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Processing and characterization of Al-Al_3Nb prepared by mechanical alloying and equal channel angular pressing

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Abstract. Mechanically alloyed Al with immiscible elements such as Nb can lead to a uniform distribution of nanoscaled precipitates which are highly stable compared to conventional alloying and with excellent interface, resulting in significant increase in strength without problems associated with nano ceramic particles in metal matrix composites. Although immiscible, Nb can be alloyed with Al through mechanical milling, forming trialuminide (Al_3Nb), either directly or upon subsequent precipitation, which possesses high strength, stiffness and stability at elevated temperatures. In the present study, Al-5 at.% Nb supersaturated solid solution was achieved after prolonged ball milling and nano Al_3Nb precipitates were formed during subsequent ageing at 530°C. The Al-Al_3Nb powder was consolidated by equal channel angular pressing (ECAP) at 400°C, resulting in a fully dense material with a uniform distribution of nanoscaled Al_3Nb precipitates in the Al matrix.

1. Introduction

Al-Nb is a promising system for high strength aluminium alloys thanks to the possible formation of hard Al_3Nb intermetallic precipitates. Al_3Nb possesses high strength and stiffness, and excellent mechanical properties at elevated temperatures [1, 2]. However, the equilibrium solubility of Nb in Al is zero at room temperature and very low (~0.065 at.%) at the maximum solubility temperature [3], making it impossible to follow the conventional solution and aging treatment to achieve precipitation strengthening. High energy mechanical alloying as a non-equilibrium processing method has been successfully employed to extend the solubility well above the equilibrium values in several Al binary systems [4-6]. One study on mechanical alloying of Al and Nb has shown that a maximum of 15 at.% Nb can be dissolved in Al [1]. Depending on the composition and milling conditions, different structures including solid solution, intermetallic or amorphous can be obtained in the Al-Nb system [1, 2, 7]. In addition, mechanical milling leads to the formation of nanostructures with improved mechanical properties [8].

The objectives of the present study are to first achieve a supersaturated solid solution of Nb in Al by mechanical alloying and to bring about nano precipitates by ageing, and then to consolidate the powder into bulk material by a severe plastic deformation (SPD) method.

The SPD processes such as equal channel angular pressing (ECAP) and high pressure torsion (HPT) have been developed to consolidate particulate materials to form bulk nanostructured materials [9, 10]. Deformation of the particles during SPD, rather than diffusion, causes their bonding [11]. a number of ultrafine and nanostructured bulk materials have been prepared by SPD consolidation

including several based on Al and Ti, leading to significantly improved mechanical properties [9, 12]. Unlike the conventional sintering process at high temperatures, which tends to destroy the nanostructures achieved by milling, ECAP enables the particles to consolidate at much lower temperatures with little porosity [13]. The preliminary results from the present investigation into the Al-Nb system demonstrated that it is possible to achieve high solubility by mechanical milling and high strength by subsequent ageing and ECAP consolidation.

2. Experiments

Elemental powders of Al (-325 mesh, 99.8% purity) and Nb (-325 mesh, 99.8% purity) were used. The powder mixture of Al-5 at.% Nb in composition was placed in a stainless steel jar filled with stainless steel balls of 10 mm in diameter with a ball to powder ratio (BPR) of 20:1. A process control agent (PCA) of stearic acid (1 wt.%) was added. High energy ball milling was performed at 400 rpm under vacuum at room temperature in a planetary mill until complete solid solution of Al-Nb was achieved. The solid solution powder was annealed at 530°C for an hour in vacuum to cause precipitation. The annealed powder was then encapsulated in a thin copper can and placed in an ECAP die with channels of 12 mm in diameter intersecting at 90°. The consolidation was carried out at 400 °C for 4 passes following Route C with a back pressure of 50 MPa. XRD (Cu Kα) was carried out to identify the phases in the powder after different milling durations. The microstructure was observed by SEM on polished surfaces and TEM on samples prepared using focused ion beam (FIB). Compression tests were performed on cylindrical samples of dimensions of 6 mm in diameter and 9 mm in height at a strain rate of 0.001/s.

3. Results and Discussion

The XRD patterns of the Al-5 at.% Nb powder after milling for 1, 45 and 90 hours are shown in figure 1, revealing the phase transformation, and the corresponding back scattered SEM images in figure 2, displaying the morphological evolution of the particles. It is clear from the XRD patterns that the peaks of the elemental Al and Nb still existed after one hour of milling, and the peaks of Nb disappeared after 45 hours, indicating the apparent formation of complete solid solution. There seems to be no further phase changes during further milling and only fcc Al peaks were observed after 90 hours of milling. The broadening of the fcc Al peaks was observed with increasing milling time as a result of the refinement of the crystallite size by heavy plastic deformation [14]. In fact, the starting grain size of ~1.3 μm in the elemental Al powder was reduced to ~23 nm after 90 hours. The Al (grey) and Nb (white) particles can be clearly distinguished after 1 hour (figure 2a), although some Al particles were elongated. With increasing milling time, the Nb particles were severely fragmented to become smaller particles and be entrapped and cold welded in the Al particles. Despite the disappearance of the Nb peaks in the XRD pattern after 45 hours, closer inspection of the corresponding SEM image (figure 2c) reveals ultrafine Nb particles of < 1 μm embedded uniformly in the Al matrix (the amount is too small for XRD to pick up). In contrast, no Nb particles were detected by the SEM images for the powder milled for 90 hours (figures 2d and e), indicating the true formation of a complete solid solution between Al and Nb. The average particle size was ~21 μm after 90 hours. The particle morphology remained nearly equiaxed with irregular boundaries after 45 and 90 hours. EDS analysis on several particles after 90 hours confirmed that they all have the same composition as the overall composition (5 at.% Nb). As expected, the lattice parameters remained constant at all the times thanks to similar atomic radii of Al (0.1430 nm) and Nb (0.1432 nm) [1].

Figures 3 a and b show the bright field TEM image after 90 hours and the corresponding selected area electron diffraction (SAED) pattern, respectively. The SAED pattern identifies fcc Al rings only, i.e. the formation of the complete solid solution, and the complete rings confirm that the prolonged milling had resulted in the formation of a nanocrystalline structure. During the early stages of milling the Al and Nb particles are subjected to high energy collisions from the impacting balls. As milling continues, fracturing and cold welding occur repeatedly, leading to severe plastic deformation. As a result, a large number of crystal defects are introduced and the amount of grain boundaries

significantly increases, leading to rapid diffusion for Nb and the eventual formation of a uniform solid solution of Al-Nb [3, 15].

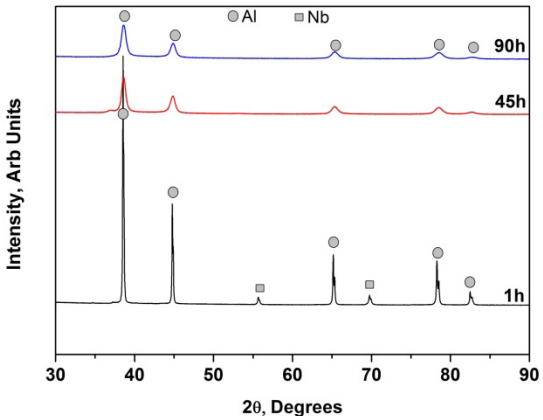

Figure 1. XRD patterns of Al-5 at.% Nb after milling for 1, 45 and 90 hours.

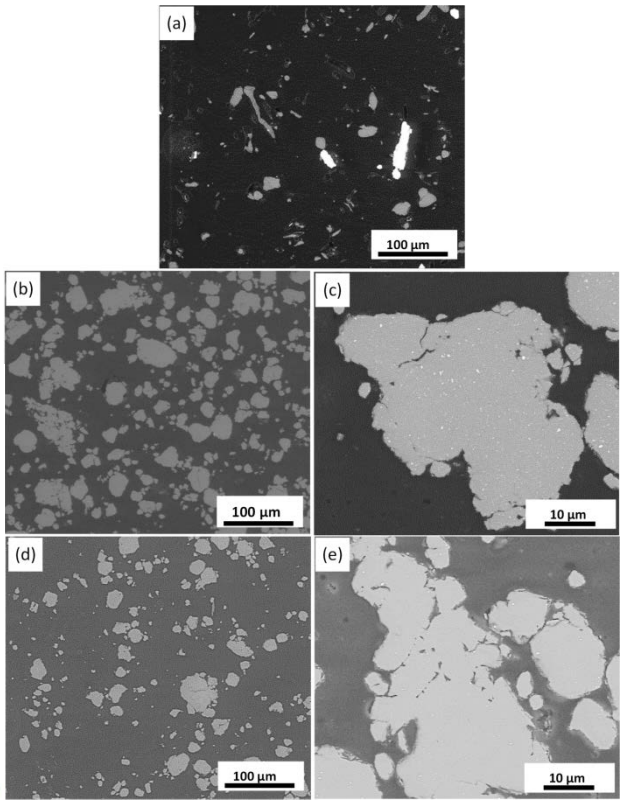

Figure 2. Backscattered SEM micrographs showing Al-5 at.% Nb particles created after milling for (a) 1 hour, (b) and (c) 45 hours, (d) and (e) 90 hours at two different magnifications, respectively.

Following mechanical milling, the solid solution powder was annealed at 530°C for one hour in vacuum to bring about the Al_3Nb precipitates. The XRD pattern of the annealed powder is shown in figure 4, confirming the formation of ~20 at.% of Al_3Nb which is the maximum amount that can result from an Al-5 at.% Nb supersaturated solid solution. It is also observed that the fcc Al peaks has become shaper compared to the solid solution peaks before annealing, indicating coarsening of the crystallites. In fact, the Al grains are now ~46 nm in size based on the peak broadening measurement, still in the nanoscale. On the other hand, the precipitates are measured to be ~29 nm in size. No other intermetallic phases are detected after the annealing process.

After ECAP consolidation of the annealed powder, a fully dense bulk material within ±0.1% of the theoretical density (3.05 g/cm^3) was obtained. The application of a back pressure ensures the shearing of the particles during ECAP, resulting in well bonded particles with no pores [12, 16]. The cross-sectional SEM microstructures (low and high magnifications) of the consolidated material are displayed in figures 5 a and b with two distinct regions, the lighter grey and the darker region between. The grey region is Al with numerous nano Al_3Nb precipitates as revealed by TEM, from the framed area in figure 5 b, in figures 5 c and d, and the darker region consist of mostly Al with a very

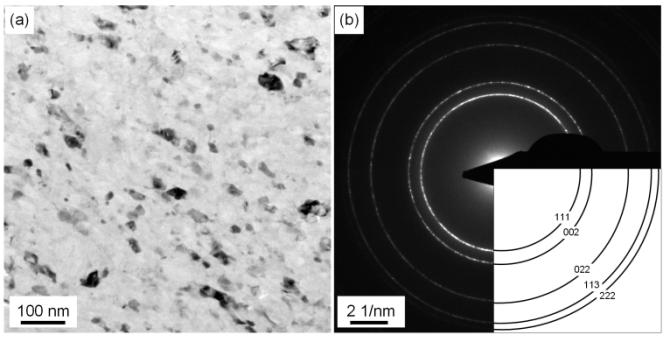

Figure 3. (a) Bright field TEM image of the Al-5 at.% Nb powder after 90 hours of milling and (b) corresponding SAED ring pattern, identifying nanocrystalline fcc structured Al.

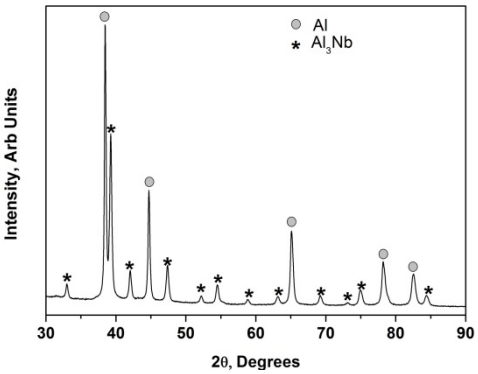

Figure 4. XRD pattern of the annealed Al-5 at.% Nb powder at 530°C for 1 hour.

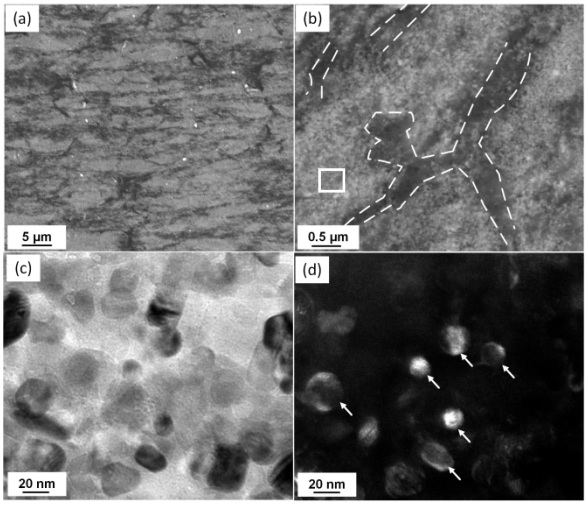

Figure 5. (a) and (b) Backscattered SEM microstructure of Al-Al_3Nb consolidated by ECAP at two magnifications, showing some pure Al areas delineated by dashed-lines. (c) Bright field TEM image from the boxed area in (b), and (d) corresponding dark field TEM image for Al_3Nb, showing uniformly distributed nano Al_3Nb precipitates (arrowed).

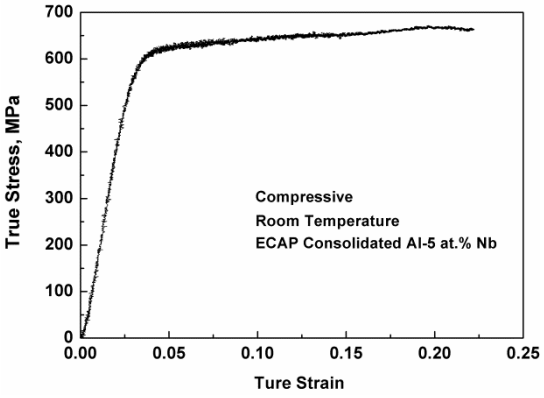

Figure 6. Compressive true stress-true strain curve for the ECAP consolidated Al-5 at.% Nb.

small number of precipitates. Image analysis indicates that the darker region accounts for an area fraction of ~12%.

Closer inspection of figures 5 c and d indicates that the Al_3Nb precipitates have an average size of ~30 nm and they are uniformly dispersed in the Al matrix. The identity of these round particles is also confirmed by EDS, giving the same composition as Al_3Nb.

No other intermetallic phases of Al and Nb are detected after the ECAP consolidation. Although the powder after milling and that after annealing appear to possess a uniform microstructure and

composition, the ECAP consolidation has resulted in the formation of the thin band with relatively few precipitates. Further investigation is in progress to understand this phenomenon.

The compressive stress-strain curve of the consolidated material is shown in figure 6, with an average 0.2% proof stress of ~590 MPa, an ultimate compressive strength of ~670 MPa and a good plasticity of ~20%. The strength is comparable to or higher than conventional high strength Al alloys, and is attributed to several microstructural factors. The presence of nano precipitates provides dispersion strengthening. The nanocrystalline structure retained after the ECAP consolidation also results in grain boundary strengthening, as reported in other Al and Ti based alloys [12]. The unique intermixed microstructure and good bonding between the particles might be responsible for the good plasticity in compression. As reported in another SPD consolidated material [12], there is a small XRD peak shift towards lower angles after annealed and ECAP, indicating the internal strain energy relief [17].

4. Conclusions

(1) Supersaturated solid solution of Al-5 at.% Nb was achieved after 90 hours of ball milling at 400 rpm.
(2) All possible Al_3Nb was precipitated after annealing the milled particles at 530°C for one hour.
(3) The Al-Al_3Nb consolidated by ECAP was fully dense with regions of uniform dispersion of nano precipitates in Al, intertwined by nearly pure Al layers.
(4) The consolidated material showed excellent yield strength of 590 MPa with a good plasticity (~20%) in compression.

5. References

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