Investigation on sintering and deformation strengthening of Mo-Cu alloy

HU Bao-quan, WANG Yan-zhong, LIU He-ping

(*School of Materials Science and Engineering, North University of China, Taiyuan 030051, China*)

Abstract: Mo-8wt%Cu nanocomposite powders were fabricated by mechanical alloying, and full density alloy was obtained via liquid-phase sintering and post-treatment process. The microstructure of Mo-8wt%Cu alloy was investigated by scanning electron microscope (SEM), and the effects of process parameters on relative density, tensile strength and elongation were studied. The results indicate that the relative density of Mo-Cu alloy is 98.6% after sintering at 1 250 ℃ for 30 min, and its microstructure is composite network. The full density of Mo-Cu alloy can be obtained when specimens are treated through deformation strengthening process of rotating forging and hydrostatic extrusion. The tensile strength and elongation rate are 576 MPa and 5.8%, respectively, when hydrostatic extrusion deformation degree is 40%.

Key words: mechanical alloying; Mo-Cu alloy; liquid-phase sintering; deformation strengthening

CLD number: TF125.24 **Document code**: A

Article ID: 1674-8042(2017)02-0173-05 **doi**: 10.3969/j.issn.1674-8042.2017.02.010

0 Introduction

Mo-Cu alloy is widely used in electrical contact, electrode, large-scale integrated circuits and high-power microwave devices for substrates, patches, connectors and cooling components and other civilian components owing to its excellent characteristics of electrical conductivity, thermal conductivity, low thermal expansion coefficient and ablation resistance. It is also used to prepare the military on a variety of missile engine nozzle, gas rudder, nose cone and other high temperature components[1-4].

Mo-Cu alloy prepared by conventional powder metallurgy (powder mixture + molding + high temperature sintering) has low density, poor composition heterogeneity and low mechanical properties, which cannot meet some requirements. Currently, the common method for the preparation of molybdenum copper alloy is that copper infiltrated molybdenum matrix alloy[5]. This method significantly improves the density and performance of Mo-Cu alloy, but it is difficult to control pore distribution in the molybdenum skeleton sintered at high temperature, and molybdenum particles are easily aggregated and grow up, which leads to the in-homogenous microstructure of Mo-Cu alloy. The performance of Mo-Cu alloy strongly depends on the particle size and composition uniformity of Mo-Cu powders[6]. Thus, the preparation of Mo-Cu powders with small particle size and uniform composition play an important role for preparing high-performance Mo-Cu alloy.

This paper proposed a new method to prepare Mo-Cu alloy using high energy ball-milling technology, and the sintering densification mechanism of nanocomposite powders and the microstructure and properties of Mo-8wt%Cu alloy were investigated.

1 Experimental

Mo powders (average size, 3.2 μm; purity, 99.9%) and Cu powders (average size, 38 μm; purity, 99.6%) were mixed as the ingredients of Mo-8wt%Cu composition. The mixture of powders was

Received date: 2017-03-15

Corresponding author: HU Bao-quan (baoquan-hu@nuc.edu.cn)

added into V-mixer and mixed for 3 h. Subsequently, the pre-mixed powders were mixed in QM-1L ball milling machine for 30 h. The dry Ar was charged into the grinding pot to prevent the oxidation of powders at high temperature for a long ball-milling time. The nanocomposite powders were pressed into green bodies at 200 t in cold isostatic pressing machine. The green bodies were sintered at 1 100－1 300 ℃ for 30 min, and then treated at 300 t by hydrostatic extrusion and 20 t by rotary forging machine for deformation processing.

The microstructure of Mo-8wt%Cu alloy was observed by using XL-20 scanning electron microscope (SEM). The crystal grain size was measured by X-ray diffraction (XRD), D/MAX-RB. The densities of specimens were determined by Archimedes method.

2 Experiments and analysis

2.1 Liquid sintering of Mo-8wt% Cu nanocomposite powders

The green body was fabricated by pressing Mo-8wt%Cu nanocomposite powders at 210 MPa using cold isostatic pressing. Its size was Φ20 mm × 180 mm and the density was 7.52 g/cm^3. The green body was sintered in a hydrogen reducing atmosphere furnace.

The relation of relative density and crystallite Mo size with sintering time at 1 250 ℃ is shown in Fig. 1.

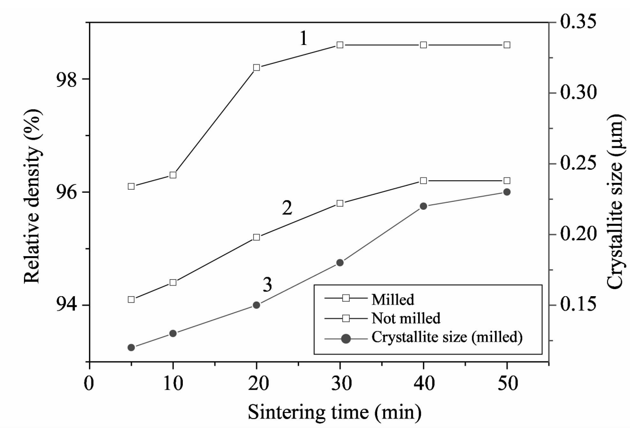


Fig. 1 Effect of sintering time on relative density and crystallite size of Mo-8wt%Cu

It can be seen that the sample achieves nearly full densification at 1 250℃ for 30 min for the milled nanocomposite powders, and the relative density is above 98%. The relative density is not continuously improved with sintering time (curve 1). The Mo grain size increases with increasing sintering time (curve 3), such as the grain size of sample is about 0.37 μm sintered for 30 min. For the no milling composite powder of Mo-8wt%Cu, the rate of densification is slower compared to the milled powders, such as the maximum relative density of sample is 95.6% sintered at 1 250℃ for 30 min (curve 2). The results indicate that nanocomposite powders have high sintering activity, and the high relative density of sample is obtained sintered at 1 250℃ for 30 min for milled nanocomposite powders.

The effect of sintering temperature on the relative density and tensile strength of Mo-8wt%Cu alloy is shown in Fig. 2.

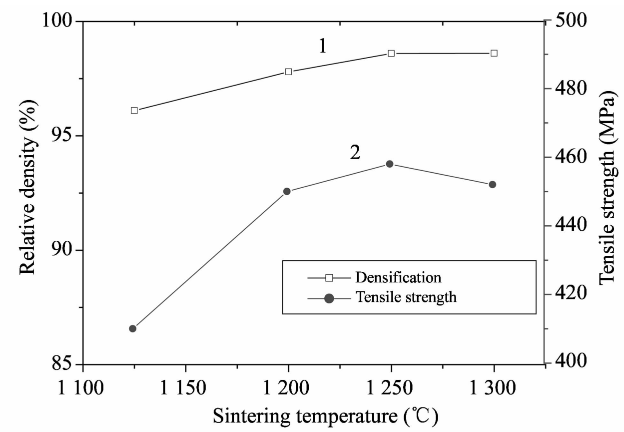


Fig. 2 Effect of sintering temperature on relative density and tensile strength

It can be seen that the relative density gradually increases with sintering temperature, and reaches the maximum value (98.6%) at 1 250℃ since the viscosity of the liquid copper reduces with the increase of sintering temperature, which induces that Mo particles are easy to flow and re-arrange to achieve the most compact arrangement[7]. However, when the sintering temperature is above 1 250 ℃, the fluid of liquid copper are sufficient to meet Mo particles adjusting the position, so the relative density does not continuously improve with further increasing sintering temperature (curve 1). In general, tensile strength improve with relative density, and thus the tensile strength also improves with increasing sintering temperature. The tensile strength reaches the maximum value at 1 250 ℃, and tensile strength is 458 MPa. When the sintering temperature is above 1 250 ℃, the tensile strength decreases due to the growing of Mo crystallite size and seeping of liquid copper from the material[8] (curve 2).

The relation of Mo crystallite size and elongation rate of sintering body of Mo-8wt%Cu with sintering temperature are shown in Fig. 3. It is seen that the crystallite size of Mo increases with sintering temperature (curve 1), and elongation rate reaches the peak value (6.1%) at 1 250 ℃ (curve 2). The elongation rate starts to decline when the sintering temperature is above 1 250 ℃, which may result from the overgrowing of Mo grain.

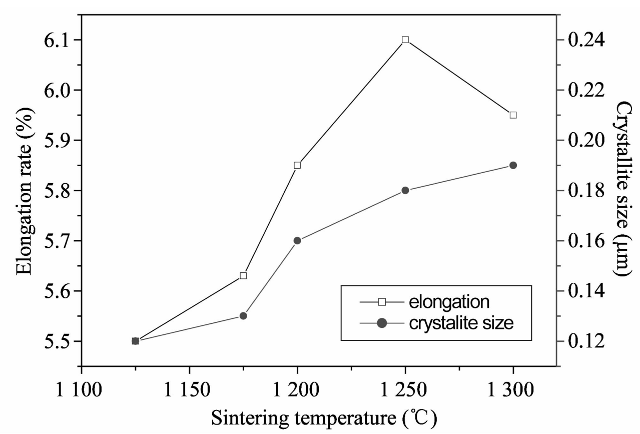
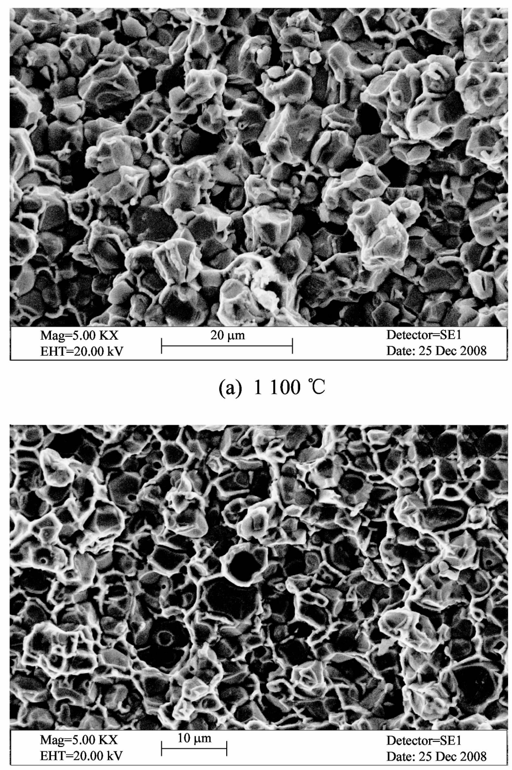


Fig. 3 Effects of sintering temperature on Mo crystallite size and elongation rate of samples

2.2 Microstructure of Mo-8wt%Cu alloy

The relation of the microstructure of Mo-8wt%Cu with sintering temperature is shown in Fig. 4. Liquid copper has high viscosity and poor fluid when the sintering temperature is below 1 100 ℃ (melting point of copper is 1 083 ℃), which leads to difficult completion of rearrangement and migration of Mo particles. The sintering mechanism mostly relies on solid-state diffusion at this stage, and Mo particles are connected more loosely, and the samples have large porosity and low relative density (Fig. 4(a)). When the sintering temperature is at 1 250 ℃, the viscosity of liquid phase copper decreases, and the sintering mechanism is changed into liquid phase sintering. The liquid Cu on Mo particles has good wettability[9,10], and the strain characteristic of Mo particles gradually disappears with increasing of sintering temperature. Mo particles grow up by the way of interconnection, and finally the microstructure becomes a composite network of Mo and Cu, and Cu is the binding phase, distributed along the grain boundary.

Comparing Fig. 4(a) with Fig. 4(b), it can be seen that the relative density of the sample sintered at 1 250 ℃ for 30 min is much higher than that sintered at 1 100 ℃.



(a) 1 100 ℃

(b) 1 250 ℃

Fig. 4 Microstructure of Mo-8wt%Cu alloy sintered at different sintering temperature for 30 min

2.3 Deformation processing of Mo-8wt%Cu

Although the relative density of Mo-8wt%Cu is very high via the liquid phase sintering of nanocomposite powders, the relative density of the sample does not yet reached the full densification. Compaction treatment process of rotary forging and hydrostatic extrusion was used for further improving the strength and relative density of Mo-8wt%Cu alloy.

The mechanical properties of Mo-8wt% Cu by strain strengthening of rotary forging and hydrostatic extrusion are shown in Table 1.

Table 1 Mechanical properties of Mo-8wt%Cu with rotary forging and hydrostatic extrusion

Strain methods	ε (%)	K (%)	σ_b(MPa)	δ (%)
No strain	0	98.6	458	6.1
rotary forging	40	99.8	513	5.8
Hydrostatic extrusion	40	100	576	5.8

It can be seen that the relative density is 100% with deformation 40%, elongation rate does not obviously falls, and the tensile strength makes a great many of improvement comparing to no deformation strengthening of Mo-8wt%Cu alloy. The main reason is the fibred microstructure is obtained, as shown in Figs. 5 and 6.

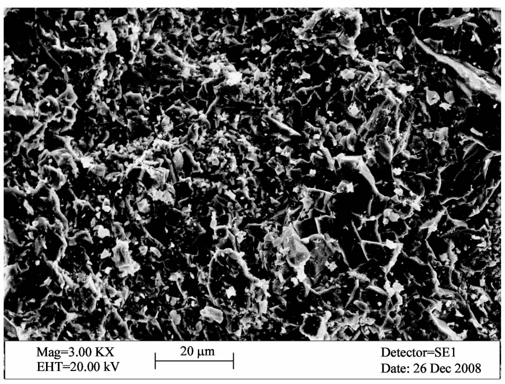


Fig. 5 Microstructrue of Mo-8wt%Cu alloy by rotating forging with deformation of 40%

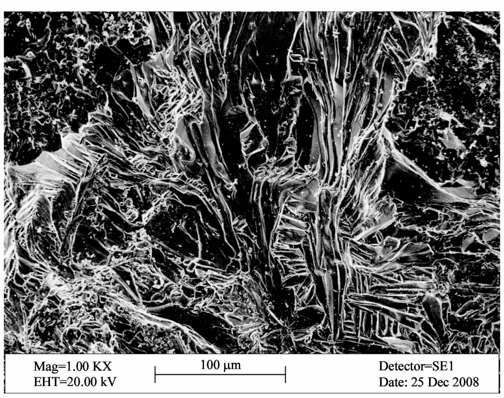


Fig. 6 Microstructure of Mo-8wt%Cu alloy by hydrostatic extrusion with deformation of 40%

It can be seen that obviously improves the tensile strength, and the plastic cannot greatly decrease because of strain hardening[11-12]. Moreover, the strain is in a situation of three directions compressive stress in hydrostatic extrusion process, which makes internal inherent micro-cracks in organization of Mo alloy constantly close up in the hydrostatic extrusion process. The reduction of inner defects also plays an important role for the strain strengthening. Thus the effect of hydrostatic extrusion on the strain strengthening is better than rotary forging.

3 Conclusions

1) The green body of Mo-8wt%Cu nanocomposite powders can quickly achieve nearly full densification at the low temperate and in short time via the liquid phase sintering, implying that nanocomposite powders have high sintering activity.

2) The Mo crystallite size increases with increasing of sintering temperature and time, which is harmful to the improvement of mechanical properties. The microstructure of the sintered sample consists of the composite network of Mo and Cu, and Cu is the binding phase.

3) The strain strengthening is an effective method for improving mechanical properties of Mo-8wt%Cu alloy. After compaction post-processing of rotary forging and hydrostatic extrusion, Mo-8wt%Cu alloy can reach full densification, and mechanical properties greatly improve. Mo-8wt%Cu alloy treated by strain strengthening of hydrostatic extrusion has fewer surface, inner defects, and finer fibred microstructure than rotary forging.

References

[1] Suryanarayana C. Mechanical alloy and milling. Materials Science and Engineering, 2001, 46: 2-6.

[2] Suryanarayana C. Nanoparticle synthesis. Materials Today, 2005, 11: 62.

[3] LIU Hai-yan, LI Zeng-feng, TANG Hui-ping. Mo-Cu composite material prepared by mechanical alloy. Journal of Functional Material, 2007, 38: 3188-3190.

[4] Alam S N. Synthesis and characterization of W-Cu nanocomposites developed by mechanical alloying. Materials Science and Engineering, 2006, 433(1/2): 161-168.

[5] Maneshian M H, Simchi A, Hesabi Z R. Structural changes during synthesizing of nanostructured W-20wt% Cu composite powder by mechanical alloying. Journal of Alloys and Compound, 2007, 445/446: 86-93.

[6] Martinez V D P, Aguilar C, Marin J, et al. Mechanical alloying of Cu-Mo powder mixtures and thermodynamic

study of solubility. Materials Letter, 2007, 61(4/5): 929-933.

[7] Ivanov E Y, Suryanarayana C, Bryskin B D. Synthesis of a nanocrystalline W-25wt% Re alloy by mechanical alloying. Materials Science and Engineering, 1998, 251: 255-261.

[8] Aguilar C, Marin J, Ordonez S, et al. Study and methods of analysis of mechanical allodyed Cu-Mo powders. Materials Science and Engineering, 2007, 464: 288-294.

[9] Johnson J L, German R M. Powder metallurgy processing of Mo-Cu for thermal management applications. The International Journal of Powder Metallurgy, 1999, 35(8): 39-48.

[10] NAN Hai, QU Hai-ping, FANG Yu-cheng, et al. Powder Metallurgy Industry, 2004, 16(6): 22-26.

[11] ZHANG Zhao-hui, WANG Fu-chi, LI Shu-kui. Microstructure and properties of tungsten alloy deformed by hydrostatic extrusion. The Chinese Journal of Nonferrous Metals, 2001, 11(31): 88-91.

[12] ZHANG Zhao-hui, WANG Fu-chi, LI Shu-kui. Deformation strengthening to tungsten alloys through hydrostatic extrusion. Acta Armamentarii, 2002, 23(2): 224-227.

Mo-Cu 合金烧结和形变强化研究

胡保全，王延忠，刘和平

（中北大学 材料科学与工程学院，山西 太原 030051）

摘　要： 用机械合金化法制取 Mo-8wt%Cu 纳米复合粉末，采用液相烧结和后处理工艺制备了全致密 Mo-8wt%Cu 合金。通过扫描电镜对 Mo-Cu 液相烧结和变形加工后合金显微组织进行了分析，研究了各种工艺参数对 Mo-Cu 合金致密性、拉伸强度和延伸率的影响。结果表明，高能球磨的 Mo-8wt%Cu 纳米复合粉末坯体，经液相烧结后，其烧结态为 Mo 和 Cu 的复合网状组织，在 1 250 ℃烧结 30 min，可获得相对密度高达 98.6%的 Mo-Cu 合金。再经静液挤压和旋转锻造变形加工处理后，可获得全致密的 Mo-8wt%Cu 合金。在室温静液挤压 40%形变率的条件下，其拉伸强度可达 576 MPa，延伸率为 5.8%。

关键词： 机械合金化；Mo-Cu 合金；液相烧结；形变强化

引用格式： HU Bao-quan, WANG Yan-zhong, LIU He-ping. Investigation on sintering and deformation strengthening of Mo-Cu alloy. Journal of Measurement Science and Instrumentation, 2017, 8(2): 173-177. [doi: 10.3969/j.issn.1674-8042.2017.02.010]