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An Interfacial Study of Borosilicate Glass and Fe-Ni-Co Alloy Joint

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Introduction

Borosilicate glass to Fe-Ni-Co alloy joint has been used for many years as hermetic and electrically insulating seals. It is not only excellent in thermal expansion matching but also in good wettability and bond strength [1]. By using electron probe microanalysis and scanning electron microscopy (SEM), it was reported that the oxide scale preformed on the alloy surface was dissolved during joining into the glass, forming a zone enriched in Fe resulting in chemical bonding and good matching of thermal expansion coefficient [1-3]. Mechanical bonding by penetration of the FeO-riched glass into the open porosity at the alloy surface was also observed [3]. However, interfacial study by transmission electron microscopy (TEM) has not been attempted, which is the aim of this work, to gain more direct evidence on the mechanism of adhesion between both materials.

Materials and Methods

The joints of a borosilicate glass to an Fe-Ni-Co alloy in a commercial, medical x-ray tube were studied. The cross-sectional TEM specimens were prepared to electron transparency by dimpling together with electrochemical polishing and ion milling. The interfacial microstructure was studied by using JEOL JSM-6335F SEM and JEOL JEM-2010 TEM-STEM operated at 200 kV.

Results and Discussion

The microstructure at the glass-alloy interface can be divided into four regions (Fig 1a). Region I is an alloy with no change in chemical composition. Region II is the porous Fe-depleted area underneath the alloy surface. The thickness of porous layers on the left- and the right-hand side interface (Fig. 1b and 1c) is approximately 5 μm and 25 μm. The glass penetrating into these open pores should create a strong mechanical bonding. These porous layers are enriched in Ni and also in Co. Region III is the Fe-riched zone in the glass, while Region IV is the bulk of the glass. X-ray line scans in TEM investigation (Fig. 2) revealed that not only Fe but also Ni and Co were dissolved into the glass. Ni and Co must therefore be involved in chemical bonding between the glass and the alloy. Moreover, particles of iron silicate (Fe_2SiO_4) and iron silicon carbonyl ($SiFe_4(CO)_{16}$) with the size of about 10-100 nm were found within the glass in this area (Fig. 3).

Conclusion

SEM and TEM investigations confirmed chemical bonding by dissolvation of Fe into the glass and mechanical bonding by penetration of the FeO-riched glass into the porous layer on the alloy surface. However, TEM observation revealed also dissolvation of Ni and Co into the glass suggesting that these elements must be involved in chemical bonding. Devitrification of Fe_2SiO_4 and $SiFe_4(CO)_{16}$ was found as fine particles within the glass at the vicinity close to the interface.

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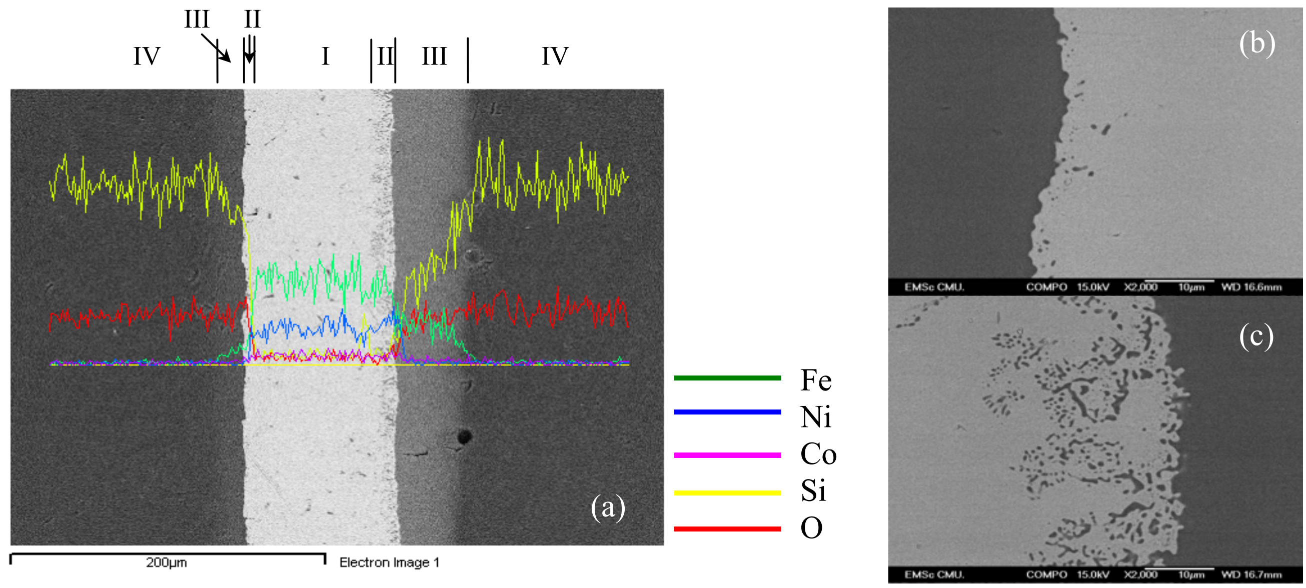

Fig. 1 (a) A SEM micrograph and x-ray line scans showing a cross section and distribution of elements at the borosilicate glass and Fe-Ni-Co alloy joint, (b) the left-hand side interface in (a), and (c) the right-hand side interface in (a).

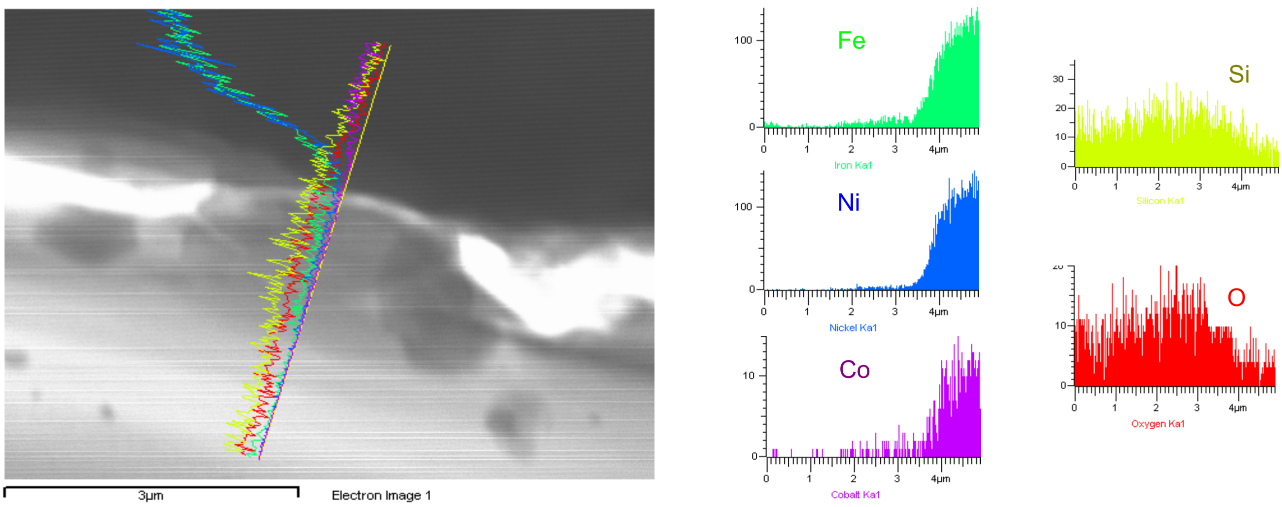

Fig. 2 A bright field TEM micrograph and STEM x-ray line scans at the borosilicate glass and Fe-Ni-Co alloy interface in Fig. 1(b).

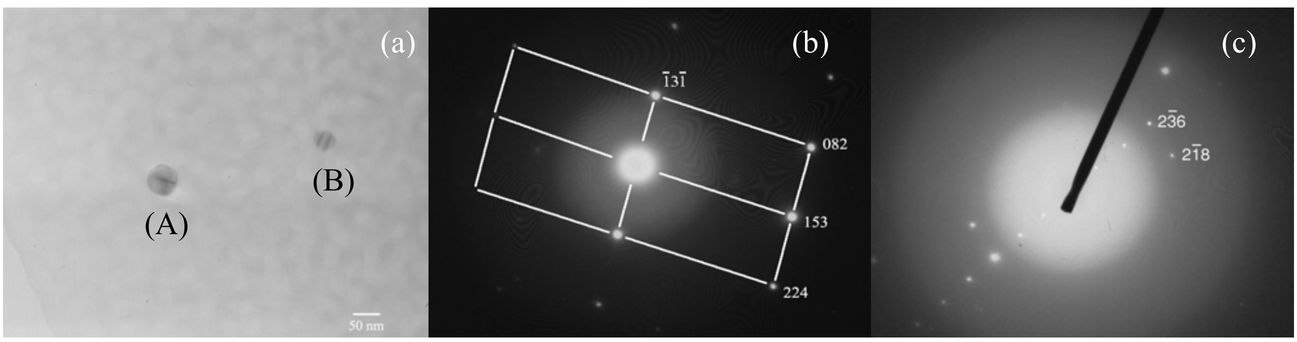

Fig. 3 (a) A bright field TEM micrograph showing precipitated particles within the glass at the area closed to the glass-alloy interface in Fig. 2, (b) and (c) are selected area diffraction patterns from particle A (Fe_2SiO_4) and B ($SiFe_4(CO)_{16}$), respectively.

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