Microstructural analysis of Co-Cr dental alloy at the metalporcelain interface: A pilot study

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ABSTRACT

Objective: The purpose of the study was to observe whether conventional porcelain firings had an effect on the underlying microstructure of cobalt-chromium alloys used in porcelain-fused-to-metal systems.

Methods: One as cast (non-veneered) and two porcelain veneered Co-Cr specimens layered with and without tungsten(W)-metal conditioner were manufactured and analysed. Electron backscatter diffraction was used to determine the crystal structures and grain size across the porcelain-fused-to-metal interface.

Results: No difference was found in the microstructure of the alloy in both with and without W-metal conditioner. For the porcelain fired specimens, disparately sized granular structures were observed adjacent to the metal-porcelain interfaces compared to the bulk of the metal. Ellipsoid shaped grains at the alloy surface ranged between 1-11 µm in diameter and averaged 2.70 µm (SD: 2.17 µm) for the specimen layered with W-metal conditioner and 2.86 µm (SD: 1.85 µm) for the specimen layered without W-metal conditioner. Grains located in the bulk were >200 µm with dendritic-like features. The depth of the fine grain structure adjacent to the surface had an average depth of 15 µm. The crystal structure of the surface layer was found to be predominantly hexagonal close-packed whereas the underlying bulk was a mixture of both face-centered cubic and hexagonal close-packed phases. For the as cast specimen, similar large grains of over 200 µm was observed but exhibited no dendritic like features. In addition, no fine grains were observed at the surface region of the as cast alloy.

Conclusion: Conventional porcelain firings altered the interfacial and bulk microstructure of the alloy while the presence of the W-metal conditioner had no influence on the underlying alloy microstructure.

INTRODUCTION

Porcelain-fused-to-metal (PFM) restorations manufactured from base metal alloys such as cobalt-chromium (Co-Cr) have become widely accepted and are often a favourable choice in attempt to achieve a balance between strength, aesthetics and expenses. These alloys have a significantly lower production cost compared to high noble alloy restorations such as gold while their high rigidity permits thinner substructures to be fabricated. Even among its own class of base metal alloys such as nickel-chromium, Co-Cr is still a favourable alternative due to the concerns raised from nickel allergy patients.(Hildebrand et al., 1989) Clinically, available studies have shown satisfactory performance with these base metal alloy restorations within the period of follow-up, while a minority of cases, which were considered failures, were a result of technical complications such as ceramic fractures or chipping.(Eliasson et al., 2007; Reitemeier et al., 2006; Sailer et al., 2007; Walton and O'Brien, 1985) Failure modes in PFM restorations are generally classified in four types: metal-metal oxide, within the metal oxide, metaloxide to porcelain and cohesive failure within the porcelain. (O'Brien, 1977) Unfortunately, the actual clinical incidence of these failure modes are often difficult to interpret since the limited pool of studies usually either fail to report the type of alloy used or the exact nature of the failure mode. However, in-vitro bond strength studies (Carpenter and Goodkind, 1979; Drummond et al., 1984; Gavelis et al., 1982; Goeller et al., 1972; Homann et al., 2006; Joias et al., 2008; Korkmaz and Asar, 2009; Lubovich and Goodkind, 1977; Malhotra and Maickel, 1980; Marker et al., 1986; Shell and Nielsen, 1962; Stannard et al., 1990; Uusalo et al., 1987; Venkatachalam et al., 2009) have indicated a higher incidence of metal-oxide related failures for base metal produced PFM restorations.

Among all these failure types, the general consensus is that failure is often associated with the metal-ceramic incompatibility, which influences the residual stress state within the porcelain. The plethora of studies available on these aspects (Baldassarri et al., 2012; De Jager et al., 2006; Lenz and Kessel, 1998; Millen et al., 2012; Tholey et al., 2011) has led to the general solution that a retarded cooling process after porcelain firing is capable of reducing the residual stress developed in the porcelain layer which in turn improves the reliability of the restoration. In addition, there have also been various bonding agents and metal conditioners introduced into the market by manufacturers which claim to be able to either improve the compatibility between the alloy and porcelain or the bonding of the two materials. However, these surface treatments have been shown to have controversial effects (Matsumura et al., 1999; Ting et al., 2014). Ting et al(Ting et al., 2014) found that W-metal conditioner used in their study reduced the adhesive strength between the metal and porcelain and also had a distinctly thicker oxide layer present. There is also a lack of knowledge in regards to the contribution of the alloy properties towards the metal-ceramic interaction aside from the fact that the elastic modulus has an especial effect (Lenz and Kessel, 1998) on the stress development at the outmost edges of the metal-porcelain interface.

Therefore, since the interest of this study was to observe and analyse the Co-Cr microstructure, an appropriate tool known as electron backscatter diffraction (EBSD) was used. EBSD is a wellestablished technique and has also been successfully used in previous studies to analyse the microstructure of biomedical Co-Cr alloys.(Koizumi et al., 2013; Kurosu et al., 2010) One of the biggest advantages of EBSD over conventional transmission electron microscopy is the ability to analyse larger areas and obtain data from specific regions of a sample more readily.

Therefore, the purpose of this study was to identify the microstructure of both the bulk and the interface after conventional

porcelain firings and observe whether the addition of a W-metal conditioner influences the microstructure of the alloy.

The hypothesis of this research is that conventional practice of porcelain firings and the application of the metal conditioner affects the underlying microstructure of the Co-Cr alloy.

MATERIALS AND METHOD

Bi-layered specimens were fabricated using Co-Cr alloy (Wirobond C BEGO, Lincoln, USA) with a co-efficient of thermal expansion (CTE) of 14-14.2 ppm/°K as the core substructure. The chemical composition for the alloy as given by the manufacturer was Co 61, Cr 26, Mo 6, W 5, Si 1, Fe 0.5, Ce 0.5, C <0.02) wt%. The veneering porcelain used was Reflex Dimension (Wieland, Pforzheim, Germany), which has a CTE of 13.1 ppm/°K. The tungsten (W)-metal conditioner used for this study was Reflex NP Safe (Wieland, Pforzheim, Germany). Three specimens in total (as cast, with W-metal conditioner bi-layer specimen and without W-metal conditioner bi-layer specimen) were manufactured for this experiment.

Three square plates (8 mm \times 8 mm \times 1.5 mm) were cut from 1.5 mm thick Erkodur thermoforming discs (Erkodent, Germany) and invested in a phosphate-bonded investment (Shera*fina* 2000, Germany) for the lost wax procedure. These were cast in Co-Cr base metal alloy using an induction casting machine (Fornax, BEGO, Germany).

Preparation of the veneering porcelain

The layering of the veneering porcelain consisted of a total of six firings excluding the glaze fire (Initial oxidation or W-metal conditioner, 1st paste opaque, 2nd paste opaque, 1st dentine and two 2nd dentine firings) and were layered as recommended by the manufacturer. A detailed summary of the firing specifications used is listed in Table 1. Initial oxidation step was only performed on the specimen without W-metal conditioner layering and was performed without vacuum. Veneered specimens were layered to a thickness of 1.5 mm. A specimen layered with W-metal conditioner was included to observe whether it had an effect on the underlying microstructure.

ELECTRON BACKSCATTER DIFFRACTION

EBSD data were obtained and analysed in the present study for the three manufactured specimen: as cast (control), with W-metal conditioner and without W-metal conditioner. The specimens were selected to identify whether the initial oxidation firing played an important role in the development of the underlying microstructure. In order to remove as much surface damage as possible on the viewed surface, the lateral surface of the metal-ceramic interface was polished to a 0.05μ m colloidal silica finish. Subsequently, EBSD analyses were performed (Zeiss Sigma VP FEG) with EBSD patterns collected (Oxford Instruments Aztec. @20kV, ~13nA) at 0.1-0.5 µm step sizes. EBSD data were post-processed (HKL CHANNEL 5 software) using methods outlined in Prior et al(Prior et al., 2009) and grains were defined as cells surrounded by boundaries with misorientations of 10 degrees or more.(Shigematsu et al., 2006) Subsequent texture orientation, phase maps grain size data were produced through the boundary conditions outlined above.

RESULTS

The observed EBSD phase maps collected for specimens with and without W-metal conditioner revealed similar findings. Firstly, the bulk of the alloy consisted of large grains with dendriticlike features well over 200 µm in diameter as shown in Fig 1 and Fig 2. These grain sizes were also consistent with the as cast specimen. As we approach closer to metal-ceramic interface (<15 µm from the interface), an accumulation of significantly smaller grains was observed in both tested specimens [Fig 1]. This fine grain structure had grain diameters which ranged between 1-11 µm and had an average grain diameter of 2.70 µm (SD: 2.17 µm) for the specimen layered with W-metal conditioner and 2.86 µm (SD: 1.85 µm) for specimens layered without W-metal conditioner. A detailed grain size distribution graph with a sample size of 206 (with W-metal conditioner) and 497 (without W-metal conditioner) is shown on Fig 3. The application of the W-metal conditioner had no significant effect (p>.05) on grain size at the alloy surface. For the analysis of the crystal structure, the as cast specimen was predominantly face-centered cubic(fcc) [Fig 2] while the specimens treated with porcelain firing was composed of an even mixture of hexagonal close-packed (hcp) and fcc crystal structure. Fine grain structure observed in the porcelain fired specimens [Fig 1] at <15 µm of the metal-porcelain interface was primarily hcp phase and had no preferred orientation.

DISCUSSION

Whether it was a typical porcelain to metal system or with the added presence of the W-metal conditioner layer, both tested specimens showed no significant difference in terms of the proportions of phases present or arrangement of these phases within the volume shown. Therefore, the presence of the W-metal conditioner appeared to have no influence on the microstructural changes observed.



Figure 1. EBSD phase map of the specimen layered without W-metal conditioner showing fine grain structure developed at the interface as compared to the bulk.



Figure 2. EBSD phase map of bulk portion of the Co-Cr alloy. (a) as cast, (b) after porcelain firing. Blue is cubic (fcc) and red is hexagonal (hcp). Other colours are non-indexed solutions.

| Firing Programs | Preheating Temperature | Drying Time [min:sec] | Heating Rate °C/min | Firing Temperature | Holding Time [min:sec] | Vacuum |
|-----------------|---------------------------|--------------------------|------------------------|-----------------------|---------------------------|--------|
| Degas | 575 | 0:00 | 80 | 980 | 5:00 | No |
| NP safe | 575 | 6:00 | 75 | 975 | 1:00 | Yes |
| Paste Opaque 1 | 575 | 6:00 | 75 | 930 | 3:00 | Yes |
| Paste Opaque 2 | 575 | 6:00 | 75 | 920 | 2:00 | Yes |
| Dentine 1 | 575 | 6:00 | 75 | 900 | 2:00 | Yes |
| Dentine 2 | 575 | 4:00 | 75 | 890 | 1:00 | Yes |
| Glaze Firing | 575 | 3:00 | 75 | 880 | 1:00 | No |

Table 1. Summary of the firing program specifications

There were two major findings observed in this study. The first is the major difference in grain size between the bulk of the metal and the grains observed <15 µm from the metal-porcelain interface. The existence of a surface metal composition which is different to the bulk metal portion of a metal-ceramic restoration has long been known and is well documented for both noble and base metal alloys.(Culha et al., 2009; Johnson et al., 2006) Studies such as Johnson et al(Johnson et al., 2006) have observed that an oxidation preparation firing prior to porcelain application was critical in creating an oxide layer and bringing elements to the surface of the metal which could be directly involved with the metal-ceramic bonding. These findings were relevant to both palladium silver and nickel chromium alloys. Elements such as tin and indium in the case of the noble metal alloys and chromium for base metal alloys had increased concentrations within several microns of the surface following porcelain firing or initial oxidation, strongly indicating that these elements had diffused towards the surface. (Anusavice et al., 1977; Lautenschlager et al., 1969; Stewart et al., 1978; Von Radnoth and Lautenschlager, 1969) Referring back to the current study, it can be seen from the grain size distribution graph of the metal-porcelain interface in Fig 3, that the majority of the grains at the surface are distributed between 1-11 µm with the highest frequency of grains found between 2-3 µm. Closer examination of the grain morphology also indicated that the majority of the grains were ellipsoid in shape with no preferred orientations. This was very different to the bulk of the alloy [Fig 2], which has a typical dendritic-like structure with grains over 200 µm in diameter. In addition, to the authors' knowledge, this fine grained layer observed at the interface has not been documented before in previous literature. The fine grained interface was also not found in the as cast specimen. Therefore, on the basis of the current EBSD observations the null hypothesis is rejected and that there is a clear distinction in grain size and crystal structure between the as cast and the bulk/ surface regions of the porcelain fired specimens.

The second major observation observed in this study was the arrangement and proportion of cubic (fcc) and hexagonal (hcp) phases present. Typical Co-Cr alloys are known to contain both fcc and hcp phases(Lee et al., 2008; López and Saldivar-Garcia, 2007; Matković et al., 2004). The cause of this can be easily deciphered thermodynamically from a binary Co-Cr phase diagram(Okamoto, 2003), which shows that phase transformation from fcc to hcp occurs below ~1000 degrees for the composition range of the alloy considered. However, due to the relatively sluggish transformation from fcc to hcp in cobalt **Figure 3.** Grain size distribution graph generated from EBSD data for both with and without W-metal conditioner specimens.



Grain size distribution

based alloys (Liu et al., 2005; López and Saldivar-Garcia, 2007; Yoda et al., 2012) and the relatively fast cooling rates involved, the resultant is that a majority of the fcc phase is retained. This observation corresponds to the as cast specimen which was found to have a predominant fcc crystal structure on the analysed surface. Concurrently, the sluggish transformation from fcc-hcp also explains the coexistence of both fcc and hcp phase found in the bulk portion of the porcelain fired Co-Cr alloy even after several subsequent porcelain sintering cycles at ~900°. It can also be seen from Fig 1 and 2 that the hcp phase appears to form striation and dendritic-like features, within the fcc matrix.(Matković et al., 2004)

The significance of these two phases has been reviewed in several recent studies.(Henriques et al., 2012; Lee et al., 2008; Matković et al., 2004; Sato et al., 2007) Matkovic's et al(Matković et al., 2004) compared two different base metal alloy systems Co-Cr-Mo and Co-Cr-Ni and observed a typical dendritic structure made up of hcp and fcc in both alloys. Little difference was observed in grain size and proportions of the phases present between the two alloys, but elemental analysis did show slight composition differences and microhardness measurement did indicate that the fcc phase had a higher hardness than the hcp phase. This signified that this property was much more dependent on the structure than a slight difference in chemical composition.(Matković et al., 2004) Other studies such as Lee et al(Lee et al., 2008) have also discussed the importance of stabilizing the fcc phase in order to improve its mechanical properties, especially strength and ductility. Relating back to the present study, it can be seen in Fig 1 that the fine grain structure at the metal-porcelain interface was predominantly hcp phase. This phenomenon has also not been previously observed and does not appear to be a result of a typical fcc-hcp martensitic transformation(López and Saldivar-Garcia, 2007; Matković et al., 2004) due to the homogenous distribution along the entire metal-porcelain interface. Therefore, this has led the authors to believe that the present grain structure and phase developments observed at the interface are likely related to the oxidizing processes undergone during sintering cycles. As known from previous studies (Anusavice et al., 1977; Lautenschlager et al., 1969; Stewart et al., 1978; Von Radnoth and Lautenschlager, 1969), certain elements such as Cr diffuse towards the surface during oxidation procedures. This increases the Cr content at the interface and shifts the phase equilibrium into a more stable region of the hcp phase, thus explaining the predominance of the hcp phase at the interface. To the author's knowledge however, there is no current literature which has discussed the impact of such fine grains developed at the interface or the hcp phase developed at the surface of Co-Cr alloys. Hcp phase is known to be a less ductile crystal structure compared to fcc which fulfils von Mises criterion in which five independent slip systems are required for good ductility (Lee et al., 2008). On the other hand, the fine grains developed at the interface should follow the Hall-Petch relationship of grain boundary strengthening with decreasing grain size, which would result in an increased hardness located at the interfacial region. (Callister, 2005). This change in hardness and embrittlement at the metalporcelain interface will impact the stresses developed in the porcelain as well as the adhesion of the metal-porcelain bond.

Further investigation is required in order to understand how the observed grains and crystal structure is developed and how this may impact the properties and reliability of this alloy; especially when bonded to conventional dental porcelain.

CONCLUSION

Within the limitations of this study, the following conclusions were drawn:

- W-metal conditioner had no effect on the underlying microstructure of the alloy.
- Co-Cr alloy which had undergone porcelain firing developed a homogenous fine grained hcp layer at the metal-porcelain interface and dendritic-like structure in the bulk.

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