Fabrication of Pure Refractory Metals by Resistance Sintering under Ultra High Pressure

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Abstract

Refractory materials, such as W and Mo, are very useful elements for use in high-temperature applications. But it is not easy to fabricat pure W and Mo with very high density and retaining very fine grain size because of their high melting point. In this paper, a newly developed method named as resistance sintering under ultra high pressure was use to fabricate pure fine-grained W and Mo. The microstructure was analysis by SEM. The sintering mechanism is primary analyzed. Basic physical property of these sintered pure W and Mo, such as hardness, bend strength, are tested.

Keywords : Tungsten, Resistance Sintering, Ultra High Pressure, Fine Grain Size

1. Introduction

Refractory metals, such as W and Mo, have many favorable properties for use as high heat flux components and high-power density structural materials in radiation environment [1]. However, it exhibits serious embrittlement in several regimes, i.e., low temperature embrittlement, recrystallization embrittlement and radiation embrittlement. In order to overcome such embrittlement, there has been an interest in fine-grain dense objects of refractory metals containing no liquid-phase sintering aids recently. Due to the high melting point of refractory metals, it is not easy to fabricate these kinds of materials with very high density and retaining fine-grain size. This has been the motivation for exploring new sintering techniques, and using ultrafine powders as the starting material. Several method have been developed for fabrication unltr-fine grained W, such as by severe plastic deformation [2], microwave sintering [3].

Ultra-high pressure (>1GPa) assisted resistance sintering has been successful in producing W-Cu composite in our former works [4]. In this work, the possibility of fabricating pure W and Mo without any sintering additive aided by the same method is studied.

2. Experimental and Results

W powder with particle size of 0.2, 1, 3μm and a purity of >99.5%, and Mo powder with a particle size of 2μm and a purity of >99.5% are used. The pure W or Mo powder are cold pressed in a steel mould to form a green compact with a diameter of 20 mm and height of 6 mm.

A special setup introduced in reference [4] was use to fabricate pure W and Mo. The sintering parameters were: under pressures of 8 GPa, an electric power input of about 20 kW and sintering times of 65s.

The density was measure by Archimedes' method. SEM was use to characterize the microstructure of the sample.

Table 1 shows the relation between pressure and the relative density of pure W and Mo. When only ultra-high pressure is loaded, the green compact can acquire very high density, especially when the particle size is larger than 1μm. Mo can get higher density than W at the same loaded pressure, due to its higher plasticity and lower hardness than those of W.

Table 1. Pressure vs relative density

Materials	W, 0.2μm	Mo, 2μm	W, 1μm	W, 3μm
Pressure, GPa	8	8	8	8
Relative density, %	91.21	96.32	94.59	95.32

Table 2. Micro hardness and bend strength

Materials	W, 1μm	W, 1μm	W, 0.2μm	Mo, 2μm
Sintering condition	8Gpa, 0KW	8Gpa, 20KW	8Gpa, 20KW	8Gpa, 20KW
Relative density, %	94.59	97.49	92.23	98.21
Hardness, HV	348.22	772.33	1078.32	352.63
Bend strength, MPa	187.56	561.12	598.25	382.12

Table 2 shows the hardness and bend strength of W and Mo fabricated at different conditions.

When only ultra-high pressure loaded on the green compact, although it can get very high density, its hardness and bend strength are not very high, as the particles are only mechanical combined, as show in Fig.1. But when a very strong curren loaded on the compressed compact at the same time, its hardness and bend strength increased obviously, althrough its relative density has little increase. When very strong current pass through the compact, lots of Joule heat will be produced quickly due to the resistance of W, thus heating and sintering the compact. Although the whole temperature of the sample is only about 1173K according to FEM analysis, but it can be supposed that the temperature at the interface of W particles should be very high due to the interspace among the particles, thus surface diffusion would occur and particles will be bonded together mainly by solid state sintering. Fig. 2 shows the morphology of the well-sintered W with particle size of 1μm. It is obvious that the fine W powder sintered and bonded well together. The grain size of W is nearly same to the initial powder size.

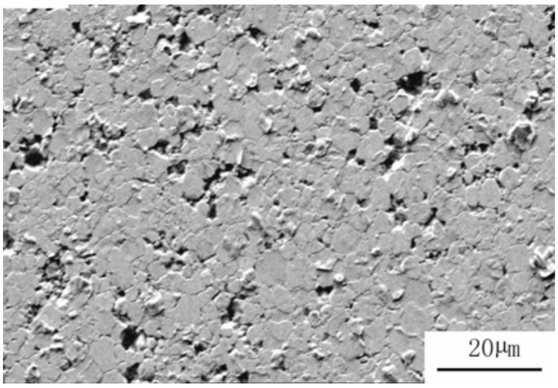

Fig. 1 SEM micrographs of tungsten loaded only b y 8GPa pressure. The particle size of W is about1μ m

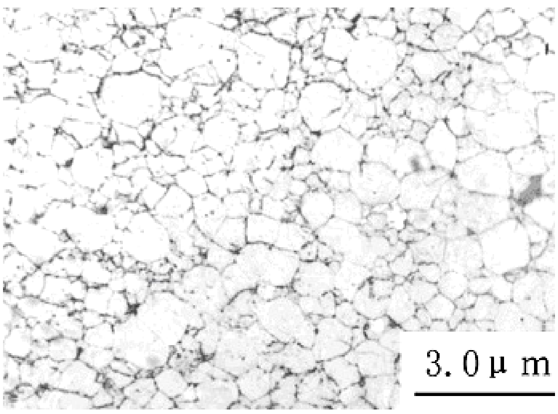

Fig. 2 SEM micrographs of W sintered at 8GPa, 2 3kW. The particle size of W is about 1μm

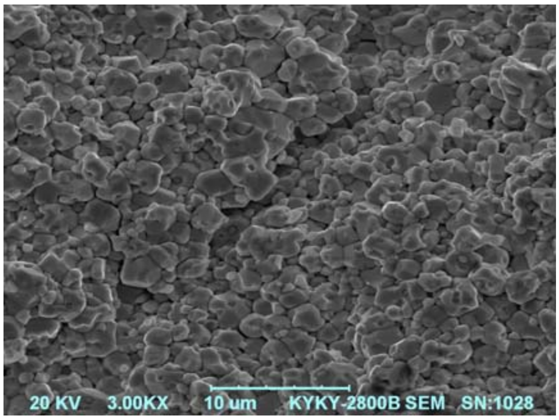

Fig. 3 SEM micrographs of W sintered at 8GPa, 20 kW. The particle size of W is about 0.2μm

Fig.3 shows the SEM micrographs of fracture surface of W with powder size of 0.2μm sinterd at 8 GPa, 20kW. The cracks spread along the grain boundary. Laminated crack is also a typical crack mode. It should be note that the grain size of the W has little growth. This shows that resistance sintering under ultra-high pressure is an effective method to fabricate nano-sized materials.

3. Summary

Resistance sintering under ultra high pressure is a very effective method to fabricate ultra-fine grained pure W and Mo. The grain size of W and Mo can be retained nearly same to its initial powder size, and thus get excellent mechanical properties. When the particle size is larger than 1μm , the relative density of sintered product is more than 97%. For sub-micro powder, the relative density of sintered product is only about 92%, further study should be done for improving the sinter density.

4. References

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