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ARTICLE

Evolution of Micro-structure and Properties of W-80Cu Sheets During Heat Treatment

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Abstract: The effect of heat-treatment on the micro-structure evolution and properties of W-80Cu sheets was investigated. The micro-structure and fracture appearance of W-80Cu sheet were observed by scanning electron microscope (SEM) and transmission electron microscopy (TEM). The electrical conductivity and mechanical properties were also investigated. The results indicate that the electrical conductivity and elongation of the specimens after heat-treatment were higher than those of the un-heated while their hardness and room temperature tensile strength was lower. The maximum electrical conductivity and elongation of W-80Cu sheet after ageing at 600 ºC for 1.0 h were achieved and the dimples were deeper and more compact, and its size and distribution became uniform. The tensile fracture of W-80Cu sheet was the main manner of the inter-granular brittle fracture with dimple fracture. When raising the heat-treatment temperature around or above 800 ºC, the size and depth of the dimples became different and the steps and quasi-cleavage appeared at the local zone. Once the time was above 1.0 h, the local dimples became big and the tearing ridges of copper got longer due to the recrystallization growth of copper grains. During the heat-treatment, tungsten phase in W-80Cu sheet had not an obvious change, but a lot of dislocations around tungsten particles and grain boundary decreased in amount. Both tungsten nano-particles and copper matrix had a good interface relation, which benefited to the strength enhancement.

Key words: heat-treatment; micro-structure; electrical conductivity; mechanical properties

W-Cu alloy has excellent properties, such as high electrical and thermal conductivity, low thermal expansion coefficient, good weldability[1-3]. This alloy has attracted much attention in many fields, such as electronic industry, metallurgy, machinery industry, and military[4-7]. But it is difficult to adopt the sintering method to fabricate W-Cu alloy with high density and properties because W-Cu alloy has low solid solubility. So the manufacture of W-Cu sheet or bar needs the follow-up process. Currently, the main manufacturing techniques of W-Cu sheet are cold and hot-rolling. Belk et al [8] researched the effect of cold rolling on W-(10~40)Cu alloy made by the liquid infiltrating way. They found that when the deformation amount of the materials was below 25%, tungsten remained un-deformed, but only when the deformation amount was up to 50%, tungsten phase began to deform. Chaojun He et al [9] pressed the powder into the plates, then sintered them to prepare W-Cu sheets (a thickness of 1.0 mm). N. Yang et al [10] fabricated successfully W-20Cu alloy sheets (a thickness of 0.5 mm) by the sintering, cold rolling and re-sintering technique. By the multi-pass hot-rolling technique, the sheet with a thickness of 0.5 mm and a relative density of 99.87% was prepared using the commercial W-20Cu alloy with a thickness of 5 mm[11].

In recent years, scholars researched that W-Cu film with higher copper content has been prepared by using magnetron sputtering and plasma spray [12-15]. These W-Cu films have the high Cu content, but are too thin to be used as the plate or sheet. S. K. Vajpai et al [16] proposed the route of

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preparing the W-95Cu, W-80Cu and W-60Cu alloy strips, which consisted of preparing W-Cu nano-composite powders by high energy ball milling, cold-compacting, sintering and hot rolling. The relevant researches of W-Cu alloy with higher copper content are still lacking and mainly focus on the fabrication processes of alloy[17-21]. The post treatment technology may affect the micro-structure, properties and application of the materials. However, the scarce studies on the effect of post treatment technology have been conducted.

So, the present work attempts to heat-treat W-80Cu sheet under the different conditions. The effect of the vacuum heat-treatment condition on the micro-structure evolution and performances of W-80Cu sheet was investigated. By studying the annealing behaviour of the materials, it should be hoped to provide the ways of manufacturing and forming these materials.

1 Experiment

By a chemical co-deposition technique, W-80Cu composite powders were prepared using copper nitrate, ammonium metatungstate (AMT), and oxalic acid as the raw materials (seen Ref.[22]). The powders were pressed under 280 MPa, and then sintered to obtain W-80Cu alloy at 1070 ºC for 1.5 h under hydrogen atmosphere. The sintered specimens have been preheated at 950 ºC for 30 min, then hot-rolled to obtain W-80Cu sheets (a thickness of 2 mm) by a rolling mill WE-600 type universal hydraulic testing machine. The relative density, electrical conductivity and hardness HV of the sheet are 98.2%, 85.6%IACS and 1415 MPa, respectively. The electrical conductivity was much higher than the literature value (66.5%) [16]. This sheet was cut into the samples with 20 mm×20 mm. Under vacuum condition, the samples have been heat-treated at 400~1000 ºC for 0.5, 1.0, 1.5, 2.0, 4.0 h, then cooled to room temperature inside the furnace.

The microstructures of W-80Cu sheets before and after heat treatment have been analyzed by SEM (VEGA3-TESCAN) and transmission electron microscopy (Titan TM themis G2 60-300). TEM samples were cut by focused ion beam. The electrical conductivity of W-80Cu sheets was tested by D60K digital metallic conductivity measuring instrument. The hardness of the samples was measured using the Vickers micro-hardness tester (Indentec 5030 SKV) under 200 g load for 10 s. Tensile strength was tested by an universal electrical material testing machine (SHIMADZU AG-I250KN).

2 Results and Discussion

2.1 Micro-structure characteristics

2.1.1 Influence of heat treatment temperature on micro structure

Fig.1 indicates the influence of the heat-treatment temperature on the micro-structure of W-Cu sheets. The black zones in the figure represent copper phase while the bright zones are the tungsten grains. The results show that the tungsten grain size of W-80Cu sheets after heat-treatment has not changed appreciably. At around 800 ºC, tungsten recrystallization occurred while tungsten phase was still not fully recrystallized at 1000 ºC[23]. The growth of tungsten grain did not occur obviously when the heat-treatment temperature was below 800 ºC, as shown in Fig.1b~1d.

During the heat-treatment process, copper recovery and recrystallization mainly occurred. The starting and finishing recrystallization temperatures depended on alloy composition[24]. Because the fine tungsten particles were well distributed among the copper substrate, the dislocation motion and sub-grain boundary coalescence were inhibited effectively. The recovery process was delayed and the recrystallization temperature was even enhanced. Copper recovery needed a relatively

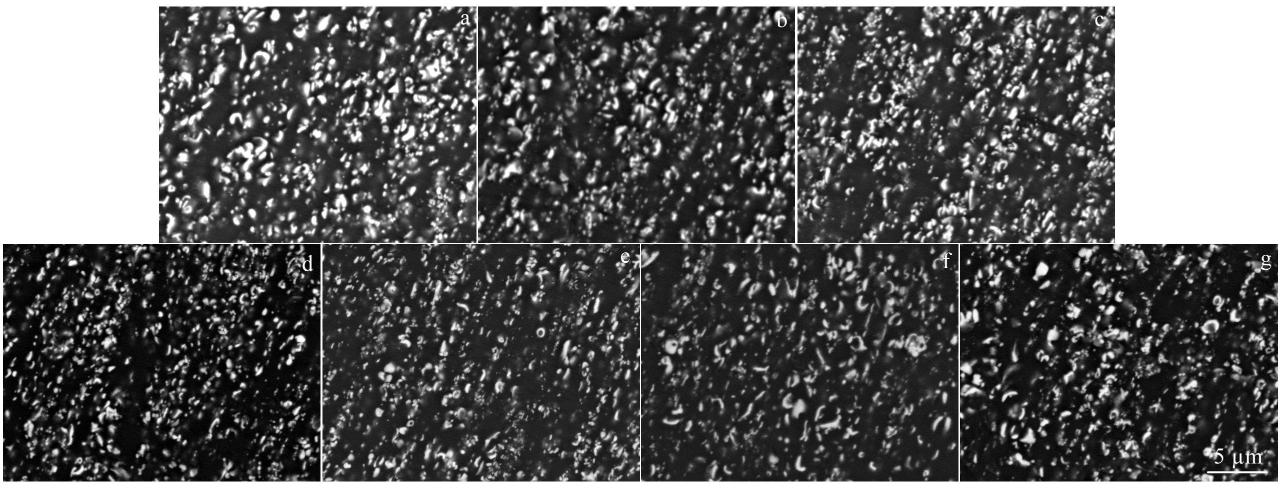

Fig.1 Back scattered electron images of W-80Cu sheets heat-treated at different temperatures for 1 h: (a) un-heated, (b) 400 ºC, (c) 500 ºC, (d) 600 ºC, (e) 800 ºC, (f) 900 ºC, and (g) 1000 ºC

long time at low annealing temperature. What's more, the diffusion rate of the atoms was relatively slow at low annealing temperature and the transport distance was not too far, which made the micro-structure of W-Cu alloy change slightly. With improving the heat-treated temperature, the earlier recovery and recrystallization began. As nano-copper particles could melt at about 550 ºC[25], liquid copper was filled into the gaps between the grains and surrounded tungsten grains through capillary action. This helped to form a better copper-net and reduce the pores in W-80Cu sheet after heat-treatment at 600 ºC. Once the temperature was up 800 ºC, copper grains and individual nano-grains of tungsten grew up with increasing the temperature, as shown in Fig.1f, 1g.

2.1.2 Influence of heat treatment time on micro-structure

Fig.2 shows the back scattered electron images of W-80Cu sheets after heat-treatment for different time at 600 ºC. The micro-structure of the sheets varied little with prolonging the heat-treatment time. At this temperature, the recovery and recrystallization of tungsten grain cannot happen while copper has experienced three processes: recovery, recrystallization and the grain growth. In the recovery stage, the density of point defects in the deformed metal decreased significantly. With prolonging continuously the heat-treatment time, the melt and diffusion of nano-copper particles made liquid copper fill in the pores and no copper content zone as well as the growth of copper. Further, the volume diffusion and grain boundary diffusion occurred sufficiently in W-80Cu sheets.

TEM images of the hot-rolled samples are provided in Fig.3. The black particles with near-spherical or polygon shape are tungsten and the bright zones are copper in Fig.3a. Also a lot of dislocation tangles are found in the copper matrix, particularly near the interface of tungsten particle, as indicated by the arrows in Fig.3a. In the hot-rolling process, the dislocations inside the material moved and proliferated under the action of external stress. Due to the repulsive force between the like dislocations, the dislocation loops released by the dislocation source were clogged, squeezed and arranged when the front dislocation encountered the strong pinning phase (W phase) in the moving process[26]. The hindrance to the grain boundary was so strong that the dislocations did not pass through the obstacles. The dislocations gradually increased and stacked at the grain boundary. So the high density dislocation clusters formed around tungsten particles, as shown in Fig.3a. The different grain orientations induced the different extent of the distortion during heat-rolling process, which led to the high density defects at the fork grain boundary. Many dislocation lines are in copper matrix, shown as the arrows in Fig.3c. Meanwhile, recovery inevitably happens during hot extrusion process and a large number of small angle grain boundaries and dislocation concentration areas are formed in the alloy, as shown in Fig.3d.

TEM images of the samples obtained by heat-treatment at 600 ºC for 1.0 h are shown in Fig.4. As shown Fig.4a, 4d, a lot of dislocations and sub-grain boundaries in W-Cu sheet disappear. Compared with Fig.3a, 3b, it can be seen in Fig.4a, 4d that the dislocation density greatly decreases. By comparing Fig.3c with Fig.4c, it is also noted that the density of the dislocation line plummets. It can also be seen in Fig.4c that a lot of tungsten nano-particles precipitate. During heat treatment process, the lattice distortion gradually recovered and the dislocation glided or climbed because the pinning effect of small particle on the dislocation was not sufficient

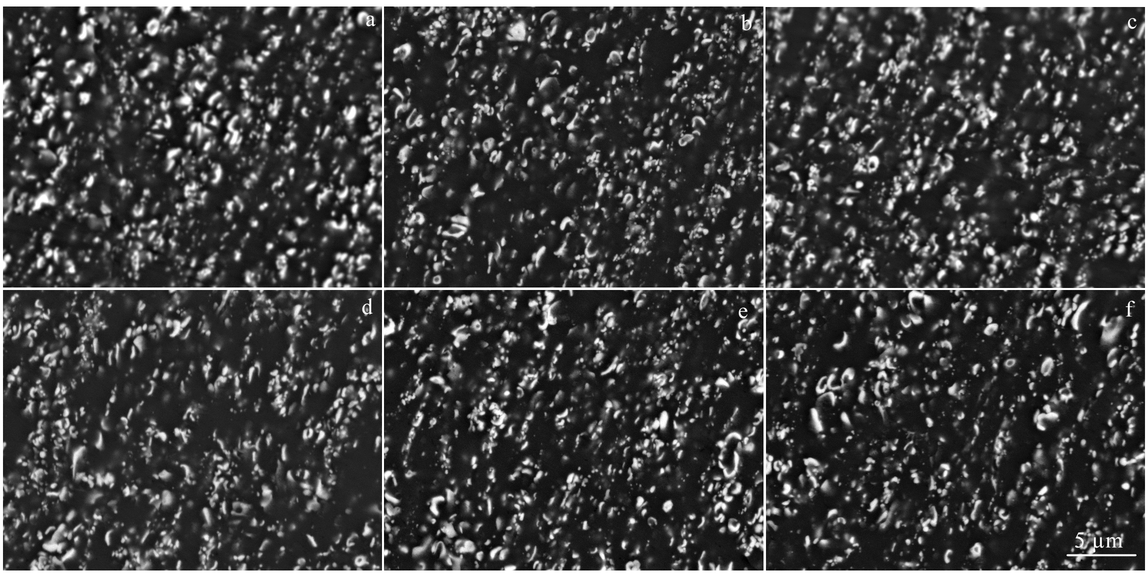

Fig.2 Back scattered electron images of W-80Cu sheets after heat-treatment for different time at 600 ºC: (a) un-heated, (b) 0.5 h, (c) 1.0 h, (d) 1.5 h, (e) 2.0 h, and (f) 4.0 h

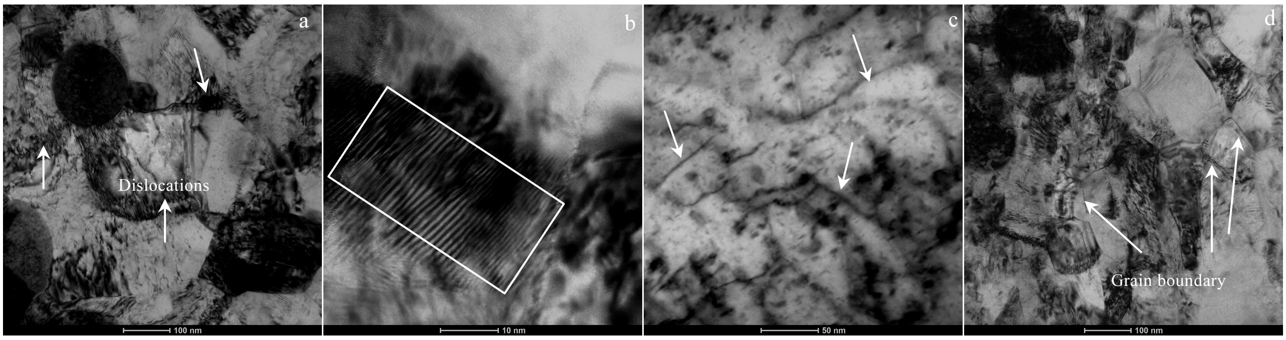

Fig.3 TEM images (a, c, d) and (b) HRTEM image of the hot-rolled sample

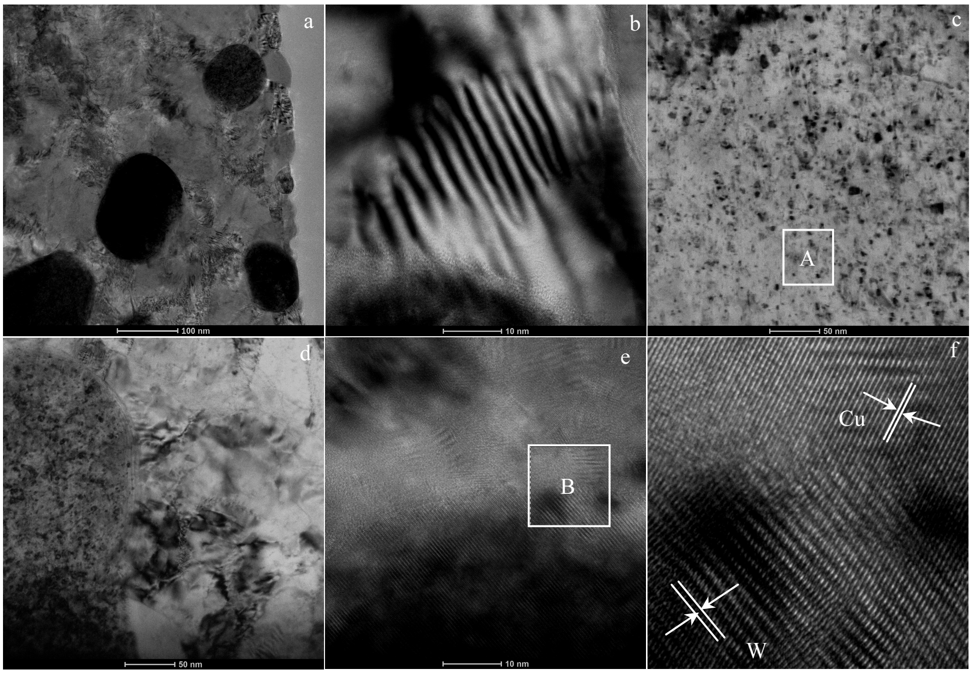

Fig.4 TEM and HRTEM images of the hot-rolled sample heat-treated at 600 ºC for 1.0 h: (a, c, d) TEM images; (b) HRTEM image; (e) HRTEM image of A zone in Fig.4c; (f) IFFT image of B zone in Fig.4e

to prevent dislocation movement. This caused that a lot of dislocations and sub-grain boundaries disappeared. An enlarged figure of A zone in Fig.4c is shown in Fig.4e. Fig.4f indicates IFFT image of B zone in Fig.4e. The interplanar spacings are 0.239 and 0.212 nm, which correspond with W (110) and Cu (110), respectively. The results in Fig.4f illustrate that tungsten nano-particles have a good interface between with copper matrix, which helps to enhance the strength and ductility of the material.

2.2 Electrical conductivity

2.2.1 Influence of heat-treatment temperature on the electrical conductivity

The influence of the heat treatment temperature on the electrical conductivity of W-80Cu sheet is indicated in Fig.5. The results reveal that the electrical conductivity rises first and then decreases with increasing the heat-treatment temperature. Because the electrical conductivity of copper is much higher than that of tungsten, the electrical conductivity is affected mainly by copper. What's more, the structural defects in metal materials (such as vacancies, dislocations, grain boundaries, pores and impurity atoms) decreased the number electrons as well as conduction paths for free electrons and enhanced the electrons scattering, which led to the decline of the electrical conductivity[27-29]. The recovery and recrystallization induced by the annealing also made the density of the machining dislocation decrease. The lattices became orderly and the scattered effect of the electrons was reduced, which enhanced the electrical conductivity. The high W-W contiguity affected the complicated current path in the structure of

W-Cu alloy[30]. Nano-copper melt and diffusion at 600 ºC destroyed W-W contiguity structure and made the microstructure of W-Cu alloy better (shown in Fig.1c), which increased the electrical conductivity. The maximum electrical conductivity is 89.4%IACS at 600 ºC. However, at higher temperature, the recovery and recrystallization of tungsten easily occurred and the new tungsten grains appeared. These resulted in decreasing the electrical conductivity.

2.2.2 Influence of heat-treatment time on the electrical conductivity

Fig.6 shows the influence of heat treatment time on the electrical conductivity of W-80Cu sheet at 600 ºC. The results indicate that the electrical conductivity increases first, and then decreased with prolonging the heat-treatment time. Furthermore, the electrical conductivity of the samples after heat treatment is higher than that of these unheated. When the heat-treatment time was 1.0 h, the maximum electrical conductivity was 89.4%IACS. But the electrical conductivity maintained the basic stability when the time continued to be prolonged.

2.3 Hardness

2.3.1 Influence of heat-treatment temperature on the hardness

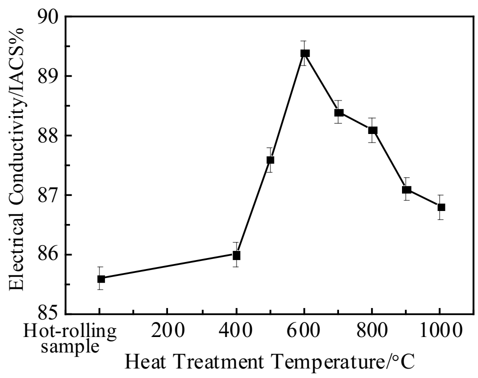

Fig.5 Effect of heat treatment temperature on the electrical conductivity

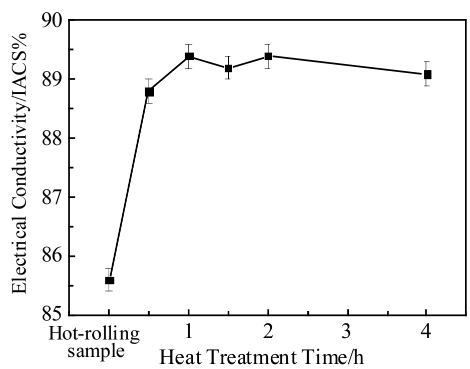

Fig.6 Effect of heat treatment time on the electrical conductivity

Fig.7 describes the influence of heat-treatment temperature on the micro-hardness of W-80Cu sheets. With increasing the heat-treatment temperature, the hardness decreased in totality, but the hardness had a slight rebound when the heat-treatment temperature was 600 ºC. This tendency is similar to the results of the Ref. [31]. The machining stress and the dislocation density in W-80Cu sheet after hot-rolling were high, which resulted in working hardening and the hardness increase. After heat-treatment, the residual stress in the alloy released and the dislocation density reduced greatly, which decreased the hardness. When the heat treatment temperature was 600 ºC, the diffusion of molten nano-copper made the micro-structure of W-80Cu sheet homogenous and dense. This resulted in an increase of the hardness. As the temperature was increased successively, the movable dislocation density and the diffusion rate of element increased, which enhanced dislocation movement and solution-diffusion[32]. Meanwhile, the grains grew and became coarse. The inhomogeneity microstructure occurred. These decreased the hardness.

2.3.2 Influence of heat treatment time on the hardness

Fig.8 depicts the influence of heat treatment time on the hardness of W-Cu alloy at 600 ºC. The results show that with extending the holding time, the hardness decreased in totality. But a slight rebound happened when the heat treatment time was 1.0 h. After heat treatment, the stress in the material released and the dislocation density decreased, which made the hardness decline. When the holding time was 1.0 h, copper recrystallization occurred and the grains grew up, as well as copper nano-particles melted and filled into micro-pore. The micro-structure was homogeneous and dense, which increased the hardness. Once the heat treatment time was too long, the growth of copper grain could destroy W-W bond and decrease the hardness.

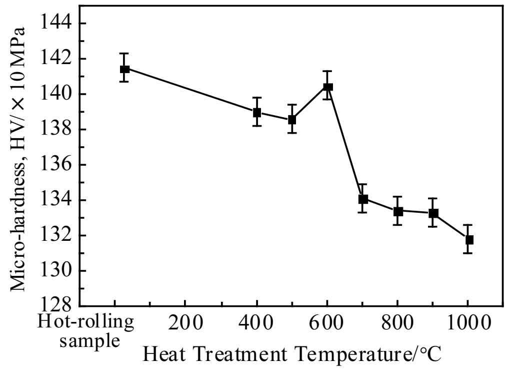

Fig.7 Effect of heat treatment temperature on the hardness

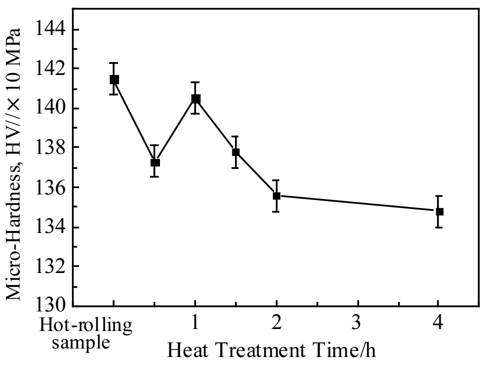

Fig.8 Effect of heat treatment time on the hardness

2.4 Tensile property

2.4.1 Influence of heat-treatment temperature on tensile property

Fig.9 shows that the room-temperature tensile strength and elongation of W-80Cu sheet vary with the heat-treatment temperature. The change of the tensile strength was in a decrease trend, but had a slight rebound at 600 ºC. This tendency is similar to that of the hardness. The elongation of the samples increased first, and then declined with increasing the heat treatment temperature. What's more, the elongation of the samples after heat treatment was higher than that of the un-heated one. Because the stress and dislocation density in the materials decreased with increasing the heat treatment temperature, the tensile strength declined while the elongation increased. The tensile strength rebounded at 600 ºC because the diffusion of molten nano-copper resulted in the dense and uniform structure. With continuing to increase the temperature, the high energy was provided for recovery, recrystallization and grain growth[33]. The individual coarse grain and the local inhomogeneity of the micro-structure reduced the strength and elongation of the material.

2.4.2 Influence of heat treatment time on tensile property

The room-temperature tensile strength and elongation of W-80Cu sheets change with the heat-treatment time, as shown in Fig.10. The results indicate that the tensile strength of the specimen after annealing is lower than that of the unheat-treated one while the elongation is higher than that of the unheat-treated one. It was explained by the fact that the stress released and the dislocation density reduced after heat treatment. The tensile strength was a minimum when the ageing time was 0.5 h because the diffusion of melt nano-copper has been incomplete in short time and the part internal stress was released. When the ageing time was 1.0 h, the tensile strength rebounded and the elongation was maximum. At this condition, the sufficient diffusion of copper phase reduced the number of micro-pores or copper-free zones in the sheets. This contributed to enhancement in the tensile strength and elongation. Still further extending the time, copper recrystallization and grain growth resulted in the inhomogeneous structure and the decline of the tensile strength and elongation.

2.4.3 Analysis fracture appearance

Fig.11 indicates the tensile fracture morphologies of W-80Cu sheets heat-treated at different temperatures for 1.0 h. The results show that copper matrix creates the continuous net while tungsten particles are distributed among copper net. The tensile fracture morphologies reveal that the dimple and intergranular fracture are both present in the tensile fracture. The partial dimples obviously form centering on tungsten particles. The fracture morphology has a lot of dimples and the tearing ridges in Fig.11. During fracture process, copper deformed plastically at first in order to reduce tensile stress, which decreased the resistance of tungsten particle slippage and retarded deformation. So, the ductile fracture of matrix copper occurred and formed the curved dimples. However, tungsten particles of the fracture surface could not coordinate the deformation because of the low plastic deformation capacity. Fracture surface of the unheat-treated samples is rough and the depth and size of

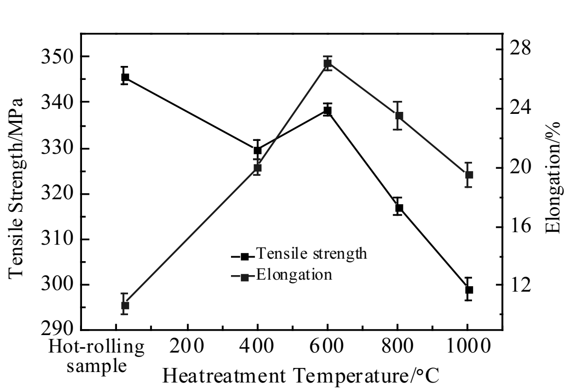

Fig.9 Tensile strength and elongation as a function of heat-treatment temperature for W-80Cu sheets

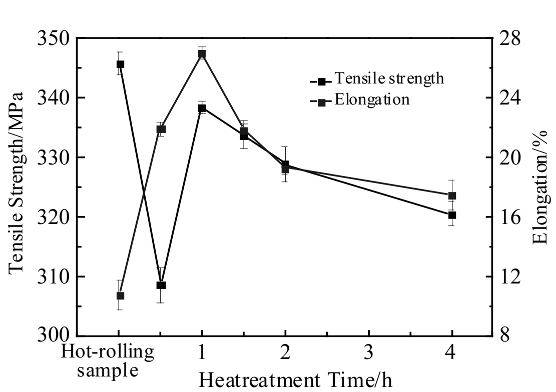

Fig.10 Tensile strength and elongation as a function of heat-treatment time for W-80Cu sheets

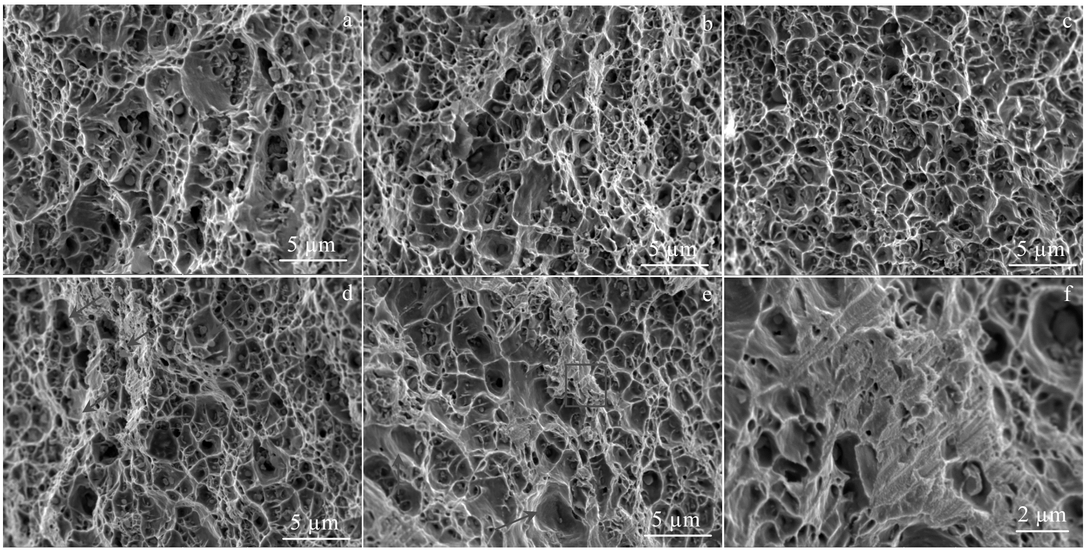

Fig.11 Fracture morphologies of W-80Cu sheets heat-treated at different temperatures: (a) unheated, (b) 400 ºC, (c) 600 ºC, (d) 800 ºC, (e) 1000 ºC, and (f) partial enlargement of Fig.11e

the dimples are different, as shown in Fig.11a. With increasing the temperature, the dimples deepened while its size and distribution became uniform. Tensile fracture morphology of W-80Cu sheet heat-treated at 600 ºC (as shown in Fig.11c) was characterized by ductile rupture and the deformation of the Cu phase was more prominent. A large number of fine and deep dimples were evenly distributed. This indicated that the strength and ductility of W-80Cu sheet was high. When continuing to increase the annealing temperature, the size and depth of the dimples on tensile fracture was different, and the local tearing ridges of copper became longer (as shown as the diamond arrows in Fig.11), which confirmed the copper grain growth. Meanwhile, the steps (as described by the arrows in Fig.11c) and quasi-cleavage (shown in Fig.11f) occurred locally. This means that the ductility decreased, which was consistent with the results in Fig.9.

Fig.12 shows the tensile fracture appearances of W-80Cu sheets heat-treated at 600 ºC for the different holding time. With extending the heat-treatment time, the dimples deepened

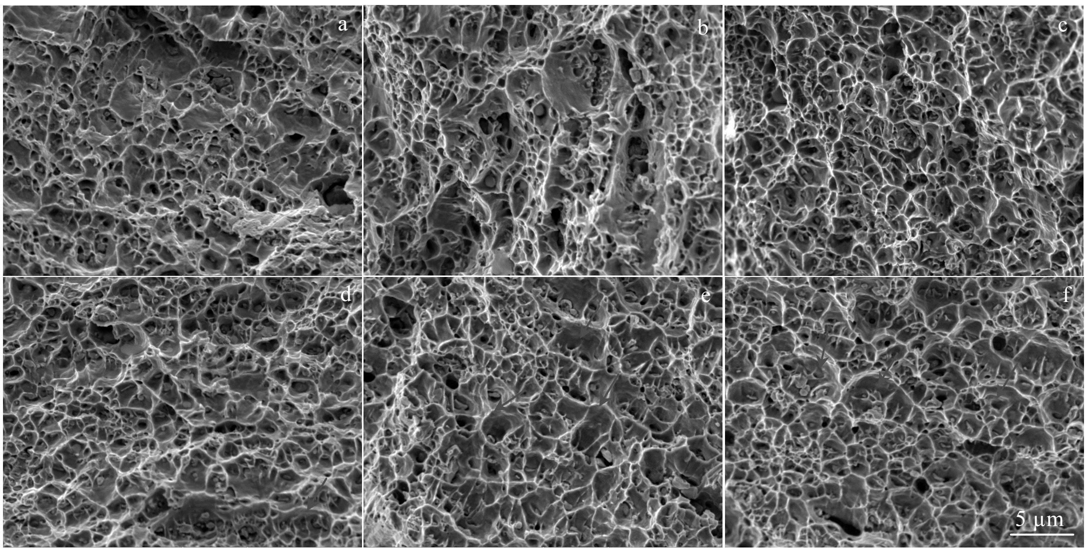

Fig.12 Fracture appearance of W-80Cu sheets heat-treated for different time at 600 ºC: (a) unheated, (b) 0.5 h, (c) 1.0 h, (d) 1.5 h, (e) 2.0 h, and (f) 4.0 h

while its size and distribution became uniform. A large number of fine and deep dimples appeared in the tensile fracture morphology of W-20Cu sheet heat-treated at 600 ºC (as shown in Fig.12c). This indicated that the strength and ductility of W-80Cu sheet were high. When continuing to prolong the heat-treatment time, the local dimples became big and the tearing ridges of copper got longer (as shown by the arrows in Fig.12d, 12e), which confirmed the copper grain growth. Meanwhile, the dimple size became different. These also indicated that the ductility and strength decreased, which chimed with the result in Fig.10.

3 Conclusions

1) The influence of heat treatment on tungsten phase in W-80Cu sheet was not significant. A lot of dislocations around tungsten particles and sub-grain boundary decreased in amount.

2) After heat treatment, the electrical conductivity and elongation of W-80Cu sheets were greater than those of the un-heated one while their hardness and tensile strength were lower.

3) After heat treatment at 600 ºC for 1.0 h, the electrical conductivity and elongation of W-80Cu sheet reached the maximum. A large number of fine and deep dimples distributed evenly on tensile fracture.

4) The fracture mode of W-80Cu sheets was the dimple and intergranular fracture. When the heat-treatment temperature was around and above 800 ºC and the holding time exceeded 1.0 h, the tearing ridges of copper on tensile fracture became longer due to the growth of copper grains.

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热处理过程中 W-80Cu 板材组织及性能演变

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摘 要：研究了热处理对 W-80Cu 板材组织演变和性能的影响。采用扫描电镜(SEM)和透射电镜(TEM)观察了 W-80Cu 板材的微观结构和断口形貌，并对其电导率和力学性能进行了研究。结果表明，热处理后试样的电导率和延伸率均高于未热处理试样，而硬度和抗拉强度均低于未热处理的试样。在 600 ℃下热处理 1.0 h，W-80Cu 板材的电导率和延伸率最大，韧窝深而密，且分布均匀。W-80Cu 板材的室温拉伸断裂方式主要是沿晶断裂和韧窝断裂的混合断裂。当热处理温度在 800 ℃左右及以上，断裂韧窝变得深浅不一，大小也不均匀，局部出现断裂台阶和准解理断裂。当时间超过 1.0 h 时，出现局部韧窝变大，铜的撕裂脊变长，这与铜晶粒的再结晶长大有关。在热处理过程中 W-80Cu 板材中的钨相没有明显变化，但试样中钨颗粒周围大量位错及材料中的晶界明显减少。纳米钨颗粒与铜基体之间存在良好的界面关系，这有利于材料强度的提高。

关键词：热处理；微观结构；电导率；力学性能

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