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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_α 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).

$2MoO_3 + 4SiO_2 \rightarrow 2MoSi_2 + 7O_2$ (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

[1] Y. Miyamoto, W.A. Kaisser, B.H. Rabin, A. Kawasaki and R.G. Ford, *Functionally graded materials: design, processing and applications*, *Mater technol series*, Kluwer Academic Publisher (1999).
[2] Umemoto, A., Hayashi, K., Hayano, K., Saito, N., Kaneko, K., Nakashima, K., *Ceramic Transactions* 198 (2007), pp. 219-224.

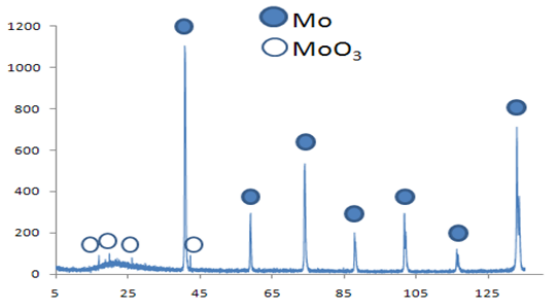

Fig.1.a .XRD pattern of as-received Mo-SiO_2 containing Mo and MoO_3 peaks at 1200 °C for 0 h.

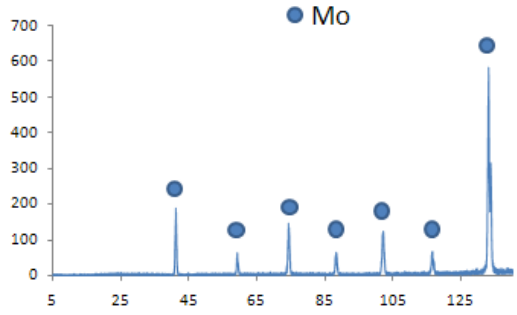

Fig. 2.a. XRD pattern of treated Mo-SiO_2containing Mo peaks at 1200 °C for 0 h without any other peaks.

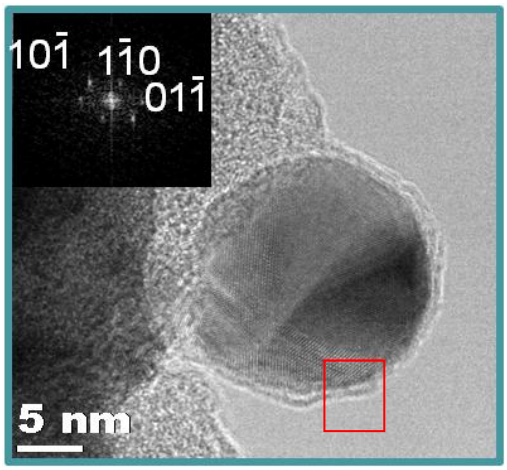

Fig. 1.b. HR-TEM and FFT pattern of as-received Mo in sintering temperature of 1200 °C for 0 h show MoO_3 on the surface of Mo.

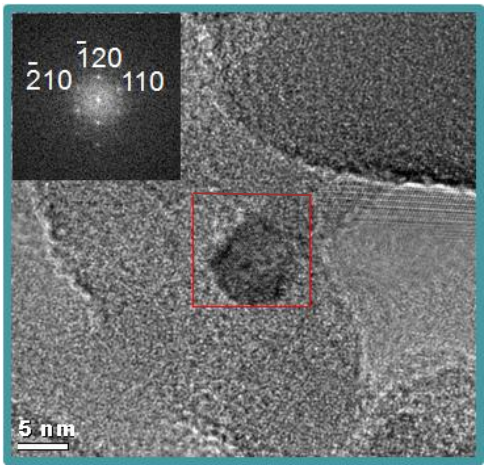

Fig. 2.b. HR-TEM image and FFT pattern of $MoSi_2$ observed in <111> direction.