

ORIGINAL ARTICLE

Effect of engraving speeds of CO₂ laser irradiation on in-ceram alumina roughness: a pilot studyBAHADIR ERSU¹, ORKUN ERSOY², BULEM YUZUGULLU³ & SENAY CANAY¹

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Abstract

Objectives. The aim of the study was to determine the effect of CO₂ laser on surface roughness of In-Ceram-Alumina-ceramic. **Materials and methods.** Four aluminum-oxide ceramic disc specimens were prepared of In-Ceram Alumina. Discs received CO₂ laser irradiation with different engraving speeds (100, 400, 600 and 800 mm/min) as a surface treatment. The roughness of the surfaces was measured on digital elevation models reconstructed from stereoscopic images acquired by scanning-electron-microscope. Surface roughness data were analyzed with One-Way-Analysis-of-Variance at a significance level of $p < 0.05$. **Results.** There was no significant difference between the roughness values ($p = 0.82$). Due to higher laser durations, partial melting signs were observed on the surfaces. Tearing, smearing and swelling occurred on melted surfaces. Swelling accompanying melting increased the surface roughness, while laser power was fixed and different laser engraving speeds were applied. **Conclusion.** Although different laser irradiation speeds did not affect the roughness of ceramic surfaces, swelling was observed which led to changes on surfaces.

Key Words: CO₂ laser, digital elevation models, glass-infiltrated alumina-ceramic, scanning electron microscope, surface roughness

Introduction

Ceramics have been traditionally preferred as a dental restorative material because of their esthetic quality and excellent biocompatibility [1]. The mechanical properties of high content alumina based ceramics make them attractive as potential materials for all-ceramics restorations in high stress-bearing areas [2]. The performance and durability of high strength aluminum oxide ceramic restorations are dependent upon the adhesive assembly between the ceramic surface and the tooth substrate [3]. The internal surface of the ceramic restoration must be prepared to optimize micromechanical retention of the cement into the ceramic micro-roughness. Surface treatments of porcelain increase the surface area and create micro-porosities on the porcelain surface, enhancing the potential for mechanical retention of the cement [4].

In-Ceram Alumina is a glass-infiltrated aluminum-oxide ceramic and is widely used in metal-free restorations. These ceramics with high crystalline content (aluminum) have been shown to demonstrate clinical success rates higher than or comparable to feldspar-, leucite- and lithium disilicate-based ceramics [5]. The densely sintered high-purity alumina and glass infiltrated aluminum-oxide ceramic are resistant to hydrofluoric acid etching and airborne particle abrasion surface treatments [6,7]. Different conditioning systems such as Rocatec and Silicoater MD [8], the neodymium:yttrium-aluminium-garnet (Nd:YAG) laser [9,10] and carbon dioxide (CO₂) laser [5] have been proposed to provide roughness and promote micromechanical retention.

Since the development of ruby lasers [11], lasers have become widely used in medicine and dentistry. The CO₂ and ND:YAG lasers are the most generally used instruments for both intra-oral soft tissue surgery

and hard tissue applications [12]. Lasers have also been used for processing dental materials, especially for fusing the materials on or into tooth surfaces [10]. The number of studies performed on the laser treatment of dental ceramic masses is gradually increasing [10,13-15]. The CO₂ laser is well suited for the treatment of ceramic materials because its emission wavelength is almost totally absorbed by the ceramic. During the process of heat induction of ceramic surfaces with a focused CO₂ laser, conchoidal tears—typical effects of surface warming—appear. These tears are believed to provide mechanical retention between resin composite and ceramics [16]. While several studies have investigated the effects of laser on hard tissue surfaces [5], studies presenting its effect on surface roughness of alumina-based ceramics are limited [5,15]. Roughness of the ceramic material was measured on digital images obtained by an atomic force microscope (AFM) [1,15] or by a surface profilometer [5]. In the presented study, roughness of In-Ceram Alumina surfaces was measured after surface treatments with CO₂ laser irradiation having different engraving speeds on digital elevation models reconstructed from stereoscopic images acquired by a scanning electron microscope (SEM). Hence, the aim of this *in vitro* study was to present the effect of engraving speed of CO₂ laser irradiation on In-Ceram Alumina roughness.

Experimental methods

Four aluminum-oxide ceramic disc specimens were prepared (6 mm in diameter and 4 mm in thickness) of In-Ceram Alumina (IA) (Vita Zahnfabrik, Bad Säckingen, Germany) according to the manufacturer's instructions. Specimens were ground on wet 240-, 400- and 600-grit silicon carbide paper to obtain a smooth flat surface. The thickness of each specimen was controlled by a micrometer (Praecimeter S, 0.01 mm; Renfert GmbH, Hilzingen, Germany) with repeated measures at four different points. After the finishing procedures, all specimens were subjected to ultrasonic treatment (Coltene Whaledent, Biosonic UC50 Ultrasonic Cleaning System, Cuyahoga Falls, OH) in distilled water to remove any surface residues and dried. Then, one side of each ceramic disc received CO₂ laser irradiation (LG500, Jinan G. Weike Science & Technology Co., Ltd., Shandong, China) with different engraving speeds (100, 400, 600 and 800 mm/min) as a surface treatment under water-cooling, with an exposure time of 10 s. The laser tip was at a 5 mm distance from the ceramic specimen surfaces. The diameter of the laser nozzle was 1 mm and the beam was delivered with a spot size of 0.5 mm. The beam irradiated an area of 500 × 300 mm. Laser energy was delivered in pulse mode with a wavelength of 10.6 μm, pulse repetition rate of 1000 Hz and power of 10 W.

Three-dimensional reconstruction from stereoscopic images (acquired at varying specimen tilt angles) is based on the measurement of the disparity (parallax), which is the shift (in pixels) of the specimen features from one image to the other [17]. There is a long list of experiments performed with the aim of realizing stereo-vision by electron microscopy [18-21]. In practice, the SEM must possess a high-quality stage so that the specimen can be rotated accurately and eucentrically (i.e. the mid-point of the specimen surface being imaged must be positioned so that it is centered on the axis of rotation); it should also be able to output digital image files [22]. The resolution of the result is the same as the resolution of the SEM images. The parallax method for elevation measurement from stereo pairs consists of imaging the same object from two viewpoints at the same plane, so the parallax or the object point displacement along an axis parallel to the straight line through these viewpoints contained on the viewer's plane is proportional to the distance between the selected point and the viewer's plane, i.e. the local elevation is proportional to the local parallax [19].

Samples were examined with SEM (Zeiss EVO50 at Hacettepe University, Department of Geological Engineering) at low accelerating voltage (5 kV) to confine the beam interaction with the specimen to regions very close to the surface. Low accelerating voltage provides an image rich in surface detail compared to those obtained at higher accelerating voltages (15–30 kV), where the beam penetrates beneath the surface and emerging signal electrons mostly carry information about the interior of the sample [23]. Commercial software (MeX, version 5.1, Alicona Imaging, Austria) was used to generate digital elevation models and to perform the roughness measurements. The improved approach (TriCreator) generated a three-dimensional model from three different tilted SEM images. In contrast to the conventional approach using two captured images (stereo pair), this method allows determining the tilt angles between the three images automatically and, thus, results in an improved reconstruction of the height [24]. The depth-of-field range is also important for the measurement performance. If this range is exceeded, blurred regions, which do not contain useful image information, appear. Even though the limited depth-of-field handicap of microscopy imaging was previously used for reconstruction of surfaces [25], for stereoscopic imaging, it is essential to acquire images where the object is all-in-focus. In-Ceram Alumina surfaces were in focus at working distances of ~10 mm. The software calculates the depth image based on the disparity in the stereo images. A large height change in relation to the image diagonal provides better results, namely the disparity can be increased by higher tilting angles. Depending on the length of the working section, the height and

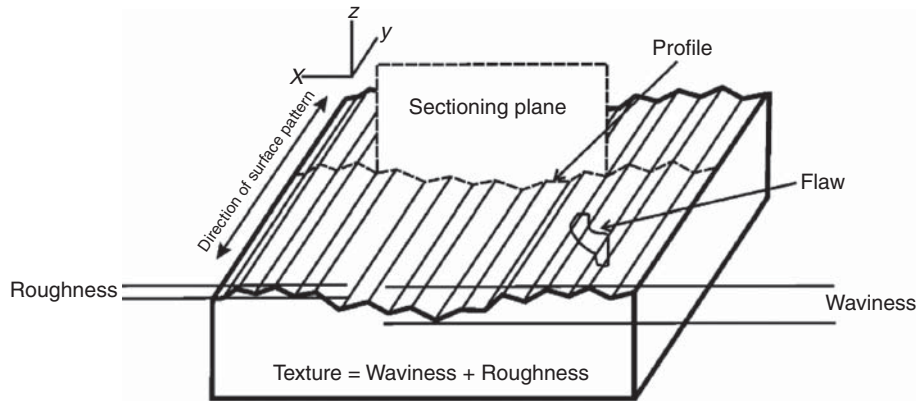


Figure 1. Surface profile (reproduced from [26]).

the image resolution, the ideal tilt angles were calculated. A tilt angle of 3° was used for this study.

Surface roughness is the micro-geometrical deviation. The roughness parameters are estimated by analyzing the topography scans of the sample's surface. Thus, everything that affects the 'real' imaging of the topography will also affect the roughness estimation. Figure 1 [26] depicts a schematic representation of a surface and the applied terms used to describe it. Roughness parameters [27] were directly calculated using the MeX software (version 5.1, Alicona Imaging, Austria) and were derived from the roughness curve which was separated from the waviness curve by a selected wavelength (L_c value of $15\ \mu\text{m}$). The average roughness (R_a) was considered in the present study, which is the area between the roughness profile and its mean line or the integral of the absolute value of the roughness profile height over the evaluation length. R_a was selected as a representative parameter of the surface roughness due to its common application on ceramic surfaces [1,5,15].

Commercial software Statistica (StatSoft, Inc, v6) was used for statistical analysis. Surface roughness data (R_a) were analyzed with One-Way Analysis of Variance. Roughness was the dependent variable and the CO_2 laser irradiation engraving speeds the influencing factor. The level of significance was set at $p < 0.05$.

Results and discussion

Surface roughness parameter (R_a) values and standard deviations are presented in Table I. There is no

Table I. Roughness values (R_a) (nm) of ceramic surfaces after laser treatment.

Sample	Speed (mm/min)	Mean	Minimum	Maximum	SD
1	100	300.43	289.74	306.79	9.31
2	400	320.48	243.14	372.38	8.27
3	600	326.83	322.94	330.72	5.50
4	800	331.11	324.69	337.53	9.08

significant difference between the roughness of surfaces after treatments with different CO_2 laser irradiation speeds ($p = 0.82$). Nonetheless, the mean roughness graph with confidence intervals (0.95) is presented in Figure 2. Representative 3D surface models reconstructed after SEM stereoscopic imaging of In-Ceram Alumina surfaces are shown in Figure 3. Linear nano-scratches and wear on ceramic surfaces formed after finishing the procedure preceding laser treatment. Surfaces show crater-like irregularities after irradiation with a CO_2 laser, confirming previous studies [5,28].

No meaningful correlation was determined between CO_2 laser irradiation speeds and In-Ceram Alumina surface roughness. Second sample stands out with the highest standard deviation (Table I) and probably prevents the meaningful correlation. When the reconstructed surface of the second sample, which was treated with a CO_2 laser speed of 400 mm/min were examined, the distinctness from other sample surfaces is conspicuous (Figure 3). Partial melting signs were observed on the surfaces (Figure 4). Tearing, smearing and swelling occur on melted surfaces. Melted parts form drop-like spherical accumulations. During the process of heat induction of ceramic surfaces with a focused CO_2 laser, conchoidal tears—typical effects of surface warming—appear. These tears are believed to provide mechanical retention between resin composite and ceramics [16].

Swellings increased the height of the existing surface so, the roughness (R_a) [15] point out the thermal effects of laser irradiation by melting a superficial layer of ceramic. This effect leads to the deposition of high amounts of radiation energy in a well-defined part of the ceramic surface over an ultra-short period of time, causing the accumulation of a very high energy density. The radiation energy is thermalized and the temperature, in a thin superficial layer, rises [7,15] represent the formation of two different phases of the ceramic within the fused zones after CO_2 laser treatments, one with crystalline structure and one with an amorphous structure, both showing different thermal expansion coefficients.

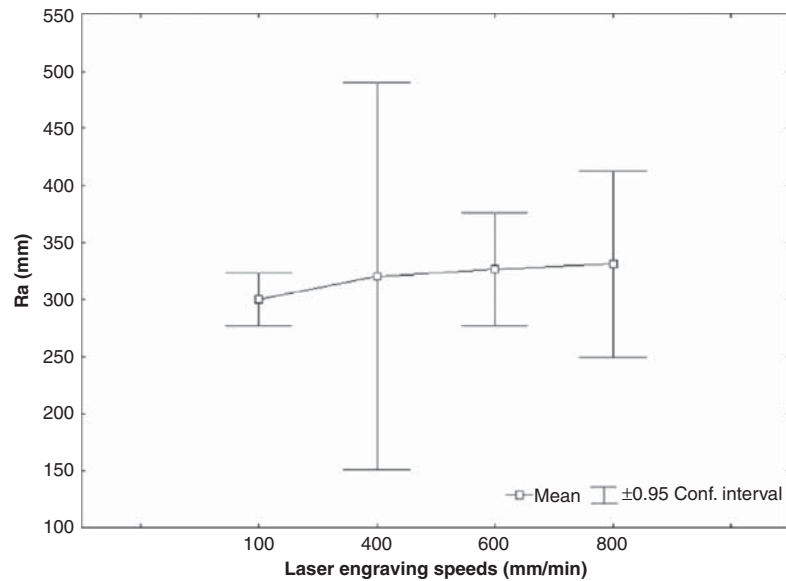


Figure 2. Mean roughness graph with confidence intervals (0.95).

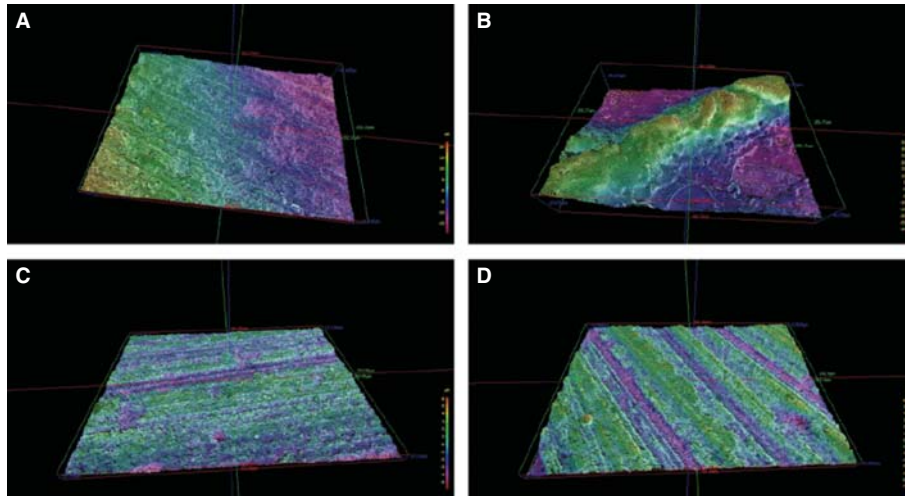


Figure 3. Representative 3D models of treated ceramic surfaces with different engraving speeds. (A) 100 mm/min, (B) 400 mm/min, (C) 600 mm/min, (D) 800 mm/min.

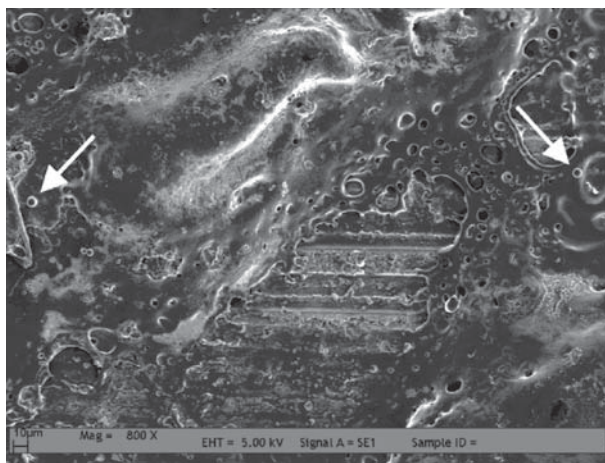


Figure 4. SEM micrograph of sample 2 surface with tears, smears and swelling. Arrows mark the drop-like accumulations (800 \times magnification).

Dissimilar surface characteristics of sample 2 are probably related to different settings of laser treatment for this sample. The power of the laser was fixed at 10 kW for each sample, but different speeds were used. However, laser treatments with higher speeds did not generate such melting signs on ceramic surfaces. In this case, the speed is not a factor on formation of such surfaces. Probably lasing duration on ceramic surfaces, which was not evaluated in this study, is also effective on roughness of surfaces. Nalbantgil et al. [29] tried to determine the amount of lasing time required to remove ceramic brackets safely without causing intra-pulpal damage by using Er:YAG laser. They established a clear relationship between time and temperature consistent with earlier studies [29]. The temperature proportionally increased with the extension of the lasing duration. Ural et al. [28] presented the surface modification on

zirconium oxide ceramics after CO₂ laser treatments with different output powers. Two Watt and 3 W laser irradiated ceramic surfaces showed irregularities such as micro-explosions. However, 4 W and 5 W output power of CO₂ laser created smoother surfaces, melted areas and micro-cracks. Ural et al. [28] used lower output powers (2, 3, 4 and 5 W) compared to this study (10 W), but anyhow melting occurred. Abdallah et al. [13] applied CO₂ laser glazing on In-Ceram Alumina and melting occurred at 10 W power. However, melted zones in previous studies [13,28] were smoother surfaces compared to surfaces treated with lower laser powers or conventionally glazed.

Conclusion

In the presented study, swelling accompanying melting increased the surface roughness while laser power was fixed and different laser engraving speeds were applied. Unfortunately, the laser pulse durations were not considered, which is the limitation of the present study. Research interested in effects of different settings of laser applications on ceramic surface roughness need to consider the laser engraving speeds, which seems to have a major effect on roughness.

Declaration of interest: The authors report no conflicts of interest. The authors alone are responsible for the content and writing of the paper.

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