MICROSCOPIC OBSERVATIONS UNDER THE EFFECT OF SURFACE PREPARATION PARAMETERS OF Ni-Cr-Mo ALLOY IN METAL-CERAMIC CONNECTION STABILITY TERMS

KLIMECKA-TATAR Dorota

Czestochowa University of Technology, Institute of Production Engineering, Czestochowa, Poland, EU,
klimt@wip.pcz.pl

Abstract

The connection between metal and ceramic occurs by physical, chemical and in small extent by mechanical forces, special attention is applied to the every stage of metal surface preparation before ceramics application. The paper presents the results of microscopic cross-sections observation of Ni-Cr-Mo coated with opaquer and dental ceramics layers. On the basis of variables sandblasting parameters found that this treatment tends to increase the diversity of the surface. Short term (because a 15-second) sandblasting makes the metal surface irregular, and the presence of hollows and hills on the surface contribute to increased adhesion between layers of opaquer and ceramic. The usage of longer sandblasting time makes that the irregularities are rather rifts and burrs. In addition, the dimensions of the resulting irregularities restrains the correct and accurate surface oxidation of the alloy, but also restrains the correct and even surface coverage within opaque Highly diverse of the alloy surface contributes to the numerous gaps, cavities and voids on the border of the metal - ceramic connection.

Keywords: Ni-Cr-Mo alloy, dental alloy, metal-ceramic system

1. INTRODUCTION

In dental technology the sandblasting technique is designed to clean the surface of the cast, to remove impurities and a byproduct after mechanical machining and heat treatment. Abrasive wear of the alloy surface caused by corundum particles striking help to roughness increase and development of the surface. Typically, combinations of metal with other materials have adhesive character, therefore, strongly developed and roughened surface enhances the strength of the connection [1-3]. Due to the fact that sandblasting has a significant impact on the quality of the metal-ceramic connection it seems to be interesting to verify how the time of sandblasting of the metal surface effect on the structure of such a connection.

Application of metals and their alloys used in dental technology is very common. Among the metallic materials used in dental technology can be mentioned: nickel, cobalt, chrome, gold, titanium and their alloys [1-3]. Metallic materials used in prosthetics are prepared by various methods include isostatic casting, using centrifugal force or the use of plastic forming techniques (squeezing and rolling), which entails many risks. The material in processes may be damaged, on the surface may appear casting cavity, cracks and impurities resulting from the chemical composition and other plasticity parameters [4-9]. This kind of defects may cause a rupture during plastic deformation, and the exploitation [10]. Therefore, so important is to plan appropriate surface treatments. In surface engineering on dental materials strengthening commonly used are surface machining, thermal treatment [11-13] as well as chemical and thermo-chemical treatments [14,15] Metal surfaces to ensure an appropriate level of aesthetics are faced and covered with ceramic and composite materials. One of the most frequently performed services in dental technology, where it meets the phenomenon of combination of metal with ceramics is to produce veneers, on a metallic base. The combination of metal with a ceramic occurs by physical, chemical, and low mechanical forces (on a metal surface is formed mechanical retention). The mechanical or adhesive connection is realized by the retention pearls or by the effect of metal surface sandblasting [16-18]. The metal selection should be guided by its chemical and physical properties. Metal to veneering with ceramic has to contain alloying elements which give the possibility to form an oxide layer which assist to produce chemical bond in conjunction with ceramic material. Preparation of the corresponding oxide layer is very important, therefore, a surfaces casts should be sandblasted to be cleaned from spontaneously arising oxides and other impurities. The surface cleaning ensures that during oxidation, a controlled amount of oxides created (recommended by the manufacturer). It can be argued that the ceramic particles of opaquer with metal oxides diffuse between them to create a combination and stable connection. Another important factor that affects on the metal - ceramic connection is suitable selection of thermal expansion coefficients between the metal and ceramic (coefficient of thermal expansion WAK and the linear coefficient of thermal expansion TEC). The range between the coefficients should be (0.5 - 1.5)10^{-6} K^{-1} at a temperature range of 25 - 500 °C [19]. The main objective of the work are the observations of metal-ceramics cross-sections micro-images. The samples were prepared from dental Remanium CSe alloy veneered with ceramics PS d. SIGN Ivoclar. 2.1. and the only variable was the time of metal surface sandblasting.

2. EXPERIMENTAL

2.1. Material characteristic

In the research the dental alloy Remanium CSe and ceramic IPS d. SIGN Ivoclar have been used. This Ni-Cr-Mo alloy is special material adapted to working with ceramics, characterized by very good ceramics adhesion, even after firing several times. It is biocompatible and does not contain harmful to the health of beryllium and has a high rate of corrosion resistance in body fluids [8, 20]. **Table 1** shows the chemical composition of the alloy and its basic characteristics.

Table 1 Chemical composition and some of the properties of Ni-Cr-Mo commercial alloy (based on manufacturer information)

	Chemical composition				
Remanium CSe	Ni (wt.%)	Cr (wt.%)	Mo (wt.%)	Si (wt.%)	(Co, Fe, Al, Ce) (wt.%)
	rest	26	11	1.5	< 1
	Properties				
	$R_{p0.2}$ (MPa)	A_m (%)	R_m (MPa)	Young Modulus (MPa)	Density (g·cm^{-3})
	340	15	550	170	8.2

The ceramic set used in the work belongs to the group of the glass-ceramic materials, adapted to perform veneers based on metallic. **Table 2** shows the composition of ceramic and its basic characteristics.

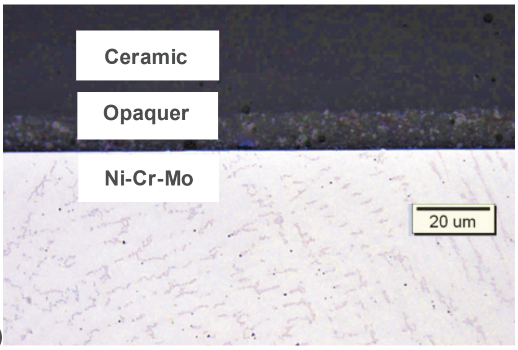
Table 2 Chemical composition and some of the properties of IPS d. SIGN (based on manufacturer information)

<table>
<tr><td rowspan="6">IPS d. SIGN</td><td colspan="8">Chemical composition (wt.%)</td></tr>
<tr><td>SiO_2</td><td>Al_2O_3</td><td>K_2O</td><td>Na_2O</td><td>CaO</td><td>P_2O_5</td><td>F</td><td>Others</td></tr>
<tr><td>50 - 65</td><td>8 - 20</td><td>7 - 13</td><td>4 - 12</td><td>0.1 - 6</td><td>0.0 - 5</td><td>0.1 - 3.0</td><td>0 - 3</td></tr>
<tr><td colspan="8">Properties</td></tr>
<tr><td>Solubility (µg·cm^{-2})</td><td colspan="2">WAK (25 - 510 °C), 10^{-6} K^{-1}</td><td>R_g (MPa)</td><td>HV</td><td>Opaquer adhesive force (MPa)</td><td colspan="2">Softening point temperature (°C)</td></tr>
<tr><td>3.8 - 8.9</td><td colspan="2">12.0 ± 0.5</td><td>80 ± 25</td><td>520 - 599</td><td>45.5 - 60.6</td><td colspan="2">510 ± 10</td></tr>
</table>

2.2. Sample preparation

Metallic samples base with 100 x 10 x 5 mm dimensions had been casted by centrifugal force using the induction furnace, then four of them have been subjected to a process of sandblasting with corundum sand (grain size150 µm). The time of the sandblasting process time for each sample was different, the shortest time is 15 seconds and followed by 1, 5, 10 minutes. For all samples after appropriate treatment has been applied ceramics IPS d. SIGN Ivoclar, according to the procedure, which is recommended by the manufacturer, i.e. after sandblasting, each sample was treated with a hot stream in order to clean metal surfaces from any dust and fine metal chips, and then inserted into a furnace to initiate the process of controlled and uniform surface oxidation. The maximum oxidation temperature was 950 °C. After the oxidation the samples surface has been purified again with a hot steam to remove possibly formed grease, then layer of opaque IPS d.SIGN A4 has been applied, after which it was possible to dentin and ceramic impose. Samples prepared in accordance with the manufacturer's instructions were fired in a temperatures exceeding 900 °C.

To properly analyze the state of metal-ceramic connection, the cross-sectional images of samples by optical microscopy have been taken. As a reference sample, a metallic sample with polished with abrasive paper grit 800 surface was used (**Fig. 1**).

a) 

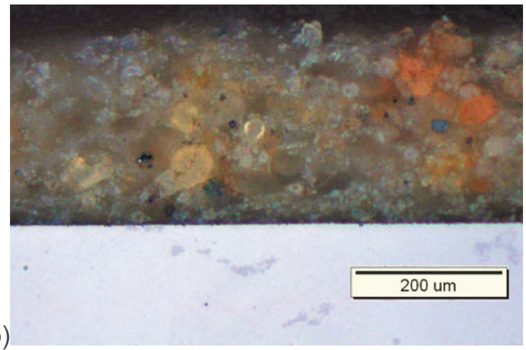
b) 

Fig. 1 The cross-section images of Ni-Cr-Mo commercial alloy coated with ceramic material - metallic sample with polished surface

3. RESULTS

Preparation of the sample surface is the most important function gives the possibility to achieve optimum mechanical properties and ensure a sufficiently strong connection on the border of two different materials. Every little mistake in the preparation of the surface affects on the connection adhesive forces. Inaccurate preparation of the surface can cause disturbances in the stability of the dental construction. In **Fig. 1** the cross-section of Remanium Cse (after sandblasting with various time) sample with ceramic coating are presented. The connection line is almost straight and small variations are probably fine metal powders that are left on the metal surface after grinding process. This type of connection probably will not show the mechanical nature of the combining forces.

The short time of sandblasting (15 s) increase diversification of metal surface The numerous inequalities have a depth and width about 20 - 30 µm. The recesses are wide hollows and hills, which in turn is conducive to good surface coverage with the opaquer (**Fig. 2 a, b**). During microscopic observation has been reported presence of the visible discontinuities of connection. Which indicates that the time of sandblasting respectively affect on the surface development, and the access of oxidizing atmosphere to the total actual surface metal is facilitated.

The use of longer sandblasting times (1, 5 and 10 minutes - **Fig. 2 c-h**), even more diversified alloy surface, but with so long time of blasting process, metal surface is more rugged and jagged (recesses have rather nature of the uneven rifts and burrs). In the other hand, the long-term corundum particles striking on the metal surface caused that a few of metal burrs were and closing creating voids and cavities. It is particularly dangerous and detrimental to the strength properties of the connection. In this case there is a possibility of non-uniform oxidation of the alloy surface, which may lead to scaling and spalling of oxides on the border of the connection and weakening functional properties of the whole system. Moreover, for a sample after 10 minutes (and longer) blasting are observed a significant amount of discontinuity and void which. Distribution area of such discontinuity is particularly vulnerable to damage caused by mechanical forces. Additionally important is the fact that the thickness of the opaquer on the entire ceramic surface is not with similar dimensions, which could have an impact on the final dimensions of the prosthetic works.

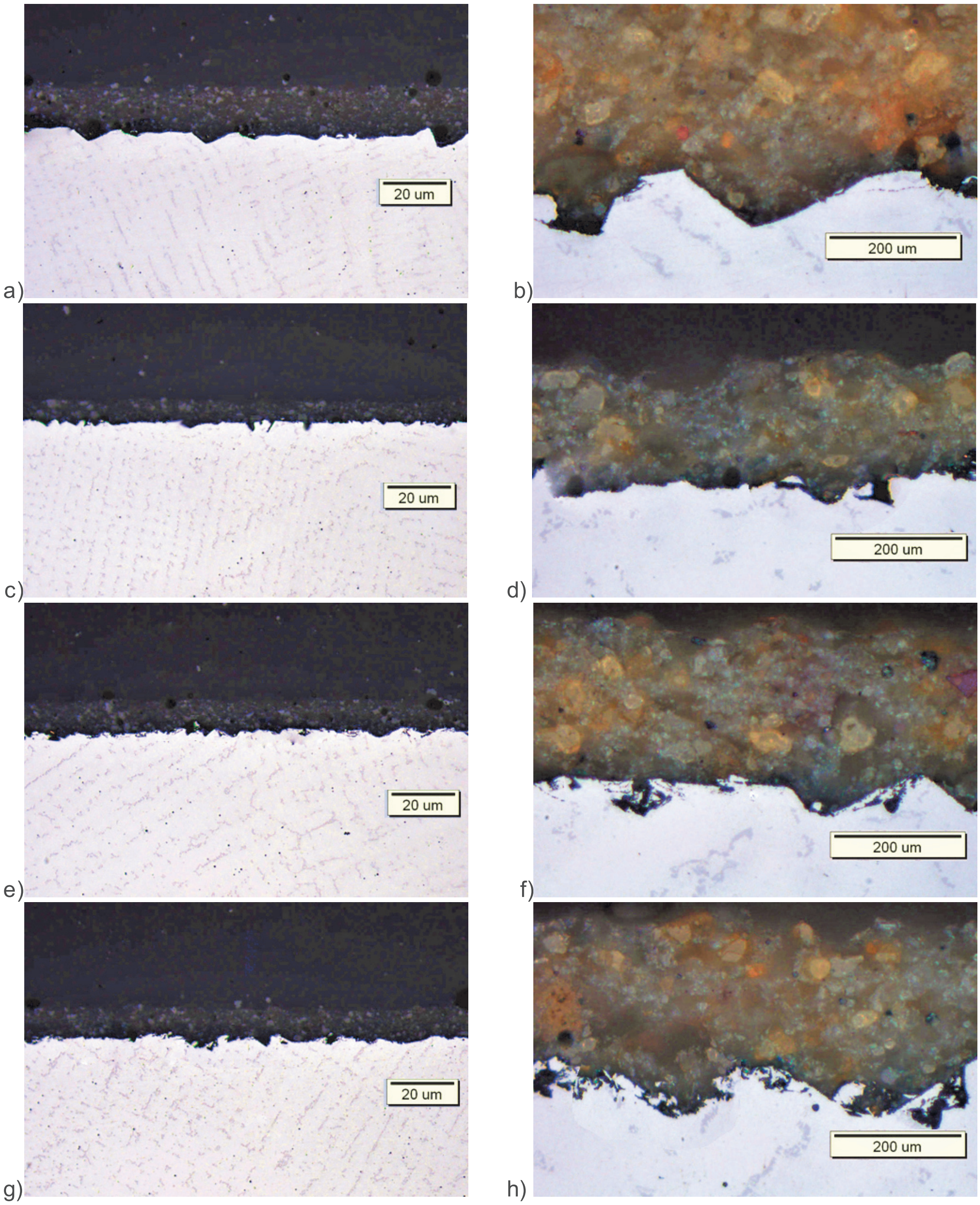


Fig. 2 The cross-section images of Ni-Cr-Mo commercial alloy coated with ceramic material. Metallic sample with sandblasted surface: a, b) 15 s; c, d) 1 min; e, f) 5 min; g, h) 10 min (a cross-section, not etched)

4. CONCLUSION

- The process of sandblasting of the alloy surface before applying a layer of dental ceramics promotes increasing the diversification the surface. Short-term, because only 15-second sandblasting makes that inequalities on the metal surface are uniform recesses and are conducive to increased adhesion forces of ceramics layers to the ground.

- The use of longer times (1, 5 and 10 min) of sandblasting process makes the created inequalities are more in the nature of rifts and burrs. Furthermore, the dimensions of the resulting inequalities prevent the proper and accurate of the alloy surface oxidation, but also prevent to evenly cover of the surface with ceramic.
- The highly diverse of surface contributes to the formation of numerous gaps, voids and discontinuities on the border of metal - ceramics connections.

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