Mechanical properties of ultrafine grained bulk copper alloy developed by cryorolling

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ABSTRACT

In the present work, we have reported the microstructures and mechanical properties of bulk Cu-4.5 Al wt%. alloy subjected to cryorolling (CR) under liquid nitrogen temperature (LN_2). Samples were plastically deformed to obtain different reduction in area (RA), i.e. 75%. The mechanical properties of cryorolled samples were investigated by tensile testing and hardness measurement. Cryorolled samples with 75% RA shows maximum tensile yield strength (600 MPa) with an elongation only ~2.1% as compared to its coarse grain. The increase in mechanical strength is due to the refinement of grain size by the combined effect of suppression of dynamic recovery and recrystallization, and accumulation of high density dislocations during cryorolling. Brittle type of fracture was identified for 75% RA cryorolled samples from fractography analysis, whereas presence of dimples in the fractured surface of it s coarse grain specimens indicated a ductile failure.

1.INTRODUCTION

In today’s era there is great technological interest in ultrafine grain materials of bulk size and superior mechanical properties of material [1-3]. Ultrafine grain materials, materials of less than 1000 nm of grain size, typically have better mechanical strength than their coarse grain counterparts. Microstructure refinement brings enhanced yield strength and toughness to attractive mechanical properties [4]. Low ductility, which may be related to the material's intrinsic properties [5], is one of the major problems with the use of ultrafine grain materials.There are several methods for the production of ultrafine / nanocrystalline bulk materials. Several plastic deformation techniques (SPD) such as equal channel angular pressing (ECAP)[6], high pressure torsion (HPT)[7], accumulative roll bonding (ARB)[8], multi-axial forging (MAF)[9], repetitive corrugation and reinforcing (RCS)[10] are can be develop ultrafine grain metals/bulk nanostructure for functional and structural use. In SPDtechniques major problem are that it requires severe plastic strains (more than unit), design difficulties, expensive tooling, production of comparatively less material quantity, and the cost factor that makes it unreliable for industrial applications to be used. Most of the above problems can be avoided by using cryorolling technique to produce ultrafine grain materials. Ultrafine grain bulk structures of pure Cu and Al alloys are reported from their bulk counterparts by using cryorolling techniques to deform them at liquid nitrogen temperatures.By rolling at cryogenic temperatures of pure metals / alloys, plastic deformation and recrystallization and suppresses dynamic recovery; and the dislocation density reaches a maximum stable level. In its highest possible deformed condition, the cryorolled material possesses Ultra-fine grain like low-angle grain substructures. If partial dynamic recovery is possible, during cryorolling, nanocrystalline material may be formed. In the formation of high-angle grain nanocrystalline materialGrain size refinement is achieved through cryorolling technique Deformed structures typically have a large fraction of low-angle grain boundaries and a unique combination of strength and ductility of the material, high-angle grain boundaries usually indicate the formation of nanocrystalline materials as reported in the literature. Many reports are available on the role of microstructural features on ultrafine grain metal and alloys mechanical properties such distribution a spacing and grain size and twin size, stacking faults, dislocation density. Ultra-fine grained materials of bulk size can be easily developing with low stacking energy fault. For everyday applications, it has a corrosion and good combination of mechanical properties of resistant and is a material for stacking fault energy (35-12 mJ / m2). The stacking energy of copper alloys can be increased by both dislocation density and twin accumulation by deforming them at sub-ambient temperatures.Therefore, it improves the work hardness rate and at the same time increases strength and ductility as reported in literature. It is observed that rolling gradually increases twin density and density of dislocations at low temperatures. An explanation for this is available. First, lowering the stacking fault energy increases the splitting into two parts of the complete dislocations with a wide stacking fault ribbon. Secondly, reducing stacking energy also encourages more twins to be produced in the cryorolling process.Twinning partials are more prevalent at sub-ambient temperatures and/or high stress rates. As reported in literature by plastic deformation at liquid nitrogen temperature, the high density Nano-scale twins were formed in Cu / Cu-alloys. Although several reports are available on Cu-4.5% Al alloys investigation for the development of ultrafine grain materials, no such report is available on mechanical behaviour and microstructural evolution of commercial grade cryorolledCu-4.5%Al alloy.With the above views, by rolling at LN_2 temperature from commercially available Cu-4.5%Al alloy, an attempt was made to prepare ultrafine grain. Samples were plastically deformed in order to obtain different area reduction (RA), i.e. 50%, 75% and 90%. Measurement of mechanical properties such as hardness of Vickers and tensile testing of all samples was examined.

2.MATERIALS AND EXPERIMENTAL DETAILS

Copper alloy (Emissions Spectrometer Cu-4.5%Al(OES). For various area reduction (RA) samples, i.e. 50 percent, 75 percent and 90 percent, the samples were cut from the plate for cryorolling. Table 1 shows the nominal chemical composition (in weight percent) of the alloy of Cu-4.5%Al

Components	Al	Cu
wt.%	4.5	Balance

Table 1. Nominal chemical composition of Cu-4.5%Alalloy analyzed by optical emission spectroscopy.

Mechanical polishing

Prior to cryorolling, the samples were mechanical polished using SiC_3 abrasive papers with grit size 2000 to obtained smooth oxidation free surface after heat treatment.

Cryorolling setup

Two high conventional rolling mill

It is used for cryorolling of the sample. The diameter of the rolls is 110mm and the rolling speed is 8 r.p.m.

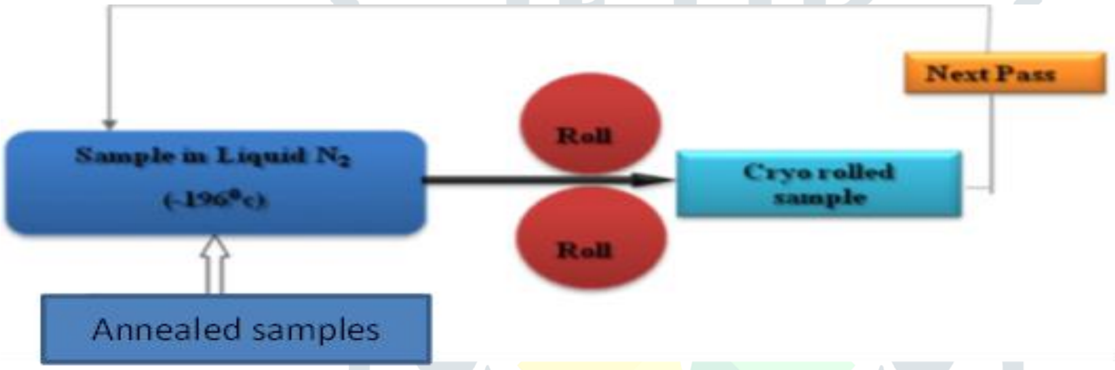


Fig.1: Flowchart of cryorolling

Accessories used

- Cryocan – used for procuring and storing liquid nitrogen.
- Sample container – to hold the sample in liquid nitrogen. It is made of stainless steel covered with refractory material i.e. glass wool to minimize the heat transfer from surrounding, so that liquid nitrogen can be liquid state for long time.
- Set of gloves – for handling the sample during cryorolling.
- Vernier caliper – for taking thickness measurement in each pass.

Multipass cryorolling by dipping all samples into containers of liquid nitrogen. Before rolling the bulk rectangular specimens (cross sectional area= 250 mm) were submerged in liquid nitrogen until liquid N2 saturation temperature was reached. Area (RA) reduction Maintained at ~2 percent per passand multiple passes were issued for maximum RA. Digital VernierCaliper was used to measure the change in thickness. The specimens were dipped in liquid N2 for sufficient time between each rolling passes to reach saturation temperature. Samples from different RA characteristics were collected, i.e. 50%, 75% and 90%. Proper care has been taken during cutting to prevent heat generation.

Microstructural characterization

Optical microscopy

As received annealed and material with different percentage reduction in area after cryorolling were cut in to proper dimension and mounted over mounting blocks for the purpose of further analysis.

Grinding

With different abrasives, copper alloys can be ground. Grinding was performed in successive steps using 320, 800, 1200, 1500 grit SiC3 abrasive papers. For each paper that follows, effective sample results turned by 900. 2000 grit size has been used to achieve a more uniform surface finish. This finer abrasive paper leads to less deformation of the surface. After each grinding, the samples were thoroughly washed to remove loose particles attached to the sample.

Mechanical cloth polishing

Cloth polishing was accommodated in two steps, rough and fine polishing. A variable velocity wheel is preferred for polishing. MgO was used for raw polishing. An abrasive tea spoon is applied near the center of the cloth, moistened with distilled water and then turned into paste. MgO solution has been used in distilled water for final polishing. After final polishing, which is a solution of 16 g ferric chloride + 5ml hydrochloric acid + 100ml distilled water for 20 seconds, the samples were cleaned and etched with reagentMicrostructure was examined under polarized light using the LEICA DMI 5000 M Optical microscope.

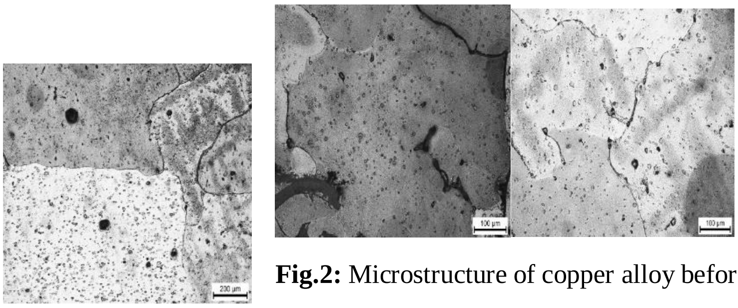
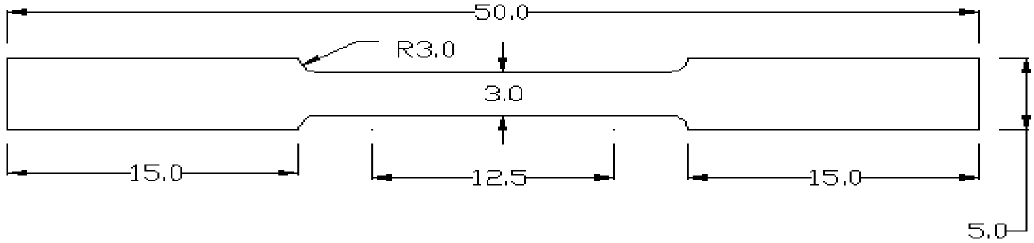


Fig.2: Microstructure of copper alloy before cryorolling

Tensile Testing

Tensile testing was performed on the TiniusOlsen S-series, H25K-S (Fig. 3) materials testing machine operated at constant cross-head speed with an initial strain rate of 5x 4 s-1 on the specimen to calculate the tensile strength, yield strength and ductility of the samples. During the production of tensile specimens, an ASTME-8 sub-size standard was considered.



All dimensions are in mm

Fig. 3: Tensile test specimen as per ASTM E8 standard.

Total tensile specimen length with 12.5 mm gage length was parallel to cryorolled and cryorolled + rolling direction. Before testing, the sample length was polished to achieve scratch-free surface and maintain uniform width throughout the gauge length. For each condition, three samples were tested to check for reproducibility. Three samples ' average tensile strength was taken.

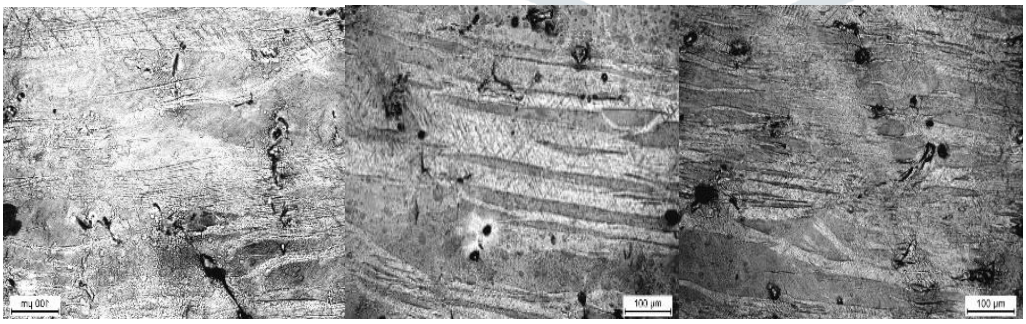


Fig.4: Microstructure of copper alloy after cryorolling

3.RESULTS AND DISCUSSION

Mechanical properties

We have already mentioned that CR has achieved a massive grain size refinement that cold rolling can not achieve. Cryorolling in metal and alloy recrystallization of cryogenic temperatures and suppresses dynamic recovery; and accumulated dislocation density with its maximum possible RA reaches its maximum saturation level. Therefore, with maximum RA achieved, materials are hardened to their maximum level. In its highest possible deformed condition, the cryorolled material has ultrafine grain like substructures with low angle grain boundaries Limits in their highest deformed condition The formation of nanocrystalline material during cryorolling is possible if dynamic recovery is not completely suppressed; otherwise the formation of high-angle grain nanocrystalline material is assisted by short-term low-temperature annealing. Grain size refinement takes place in the technique of cryorolling. SPD deformed structures usually have a large fraction of low-angle grain boundaries and where the material has a unique combination of strength and ductility, higher grain boundaries usually predominate indicating the formation of nanocrystalline materials as reported in the literature. The increase in hardness is therefore due not only to the strain hardening of the material, but also to the reduction in the size of thegrain. Indeed, the vast refinement of the grain size may dominate the increase in durability

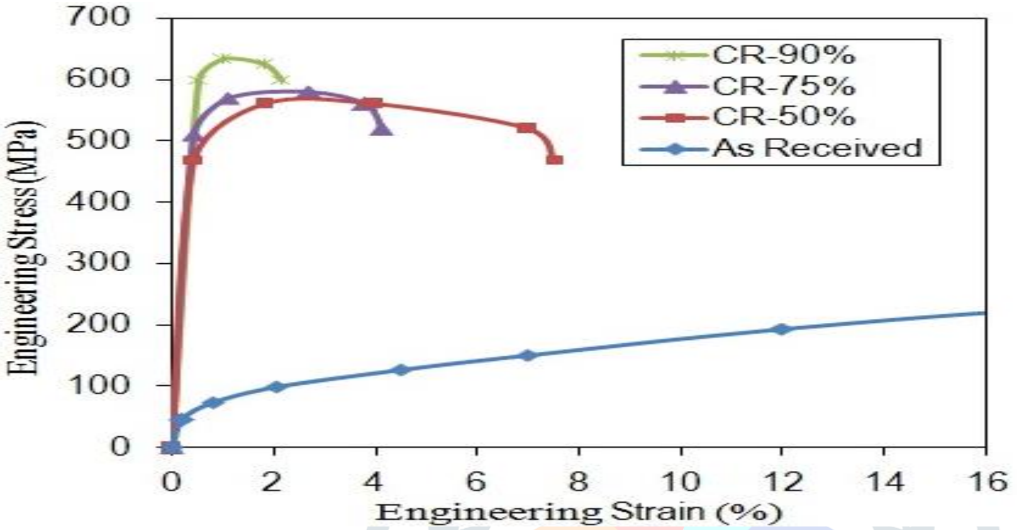


Fig. 5: Engineering stress-strain curves for as received annealed and various % RA specimens.

The mechanical tensile behaviour of the annealed and cryorolled samples received is shown in Fig5. It can be noted that the yield strength (YS) of the annealed material received and the ultimate tensile strength (UTS) of 80 and 266 MPa respectively. Up to 50% of RA, YS and UTS were 470 and 560 MPa respectively after cryorolling. The additional deformation of the RA, YS and UTS of the specimen increased further to 75% to 510 and 581MPa. Measured to be 600 and 633 MPa of the same material with a maximum reduction (90% RA) for LN2, YS and UTS respectively. Plastic deformation at sub-ambient temperature consequences is expected to have high strength accompanied by good ductility value in the suppression of dynamic recovery, promotion of twin activity, and higher dislocation density. The current stacking fault energy of the alloy is about 17 mJ / m2; reduced stacking fault energy restricts the movement of dislocation by slip and therefore the twinning mechanism for plastic deformation is preferred.Therefore, rates of work-hardness are reported to rise and cause simultaneous increases in strength and value of ductility. Because the available minimum grain size is proportional to the stacking fault energy, low stacking fault energy enhances grain refinement and improves mechanical properties compared to high SFE alloys. It's visible from the Fig. 4 That the YS and UTS of the Cu-20 percent Zn alloy will increase as the RA percentage increases, but at a high ductility cost.With an average grain size of 150 μm (Fig. 4), the received material's elongation percentage was 39 %. The ductility was reduced to ~2 percent corresponding to its 600 MPa YS for its maximum possible reduction, i.e. 90 percent RA. It is a common phenomenon observed in highly deformed metals and alloys due to the inverse relationship between strength and ductility.

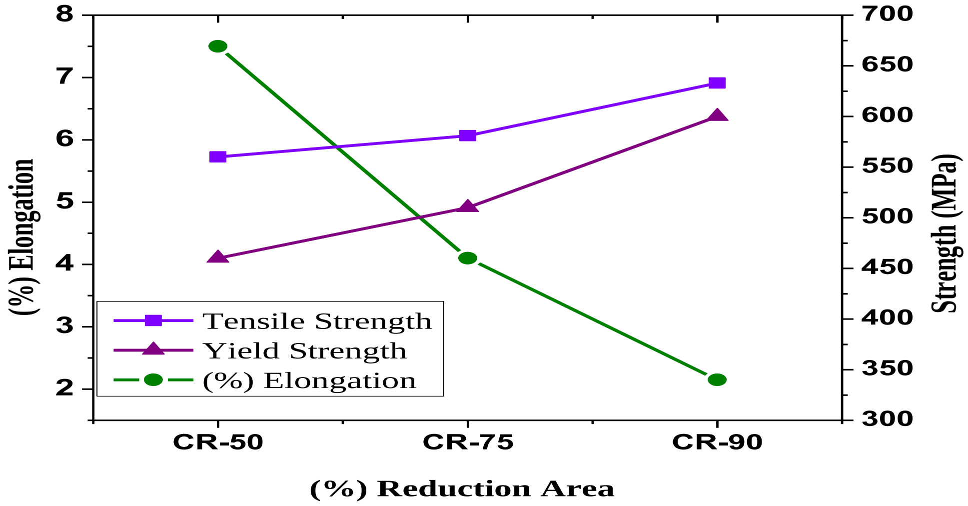


Fig. 6: Showing the variation in UTS, YS and % elongation with reduction in area (RA).

The variation in UTS, YS and elongation percentage due to area reduction is shown in Fig. 6. Thus while tensile YS increased to a very high value (600 MPa), due to its very low ductility, the material could not be useful for its structural applications. Therefore, to achieve the best combination of strength and ductility, it is necessary to compromise the strength a bit to increase the elongation. Therefore, for 90% RA samples at different temperatures, i.e. 225, 250 and 300°C for 20 min. under high purity argon atmosphere, a short annealing was performed and mechanical properties were investigated.

Table 2: Variation in hardness of the cryorolled sample

S1.No.	Sample condition	Hardness (VHN)	Hardness (GPa)
	As received (annealed at 800°C-3hours)	56	0.549
2.	CR-50%	196	1.92
3.	CR-75%	216	2.11
4.	CR-90%	226	2.21

CONCLUSION

1. Ultrafine grain brass was prepared from commercially available Cu-4.5%Al wt by rolling at LN2 temperature. Detailed investigation of microstructure and mechanical properties was carried out. Based on the results obtained and their analysis, the following conclusions can be drawn.

2. Cryorolled samples with 90% RA have the highest value of mechanical properties (YS-600 MPa and UTS-633 MPa) . Because of the grain size refinement by the combined effect of recrystallization and suppressing dynamic recovery the accumulated dislocation density reaches its maximum saturation status.

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