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# Effect of Nb, Y and Zr on thermal stability of nanocrystalline Al-4.5 wt.% Cu alloy prepared by mechanical alloying



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# ABSTRACT

In the present study, effect of Nb, Y and Zr (having low equilibrium solid solubility in Al) in the formation of nanocrystalline Al-4.5 wt.% Cu-1at.% X (X = Nb, Y & Zr) solid solutions by mechanical alloying and their thermal stability have been investigated. The mechanically alloyed samples were annealed in batches for 1 h at different temperatures ranging from 150 to 550 °C. The thermal stability has been studied through X-ray diffraction (XRD) phase analysis and variation of microhardness as a function of temperature. Transmission electron microscopy (TEM) and SAED analysis showed that 1 at.% Y and 1 at.% Nb could be dissolved into Al-4.5% Cu solid solutions after mechanical alloying up to 8 h of milling. The spontaneity of formation of Al-4.5%Cu–Y, Al-4.5%Cu–Nb and Al-4.5%Cu–Zr solid solutions has been explained from the change of Gibbs free energy as per Miedema's and Toop's models. It demonstrated that the summative energy due to the reduction in crystallite size and accumulation of dislocation density exceeds the theoretical energy barrier required for the formation of the Al-4.5%Cu–Y, Al-4.5% Cu–Nb disordered solid solutions. The grain sizes were found to retain <100 nm even after annealing at 550 °C. This is attributed to the stabilization of metastable grains by segregation of large size solute atoms (Nb, Y and Zr) along grain boundaries and/or Zener pinning by intermetallic precipitates like Al<sub>3</sub>Nb in Al– Cu–Nb, Al<sub>3</sub>Y and Al<sub>3</sub>Y<sub>5</sub> in Al–Cu–Y and Cu<sub>10</sub>Zr<sub>7</sub>, Al<sub>3</sub>Zr and Cu<sub>5</sub>Zr in Al–Cu–Zr alloys.

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# 1. Introduction

Recent developments in the automobile, aerospace, defence and construction industries have led to increased interest in developing Al–Cu alloys with improved elevated temperature strength, stiffness and reduced density [1–4]. Achieving these properties would extend the lifetime of the components and reduce fuel cost significantly. The need for such improvement is a major drive for processing and alloy development in the structural aluminium alloys. Addition of Cu up to about 5 wt.% in Al has resulted in good toughness and increased strength subjected to aging conditions [1].

Though Al–Cu alloys possess excellent room temperature properties, their properties at elevated temperatures (above 423 K) is limited due to the coarsening of the  $\theta'$  (CuAl<sub>2</sub>) precipitates at higher temperatures leading to decline in hardness and creep resistance of the alloy [4]. Earlier reports demonstrate that an addition of rare earth (RE) elements could significantly increase the peak age hardness and improve the thermal stability of the Al–Cu

alloys [2,3]. For instance, addition of praseodymium (Pr) brought about the refinement of grains and improved the thermal stability of the  $\theta'$  precipitates [4]. Refinement of the alloys to ultrafine/ nanocrystalline range has gained great attention due to their superior mechanical and physical properties. But, the increased surface area causes pure ultrafine/nanocrystalline materials to have extremely unstable microstructure. They tend to reduce it in order to decrease the grain boundary energy associated with it and tend to coarsen at elevated temperatures, losing their unique properties acquired in ultrafine/nanocrystalline structures [5-7]. Addition of dopant material has been found to reduce grain coarsening at elevated temperatures by means of two strategies: kinetic or thermodynamic stabilization. Kinetic stabilization utilizes effects such as solute drag, second-phase particle pinning (Zener pinning), chemical ordering, or porosity. In the case of thermodynamic stabilization, the solution of the Gibbs interface energy equation leads to the relation.

$$\gamma = \gamma_0 + \Delta G_{seg} \Gamma_s \tag{1}$$

 $\gamma_0$  is the non-segregated grain boundary energy and  $\Delta G_{seg}$  is the

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free energy change associated with segregation of the solute [8]. According to this equation when  $\Delta G_{seg}$  <0, a metastable thermodynamic equilibrium can be achieved by a nanocrystalline material at  $\gamma = 0$ . Development of nanocrystalline material by mechanical alloying strains the material, which enhances its grain boundary area and consequently its energy. Nonequilibrium processing methods like mechanical alloying can be exploited to bring solutes having high atomic radius and lower solubility in Al (under equilibrium conditions) into the solid solution of Al easily [17].Annealing of the nanocrystalline material at suitable temperature allows solute diffusion to the high energy grain boundary sites to decrease its energy, as compared to that of the metastable as-milled alloy. These solutes occupy the grain boundary regions and thermodynamically reduce the grain boundary energy, which eliminates the driving force for grain boundary migration and grain growth [9].

Processing of nanocrystalline Al alloys by mechanical alloying is well established technique to produce tonnage of nanopowders [10]. But, consolidation is unavoidable in the processing of the nanocrystalline bulk materials and often involves high temperature and pressures, leading to change in the microstructure. Haber et al. observed grain growth at room temperature in nanocrystalline pure Al prepared by chemical methods [11]. But in many works, exceptional thermal stability of nanocrystalline aluminium alloys has also been observed [12–15]. Their thermal stability has been ascertained to the presence of impurities or second phase particles due to their interaction with the process control agent (PCA), reactive ball milling or the milling medium. For instance, Zhou et al. reported an initial grain size of 26 nm and maintains a grain size of about 50–60 nm even at 0.78T<sub>m</sub> [12]. Addition of Cu improves the strength of aluminium due to the solid solution strengthening of Cu. Shanmugasundaram et al. [16] also reported a high thermal stability of Al-4wt.% Cu alloy. They reported that the frictional stress improved from 15 to 30 MPa for pure Al to 170 MPa for Al-4wt.% Cu alloy. Addition of Zr in Al by inert gas condensation increased the hardness (to 1.7 GPa) about 5 times as compared to coarse grained Al (0.29 GPa) [18]. The effect of Ti on the grain size refinement and thermal stability of Al-10 wt.% Cu alloys was demonstrated explicitly in Ref. [19]. The effect of misfit solutes on the thermal stability of Cu based alloys [9,20–23] like Cu-Nb [20], Cu-Y [21], Cu-Zr [9,22], Cu-Ta [23] prepared by mechanical alloying was studied by various researchers and it was found that a higher extent of thermal stability was exhibited by Nb, Y and Zr. The effect of Zr on thermal stabilization of pure Fe [24], Fe-10 and 18 wt.% Cr [25] was reported to enhance to a great extent. Similarly, effect of Y and Hf on the thermal stability of Fe-Cr alloys was also reported to increase significantly [26,27]. Effect of Y addition in Ni and its grain growth kinetics were investigated in Ref. [28].

Though, addition of large size insoluble solutes in various metals has been found to improve their thermal stability significantly, effects of Zr, Nb or Y addition in Al–Cu alloys and their thermal stability have not been reported so far. The Al–Cu alloys (duralumin) are extremely important group of aerospace and automobile materials, and high temperature strength and stiffness are strongly dependent on the thermal stability of the ultrafine/nanocrystalline grains of such alloys. Thus, it would be highly reasonable to investigate the stabilizing ability of Nb, Y and Zr on nanocrystalline Al-4.5 wt.% Cu alloy. The thermodynamic feasibility and mechanisms of thermal stability using Toop's model have been discussed in detail.

## 2. Experimental details

Elemental powder blends (Alfa Aesar, purity  $\geq$ 99.7%) of Al-4.5 wt.% Cu-1at.% X (X = Zr, Nb,Y) compositions were milled in 440 stainless steel grinding media using SPEX 8000 M high energy ball mill. The grinding media consists of 8 mm (17 in numbers) and 6 mm (16 in numbers) diameter balls, and a ball-to-powder weight ratio of 10:1 was maintained throughout the milling time. The vial was sealed in high purity argon atmosphere (purity <10 ppm  $O_2$ ) prior to milling and the milling was carried out for 8 h at room temperature. The as-milled powder samples were compacted as 10 mm diameter disk samples in a stainless steel die-punch at an applied pressure of 300 MPa using a hydraulic press. Then the disk samples were annealed in batches at different temperatures from 150 to 550 °C under Ar+2%H<sub>2</sub> atmosphere (purity <10 ppm  $O_2$ ). XRD analysis of the as-milled and annealed powders was performed using Cu-K $\alpha$  radiation ( $\lambda = 0.154$  nm) at a scan rate of 1°/ min in a Rigaku X-ray diffractometer. The crystallite size of the Albased alloys has been calculated from five major XRD peaks after eliminating the broadening effects due to strain by using the plot between  $B_r \cos\theta vs. \sin\theta$  [29,30]. Precise values of lattice parameter of the Al-based alloys  $(a_{Al})$  were calculated from the 5 major XRD peaks by the extrapolation of  $a_{A1}$  vs.  $(\cos^2\theta/\sin\theta)$  plot to  $\cos\theta = 0$ [29,30] The disk samples were polished to a mirror finish surface and Buehler microhardness (Model: UHL Technische Mikroskopie VMHT) tester was employed to perform Vickers microhardness measurements. Microhardness test was carried out using 50 g load at a speed of 15  $\mu$ m per second with a dwell time of 25 s for each indentation. The each reported hardness value is the average of at least 6 indentations. Transmission electron microscopy (TEM) analysis was carried out for some selected as-milled and annealed samples using a JEOL 2000FX at a beam energy of 200 keV. The TEM samples were prepared by drop cast technique using a carbon coated copper (Cu) grid.

# 3. Results and discussion

#### 3.1. XRD analysis of metastable solid solutions

Fig. 1 shows the XRD patterns of the Al-4.5 wt.% Cu-1at.% X



Fig. 1. XRD patterns of the as-milled samples of Al-4.5 wt% Cu-1at% X (X = Nb, Y & Zr) in comparison with that of the Al-4.5 wt% Cu and pure Al.

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