Journal of Alloys and Compounds 507 (2010) 236–244

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

journal homepage: www.elsevier.com/locate/jallcom

Effect of strengthening mechanisms on cold workability and instantaneous strain hardening behavior during grain refinement of AA 6061-10 wt.% TiO_2 composite prepared by mechanical alloying

S. Sivasankaran[a], K. Sivaprasad[b], R. Narayanasamy[a,*], Vijay Kumar Iyer[c]

[a] Department of Production Engineering, National Institute of Technology, Tanjore Main Road, Tiruchirappalli 620015, Tamil Nadu, India
[b] Department of Metallurgical and Materials Engineering, National Institute of Technology, Tiruchirappalli 620015, India
[c] Powder Metallurgy Shop, Heavy Alloy Penetrator Project, Tiruchirappalli 620025, India

ARTICLE INFO

Article history:
Received 2 July 2010
Accepted 24 July 2010
Available online 4 August 2010

Keywords:
Mechanical alloying
Cold workability
Strengthening mechanism
Strain hardening

ABSTRACT

The mechanical alloying (MA) of AA 6061 alloy reinforced with 10 wt.% fine anatase-titania composites powder milled with different timings (1, 5, 10, 20, 30, and 40 h) was cold consolidated and sintered. The main purpose of this study is to investigate the effect of microstructure and the various strengthening mechanisms such as solid solution, grain size, precipitate, dislocation and dispersion strengthening during grain refinement of AA 6061-10 wt.% TiO_2 composite via MA on cold working and strain hardening behavior. The sintered composite preforms were characterized by X-ray diffraction, scanning electron microscope, and transmission electron microscope. The strengthening mechanisms were estimated by using simplified models available in the literatures. The evaluation of cold deformation behavior under triaxial stress condition through room temperature cold-upsetting tests (incremental loads) was studied by correlating the strengthening mechanisms. Among the developed strengthening mechanisms the grain size and dislocation strengthening mechanisms diminished the deformation capacity of the composites. The strain hardening behavior was also examined by proposing instantaneous strain hardening index (n_i). The value of maximum instantaneous strain hardening index for ultrafine-grained composite was to be about twice that of coarse-grained composite at lower true axial strain value. Also, the coarse grained 5 h composite was observed to be the best one as it exhibited a better strain hardening or deformation behavior.

1. Introduction

Ultrafine-grained (UFG), fine-grained (FG) and nanocrystalline (NC) materials are increasingly being studied owing to their improved mechanical properties. Bulk UFG or NC metals, alloys and composites are commonly produced by high-energy ball milling [1], equal channel angular pressing [2], and high pressure torsion [3]; which are under the category of severe plastic deformation (SPD). During SPD, very large plastic strains have been introduced which led to grain refinement in coarse-grained (CG) powder. Grain refinement plays a vital role in Al-alloy based composites. Apart from cast Al-alloy based composites; grain refinement has several benefits via mechanical alloying (MA) followed consolidation by conventional powder metallurgy (P/M) process; which improves the mechanical properties, distribution of second phase particles, better strength and fatigue life.

Structural applications of Al-alloy based composites at high or moderate temperatures require a fine, homogeneous and stable distribution of particles to guarantee the dispersion hardening up to the temperature of use. Although particle dispersion can be obtained by conventional ingot metallurgy, problems as formation of coarse intermetallic particles and segregation during the ingot solidification usually takes place [4,5]. These problems can be overcome by using P/M and more specifically MA as processing route [3,6–8]. MA is a solid-state processing that enables refinement of the microstructure, homogenization and extension of solid solubility limits [9–11]. The possibility of higher solubility could be interesting to enhance precipitation of equilibrium phases in the heat treatable Al-alloys via MA. The highest strength Al-alloys with applications in the aircraft and automated industry are those of 6XXX series; which have a good formability and heat treatable alloy. Also, to facilitate the prevention of reinforcement clusters or agglomerates on the matrix, especially in the case of small size reinforcement particles, MA is one of the P/M processes that produces uniform dispersion of the reinforcement particles in the matrix.

---

* Corresponding author. Tel.: +91 431 250 3504; fax: +91 431 250 0133.
E-mail addresses: sivasankarangs1979@gmail.com (S. Sivasankaran), ksp@nitt.edu (K. Sivaprasad), narayan@nitt.edu, narayan10455@yahoo.co.in (R. Narayanasamy), yajiv29@yahoo.com (V.K. Iyer).

0925-8388/$ – see front matter © 2010 Elsevier B.V. All rights reserved.
doi:10.1016/j.jallcom.2010.07.168

Generally, the reinforcement clusters on the matrix has occurred when the particle size of matrix-to-reinforcement ratio exceeds one, which deteriorates the mechanical properties. This problem is encountered in conventional P/M process [12], however, this can be avoided in MA [13,14].

Numerous investigations have been carried out regarding strain hardening behavior through tensile tests during grain refinement [15–17]. In fact, the uniaxial tensile test would not be able to sustain a uniform tensile deformation at ambient temperature for more than a couple of percent of plastic strain especially in refined grain materials. Hence, compression tests (here, cold-upsetting) are needed to provide a direct evaluation of the strain hardening response as a function of true strain because the compressive behavior is not strongly influenced by superfluous factors such as surface or internal blemish [18]. Further, limited studies are reported on the strain hardening behavior of materials through compression tests [19–21]. But, there is no work on cold workability and strain hardening behavior under cold-upsetting (triaxial stress condition) of AA 6061-10 wt.% TiO_2 composite during grain refinement processed by MA.

Workability is a term used to evaluate the capacity of a material to withstand the induced internal stresses of forming prior to the splitting of material occurs. The cold workability and the strain hardening behavior of porous P/M composites under uniaxial, plane and triaxial conditions were elaborately analysed and discussed in the previous works by Narayanasamy et al. [22–25]. Their results have shown that the workability behavior of metals/alloys/composites of P/M components depends on the aspect ratio, the preform geometry, the particle size and the percentage of reinforcement for the composites, the die geometry, the lubricants, and the compacting load. However, this is not the aim of the present work, instead, to study the effect of grain refinement on the cold workability and its strain hardening behavior of AA 6061-10 wt.% TiO_2 composite via MA. According to the findings of Narayanasamy et al. [22–25], porous P/M material during cold working not only experiences the usual strain hardening but also experiences 'geometrical work-hardening (GWH)' (due to a continued increase in density which leads to the enhancement in area of the cross-section) and 'matrix work-hardening (MWH)' (due to continuous increase in strength of the matrix with increase in strain). In general, GWH mainly depends on stress required for closing of pores during compressive plastic deformation [22–25].

As reported in previous work [13], a commercial AA 6061 Al-alloy based composite was produced by MA with titania addition. The main objective was to produce a dispersion-hardened alloy by TiO_2 particles [13]. The precursor powders produced by MA were cold compacted and then sintered in different temperatures. The consolidated samples in the as-sintered condition were characterized. Results showed that MA increased the mechanical properties of the AA 6061 Al-alloys and the respective composites when compared to the commercial one. This was attributed to contributions of various strengthening mechanisms resulting from the milling such as grain size refinement to nanometric scale and increase in dislocation density due to severe deformation caused by the MA process. The strengthening mechanism was not carried out in the previous work. Hence, the main objectives of the present work are threefold. Firstly to investigate the dominant strengthening mechanisms and suggest a strength model for AA 6061 reinforced with 10 wt.% TiO_2 (higher percentage, ≈45 matrix-to-reinforcement particle size ratio) particles with different milling time in as-sintered condition. The strengthening mechanism includes the solid solution, dispersoid, precipitate, forest dislocation and grain size strengthening. Secondly to study the effect of strengthening mechanisms on the cold workability during grain refinement and at last its strain hardening behavior by proposing instantaneous strain hardening index (n_i) under triaxial stress state condition.

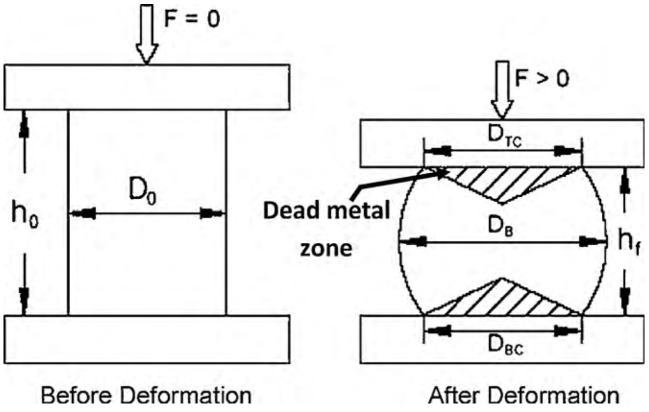

Fig. 1. The upset forging test-before and after deformation.

2. Experimental details

As reported elsewhere [13], powder of an AA 6061 based alloy with composition 99.68Al–0.68Si–0.7Fe–0.275Cu–0.15Mn–1Mg–0.195Cr–0.25Zn–0.15Ti (wt.%) with addition of 10 wt.% TiO_2 (anatase form with tetragonal structure, <1 μm average particle size, 3.84 g/cm^3 density) was produced by MA using pure elemental powders as starting materials. MA was carried out using a planetary ball mill (Insmart systems, Hyderabad, India) in toluene at 280 rpm and a ball-to-powder ratio of 10:1 using a hardened stainless steel media in different milling times, 1, 5, 10, 20, 30, and 40 h. Further details of milling, powder morphology and structural evaluation of the milled powders were reported in [13,14]. The precursor powders obtained by MA were consolidated with diameter 30 mm by cold compaction at 350 MPa (machined to get aspect ratio 0.375 related to P/M components) followed degassing at 623 K (45 min) and then sintering (N_2 atmosphere with mass flow rate of 6 m^3 per hour) at 848 K for 90 min in a mechanical pusher furnace as applicable to P/M industries. Phase identification and structural properties of the sintered composites were characterized by X-ray diffraction (XRD) using Cu-Kα radiation (1.5406 Å) in a D/MAX ULTIMA III diffractometer (Rigaku Corporation, Japan) operating at 30 mA and 40 kV. From the XRD peak profile, instrumental broadening and $K_{\alpha2}$ components were subtracted and then the crystallite size was estimated using Williamson and Hall method [26]. Lattice parameter of the matrix was calculated from the first five peaks using Cullity method [27].

The microstructure of the as-sintered composites was examined by HITACHI S 3000H scanning electron microscope with back scattered electron image (SEM/BSEI) and PHILIPS CM12 transmission electron microscope (TEM). Hardness was measured using PC based Ratnakar Vickers tester at a load of 1 kg. Averages of at least 15 measurements were taken for each hardness value. The resulting sintered composites were subjected to incremental compressive load (cold-upsetting) of 5 kN between two flat mirror finished open dies on a hydraulic press (100 tones capacity). The initial diameter (D_0), height (h_0) and density (ρ_0) were measured and recorded. The deformation was carried out until the appearance of the first visible crack on the free surface. After each interval of loading, dimensional changes in the specimen such as height after deformation (h_f), top contact diameter (D_{TC}), bottom contact diameter (D_{BC}), bulged diameter (D_B) and density of the preform (ρ_f) were measured. At least five readings were taken and the average was used for investigation. The schematic diagram showing the various parameters measured before and after deformation is provided in Fig. 1. The density (average of five readings) of cold-upset preforms after every incremental loading was measured using Archimedes principle.

3. Results and discussion

3.1. X-ray diffraction and TEM analysis

Fig. 2 shows the XRD patterns of AA 6061-10 wt.% TiO_2 composite to different milling times after sintering. The sample consists of mainly α-Al, TiO_2 and Al_2O_3 phases. The observed Al_2O_3 phase as a precipitate was expected to form due to the oxidation of matrix of α-Al phase during sintering; which was of unknown type as given by the JCPDS data base (31-0026). Though the samples were sintered at N_2 atmosphere in mechanical pusher furnace, the presence