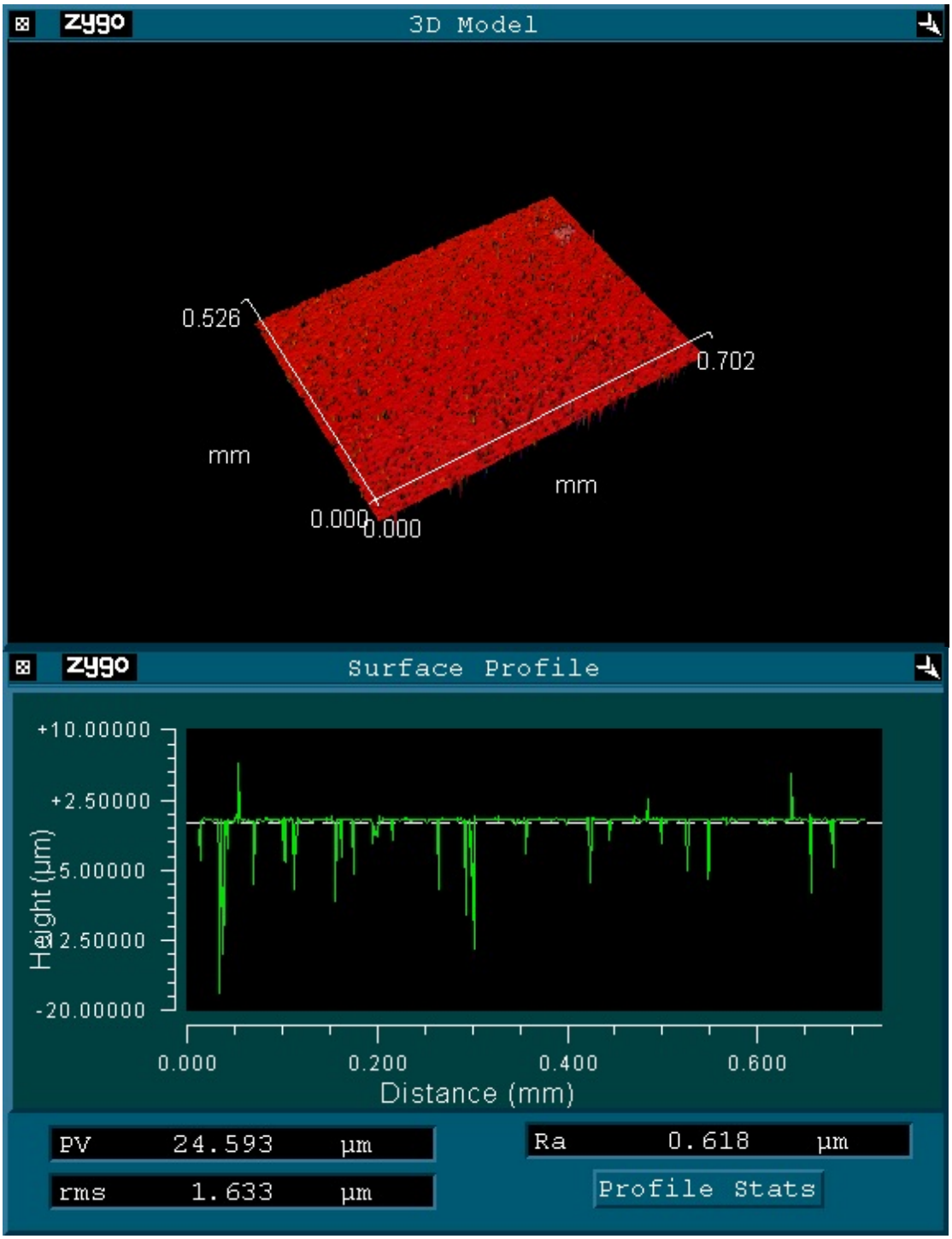


(c)

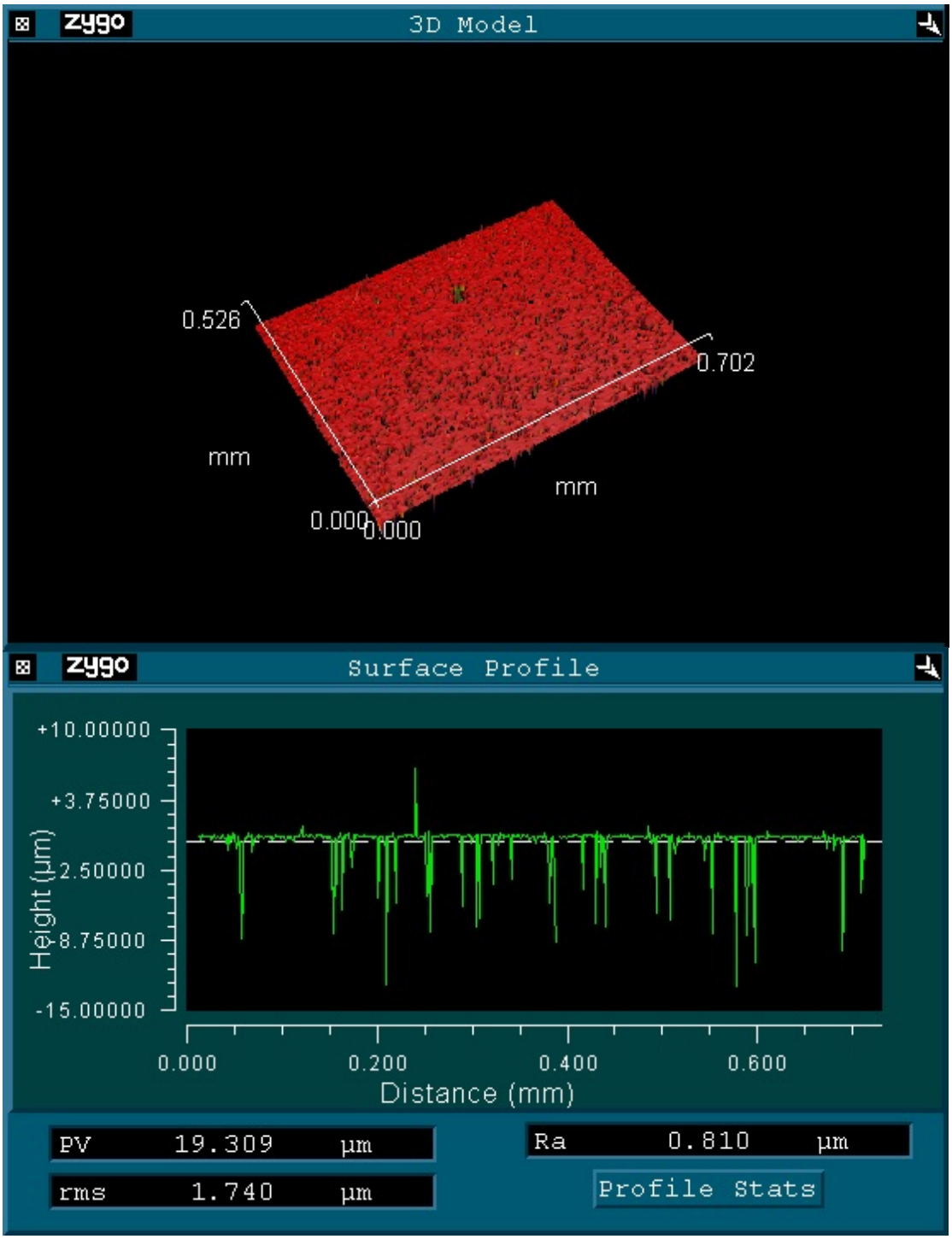
Figure 17: 3-D models and surface profiles of BroxZir Solid zirconia at baseline (a), 45 hours (b), and 91 hours (c).


zygo
3D Model
0.526
0.702
mm
mm
0.000
0.000
zygo
Surface Profile
+5.00000
+1.25000
-2.50000
-6.25000
-10.00000
Height (µm)
0.000
0.200
0.400
0.600
Distance (mm)
PV 10.791 µm
rms 0.967 µm
Ra 0.321 µm
Profile Stats

(a)

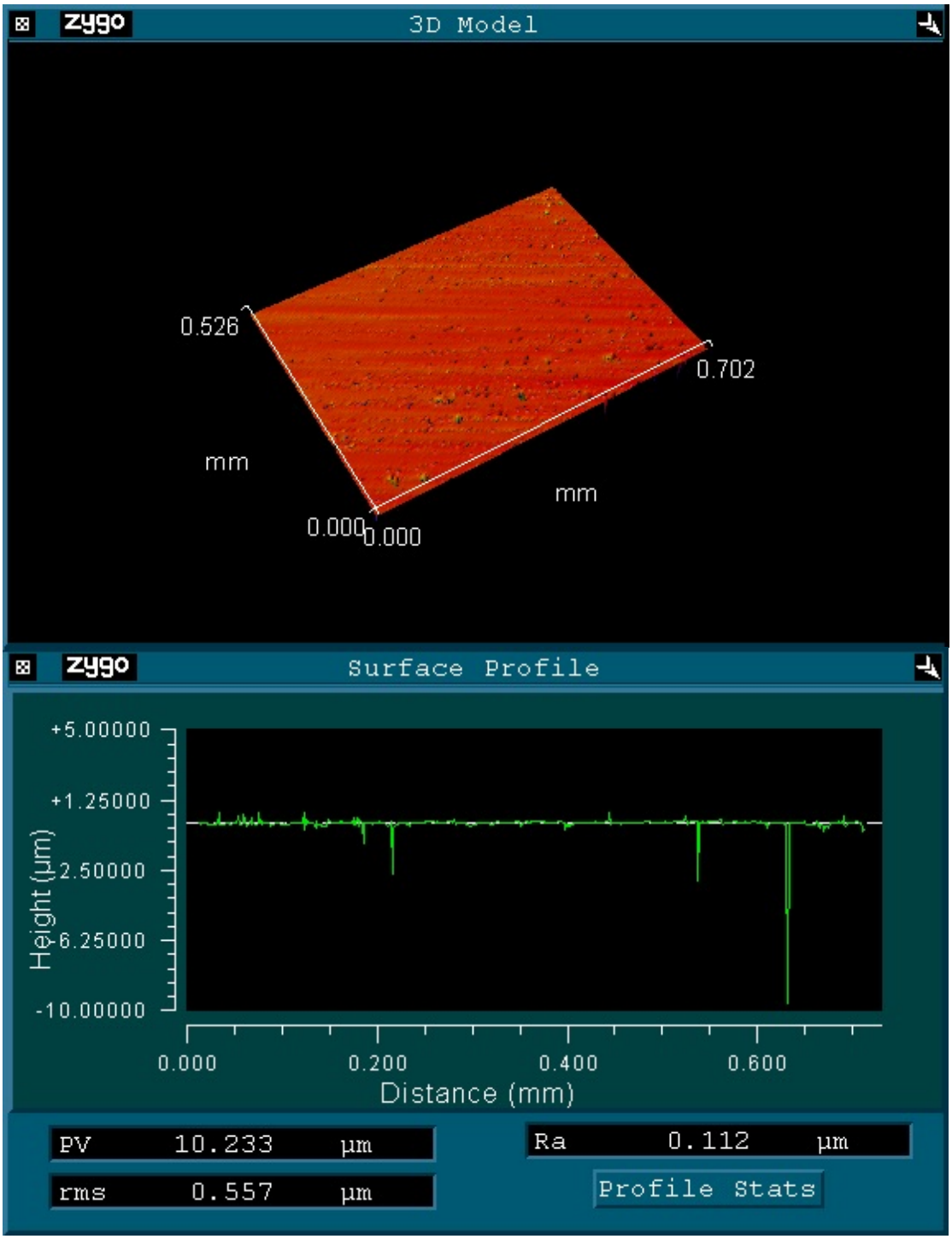

zygo
3D Model
0.526
0.702
mm
mm
0.000
0.000
zygo
Surface Profile
+10.00000
+2.50000
-5.00000
-12.50000
-20.00000
Height (µm)
0.000
0.200
0.400
0.600
Distance (mm)
PV 24.593 µm
rms 1.633 µm
Ra 0.618 µm
Profile Stats

(b)

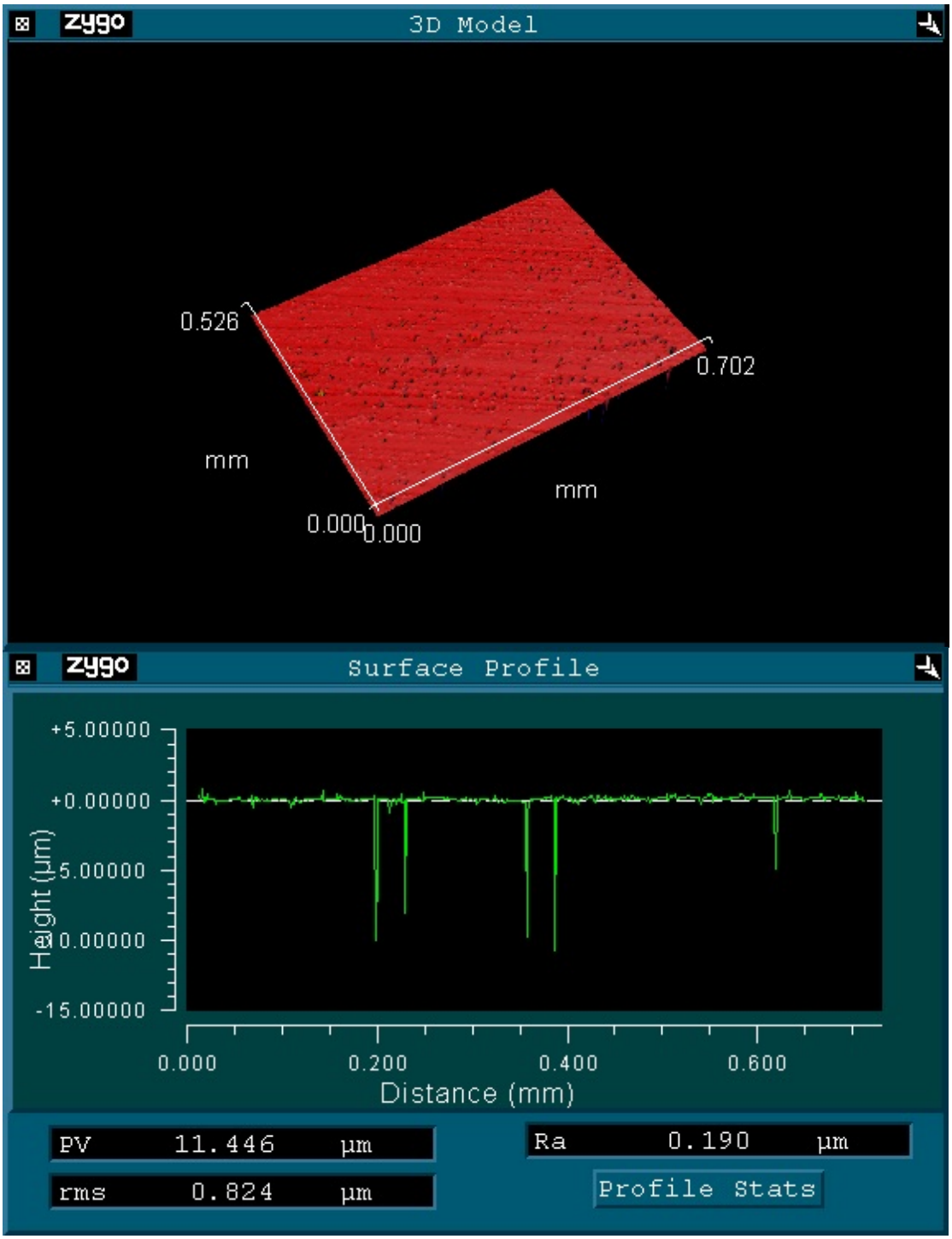


(c)

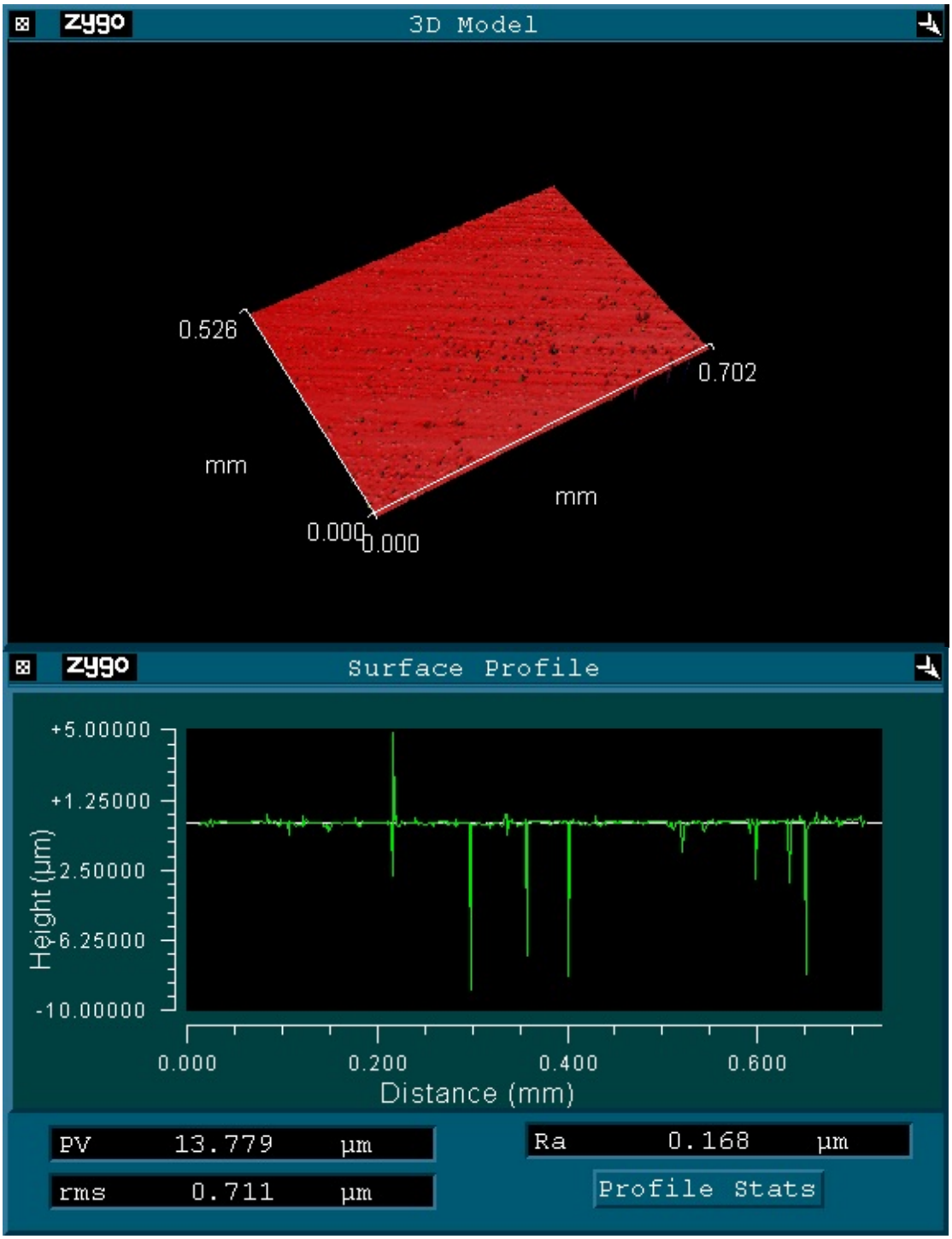
Figure 18: 3-D models and surface profiles of IPS VITA ENAMIC at baseline (a), 45 hours (b), and 91 hours (c).


zygo
3D Model
0.526
0.702
mm
mm
0.000
0.000
zygo
Surface Profile
+5.00000
+1.25000
-2.50000
-6.25000
-10.00000
Height (µm)
0.000
0.200
0.400
0.600
Distance (mm)
PV 10.233 µm
rms 0.557 µm
Ra 0.112 µm
Profile Stats

(a)

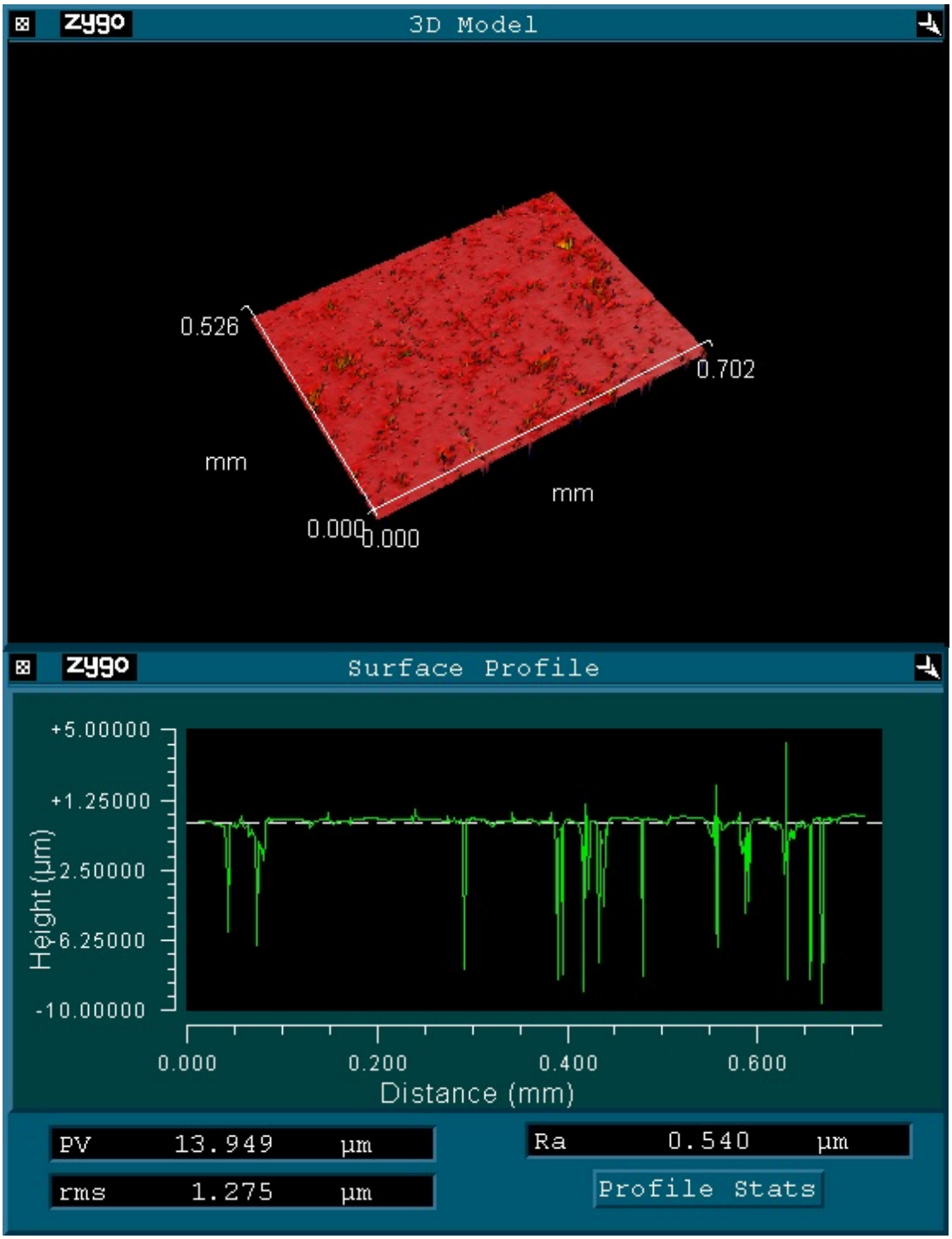

zygo
3D Model
0.526
0.702
mm
mm
0.000
0.000
zygo
Surface Profile
+5.00000
+0.00000
-5.00000
-10.00000
-15.00000
Height (µm)
0.000
0.200
0.400
0.600
Distance (mm)
PV 11.446 µm
rms 0.824 µm
Ra 0.190 µm
Profile Stats

(b)

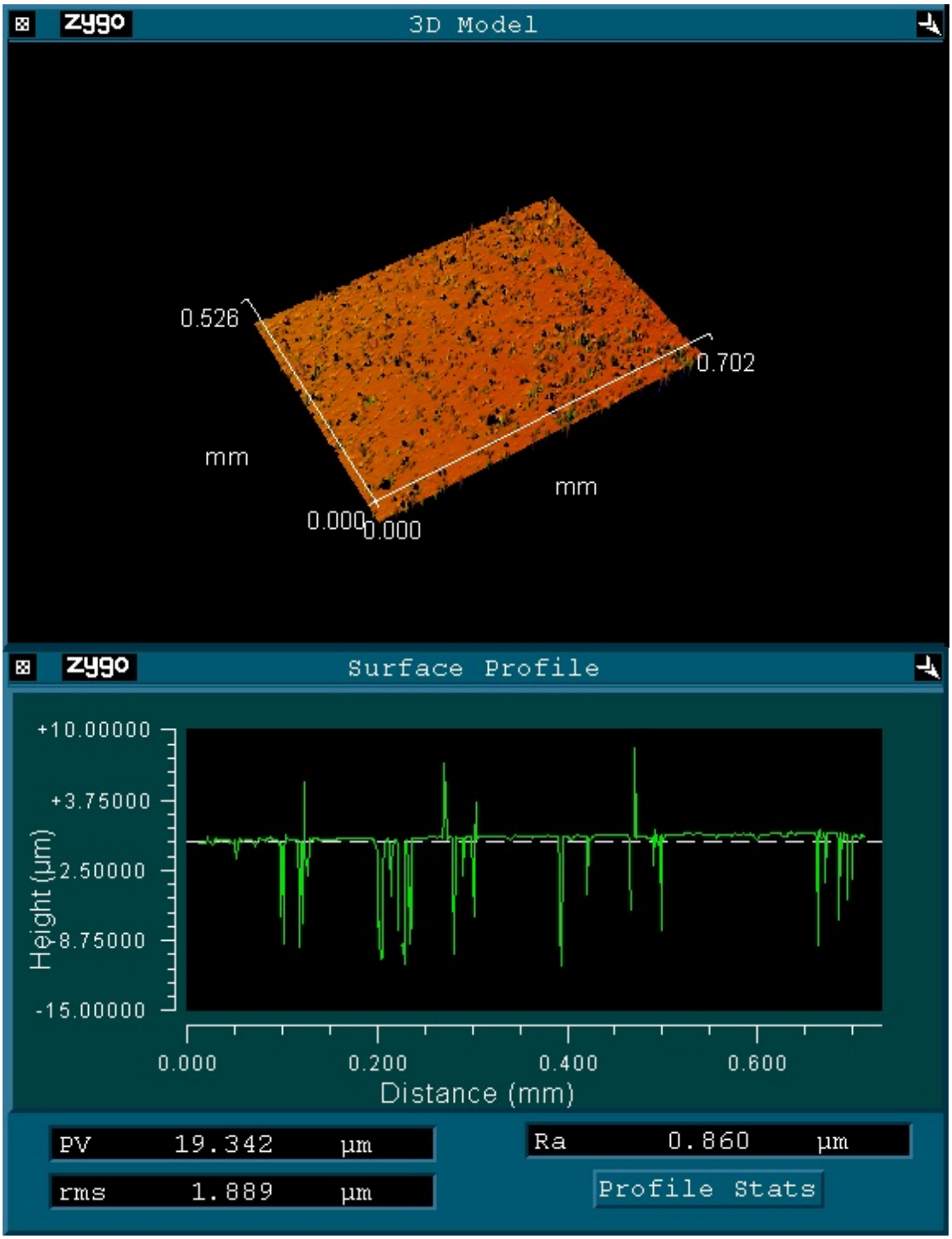


(c)

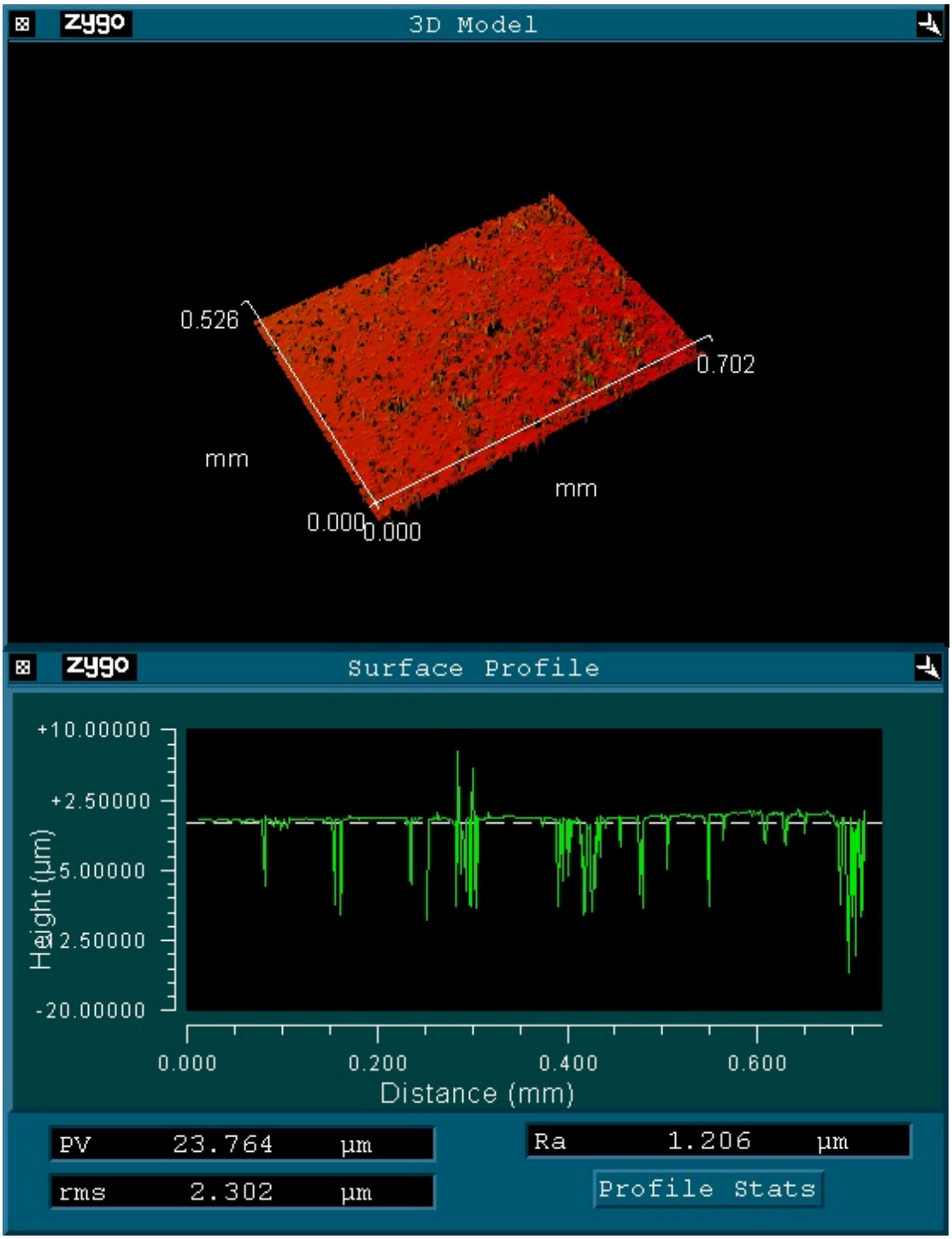
Figure 19: 3-D models and surface profiles of IPS e.max CAD at baseline (a), 45 hours (b), and 91 hours (c).


zygo
3D Model
0.526
mm
0.000
0.000
mm
0.702
zygo
Surface Profile
+5.00000
+1.25000
-2.50000
-6.25000
-10.00000
Height (µm)
0.000
0.200
0.400
0.600
Distance (mm)
PV 13.949 µm
rms 1.275 µm
Ra 0.540 µm
Profile Stats

(a)


zygo
3D Model
0.526
mm
0.000
0.000
mm
0.702
zygo
Surface Profile
+10.00000
+3.75000
-2.50000
-8.75000
-15.00000
Height (µm)
0.000
0.200
0.400
0.600
Distance (mm)
PV 19.342 µm
rms 1.889 µm
Ra 0.860 µm
Profile Stats

(b)



(c)

Figure 20: 3-D models and surface profiles of VITABLOCS Mark II at baseline (a), 45 hours (b), and 91 hours (c).