Surface characterization of monolithic zirconia submitted to different surface treatments applying optical interferometry and raman spectrometry

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This study evaluated roughness parameters and phase transformation of monolithic zirconia ceramics after various mechanical and laser thermal treatments. Fully sintered monolithic zirconia cylinder specimens were divided to five groups, according to the applied surface treatment: CL: control, GB: grit-blasted with glass particles, AL50: grit-blasted with 50 μm alumina particles, AL90: grit-blasted with 90 μm dyed-alumina particles and FEML: subjected to femto laser thermal treatment. Six roughness parameters (Sa, Sq, Sz, Sci, Svi and Sdr) were measured by optical profilometry. Phase transformation in zirconia was determined by micro-Raman spectroscopy. The highest roughness values were recorded in AL90 and FEML groups, followed by AL50. AL90 presented statistically higher monoclinic phase content compared to all other groups. Control and GB groups presented similar roughness without phase transformation. Laser thermal treatment causes minimal destruction of the zirconia surface, and can be suggested as an alternative to other roughening treatments, for enhancing the adhesive potential to dentin.

Keywords: Roughness, Zirconia, Laser, Raman spectroscopy

INTRODUCTION

Nowadays Yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) ceramics are very popular in prosthetic dentistry. Their excellent mechanical properties and biocompatibility encouraged many clinicians and dental laborotaries to fabricate a large number of zirconia based restorations1-2). However, the interface between Y-TZP ceramic cores and ceramic veneering material is considered to be the weak link responsible for the high degree of ceramic chipping. Strong adhesion of veneer ceramics to Y-TZP surface is a prerequisite for chipping resistance. Clinical failures due to veneer debonding or fracture, present an incidence of 20% after 5 years of function3). In order to eliminate veneer failures full contour monolithic zirconia restorations were proposed as an alternative4). Monolithic zirconia follows different sintering and fabrication processes compared to conventional zirconia5). However, the last generation of translucent monolithic zirconia with increased Yttria (5–6%) and less alumina (0.5%) content6) is expected to present different surface characteristics after surface roughening procedures.

In cases of increased aesthetic demands, monolithic cores are combined with veneer ceramics to imitate physical dentition2). Consequently, two major interfaces are involved for the long term success of a dental restoration. One is between zirconia surface and the adhesive cement and the other between veneer ceramic and zirconia core7-8). In order to achieve high bond strength in both interfaces, a series of different zirconia surface modifications has been suggested3,7).

Air abrasion with alumina particles is considered as the gold standard surface treatment, but induces phase transformation9-11). Milder abrading materials, such as polymer or glass beads, have been used for years in other applications. Microscope-controlled glass bead blasting was introduced as an alternative to periodontal surgery12). Only in a recent study, glass beads blasting to presintered zirconia appeared to enhance resin cement adhesion with promising results13).

An alternative concept is the laser thermal treatment of the Y-TZP surface. Most lasers used in dental practice create extensive cracks and surface damage on zirconia surfaces14). Industrial ultra-fast lasers, such as pico and femto second laser, allow surface ablation in hard materials with extreme precision and reproducibility with less collateral damage to the adjacent material than any other thermal or mechanical process15).

Optical scanning methods of interferometry are appropriate for surface roughness analysis. Most researchers measure only the Sa parameter (the arithmetic mean of the absolute values of the surface departures above and below the mean plane within the sampling area) which provides limited information on surface outline and could result in misleading conclusions when used alone. In order to investigate the actual correlation between surface roughness and

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Received Nov 6, 2018: Accepted Feb 10, 2019
doi:10.4012/dmj.2018-358 JOI JST.JSTAGE/dmj/2018-358
adhesive potential, more specified roughness parameters should be adopted\(^\text{16}\). Moreover, the destructive result of surface pretreatments can be defined by the tetragonal to monoclinic phase transformation. Raman spectroscopy is a reliable method for monitoring and quantification of this transition\(^\text{17}\).

The purpose of this study was to evaluate the effect of laser induced thermal treatment and particle abrasion methods to surface roughness and phase transformation of a monolithic zirconia ceramic.

**MATERIALS AND METHODS**

A single type of Y-TZP ceramic material (Bruxzir Solid Zirconia HT 2.0, Glidewell, Newport Beach, CA, USA) was used in this study. This ceramic consists of 92% ZrO\(_2\), 5.2% Y\(_2\)O\(_3\), HfO\(_2\)<4%, and Al\(_2\)O\(_3\)<0.5%, SiO\(_2\)<1% by weight\(^\text{16}\). Cylindrical monolithic zirconia specimens (diameter=10 mm, \(n=50\), BruxZir Solid Zirconia, Glidewell) were prepared from pre-sintered zirconia blocks. After sintering, all specimens were embedded in epoxy resin (Epofix, Struers, Ballerup, Denmark) leaving one of the surfaces uncovered and ground in a grinding/polishing machine (DAP V, Struers). A customized metal base was fabricated to ensure identical vertical force (20 N) for each specimen during the grinding procedure. Rotary polishing machine was stabilized at a speed of 200 rpm and silicon carbide papers (220 and 1200 grit-size, Struers) were used under water-coolant for 10 min. Specimens were finally ultrasonicated in ethanol for 3 min, water-rinsed and air-dried and equally divided into 5 groups (10 specimens/group) which were subjected to the following treatments:

- **CL:** Control, no further treatment.
- **GB:** Specimens were air-abraded with crushed glass beads using an intraoral sandblaster (Microetcher II A, Danville Materials, S. Ramon, CA, USA) operated for 10 s at 0.25 MPa air pressure, 10 mm distance and 45° incidence angle. Blasting powder contained 0–10% 45 μm grains, 90–100% 63 μm grains and 0–1% 90 μm grains.
- **AL50:** Specimens were air-abraded on the surface with alumina 50 μm grains operating under the conditions described in the GB treatment.
- **AL90:** Specimens were air-abraded on the surface with dyed alumina 90 μm grains operating under the aforementioned conditions.
- **FEML:** Specimens were subjected to laser thermal treatment and were grooved by continuous horizontal line scans with an overall area of 5×5 mm with line spacing of 50 μm.

**Femto laser thermal treatment**

The experimental apparatus used for the direct laser processing of the samples contained the following: An Yb:KGW laser source produced linearly polarized pulses of 170 fs, 1 KHz repetition rate and 1.026 nm central wavelength. The laser pulses were focused on the sample via an achromatic convex lens of 100 mm focal length while the Gaussian spot diameter, measured by a CMOS camera closed to the focal plane at 1/e\(^2\), was 35 μm diameter. Samples were fixed onto a 3-axis motorized stage and positioned perpendicular to the incident beam. All irradiations were performed in ambient environment at normal incidence. At the same time, power modulation was achieved by the means of an automatically rotating zero-order half wave plate behind a high extinction ratio, glan-taylor linear polarizer. The design and successful irradiation of a specific laser pattern was performed by continuous horizontal line scans with overall area dimensions of 5×5 mm with line spacing of 50 μm. For the fabrication of test surfaces on the zirconia specimens laser parameters were: laser fluence, \(\phi=9.6\ J/cm^2\) and sample speed during laser scanning, \(v=1\ mm/s\) (Fig. 1).

**Optical microscopy**

All specimens after treatment were examined under a stereomicroscope (M80, Leica, Weltzkar, Germany) at 10×–60× magnifications to assess defects or discontinuities of zirconia surfaces.

**Surface profilometry**

In order to measure changes in the surface roughness of the specimens \((n=5/group)\) an optical interferometric profiler was employed (WykoNT 1100, Veeco, Tuscon, AZ, USA) under the following conditions: Mirau lens (F), vertical scanning interferometry mode (VSI), 304×231 μm analysis area (20× magnification) and 0.1 nm (z-axis) and 0.2 μm (x- and y-axes) resolution. Three amplitude (\(Sa, Sz, Sq\)), one hybrid (\(Sdr\)) and two functional (\(Sci, Svi\)) parameters were measured. In this context, the definitions of these parameters are: \(Sa\) is the arithmetic mean of the absolute values of the surface departures above and below the mean plane within the sampling area and represents an overall measure of the surface texture. \(Sq\) is similar to \(Sa\) and is the root mean square (rms) roughness. \(Sz\) is defined as the average value of the
absolute heights of the five highest peaks and the depths of the five deepest pits or valleys within the sampling area. Sdr expresses the ratio of the increment of the interfacial area of a surface over the sampling area. Sci defines the ratio of the void volume of the unit sampling area at the core zone and is related to the volume of fluid retention in the core zone. Svi is derived from the analysis of the bearing surface area, quantifying the volume of fluid filling the core and the valleys of the given surface, respectively18).

Raman spectroscopy
The monoclinic phase content on each zirconia specimen (n=3/group) was determined by Raman spectroscopy. Raman spectra were obtained by an HR800 UV-Raman setup (Horiba group, Montpellier, France) using the 441.6 nm wavelength of a HeCd laser as the excitation radiation. Focusing of the excitation beam as well as collection of the scattered radiation was performed by a microscope objective (50×) which offered spatial resolution of ~1–2 μm and in addition enabled visualization of the sample’s surface. The laser power on sample for all measurements was ~10 mW which did not result in any observable light/thermal induced structural alterations. The scattered photons were directed to an appropriate edge filter, which rejected most of the elastically scattered ones. The inelastically (Stokes-side) scattered photons were focused on the entrance slit of a single monochromator (800 mm). Dispersion with respect to their wavelength was achieved by an 1,800 grooves/mm grating and detection was done by a liquid nitrogen cooled CCD detector. Resolution was set to <4.5 cm⁻¹ within the whole spectral range recorded. For averaging purposes a set of ~100 spectra were obtained from the sample’s surface under investigation. Manipulation of the collected spectra was performed according to the methodology described in detail in reference19).

Statistical analysis
One-way anova with associated Bonferroni multiple comparisons tests was conducted for the Sci parameter that presented normal distribution and homogeneity of variance evaluated through Shapiro-Wilk test and Levene’s test of homogeneity of variance accordingly. Nonparametric analysis was used in measured quantitative variables that were not normally distributed. Kruskal Wallis and Mann-Whitney tests were performed in Sa, Sq, Sz, Svi and Sdr parameters. Raman spectroscopy results followed non-parametric Kruskal-Wallis test due to lack of homogeneity of variances. The analysis was performed with the IBM Statistics SPSS 20.0 software and the significance level was set at p<0.05.

RESULTS
Optical microscopy
In FEML treatment the change of colour of the processed area (brown) was visible even without magnification. In CL group even with maximum reflectance (when optically viewed) only minor surface scratches were visible and were not different to the GB group. In alumina blasted groups the loss of surface gloss was visible.

3D-profilometry
Representative 3D-profilometric images of the zirconia surfaces per treatment group are illustrated in Figs. 2(a–e). The mean values of the six roughness parameters

Fig. 2  (a–e) All groups of monolithic zirconia surface treatments.
Representative 3D-images (20× magnification) of CL (a, scale range 0.87–1.66 μm), GB (b, scale range 1.16–0.64 μm), AL50 (c, scale range 2.14–3.71 μm) AL90 (d, scale range 4.7–8.8 μm) and FEML (e, scale range 8.6–43.6 μm).
Fig. 3 Raman spectra of the control as well as the treated samples.

The spectra presented derived after averaging 100 spectra obtained from various positions on the surface of each sample. Inset indicates the characteristic peaks of the monoclinic and tetragonal zirconia phase which have been used for the calculation of the monoclinic phase vol%.

Table 1 Mean values and standard deviations of roughness parameters in all experimental groups

| Group | Sa nm  | Sq nm  | Sz μm | $S_v$ | $S_i$ | Sdr % |
|-------|--------|--------|-------|-------|-------|-------|
| CL    | 73.9±10.7 d | 98.5±13.5 d | 1.15±0.1 d | 1.22±0.03 b | 0.14±0.01 a | 1.2±0.35 e |
| AL50  | 340.3±49.2 b | 436.2±57.37 b | 3.32±0.55 b | 1.41±0.06 a | 0.13±0.01 a | 15.19±4.76 b |
| AL90  | 1,155.1±97.76 b | 1,458.2±119.4 a | 8.02±1.16 a | 1.43±0.12 a | 0.11±0.01 b | 9.92±1.14 c |
| GB    | 99.4±16.6 b | 137.95±19.2 a | 1.49±0.19 c | 1.41±0.09 b | 0.10±0.01 b | 2.35±0.6 d |
| FEML  | 307.3±32.7 b | 699.8±97.5 a | 8.31±1.98 a | 0.86±0.08 b | 0.09±0.01 b | 656.1±179.03 a |

Same letters indicate non statistically significant differences at $\alpha=0.05$.

are summarized in Table 1. For Sa statistically significant differences were recorded among all groups, except between Al50 and FEML, for Sq except between AL90 and FEML and for Svi except between GB and FEML. All surface parameters presented significant differences between CL and the rest groups and between FEML and the rest groups. The ranking for amplitude parameters was the following: for Sa AL90>AL50>FEML>GB>CL, for Sq AL90>FEML>AL50>GB>CL and for Svi AL90>FEML>AL50>GB>CL. For the functional parameters, the ranking was: for Sci AL90, AL50, GB>CL>FEML, for Svi AL50, CL>AL90, GB>FEML and finally, for the hybrid parameter Sdr FEML>>AL50>AL90>GB>CL.

Raman spectroscopy

Raman spectra were collected from the treated surface of the samples. Based on the methodology described in detail$^{19}$ as well as on the early work of Katagiri$^{17}$ we calculated the monoclinic volume fraction from all the recorded Raman data after appropriate manipulation. Due to the low spatial resolution (~1 μm) offered by the technique, a series of ~100 spectra for each sample were apparently acquired and averaged in order to be processed and quantify the monoclinic phase volume fraction. Averaged spectra corresponding to the different types of surface treatments are shown in Fig. 3. Most of the bands observed are assigned to the tetragonal phase (e.g. 146, 260, 317, 462, 607 and 640 cm$^{-1}$) while only weak bands attributed to the monoclinic phase contribute to the spectra (for example peaks at ~180 and 190 cm$^{-1}$). The inset depicts the spectral range where one clearly observes the peaks of the monoclinic phase (~180 and 190 cm$^{-1}$) and the characteristic one of the tetragonal phase (146 cm$^{-1}$) which are used for the quantification of the monoclinic phase volume fraction. There is no monoclinic phase in the control samples indicating that the polishing process does not alter the initial structure of the material. Glass treatment appears to have only minor effect on the material’s structure (monoclinic phase peaks are hardly observed).

Micro-photographs of the laser treated “grooved” samples are given in Fig. 4. The width of the successive pits/bumps is ~45/55 μm thus the periodicity of the grooves is 100 μm. A picture of a single groove focusing on the top sample surface is shown in Fig. 4(a), while the same area when focusing at the lower region of the groove is shown in Fig. 4(c). Raman spectra from the laser treated areas of the samples (similar to the one shown in Fig. 4(c)) are characterized by weak monoclinic bands. On the contrary, spectra obtained at the untreated top sample surface are totally tetragonal. Alumina treated samples possess structural characteristics that depend on the size of particles used. Thus, sandblasting with smaller particles results in lower surface roughness and leads to low monoclinic phase volume fraction. Samples treated by larger particles exhibit greater roughness as well as monoclinic volume fraction. The results derived from the Raman measurements that quantify the monoclinic volume fraction for each treated sample are given in Fig. 5. Kruskal-Wallis analysis showed statistical significant differences among all groups (Table 2), with the order of the highest monoclinic content to the lowest being the following: AL90>FEML>AL50>GB>CONTROL.
Fig. 4 Microphotographs obtained from the surface of FEML sample (a) low magnification, (b) high magnification focusing on the upper part of the grooves (non-illuminated areas of the sample), (c) the respective photo captured after focusing at the bottom part of a groove (illuminated area of the sample). Notice the visual differences with respect to the roughness between illuminated and non-illuminated areas.

Table 2  Kruskal-Wallis statistical analysis results for monoclinic phase content

| Null hypothesis                                                                 | Test               | Sig.   | Decision                  |
|---------------------------------------------------------------------------------|--------------------|--------|---------------------------|
| The distribution of monoclinic phase content (vol%) is the same across categories of treatment | Indipendent samples Kruskal-Wallis tests | 0.000  | Reject the null hypothesis |

Asymptotic significances are displayed. The significant level is 0.05.

Fig. 5 Monoclinic phase vol% extracted by Raman spectroscopy for the samples under investigation. Different letters show that monoclinic phase content differed significantly among all groups (p<0.001).

DISCUSSION

Both surface roughness and monoclinic phase volume fraction were influenced by surface treatment. The observation of amplitude parameters (Sa, Sq and Sz) revealed an overall estimation of roughness in all groups. AL90 group presented statistically higher average roughness compared to all other groups (Sa, Sq), followed by AL50 and FEML. Differences in Sq but not in Sa were found between AL50 and FEML group. Significant lower measurements were recorded in CL and GB groups for the Sa and Sq values. An initial estimation was that abrading with 90 μm hard alumina particles created rougher surface. Compared to AL50 group, larger particle size at the same pressure induced significant surface alteration. Increased kinetic energy of AL90 particles may have caused deeper valleys and larger imprints in zirconia surface and a remarkable increase of stress that may have accelerated the tetragonal to monoclinic phase transformation. Laser grooves (FEML) were rougher than those on the CL surface, indicating that ablation resulted in different surface pattern. Increase of amplitude parameters after femto laser ablation was identified on zirconia dental implants in an early work of Delgado-Ruiz et al.\(^{20}\) and the increase in roughness parameters was similar to the present study, although initial roughness of zirconia dental implants was significantly higher. GB treatment resulted in minimal change in the initial roughness, which was expected, since glass particles are softer than alumina and zirconia particles. The same ranking was followed in the value of Sz, with the exception of AL90 and FEML presenting similar values.

The hybrid parameter Sdr is essential for adhesive protocols. AL90 and AL50 presented statistically increased values compared to the CL and GB groups. Moreover, AL50 was statistically higher than AL90. A possible explanation could be that smaller particles may have invaded in extended area and the result could be beneficial for adhesion with different substrates. The extreme Sdr values of the FEML group may have been
the result of the new surface pattern caused by the laser. Increased waviness of the surface or densely distributed sharp edges and valleys could explain this observation. The results for Sdr are similar with a previous work of Queiroz. In an earlier work, Jevnikar et al. measured higher Sdr values (73.2) after 110 μm alumina blasting in total different abrasion variables and bulk zirconia material implying that more aggressive abrasive method could be also beneficial for adhesion.

Functional parameters (Sci and Svi) can be related to mechanical interlocking mechanisms, since the fluid volume retention in core and valley zones affects the penetration of the adhesive substrate on the zirconia surface. In all blasted groups, Sci was significant higher compared to the CL and FEML groups. Low values of Sci and Svi in FEML group were observed. This indicates that the extended lased surface (as measured by Sdr) might present very condensed and sharp peaks and valleys that cannot support maximum fluid volume.

Raman spectroscopy revealed that initial polishing in the CL group did not induce measurable t-m transformation. Similar result for monolithic zirconia material, after gentle polishing with rotary instruments containing diamond and silicone carbide abrasives, was described by Huh et al. All air-abraded samples (either with crushed glass beads or with alumina grains) exhibited monoclinic phase vol% characterized by significant error bars which indicates the inhomogeneity of the phase transformed material on the samples’ surface. On the contrary, the phase transformation induced on the laser treated areas of the respective samples (FEML treated samples) was rather uniform, as shown by the considerably lower error bars in Fig. 3. Furthermore, the monoclinic phase for the FEML sample was calculated only in the laser treated regions (50% of the total surface). The remaining untreated regions presented no monoclinic phase, similarly to control surfaces.

In the present study, the polishing of Zr followed a strict protocol, standardizing the grit size, time and pressure to each specimen by the manufacturing of a special metal holder base. Polishing and glazing techniques significantly reduce zirconia roughness. The initial roughness of as-sintered specimens was significantly decreased after polishing. Owing to the fact that zirconia is a very hard material, the primary abrasive must be diamond, SiC, alumina or zirconia. Blasting pressure for all groups was standardized at 0.25 MPa, since further increase could cause deposition related damage without significant gain in roughness parameters.

The GB treatment resulted in similar roughness compared to the CL, which was expected, since glass beads are softer than alumina particles. Minimal roughness change is not recommended for better adhesion, but GB can be considered as an excellent alternative for decontamination or removal of debris on zirconia surface. Additionally, blasting with glass beads is a slow and mild process that effectively strips the zirconia surface, without causing phase transformation. This method has been introduced in combination with silane and resin cement with promising bond strength results.

Laser patterning is a new concept to increase the adhesive potential, since zirconia surface is extended in both macro and micro scale with minimal structural changes. FEML grooving resulted in a rough surface pattern and the laser induced lines presented repetitive morphology. The zirconia material, in between the grooves, was left intact with the same characteristics as the CL group. The surface appearance was distinct from other treatments with a repetitive regular structure that bears the potential for increased retention of resin and ceramic dental materials. The size of FEML grooves ≤50 μ depth, ≤50 μ width) multiplies macroscopically the adhesive surface and seems advantageous compared to the AL50 and AL90 treatments. The pulse duration was used on the fs regime to minimize the thermal effect and collateral damage during the ablation process. The laser wavelength was fixed at 1.026 nm since no other option was available, and the velocity 1 mm/s was decided as it was efficient for accumulating more than 1 number of pulses for efficient material removal and relatively rapid laser scanning according to the following equation: Neff = 2 × w0 × f/v, where Neff is the effective number of pulses receptive to the sample surface during the laser scanning, w0 is the beam waist at the focal plain, the f is the laser repetition rate and the v is the translation stage velocity. The laser fluence was determined at 9.6 J/cm² as it was enough to cause material removal of 50 μm depth and 50 μm width. This volume was considered sufficient for the retention of a resin cement material or a veneering ceramic.

The optimization and the characteristics of each groove is a very promising project for future research to further optimize surface roughness. Some researchers have already introduced laser patterning to improve zirconia adhesive performance. Encouraging results were obtained from adhesion of veneer ceramics or resin cements to femto lased zirconia surfaces.

Three dimensional (3D) surface roughness describes surface characteristics better than 2D roughness measurements. The large number of existing roughness parameters poses difficulties to adequately compare processed surfaces. Researchers measure a series of different parameters for more accurate assessment of roughness. The parameters selected in this study focus on the adhesion potential of zirconia surface with veneering and cementation materials.

The present study represents a preliminary report of novel zirconia surface treatments. One type of translucent
monolithic zirconia (Bruxzir HT 2.0) was tested. Further investigations are necessary to assess the influence of specific roughness parameters to bonding performance with different zirconia substrates. New methods that might combine femtosecond laser patterns, with other additive or more aggressive surface pretreatment methods paves way for new perspectives regarding the optimization of zirconia adhesion to resin and ceramic materials.

CONCLUSION

Surface roughness parameters and monoclinic phase content are influenced by zirconia surface pretreatments. Alumina abrasion increases zirconia roughness inducing a significant inhomogeneity in zirconia phase transformation. Glass bead blasting has the least effect on surface roughness compared to all other surface treatments. Femto laser treatment increases zirconia roughness and monoclinic phase content, affecting only laser damaged areas, must be considered as a promising alternative method for the adhesion of veneering ceramics and luting materials.

ACKNOWLEDGMENTS

This post-doctoral research was carried out within it “AID TO POST DOCTORAL RESEARCHERS” of the scientific field “Development, Human Resources, Education and Lifelong Learning”, which is being implemented from the IKY and was co-funded by the European Social Fund and the Greek public.

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