

## Influences of Doping Methods on Microstructure and Fracture Toughness of Mo-La Alloys

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### Abstract

Lanthanum oxide was introduced to molybdenum powder by liquid-liquid doping and liquid-solid doping respectively. Mo alloys were prepared by powder metallurgy technology. The size distribution and feature of dopant particles and the fractographs of Mo alloys were investigated by TEM and SEM respectively. The results indicated that liquid-liquid doping method is favorable for refining and dispersing  $\text{La}_2\text{O}_3$  particles uniformly in matrix. Fracture toughness of Mo alloys prepared by liquid-liquid doping showed better results than that of liquid-solid doping. Furthermore, the influences of the size distribution of  $\text{La}_2\text{O}_3$  on properties of Mo alloys was discussed by dislocation pile-up theory.

**Keywords :** doping, fracture toughness, microstructure, dopant particles, Mo-La alloys

### 1. Introduction

It is well known that  $\text{La}_2\text{O}_3$  particles in Mo matrix could refine grains, purify and strengthen the grain boundaries, increase the recrystallization temperature of Mo alloys, and decrease the ductile-to-brittle transition temperature (DBTT)<sup>[1-4]</sup>.

There are three methods to introduce  $\text{La}_2\text{O}_3$  to Mo alloys: Solid-solid doping, Liquid-solid doping, and Liquid-liquid doping. Different methods would lead to different properties of Mo alloys. Due to the size distribution of  $\text{La}_2\text{O}_3$  in alloy is affected by the properties of  $\text{La}_2\text{O}_3$  powder, solid-solid doping isn't discussed in this paper. For liquid-solid doping and liquid-liquid doping, the doping medium is the solution of lanthanum's compound, which is independent of  $\text{La}_2\text{O}_3$  powder. It is necessary to discuss the distribution of  $\text{La}_2\text{O}_3$  and properties of Mo alloys prepared by different doping methods to optimize doping processes.

### 2. Experimental and Results

Mo rods with a diameter of 17 mm was sintered after compressed by CIP. Doped methods, contents of La and densities of samples were listed in table 1. Fracture toughness test was performed in three-point bending method at room temperature according to the standard ASTM E399. Slices 0.2mm in thickness were cutted from sintered Mo alloy. The slices were grinded and electro-polished in a twin-jet apparatus. Dopant particles were observed by TEM. The component of second particles were determined by diffraction spots. The micrograph and fractographs were observed by SEM, the ingredients of impurities were determined by EDS.

**Table 1. Doping methods, contents of La and density of Mo alloy**

No.	Doping Method	Contents of La (wt.%)	Density (g/cm <sup>3</sup> )
SY-1	liquid-liquid	0.26	10.0
SY-2	liquid-solid	0.26	9.75

Figure1a, b were micrographs of SY-1 and SY-2. Black grains were determined as  $\text{La}_2\text{O}_3$  by diffraction patterns.  $\text{La}_2\text{O}_3$  grains in SY-1 dispersed uniformly and showed spherical of nearly the same diameter, and dislocations were pined by  $\text{La}_2\text{O}_3$  grains. The diameter of 97.8%  $\text{La}_2\text{O}_3$  grains in SY-1 was below 40nm. In SY-2, big  $\text{La}_2\text{O}_3$  particles were ellipsoid-shape, and agglomerated together, small particles were sphere-shape, and dispersed. The diameter of 78%  $\text{La}_2\text{O}_3$  grains in SY-2 was below 200nm. From the results, it could be concluded that liquid- liquid doping can refine and disperse  $\text{La}_2\text{O}_3$  grains sufficiently in Mo alloys, the shape of  $\text{La}_2\text{O}_3$  are uniform.

Fracture toughness,  $K_{IC}$ , of SY-1 was 11.9  $\text{MPa}\cdot\text{m}^{1/2}$ , but that of SY-2 was only 7.3  $\text{MPa}\cdot\text{m}^{1/2}$ . Fractographs of the specimens are as shown in Fig.2. The fracture surface of SY-1 was unequal with concaves and protrusions, and cracks can be seen to become deflected during propagating. The sintered pores were not observed in Mo alloy except concaves in triangle boundaries. The size distribution of Mo grains was extensive, and the fracture mode of some grains is transgranular, whereas those of others is intergranular as shown in Fig2a. However, the fracture surface of SY-2 was plain, and the fracture mode was typically intergranular. There were many sintered pores in triangle boundaries, Mo grains were uniform in size as shown in Fig2b. The results of EDS showed that there were impurities, such as Ca, K, at

the interface of  $\text{La}_2\text{O}_3$  particles and matrix in Mo-La alloys.

Nano  $\text{La}_2\text{O}_3$  with high surface energy in SY-1 may form liquid phase with other impurities at sintering temperature. It would benefit extruding pores, facilitating sintering, and increasing the densities of Mo alloys. The clustering impurity at the boundaries was decreased by dopant particles, so that the boundary strength was increased<sup>[5-10]</sup>. The more the dopant particles are, the less the impurity at the boundaries is. A number of  $\text{La}_2\text{O}_3$  in Mo alloy would distribute dislocations uniformly<sup>[12]</sup>, reduce dislocation density, decrease the stress concentration, and passivate the tip of cracks, and lead to the crack deflected and the fracture energy is absorbed. The more the dopant particles are, the more the energy absorbed is. So the fracture toughness of SY-1 is higher than that of SY-2.

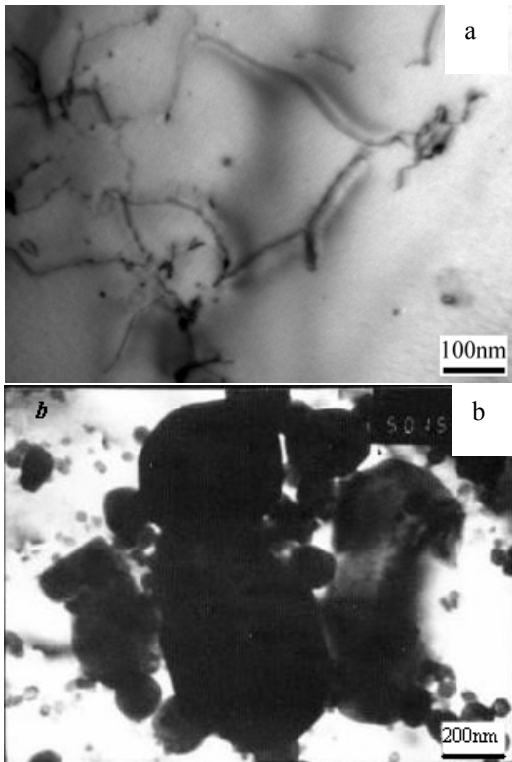


Fig. 1 Micrograph of Mo alloys a.SY-1 b.SY-2

### 3. Summary

Liquid-liquid doping method was confirmed to be able to refine and disperse  $\text{La}_2\text{O}_3$  particles uniformly. Fracture toughness of SY-1 was higher than that of SY-2.

Many nano  $\text{La}_2\text{O}_3$  in SY-1 were found to purify and increase the boundary strength, distribute dislocations uniformly, promote sintering of Mo alloy, increase density of sintered compact and strength of Mo alloy.

A number of  $\text{La}_2\text{O}_3$  in Mo alloy were found to passivate the tip of cracks, and the fracture energy could be absorbed. The more dopant particles there are, the more the energy is absorbed.

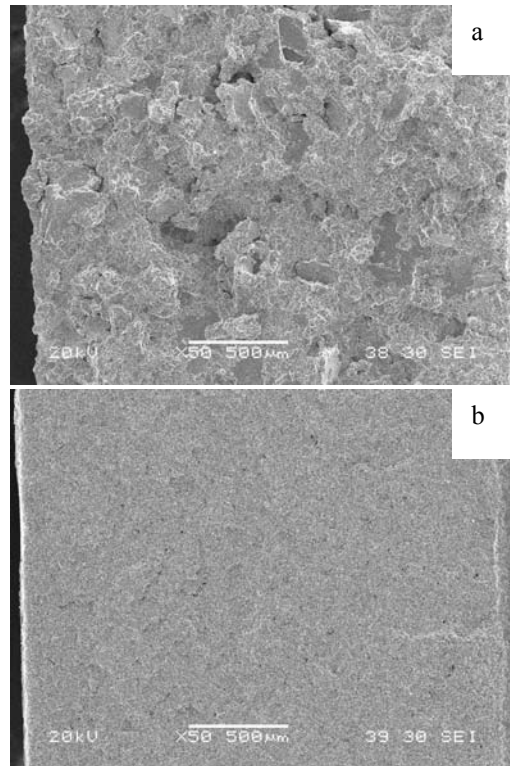


Fig. 2 Fractograph of Mo alloys a. Fractograph of SY-1; b. Fractograph of SY-2

### 4. References

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