Microstructure and mechanical properties of pure copper subjected to skin pass asymmetric rolling

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Abstract. Pure copper after 300°C annealing has been processed by skin pass asymmetric rolling up to small and medium total reductions in this work to study the evolution of microstructure, mechanical properties and texture. Subsequent EBSD characterization shows that after 300 °C annealing, the grains equiaxed but the texture is still typical rolling texture. At small rolling reductions (5.0% and 11.8%), the microstructure did not change much, the stress increased, and the ductility were still high (>20%), and the texture remained the same. At higher rolling reduction (32.0%), the grains were refined, the stress increased but the ductility decreased at the same time, and the texture remained the same. At medium rolling reduction (63.6%), the grains were further refined, the stress further increased, and the ductility further decreased, and the texture changed from typical rolling texture to shear texture at the edge and rolling texture at the centre.

1 Introduction

Ultrafine grained (UFG) or nanograined (NG) materials have been paid much attention in recent decades for their extremely high strength and high corrosive resistance. Severe plastic deformation is the most applausive method to produce bulk UFG materials, including accumulative roll bonding (ARB) [1] and equal channel angular pressing (ECAP) [2] and so on. The newly introduced asymmetric rolling [3] has also been widely used for manufacture bulk UFG or even NG materials [4,5].

In asymmetric rolling, the working pieces are rolled between rolls of different diameters or same diameter rolls rotating at different velocities. Asymmetric rolling is considered an innovative process for the strain state imposed on the sheet is an integration of plane strain and shear though it can be achieved in the conventional rolling system [6]. Several studies [7-10] have shown that asymmetric rolling can generate grain rotation and grain subdivision which result in grain refinement and texture transformation and improve the properties of the materials. Lee's work [7] indicated that strong <111> //ND orientation induced by the shear deformation during asymmetric rolling might improve the deep drawability and the refined grains could improve the mechanical property. In asymmetric

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rolling, rolling reduction per pass also plays a significant role in the final properties. Ma [9] studied the influence of thickness reduction per pass (TRPPs) in the range of 15-75% on the properties of 7075 aluminium alloy and found that smaller TRPPs led to fine sub-grains generation with an average size less than 0.5 μm which improved the mechanical properties.

Most studies to date focused on the properties and structures after asymmetric rolling up to medium and large reduction, or large rolling reduction per pass. In this regard, this work applied a so-called skin pass rolling method and combined with asymmetric rolling to obtain refined grains and rather high ductility. Subsequent study concentrates on the microstructure and mechanical properties of pure copper after skin pass asymmetric rolling up to small to medium reduction.

2 Experiments

The materials used in this work were pure copper with a purity of 99.95%. The as received copper was in sheet form with a thickness of 2.97 mm. Prior to rolling, the copper sheet was heat treated at 300 °C for two hours, and then air cooled to room temperature. After heat treatment, the working pieces were subjected to skin pass rolling using small rolling reduction per pass for 10, 20, 50 and 100 rolling passes, which gave total reductions of 5.0%, 11.8%, 32.0% and 63.6%, respectively. The reduction per pass is nearly 0.6%. A multi-function rolling mill with the roll diameter of 120 mm was adopted. The rolls were independently driven by two motors. The rolling speed ratio was kept at 1:1.4 during the skin pass asymmetric rolling.

Dog-bone shaped tensile samples were prepared from the skin pass rolled sheets according to ASTM E8 and the gauge length of the specimen was 25mm. The tensile tests were carried out on an Instron tensile machine at an initial strain rate of 5×10-4s-1. To ensure the accuracy, digital image correlation (DIC) was used to measure the strain of the samples.

Electron backscatter diffraction (EBSD) technique was adopted to characterize the microstructures and textures of each skin pass rolled sheet. The specimens for EBSD were Electron polished using Struers LectroPol-5 after grinding and mechanical polishing. An electrolytes of 500 ml distilled water with 250 ml phosphoric acid (85%) and 250 ml ethanol (85%) were used. Electron polishing was conducted at 22 V for 7-10 seconds with a flow rate of 13 at 25 °C. EBSD was carried out using JSM 7001F (JEOL) field emission scanning electron microscope (FESEM) on the RD-ND plane at both the edge and the centre of the sheets.

3 Results and discussions

Figure 1 shows the engineering stress-strain curves of the 300 °C annealed and skin pass rolled samples. As it is shown in the figure that the yield stress (YS) $\sigma_{0.2}$ and ultimate tensile strength (UTS) σ_{UTS} of the annealed sample is 184 MPa (using the 0.2% offset method) and 248 MPa, respectively. The uniform elongation (UE) ε_u of the anneal sample is 30.1%. After 10 passes asymmetric rolling, $\sigma_{0.2}$ increases by 36% to 250 MPa compared to the annealed state. As the rolling passes continue to increase, the increment rate of strength gradually decreases. After 20 passes rolling, $\sigma_{0.2}$ increases by 50% from the annealed state while the total reduction reaches 11.8%. The maximum strength σ_{UTS} reaches 312 MPa after 100 passes rolling. Although the total reduction from 50 passes to 100 passes increases by 100%, the strength does not increase dramatically. The detailed statistics of the tensile tests results are listed in Table 1.

The shape of the stress-strain curves also changes with the number of rolling passes. The annealed sample has a long work hardening stage and large uniform elongation. The work hardening stage drastically decreases with rolling passes which results in dramatic decrement of uniform elongation. The samples up to 20 passes maintain a rather stable deformation stage which keeps the total elongation larger than 25%. At larger reduction, such as the specimens of 50 and 100 passes, the stress-strain curves exhibit typical characteristics of high strength with lower ductility materials as it is referred in Ref [4,11]. When the total reduction reaches 63.6%, the strength does not increase much as compared to that of the 11.8% reduction. The uniform elongation, on the other hand, dramatically drops from 13.6% to 1.4%.

Compared to the 35% reduction specimen in Ref. [12], in which the yield stress is near 350 MPa while uniform elongation goes to peak immediately after yield, the 50 passes (32% reduction) sample in this study shows a similar strength with the 35% reduction specimen but have better ductility.

Figure 2 presents the EBSD images of pure copper at edge and centre after asymmetric rolling up to various strains. The upper images (Figure 2 a to e) shows the orientation maps at the edge while lower images (Figure 2 f to j) are from the centre of the sheets. According to Figure 2, the microstructure of the annealed, 10 and 20 passes rolled samples exhibits equiaxed grains at both edge and centre, but the grains gradually become smaller with increasing rolling deformation. After 50 passes, the grains start to elongate along the rolling direction. After 100 passes, the grains become pancake shaped at both edge and centre and the grain orientation also changes dramatically: from 85μm at 300 °C anneal sample and 48μm at 10 passes to about 4μm at 100 passes.

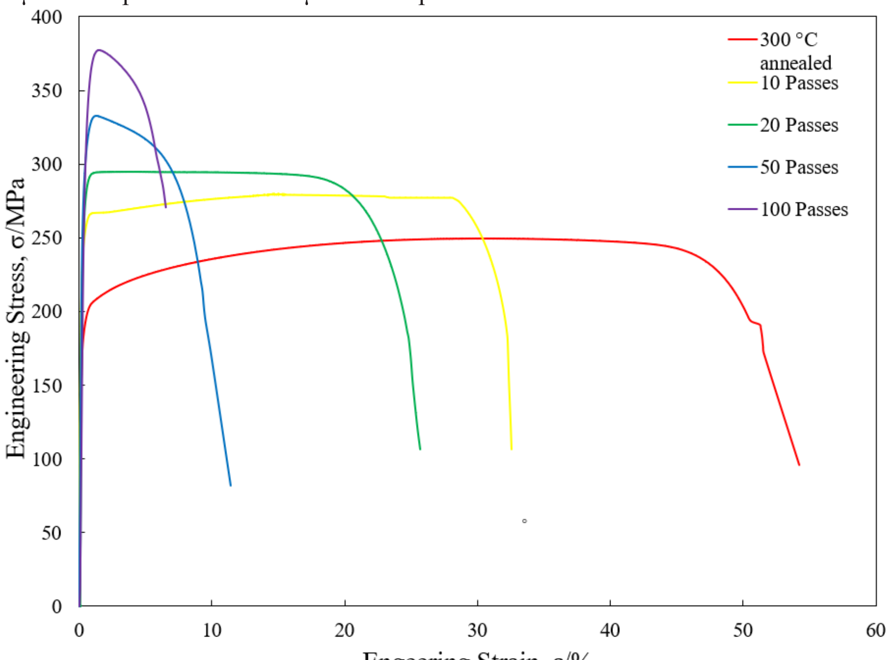

Fig. 1. The engineering stress-strain curves of annealed and skin pass rolled samples.

Table 1. The tensile properties of annealed and skin pass rolled pure copper samples.

Specimens	Total Reduction/ %	$\sigma_{0.2}$/MPa	ε_u/%	σ_{UTS}/MPa
300°C annealed	0	184	30%	248
10 passes rolled	5.0	250	13.6%	279
20 passes rolled	11.8	276	4.3%	294
50 passes rolled	32.0	303	1.2%	333
100 passes rolled	63.6	312	1.4%	375

Combining with the inverse pole figure, it can be seen that the initial orientation of the sample was mainly dominated by [111] direction. With the development of deformation, the grain orientation turns to [101] direction which is the result of grain rotation during the deformation.

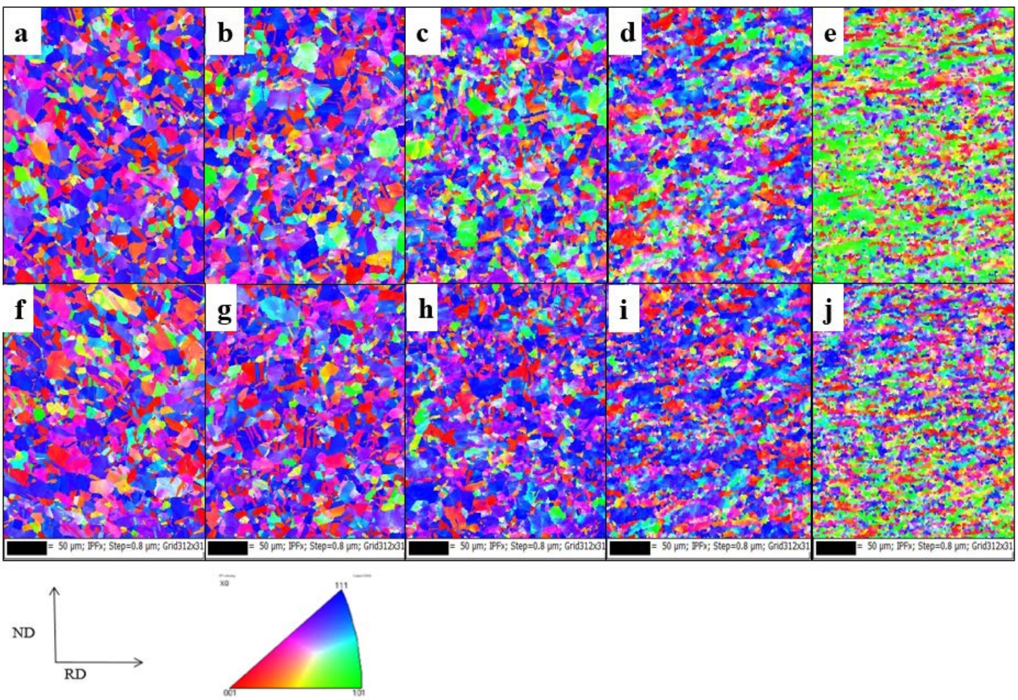

Fig. 2. The orientation maps at edge and centre of the examined specimens, a-e shows the edge and f-j is the centre (a, f: 300 °C annealed, b, g: 10 passes, c, h: 20 passes, d, i: 50 passes, e, j: 100 passes).

Figure 3 gives the distribution of misorientation angle at each rolling pass. As is shown in the figure, the misorientation angle in the 300 °C annealed sample presents two peaks at near 2° and 60° which means there exist large amount of both low and high angle grain boundaries. When rolled to 10 and 20 passes, the low angle grain boundaries begin to increase dramatically, and the peak around 2° comes to the maximum value at pass 20. After 50 passes, both peaks decrease and further decrease after 100 passes. The volume fraction of low angle grain boundaries (LAGBs), high angle grain boundaries (HAGBs) and twinning boundaries (TBs) of the annealed and asymmetrically rolled pure copper sheets is depicted in Figure 4. It can be seen from Figure 4 that both the edge and centre have the same trend. Before 20 passes, the volume fraction of LAGBs increases at both edge and centre. From 50 passes on, it begins to decrease. The trend of HAGBs is exactly the opposite to LAGBs: before 20 passes, the volume fraction decreases with the number of passes, then increases after 50 passes. The fraction of TBs is gradually decreasing before 50 passes but raises slightly at 100 passes.

Compared with Figure 3 and Figure 4, the rise of LAGBs indicates an increment of subgrain boundaries at the early stage of rolling as the result of dislocation kinking and tangling. With the development of rolling passes the newly formed subgrain boundaries evolve towards HAGBs which was well interpreted in Ref. [13]. Ma [11] has proposed that using lower TRPPs can help to store higher energy which increases the potential of recrystallization and the discontinuous recrystallization process (DDRX) will increase volume fraction of HAGBs. The phenomenon depicted in Figure 4 of both edge and centre can confirm that the rolling deformation consists of some DDRX process.

Figure 6 depicts further the evolution and transformation of texture via three major fibres at both edge and centre. Prior to rolling (red line), the texture type is Brass at the edge and Copper at the centre. With the development of deformation, the intensity along α fibre decrease. Along τ fibre, the highest intensity stays at the position of Copper component for the specimens rolled up to 50 passes at both edge and centre. The highest intensity shifts to Rotated Cube at the edge of the 100 passes sample, while at the centre, it stays close to the Copper component, but relative intensity is much lower than the 50 passes and the less reduction samples. At the same time, the intensity of Rotated Cube is the highest compared to the samples with less rolling passes. The intensity distribution along β fibre also confirms the mentioned trend.

In α fibre line, the Goss component is relatively intense in low strain and initial state, and then the intensity decreases at larger strain, because the Goss orientation is metastable and can be transformed into other components easily. S component is not strong as Copper but still maintains relatively high intensity in the rolling passes up to 50. Compared to the edge and centre part of specimen, the edge one shows more Rotated Cube texture than the centre part which indicates that at topmost of the sample, the shear stress dominates.

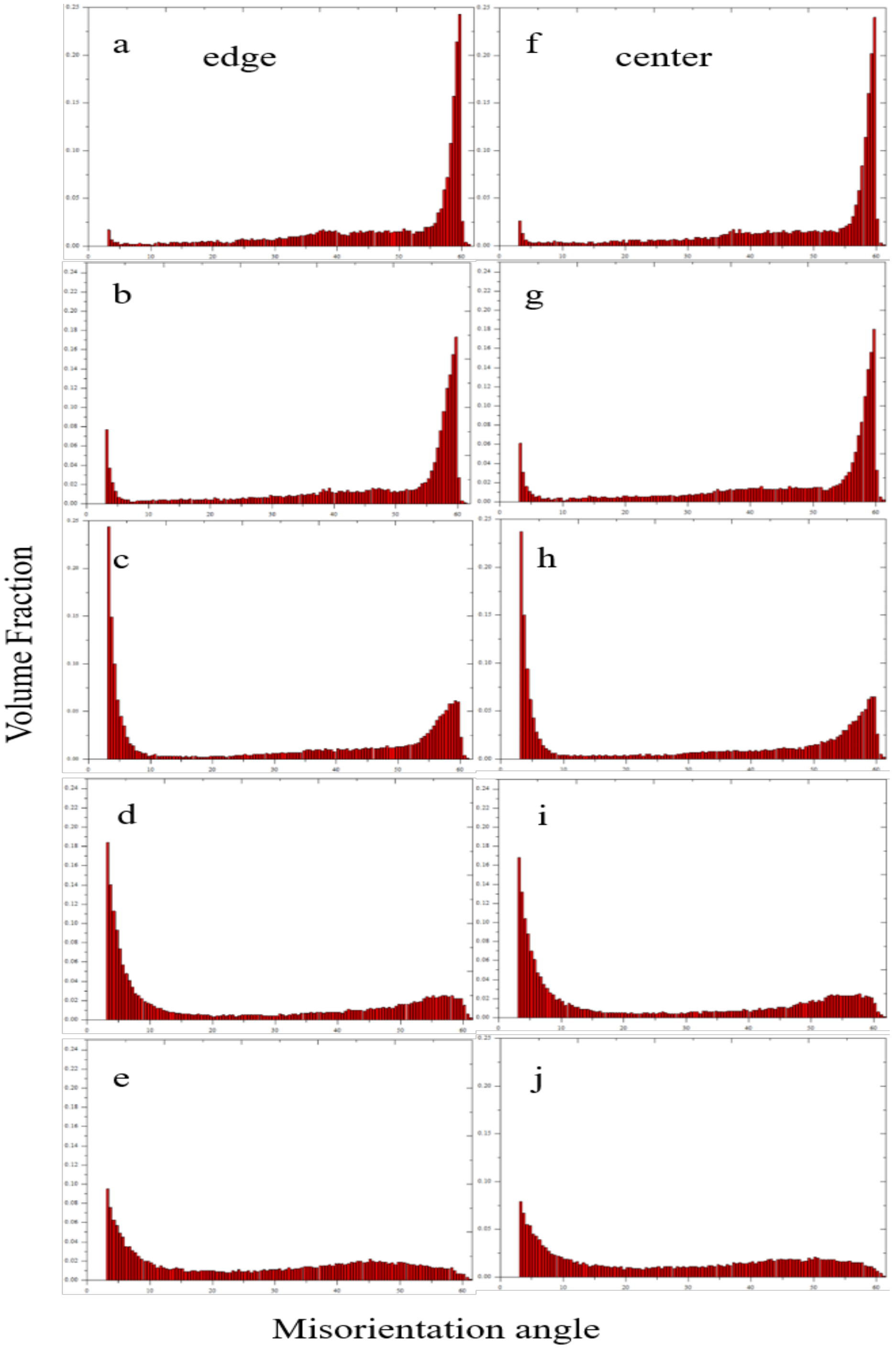

Fig. 3. Misorientation angle distribution of the annealed and skin pass rolled pure copper: (a, f: 300°C 2h, b, g: 10 passes, c, h: 20 passes, d, i: 50 passes, e, j: 100 passes). The horizontal axis range: 0~60°, the vertical axis ranges from 0 to 0.25 in intensity.

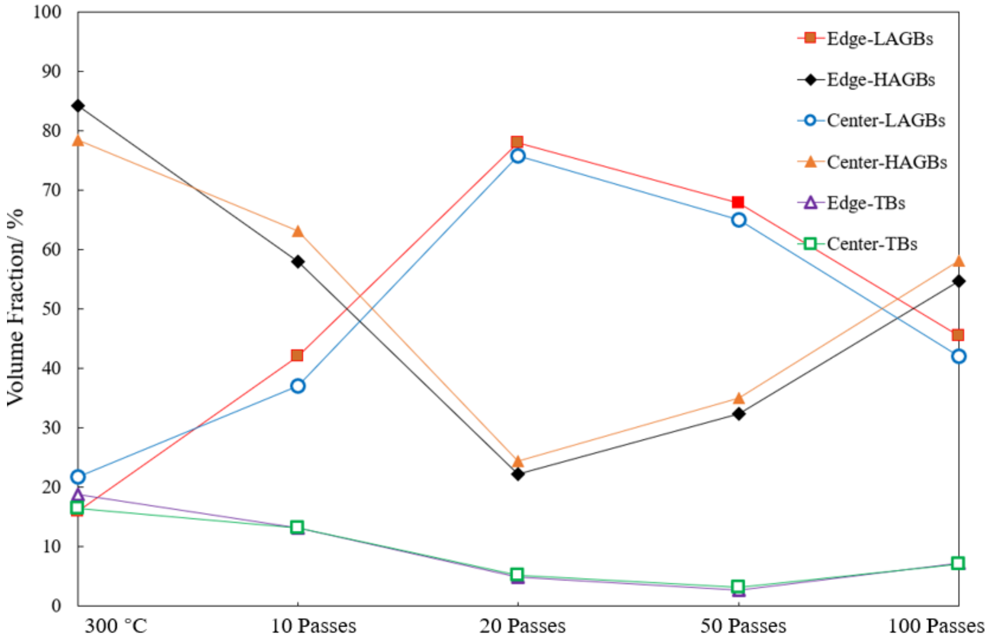

Fig. 4. Volume fraction distribution of LAGBs, HAGBs and TBs of annealed and skin pass rolled specimens.

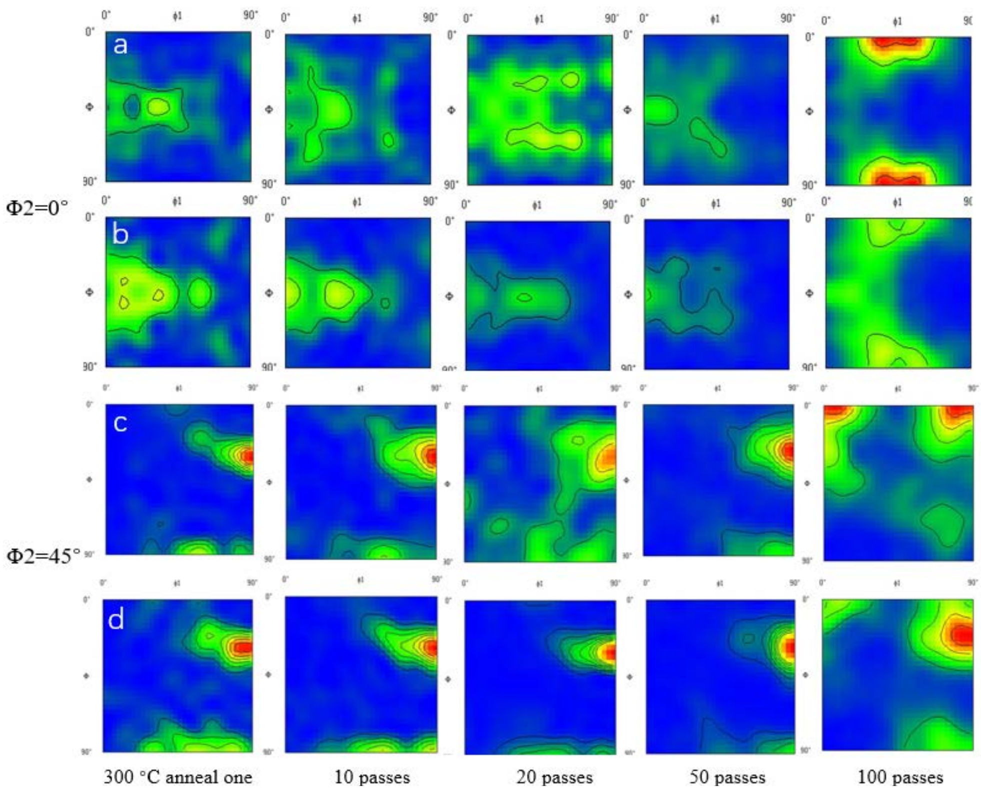

Fig. 5. ODF section of annealed and skin pass rolled specimens: a edge part at φ2=0°, b centre part at φ2=0°, c edge part at φ2=45°, d centre part at φ2=45°.

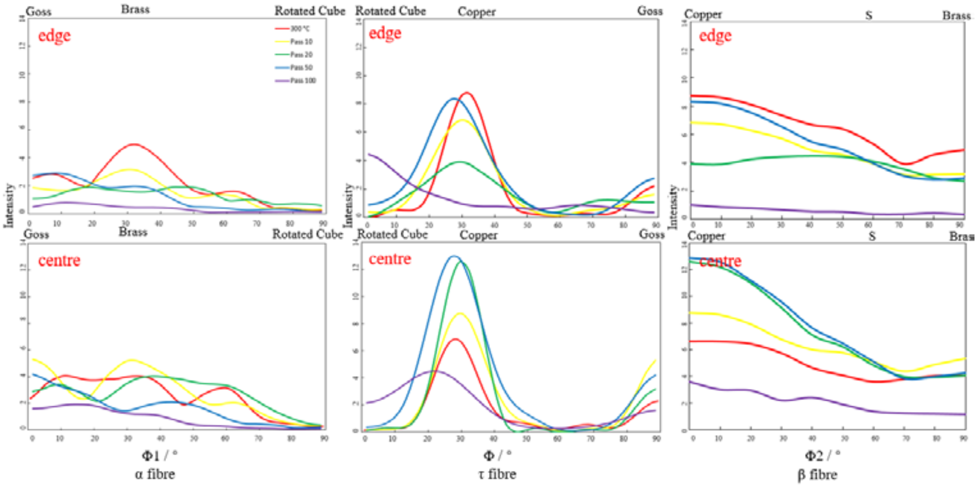

Fig. 6. Intensity distribution along α-fibre (Φ=45°, φ2=0°), τ-fibre (φ1=90°, φ2=45°) and β-fibre for the annealed and skin pass rolled specimens. The horizontal axis ranges from 0 to 90°, and vertical axis ranges from 0 to 14 in intensity of texture fibre.

4 Conclusions

Pure copper after 300°C annealing was processed by skin pass asymmetric rolling up to small and medium total reductions in this work to study the evolution of microstructure, mechanical properties and texture. The conclusions are:

1. The 300°C annealed samples still exhibited rolling/deformation microstructure and the initial grain size is about 85μm, after 100 passes skin pass rolling, the grains greatly refined to about 4μm.
2. Tensile testing results showed that at small rolling reductions (5.0% and 11.8%), the ductility of the sheets was still relatively high (more than 20 %) while the strength increased by 35.8%. At medium rolling reductions (32.0% and 63.6%), the strength of the sheets increased dramatically but at the same time, the ductility decreased drastically. Although the increment at strength with decrement of ductility is still existing, and the decrement of 10 passes is much smaller compared to the reported results of similar asymmetrically rolled samples.
3. At small rolling reductions, LAGBs dominated. With the increment of rolling passes and accumulated rolling reduction, LAGBs evolved into HAGBs.
4. The texture of the annealed and low rolling reductions samples was dominated by Copper component at both edge and centre. After rolling up to medium reduction (63.6%), the texture at the edge was Rotated Cube and the texture at the centre was dominated by Copper component with weak Rotated Cube.

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