Original Article

Effect of different air-abrasion particles on metal-ceramic bond strength

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Abstract *Background/purpose:* Chipping and delaminating of veneering ceramics are critical problems in the fabrication of metal-ceramic restorations. The purpose of this study was to evaluate the effect of different air-abrasion particles on the shear bond strength of a ceramic to nickel-chromium (Ni-Cr) and cobalt-chromium (Co-Cr) alloys.
Materials and methods: Forty square specimens (10 mm × 10 mm × 1.5 mm) were cast for each Ni-Cr and Co-Cr alloys. Specimens were divided into four groups for the air-abrasion procedures: 50-μm aluminum oxid particles (Al_2O_3), 110-μm Al_2O_3 particles, 30—50-μm synthetic diamond particles, and 60—80-μm cubic boron nitride particles. After the air-abrasion procedures, specimens received ceramic veneering. The shear bond strength was determined at a crosshead speed of 1 mm/min. Data were statistically analyzed by a 2-way analysis of variance with post-hoc Tukey's test ($\alpha = 0.05$). Effects of the air-abrasion particles on each alloy were examined with a scanning electron microscope.
Results: The highest bond strengths were obtained with application of air abrasion with 110-μm Al_2O_3 particles and the lowest bond strengths were obtained with 50-μm Al_2O_3 particles ($P < 0.05$).
Conclusions: None of the tested alternative air-abrasion particles provided superior bond strengths compared with 110-μm Al_2O_3 particles.
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Introduction

Noble metal alloys are widely used for porcelain veneering in dentistry.[1] However, with continuing price fluctuations of noble metals, more attention has been given to alternative alloys. Base-metal alloys are economical alternatives to expensive gold alloys.[2] These alloys allow the fabrication of

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thinner infrastructures because they have greater rigidity, which is related to the modulus of elasticity. Nickel-chromium (Ni-Cr) and cobalt-chromium (Co-Cr) alloys are the most widely used when cost and rigidity are considered.[3,4]

The longevity of metal-ceramic restorations depends on the formation of a stable adhesive layer between the two materials. The adhesion mechanism between the metal and ceramic has not been completely defined, but it is believed to generally result from suitable oxidation of the metal and interdiffusion of ions between the metal and ceramic.[5-8]

Stress concentrations during ceramic cooling can result in ceramic chipping, either immediately or with a delayed response.[9] Chipping and delaminating of veneer ceramics are critical problems in fabricating metal-ceramic restorations, for both base-metal and noble-metal alloys. The primary requirement for the success of a metal-ceramic restoration is the development of reliable bonding between the veneering ceramic and alloy.[10-12]

Bond strength is determined by many factors: the strength of the chemical bonds, mechanical interlocking, the type and concentration of defects at the interface, wetting properties, and the degree of compressive stress in the veneer layer due to differences in the coefficients of thermal expansion between the metal and veneering ceramic.[13,14]

Airborne-particle abrasion of bonding surfaces increases the metal surface energy by improving the wettability of the opaque ceramic and consequently, the bond strength, through micromechanical bonding.[15] Aluminum oxide (Al_2O_3) particles are the most commonly used air-abrasion particles for this purpose. In dentistry, one alternative air-abrasion particle, synthetic diamond particles, which are used to increase the surface roughness and surface area, was previously applied to a glass-infiltrated aluminous oxide ceramic surface (In-Ceram, Vita Zahnfabric, Bad Sackingen, Germany).[16]

The search for alternative super-hard materials, some of which might even be harder than diamond, has stimulated research on high-pressure materials for more than 50 years. High-pressure synthesis on an industrial scale is applied to obtain synthetic diamonds and cubic boron nitride (CBN), which are super-hard abrasives of choice for cutting and shaping hard metals and ceramics.[17,18] CBN is the hardest material presently available, next to diamond, and may be an other alternative material for air-abrasion procedures.

Metal-ceramic crown applications have been a successful dental restoration for more than 30 years; however, questions remain about the optimal conditions for the alloy surface during application of an opaque ceramic and the firing procedures. Many studies focused on metal-free restorations, although the use of metal-ceramic restorations continues, additional investigations of metal-ceramic bonds are indicated.[19] However, little is known about the effects of different mechanical surface pretreatment methods and the bond strengths of ceramic to Ni-Cr and Co-Cr alloys.

The purpose of this study was to evaluate the shear bond strengths of a ceramic to four differently treated Ni-Cr and Co-Cr alloys. The research hypothesis was that the alternative air-abrasion particles tested would improve the shear bond strengths of the ceramic-to-metal surface.

Materials and methods

The materials used in this study are presented in Table 1. Eighty wax patterns (sculpting wax FC; Bego, Bremen, Germany) were fabricated from a silicone mold with dimensions of 10 mm × 10 mm × 1.5 mm. Wax patterns were invested in a phosphate-bonded investment (Multi-Vest, Dentsply International, York, PA, USA). Forty Ni-Cr and 40 Co-Cr alloys were prepared to complete the castings. The alloys were cast with an induction casting machine (Fornax, Bego). The complete castings were divested and the sprue was removed. Any remaining investment was carefully removed by air abrasion with 50-μm Al_2O_3 (Korox 50, Bego). Metal surfaces were finished by grinding with a 600-grit silicone carbide abrasive paper (3M ESPE, St. Paul, MN, USA) for 10 seconds at 300 rpm on a grinding machine (Buehler Metaserv, Buehler, Germany) under running water, ultrasonically cleaned for 15 minutes in distilled water, and then air-dried.

All specimens were divided into 4 groups, each containing 10 specimens for the air-abrasion procedures. The groups included 50-μm Al_2O_3 particles (Korox 50) (Group K1), 110-μm Al_2O_3 particles (Korox 110) (Group K2), 30—50-μm synthetic diamond particles (Micron + MDA) (Group D), and 60—80-μm CBN particles (ABN 600) (Group CBN). Air-abrasion procedures were performed using an intraoral air-abrasion device (Microetcher, Danville Engineering, San

Table 1	Materials used in the study.	
Material	Type of material (in wt%)	Manufacturer
Wiron 99	Ni-Cr alloy (Ni 65, Cr 22.5, Mo 9.5, Nb 1, Si 1, Fe 0.5)	Bego, Bremen, Germany
Wirobond C	Co-Cr alloy (Co 61, Cr 26, Mo 6, W 5, Si 1, Fe 0.5, Ce 0.5, C max 0.02)	Bego, Bremen, Germany
IPS d.SIGN	Veneering ceramic (SiO_2 50—65, Al_2O_3 8—20, K_2O 7—13, Na_2O 4—12, CaO 0.2—6, P_2O_5 0.2—5, F 0.1—3, additives 0.0—3.0)	Ivoclar Vivadent, Schaan, Liechtenstein
Korox 50	50 μmAl_2O_3 particles	Bego, Bremen, Germany
Korox 110	110 μm Al_2O_3 particles	Bego, Bremen, Germany
Micron + MDA	30—50 μm synthetic diamond particles	Elementsix, Shannon, Ireland
ABN 600	60—80 μm cubic boron nitride particles	Elementsix, Shannon, Ireland

Al_2O_3 = aluminum oxide.

Table 2   Firing cycle of IPS d.SIGN ceramic.

Material	Dt (min)	T↑ (°C)	T1 (°C)	T2 (°C)
Opaque layer 1	6	60	450	899
Opaque layer 2	6	60	450	899
Body ceramic	4—6	60	450	869

Dt = Drying time; T↑ = heating temperature per minute; T1 = initial temperature under vacuum; T2 = final temperature under vacuum.
  Source: Ivoclar Vivadent, Schaan, Liechtenstein.

Ramon, CA, USA) at an air pressure of 315 Pa for 15 seconds at a distance of approximately 10 mm.

After the air-abrasion procedures, specimens were ultrasonically cleaned for 1 minute in distilled water and air-dried and then received treatments of veneering. Ceramic sintering programs are listed in Table 2. No initial oxidation step was performed on the base-metal alloy specimens before application of the opaque porcelain. Thicker oxide layers occur with nickel- and cobalt-based alloys because they contain elements that easily form oxides during the initial step of oxidation.[19] According to the manufacturer, the development of a heavy oxide layer at this stage can obstruct the mechanical bond. A thin layer of opaque was applied to the metal surface, followed by a second layer and two dentine body layers, each of which was separately fired according to the manufacturer's instructions (IPS d.SIGN, Ivoclar Vivadent, Schaan, Liechtenstein). The ceramics were applied to the metal specimens with the aid of a polytetrafluoroethylene mold (Isoflon, Diemoz, France) to standardize their dimensions. The mold consisted of two independent pieces. The first layer of dentin ceramic was applied and the mold was carefully removed and fired as recommended by the manufacturer. Then a second layer of dentin ceramic was applied and fired to compensate for ceramic shrinkage. A 3-mm layer of veneering ceramic was fired, covering an area of 5 mm × 10 mm at one edge of the face (Fig. 1). After application of the ceramic, each specimen was embedded in an acrylic resin mold and seated in a shear test jig. Specimens were stored in distilled water at 37°C for 24 hours before the shear bond strength test.
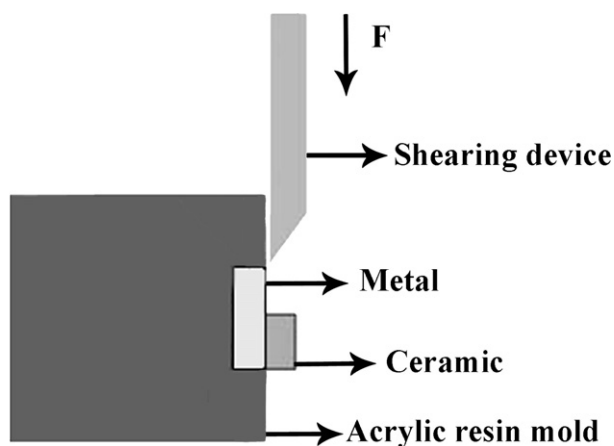

Figure 1   Test design (F = shear force).

A universal test machine (Lloyd LRX; Lloyd Instruments, Fareham, Hampshire, UK) was used for the shear bond strength test at a crosshead speed 1 mm/min. Specimens were loaded to failure by applying a shear force to the veneering material at the alloy interface. Data were statistically analyzed. The Kolmogorov-Simirnov test showed that the data had a normal distribution ($P > 0.05$). A homogeneity of variance test was done using Levene's test (F ratio = 2.748, $P < 0.05$). Means and standard deviations of bond strengths were calculated and mean values were compared by two-way analysis of variance (SPSS 12,0; SPSS, Chicago, IL, USA), followed by a multiple-comparison test performed using post-hoc Tukey's test. Statistical significance was set at $\alpha = 5\%$.

All specimens were visually inspected for the fracture mode (cohesive, adhesive, or mixed). The fracture mode was classified as cohesive if the failure occurred within the body of the veneering ceramic, adhesive if the failure occurred at the junction of the metal and the veneering ceramic, and mixed if adhesive failure occurred between the veneer and metal together with cohesive fracture of the veneering ceramic.

To evaluate the effects of air-abrasion particles on the surface morphology of the Ni-Cr and Co-Cr alloys, an additional five specimens (one for a control after-casting surface) for each alloy were treated with the same experimental protocol as described above. After casting, the surface was not used for shear testing but was presented as a reference for the prepared surfaces. All specimens were coated with gold using a sputter coater (S150B; Edwards, Crawley, UK) and examined under a field emission scanning electron microscope (SEM) (JSM-6335F; JEOL, Tokyo, Japan) at 20 kV. SEM photomicrographs were developed at 500× magnification for visual inspection.

Results

Mean values and standard deviations of the shear bond strengths are presented in Table 3. Results of the two-way analysis of variance showed that interactions between air-abrasion particles and the metal alloy were not significant ($P > 0.05$) (Table 4). There was no significant difference between the Ni-Cr and Co-Cr alloys ($P > 0.05$). The statistical analysis revealed that air-abrasion particles had a significant influence on the shear bond strength of the ceramic-to-metal surface ($P < 0.05$). The bonding strength depended on the type and particle size of the air-abrasion particles. Air abrasion with 110-μm Al_2O_3 particles showed higher bond strengths of the ceramic-to-metal surface, and no significant differences were found compared with air abrasion with 60—80-μm CBN particles ($P > 0.05$) in each metal group.

Air abrasion with 50-μm Al_2O_3 particles showed lower bond strengths and no significant differences were found when compared with 30—50-μm synthetic diamond particles ($P > 0.05$) in each metal group.

In Groups K2 and CBN, four of the specimens in each alloy fractured within the opaque (cohesive mode), whereas the rest fractured at the alloy-ceramic material interface (adhesive mode) (Table 5).

SEM photomicrographs of the air-abraded metal alloy surfaces are presented in Figs. 2 and 3. The topographic

Table 3 Mean shear bond strength values in MPa for ceramic bonded to metal specimens.

Air-abrasion particles	Mean (SD)	
	Ni-Cr	Co-Cr
50 μm aluminum oxide	40.48 (4.17) A a	41.73 (3.77) A ab
30—50 μm synthetic diamond	44.21 (2.01) B ab	45.39 (1.67) B b
60—80 μm cubic boron nitride	51.87 (3.49) C c	52.72 (3.73) C c
110 μm aluminum oxide	52.52 (2.77) D c	54.55 (3.54) D c

*Values having same letters (lowercase: within groups; uppercase: between groups) were not significantly different for post-hoc Tukey's test ($P > 0.05$).
Co = cobalt; Cr = chromium; Ni = nickel; SD = standard deviation.

patterns differed among specimens air-abraded with Al_2O_3, synthetic diamond, and CBN particles. The air-abrasion particles showed similar effects on both metal alloys. The SEM photographs showed that the air-abrasion procedure modified the surface topography of the metal by increasing the irregularities on the surface compared with the surface after casting (Figs. 2A and 3A). Air abrasion with 50-μm Al_2O_3 (Figs. 2B and 3B) and 30—50-μm synthetic diamond particles (Figs. 2C and 3C) created similar rough surfaces. Air abrasion with 60—80-μm CBN (Figs. 2D and 3D) created macro-irregular surfaces. Air abrasion with 110-μm Al_2O_3 (Figs. 2E and 3E) created more-irregular and rougher surfaces than with the other particles.

Discussion

The data support rejection of the hypothesis that the alternative air-abrasion particles tested would improve the shear bond strengths of the ceramic-to-metal surface. Air abrasion with different particles, which have different particle sizes and forms, affected the shear bond strengths ($P < 0.05$). Shear bond strengths were significantly higher in groups K2 and CBN than in groups D and K1 ($P < 0.05$). Although the highest shear bond strength was obtained in Group K2, no significant differences were found when compared with Group CBN.

It is difficult to compare the results of the present study with those obtained in other studies because different methods were used to evaluate the metal-ceramic bond strength. There are questions regarding the testing methods for evaluating the actual strength of metal-ceramic bonds, because a method that can accurately measure this property is unknown. Some authors stated that there is no methodology capable of measuring only shear forces along the metal-porcelain interface.[2,20,21]

Hammad and Talic[20] carried out a critical analysis of tests used for metal-ceramic systems. These authors stated that shear tests with a flat interface mostly direct the tension to the interface and does not evaluate the elasticity modulus of the metal as in flexural tests.[20] According to ISO 9693, which uses a three-point bending test rather than shear bond strength used in the present study, the mean debonding strength/crack initiation strength should be >25 MPa to meet ISO requirements.[22]

De Melo et al.[19] evaluated the shear bond strength between porcelain and four alloys (two Ni-Cr and two Co-Cr alloys). The authors reported 54.0 MPa and 63.0 MPa for shear bond strengths of the Ni-Cr alloys and 71.7 MPa and 55.2 MPa for the Co-Cr alloys. The specimens were air-abraded with 100-μm Al_2O_3 for 10 seconds at a 2-cm distance with 210 Pa pressure and at 45°angle. The authors concluded that none of the base-metal alloys studied demonstrates superior bond strength to the porcelain tested.[19]

Joias et al.[9] evaluated the shear bond strengths of a dental ceramic to five Co-Cr alloys. They reported shear bond strengths of 94.0 MPa, 96.8 MPa, 75.1 MPa, 71.2 MPa, and 63.2 MPa. Specimens were air-abraded with 110-μm Al_2O_3 for 10 seconds at a distance of 2 cm under 551580.6 Pa of pressure. They used a modified apparatus to test the shear bonding, based on a study by de Melo et al.[19] They concluded that the bond strength of a dental ceramic to a Co-Cr alloy is dependent on the alloy composition.[9]

Neto et al.[15] evaluated the metal-porcelain bond strength of three ceramic systems associated with three Ni-Cr alloys and one experimental Co-Cr-Ti alloy. They reported bond strengths of 22.08—44.38 MPa with the Ni-Cr alloys. They used a modified rectangular parallel shear test, which determined the shear strength required to break the metal-ceramic bond of a ceramic ring constructed around cylindrical metal rods. This test was based

Table 4 Results of 2-way analysis of variance.

Source of variation	Sum of squares	df	Mean square	F ratio	P
Metal alloy	35.245	1	35.245	3.325	>0.05
Air-abrasion particle	2136.934	3	712.311	67.195	<0.001
Air-abrasion particle × metal alloy	3.746	3	1.249	0.118	>0.05
Error	763.253	72	10.601		
Total	186750.730	80			

Table 5 Failure types.

Groups	Ni-Cr			Co-Cr		
	AD (%)	CO (%)	MI (%)	AD (%)	CO (%)	MI (%)
K1	100	—	—	100	—	—
D	100	—	—	100	—	—
CBN	60	—	40	60	—	40
K2	60	—	40	60	—	40

AD = Adhesive failure; CBN = cubic boron nitride; CO = Cohesive failure; Co = cobalt; Cr = chromium; MI = Mixed failure; Ni = nickel.

on the test proposed by Shell and Nielsen.[23] The area for porcelain bonding was air-abraded with 50-μm Al_2O_3. They concluded that the bond strengths of the three ceramic systems to the Ni-Cr and Co-Cr-Ti alloys significantly varied, indicating that metal/ceramic compatibility was very important for bond strength.[15]

Pretti et al.[24] evaluated the shear bond strength of a metal-ceramic bond of two Co-Cr alloys. They reported shear bond strengths of 48.39 MPa and 55.96 MPa for the two alloys and stated that no significant difference was observed. The metal surfaces were air-abraded with 100-μm Al_2O_3 for 10 seconds at a distance of 2 cm in their study.[24]

Çiftçi et al.[25] evaluated the shear bond strengths of four esthetic veneering materials on a Ni-Cr alloy. They reported a shear bond strength of 34.96 MPa for the ceramic veneer. The alloy surface was air-abraded with 50-μm Al_2O_3 for 15 seconds at a 5-mm distance under an emission pressure of 0.5 MPa.[25]

It is obvious that different bond strengths are expected with different test methods. In contrast to a previous study,[26] which reported that the grain size of Al_2O_3 had only

a little effect on the bond strength, higher bond strengths were obtained in the present study with larger particle sizes. Although the particle sizes of CBN (60—80 μm) were smaller than those of the 110-μm Al_2O_3 particles, similar bond strengths were obtained. This result may have been due to the hardness of the CBN particles. The hardness of Al_2O_3 particles was lower than that of synthetic diamond abrasives and CBN. According to Mohs' scale, hardness values of Al_2O_3, CBN, and synthetic diamond abrasives are 9, 9.9, and 10 respectively.[17] It was also shown and verified by SEM photomicrographs in the present study that air-particle abrasion with 60—80-μm CBN (Figs. 2D and 3D) created nonuniform rough surfaces compared with 110-μm Al_2O_3 particles (Figs. 2E and 3E). But this nonuniform rough surface was sufficient to increase the bond strength. Analysis of the fracture type revealed that 40% of specimens presented with mixed fracture in Groups CBN and K2. This is evidence of higher bond strengths between the metal and ceramic. The mechanism responsible for this strength increase is probably the roughness causing the fracture path to deviate into the porcelain. Furthermore, interfacial roughness causes the fracture path to deviate,
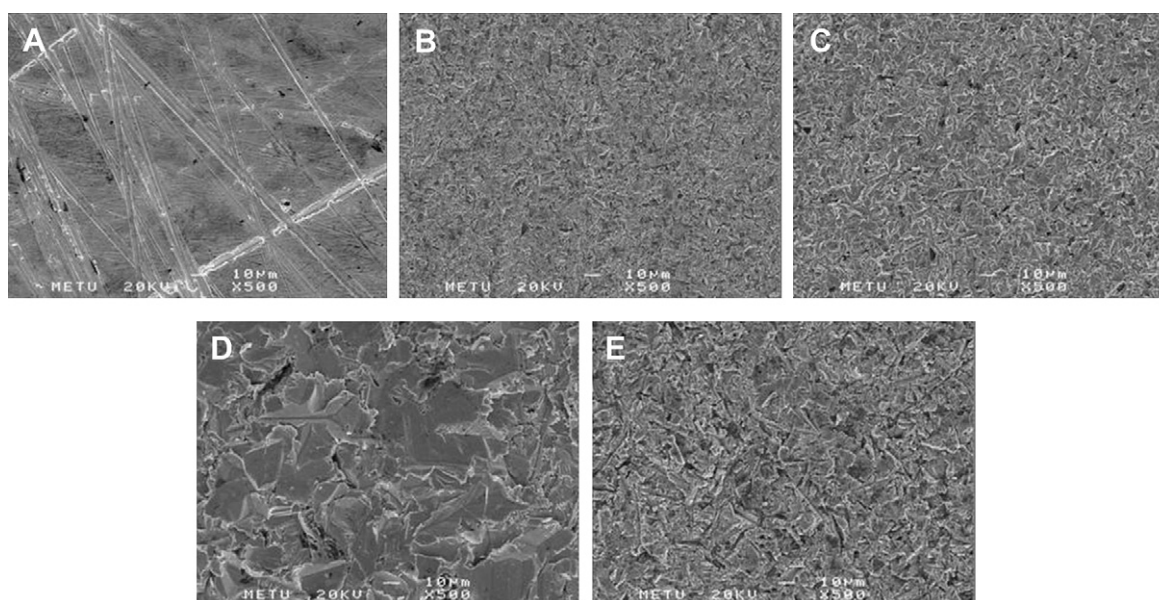
Figure 2 Scanning electron microscope images of the Ni-Cr alloy surface after different surface pretreatments. (A) After-casting surface; (B) air abrasion with 50 μm Al_2O_3; (C) air abrasion with 30—50 μm synthetic diamond particles; (D) air abrasion with 60—80 μm CBN; (E) air abrasion with 110 μm Al_2O_3. After-casting surface (A) was not used for shear testing but presented for the reference to the prepared surfaces. Original magnification 500×; bar 10 μm.
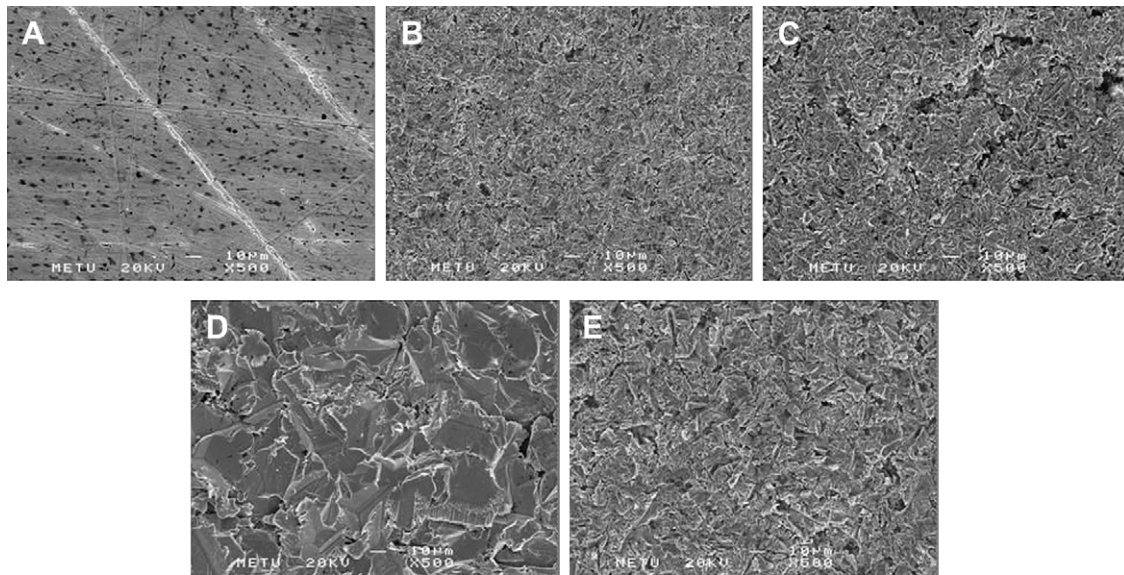
Figure 3 Scanning electron microscope images of the Co-Cr alloy surface after different surface pretreatments. (A) After-casting surface; (B) air abrasion with 50 μm Al_2O_3; (C) air abrasion with 30—50 μm synthetic diamond particles; (D) air abrasion with 60—80 μm CBN; (E) air abrasion with 110 μm Al_2O_3. After-casting surface (A) was not used for shear testing but presented for the reference to the prepared surfaces. Original magnification 500×; bar 10 μm.

increasing the fracture surface area and the total energy required for failure.[27]

Most of the specimens tested in this study failed at the alloy-opaque interface (adhesive mode), suggesting that the oxide layer was weaker than the ceramic. Chemical bonds and mechanical interlocking are believed to play the most prominent roles in the bond strength of a ceramic to a metal.[23,28] The most important is the chemical type; and an oxide layer is responsible for adhering ceramic to metal. Sandblasting was shown to affect the oxide layer thickness; the oxide layer formed before sandblasting differed from the one obtained after sandblasting.[29] It is well known that the formation of metal oxides during the oxidation process is dependent on the alloy composition and surface treatment.

Brantley et al.[29] showed that the formation of metal oxides during the oxidation process varies depending on the type of alloy, the method of surface finishing, and the duration of the oxidation process.

Although bond strength data like these can be helpful at the clinical level for selecting the most suitable materials for use, it is also important to point out that the framework strength does not exclusively depend on the alloy used but also on its design. Sharp shapes often have very low resistance to fracture. Therefore, alloys for use in metal-ceramic restorations cannot be chosen by clinicians exclusively based on material bond strengths.[11]

Although there is a relationship between high bond strength values and compatibility between the metal and ceramic, wet-fatigue studies may provide simulations of bonding at the interface.[30] The thickness and composition of the oxide layer after different air-abrasion particles are used on both Ni-Cr and Co-Cr alloys should also be investigated in a future SEM study using high magnification and energy-dispersive spectrometric analyses. The surface roughness and volume loss from the metal surfaces were not investigated in the present study. Atomic force microscopy can be used in a future study to investigate changes in the surfaces.

Within the limitations of the present study, the following conclusions were drawn. No statistically significant differences were observed between the two tested alloys. None of the tested alternative air-abrasion particles provided superior bond strengths compared with 110-μm Al_2O_3 particles. Further evaluation of the effects of alternative air-abrasion particles on bond durability is required before making any clinical recommendations.

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