

Compressive deformation study on bimodal AA 4032 alloy produced by ball milling

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The microstructure, mechanical properties and deformation behavior of bimodal structured nanocrystalline AA 4032 alloy are investigated in the present work. The bimodal structure alloy powders are fabricated using blending of coarse grained powders with nanocrystalline powders. The nanocrystalline powders are prepared by mechanical alloying using high energy ball mill for 30 h. A different mass fractions of coarse grained powders (micron size powders or micro powders) are taken for blending and the powders are consolidated by hot pressing to produce bulk bimodal alloys. The addition of micron sized powders is improved the ductility of the bimodal alloy along small reduction strength when compared with nanocrystalline alloy. The deformation of the bimodal alloy is due to highly localized plastic deformation under uniaxial compression. The failure of the alloy is due to shear localization and cavitations.

Keywords: Bimodal, Ball milling, Nanocrystalline, Deformation, AA 4032 alloy, Mechanical properties

The mechanical properties of the materials mostly depend on the grain size and the microstructure. Usually microcrystalline materials give more strength by suitable reinforcement, reduction in their crystallinity nature¹. Nanocrystalline materials exhibit high strength based on Hall-Petch relationship when compared with microcrystalline materials. But bulk nanocrystalline materials show a poor ductile properties at room temperature which is due to strain hardening during synthesis². However, a combination of both micro and nanocrystalline grain size distributed materials exhibits a high strength with better ductility³⁻⁵. Many researchers suggest that the bimodal grain size distribution enhances the ductility of nanocrystalline materials. But fabrication of dual structure by warm compaction, HIPing and hot extrusion is very difficult since uniform distribution of the dual structure is not possible^{6,7}. To avoid all these problems blending of coarse grain and nanocrystalline powders followed by consolidation is the easiest method to achieve bimodal grain size distribution. In the present work, mechanical alloyed nanocrystalline and coarse grained (unmilled) powders can be combined with different proportion to achieve high strength with improved ductile properties of nanocrystalline materials.

Experimental Procedure

Nanocrystalline AA 4032 powder was produced by using Fritsch P5 high energy ball mill at room temperature. Elemental powders of AA 4032 were mechanically alloyed with WC media and the BPR was maintained as 10:1. A small amount of toluene was added to the powders to prevent sticking of powders and heat built during milling. The ball mill was operated at 300 RPM and the milling time was 30 h. The phase evolution and the crystallite size were measured by X-ray peak broadening technique using Williamson Hall analysis. The nanocrystalline powders were mixed with three different proportions (10, 20 and 30 wt%) of unmilled powders to achieve bimodal grain size distribution. The bimodal powders along with nanocrystalline and microcrystalline powders were consolidated using hot compaction press at 450 MPa at 480°C for bimodal and nanocrystalline compacts. In the present study, an optimized value of 20% coarse grain powders added to the nanocrystalline powders were taken based on the hardness value. The phase evolution during ball milling and the crystallite size was calculated using X-ray diffraction technique. The microstructure analysis was done by using scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The hardness and compressive strength of the sintered compacts were measured.

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Results and Discussion

Phase evolution

Figure 1 shows the XRD pattern of microcrystalline, nanocrystalline and bimodal crystalline powders along with peak shift. It shows that nanocrystallinity nature of the powders were achieved through the broadening of the peak and peak shift (Fig. 1b) in ball milled powders. The as received or unmilled powders shows aluminium peaks along with major alloying element peak silicon, but after 30 h of milling, more silicon atoms diffused into the aluminum atoms which forms a solid solution. Moreover large amount strain was accumulated into powders during mechanical alloying which reduces the crystallite size with increased lattice strain. The crystallite size of nanocrystalline powders were calculated using modified Williamson-Hall analysis and the final crystallite of the powders after 30 h of ball milling is 17 nm and 0.176% of strain⁸.

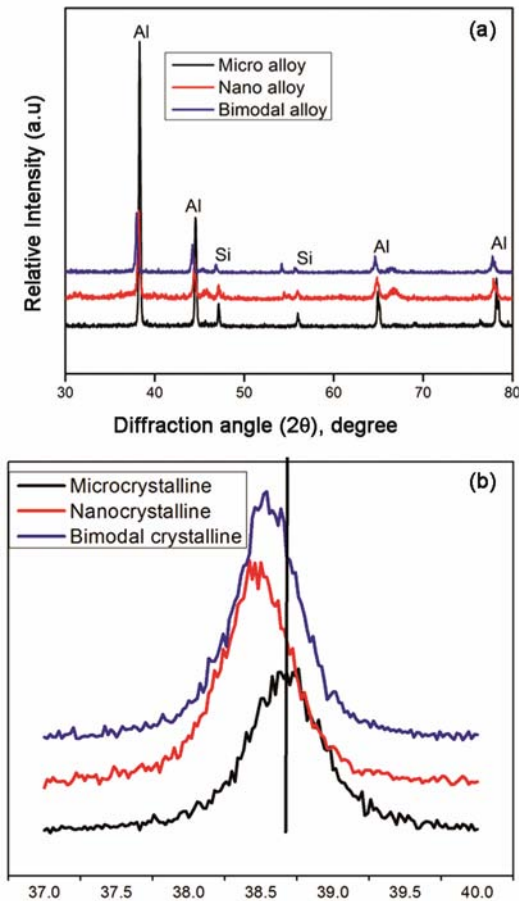


Fig. 1 –(a) XRD pattern of micro, nano and bimodal alloys and (b) peak shift of 3 modal alloys

$$\beta_{hkl} \cos \theta = \frac{k \lambda}{t} + \left(4 \sin \theta \left(\frac{2u}{E_{hkl}} \right)^{1/2} \right)$$

where β is the full width half maximum (FWHM), k is the shape factor; λ is the wavelength of Cu-K α radiation; θ is the diffraction angle E_{hkl} is the Young's modulus in the direction perpendicular to (hkl) plane and u is energy density (energy per unit volume).

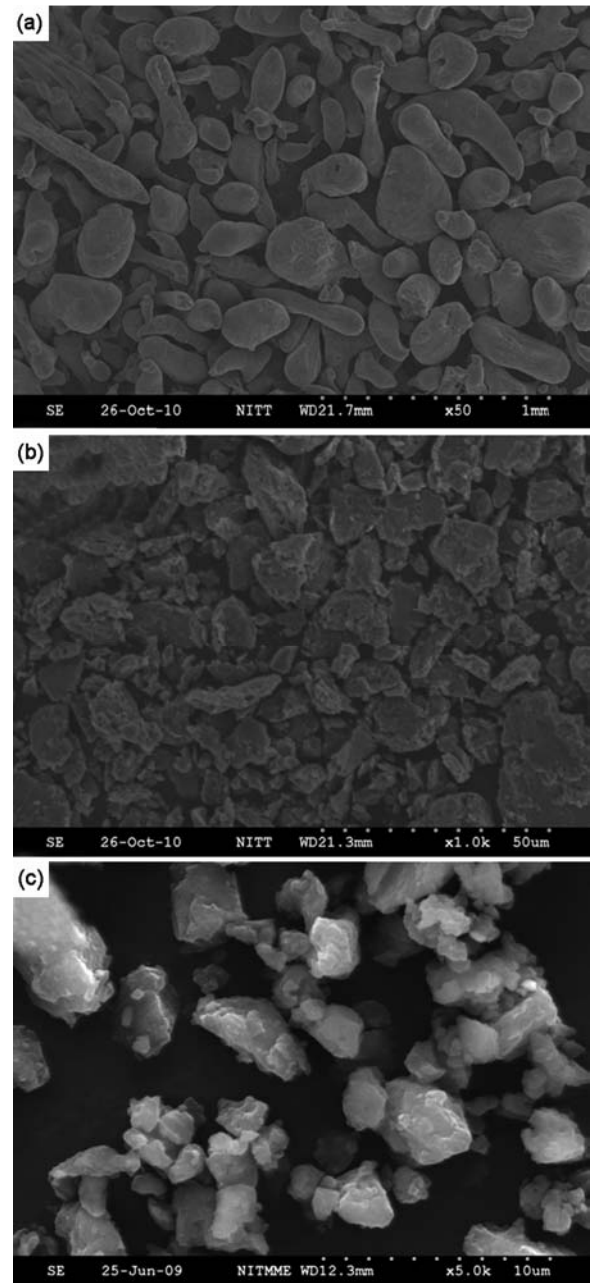


Fig. 2 – SEM micrographs of (a) unmilled powders, (b) bimodal distributed powders and (c) nanocrystalline powders

Microstructural analysis

The morphology of the microcrystalline powders, bimodal grain sized powders and nanocrystalline powders are shown in Fig. 2. The initial powders had uniform shape and size without any defects whereas the nanocrystalline powders shows a changes in their morphology. Mechanical alloying imparts a heavy structural disintegration followed with cold welding results in accumulated strain with reduction in crystals size⁹. The bimodal structure of AA 4032 powders was shown in Fig. 2b and there exists a uniform distribution of a small amount of unmilled powders with nanocrystalline materials.

The TEM microstructure of nanocrystalline AA 4032 powders SAED pattern is shown in Fig. 3. It is evidenced that alloy powders are in nanocrystalline in nature and most of the elemental powders were diffused into the Al matrix forming a solid solution.

The diffusion of Si atoms in aluminum matrix is proved by the lattice parameter variation of ball milled powders. Table 1 shows the variation in lattice parameter of AA 4032 powders during milling. The lattice parameter of Al does not change considerably in the initial milling hours. At the end of 5th hour milling, the lattice parameter decreases which is due

to the dissolution of Si, Cu and Ni atoms into the aluminium matrix during ball milling. The atomic radii of Si, Cu and Ni are 1.17, 1.28 and 1.25 Å respectively, which are smaller than aluminum (1.43 Å). Thus the lattice parameter starts to decrease and form substitutional solid solution. It is interesting to note that the lattice parameter increases between the 20th and 25th milling hour which may be due to the dissolution of magnesium present in alloy composition. The atomic radius of Mg (1.6 Å) is greater than aluminum which attributed to the increase in lattice parameter value. After 25 h of milling, almost all the elemental powders were dissolved into the Al matrix.

The microstructure of bimodal grain size structured compact is shown in Fig. 4. The optical microstructure, the SEM and the TEM are shown in Figs 4a, 4b and 4c, respectively.

The chemically etched sample of bimodal alloy is shown in Fig 4a. The microstructure clearly indicates the presence of coarse grains. The same is resulted in both SEM and TEM microstructure.

Mechanical properties

Figure 5a illustrates the variation in hardness with weight fraction of micro grains from microindentation test. There is decrease in hardness with increasing weight fraction of micrograins. In the specimen with weight fraction of micro grains, there is significant variation in hardness. Thus, the bimodal grain structure samples have possibility of nano and micro grains to deform at different stresses. The bimodal structure samples exhibits an unusual deformation which is similar to ductile toughening in brittle materials^{10,11}.

The decrease in compressive strength with increasing the fraction of micro sized grains is well known because coarse grained material is softer than nanostructured materials. The compressive deformation strength of bimodal structured AA 4032 compared with nanocrystalline AA 4032 is shown in Fig. 5b. The ductility of the sample is improved due

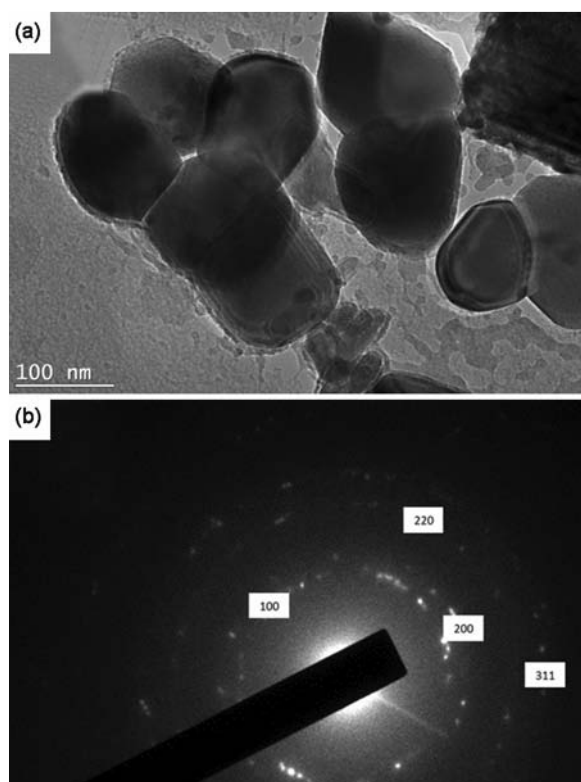


Fig. 3 – (a) TEM microstructure of nanocrystalline AA 4032 alloy and (b) SAED pattern

Table 1 – Lattice parameter variation during ball milling

Sl. No	Milling time	Lattice parameter (Å)
1	0 h	4.0507
2	5 h	4.0507
3	10 h	4.0467
4	15 h	4.0467
5	20 h	4.0467
6	25 h	4.0549
7	30 h	4.02268

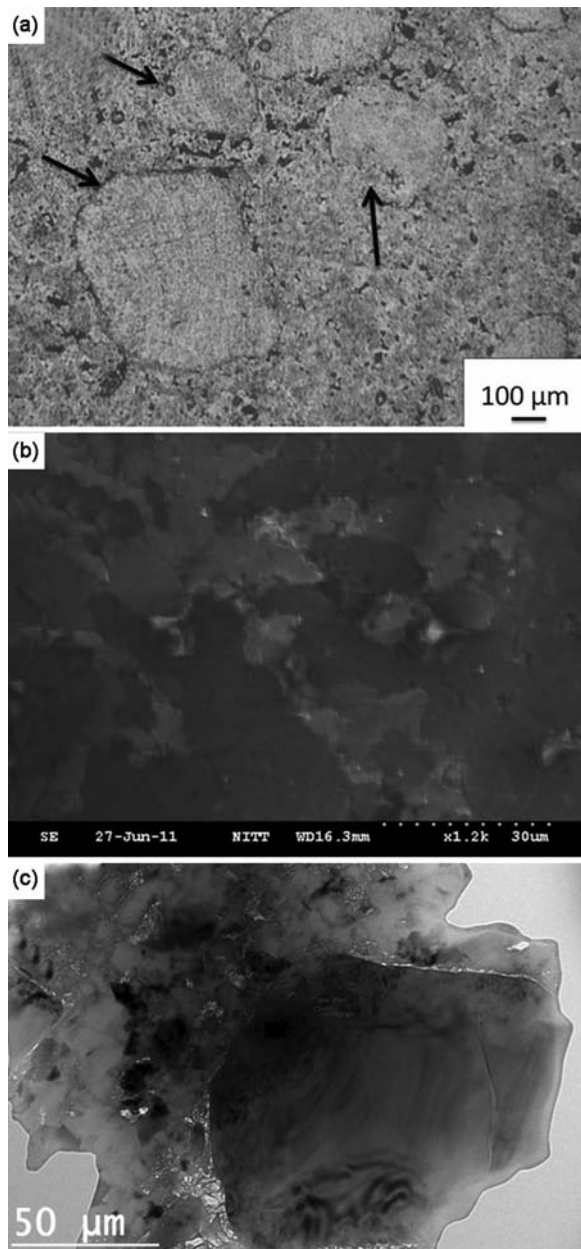


Fig. 4 – Microstructure of bimodal alloy (a) optical microstructure, (b) SEM and (c) TEM

to elastic-perfectly plastic behavior after yielding. This anisotropy nature in ductility of bimodal structure is increase with increasing the fraction of coarse grain¹²⁻¹⁵. The failure of bimodal structured sample is attributed to following reasons:

- (i) work hardening of nano grains
- (ii) structural defects in the interface of nano grains and micro grains
- (iii) during compression, microcracks will form easily at the end of micro grains which cause a shear failure. It is proved that strength is decreased in

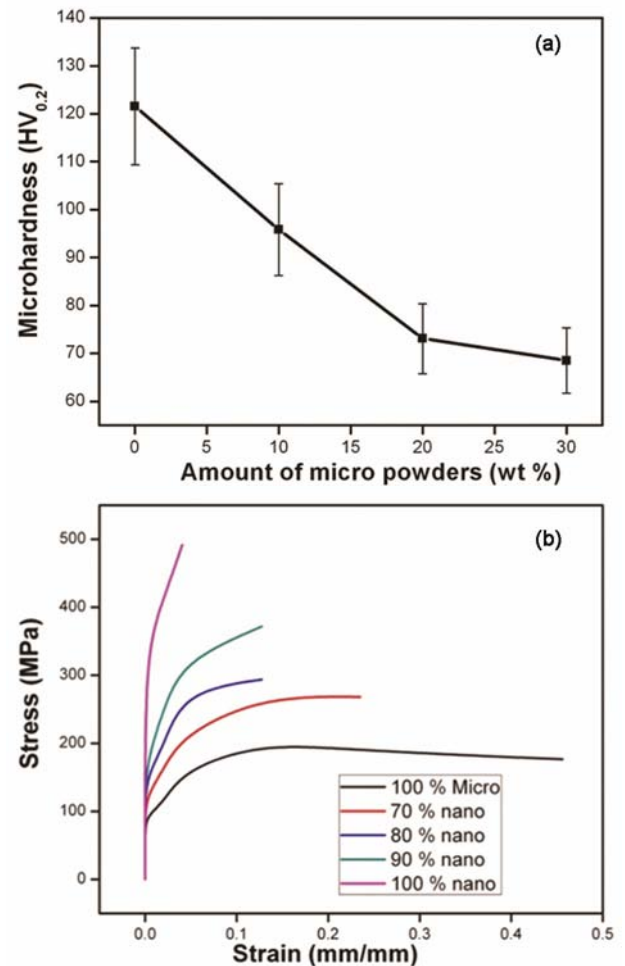


Fig. 5 – (a) Microhardness of bimodal alloys and (b) compression curves of nano, micro and bimodal alloys

bimodal structure with increase in the coarse grain content. But the ductile property is restored quantitatively than nanocrystalline AA 4032. The increase in the ductility is attributed to the plastic deformation of the coarse grained structure¹⁶. The enhanced strength of mechanically alloyed AA 4032 alloy is due to the grain refinement, solid solution strengthening by dissolution of Si atoms into the Al matrix. The fracture of the alloy was attributed to shear localization and a combined effect of shear localization and cavitations.

Conclusions

Bimodal structures of AA 4032 alloy composed of nanocrystalline grains and coarse grains were produced by consolidation of ball milled powders resulting in ductile toughening. The bimodal structured AA 4032 alloy showed balanced mechanical properties with better yield and ultimate

strength, and improved ductility compared with both microcrystalline and nanocrystalline AA 4032 alloys.

The present work resulted in the new method for processing of bulk nanocrystalline materials with improved strength and ductility. Difficulty in blending different grain size powders is overcome by the present technique. Using the present technique, fabrication of single phase microstructure with dual grain size can be done to achieve unique combinations of properties.

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