

Effect of Hydroxyapatite Coating in combination with physical modifications on Micro-shear Bond Strength of Zirconia to Resin Cement

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Research Article

Keywords: In-Ceram Zirconia, hydroxyapatite cement, Resin Cements, Shear Strength, Air Abrasion, Dental

Posted Date: April 21st, 2022

DOI: <https://doi.org/10.21203/rs.3.rs-1555924/v1>

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Abstract

Background

Zirconia has been used as a reliable core material in dental restorations for years, however, its bonding to resin cement is a matter of challenge. Physical, chemical, and a combination of these techniques have been investigated to boost the properties of zirconia surface bonding. The objective of this work was to evaluate the effect of hydroxyapatite coating as a chemical therapy in combination with physical modifications on micro-shear bond strength of the resin cement over zirconia.

Methods

In the present research, 60 sintered zirconia blocks (4×4×4 mm) were randomized into four groups of 15, including Al₂O₃ particle abrasion (group 1), HA coating (group 2), Al₂O₃ particle abrasion + HA coating (group 3), and Er, Cr:YSGG laser irradiation + HA coating (group 4). The micro-shear bond strength was determined by bonding the blocks to the resin cement.

Results

Bond strength (mean ± standard deviation) of modified zirconia surfaces were 16.93±4.94 MPa, 16.14±5.4 MPa, 19.4±5.27 MPa and 16.21±3.7 MPa in the group 1 to 4, respectively. ANOVA tests results clarified no significant difference regarding bond strength values of zirconia surfaces to the resin cement between studied preparation modalities. (P>0.05)

Conclusion

Observations of the present study showed that HA coating can be as effective as airborne-particle abrasion technique in the improvement of bond strength to zirconia surface. Moreover, sandblasting by aluminum oxide or Er,Cr:YSGG laser irradiation prior to HA coating of zirconia showed no significant effect on the reinforcement of bond strength values when comparing with HA coating alone.

1. Background

All-ceramic materials optimized for mechanical properties have recently attracted special attention for use in the manufacture of fixed metal-free restorations. Among these, zirconium oxide (ZrO₂) is of the most popular ceramics [1]. This ceramic has desirable characteristics such as, biocompatibility, good appearance and chemical stability. Thus, it becomes a favorable material with various applications in dentistry such as dental braces, post, implant restoration abutments, and framework of fixed partial prostheses. Likewise, it is applicable in conservative restorations including veneers, inlay, onlay, and Maryland bridges. The non-retentive preparation in these restorations has led to a special importance to the bond strength between tooth and restoration [2]. More conservative tooth preparation, improved marginal adaptation, increased restoration retention, and reduced microleakage are among the advantages of a chemical bond of the restoration with the resin cement [3]. Hence, it is of clinical

importance to achieve a chemical bond in zirconia surface especially, when the macro-mechanical retention is compromised. Moreover, it has been shown that the coupling agents were not successful in attainment of an acceptable chemical bond between the resin cements and zirconia disparate silica-based ceramics due their poly-crystalline glass-free structure that cannot be etched by common acids [4]. Resin-bond strengths is associated with their potential to penetrate into the surface irregularities of the underlayer [5]. The methods to achieve improved bond strength of zirconia with resin cements can be classified into 3 groups including physical, chemical and physicochemical techniques. Tribochemical treatment by Rocatec™ is the available physicochemical technique regarding the ceramics like Y-TZP. In this technique, silica-coated aluminum particles are subjected to low pressure sandblasting, followed by silica-mediated chemical modification of ceramic surface [6]. Application of primers containing organophosphate monomers and carboxylic acid are among the chemical techniques for improving the adhesion [6]. Resin cements containing 4- methacryloxyethyl trimellitate anhydride (4-META), methacryloxy decyl phosphoric acid (MDP), or 3-trimethoxysilylpropyl methacrylate (MPS) monomers could have a reaction with oxide groups in the structure of Y-TZP crystalline. This process resembles the interaction between the silane coupling agent and silica-based ceramics [7]. Air-borne particle abrasion with Aluminium Oxide and laser etching are examples of creating surface roughness as a physical technique [8]. Alumina sandblasting can successfully enhance surface area and create active surface with increased wettability in the dental materials [9]. Nonetheless, this method has been criticized for its adverse effects on the mechanical profiles of zirconia like flexural strength as well as the possibility to induce subcritical crack growth within the material [9]. Lasers have different applications in dental treatments. The neodymium-doped: yttrium aluminum garnet (Nd: YAG) laser can be used in reducing teeth sensitivity, caries removal, bleaching and producing surface roughness in high-strength core ceramics [10]. Liu et al. [11] demonstrated rougher surface of zirconia ceramics with higher output power of Nd: YAG laser. However, shear bond strength of the laser group was not obviously increased. The pulsed erbium lasers (i.e. Er,Cr:YSGG and Er:YAG) has been applied for the treatment of surface. Kasrae et al. [12] revealed that the bond strength of zirconia with resin cement was greater in groups treated with Er: YAG laser in comparison to CO₂ laser treated samples. Referring to the findings, the Er,Cr:YSGG laser can generate comparable surface roughness rather than acid etching of dentin or enamel surface [13]. Likewise, it could effectively change the surface roughness of the lithium disilicate ceramics and increase the shear bond strength of this ceramic with resin cement [14]. Contradictory findings are available regarding the preference of different surface-conditioning approaches to improve the adhesion of resin cement with zirconia. The inorganic matrix of bone and teeth of human beings can be found in Hydroxyapatite or HA [Ca₁₀ (PO₄)₆·2(OH)] as phosphocalcic hydroxyapatite [15]. It exhibits an admirable biocompatibility, with a crystal structure and composition like apatite in the skeletal and dental architectures of human. Seo et al. [16] fabricated a coating of HA zirconia substrates by a room temperature spray process. They exhibited no severe dissolution being observed during in vitro experimentation [17]. Significant advances in nanotechnology have suggested practical applications for nano-HA in dentistry, with crystals ranging from 50 to 1000 nm. Nano-HA possess bioactive and biocompatible properties. Moreover, they are similar to the tooth enamel apatite crystal morphologically, and in terms of crystallinity and crystal architecture [18] Further, Nano-HA has a filler function because of

repairing tiny depressions and holes on the surface of enamel, an action that increases with the small size of the constituent particles [15]. Nanoparticles produce a thin and consistent coating layer with adequate strength [19]. Nano-sized HAp increases the level of crystallinity in glass ionomer cements and enhances mechanical features such as micro-shear bond strength [20]. Panavia F 2.0 is an extensively applied resin cements in dentistry, which contains a bifunctional monomer, 10-methacryloyloxydecyl dihydrogen-phosphate (MDP). According to the favorable adhesion of the resin cements to the tooth structure which is mainly composed of HA, it is hypothesized that a coated layer of HA on the zirconia could result in a suitable bond strength. The present study investigated the influence of HA coating on micro-shear bond strength of zirconia to Panavia resin cement. In accordance with the study null hypothesis, no difference is there in the μ SBS between the study groups. Likewise, no difference in the patterns of failure.

2. Materials And Methods

In the current in vitro work, 60 pre-sintered commercial dental zirconia blocks (Cercon, Dentsply, Amherst, N.Y.) with dimensions of 4 × 4 × 4 mm were ground for 60 s via 600 and 800 Struers RotoPol 11 silicon carbide grit abrasive (Struers A/S, Rodovre, Denmark), followed by cleaning in a i600B steam cleaner (Italy ELT) for 10 minutes using ethanol (96%) and subsequently air-drying for 30 s. In accordance with an earlier identical work [21] and regarding the Bonferroni formula for determination of the sample size, the number of specimens in each group was determined as n = 15. The sampling method was non-randomized in this study. However, the allocation of specimens to each group was randomized.

Group 1 consisted of zirconia blocks sandblasted with Al₂O₃ particles (50 μ m) under a pressure 4 kg/cm² and a distance of 10 mm for 15 s in a MESTRA sandblast apparatus (TALLERES MESTRAITUA S.L, Espana), followed by cleaning via ethanol (96%) for 10 min in ultrasonic cleaner. Group 2 consisted of zirconia blocks whose surface were coated via HA thermal protocol so that a slurry solution was prepared by adding 10 gr of nano particle HA powder (less than 100 nm) (Merck, Germany) to distilled water (50 cc). Then, 1gr of poly vinyl alcohol (Merck, Germany) was also added as the binder of the suspension, followed by heating on a magnet stirrer with 1000 rpm at 100°C for 60 min to achieve a uniform suspension. At last, the zirconia blocks were placed into in this slurry at angle of 45° for 5 s.

Group 3 consisted of the zirconia blocks sandblasted with the same condition as described in group 1 and then layered with a coating of HA following the same procedure as described in group 2.

Group 4 consisted of the zirconia blocks whose surfaces were first exposed to Er,Cr: YSGG laser irradiation with the following details: wavelength of 2.78 μ m, and 140- μ s pulse duration with a 20-Hz repetition rate and 4-W output power. The laser optical fiber (in a diameter of 600 μ m and a length of 6 mm) with the gold handpiece was positioned to the surface at a distance of 10mm perpendicularly and moved in a sweeping manner manually within a 30-s exposure time over the whole area. Continuous water (55%)-air (65%) flow were applied while performing irradiation. After laser treatment, the surface was coated with HA as explained for group 2.

Subsequent to the surface treatments, all the blocks in groups 2, 3, and 4 were sintered following this protocol: ambient temperature to 300°C for 10 minutes, 300–600°C for 10 minutes, 600–900°C for 30 minutes, 900–1200°C for 40 minutes, maintenance at 1200°C for 120 minutes and cooling for annealing, employing a CWF Furnace (Keison Products, UK). Following the surface treatment in all the study groups, a mold was selected to be Tygon Norton Performance Plastic tubes (with an inner diameter of 0.8 mm and a height of 1 mm (Cleveland, OH, USA) for the Panavia F2.0 cement bonding (Kuraray Medical Inc.) to prepared surfaces. Then, the tubes were removed with a heated sharp scalpel. All samples were placed into distilled water, for 24 hours incubated (Incubator, Model PL-455G PecoPooya Electronic Co.) at 37°C, and positioned in a micro-tensile tester (Bisco Inc., USA) for the determination of micro-shear bond strength. A metal loop (0.2 mm thick; Ligatur, dentsply GAC_SOF) was put around the cement cylinder in the bonding site for the exerted tensile load conversion to shear load through vertical soldering of casting molds to jig, as seen in Fig. 1. The load extent at failure (crosshead speed = 0.5 mm/min) was determined, followed by computing the values of micro-shear bond strength in accordance with the equation of $S \text{ (MPa)} = F \text{ (N)} / A \text{ (mm)}$. Following the shear experiment, a SEM was used to determine the fractured surfaces for achieving the failure variants that included Cohesive failure, Adhesive failure and Mixed failure

Statistical analysis method

To analyze data the SPSS software version 21.0 was used. The statistical comparison of the bond strength was conducted with one-sided analysis of variance: ANOVA among the 4 groups. In the present study, the type 1 error rate (α or p value) was considered equal to 0.05.

3. Results

The mean μ SBS in group 1, 2, 3, and 4 was calculated as 16.93 ± 4.94 MPa, 16.14 ± 5.4 MPa, 19.4 ± 5.27 MPa, and 16.21 ± 3.7 MPa, respectively. (Table 1). According to one-sided analysis of variance results, the differences in the amount of μ SBS were not significant among the study groups ($p = 0.23$). Since there were no significant differences as using the ANOVA analysis, no pairwise comparison was done between the groups.

Table 1
Central dispersion indices of SBS of resin cement to zirconia surfaces in different preparation groups

μ				
Groups	Mean (MPa)	Standard deviation	Minimum	Maximum
1: Sandblasting with Al ₂ O ₃	16.93	4.9	10.3	26.4
2: HA coating	16.14	5.4	7.9	26
3: Sandblasting + HA coating	19.4	5.2	9.5	28.6
4: Er, Cr: YSGG + HA coating	16.21	3.7	10.9	23.6

The most common failure mode in all the study groups were the adhesive type. All the cohesive fractures occurred in the cement cylinder. The mixed type of failure was less common type of fracture in group 1 and 3. Table 2 describes the number of different types of fractures in the study groups. (Fig. 2)

Table 2
Dispersion and type of failure mode among the 4 study groups

Groups	Adhesive	Cohesive	Mixed
1: Sandblasting with Al ₂ O ₃	11 (73.3%)	1 (6.7%)	3 (20%)
2: HA coating	8 (53.3%)	2 (13.3%)	5 (33.3%)
3: Sandblasting + HA coating	9 (60.0%)	3 (20.0%)	3 (20.0%)
4: Er, Cr: YSGG + HA coating	8 (53.3%)	2 (13.3%)	5 (33.3%)

4. Discussion

According to the mineral tooth structure which is mainly composed of hydroxyapatite crystals, and favorable resin cement bond strength to such structure, it is hypothesized that coating the zirconia surface with the Nano-sized HA might boost the bond strength with resin cements. The current effort investigated the influence of hydroxyapatite coating solely and in combination with physical surface modification via Er,Cr:YSGG laser irradiation and air borne particle abrasion. The results indicated that the null hypothesis should be accepted, as the μ SBS was not significantly different among the study groups. While Y-TZP offers many advantages such as high strength and fracture toughness, and good wear resistance, there are some disadvantages such as the non-polar nature leading to negligible bonding capacity to dental structure and/or overlaying ceramics [22]. Multiple chemical and mechanical methods have been reportedly combined for the enhancement of bonding activity of dental zirconia [1]. The surface of zirconia can be modified by a HA coating, which has been previously used as a promising method to modify bioinert metallic surfaces in implant dentistry [16]. As reported by Sagsoz, Omer, et al. the highest bond strength within resin-ceramics and resin cement was among HAp group. According to the conclusions by them, HAp coating can be used instead of hydrofluoric acid etching and sandblasting

in CAD-CAM materials and resin cement to improve bond strength [23]. There are diverse coating approaches available for exploiting the merits of HA. No standard instructions have been issued so far for the various dimensions of a particular approach, such as surface profiles, porosity, texture, coating thickness and crystallinity or crystal size effects. Several methods of coating a HA layer onto bioinert metallic implant surfaces have been previously reported which include the plasma spray and sol-gel methods. The present work benefited from thermal coating approach because of fine mechanical profiles, practically feasibility and admirable phase composition [21]. Thermal coating technique has possibilities to modify surface features of Zirconia and might be capable of shifting the bonding pattern of Zirconia ceramics [21]. As concluded by Okada, Masahiro, et al. thermal coating of the zirconia after applying silane coupling agents has capacity to boost the shear-bond strength within the zirconia and composite resin cement [24]. Previous research have indicated that in comparison to control group, air borne particle abrasion boosted significantly the bond strength of zirconia surface to Panavia F2.0 resin cement [21]. In addition, one of the best surface treatment approach is sandblasting for surface modification of zirconia ceramics [25]. Thus, it was employed as control group in our work. Controversies and limitations of sandblasting method can be addressed by coating zirconia surface via HA regarding its merits, some of which are great surface tension, radiopacity, optimal hardness as for natural teeth, potent wear profile, and admirable bonding and wettability. The present study revealed that the HA-decorated zirconia surface boosted bond strength to resin cement similar to sandblasted group. However, combination of other surface treatments such as Er, Cr: YSGG laser irradiation or air abrasion has shown to have no additional benefit in improvement of bond strength. The lowest range for acceptable clinical bonding was suggested to be between 10 and 13 MPa [26] The μ SBS values of all study groups were beyond the acceptable range and their differences were not statistically significant. As stated by Ranjbar Omidi B et al., silica coating through Cojet sand, presented substantially greater μ -shear bond strength compared with Er: YAG laser and sandblasted group. Also, μ SBS values of sandblasted group were higher than Er: YAG laser [27]. Sandblasting can increase the bond strength between zirconia and resin cement [21]. Tanis et al. [28] indicated that the values of shear bond strength between Y-TZP and Panavia F2.0 significantly were greater in the group modified with sandblasting + tribochemical silica coating + silane in comparison to the sandblasted specimens. Consistent with this, Zandparsa et al. [25] reported that sandblasted zirconia surface showed inferior shear bond strength to enamel in comparison to Al₂O₃ air abraded + Z-PRIME Plus group. According to the findings of these two studies and the other similar studies [1] it might be assumed that an increase in surface roughness via laser irradiation or air-borne particle abrasion in combination with modifying the surface with a layer of HA might result in higher values of μ SBS. Although the μ SBS value was higher in group 3 in comparison to group 1 but such difference was not statistically significant. This might be due to the replacement of the surface irregularities with Nano-sized HA that would leave no efficient indentation for micromechanical retention of the cement layer. We coated the surface of zirconia with Nano-HA on the basis of a thermal protocol. Nano-particle provided a thin and uniform controllable coating layer with adequate strength. Also, AL-Murad LM et al. and Abdulkader SW et al. reported that addition of Nanohydroxyapatite (nHAp) to Self-Adhesive Resin Cement reinforces the mechanical features and bond strength to zirconia [29, 30]. There is limited scientific evidence comparing the amount of surface roughness produced via Er,Cr:YSGG laser

irradiation and Al₂O₃ particle abrasion. An SEM observation revealed more surface irregularities with 50 µm Al₂O₃ abrasion than the Er, Cr: YSGG laser (2W and 3W) treated Y-TZP surface [26]. Although specific surface roughness tests are required for more accurate conclusion on differences in surface roughness, the higher values of µSBS in group 3 of the present study, in comparison to group 4, could be as a result of better micromechanical bond. While several studies have investigated the impact of Al₂O₃ particle abrasion and its influencing factor on the sintered zirconia surface roughness [31]. In the present study, particle size was utilized. The data of the previous studies reported that the 50-µm Al₂O₃ particle abrasion could result in elevating flexural strength because of forming compressive fields owing to induced tetragonal–monoclinic transformation of crystals present in the surface [32]. However, such transformation and formation of small flaws might compromised the fatigue strength of Y-TZP restoration in long term [33]. In a work by Wegner and Kern [34], the phosphate ester monomer of MDP exhibits a water resistant durable chemical bonding. The MDP monomer with bonding agents reportedly could boost the bond strength of sandblasted zirconia with resin cement [35]. Hence, we recruited Panavia F2.0 in our experiments. The SEM images captured to analyze the failure mode revealed that the majority of fractures were adhesive in solely air abraded group (group 1) and other groups coated with HA were showed mixed fracture more than group 1. It can be related to the high quality of chemical bond of resin cement with coated HAp Zirconia [19]. The present paper is a continuation of previous work on improvement the bonding characteristic of Zirconia ceramics [26, 28]. Similarly, Azari et al. concluded that failure patterns in the HAp coated group were mixed and adhesive in the sandblasting, and control [19]. SEM observations also revealed that some of adhesive failures in Er, Cr: YSGG group. While it seems that HA coatings can be considered for preparation of zirconia surfaces before using luting cements, the effect of temperature, moisture environment, and cyclic loads is still unclear on this modality of surface modification. In addition to more in vitro evidence on the applicability of this technique, further clinical investigations are required in this regard. The main limitation of this research was to achieve a uniform HA coating on the zirconia. It was a technique sensitive procedure. The use of shear bond strength test due to easy application protocol was a limitation of this study [28].

5. Conclusion

The following conclusions were obtained in the present work:

- The µSBS of zirconia is improved with HA coating surface treatment.
- Improvement of µSBS with HA surface treatment is comparable with air borne particle abrasion.
- To improve bond strength, there is no significant additional benefit in combining air borne particle abrasion or laser irradiation with HA coating.

HA coating might be a promising alternative method for air borne particle abrasion for zirconia surface

Declarations

Ethics approval and consent to participate

Not applicable.

Consent for publication

Not applicable.

Availability of data and materials

All data generated or analysed during this study are included in this published article.

Competing interests

The authors have no relevant financial or non-financial interests to disclose.

Funding

No funding was received to assist with the preparation of this manuscript.

Authors' contributions

All authors contributed to the study conception and design. Material preparation, data collection and analysis were performed by [Faezeh Atri], [Vania Rasaie] and [Sakineh Nikzad Jamnani]. The first draft of the manuscript was written by [Saba Mohammadi] and all authors commented on previous versions of the manuscript. All authors read and approved the final manuscript.

Acknowledgements

Not applicable

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Figures



Figure 1

The micro-shear test

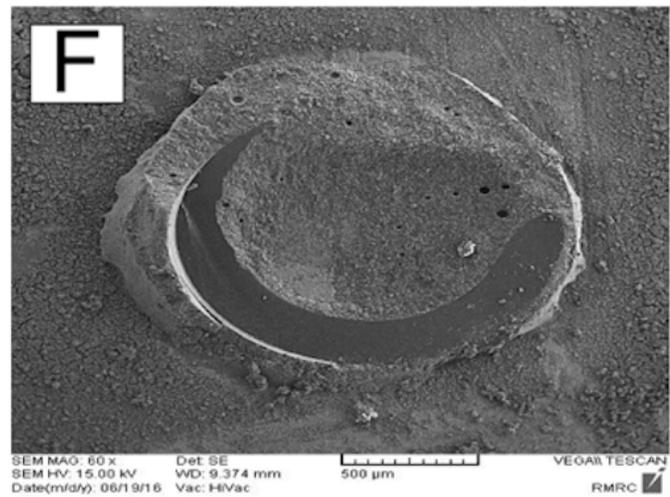
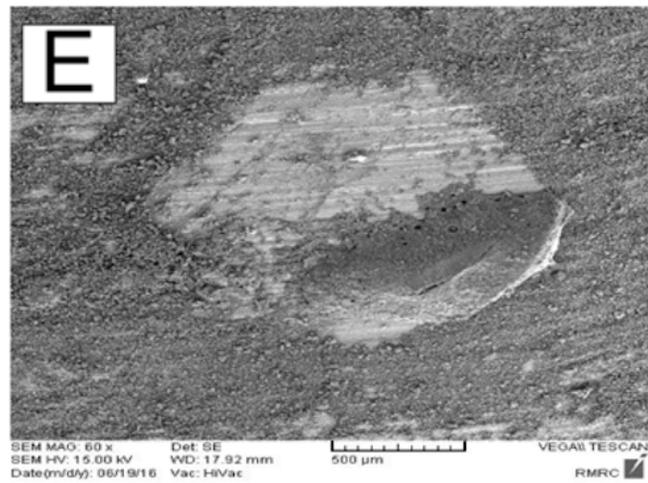
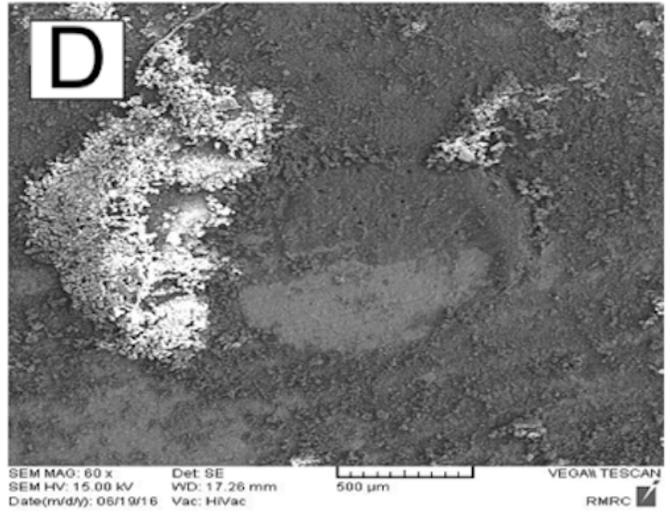
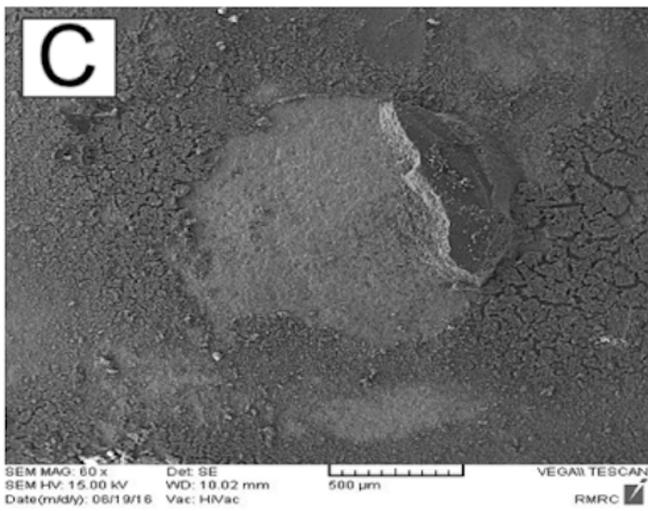
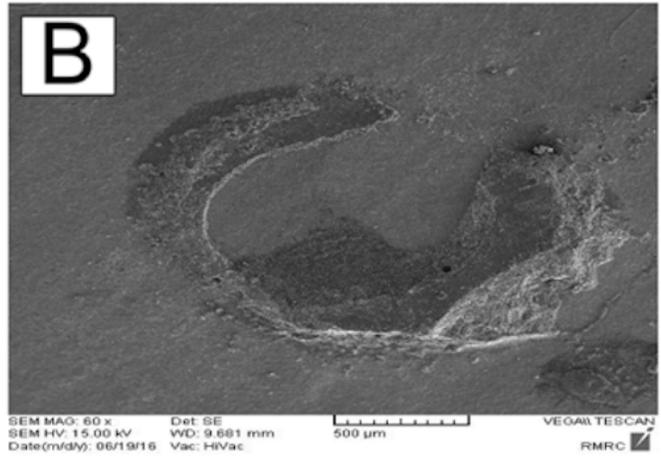
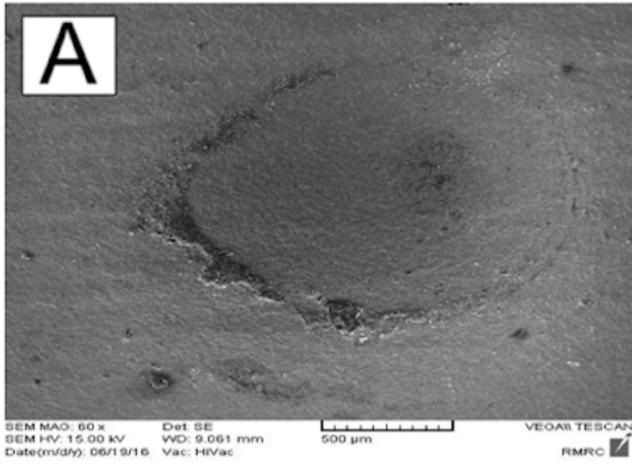


Figure 2

Al₂O₃ particle abraded group (group 1). A: Adhesive failure mode. B: mixed failure mode

Mixed failure mode C: sandblast + HA coating group (group 3) D: HA coated group (group 2)

Er, Cr: YSGG + HA coating group (group 4). E: Mixed failure mode. F: Cohesive failure mode