Microstructures and mechanical properties of interface between porcelain and Ni–Cr alloy

J. Liu¹, X.M. Qiu¹, S. Zhu², D.Q. Sun³,∗

¹ Key Laboratory of Automobile Materials, School of Materials Science and Engineering, Jilin University, Changchun 130022, China
² Stomatology Hospital, Jilin University, Changchun 130041, China

ABSTRACT

Microstructures and mechanical properties of interface between porcelain and Ni–Cr alloy have been investigated using scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDS), X-ray diffraction analyzer (XRD) and electronic universal testing machine. Experimental results show that there exists a reaction layer in the Ni–Cr/porcelain bond interface and the thickness of the reaction layer increases with the increase of firing temperature and firing time. The interdiffusion of atoms occurred during firing, Al, Si, Sn and O diffusing from porcelain material into Ni–Cr alloy, and Ni and Cr diffusing into the porcelain material. The phase composition of Ni–Cr/porcelain interface is complicated and mainly contains SnO₂, AlNi₃, SnCr₀.₁₄Oₓ and KAlSi₂O₆ compounds. Firing temperature and firing time have obvious effect on the shear bond strength of Ni–Cr/porcelain interface relative to the different reaction layer structures. The maximum shear bond strength reached 43.8 MPa at firing temperatures of 990 °C for firing time of 2.5 min.

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1. Introduction

Porcelain fused to metal (PFM) restorations have been widely used in clinical treatment and restoration of teeth because of their excellent characteristics such as natural beauty, obdurability, impact resistance, wear resistance and corrosion resistance, and are welcomed more and more by doctors and sufferers [1–4]. Initially, the alloys used as the substructure of metal/porcelain restorations were high-gold alloys, then low-gold alloys. However, Ni–Cr alloys were introduced into dentistry as a possible replacement for precious alloys due to the sharp increase in cost of gold in 1973. In addition, Ni–Cr alloys also offer the advantage of an increased modulus of elasticity compared with gold, which allows thinner sections of the alloys to be used, and consequently reduces polish of natural teeth during the restoration. At present, Ni–Cr alloys have been widely used for the restoration of teeth in some developing countries because of the economic and other reasons, although doubts remain as to the biocompatibility of Ni–Cr alloys.

The success of the PFM restorations acutely depends on the success of the strong bond between porcelain and the metal substructure [5–8]. However, the clinical failure rate of PFM restorations due to fracture and exfoliation of porcelain is 59.1% of the whole clinical failure. In the current study, a lot of researches done on the PFM restorations had focused on the nature and kinds of the alloy, but reports about the microstructure and composition of the metal/porcelain interface are limited [9–11]. Better understanding of interface reactions between porcelain and Ni–Cr alloy is necessary if more compatible metal/porcelain bond is to be developed.

The present work investigates influences of firing temperature and firing time on microstructures and mechanical properties of interface between Ni–Cr alloy and porcelain. Its purpose was to obtain better understanding of the Ni–Cr/porcelain interface bond and provide some foundation for improving the quality of PFM restorations.

2. Experimental

Ni–Cr alloy (SHOFUUNIMETALITI, Japan) was used with dental porcelain (SHOFU, Japan) in this investigation. The chemical compositions of the Ni–Cr alloy and dental porcelain are presented in Tables 1 and 2, respectively.

The Ni–Cr alloy plates (30 mm × 30 mm × 1 mm) were cast with centrifugal casting machine according to proper operating instructions. Every alloy plate was cut into six base metal specimens with dimensions of 15 mm × 10 mm × 1 mm by electrical discharge machining. Prior to porcelain veneering, surfaces of base metal specimens (15 mm × 10 mm) were polished with 800-grit SiC sandpaper, and then ultrasonically cleaned in an acetone bath and rinsed...
in deionized water for 5 min to remove surface contaminants. Two coats of opaque porcelain (PA2O) condensed by vibration and blotting procedures were applied on the specimen surface, each being about 0.2 mm thick, and then the body porcelain (A2B) was subsequently formed by use of a brush and a vibrator. The porcelain on metal specimen surface was fired in the vacuum porcelain furnace (Multimat 99 VACV MAT 2500, Germany). Effects of firing parameters on microstructures and mechanical properties of metal/porcelain interfaces were investigated by changing firing temperatures (930–1040 °C) and firing times (0.5–15 min). The firing technological parameters are shown in Table 3. After porcelain firing, a uniform porcelain with 1.0 mm in thickness was applied along an 10 mm length in the central portion of the metal specimen surface. The cross-sections of specimens with porcelain were ground, polished, and etched in 2 g CuCl$_2$·2H$_2$O, 40 ml concentrated hydrochloric acid and 50 ml 95% ethyl alcohol for metallographic examination.

The microstructures of metal/porcelain interfaces were examined using SEM (Model JSM-5310, Japan), EDS (Model Link-Isis, Britain) and XRD (Model D/Max 2500PC, Japan). The shear test was carried out at room temperature by means of electronic universal testing machine (Model CSS-44100, China) with a loading rate of 0.5 mm/min. The schematic of shear test is shown in Fig. 1. The shear bond strength of metal/porcelain interface was determined, based on the average of three measurements per condition. After the shear test, fractured surfaces of the metal and porcelain were examined.

3. Results and discussion

3.1. Microstructures of Ni–Cr/porcelain interface

Fig. 2 shows SEM microstructures of Ni–Cr/porcelain interfaces produced at firing temperatures of 930 °C, 990 °C and 1040 °C for firing time of 1.0 min. As can be seen, the bond interface is smooth and compact, there exists a reaction layer in the interface, and the thickness of reaction layer increases with rising firing temperature. Some microcracks were observed at porcelain side of the metal/porcelain interface produced at firing temperature of 1040 °C, as shown in Fig. 2(c). Fig. 3 illustrates EDS element line distribution of Ni–Cr/porcelain interface produced at firing temper-
Fig. 3. Element line distribution of Ni–Cr/porcelain interface produced at firing temperature of 990 °C for firing time of 1.0 min.

Fig. 4. X-ray diffraction patterns of Ni–Cr/porcelain interfaces at different firing temperatures for firing time of 1.0 min.

Fig. 5. The influence of different firing temperatures for firing time of 1.0 min on shear bond strength of Ni–Cr/porcelain interface.
strength of the interface was significantly improved because the interface reaction resulted in forming new phases \( \text{SnCr}_{0.14} \text{O}_x \) and \( \text{KAlSi}_2 \text{O}_6 \) during the firing process, which is in agreement with the XRD results shown in Fig. 4. The shear bond strength of the Ni–Cr/porcelain interface reached 37.5 MPa at firing temperature of 990 °C for 1.0 min. Fracture also occurred at metal/porcelain interface, but a large amount of retained porcelains were observed on the metal surface, as shown in Fig. 6. Fig. 6(a) shows the morphology of the retained porcelain on Ni–Cr alloy with a macro-image, and Fig. 6(b) illustrates the typical fracture surface morphology of the retained porcelain at higher magnification. These results confirm that the Ni–Cr/porcelain interface is an integration of diffusion type and compound type. The increased shear bond strength of the interface is mainly attributed to the formation and growth of \( \text{SnCr}_{0.14} \text{O}_x \) and \( \text{KAlSi}_2 \text{O}_6 \) phases producing strong metallurgical bond force between metal and porcelain. Further rising firing temperature to 1040 °C resulted in sharply decreasing the shear bond strength of Ni–Cr/porcelain interface. According to the research results of interface microstructures, there exist some microcracks at porcelain side of metal/porcelain interface produced at firing temperature of 1040 °C. During shear test, the specimen with porcelain was subjected to applied mechanical force and the microcracks act as preferential sites for the initiation and propagation of cracks due to stress concentration in microcrack tips. The existence of the microcracks at porcelain side of metal/porcelain interface caused the bonding strength of metal/porcelain interface to be even lower than that of Ni–Cr/porcelain interface. These results suggest that firing temperature is one of controlling factors for the bond strength of metal/porcelain interface, and it is favorable to select proper firing temperature for improving the bond strength of the interface.

Fig. 7 shows the influence of different firing times at firing temperature of 990 °C on shear bond strength of Ni–Cr/porcelain interface. As can be seen, the influence of firing time on the shear bond strength is similar to that of firing temperature. At firing time of 0.5 min, the shear bond strength of Ni–Cr/porcelain interface was 13.6 MPa. Fracture occurred at metal/porcelain interface, and the interface completely peeled off. With an increase of firing times from 0.5 min to 2.5 min the shear bond strength of interface increased and the maximum shear bond strength reached 43.8 MPa at firing time of 2.5 min. Fracture also occurred at metal/porcelain interface and there exist a large amount of retained porcelains on the metal surface. It should also be related to the formation and growth of new phases producing strong metallurgical bond force between metal and porcelain. Further increasing firing times from 2.5 min to 15 min, the shear bond strength of the interface decreased. Therefore, it is also favorable to select proper firing time for improving the bond strength of metal/porcelain interface.

4. Conclusions

(1) There exists a reaction layer in Ni–Cr/porcelain bond interface. The phase composition of the metal/porcelain interface is complicated and mainly contains SnO\(_2\), AlNi\(_3\), SnCr\(_{0.14}\)O\(_x\) and KAlSi\(_2\)O\(_6\). The increase of firing temperature and firing time favors the formation and growth of new phases \( \text{SnCr}_{0.14} \text{O}_x \) and \( \text{KAlSi}_2 \text{O}_6 \) in Ni–Cr/porcelain interface.

(2) Firing temperature and firing time strongly affect the shear bond strength of Ni–Cr/porcelain interface relative to the different reaction layer structures. The shear bond strength reaches 43.8 MPa at firing temperature of 990 °C for firing time of 2.5 min. It is necessary to select proper firing parameters for improving the bond strength of metal/porcelain interface and the quality of PFM restorations.

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References