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ORIGINAL ARTICLE

The effect of simulating porcelain firing on the elemental composition, microstructure, and mechanical properties of electroformed gold restorations

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Abstract *Background/purpose:* The mechanical properties of pure gold (Au) are modified by thermal treatments. Thus, the aim of this study was to evaluate the effect of porcelain firing on the elemental composition, microstructure, and mechanical properties of electroformed Au crowns.
Materials and methods: Twenty electroformed Au specimens were prepared and divided into two groups. The first group did not receive any treatment (ELEC), and the other group was subjected to porcelain firing (PFIR). After metallographic grinding and polishing, all were investigated by scanning electron microscopy, and elemental composition was determined using energy-dispersive X-ray spectroscopy (EDX). Internal porosity was identified by quantitative image processing. Mechanical properties including Martens hardness (HM), indentation modulus (E_{IT}), elastic index (η_{IT}), and Vickers hardness (HV) were determined by instrumented indentation testing. The results were statistically analyzed using unpaired *t* test ($\alpha = 0.05$).
Results: A random distribution of tiny pores was identified in cross section, but no significant difference was found between groups [ELEC (%), 0.24 ± 0.13; PFIR (%), 0.31 ± 0.7]. Backscattered electron images revealed no mean atomic number contrast for both groups, indicating that the material was a single-phase alloy, whereas no differences between groups were

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identified in the composition of C, N, O, and Au after EDX analysis. By contrast, all mechanical properties tested showed statistically significant differences, with the PFIR group showing significantly lower HM, η_{IT}, and HV but increased E_{IT} compared with those of the ELEC group.
Conclusion: Although microstructure and elemental composition of electroformed Au crowns remain unchanged, the mechanical properties are significantly affected by the thermal treatment of porcelain firing.

Introduction

Electroforming has been adopted by dental technology since 1991 for the manufacture of gold (Au)-based metallic substrates for metalloceramic and telescopic crowns.[1–4] Metallic substrates made of electroformed Au are free of pores,[3] with excellent corrosion resistance and biocompatibility,[5] and with adequate to excellent marginal accuracy.[6–9] Low bond strength with dental porcelains,[10,11] technique sensitivity,[3] and the low mechanical properties of pure Au are among the drawbacks of this technology. However, analysis of clinical data has shown adequate longevity of electroformed crowns in both anterior and posterior regions.[12–14]

Although these structures are characterized as free of pores,[3] their mechanical properties are of great concern, because they are composed of a thin foil of pure Au with 0.2-mm mean thickness.[6] The mechanical properties of a substrate are of paramount importance for the performance of both telescopic crowns and galvanoceramic restorations, but until now there have been no studies on the mechanical properties of electroformed structures themselves. One serious limitation to conducting such studies is associated with sample preparation. Conventional mechanical tests (i.e., tensile, bending, and other tests) require bulky specimens, which cannot be prepared by electroforming technology, because the maximum achievable thickness is <0.3 mm.[3,10] As an alternative to conventional testing, the instrumented indentation test (IIT) has been determined by the International Organization for Standardization (ISO) as a reliable methodology for testing mechanical properties such as hardness, elastic index, relaxation, modulus of elasticity, and creep.[15] The principle of the technique is based on monitoring, in real time, the force applied to a hardness indenter and the indentation depth of the indenter in the sample. One advantage of this methodology is that irregular and small samples can be tested, thereby eliminating the need for bulky specimens.

In addition, previous studies have reported that electroformed structures contain light elements such as C, O, and N, which are retained in their structure, probably during the manufacturing process.[10] These low-atomic-number elements with small atomic radii can be dissolved in a metallic structure, forming an interstitial solid solution with increased hardness and tensile strength at the expense of ductility and fracture strength, as in the case of hydrogen embrittlement.[16] In addition, they might be implicated in further changes in mechanical properties during porcelain firing, because the firing temperature (780°C) is very close to the melting point of pure Au (1064°C), and various mechanisms might be triggered. N and O might evaporate during firing because of the degassing effect of vacuum, a technology extensively used to remove contaminating substances from metals and alloys (i.e., the removal of H_2 from Ti).[17] Thermal treatment might trigger the precipitation of new phases, modifying the microstructure and thus the mechanical properties. Interestingly, previous studies have shown that the mechanical properties of pure Au do indeed change after annealing above 300°C.[18] If this is a true hypothesis, then mechanical properties might be further modified with thermal treatment to enhance the performance of electroformed Au structures in telescopic and metalloceramic applications.

Therefore, the aim of this study was to determine the effect of porcelain firing on the elemental composition, microstructure, and mechanical properties of electroformed galvanic crowns. The null hypothesis was that the mechanical properties of electroformed Au structures are changed after porcelain firing.

Materials and methods

Sample preparation

One master silicone mold was prepared by the taking of an impression of a stainless steel rod (4 mm diameter and 5 mm high) with polyether materials (Impregum; 3M ESPE, St. Paul, MN, USA). Twenty cylindrical master dies were prepared by the pouring of a die stone (Jade Stone; Whip Mix Corporation, Louisville, KY, USA) into the master mold, which was left to set at ambient conditions. Then, 20 electroformed Au specimens were prepared (Figure 1) on the master dies in an electroforming machine (Ephos Galvano; Elemental Dental, Thessaloniki, Greece). The 20 specimens were equally divided into two groups. The first group received no further treatment (ELEC), and the second group (PFIR) was subjected to a complete firing schedule according to that used for a low-fusing porcelain (DuceraGold; Degudent, Hanau-Wolfgang, Germany) suitable for covering electroformed Au crowns. The firing schedule simulates two layers of opaque and dentin and a single glaze according to the manufacturer's instructions (Table 1). The firing of crowns were carried out in a dental oven (MultimatNTpress; Dentsply, York, PA, USA)

All specimens were then embedded along their longitudinal axis in an epoxy resin (Epofix; Struers, Belarup,