# Scanning Electron Microscopy and Energy-dispersive X-ray Spectroscopy in Determining the Metallic-shine-loss Causes of a Low Gold Content Conventional Alloy

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*The aim of this study is to determine the causes which lead to a significant colour change and loss of the metallic shine of a 4-element bridge made of a low-gold-content conventional alloy, only a few months after cementation. The authors used scanning electron microscopy (SEM) with retrodiffused electrons and energy-dispersive X-ray spectroscopy (EDX), applied to the restoration surface and in transversal section.*
*The failure occurred due to the incorrect melting conditions of the alloy which leads to internal porosities and areas with very different superficial composition. On the other hand, the improper polishing of the surface led to diminished corrosion resistance and the degradation of the prosthetic piece only a few months after cementation in the mouth.*

*Keywords corrosion, scanning electron microscopy, retrodiffused electrons, energy-dispersive X-ray spectroscopy, low-gold-content conventional alloy*

A 4-element bridge (fig. 1) presented a significant colour change and the loss of the metallic shine, only a few months after cementation. It presented darkened and shiny areas. The 4-elemens bridge (23-26), made of a low-gold-content conventional class E alloy (Au, Ag, Pd, Cu, Zn), was removed and analyzed in order to determine the causes of this failure.

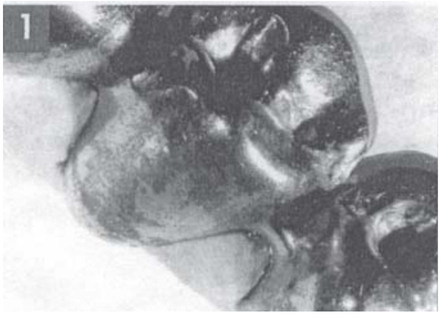

Fig. 1. The 4-element bridge is characterized by coloration and loss of surface shine

## Experimental part

For analytic purposes, we prepared 2 samples. One part of the restoration (24-25) was used as a sample to analyze the degraded surface by scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX), without altering the surface.

The other part of the restoration (26) was transversally sectioned in order to observe, in the same conditions, the superficial structure of the alloy.

## Results and discussions

The surface analysis performed with retrodiffused electrons (fig. 2) and energy-dispersive X-ray spectroscopy (EDX) (fig. 3, 4), shows that the coloration is associated with a non-homogeneous surface composition. It shows areas rich in copper and sulfur (area 2 , fig. 4), areas rich in silver and sulfur (area 3, fig. 5) and areas without sulfur (area 1, fig. 3).

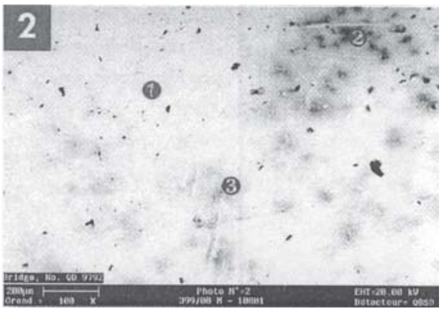

Fig. 2. Retrodiffused-electron image ( chemical contrast) of the 25 crown surface

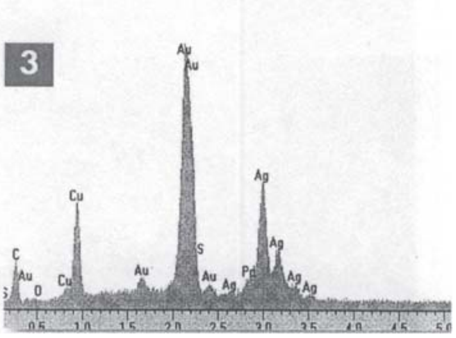

Fig. 3. EDX spectrum of area 1 (area without sulfur)

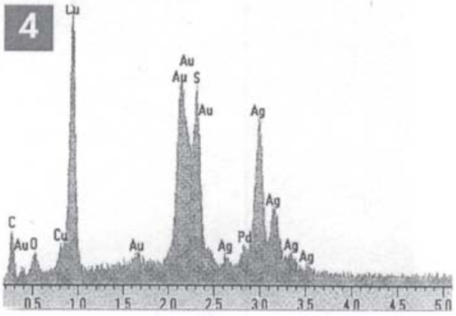

Fig. 4. EDX spectrum of area 2 (area rich in copper and sulfur)

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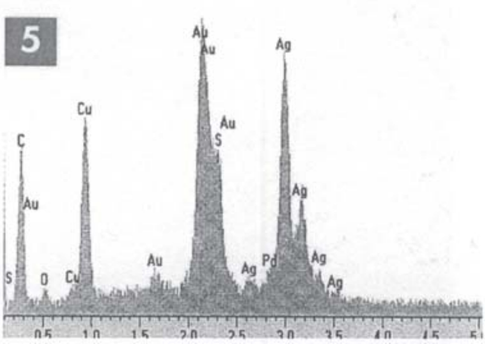

Fig. 5. EDX spectrum of area 3 (area rich in silver and sulfur)

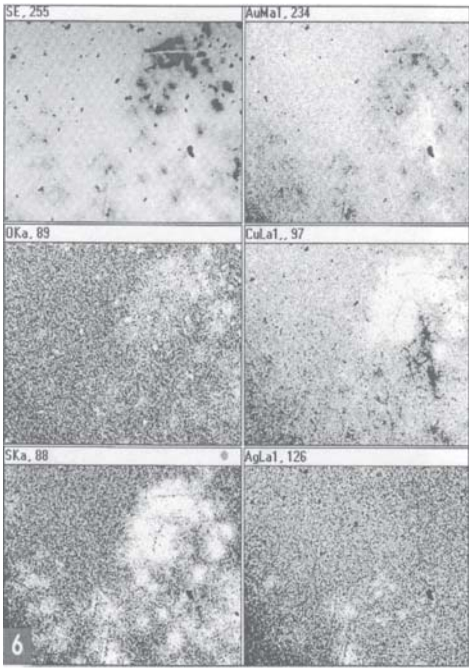

Fig. 6. Retrodiffused electron analysis (chemical contrast) and allocation of the elements (Au, O, Cu, S and Ag)

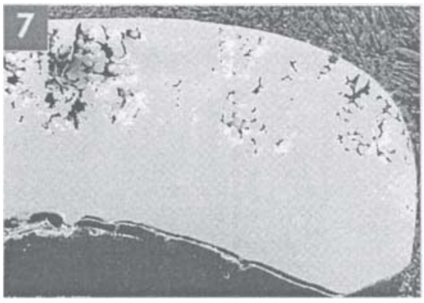

Fig. 7. Section observation with secondary electrons (topographic contrast)
SEM analysis with retrodiffused electrons (fig. 8) shows a dendritic non-homogeneous structure, close to the surface.

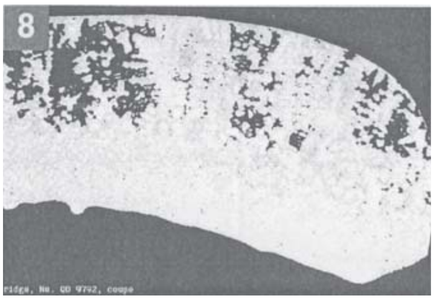

Fig. 8. Section observation with retrodiffused electrons (chemical contrast)

**Table 1**
RELATIVE CHEMICAL COMPOSITIONS IN Au, Ag AND Cu MEASURED IN 4 AREAS: I AND II – INTERNAL COMPOSITION OF THE MATRIX AND EXTERNAL COMPOSITION OF THE INCLUSIONS, III – INCLUSIONS RICH IN Au AND Ag (FIG. 9), IV – INCLUSIONS RICH IN Cu (FIG. 10).

| | Au* | Ag* | Cu* |
|---|---|---|---|
| I | 560 | 243 | 197 |
| II | 574 | 237 | 189 |
| III | 611 | 354 | 35 |
| IV | 253 | 123 | 624 |

* ‰ weight

Retrodiffused electron observation and the location of the elements (Au, O, Cu, S and Ag) (fig. 6) indicated that the shine loss area 2 was characterized by excess of sulfur and copper. The presence of oxygen was associated with copper localization.

SEM section analysis using secondary electrons (fig. 7) shows a very important millimetric internal porosity, localised on the external parts and merging at the surface, in some areas.

SEM analysis with retrodiffused electrons (fig. 8) shows a dendritic non-homogeneous structure, close to the surface.

The allocation of the main alloy elements (Au, Ag, Cu), close to the surface, is not homogeneous, mainly in the proximity of the porosity areas. This non-homogeneity is more important in the sub-superficial darkened areas than in the sub-superficial shiny areas. Independent from the porosities coloured in black (fig. 9) we observe, in the darkened sub-superficial area, the presence of the inclusions rich in Au and Ag and poor in Cu. In the shiny sub-superficial areas we observe, in addition to the internal porosities, tiny inclusions very rich in Cu. (fig. 10).

Comparing the nominal chemical composition to that of the analyzed areas (areas I, II, III and IV), we noticed

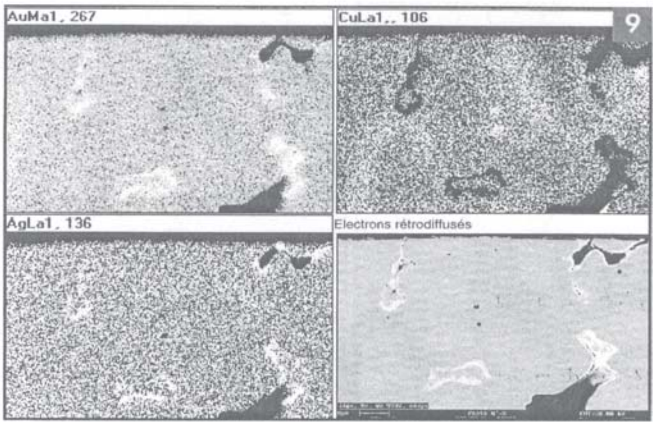

Fig. 9. Observation of the sub-superficial layer of the dark area with retrodiffused electrons (chemical contrast) and element allocation (Au, Ag and Cu)

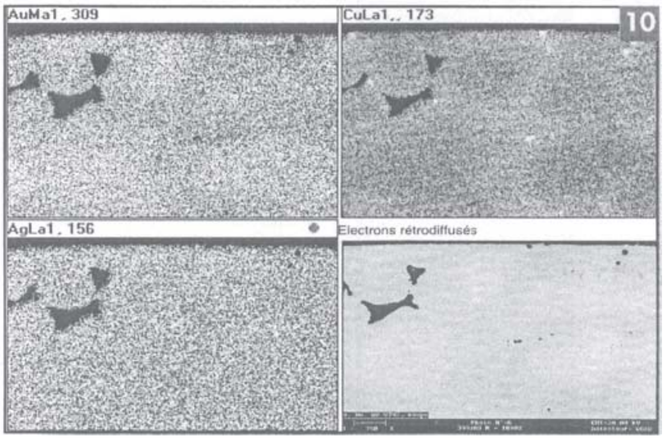

Fig. 10. Observation of the sub-superficial layer of the shiny area with retrodiffused electrons (chemical contrast) and the elements allocation (Au, Ag and Cu)

differences (table 1). It is well known that any change in the composition of an alloy (decreasing or increasing with one or more elements) has catastrophic consequences for the corrosion resistance [1, 2]. The corrosion process will be accelerated by the unstable phases in the alloy structure [3]. A galvanic cell will appear between these phases and the alloy matrix, encouraging selective corrosion [4-6]. In our case, we can talk about a shine loss. Composition analysis indeed shows the presence of sulfur and oxygen. Areas rich in copper or silver reacted with the sulfurous compounds in the saliva, which explains their presence in the coloured, mat areas. In this case we can talk about a selective corrosion process with formation of insoluble chemical products as sulphides, copper and silver [7].

## Conclusions

The failure is due to the incorrect alloy usage. The alloy's normal melting conditions were not respected. This incorrect melting was the cause for the internal porosities and for the areas with very different superficial composition. Independent from the porosity, the surface was not properly polished. The surface, porous and chemically non-homogeneous, due to the incorrect melting and surface treatment, led to diminished corrosion resistance and the degradation of the prosthetic piece only a few months after it's cementation in the mouth.

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