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# Neutron Diffraction Analysis of Tungsten-Molybdenum-Disilicide Powders Formed by Self-propagating High Temperature Synthesis

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**Abstract**

*Tungsten-molydiside $W_xMo_{1-x}Si_2$ was synthesized by self-propagating high temperature synthesis (SHS). The SHS product with the initial composition of (0.5Mo+0.5W+2Si) contains 23.9% $MoSi_2$, 40.89% $WSi_2$ with remaining 9.11% Mo, 9.16% Si and 16.94%W. Lattice parameters of the $MoSi_2$ and $WSi_2$ determined by Rietvelt analysis were a=0.3206 nm, c=0.7841 nm and a=0.3212 nm, c=0.7822 nm, respectively.*

**Keywords: tungsten molydisilicide, self-propagating high temperature synthesis, neutron diffraction**

## 1. Introduction

Moly-disilicide widely used as electric heating element includes 2-10 vol.% of vitreous silicon dioxide($SiO_2$) phase to prevent oxidation during the reaction and/or milling process, that results in decreasing both the interface bonding strength and the high temperature strength.[1] In this study, moly-disilicide with tungsten addition was synthesized by SHS and the final phase was analyzed by neutron diffractometry.

## 2. Experimental method

Each powder mixture with stoichiometric amounts of molybdenum, silicon and tungsten powders (Aldrich, USA) was mechanically blended and compacted into a disk-shaped pellet under 100 MPa pressure. The green pellet with 12 mm in diameter and 25 mm in height was ignited in a SHS reaction chamber under argon atmosphere. The morphology of the SHS products was analyzed using scanning electron microscope (Jeol 35C). The powder neutron diffraction spectra from $5^o$ to $155^o$ were measured using the 32-detector high resolution powder diffractometer (HRPD).

## 3. Results and Discussion

Fig. 1-a is typical neutron diffraction pattern of (W, Mo)$Si_2$ prepared by SHS. The Rietveld refinement of each pattern converged to good agreement ($\chi^2$ = 1.88). The lattice parameter of the $Si_2Mo$ phase, a = 0.3204 nm, c = 0.7844 nm obtained from the Rietveld refinement. In the sample prepared by the powder mixture with the initial composition of (0.5Mo+0.5W+2Si), the Rietveld refinement of a structural model consists of the $Si_2Mo$(23.9%), $Si_2W$(40.89%), Mo(9.11%), Si(9.16%) and W(16.94%). The lattice parameter of the $Si_2Mo$ and $Si_2W$ phases were a = 0.3206 nm, c = 0.7841 nm and a = 0.3212 nm, c = 0.7822 nm, respectively. The combustion temperature of the initial composition of (Mo+Si) powder mixture was about $1450^oC$. The tungsten added in the initial powder mix retarded the reaction. The combustion temperature of the (0.5W+0.5Mo +Si) powder mixture was about $1415^oC$. Considering the binary phase diagrams of Si-W and Si-Mo, both combustion temperatures were above silicon melting point ($1414^oC$) and below molybdenum and tungsten melting points and above the eutectic temperatures of Si-$MoSi_2$ ($1410^oC$) and Si-$WSi_2$ ($1392^oC$). This means that the combustion reaction occurs between liquid silicon and solid molybdenum and tungsten.

Fig. 1-b is morphologies of final products prepared by SHS reaction with initial molybdenum particle size of 10μm. The reaction product was of partially sintered and its particle diameters are in a range from 1 to 2μm, which are smaller than that of initial molybdenum powder. Inner particle size is larger than outer particle size. This means that the microstructure of the SHS products depends on the combustion behavior. Since the diffusivity of the elements significantly depends on the temperature, the addition of tungsten results in retarding the formation and changing the composition of the final products. This supports that molybdenum and tungsten atoms diffuse into liquid silicon and nucleation occurs on a solid powders surface followed by heterogeneous nucleation mechanism.[2, 3] Hence, the formation mechanism suggested in this study include solution-precipitation in which molybdenum and tungsten

powders are melted and supersaturated in liquid phase, and heterogeneous nucleation occurs on solid powder to form complete solid solution.

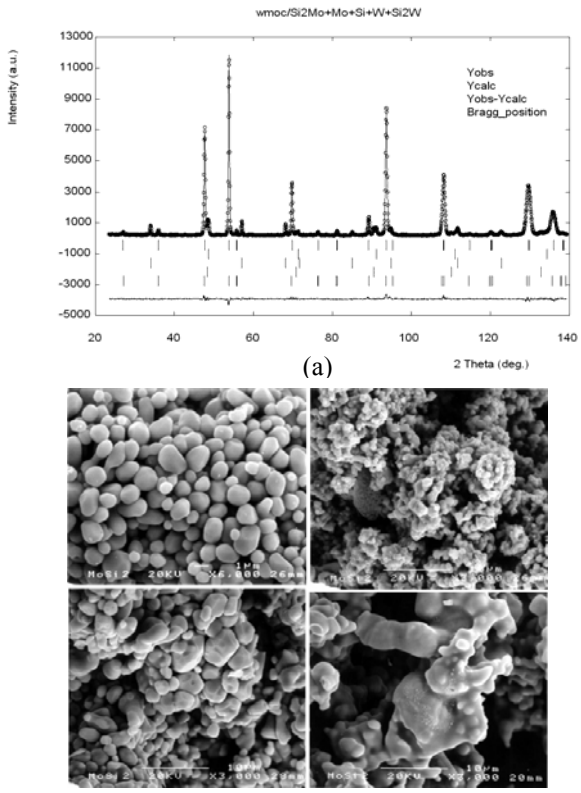


**Fig. 1 Typical neutron diffraction spectra of tungsten moly-disilicide formed by SHS and Scanning electron micrographs of final product with initial powder size of molybdenum and position (top left and bottom left : outer and inner surface formed by using 10 μm Mo, top right and bottom right : outer and inner surface formed by using 75 μm Mo)**

## 4. Summary

Moly-disilicide with tungsten addition was synthesized by SHS. Tungsten addition decreases the combustion temperatures from 1450°C and 1415°C. Lattice parameter of the $Si_2Mo$ phase, is a = 0.3204 nm, c = 0.7844 nm. Final product with 25 % tungsten addition contains $Si_2Mo$ 23.9%), $Si_2W$(40.89%), Mo(9.11%), Si(9.16%) and W(16.94, in which lattice parameter of the $Si_2Mo$ and $Si_2W$ phases are a = 0.3206 nm, c = 0.7841 nm and a = 0.3212 nm, c = 0.7822 nm, respectively. The formation mechanism of the tungsten moly-disilicide during SHS includes solution-precipitation in which molybdenum and tungsten atoms diffuse into liquid silicon and heterogeneous nucleation occurs on solid molybdenum and tungsten powders to from complete solid solution.

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