Microstructure and Chemical Homogeneity of Cast Dental Crowns Made from CoCrMoW Alloy and Ceramic Mass

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The aim of this study was the verification, using scanning electron microscopy, of microstructural integrity of metal-ceramic fixed dental crown restorations, made from CoCrMoW alloy (Girobond) and porcelain. SEM results on the cross-section of dental crown evidenced three layers: CoCrMoW bulk alloy mass, opaque zone and translucede porcelain mass. Defects as cracks and oxidation zone, as well as the presence of intermetallic compounds in boundaries were found in metallic part. The opaque and porcelain layers have different morphologies (microstructures). Also, different elemental compositions were found by EDX analysis. The distributions of elements along a selected analysis line are evidenced.

Keywords: CoCrMoW alloy, porcelain, physiognomic metal-ceramic crown, microstructural homogeneity

Prosthetic treatment of partial edentulous involves implanting of a foreign object in the oral cavity of the patient, which requires special attention to the compatibility of materials used with the biological environment [1,2].

The placement of a material into the oral cavity creates active interfaces through which the body affects the material and the material affects the body. Regardless of the material placed, these interactions occur depending on the material, the host, and the forces and conditions placed on the material [3].

In addition to a number of mechanical, physical and chemical conditions, the materials from which the prosthetic restorations are made must present biological compatibility [4].

Numerous factors act on dental materials which in time could lead to an alteration of their properties under various influences as: mechanical factors (represented by occlusion pressure exerted during mastication, which can plastic or elastic deform or even can induce a fracture of the material used), chemical factors (represented by reactivity of saliva, which has a variable pH depending on the nature of the food ingested, the use of drugs, various general diseases, etc.), and microbial factors (represented by the microbial flora present in the oral cavity) [5].

Another category of factors that influence the properties of biomaterials used in fixed prosthodontics is the technological correctness of the stages of making proper restoration [6].

Co-Cr alloys have the advantages over the Ni-Cr alloys due to their biocompatibility and the lack of allergic reactions that the latter can develop [7]. Generally, the dental casting defects can be avoided by a strict manipulation of the involved materials and following of the procedures governed by certain fundamental rules and principles [8].

Also, Co-Cr alloys are highly resistant to corrosion due to their chromium content, which forms a protective oxide film on the alloy surface [9-14]. In dentistry, alloys usually contain at least 4 metals, and often 6 or more, thus being metallurgical complex materials. More than 25 elements in the periodic table of elements can be used in dental alloys. The complexity and diversity of these alloys make understanding their biocompatibility difficulty, because any element in an alloy may be released and may influence the body. Dental casting alloys are widely used in applications that place them into contact with oral tissues for many years and the practitioners must choose among hundreds of alloy compositions, often without regard to biologic properties [15].

Interactions of dental cast alloys with the oral tissues take place by different mechanisms leading to bacterial adherence promotion, toxic and subtoxic effects and allergy. Whereas bacterial adhesion promotion may be avoided by adequate oral hygiene measures, the other mechanisms may lead to clinically adverse local reactions due to the metal presence [16]. According to Minciuna et al. [17], an oxidation of alloy may occur at high temperatures, with formation of intergranular iron oxide films, a presence which reduces the corrosion resistance of dental material.

A previous study that we performed [1] revealed that dental non-physiognomic metal crowns, made from CoCrMoW alloy (Girobond/Amann Girrbach) in rough cast state, and had inhomogeneity from microstructural and chemical point of view. In a similar manner, we made mixed metal-ceramic crowns, in which the metal component was the same CoCrMoW alloy and the physiognomic component (which covers the metallic substrate) is a porcelain. In order to determine if there must be applied or not a heat treatment of annealing (burning), we tried further to understand what is happening to the metallic material in the next technological step in the dental laboratory. The aim of study presented in this paper was to verify, using scanning electron microscopy and EDX elemental analysis, the microstructural integrity of fixed joint metal-ceramic restorations made from CoCrMoW alloy and porcelain.

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Experimental part

The infrastructure (support) of dental crown is CoCrMoW alloy (Girobond) purchased from Amann Girrbach Company, with similar characteristics as described in [1]. An opaque layer (ceramics) is applied on metallic support and this ceramic paste was burned at the temperature about 910°C. The final stage was the application of the translucence ceramic. The ceramics was a HeraCeram porcelain purchased from Heraeus Kulzer Company. The scanning electron microscopy and X-ray spectroscopy with energy dispersion (EDX) analyses were conducted with a Philips microscope, model ESEM XL 30 TMP equipped with an EDX spectrometer. The preparation procedure of joint metal-ceramics samples was similar with procedures described in [1,2].

The bonding and ceramic layer are deposited by painting on the metallic crown, after grinding, layer after layer. After each layer deposition, the metallic crown is heat treated (burned) in a laboratory furnace at different temperature (950°C for bonding, 980°C for 1st ceramic layer, and 850°C for the 2nd ceramic layer.)

Results and discussions

Examination with scanning electron microscope of the metal microstructure of the metal-ceramic crown before layering the physiognomic component has showed an imbedded structure inhomogeneity caused by casting (fig. 1).



Fig. 1. SEM image of casted metal substrate of the crown, 500x

The metal substrate microstructure consists in dendritic solid solution, as expected for the casted state [1], and interdendritic separations of intermetallic compounds.

At a magnification of 2000x, we found that the porcelain layer was not deposited uniformly and detected an opaque layer at the metal-ceramics interface. In fact, the porcelain mass was applied onto this opaque layer. We have also noted the presence of surface casting defects, represented by cracks of the metallic part of the crown on the outside and the presence of oxidation zones in the interior (fig. 2).

The opaque layer bonded to the metallic zone of crown was analyzed in terms of the EDX spectral characteristics



Fig. 2. SEM image of cross-section in the metal-ceramic crown, 2000x. From right to left, three layers (physiognomic porcelain, opaque zone, alloy mass) are observed





Wt %	At %
21.44	42.65
2.49	3.45
4.44	5.24
13.61	15.43
3.91	0.64
1.81	0.6
6.54	5.32
0.91	0.71
16.33	10.85
2.74	0.62
8.38	5.13
0.89	0.51
15.23	8.23
1.28	0.62
100	100
	Wt % 21.44 2.49 4.44 13.61 3.91 1.81 6.54 0.91 16.33 2.74 8.38 0.89 15.23 1.28 100





Fig. 4. SEM image of ceramic (detail) layer, 2000x



(fig. 3). The results of EDX chemical analysis in this area are shown in table 1.

A SEM image (with 2000x magnification) of the area corresponding to the dental ceramic mass (porcelain) is shown in figure 4.

As shown in figure 5 and table 2, the EDX results of the composition of ceramic layer showed the presence of elements such as Si, K, Ca, O, Al.

In figure 6, a SEM image at magnification of 500x of the metal alloy mass reveals the homogenization of the solid

Element	Wt %	At %
0	34.98	50.61
Na	3.16	3.18
Al	6.43	5.51
Si	37.98	31.3
K	14.35	8.49
Ca	0.95	0.55
Ce	2.15	0.36
Total	100	100

Table 2EDX CHEMICAL ANALYSISOF THE CERAMIC LAYERIN A SINGLE LOCATION



Fig. 6. SEM image of bulk metallic mass, 500x

solution and the presence of intermetallic compounds in grains boundary proximity. Of course, these defects will be visibly removed by thermal cycles necessary to obtain aesthetic ceramic layer. The EDX spectrum is shown in figure 7.



Element	Wt %	At %	
Al	0.64	1.38	
Si	2.55	5.29	
Мо	4.54	2.75	EDX CH
Cr	24.84	27.77	IN A
Mn	0.87	0.93	
Со	60.89	60.09	
W	5.67	1.79	
Total	100	100	

Table 3DX CHEMICAL ANALYSIS OFBULK METALLIC MASSIN A SINGLE LOCATION

The results of EDX chemical analysis, which are listed in table 3, showed the major presence of chromium and cobalt, as well as the existence of micro-percentages of aluminum, silicon, molybdenum, manganese and tungsten.

Figure 8 shows the spatial distribution of elemental materials inside the composite crown. We found the presence of cobalt, chromium, manganese and molybdenum (in metallic layer), silicon and zinc (in ceramics), titanium (in opaque layer), potassium and aluminum (both in the opaque layer and dental ceramic mass), tungsten (in metallic part, into the opaque layer and into dental ceramic mass, respectively). Also, in the dental ceramic mass we found the presence of individual



Fig. 8. Distribution of elements, indicated in SEM mapping

particles containing platinum, and uniformly distributed oxygen and cerium elements.

Form the SEM mapping of the microstructure (a) one can be mentioned some relevant aspects: i) in the bonding layer there are present: Ti, O, K, Al, W, islands of Pt, Si, and Zn; ii) in the first ceramic layer there are present: Si, Ce, K, Al W, O, islands of Pt, and Ca; iii0 in the second ceramic layer there are present: Si, K, Al, W, O.

We present in the followings some graphical representations about the distribution of elements along the analysis line is shown in figure 9.



Fig. 9. SEM image with indication of analysis line for elements distribution counting

The distribution along the analytical line of cobalt, chromium, oxygen, silicon and vanadium (fig. 10), reveals that cobalt and chromium are found mainly in metallic mass, whereas they have significantly reduced quantities in the opaque layer and ceramic mass, where they show up as residues. Oxygen and silicon are found in increased amounts in the ceramic mass. The vanadium element is uniformly distributed in all three layers.

The distribution of molybdenum, manganese and calcium along the analysis line (fig. 11) shows a relatively uniform distribution in all layers of calcium, and molybdenum, but the manganese shows up mostly in the



Fig. 10. Comparative elemental distribution of O, Si, W, Cr, and Co in the analysis line



metallic mass.

The elemental distribution of zinc, sodium and aluminum along the analysis line (fig. 12) demonstrates their reduced presence in metal and a significant presence in ceramic material.

The elemental distribution of zinc, sodium and aluminum along the analysis line (fig. 12) demonstrates their reduced presence in metal and a significant presence in ceramic material.

Comparative distribution of platinum, titanium, cerium and potassium (fig. 13) along the analysis line shows that titanium is present in large quantities in the opaque layer, but its content decreases in the other zones. Platinum is found in large quantities in the first ceramic layer, and its content decreases in the other zones. Cerium and potassium are present in both opaque and ceramic layers.

Conclusions

SEM results on the cross-section of fixed dental crown evidenced three layers: CoCrMoW bulk alloy mass, opaque zone and translucede porcelain mass. Defects as cracks and oxidation zone, as well as the presence of intermetallic compounds in boundaries were found in metallic part. The opaque and porcelain layers have different morphologies (microstructures). Also, different elemental compositions were found by EDX analysis performed in single location for each layer. The distributions of elements along a selected analysis line are evidenced.

It is expected an improvement of the internal microstructure of the metallic part and hence of its mechanical behavior due to repeated reheating in the burning cycles of the dental ceramic mass. Of course, a diffusion of the elements constituting the various layers of dental crown may have place, especially between the layers of ceramic, thus creating a link between porcelain layers deposited successively.

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