Analysis by Microscopy Techniques of Metal-Ceramic Dental Restorations with Ni-Cr Support

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Dental biomaterials are designed to improve oral health by replacing the missing maxillary structures. The prosthesis partial edentulous restorations are widely used as metal-ceramic fixed prostheses having metal support from Ni-Cr or Co-Cr alloys. We have conducted metallographic studies on Ni-Cr alloy samples of metal-ceramic biomaterial commonly used in dentistry, in two characteristic zones: the metallic body and the metal-ceramic interface. Optical microscopy examination revealed precipitates of intermetallic compounds both in grain boundaries and interdendritic. The examination of the metal-ceramic interface did not revealed separations or cracks, but relatively uniformly distributed pores, resulting from the burning of opaque zone were detected.

Keywords: SEM, optical microscopy, Ni-Cr alloy, fixed prosthetic restorations, ceramics

Park and Lakes [1] defined the biomaterials as synthetic materials that replace a missing part of a living organism or compensate the missing function, having intimate contact with living tissues. Biomaterials must be effective, economic and physiologically acceptable. In dentistry, the prosthetic treatment of reduced partial edentulous involves introduction of a foreign object in the oral cavity of the patient, which requires special attention in terms of biocompatibility and corrosion resistance of materials used in this biologic environment [2,3]. In the prosthetics of partial edentulous restorations are widely used metal-ceramic fixed dentures, with metallic holders made from Ni-Cr or Co-Cr alloys. The quality and performance of dental materials depend on the correct composition of dental alloys, mechanical properties, corrosion resistance and biocompatibility [3, 4].

Numerous testing methods have been developed and selected by researchers to evaluate metal-ceramic bond strength and the minimum value, recommended by ISO 969315 for restorative dental metal-ceramic systems, is 25MPa for the three-point bending test [5]. However, it can be said that this value is related more to the flexural strength of the metal substrate than that of ceramic [5-7], this causing difficulties when attempting a comparison between different metal substrates.

Early, the noble metal alloys have been widely used in dental ceramic cladding. However, given the noble metal price fluctuations, greater attention was paid to alternative alloys. The Ni-Cr and Co-Cr alloys have shown good mechanical properties, such as high hardness, low density, and high tensile strength. Also, low cost and relatively simple manufacturing technology of Ni-Cr alloy and Co-Cr led to widespread use of them in fixed dental prostheses lately. By adding beryllium in these alloys there are improved casting properties of them and gave bond durability of porcelain and alloy [8-11].

Compatibility of the metal-ceramic system depends on the harmony of complex properties of both materials. A good restoration requires compatibility acceptable from chemical, thermal, mechanical and aesthetic point of view between metal and ceramics [12]. Chemical compatibility with the oxide layer involves formation of a strong bond that will resists to stresses resulting from thermal and mechanical incompatibility without compromising aesthetics. The oxide layer necessary for connection is created during degassing, oxidation or combustion. The oxidation time and temperature must be sufficient to create an oxide layer suitable for the metal-ceramics connection. The connection can be produced by chemical compatibility strong enough to overcome both transient and residual thermal stresses and mechanical forces encountered in clinical function [12]. Thermal and mechanical compatibility includes a ceramics firing temperature which does not cause distortions in the structure of metal, in conjunction with the optimum combination of thermal expansion coefficients [13]. In clinical applications, metal-ceramics systems compatibility must be able to simulate a number of forms of type tooth shades, translucency, fluorescence and finished surface [12].

In this paper we report the results of metallographic studies on Ni-Cr alloy samples of metal-ceramic biomaterial commonly used in dentistry, in two characteristic zones: the metallic body and the metal-ceramic interface.

Experimental part

Optical metallography studies on the materials used in the metal-ceramic reconstructions were performed on samples in molded state and after the three thermal cycles, with the following parameters: $V_{heating}$ = 50 °C/min, with 5 min holding at approx. 1000°C, cooling with maximum rate of 70°C/min (in the range of 1000°C–600°C), and then with approx. 100°C/min. Microstructure was highlighted by using the well-recommended metallographic reagent (20 mL HNO_3 and 80 mL HCl solution). Examinations were performed by routine techniques for optical (Olympus BX5

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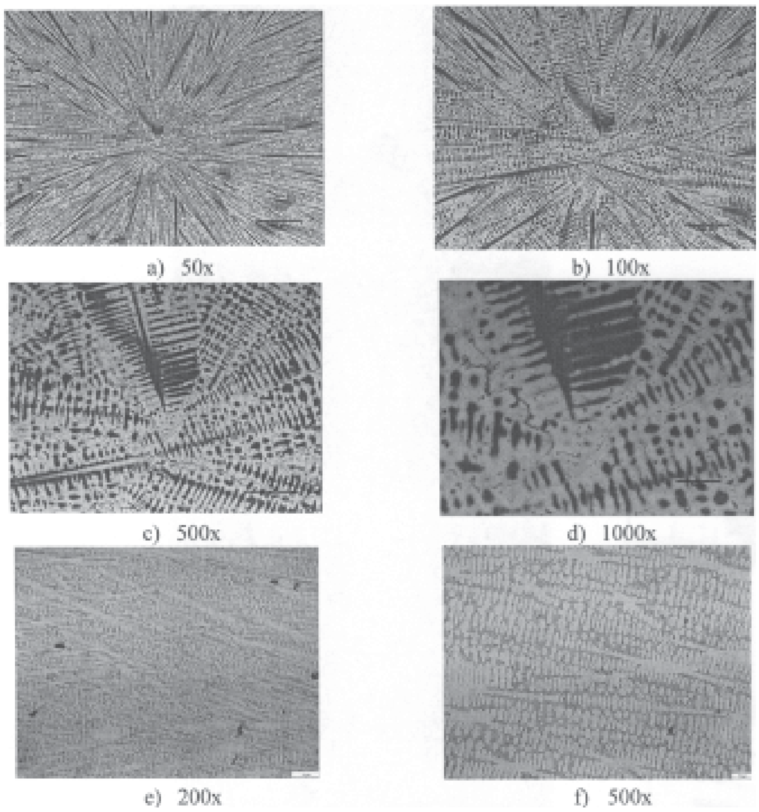

Fig. 1. Optical micrographs of the sample taken Ni-Cr alloy in the following states: (a-d) –as cast; (e,f) –after three thermal cycles (Attack reagent: 20mL HNO_3, 80mlHCl)

microscope) and scanning electron microscopy (Philips ESEM XL 30 TMP equipment with an EDS spectrometer).

Results and discussions

Figure 1 shows the optical micrographs of the Ni-Cr alloy sample in the cast state (a, b, c, d) and after three thermal cycles (e and f) at different magnifications

Within the cross-section of the sample it can be observed a star type orientation of the principal axes of dendrites due to the directional solidification in the shaped metal casting. With a magnification of about 500x it can be observed the images show that both grain boundaries and dendrites developed almost perfectly symmetrical to the principal axes. The second axes of the dendrites and the distance between them, due to the high speed solidification are of the order of microns. At high power magnification is observed that there is neither precipitated nor interdendritic grain boundary, which leads to the assertion that the structure is a solid solution specific to chemical heavily segregated. After the three thermal cycling, there is a pronounced reduction of segregation and a tendency to rounding and enlargement of secondary axes of the dendrites, these phenomena being attributed to decreasing concentration gradient of the main alloying elements, i.e., Cr and Mo. In terms of inclusions condition, since the samples were prepared by re-melting the commercial alloy and both re-melting and casting were performed in a controlled atmosphere with sandblasted and etched metal molds, there are not observed exogenous inclusions and the endogenous ones are have dimensions of maximum 10 μm (fig. 1-e). Regarding the chemical composition of the alloy it can be stated that the grain boundary precipitates in the form of Ni_2Cr compound (dark compound) with a Cr content that can vary between 21

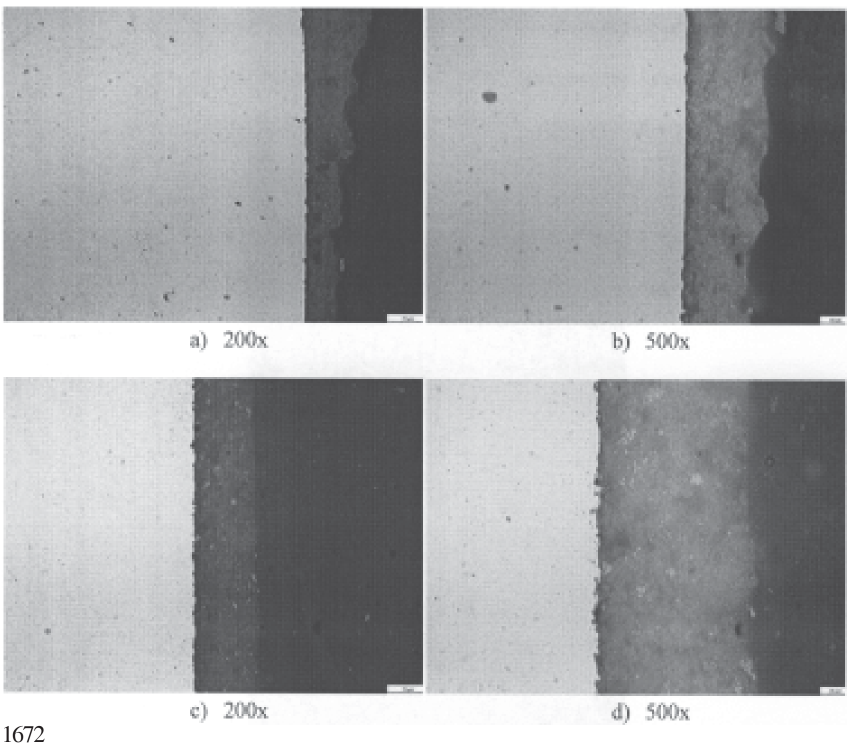

Fig. 2. Optical micrographs of the metal-opaque interface (a, b), and the opaque-dentin-metal interfaces (c, d) for non-etched samples of Ni-Cr alloy

Table 1
CHEMICAL COMPOSITION OF THE THE Ni-Cr ALLOY SAMPLE (FROM EDS ANALYSIS)

Sample	Chemical composition [At. %]									
	Co	Cr	Mo	Ni	Si	Mn	Al	Be	Fe	Total
Ni-Cr	0	26.4	7.4	57.7	4.2	2.8	0	0	1.5	100

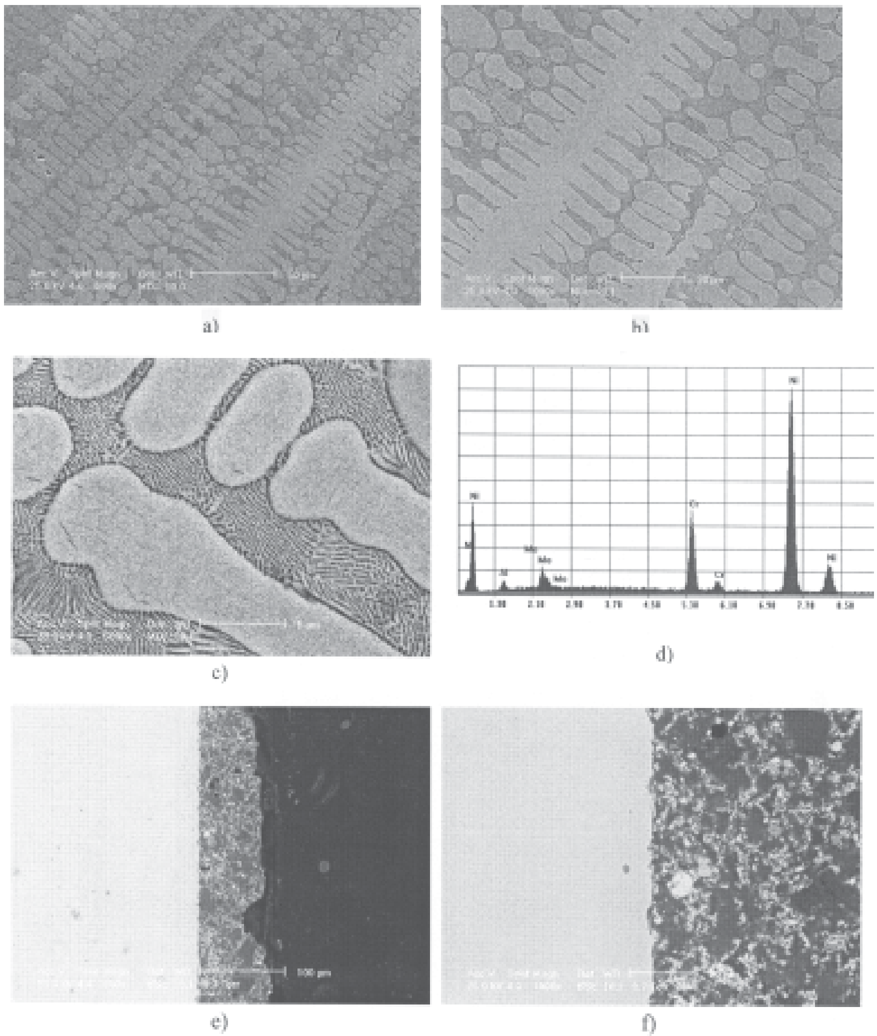

Fig. 3. SEM images (a-c,e,f) and EDS spectrum (d) of Ni-Cr alloy sample

and 37%; this compound can appear in the structure at temperatures lower than 590°C (as Ni-Cr ASM phase equilibrium diagram shows). The light compound, placed between the branches of dendrites, taking into account the Mo microsegregation, is a metastable compound and can be attributed (accordingly to Ni-Cr phase equilibrium diagram) to the intermetallic compounds: Ni_4Mo, $Ni_{17}Mo_5$, or even Ni_3Mo.

Regarding the analysis of metal-ceramic interface, in figure 2 are shown optical micrographs in cross-section (non-etched samples) of the interface opaque metal layer (a, b), metal-coating opaque-dentine (c, d). From the presented micrographs it can be seen that after the second thermal cycle (a, b) and the third cycle (c, d), the metal-ceramic interface is continuous, with no separation. It is observed, instead micron-sized pores, a relatively uniform distributed structure, resulted from opaque firing. Similar pores can be found in the opaque mass (bulk). No micro-cracks are observed after 2nd or 3rd thermal cycle.

In the case of the substrate of Ni-Cr alloy (fig. 2), there is also an almost perfect continuity of metal-ceramic interface, with no pores in the interface. The pores presence in the vicinity of the interface layer in the opaque zone after 3rd thermal cycle can be attributed to a chemical reaction within opaque-dentin zone. Also, here there are not observed micro-cracks.

Table 1 shows the chemical composition of the alloy as determined by EDS elemental analysis. We can notice that the employed Ni-Cr alloy corresponds to the compositional admitted limits for this alloy type.

Scanning electron microscopy (SEM) images (fig. 3) come to complete the above exhibited metallographic aspects, from both morphologically and compositionally points of view. SEM studies have been carried out both on samples in the as cast state and after the heat cycle tests with the deposit (layering) of ceramic layer. In figure 3c, there is shown in detail a portion of a dendritic structure, where it can be seen a lamellar constituent in the interdendritic space, which is characteristic for a mechanical mixture. Corroborating with the information from phase equilibrium diagrams for Cr-Ni, Mo-Ni and Ni-Cr-Mo alloys systems it can say that in this image it is a peritectoid compound, most probably $CrMo_4Ni_5$, in a relatively small proportion.

Conclusions

In the metal alloy mass (bulk), with the optical microscopy at high enough magnification it can be observed a compounds precipitation on grains boundary and interdendritic. The micrographs illustrating the metal-ceramic interface show that for both the 2nd (fig. 1, a, b) and the 3rd thermal cycle (fig. 1, c, d) the metal-ceramic interface is continuous, without detachments or micro-cracks. Instead, there are observed micron-sized pores relatively evenly distributed, resulting from the opaque zone resulted from firing. In SEM images it can be observed the details of the optimal morphology of metal-ceramic interface.

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