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High strength and high electrical conductivity CuMg alloy prepared by cryorolling

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Abstract: The microstructure, mechanical properties and electrical conductivity of the room-temperature and cryogenically rolled Cu−0.2wt.%Mg alloy were investigated by transmission electron microscopy (TEM), electron backscattered diffraction (EBSD), hardness measurement, tensile tests and electrical conductivity measurement. The results show that for the cryorolled sample, the grain size is decreased by 41% compared with the sample processed at room temperature. With increasing thickness reduction, the microhardness of the alloy continuously increases and the electrical conductivity decreases. For the sample with 90% thickness reduction rolled at cryogenic temperature, the tensile strength and the electrical conductivity are 726 MPa and 74.5% IACS, respectively. The improved tensile strength can be mainly attributed to the grain boundaries strengthening and dislocation strengthening.

Key words: CuMg alloy; cryorolling; mechanical properties; grain size; twin

1 Introduction

In recent years, Cu-based alloys have attracted much attention because of heavy demand for high mechanical strength and high electrical conductivity from industry fields [1−5]. For example, contact wire of high-speed trains requires that Cu-based alloys should have a tensile strength higher than 500 MPa and an electrical conductivity larger than 60% International Annealed Copper Standard (IACS) [4]. In this aspect, CuMg alloys have been successfully used in the high-speed railway of China which have a tensile strength of 522 MPa and an electrical conductivity of 68.6% IACS [4].

Previous report showed that the addition of Mg into Cu remarkably increased the tensile strength due to solid−solution strengthening without greatly sacrificing the electrical conductivity [6]. Several methods have been proposed to further improve the mechanical properties of CuMg alloys, including the equal channel angular pressing (ECAP) combined with the cold working [4,7,8] and addition of the third element [8,9]. ZHU et al [4] investigated the mechanical properties and the electrical conductivity of two CuMg alloys processed by four ECAP passes followed by cold working and annealing, and reported a maximum ultimate tensile strength (UTS) of 627.4 MPa and an electrical conductivity of 72.4% IACS in Cu−0.4wt.%Mg alloy. RODRÍGUEZ-CALVILLO et al [7] analyzed the strengthening mechanism of two CuMg alloys processed by ECAP and concluded that the subgrain structure is the major contribution to the strengthening. The addition of the third element may influence the properties of CuMg alloys through the following ways: reacting with some impurity elements or refining microstructure, as demonstrated in CuMgCe alloys [8] and CuMgCa alloys [9]. It has been reported that Cu−0.29wt.%Mg−0.21wt.%Ca alloy showed excellent performance with an UTS of 545 MPa and an electrical conductivity of 71.79% IACS [9].

During fabrication of Cu-based alloy contact wire, cold rolling is an essential processing. As compared to rolling at room temperature, rolling at cryogenic

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temperature (cryorolling) is a more effective method to prepare ultrafine grained (UFG) Cu-based alloys. This has been confirmed by the reported results in CuZn alloys [10], CuAl alloys [11] and CuCrZr alloys [12] etc. Deformation at cryogenic temperature can suppress dislocation activity and favor the presence of deformation twins. Furthermore, the addition of Mg into Cu significantly reduces stacking fault energy [7], which also benefits the introduction of deformation twins. However, little information is available in literatures on the microstructure, mechanical properties and electrical conductivity of cryorolled CuMg alloy.

In the present work, the microstructure, mechanical properties and electrical conductivity of cryorolled Cu−0.2wt.%Mg alloy were investigated. For comparison purpose, this alloy was also processed at room temperature. The effects of cryorolling on mechanical strength and electrical conductivity were discussed based on microstructural evolution.

2 Experimental

A commercial Cu−0.2wt.%Mg alloy was provided by China Railway Construction Electrification Bureau Group. The as-received alloy was in cold-rolled state. Before processing, the as-received alloy was annealed at 300 °C for 30 min, followed by air cooling. Hereafter, this state was taken as the initial one. Samples were rolled at room temperature and cryogenic temperature without intermediate annealing, respectively. For cryorolling, the samples were dipped into liquid nitrogen for 15 min to achieve the saturation temperature before the first pass. The total thickness reduction (90%) was obtained in multi-pass with a reduction per pass less than 10%. After each pass, the samples were immediately dipped into liquid nitrogen for 5 min before next pass. For rolling at room temperature, the samples were also processed in the same way with cryorolling samples. For simplicity, the processing at room temperature and cryogenic temperature for 90% reduction were expressed by RTR and CR, respectively.

Microhardness was measured by using a HVS−1000 type tester under a load of 100 g for 10 s. Tensile samples were cut along the rolling direction. Tensile properties were tested using the samples with dimensions of 1 mm × 1 mm in cross section and 12.5 mm in gauge length. All the tensile tests were carried out on an Instron−3365 universal tensile machine with an initial rate of 1.5%/min at room temperature. Electrical conductivity was measured by using a FIRST FD−102 type eddy current conductivity meter. At least, nine measurements were taken and the average values were calculated. X-ray diffraction (XRD) test was carried out on a Rigaku/TTR-III diffractometer with Cu K_α radiation by step-scanning in the 2θ range of 30°−95°. The voltage and current were 40 kV and 150 mA, respectively. The silicon standard sample was used to calibrate the diffractometer. Microstructure was carefully observed by electron backscattered diffraction (EBSD) and transmission electron microscopy (TEM). EBSD was carried out on an FEI Nova Nano scanning electron microscope (SEM). The scanning step was 0.15 μm. TEM observation was performed by using a JEOL 2100 transmission electron microscope operated at 200 kV. The thin foils were prepared by mechanical grinding, followed by ion-milling.

3 Results and discussion

3.1 Microstructure

Figure 1 shows the EBSD inverse pole figure map of initial sample. The initial microstructure is characterized by the nearly equiaxed grains with a size of about 2.8 μm. After rolling at different temperatures, the deformed microstructure was investigated by TEM observation. Figure 2 shows the bright field TEM images of transverse plane of RTR sample from different regions with different magnifications. The severely elongated grains along the rolling direction with low aspect ratio are observed. The average width of lamellae is about 96 nm. In some regions, high-density dislocations in tangles and cells and a small number of deformation twins are observed, as shown in Fig. 2(b). The average twin spacing is about 40 nm. The above results are consistent with the reported microstructure evolution of pure Cu which is dominated by the dislocation movement [13].

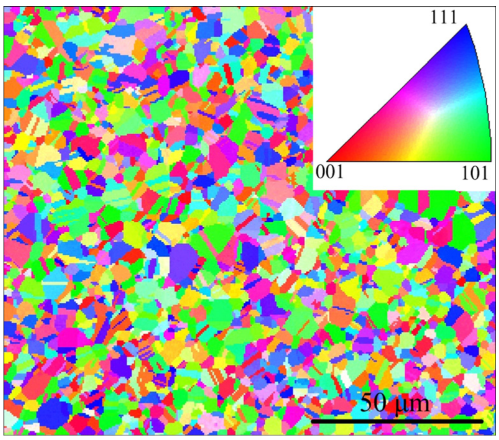

Fig. 1 EBSD inverse pole figure map of initial CuMg alloy

Figures 3(a) and (b) show the microstructures of transverse plane of CR sample from different regions with different magnifications. As compared to the RTR sample, the cryorolled sample shows finer grains and the width of lamellae is estimated to be 57 nm. The grain boundaries are curved, which are different from the

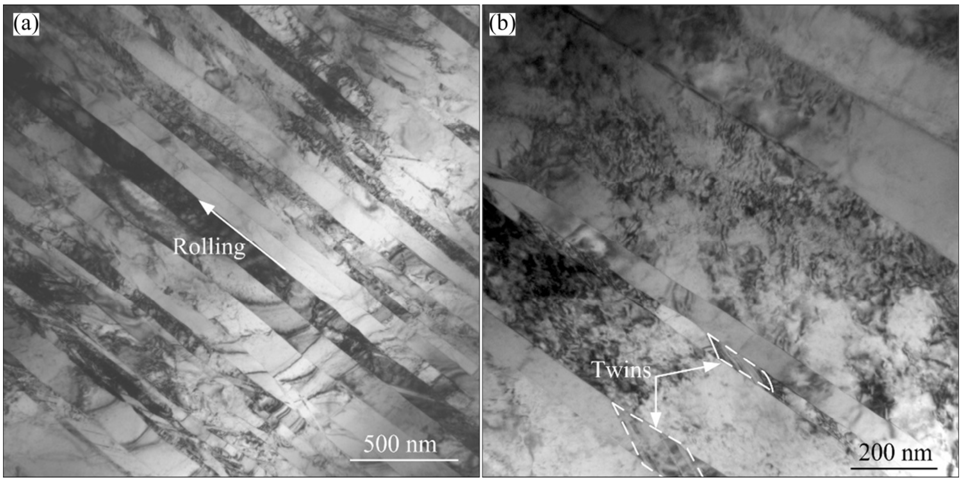

Fig. 2 Bright field TEM images of CuMg alloy rolled at room temperature for 90% thickness reduction: (a) Lower magnification; (b) Higher magnification

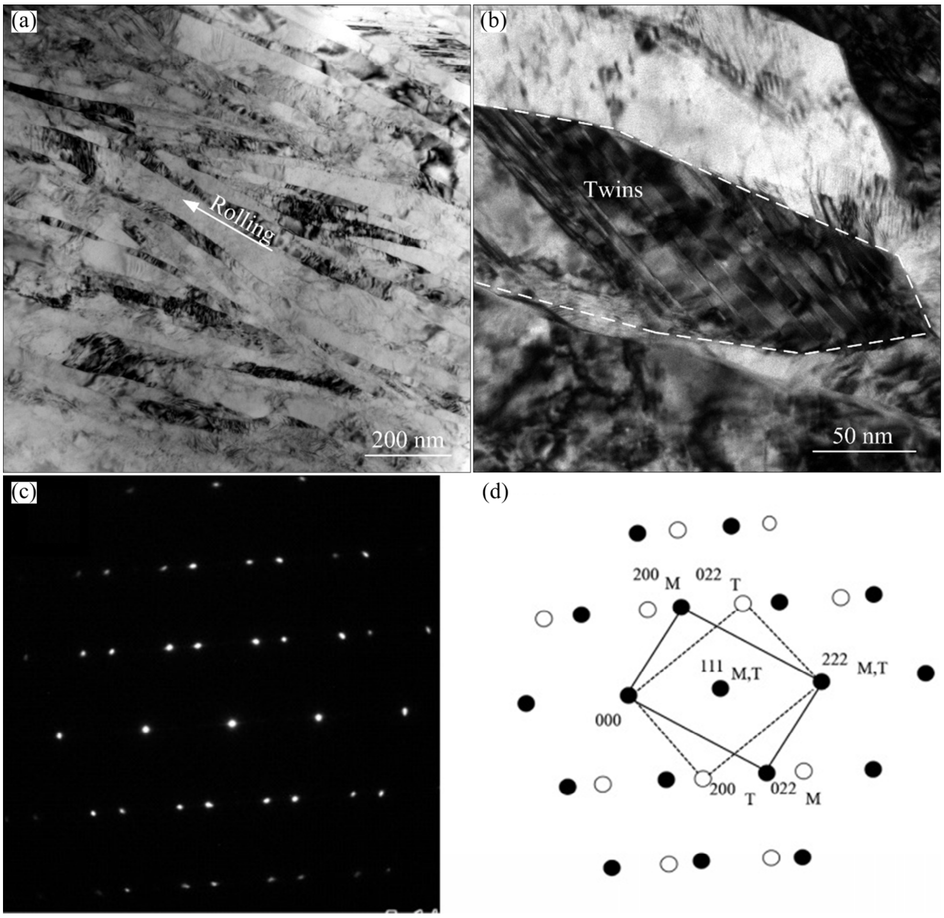

Fig. 3 Bright field TEM images of CuMg alloy cryorolled for 90% thickness reduction at lower (a) and higher (b) magnifications, SAED pattern (c) and corresponding index results (d)

straight grain boundaries of RTR sample. High-density dislocations and some deformation twins are also visible in some regions, as shown in Fig. 3(b). The average twin spacing (19 nm) is smaller than that of RTR sample, which is ascribed to the decreased stack fault energy as a result of the decreased rolling temperature [14,15]. Figures 3(c) and (d) show the selected area electron diffraction (SAED) pattern of the twin in Fig. 3(b) and the corresponding index results, respectively. The SAED pattern shows the typical twin relationship of {111}/[112] type in Cu-based alloy. It is seen that the twin boundary is curved. This may suggest that these twins form during the rolling process and are deformation twins. It should be mentioned that the twin shown in Fig. 2(b) has the same twin relationship with that in Fig. 3(b). The comparison between Fig. 2 and Fig. 3 shows that cryorolling favors obtaining finer microstructure in CuMg alloy. This can be ascribed to the fact that the decrease in rolling temperature suppresses the dynamic recovery by reducing stacking fault energy [11,16,17].

3.2 Mechanical properties

Figure 4 shows the dependence of microhardness of CuMg alloys on thickness reduction of differently rolled samples. It is seen that the average microhardness of samples gradually increases with increasing thickness reduction, irrespective of deformation temperature. The cryorolled samples show the overall larger micro-hardness than those processed at room temperature. Figure 5 shows the stress−strain curves of initial and differently rolled samples. The initial sample shows obvious strain hardening, low UTS and high uniform elongation (37%). The rolled samples exhibit little strain hardening. After 90% rolling reduction in thickness, the UTS and yield strength of CR sample can reach up to 726 MPa and 640 MPa, respectively, which are much higher than those of initial sample (303 MPa, 119 MPa) and RTR sample (562 MPa, 516 MPa). To the best of our knowledge, the strength of CR sample is the highest value obtained in Cu−0.2wt.%Mg alloy. ZHU et al [4] once reported that the UTS of Cu−0.2wt.%Mg alloy increased from 268.9 to 574.8 MPa after being processed by ECAP plus cold working. The uniform elongation of CR sample (1.5%) is also slightly larger than that of RTR sample (1.2%). As compared to ECAP plus cold working, the present CR processing is more effective in improving the strength of Cu−0.2wt.%Mg alloy. The width of lamellae of the alloy processed by ECAP plus cold working is about 100 nm [4], which is much larger than that of the present work (57 nm). This is possibly responsible for the difference in mechanical strength.

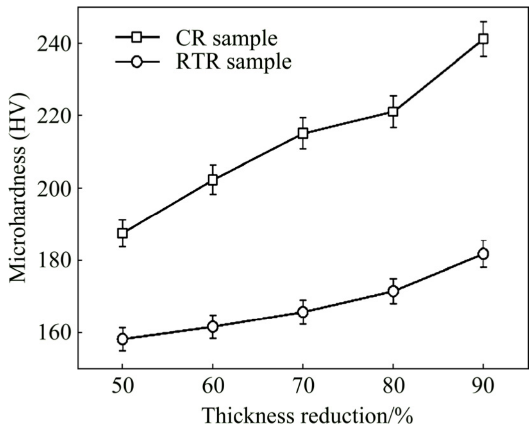

Fig. 4 Microhardness of differently processed CuMg alloys as function of thickness reduction

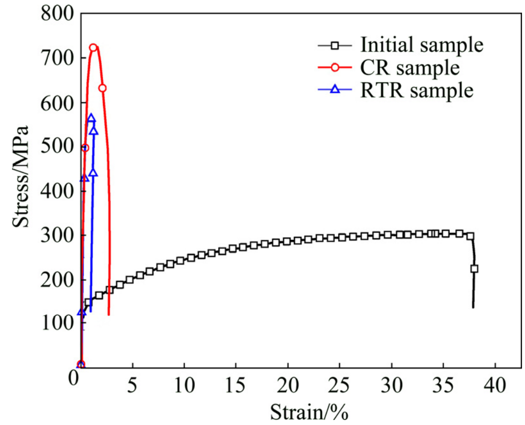

Fig. 5 Typical tensile stress−strain curves of initial sample and samples processed at different temperatures

3.3 Electrical conductivity

Figure 6 shows the effect of thickness reduction on electrical conductivity of differently treated CuMg alloys. With the increase of thickness reduction, the electrical conductivity of all samples decreases, irrespective of deformation temperature. This can be rationalized by the increased grain/twin boundaries and dislocations introduced during rolling with the increase of thickness reduction. When the thickness reduction is 90%, the electrical conductivities are reduced from 85% IACS (initial sample) to 79% IACS and 74.5% IACS for RTR and CR samples, respectively, which still meet the requirement for contact wire of high-speed railway [14]. Compared with the samples processed at room temperature, the cryorolled samples exhibit larger number of grain/twin boundaries and high-density dislocations which are responsible for the smaller electrical conductivity because of the enhanced scattering of electrons [5].

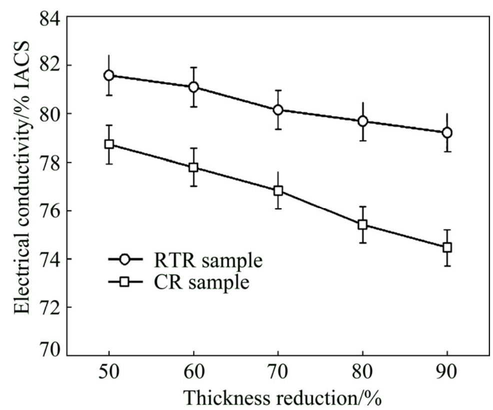

Fig. 6 Effect of thickness reduction on electrical conductivity of differently processed CuMg alloys

3.4 Discussion

The above results indicate that cryorolling is more effective in improving mechanical strength than room temperature rolling for CuMg alloys. Based on the microstructural results, several strengthening mechanisms are suggested to be involved in the present case, i.e. grain boundary strengthening (σ_{GB}), twin boundary strengthening (σ_{TB}) and dislocation strengthening (σ_d). Generally, the yield strength (σ_{YS}) can be calculated by the following equation [10]:

$$\sigma_{YS}=\sigma_0+\sigma_{GB}+\sigma_{TB}+\sigma_d=\sigma_0+k(1/d+1/\lambda)^{1/2}+\alpha Gb\rho^{1/2} \quad (1)$$

where σ_0, k, d, λ, α, G, b and ρ are the frictional stress, a constant, grain size, twin thickness, Taylor factor, shear modulus, Burgers vector value and dislocation density, respectively. σ_0 is 25 MPa for pure copper [18,19]. Following the reported work [20], cryorolling increases the volume fraction of deformation twin and decreases the twin thickness, which is also confirmed by the results shown in Figs. 2 and 3. According to Eq. (1), the contribution of twin to yield strength in CR sample should be larger than that in RTR sample. However, in the present work, the volume fraction of twin is low and its distribution is not homogeneous, thus the contribution of twin to yield strength is not determined. Therefore, the strengthening contribution from grain boundary is considered. K_{GB} is 0.18 MPa/m$^{1/2}$ for Cu alloy [9]. In the case of the lamellar structure, the effective grain size was calculated as twice the width of the lamellae [21,22]. Therefore, the strengthening contributions from grain boundary were calculated to be 533 and 411 MPa for CR and RTR samples, respectively.

Regarding the strengthening contribution to yield strength from dislocation, constant α is taken as 0.24 [7,23,24], shear modulus G is 45.6 GPa [7] and Burger vector value is equal to $\sqrt{2}\,a/2$ (a is lattice constant) for Cu alloys and is usually taken as 0.26 nm [10]. The dislocation density can be calculated by using the following equation [17]:

$$\rho=\frac{16.1\varepsilon^2}{b^2} \quad (2)$$

where ε is microstrain which can be obtained from XRD results. Figure 7 shows the XRD results of differently treated samples. By means of XRD peak broadening analysis of the {220} reflection, the dislocation densities of CR and RTR samples were calculated to be 8.8×10^{14}/m^2 and 6.3×10^{14}/m^2. This agrees well with other report in which the dislocation density of cryorolled CuZnSi sample is also larger than that of the room temperature rolled sample because of the suppression of dynamic recovery [23]. Then, the strengthening contributions to yield strength from dislocation were determined to be 84.3 MPa and 71.6 MPa for CR and RTR samples, respectively.

Table 1 summarizes the experimental and calculated yield strengths of CR and RTR samples. It is seen that grain size may take a dominant role in determining the mechanical strength of CuMg alloy. The calculated value is close to the experimental one. It should be emphasized that the difference between the calculation of idea case and the measurement of practical alloy and the error of measurement and calculation are unavoidable [25]. For example, the calculated σ_{GB} value may be overestimated since the effective grain size is not accurately enough.

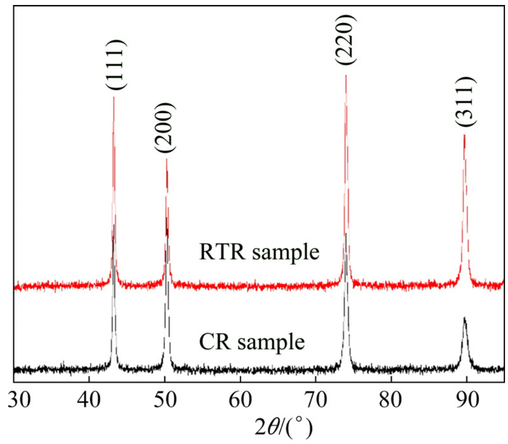

Fig. 7 XRD patterns of differently processed CuMg alloys

Table 1 Calculated yield strengths from different strengthening mechanisms and experimental values

Sample	Measured σ_{YS}/MPa	Calculated strength/MPa		
		σ_{GB}	σ_d	σ_{YS}
CR	640	533	84.3	642.3
RTR	516	411	71.6	507.6

4 Conclusions

(1) Cryorolling is more effective in refining the microstructure of CuMg alloy than rolling at room temperature. The microstructure of the rolled samples is characterized by lamellar structure. For CR and RTR samples, the widths of lamellae are about 57 and 96 nm, respectively.

(2) After the 90% thickness reduction at cryogenic temperature, the UTS and the electrical conductivity of CuMg alloy are 726 MPa and 74.5% IACS, respectively. The improved strength can be mainly attributed to the grain boundary strengthening and dislocation strengthening.

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深冷轧制制备高强、高电导率 CuMg 合金

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摘 要：利用透射电子显微镜观察(TEM)、电子背散射衍射(EBSD)技术、硬度测试、拉伸测试与电导率测试研究室温轧制与深冷轧制 Cu−0.2wt.%Mg 合金的显微组织、力学性能与电导率。结果表明，与室温轧制样品相比较，深冷轧制样品的晶粒尺寸减小了 41%。随轧制变形量增加，合金的显微硬度持续增加而电导率下降。对于深冷轧制样品，当厚度减小 90%时，其抗拉强度和电导率分别达到 726 MPa 和 74.5% IACS。抗拉强度的提高主要归因于晶界强化与位错强化。

关键词：CuMg 合金；深冷轧制；力学行为；晶粒尺寸；孪晶

(Edited by Wei-ping CHEN)