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# Surface characteristics and corrosion properties of selective laser melted Co–Cr dental alloy after porcelain firing

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## ARTICLE INFO

### Article history:

Received 15 September 2012

Received in revised form

4 August 2013

Accepted 27 November 2013

### Keywords:

Co–Cr alloy

Selective laser melting (SLM)

Corrosion

Firing

## ABSTRACT

**Objective.** We examined the surface characteristics and corrosion properties of selective laser melted (SLM) cobalt–chromium (Co–Cr) dental alloys before and after porcelain-fused-to-metal (PFM) firing.

**Methods.** Samples were manufactured utilizing SLM techniques and control specimens were fabricated using traditional casting methods. The microstructure and surface composition were examined using metallographic microscopy, X-ray diffraction (XRD), and X-ray photoelectron spectroscopy (XPS). Corrosion properties were evaluated using electrochemical impedance spectroscopy. Student's t-test was used to evaluate differences in numerical results of electrochemical corrosion tests between SLM and cast specimens before or after PFM firing. The results of electrochemical corrosion tests of the SLM and cast samples before and after firing were analyzed using one-way ANOVA.

**Results.** Although PFM firing altered the microstructure of the SLM specimens, they still exhibited a compact and homogeneous structure, and XPS analysis indicated that there were no significant differences in the surface composition of the specimens after firing. In artificial saliva at pH 5, the  $R_p$  value of the SLM specimens was  $6.21 \text{ M}\Omega \text{ cm}^{-2}$  before firing and  $2.84 \text{ M}\Omega \text{ cm}^{-2}$  after firing, suggesting there was no significant difference in electrochemical corrosion properties ( $P > 0.05$ ). In artificial saliva at pH 2.5, the  $R_p$  value of the SLM group was  $4.80 \text{ M}\Omega \text{ cm}^{-2}$  before firing and  $2.88 \text{ M}\Omega \text{ cm}^{-2}$  after firing, again indicating no significant difference in electrochemical corrosion properties ( $P > 0.05$ ). At pH 2.5, there was a significant difference in corrosion behavior between the cast and SLM groups, with the  $R_p$  value of the cast group being  $0.78 \text{ M}\Omega \text{ cm}^{-2}$  vs.  $2.88 \text{ M}\Omega \text{ cm}^{-2}$  for the SLM group.

**Significance.** The improved post-firing corrosion resistance of SLM specimens provides further support for their use in prosthodontic applications, as the oral environment may become temporarily acidic following meals.

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## 1. Introduction

Selective laser melting (SLM) is a widely used production process in which three-dimensional objects are created by selectively melting regions of a powder layer using a laser heat source [1–3]. The melted areas are specified using a CAD file, and the solid object is progressively formed by melting layers on top of one another. Compared with traditional casting techniques, SLM reduces the probability of operator error and minimizes casting defects, providing greater production precision [4,5]. SLM products exhibit higher density and improved corrosion and surface properties [6]. Because of these advantages, the technique has recently been applied to denture manufacture and has attracted the attention of dentists. Traditional casting methods have been used to manufacture Co–Cr restorations for many years [7,8], but SLM has only recently been employed to fabricate Co–Cr products [3,6]. For the sake of clinical safety, it is important to investigate the properties of Co–Cr products fabricated using SLM techniques.

Dental alloys used in fixed prostheses typically have a porcelain veneer fired onto the base metal for esthetic reasons. This type of metal–ceramic restoration is known as a porcelain-fused-to-metal (PFM) restoration. The facial surfaces of PFM restorations are veneered while the lingual and occlusal surfaces and sub-gingival margins are exposed. The porcelain firing process requires high-temperature treatment consisting of four operations between 950 and 1010 °C. The heating process inevitably changes the microstructure of the dental alloys, which may in turn affect their corrosion behavior.

Materials for dental applications have unique requirements including non-toxicity, biocompatibility, and mechanical strength [9]. The biocompatibility of a material is influenced by the corrosion behavior, as corrosion may induce the release of toxic ions. It is therefore important to study the influence of the porcelain firing process on the corrosion behavior of the final product. Relatively few researchers have examined the effect of the PFM firing process on the surface characteristics and corrosion properties of alloys such as Ni–Cr [10]. Other studies have evaluated the corrosion properties of Co–Cr alloys in artificial saliva [11,12], and Qiu et al. [13] described the effects of the PFM firing process on the surface and corrosion properties of these alloys. However, the influence of the PFM firing process on the surface and corrosion properties of Co–Cr components fabricated using SLM has not been assessed. The pH within the oral cavity can become substantially lower after meals. The resulting ion exchange may affect the surface properties of the alloy and potentially modify the corrosion behavior [14,15].

We evaluated the effects of a simulated PFM firing process on the surface characteristics and corrosion properties of Co–Cr samples fabricated using SLM. The properties were compared to those of cast control specimens in artificial saliva at pH 2.5 and 5.

## 2. Materials and methods

### 2.1. Materials and sample preparation

The specimens were manufactured from a commercially available Co–Cr alloy containing 63.9 wt% Co, 24.7 wt% Cr, 5.4 wt% W, 5.0 wt% Mo, and trace quantities of Si (Wirobond C+, Bego Dental, Bremen, Germany). The 16 cylindrical specimens (10 mm in diameter and 3 mm thick) were fabricated using an SLM system (BEGO MEDIFACTURING-SYSTEM, BEGO Medical, Germany) equipped with a Yb (Ytterbium) fiber laser with a wavelength between 1060 and 1100 nm and a maximum power of 200 W. The laser translation speed utilized in the study was under 7000 mm/s. The beam diameter was approximately 0.1 mm. Another 16 control specimens of the same shape and size were produced using a flame-casting method employing an oxygen–propane (50/50, v/v) gas mixture [16].

The specimens were polished with a series of silicon carbide (SiC) papers (400, 800, and 1200 grit) on a grinding and polishing machine (Beta, Buehler, Lake Bluff, IL, USA). The polished samples were ultrasonically cleaned in ethanol and deionized water.

In order to imitate the porcelain veneering procedure, half of the samples were selected at random and subjected to the firing cycle used in fabricating PFM restorations. Firing was performed under vacuum in a dental porcelain furnace (Multimat C, Dentsply Int., York, PA, USA). Briefly, the specimens were degassed at 1010 °C under vacuum for 5 min, opaque fired at 980 °C under vacuum, and cooled in air. Body firing was performed under vacuum at 970 °C and was followed by a second air cooling step. The final procedure consisted of glaze firing at 980 °C under vacuum followed by air cooling. After firing, the samples were re-polished and cleaned according to the procedure described above [13].

### 2.2. Microstructural observation

One specimen of each group was selected before and after firing and etched for 30 s using hydrochloric acid/hydrogen peroxide (80:20, v/v) at room temperature [16]. The microstructure of the specimen was examined using a metallographic microscope (Carl Zeiss Image AIM, Germany). Micrographs were obtained at 100 $\times$ .

An additional sample from each group was selected for X-ray diffraction (XRD) analysis and was processed as described above. The X-ray diffraction pattern was recorded between 15 and 100° 2 $\theta$  using Cu K $\alpha$  radiation (Rigaku Ultima IV, Japan).

### 2.3. Surface analysis

X-ray photoelectron spectroscopy (XPS) (Axis Ultra DLD surface analysis system, Kratos Analytical, Hadano, Japan) was utilized to identify the elemental constituents and chemical composition of the sample surfaces. One specimen of each group was examined before and after firing. The analysis was carried out using a monochromatic Al K $\alpha$  electrode at 15 kV

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