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The Shear Bond Strength of Porcelain Bonding to Cobalt-Chromium Dental Alloys Before and After Thermal Cycling

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Abstract: Metal-ceramic crowns and bridges provide a stable and aesthetic tooth reconstruction. Hot pressing of veneering ceramics to metal substructure minimizes the formation of pores. Within the oral cavity, prosthetic structures are exposed to thermal stress. The study examines the influence of temperature changes on the quality and durability of the metal-ceramic bond using thermocyclic loading. Prior to the ceramic pressing, 40 cylindrical alloy specimens (height: 15 mm, diameter: 7 mm) were treated with abrasive blasting using Al_2O_3 (250 µm) or SiC (250 µm). They were divided into four groups: two control groups (sandblasted with Al_2O_3 or SiC) and two groups subjected to thermocyclic simulation. The ceramic-to-metal bond strength was measured with a shear test before and after thermocyclic loading. A statistically significant reduction in the bond strength between the metal substructure and the pressed ceramic was found after thermocyclic exposure. The bond strength decreased by 57% for Al_2O_3 sandblasted specimens and 36% for those sandblasted with SiC, as against the control group. This indicates that thermocycling weakens the bond between the metal alloy and pressed ceramic. The SiC blasting of the cobalt-chromium alloy results in a higher shear bond strength of the material connection after thermocyclic stimulation than the abrasive blasting with Al_2O_3.

Keywords: shear strength; dental porcelain; metal-ceramic; thermocycles; abrasive blasting; joint

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1. Introduction

Metal-ceramic crowns and bridges, used as permanent replacements for lost tooth hard tissues, typically consist of an internal metal substructure (that provides structural strength and flexibility) and an external ceramic veneering layer (that delivers an aesthetic finish). This type of prosthetic restoration is widely preferred by both patients and prosthodontists due to their advantageous cost-to-property ratio.

The clinical success of a metal-ceramic connection largely depends on the effectiveness of the bond between the ceramic and metal structure [1,2]. Factors directly influencing the ceramic-metal alloy connection include the oxide layer, which enables the chemical bond and is formed on the alloy surface through the oxidation process [3], and microretentive cavities, which enhance mechanical bonding and are created through abrasive blasting. These microcavities allow the liquid ceramic to flow and anchor within the surface irregularities. Another factor is the proper matching of the coefficients of thermal expansion (CTE) of both materials. CTE is responsible for the induction of compressive stress at the material

interface [4]. The optimal thickness of the oxide layer affects the bond strength between the metal base and the dental ceramic, as it is responsible for the mutual diffusion of atoms between both materials. The dimensions of the microretentive cavities created through abrasive blasting are influenced by factors such as the type and size of the abrasive material, as well as the parameters of the blasting process (the working pressure—air pressure; the angle of incidence; the distance between the nozzle; and the workpiece). In dental laboratories, aluminum oxide (Al_2O_3) with a grain size of 110–250 µm is typically used for abrasive blasting under a pressure of 0.3–0.4 MPa. These parameters are considered standard as they provide optimal conditions for achieving strong adhesion between the veneering ceramic and the metal substructure. In practice, other parameters, such as the angle of incidence and the distance between the material surface and the nozzle, are generally considered less critical due to the fact that it is impossible to maintain their constant level while processing complex geometries of prosthetic components. Nevertheless, the abrasive treatment significantly enhances the bond by creating microretentive cavities that increase the actual contact surface area between the ceramic and metal. This larger interface improves both the mechanical interlocking and the chemical bonding potential of the materials. Additionally, abrasive treatment increases the surface energy and enhances wettability, creating more favorable conditions for liquid ceramics to flow into surface irregularities and leading to satisfactory adhesion [4]. For bonding components generated by thermal shrinkage, optimal conditions are achieved when the thermal expansion coefficient of the ceramic is approximately 0.5×10^{-6} K^{-1} lower than that of the metal alloy onto which it is fired [5]. When this condition is met, the ceramic layer shrinks slightly during the cooling process, inducing mild compressive stresses that, apart from enhancing the durability and integrity of the bond, also counteract tensile stresses induced by mechanical loading [2,5].

Prosthetic structures are subjected to a range of mechanical stresses, including biting forces and chewing loads, which encompass both static and dynamic stresses as well as fatigue loads. The durability of these structures and their susceptibility to damage under such conditions remain critical areas of ongoing research [6]. One of the most frequent failures observed in ceramic-metal prostheses is the delamination and chipping of the veneering ceramic from the metal substructure [7]. Alongside mechanical stresses, these structures are also subjected to thermal loads. Temperature fluctuations, such as those experienced during the consumption of hot and cold foods and beverages, as well as the disparities in thermal conductivity and thermal expansion between the ceramic and metal components, can significantly influence the performance and durability of the ceramic-metal bond.

Although the durability of the discussed bond is influenced by three primary factors: chemical connection, mechanical interlocking, and thermal shrinkage stress, the most critical is the second one. Proper surface preparation promotes the flow of liquid ceramics into surface irregularities, facilitating stronger mechanical adhesion. While aluminum oxide (corundum) is the most commonly used abrasive material for surface treatment, other materials, such as silicon carbide (SiC), have shown potential benefits. SiC grains have sharper edges, making them more effective in surface preparation than corundum. Given that the most common failure in metal-ceramic prosthetic restorations occurs at the metal/ceramic interface, the aim of this study was to evaluate the impact of the abrasive material on the bond strength between a cobalt-chromium alloy and veneering ceramic. Recognizing the variable temperature conditions within the oral environment, the study included not only standard strength tests but also strength evaluations following exposure to thermal cycling.

2. Materials

Magnum Nitens cobalt-chromium alloy (Degudent GmbH, Hanau, Germany) was used for the research. The alloy contains, according to the manufacturer's data, 63 wt% of cobalt and 28 wt% of chromium. The remaining 9 wt% are admixtures of molybdenum, tungsten, silicon, niobium, and iron. Thermal expansion coefficient: 14.5×10^{-6} K^{-1} (25 ÷ 500 °C), 14.7×10^{-6} K^{-1} (25 ÷ 600 °C). The pressed ceramics used in the tests were Cergo Kiss ceramics (Degudent GmbH, Hanau, Germany) with an expansion coefficient of 14.1×10^{-6} K^{-1} (25 ÷ 500 °C) and 14.3×10^{-6} K^{-1} (25 ÷ 600 °C).

3. Research Methodology

Forty cylindrical material specimens were prepared with a height of 15 mm and a diameter of 7 mm. They were divided into two subgroups: A (n = 20) and B (n = 20). Prior to the ceramic pressing process, one of the front surfaces of each specimen underwent abrasive blasting. Aluminum oxide was used as the abrasive material in subgroup A, whereas silicon carbide was used to sandblast specimens from subgroup B. In both cases, the abrasive material had a grain size of 250 μm. The abrasive blasting was performed at a pressure of 0.4 MPa, with the working nozzle positioned approximately 50 mm from the material surface. The nozzle axis was oriented at an angle of approximately 45° relative to the specimen. Following abrasive blasting, any loosely bound abrasive grains that were not embedded in the metal surface were removed using a pressurized steam generator. The specimens were then thoroughly dried. Part of these specimens (n = 10 from subgroup A and n = 10 from subgroup B) was utilized for surface characterization tests, including roughness and free surface energy measurements. On the remaining specimens (n = 10 from subgroup A and n = 10 from subgroup B), Cergo Kiss ceramics (Degudent GmbH, Hanau, Germany) was pressed in a multi-step process. An opaquer layer was first applied in two firing stages on one of the frontal surfaces of each specimen. Subsequently, a 2 mm thick wax layer was applied over the opaquer. A sprue was then attached to each specimen axially in order to prevent investment fracture during the pressing stage. Sprues were approximately 6 to 8 mm long and had a 7 mm diameter. The ceramic mass was then pressed under vacuum conditions at a temperature of 950 °C, with the pressing process lasting 45 min. After cooling, the samples were released from the investment material. Residual fragments of the investment were completely removed using glass beads with a grain size of 50 μm, applied under a working pressure of 0.2–0.3 MPa. Thus, prepared specimens were subsequently subjected to technological shear tests.

Thermocyclic loading involved alternating immersion of the specimens in water baths at 5 °C for 20 s and at 60 °C for 20 s, with a 5 s transition period in-between. These thermocycles were performed in order to simulate the temperature fluctuations that occur in the mouth during food consumption [8,9] and respiration [9].

The testing was conducted using the THE-1100 thermocycler (SD Mechatronics, Feldkirchen-Westerham, Germany). Following the standard recommendations, a total of 5000 thermocycles were performed [10]. Thus, prepared specimens, both before and after thermocyclic loading, were evaluated for the strength of the ceramic/metal bond. The strength was assessed using a shear test in the Zwick Z020 device (Zwick-Roell, Ulm, Germany), with a crosshead displacement speed of 2 mm/min.

The results of the strength tests were statistically analyzed. The shear strength values of the specimens that were not subjected to thermocyclic loading were compared to those that underwent thermocycling. Statistical analysis was performed using the Statistics v. 12.5 software package. To evaluate the shear strength differences between groups, an ANOVA test was conducted, as the data met the assumptions required for this test. A significance level of alpha = 0.05 (p = 0.05) was adopted for all evaluations. The fragments of the

samples obtained after the endurance tests were further analyzed through fractographic studies using the HITACHI S-3000N scanning electron microscope (Hitachi, Ltd., Tokyo, Japan). The aim was to investigate the nature and origin of the fractures. The surfaces of the fragments were examined using backscattered electron imaging at a magnification of 500×. Additionally, maps of element distribution on the fractured surfaces were created using NORANs EDS (Energy Dispersive X-ray Spectroscopy, Thermo Electron Scientific Instruments LLC, Madison, WI, USA) analyzer, integrated with the scanning electron microscope and VANTAGE control and analysis software (Ventage 3.1, Thermo Fisher Scientific, Waltham, MA, USA).

4. Results

Table 1 presents the results of the strength tests for the pressed ceramic and cobalt-chromium alloy bond. Tables 2–5 provide the *p*-values for individual comparisons between the different groups. The collective graphic analyses are illustrated in Figure 1.

Table 1. Results of the shear strength tests of Co-Cr conection after abrasive treatment using Al_2O_3 and SiC, without and with thermocyclic loading.

Process Type	Mean (MPa)	Standard Deviation SD (μm)
Al_2O_3 without thermocycling	8.31	2.93
Al_2O_3 with thermocycling	3.51	1.34
SiC without thermocycling	8.37	2.19
SiC with thermocycling	5.25	1.21

Table 2. Comparison of the influence of aluminum oxide and silicon carbide abrasive treatment on the bond strength of specimens without thermocycling ($p < 0.05$).

	Al_2O_3 Without Thermocycling	SiC Without Thermocycling
Al_2O_3 without thermocycling	-	0.6932
SiC without thermocycling	0.6932	-

Table 3. Comparison of the influence of thermocycling on the bond strength of specimens treated with aluminum oxide ($p < 0.05$).

	Al_2O_3 Without Thermocycling	Al_2O_3 with Thermocycling
Al_2O_3 without thermocycling	-	0.0059
Al_2O_3 with thermocycling	0.0059	-

Table 4. Comparison of the influence of thermocycling on the bond strength of specimens treated with silicon carbide ($p < 0.05$).

	SiC Without Thermocycling	SiC with Thermocycling
SiC without thermocycling	-	0.0122
SiC with thermocycling	0.0122	0.0434

Table 5. Comparison of the influence of aluminum oxide and silicon carbide abrasive treatment on the bond strength of specimens after thermocycling ($p < 0.05$).

	Al_2O_3 with Thermocycling	**SiC with Thermocycling**
Al_2O_3 with thermocycling	-	0.0434
SiC with thermocycling	0.0434	-

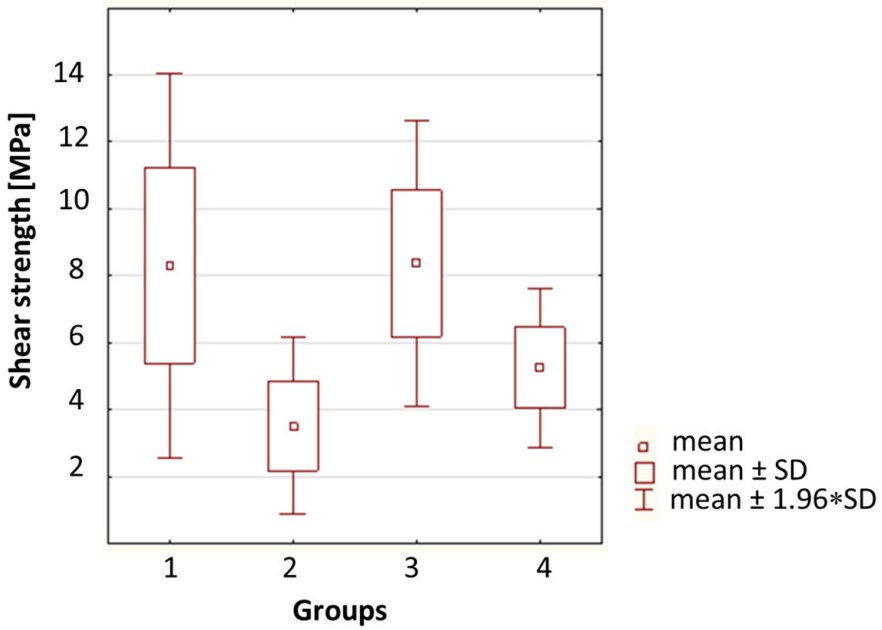


Figure 1. Shear strength results depending on the type of abrasive material used. **1**—Al_2O_3; **2**—Al_2O_3 with thermocycling; **3**—SiC; **4**—SiC with thermocycling.

Based on Levene's test, it was found that the variances were homogeneous ($p < 0.05$). To assess statistically significant differences, the Anova test and Tukey's test were used for unequal frequencies.

- Al_2O_3 vs. Al_2O_3 after thermocycling—$p < 0.05$, where higher values were obtained for Al_2O_3
- Al_2O_3 vs. SiC after thermocycling—$p < 0.05$, where higher values were obtained for Al_2O_3
- Al_2O_3 after thermocycling vs. SiC—$p < 0.05$, where higher values were obtained for SiC

The results of the research indicated that the bond strength between pressed ceramics and metal is consistent and independent of the abrasive material used for surface treatment of the metal alloy. The shear strength of the Co-Cr connection after abrasive treatment with Al_2O_3 (0.4 MPa, 110 μm) and pressed ceramics was 8.3 ± 2.9 MPa, while the same measurement for the Co-Cr connection treated with SiC (abrasive treatment at 0.4 MPa) and pressed ceramics was 8.4 ± 2.2 MPa. Statistical analysis revealed no significant differences.

Specimens subjected to thermocyclic loading exhibited a decrease in the ceramic-to-metal shear bond strength compared to the specimens that did not undergo thermocycling. The bond strength values of the Co-Cr-ceramics after thermocyclic treatment were equal to 3.5 ± 1.3 MPa for specimens with Al_2O_3 surface abrasion and 5.2 ± 1.2 MPa for specimens where the alloy surface was treated with SiC. The shear bond strength in the specimens subjected to thermocycles decreased by 57% for the Al_2O_3-treated material and 36% for the specimens where the metal was treated with SiC, compared to the strength of the samples

without thermocycling. Statistical analysis revealed a statistically significant difference between the metal/ceramic shear bond strength in the specimens without thermocyclic loading and in those subjected to thermocycles. This was observed for both researched abrasive materials used for sandblasting the specimens. Additionally, differences in shear bond strength were noticed between the specimens subjected to thermocycles. The specimens in the silicon carbide abrasive treatment group exhibited significantly greater variations in bond strength compared to the aluminum oxide-treated specimens.

Figures 2 and 3 demonstrate that the majority of the area is dominated by the metal alloy elements, chromium and cobalt. When comparing the fractured surfaces before and after thermocyclic loading, it can be observed that the area occupied by silicon (the ceramic component) is smaller after thermocycling than without it. This indicates that the cracking primarily occurs at the metal/ceramic interface, with only minimal fracture within the ceramic material itself.

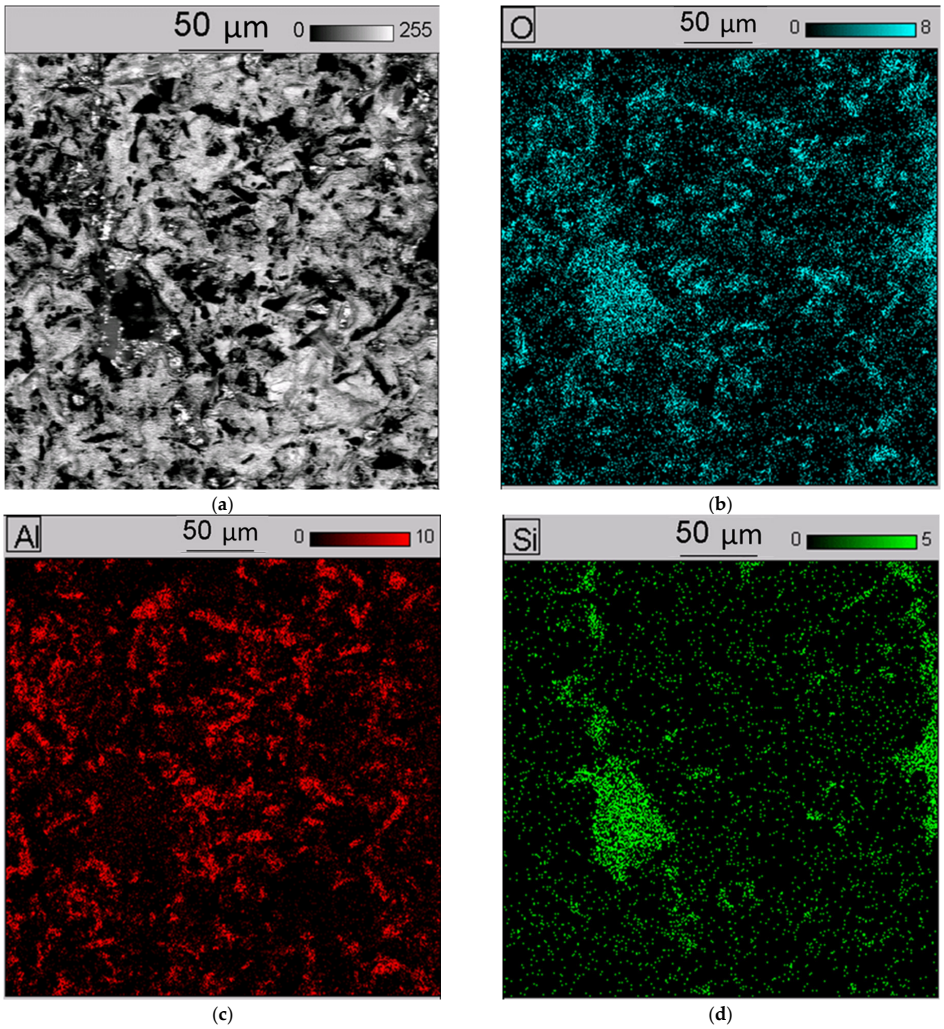
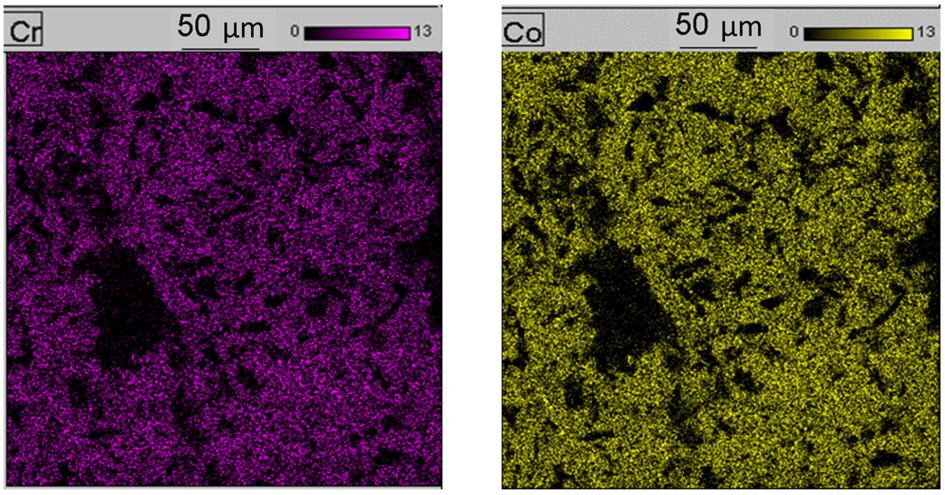
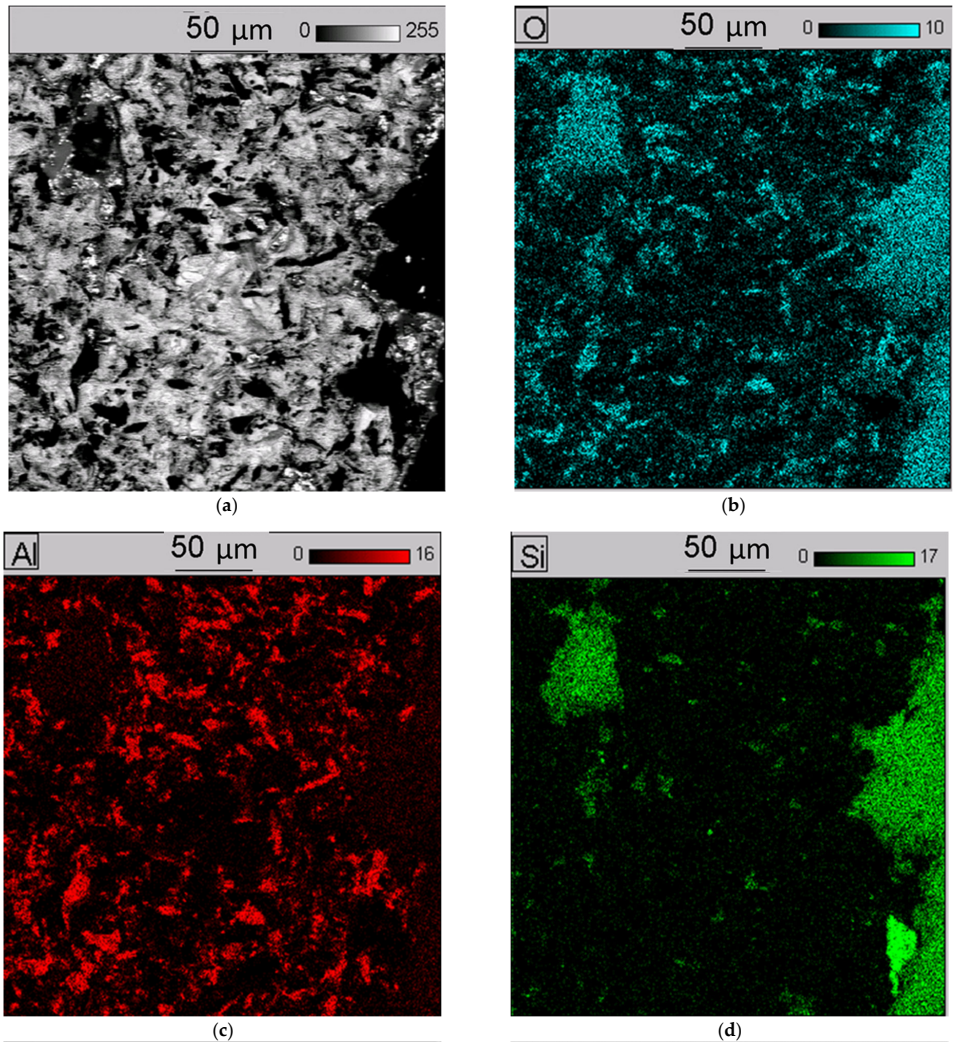


Figure 2. *Cont.*



(**e**) (**f**)

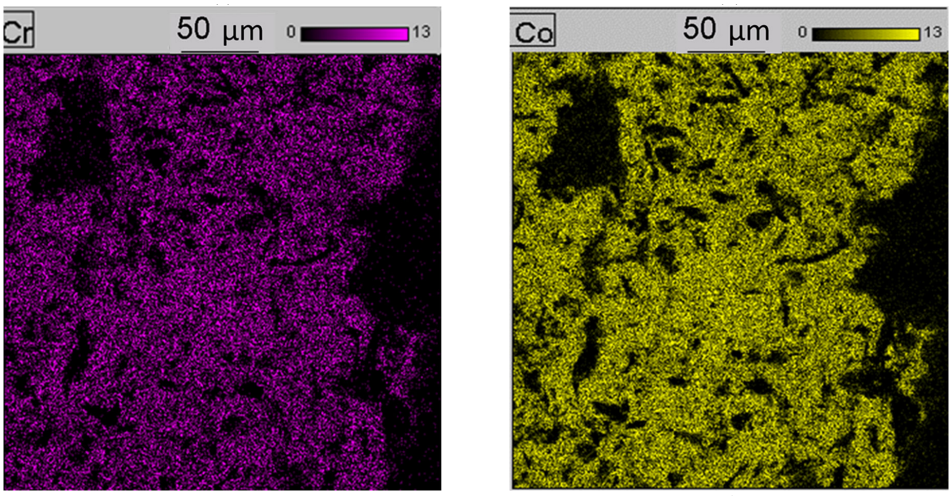
Figure 2. Fragments of specimens abrasively treated with Al_2O_3 without thermocycles: (**a**) Microscopic image of the fractured fragment of the specimen in backscattered electrons (BSE); (**b**) Surface distribution of oxygen in the area shown in (**a**); (**c**) Surface distribution of aluminum in the area shown in (**a**); (**d**) Surface distribution of silicon in the area shown in (**a**); (**e**) Surface distribution of chromium in the area shown in (**a**); (**f**) Surface distribution of cobalt in the area shown in (**a**).



(**a**) (**b**)

(**c**) (**d**)

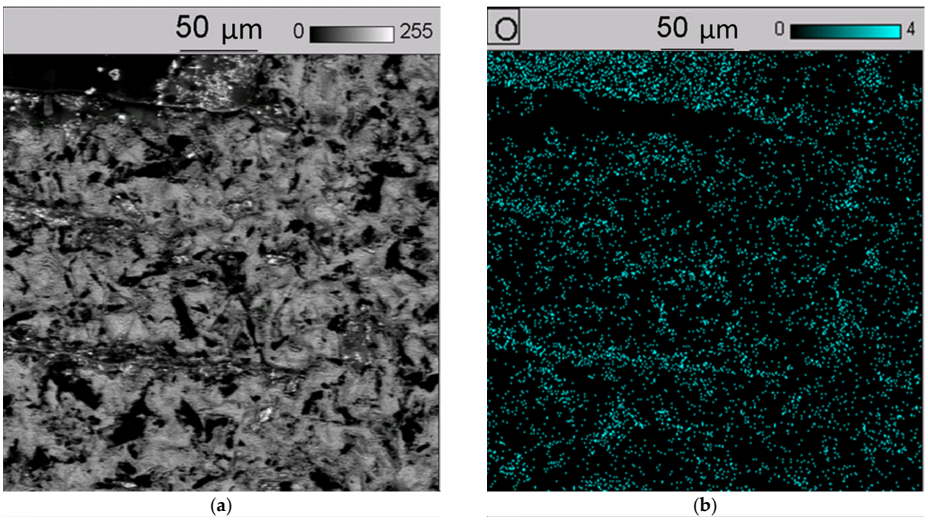
Figure 3. *Cont.*



(**e**) (**f**)

Figure 3. Shows fragments of specimens abrasively treated with Al_2O_3 with thermocycles: (**a**) Microscopic image of the fractured fragment of the specimen in backscattered electrons (BSE); (**b**) Surface distribution of oxygen in the area shown in (**a**); (**c**) Surface distribution of aluminum in the area shown in (**a**); (**d**) Surface distribution of silicon in the area shown in (**a**); (**e**) Surface distribution of chromium in the area shown in (**a**); (**f**) Surface distribution of cobalt in the area shown in (**a**).

Figures 4 and 5 illustrate that the area occupied by cobalt and chromium, the alloy components, is much larger than the area occupied by silicon and aluminum, the ceramic components. However, before thermocyclic loading, the aluminum, silicon, chromium, and cobalt were more dispersed compared to their distribution after thermocyclic loading (Figures 4c–f and 5c–f). Without thermocycles, the areas occupied by aluminum and silicon (ceramic components) are larger on the surfaces of specimens treated with Al_2O_3 (Figure 2c,d) compared to those treated with SiC (Figure 4c,d). After thermocyclic loading, the areas occupied by aluminum and silicon (ceramic components) are larger on the surfaces of the SiC-treated specimens (Figure 5c,d) than on the Al_2O_3-treated specimens (Figure 3c,d).



(**a**) (**b**)

Figure 4. *Cont.*

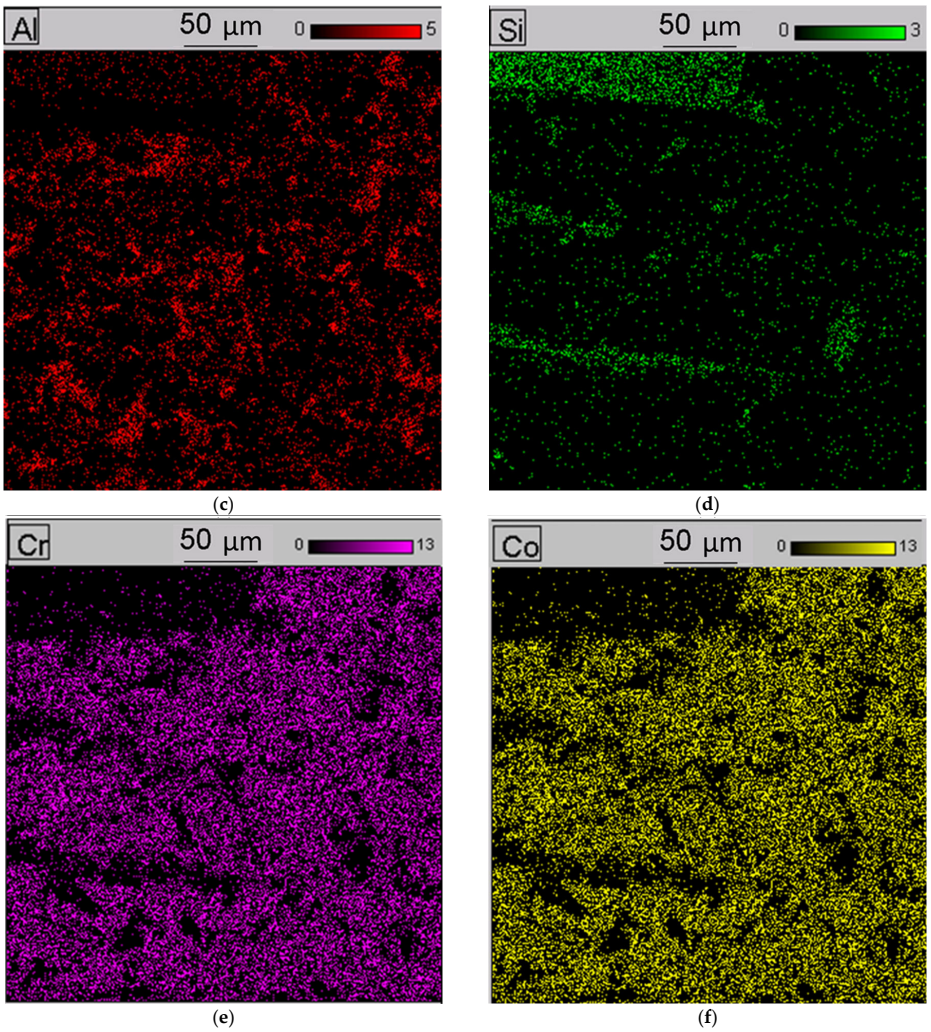


Figure 4. Shows fragments of specimens abrasively treated with SiC without thermocycles: (**a**) Microscopic image of the fractured fragment of the specimen in backscattered electrons (BSE); (**b**) Surface distribution of oxygen in the area shown in (**a**); (**c**) Surface distribution of aluminum in the area shown in (**a**); (**d**) Surface distribution of silicon in the area shown in (**a**); (**e**) Surface distribution of chromium in the area shown in (**a**); (**f**) Surface distribution of cobalt in the area shown in (**a**).

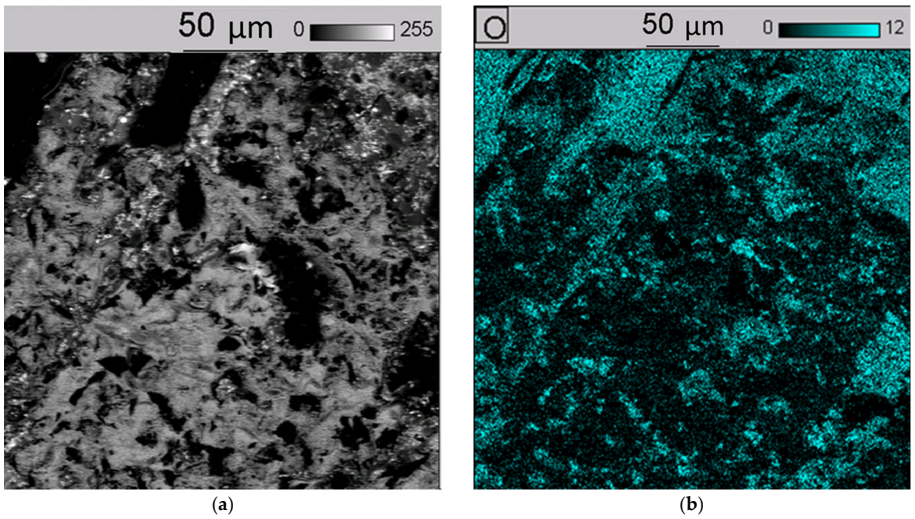


Figure 5. *Cont.*

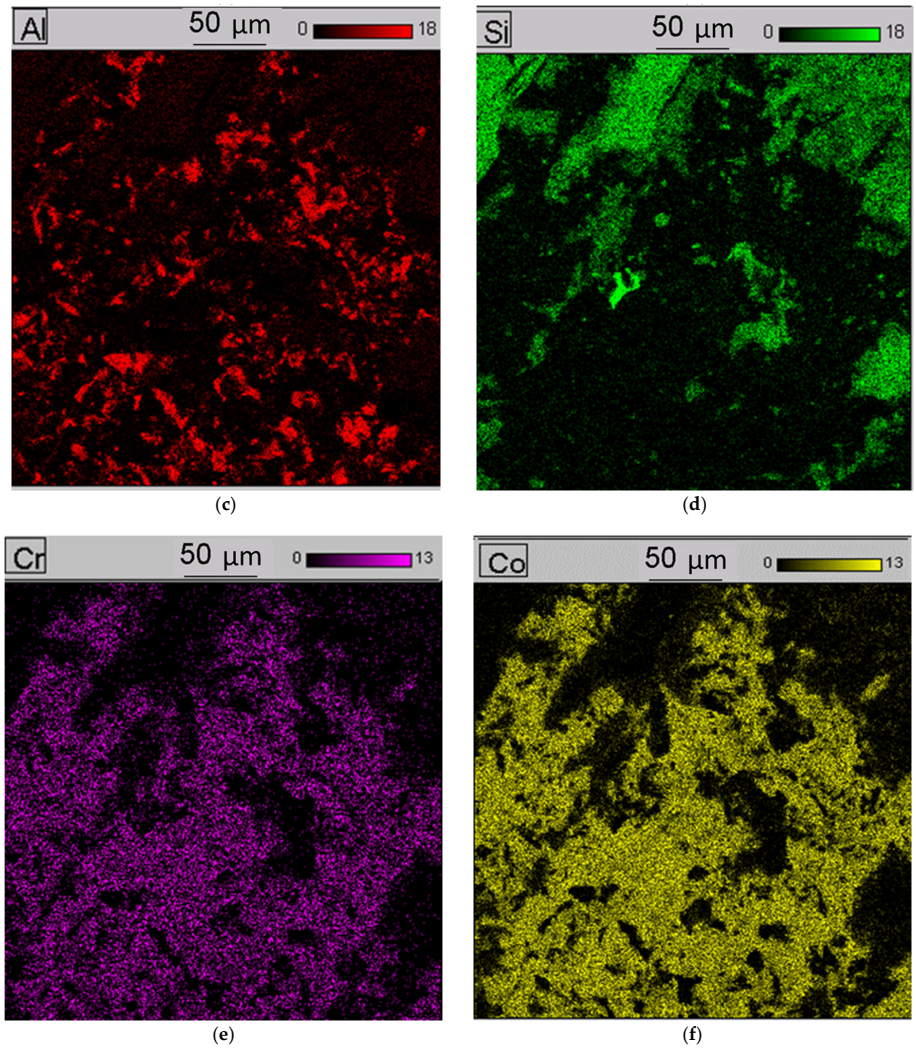


Figure 5. Shows fragments of specimens abrasively treated with SiC with thermocycles: (**a**) Microscopic image of the fractured fragment of the specimen in backscattered electrons (BSE); (**b**) Surface distribution of oxygen in the area shown in (**a**); (**c**) Surface distribution of aluminum in the area shown in (**a**); (**d**) Surface distribution of silicon in the area shown in (**a**); (**e**) Surface distribution of chromium in the area shown in (**a**); (**f**) Surface distribution of cobalt in the area shown in (**a**).

5. Discussion

The literature indicates that in prosthetics the metal-ceramic connection is mostly subjected to shear stress, which is why a technical shear test was used to assess the bond strength. In practical applications, particularly when fabricating multilayer joints, detailed observations regarding the nature of joint decohesion and the impact of technological factors are critical for ensuring optimal performance. The recommends evaluating the ceramic/metal bond strength using a three-point bending test [11]. However, this method primarily measures the force required to fracture the ceramic/metal bond by delaminating the ceramic layer rather than providing a clear determination of the connection's strength due to various stress conditions during the fracturing process. The destructive force recorded during the test may result in a fracture at the ceramic/metal interface, a fracture within the ceramic itself, or a simultaneous combination of both. The authors of the study [12] highlight that the bending method is characterized by low repeatability of results and a weak correlation between the bending force and adhesion strength. In contrast, the shear test provides a reliable means of assessing the relationship between bond strength and technological parameters.

From the perspective of the long-term performance of permanent prosthetic restorations, analyzing the factors influencing the ceramic-to-metal bond is crucial, as one of the most common failures involves the delamination or chipping of the ceramic layer from the metal base. Material factors, including the type of materials used, their thermal properties, and surface preparation methods, play an important role [13,14]. For instance, Schmage et al. [15], in their study on the durability of the bond between metal clasps of partial dentures and dental ceramics, found that shear strength is influenced not only by the surface roughness of the bonded materials but also by the method used to achieve this roughness.

An essential factor to consider is the connection's resistance to the oral environment, including variations in food temperature and humidity. Consequently, it was deemed necessary to evaluate the impact of thermocyclic simulation on the strength of the cobalt-chromium alloy and pressed ceramic bond. The chosen number of thermocycles in this study, 5000, aligns with parameters reported in other research papers [16–18] and corresponds to approximately five years of use in ceramic-metal restorations.

The test results for specimens not subjected to thermocyclic loads indicate that the shear strength of the metal/ceramic bond does not exhibit statistically significant differences, irrespective of the type of abrasive material used for surface preparation. Currently, there are no established guidelines specifying the required strength for such connections. Consequently, it is challenging to assess whether values in the range of 8–9 MPa are adequate. A point of reference could be the strength values recommended for bonding metal alloys with fired ceramics, where the acceptable threshold is 25 MPa [11]. The obtained strength values appear to be significantly lower. However, the recommended value applies to strength tests conducted using the three-point bending method. Based on available research on correlations between results obtained through different testing methods, the values observed in this study should be multiplied by a factor of $2 \div 2.5$ to provide a comparable estimate [19]. After applying the correction factor, the adjusted strength values range between 16 and 20 MPa, which appear to be acceptable. This is particularly reasonable when the fact that the semi-liquid state of the ceramic during the pressing process with a significantly higher viscosity than the fired liquid ceramic is taken into consideration. The higher viscosity of the pressed ceramic reduces its ability to flow into surface irregularities, leading to less effective mechanical anchoring and, consequently, lower bond strength.

The simulation of temperature fluctuations in a thermal cycler significantly reduced the shear strength of the evaluated metal/ceramic system (Table 1). This aligns with findings of M. Antanasov et al. [18,20], who demonstrated that thermo-mechanical cycling substantially diminishes the shear strength of ceramic/metal connection, regardless of the fabrication method of the metal cobalt-chromium alloy (e.g., casting, milling, and selective laser melting). Their research suggests that the reduction in strength is likely related to cracks forming at the ceramic/metal interface, which weaken the bond. Metal and ceramics have different coefficients of thermal expansion, thermal conductivity, and mechanical properties (modulus of elasticity, plasticity). This phenomenon cannot be avoided. The guidelines for manufacturing metal-ceramic restorations even recommend slight differences in thermal expansion coefficients in order to create compressive stresses via shrink-fit mechanisms. Metals, with higher thermal conductivity, heat and cool more rapidly than ceramics, generating thermal stresses deriving from volume differences. These stresses can initiate microcracks, which propagate with repeated thermal cycling. Additionally, the mismatch in the modulus of elasticity between the metal and ceramic exacerbates the stress during mechanical loading [17,21]. Thermal fluctuations are especially pronounced in the external parts of the prosthetic restorations, which directly contact hot or cold substances.

Research has shown that the outer layers of veneering porcelain in those parts are most prone to damage [22].

Fractographic studies confirm that the metal/ceramic interface is the weakest link in the system. Analysis of the fractured surfaces reveals that the metal portion (characterized by the presence of Co and Cr on the elemental distribution maps) constitutes a significantly larger area than the regions containing ceramic components (Al, Si, O). This indicates that the majority of the observed fractures are adhesive in nature, with only a minor portion being cohesive within the ceramics. When comparing fracture surfaces of specimens without and with thermocycling, it can be noticed that the latter exhibit larger areas containing ceramic elements (Al, Si, and O) (Figures 2b–d, 3b–d, 4b–d and 5b–d). This suggests an increase in cohesive fractures through the ceramic material in specimens subjected to thermal cycling. This phenomenon can be explained by the fact that the thermal stresses mainly affect the ceramic component, which is brittle and, contrary to metal, lacks the capacity for plastic deformation. The predominance of the crack occurrence within the ceramics aligns with their higher share in the final elemental diffusion of the fractured specimens. Nevertheless, the presence of adhesive fractures at the metal/ceramic interface highlights that this part remains the weakest.

Given the observed reduction in the metal/ceramic bond strength after thermocycling, it is essential to establish specific requirements for the connection's resistance to temperature fluctuations, similarly to the standard, which recommends the minimum strength of this connection [11]. Such standards would be particularly relevant given the exposure of prosthetic restorations in the oral cavity to variable thermal loads. This consideration is critical because thermal loads combine with the mechanical stresses arising from chewing forces, and both of these phenomena can occur simultaneously.

6. Conclusions

1. Thermocyclic simulation reduces the strength of the metal-ceramic bond.
2. The weakest part of the connection is the metal/ceramic interface.
3. In all cases, the damage to the structure predominantly occurs as adhesive failure at the metal/ceramic interface.
4. When designing metal/ceramic connections, it is essential to evaluate their strength after thermal fluctuation exposure.
5. The use of SiC, compared to Al_2O_3, gives a better bond between metal and ceramics.

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