Improvement of mechanical properties of Cu-10% Zn alloy processed by cryoforging

Dasharath S M, Suhrit Mula*

Department of Metallurgical and Materials Engineering, Indian Institute of Technology, Roorkee 247667, Uttarakhand, India

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Abstract

The microstructural evolution and mechanical properties of cast brass (Cu-10% Zn) subjected to cumulative strains of 3.0, 5.4 and 7.2 through multiaxial forging (MAF) at cryogenic temperature (77 K) were investigated. The mechanical properties of the homogenized and MAF treated alloy were measured through Vickers hardness testing. The brass deformed up to a cumulative strain of 7.2 showed improvement in the hardness of 2.3 GPa as compared with the as-homogenized annealed (0.55 GPa) alloy. The microstructural evolutions of cryoforged samples were characterized by optical microscopy, atomic force microscopy (AFM), transmission electron microscopy (TEM) and X-Ray diffraction (XRD). Thereby, it helps to increase twining activity for further deformation. TEM and AFM investigations confirmed that the formation of subgrains as well as nanotwins is responsible for the improvement of the mechanical properties. Copyright © 2018 VBRI Press.

Keywords: Copper based alloys; cryoforging; ultrafine grain; dislocation substructure; electron microscopy.

Introduction

Reduction of grain size has been an elongated approach for improving the strength and ductility of the metals and alloys [1]. More number of literature studies has been shown that grain size reduction from the coarse grain to nano/ultrafine range, which leads to significant strengthening due to the occurrence of large grain boundaries [2]. It is believed that the UFG impart high strength as expected from the Hall-Petch relationship, whereas the micrometer grains contribute to the enhancement of the ductility [2]. The microstructure and mechanical properties of materials processed by severe plastic deformation, such as equi-channel angular pressing (ECAP), high pressure torsion (HPT) and accumulative roll bonding (ARB), Ball milling (BM), and Multiaxial forging (MAF) have widely developed to produce the ultrafine-grained (UFG) or nanocrystalline (NC) materials [1]. Out of above these, MAF is a simple processing technique in which the material undergoes repeated forging in three orthogonal directions. The advantage of MAF is that, the original shape remains same with least distortion even after several cycles. Compared with the MAF at room temperatures or elevated temperatures, suppression of dynamic recovery and recrystallization during deformation at cryogenic temperatures preserves a high density of defects generated by deformation, which can act as the potential recrystallisation sites. Accordingly, the cryogenic deformation requires less plastic deformation for achieving ultra-fine grained structure, compared to the SPD processes at room temperature [3].

In the present study, Microstructural evolution and mechanical properties of Cu-10wt.% Zn alloys produced by cryoforging. The influence of cumulative strain on hardness of the brass at intermediate steps was examined through the Vickers hardness. A systematic study on microstructural evolution with increasing strain at mesoscale and microscale by using the optical microscopy, atomic force microscopy, transmission electron microscopy and X-Ray diffraction was made in the present work.

Materials and methods

Brasses with 10. % Zn (wt. %) were produced by metal mould casting technique from electrolyte grade copper (> 99.99%) and zinc (purity level > 99.99%). The zinc was added to the copper melt in proper ratio and stirred for 1 minute, and then molten alloy was poured into the cast iron mould of 72 mm×42mm×42mm size. After that the material was machined out to remove casting surface and homogenized at 800˚C for 4 h in a high purity argon atmosphere. The composition of the above cast sample was analyzed using optical emission spectroscopy. The samples of dimensions of 32.91mm×30.5mm×27 mm were cut for cryoforging at LN_2 temperature. The samples were subjected to MAF at 77 K using friction screw forging machine at a strain rate of 10 s^{-1}. The direction of the sample is changed for every pass at an angle of 90 degree. The sample dimension ratio 1:1.13:1.22 was maintained constant throughout the processing. MAF at cryogenic temperature was performed by filling the die with liquid nitrogen and its level was maintained up to sample height during forging. After every pass, the sample is allowed to attain thermal equilibrium with liquid nitrogen by giving 5–10 min soaking time. **Fig. 1** shows the schematic diagram of MAF for a single cycle. Strain per pass is fixed as $\Delta\varepsilon_i = 0.2$ (where ‘i’ number of passes) and maintained constant throughout all passes. The cumulative strain after one cycle of MAF was $\Sigma\Delta\varepsilon_{n=1} = 0.6$ (where ‘n’ is number of cycles). In the present study, MAF was carried out to cumulative strains of 3.0, 5.4, 7.2 i.e., 5, 9, 12 cycles, respectively. Samples were successfully forged up to 12 cycles without any cracking. Microstructure evolution and mechanical properties were studied after MAF at different cumulative strains ($\Sigma\Delta\varepsilon_{n=5} = 3.0$, $\Sigma\Delta\varepsilon_{n=9} = 5.4$, $\Sigma\Delta\varepsilon_{n=12} = 7.2$). To assess the strength, Vickers hardness measurements were carried out using a load 5 kg for a dwell time of 15s. Each reported hardness value is the average of at least 8 indentations. Microstructural analysis was carried out by optical microscopy, AFM, TEM and XRD analysis. Some selected samples were cut for TEM study from the center of the forged specimen. The cut specimens were thinned down to 80µm through grinding using silicon carbide abrasive papers of 800, 1200, 1500 grit size. 3 mm disc samples were punched out from this thin foil. Electro polishing was performed in a FEI twin jet electro polisher (operated at 40V) using a solution of methanol +20% nitric acid maintained at -40°C temperature. TEM investigation was carried out using FEI Technai 20 G2S-Twin transmission electron microscope operated at 200 kV.

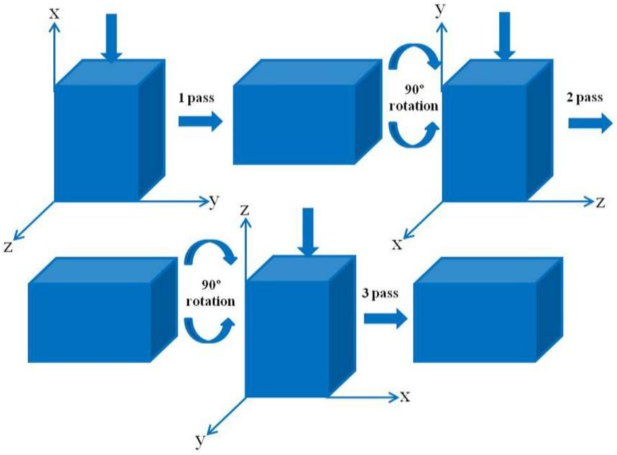

Fig. 1. Schematic of multiaxial forging corresponding to one cycle and cryoforging process flowchart.

Results and discussion

Microstructural investigation

Figs. 2 show the optical microscope of the as homogenised annealed and cryoforged samples, TEM and AFM analysis of the cryoforged samples. Average grain size of the as-received annealed specimen was estimated from optical microstructure and is shown in Fig. 2(a). The presence of large equiaxed grains can be observed with an average size of 172 µm, it is calculated by line intercept method. A large number of twins of various thicknesses may be seen from optical micrograph **(Fig. 2a)**. Forging at

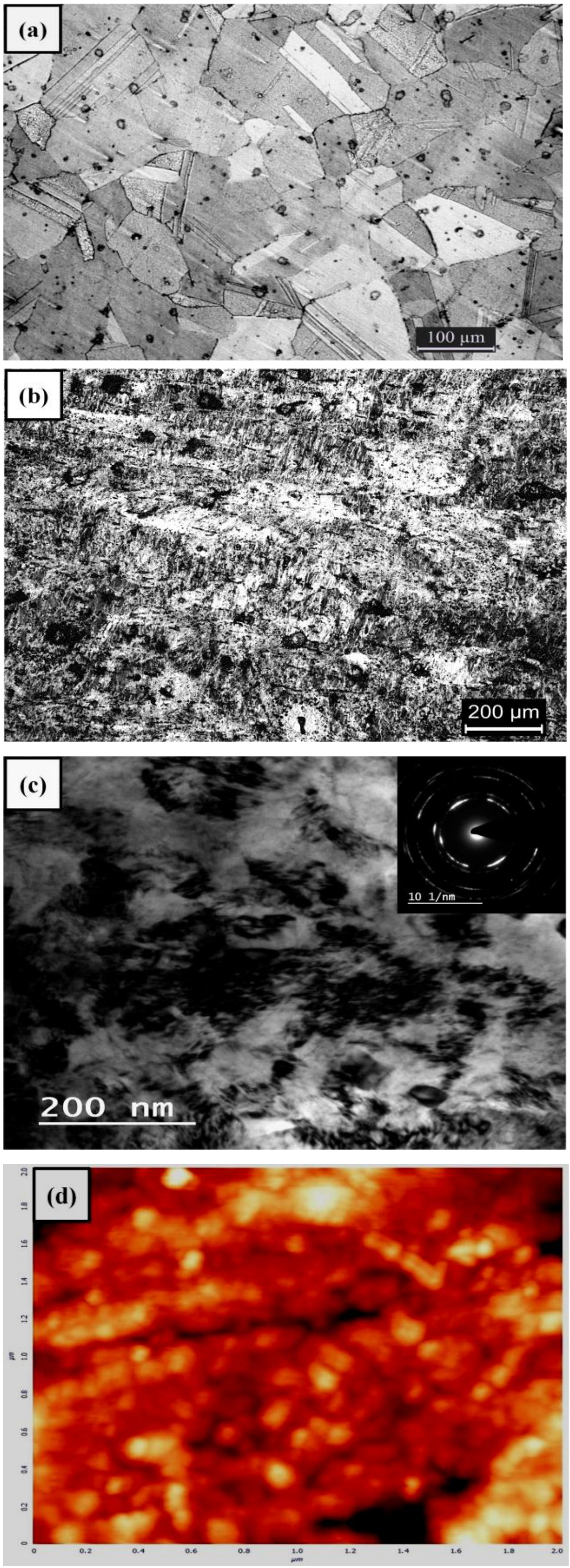

Fig. 2. Optical micrographs of (a) As homogenised Cu10%Zn, (b) 12 cycles, (c) TEM micrographs of 12 cycles bright field image, (d) AFM image of 12 cycles.

LN_2 up to 12 cycles, the microstructure **(Fig. 2b)** got full disturbed and filled with deformation bands and equiaxed fine grains. Observed from the Fig. 2b the band gap was reduced and small equiaxed grains was formed this may due to the high density of deformation bands with increasing the MAF strain. Under severe plastic deformation at low temperatures, several geometrically necessary boundaries (GNBs) such as micro bands or deformation bands [4] are easily developed. The mutual crossing, number of deformation bands and misorientation of deformation bands increases with strain; finally resulting in evolution of new fine grains [5-6]. Basically, the optical micrographs show highly deformed structures, and it is not possible to reveal the detailed microstructural features from the optical micrograph. Therefore, AFM and TEM analysis were carried out to confirm the grain size refinement achieved by cryoforging. **Fig. 2c** shows the TEM micrograph of MAF at cycles. Average grain size estimated from the TEM micrograph is ~ 90 nm. Similarly, Average grain was measured from **(Fig. 2d)** the AFM is~ 90nm; it is almost similar to TEM analysis.

X-ray diffraction analysis

Fig. 3 shows the XRD patterns of as-homogenised and cryoforged with different cycles of Cu-10%Zn alloy. It can be noticed from the **Fig. 3** that with increasing the number of cycles, the peak intensity of (111) and (200) decrease and width of the peak increased. The X-ray peak broadening is due to the combined effect of the grain refinement (finer crystallite size) and increase in residual strain [7]; whereas, the peak intensity of (220) increased, but it is also broadened with increase in the number of cycles. Compared to the as homogenized samples, the cryoforged samples exhibited peak shift towards small angles as well as peak broadening. Ungar [8] revealed the peak shift to the various internal stresses and peak broadening to the formation of fine grains. Hence it is believed that cryoforged samples have undergone the extensive plastic deformation and grain refinement which is also evident in the microstructures **(Fig. 1(b-d))**.

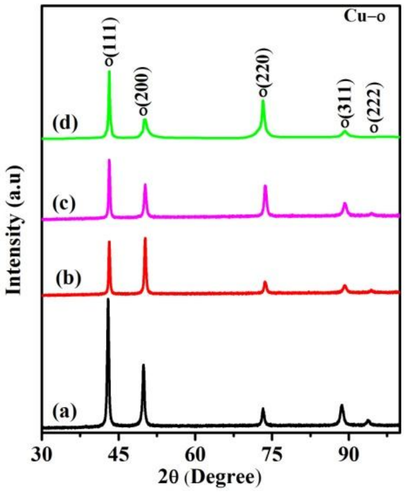

Fig. 3. XRD patterns of (a) As homogenised, (b) 5 cycles, (c) 9 cycles, (d) 12 cycles.

Mechanical properties

Fig. 4 shows the hardness of the as homogenised annealed and cryoforged samples. The Vickers hardness of the as received annealed material was measured to be 0.55 GPa. After forged under liquid nitrogen temperature up to 5 cycles, it increased to 2.0 GPa. On further forged to 9 cycles, the hardness values further enhanced to 2.1GPa. Maximum number of cycles was achieved by cryoforging was 12 cycles and corresponding measured hardness values is 2.3 GPa, which is more than 4 times as compared to the homogenised annealed samples. We have already mentioned that there is a massive grain size refinement achieved by cryoforging, which cannot be achieved by cold forging. Cryoforging of metals and alloys in cryogenic temperatures suppresses dynamic recovery and recrystallisation [9]; and the density of accumulated dislocations reaches its maximum saturation level with its highest possible number of MAF cycles. Therefore, materials get strain hardened to its maximum level with maximum cycles achieved. The cryoforged material possesses ultrafine-grain like substructures with high angle grain boundaries [10] in its highest possible deformed condition. The formation of nanocrystalline material is possible during cryoforging if dynamic recovery is not completely suppressed; otherwise a short time low-temperature annealing helps in the formation nanocrystalline material of high-angle grain boundaries. Thus, grain size refinement occurs in cryoforging technique. The SPD deformed structures usually contain a large fraction of low angle grain boundaries [1], and if the material possesses unique combination of strength and ductility, presence of high angle grain boundaries normally predominant indicating the formation nanocrystalline materials as reported in the literature [1,9,10]. Therefore, the increase in hardness is not only due to strain hardening of the material but also due to grain size reduction [11]. In fact, increase in hardness is possibly dominated by the vast grain size refinement.

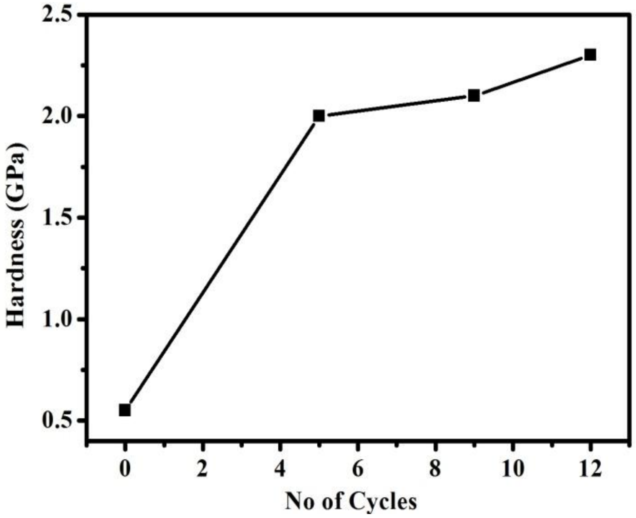

Fig. 4. Hardness of as Homogenised and Cryoforged with different number of cycles.

Conclusions

Ultrafine grain brass was prepared from commercially available Cu-10 wt.% Zn alloy by cryoforging. Detailed Investigation of microstructure and mechanical properties were made. On the basis of obtained results and their analysis, the following conclusions can be drawn. The homogenized annealed samples of Cu–10% Zn alloy showed the presence of more number of fine twins due to reduction of the SFE in presence of more amounts of Zn. Cryoforged samples with 12 cycles show highest value of hardness, as compared to that of the homogenised annealed samples. It is due to the refinement of grain size by the combined effect of suppression of dynamic recovery and recrystallization and the density of accumulated dislocations reaches its maximum saturation state with its highest possible number of cycles.

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