Fabrication and Characterization of Mo-SiO$_2$ Composite

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1. Introduction
Functionally graded materials (FGM) are composite materials where the material composition and/or microstructure vary gradually to achieve enhanced physical properties with respect to the spatial coordinates [1]. Umemoto et al. fabricated Mo-SiO$_2$ FGM by the slip-casting method and proposed a new class of hermetically sealed HiDLs by using this Mo-SiO$_2$ FGM [2]. In general, metallic Mo and SiO$_2$ glass have a low wettability because of adhesive forces between a metal and glass; however their composite materials can be fabricated to form FGM. In this work, the interfaces between Mo and SiO$_2$ were mixed and sintered at different temperatures, then their interfaces were studied in detail by XRD and TEM.

2. Experiment
Mo powder (average size: 1.5 μm) and SiO$_2$ powder (average size: 1.4 μm) were used as starting materials. A part of this Mo powder (as-received Mo) was rinsed with hydrochloric acid (treated Mo). The mixture of Mo-SiO$_2$ water suspension was prepared under ultrasonic stirring. Combined sedimentation and pressurized slip-casting method were carried out, and then the slurry was cast in plastic molds on a porous ceramic block. After being dried at room temperature in vacuum for 24 h, samples 1, 2, 3 and 4 were sintered in vacuum at 1200 °C for 0 h, 1200 °C for 5 h, 1300 °C for 0 h and 1735 °C for 0 h, respectively. Further, the microstructure of each sample was observed by Co K$_\alpha$ radiation XRD (at 40 kV and 40 mA) and TEM (JEOL JEM-3200FSK electron microscope operating at 300 kV).

3. Results and discussion
Fig. 1.a shows the XRD pattern of as-received Mo-SiO$_2$ containing Mo and MoO$_3$ peaks. This suggests before sintering, MoO$_3$ layers exist on the surface of Mo particles (see also Fig.1.b). According to XRD results obtained from as-received Mo-SiO$_2$ at higher temperatures, MoO$_3$ peaks gradually distinguished. Meanwhile, MoSi$_2$ peaks started to appear due to a possible chemical reaction between Mo and SiO$_2$ (Equation 1). Contrary to as-received Mo, treated Mo XRD results showed Mo peaks without any other peaks (Fig. 2.a).

$$2\text{MoO}_3 + 4\text{SiO}_2 \rightarrow 2\text{MoSi}_2 + 7\text{O}_2 \quad (1)$$

In order to confirm this chemical reaction in the composites, we also performed
transmission electron microscopy (TEM). Fig. 2.b represents HR-TEM image and FFT pattern of MoSi$_2$ observed in $<$111$>$ direction which confirms the presence of MoSi$_2$.

References
