Analysis of the connection of a nickel-chromium alloy with dental ceramics after aluminum oxide abrasive blasting

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Abstract. Fixed prosthetic restorations in the form of ceramic crowns or bridges are characterized by good aesthetics and durability. The most important turn out to be the mechanical attachment of the ceramic material in the unevenness of the metal, formed after abrasive blasting. The aim of this research is to investigate the impact of abrasive blasting of a nickel-chromium alloy on the covering the unevenness with ceramics. In the research was used a Ni-Cr alloy (Heraenium® NA, Hera). The metal surface was blasted with alumina of variable size (50, 110 and 250 µm) and pressure (0.4 and 0.6 MPa), which was then fired with ceramics in the form of 2 layers of IPS Classic Opaque (Ivoclar Vivadent). Samples were hot mounting and cut to obtain a cross-section of the connection. The samples was examined under a Scanning Electron Microscope. The results show the presence of air bubbles at the metal-ceramic interface and in the layers of ceramic material. The photographs show a difference in the appearance of the unevenness of the metal surface by comparing samples after blasting with an abrasive of different sizes. No significant differences in the appearance of metal roughness are observed when comparing samples after treatment with the same grain size and different pressures.

1. Introduction

Ceramic crowns and bridges on a metal substructure are one of the dental restorations made in dental prosthetics. They are characterized by good aesthetics and durability [1]. The bond strength between the metal and the ceramic material is associated with the occurrence of several mechanisms that are responsible for creating a strong connection. One of them concerns the presence of chemical bonds between materials that are formed due to the presence of an oxide layer on the metal surface. Another owes its presence to differences in coefficients of thermal expansion, where during cooling of the restoration after firing ceramics, it has compressive stress, increasing the connection strength. The last mechanism concerns the anchoring of the ceramic material in the unevenness of the metal, formed after abrasive blasting. Preparation of the surface of the metal restoration for the joint is one of the main principles aimed at ensuring the permanent use of the restoration. It is known that the shear strength of the connection between metal and ceramics depends on the development of the surface, which is why works are carried out where Co-Cr alloy [2], [3] or titanium [4] is analyzed. Sandblasting is also used in the development of zirconia surface, which is commonly used in the creation of substructures for ceramic restorations, due to its good aesthetic properties and biocompatibility. The durability of the connection is influenced by the development of the material surface, i.e. the size and shape of the unevenness created. To provide them, various treatments are used, such as chemical surface etching or mechanical roughness formation [5], [6]. The process of mechanical surface development can be performed by machines with rotary instruments or sandblasting. A combination of these treatments is
also used to ensure the best possible surface properties [7]. Abrasive blasting directly affects the formation of the right surface roughness and the parameters used also affect its wettability or the amount of abrasive particles stuck in it. From the dental point of view, surface properties that give information about its Surface Free Energy and the wetting ability of the liquid may be an important aspect. Wetness of the surface with liquid ceramics is also important for the quality of the connection. When firing a ceramic material, depending on the surface properties, it may cover to varying degrees of metal unevenness leading to a reduction in joint durability. The influence of metal surface preparation for connection with dental ceramics is described in the literature, where its strength is analyzed in correlation to the condition of the substructure surface. The purpose of our research was to expand knowledge about the connection by analyzing its appearance for the surface of a Ni-Cr alloy subjected to various blasting processes.

2. Materials and methods
Six cylindrical samples made of nickel-chromium alloy (Heraenium® NA, Heraeus Kulzer), whose diameter was 8 mm and height 7 mm was used in the test. The surfaces were blasted with alumina with variable parameters, where the processes differed in the pressure used (0.4 MPa, 0.6 MPa) and the abrasive grain size (50 µm, 110 µm, 250 µm). The treatment lasted 20 seconds and the nozzle distance from the material surface was 15 mm. The angle between the nozzle and the metal for all samples was 45°. After sandblasting, the samples were cleaned in an ultrasonic cleaner (Emmi-55HC-Q, Emag) in deionized water for 8 minutes, and then dried under compressed air. Dental ceramics in several layers were fired on prepared and cleaned surfaces. The first two were the opaque IPS Classic Opaquer (Ivoclar Vivadent) and the next one was IPS Classic Dentin (Ivoclar Vivadent) in the firing parameters given in Table 1.

| Table 1. Firing parameters for IPS Classic dental ceramics (Ivoclar Vivadent). |
|---------------------------------|-------------|-------------|-------------|-------------|
|                                 | The starting | The final   | Heating rate | Heating time |
|                                 | temperature | temperature | [°C/min]     | [min]        |
| Opaque layer                    | 403         | 980         | 80          | 1           |
| II opaque layer                 | 403         | 970         | 80          | 1           |
| I dentin layer                  | 403         | 920         | 60          | 1           |
| II dentin layer                 | 403         | 910         | 60          | 1           |

Then the samples were cut and included, and then metallographic specimens were created to ensure the observation of the metal-ceramics boundary. Chemical composition studies using X-ray microanalysis with EDS energy dispersion and observation of surface topography were carried out on a Scanning Electron Microscope (JSM-6610LV, JEOL).

3. Results
Point x-ray EDS microanalysis was performed in 3 places on the tested surface in order to distinguish between the present materials on the connection cross-section. At each of the analyzed points, elements characteristic of the materials used are observed (Table 2).

| Table 2. Elements in the material obtained from the EDS study. |
|---------------------------------------------------------------|
| Measuring point  | Current elements                         |
| Dentin           | O, Si, Al, K, Na                         |
| Opaque           | O, Si, Zr, K, Al, Na, Zn, Ca             |
| Ni-Cr alloy      | Ni, Cr, Mo, Si, Mn                       |

The results of the microscopic observation of the cross-section of the Ni-Cr-ceramic alloy connection are presented in the pictures from Figure 1 to Figure 6.
Figure 1. SEM photography of a metal-ceramic joint after treatment of a metal surface with 50 µm alumina at 0.4 MPa (approx. x800).

Figure 2. SEM photography of a metal-ceramic joint after treatment of a metal surface with 50 µm alumina at 0.6 MPa (approx. x800).

Figure 3. SEM photography of a metal-ceramic joint after treatment of a metal surface with 110 µm alumina at 0.4 MPa (approx. x800).

Figure 4. SEM photography of a metal-ceramic joint after treatment of a metal surface with 110 µm alumina at 0.6 MPa (approx. x800).

Figure 5. SEM photography of a metal-ceramic joint after treatment of a metal surface with 250 µm alumina at 0.4 MPa (approx. x800).

Figure 6. SEM photography of a metal-ceramic joint after treatment of a metal surface with 250 µm alumina at 0.6 MPa (approx. x800).
It is noticeable that the metal surface after blasting with alumina changes its structure due to the size of the abrasive used. For samples after sandblasting with the smallest particles (50 µm), the widths of the observed surface irregularities are much smaller, comparing them with the surface treated with grain size 110 and 250 µm. The use of higher pressure does not cause significant differences in the shape of the metal surface.

Figure 7. Surface maps of the elements on the metal-ceramic connection for sample A54.

Figure 7 contains surface maps of the elements included in the metal alloy, opaque and abrasive. On the border between metal and ceramics, an increased occurrence of aluminum is observed, which is caused by the presence of embedded abrasive particles.

4. Discussion

The study focused on the analysis of the boundary between a nickel-chromium alloy and ceramics, formed after firing a ceramic material on a metal surface. After the EDS analysis, it is possible to observe the presence of more elements contained in the first layers of ceramics and this is characteristic of this material. The opaque is a material that directly contacts the alloy, creating chemical bonds that affect the strength of the connection and covers the unevenness of the metal after abrasive blasting, creating mechanical catches [8]–[10].

Observation of the metal-ceramic connection in SEM photographs gives information on the occurrence of air bubbles in the ceramic material, where for each sample their quantity is small. The method of applying ceramic layers to the metal surface may be responsible for the main reason for their appearance in the structure. In this study, the opaque was used in the form of a paste, which is applied with a brush. This method of application is also used in creating the dentin layer. This involves the possibility of enclosing air in one of the layers (Figure 8) and this is a situation widely encountered, both in the case of research on ceramics [11]–[13] as well as in clinical cases related to failures [14].
Figure 8. SEM photography of a metal-ceramic joint after 250 µm grain processing and 0.6 MPa pressure. Arrows indicate the place of occurrence of air bubbles.

The ability of the liquid to wet the alloy surface is also significant. In prosthetics, when the ceramic material is applied to the metal, it is an aqueous suspension, so its character may be more polar. When firing ceramics, by increasing the temperature, water comes out of the material and changes the character of the material to apolar, so in this case, it is important to assess the ability of ceramics to wet the metal surface [7]. The presence of roughness can also affect the cover of semi-liquid ceramics in unevenness in a different way, which is associated with a change in the viscosity of the material as well as the occurrence of various development of the metal surface. The firing temperature of the ceramics also has an impact, where better wetting properties of the ceramics are observed at higher temperatures [7]. The SEM photographs show a change in the appearance of the metal surface with a change in the used abrasive particle size. For samples whose surface has been treated with alumina with a particle size of 50 µm observed surface irregularities, which are not deep. With the increase in the particle size of the abrasive, an increase in the depth of unevenness is noted, where for the largest grain the metal surface has the largest amount of splinters. The wettability of the surface according to the Wenzel model increases as its unevenness increases [15]. Therefore, the lower the surface roughness, the less the wetting ability of the liquid should be. In addition, according to Cassie and Baxter, the rough surface consists of a solid and air, because depending on the width and depth of the unevenness, when it is wetted, the air can be enclosed in the form of bubbles at the boundary of the material. Such a mechanism can occur for roughness, whose width is small and is characterized by a large depth. In the case of wider unevenness, the material or liquid wetting the surface will not lead to air clumping at the border. Therefore, the presence of roughness and nano-roughness causes the surface to become hydrophobic. This is due to the energy state of the surface [15]. However, the SEM observation does not show a tendency to increase the occurrence of air bubbles depending on the surface roughness. They are most likely the result of the above-mentioned possible causes. The wettability of the metal may be related to the strength of the described joint due to adhesive forces. Not without significance and in this case the surface roughness turns out. Research on the impact of variable abrasive blasting on the strength of a Co-Cr alloy connection was presented by Pietnicki et.al., where durability was less for samples after 50 and 250 µm particle treatment [2]. Similar results are also observed for titanium in the studies of Gołąbiowski et.al. [16]. For this material the lowest values are recorded for grains 50 and 250 µm, but also for the highest pressure used (0.6 MPa). Tests associated with the use of chemical etching surfaces in the case of titanium also results in a decrease in the strength of the metal-ceramic joint, which was
presented in the study of Parchańska-Kowalik et. al. [17]. Tests showed a reduction in connection durability from 32 MPa obtained for sandblasted surfaces to approx. 16 MPa for sandblasted and chemically etched surfaces. This may be due to the removal of abrasive particles from the surface, or the reduction of unevenness on the surface. The same relationship is observed for testing the strength of the connection between titanium and zirconia [18].

Observation of the surface of the Ni-Cr alloy in our study shows that for the smallest grain treatment the irregularities are not deep, and their width is small. In this case, the ceramics are not sufficiently hooked into the roughness, which is why the joint strength turns out to be smaller than in samples after 110 µm particle treatment. In the case of samples where the metal surface has been treated with the largest grain, it may turn out that the depth of the unevenness is adequate, but their width leads to a weaker attachment of the ceramic material to them. For the Co-Cr alloy from the Pietnicki test, the optimum surface unevenness is obtained for blasting with 110 µm abrasive.

To sum up, air bubbles are observed on the border of the tested materials, which are associated with the technique of applying ceramic material to the metal. The connection cross-section shows the presence of an abrasive embedded in the surface of a Ni-Cr alloy. No significant differences in the appearance of the metal surface are observed for samples treated with the same grain size but with different pressures.

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