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Thermal stability and mechanical properties of mechanically alloyed Al–10Ti alloy

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The mechanical properties of Al–10Ti alloy prepared by mechanical alloying and subsequent hot hydrostatic extrusion were evaluated at room and elevated temperatures. Transmission electron microscopy was used to characterize the microstructural changes of this alloy on heat treatment at 500 °C for various times. The results show that the mechanically alloyed Al–10Ti has high strength and high thermal stability at elevated temperature. The strength and stability of this alloy are attributed to its fine grain size and to the high volume fraction of small Al_3Ti intermetallic compounds dispersed in the aluminium matrix. After 50 h annealing at 500 °C, no serious coarsening of either the Al_3Ti dispersoids or the grains was observed.

1. Introduction

Light weight materials that possess good strength at elevated temperature are needed for high temperature engineering applications. Mechanical alloying processes offer unique advantages in producing high strength aluminium alloys for elevated temperature structural components. Previous studies have shown that mechanically alloyed Al–Ti, containing a high volume fraction of Al_3Ti dispersoids, possesses good stability of mechanical properties up to 500 °C [1, 2], and is considered as a challenging material for titanium base alloys [3]. The improved elevated temperature strength and stability of this alloy are attributed to the slower coarsening rate of the Al_3Ti particles. Additionally, the high volume fraction of Al_3Ti particles may inhibit to a certain extent grain growth, and thus limit losses in strength due to long term, high temperature annealing heat treatments. As such, this study was undertaken in order to compare mechanical and microstructural changes that occur during high temperature static annealing heat treatments.

2. Experimental procedure

Aluminium powders (99% pure, − 80 mesh) and titanium powders (98.5% pure, − 200 mesh) were mixed in the composition of Al–10 wt % Ti, and mechanically alloyed in an attritor ball mill under the protection of pure argon gas using hardened steel balls and vial. 0.5 wt % stearic acid was added as a process control agent. Chemical analyses show that 0.18 wt % Fe was introduced due to debris wear from the milling medium after 43 h milling. Milled powders were hot pressed to cylindrical billets with 98% theoretical density at 400 °C and 500 MPa in a vacuum of better than 4×10^{-2} Pa, and then hot hydrostatically extruded to rods at 400 °C with an extrusion ratio of 14:1. The preheating time was 10 min before extrusion, and the extruded rods were cooled to room temperature in air. Thermal exposure experiments were conducted at 500 °C for various times. The mechanical property measurements were performed on an Instron universal testing machine, the direction of loading was parallel to the extrusion direction, and the initial strain rate was $2 \times 10^{-4} s^{-1}$.

The microstructure of the consolidated materials was examined by transmission electron microscopy (TEM, Philips EM420). Thin foils for TEM observation were prepared by twin jet electropolishing with an electrolyte of 1:3 nitric acid:methanol at − 30 °C.

The average precipitate size was determined from the TEM images. Random lines were drawn across the micrographs and the diameters of the precipitates intersecting these lines were measured. At least 100 different measurements were made to determine the average precipitate size.

3. Results and discussion

Fig. 1 shows the mechanical properties of the Al–10Ti alloy consolidated from powders milled for 18 and 30 h. Evidently, the tensile strength decreases with increasing temperature, and the ductility varies slightly. Materials consolidated from powders milled for long times have high strength at elevated temperature and at room temperature. The 0.2% offset yield strength and ultimate tensile strength increase with increasing milling time; however, elongation decreases as shown in Fig. 2. The increase of tensile strength with milling time may result from grain size refinement. Micrographs of the materials consolidated from the powders milled for 18 and 43 h are shown in Fig. 3; the longer the powders were milled, the smaller the grain sizes of materials consolidated at the same condition. For materials with grain sizes greater than about 100 nm, dislocations are present. While for

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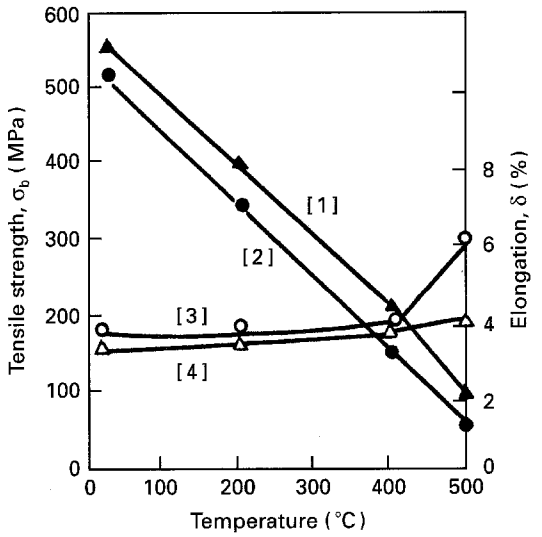


Figure 1 Tensile strength (●, ▲) and elongation (○, △) versus temperature for materials consolidated from milled powders for 18 [2, 3] and 30 h [1, 4].

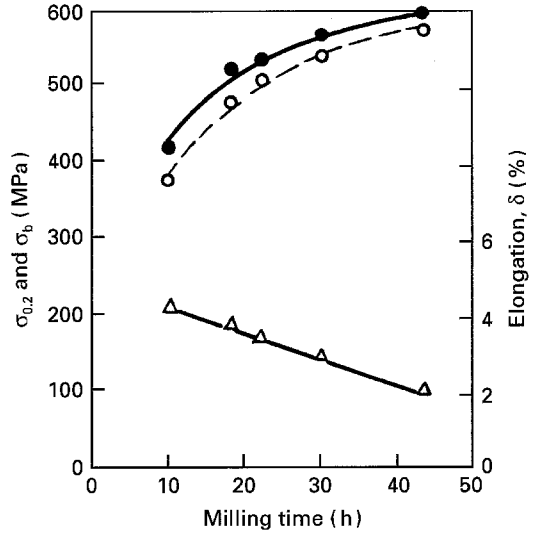


Figure 2 Tensile properties of extruded alloy as a function of milling time: (●) σ_b, (○) $\sigma_{0.2}$, (△) δ.

materials with grain sizes less than 100 nm there is no sign of dislocation within grains.

Fig. 4 shows the room temperature tensile strengths for samples annealed at 500 °C for various times. The tensile strengths drop greatly after short annealing times, and level off with continued increases of annealing times. The materials consolidated from powders milled for long times have higher thermal stability and tensile strength than those materials consolidated from powders milled for short times.

Examination of the microstructure of annealed alloys by TEM reveals that the grain size and dispersoid size increase with increasing annealing time. Fig. 5 shows that the Al_3Ti dispersoids inhibit grain growth. The variation of grain size with annealing time is shown in Fig. 6. The grain size coarsens obviously at the start of the annealing stage, this is the reason that the strength drops rapidly at the initial annealing stage.

Grain size may be expected to be related to the size and distribution of dispersoids which act to pin the

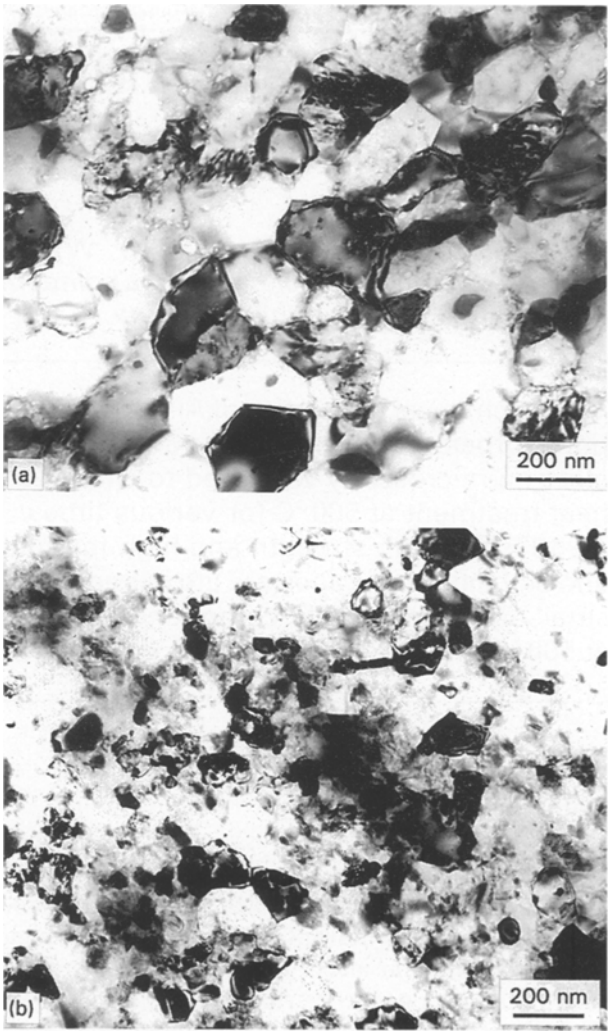


Figure 3 TEM structure of materials consolidated from milled powder for (a) 18 h, and (b) 43 h.

boundaries. An initial analysis, due to Zener [4] relates the grain size, d, to the dispersoid radius, r, and volume fraction, f, as

$$d = \frac{4r}{3f} \quad (1)$$

if all the Ti, C and O elements have transformed to the respective compounds, i.e. Al_3Ti, Al_4C_3 and Al_2O_3, the total volume fraction will be 24.1%. Providing $r = 10$ nm, $d = 55.3$ nm is obtained according to Equation 1, d is too small to fit the experimental results. The reason is that not all the dispersoids are boundary pinners.

The mean dispersoid sizes obtained in the present work are plotted against coarsening time; the cube of the mean dispersoid radius versus time gives the best apparent fit to a straight line, as shown in Fig. 7. This indicates that the mechanism for the Ostwald ripening of dispersoids is lattice diffusion-controlled, and follows the Lifshitz–Slyozov–Wagner (LSW) equation [5, 6]

$$r^3 - r_0^3 = Kt \quad (2)$$

where r_0 is the mean radius of dispersoids at time $t = 0$, and K is a material parameter.

From the above results, one could infer that the strengths of these alloys must be mainly attributed to

their fine grained nature. The increase in grain size is clearly related to the loss in 0.2% offset yield strength, as shown in Fig. 8a. The relationship of $\sigma_{0.2}$ with $d^{-1/2}$ follows the Hall–Petch equation for materials in as-extruded conditions, and can be written as the following linear equation

$$\sigma_{0.2} = 190 + 216.2d^{-1/2} \quad (3)$$

where the lattice frictional stress required to move individual dislocation, σ_0 is 190 MPa, and a positive

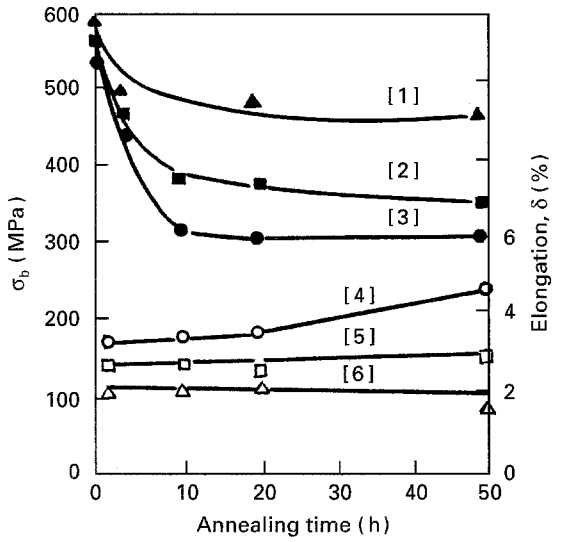


Figure 4 Tensile strength (▲, ■, ●) and elongation (△, □, ○) of the alloy consolidated from milled powders for 18 h [3, 4], 30 h [2, 5] and 43 h [1, 6] after annealing at 500 °C for various times.

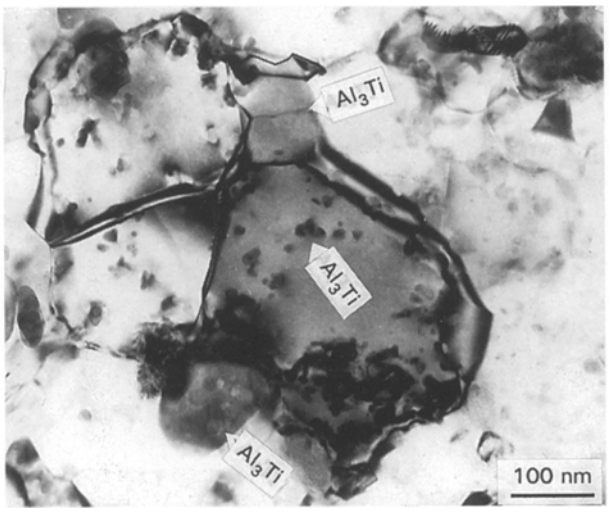


Figure 5 TEM structure of the alloy consolidated from 43 h milled powder after annealing at 500 °C for 50 h.

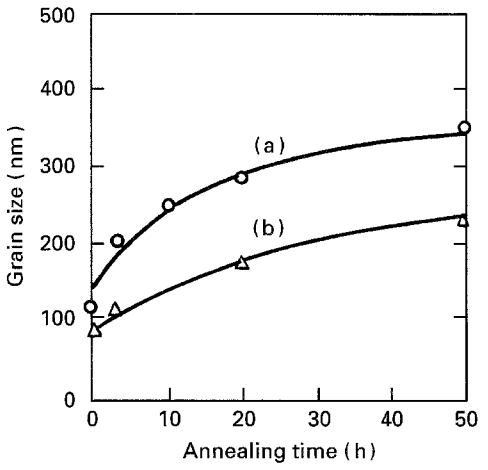


Figure 6 Variation of average grain size with annealing time for materials consolidated from milled powders for (a) 30 h, and (b) 43 h.

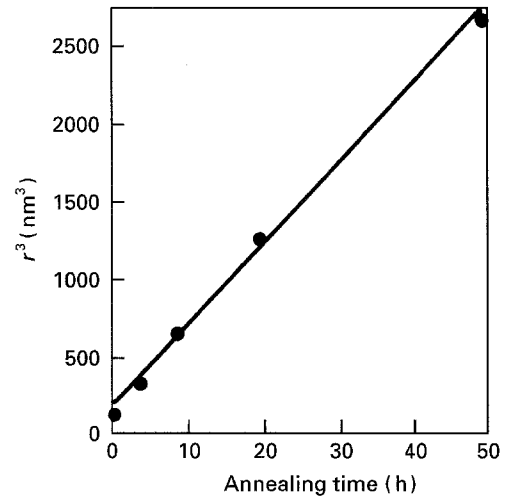
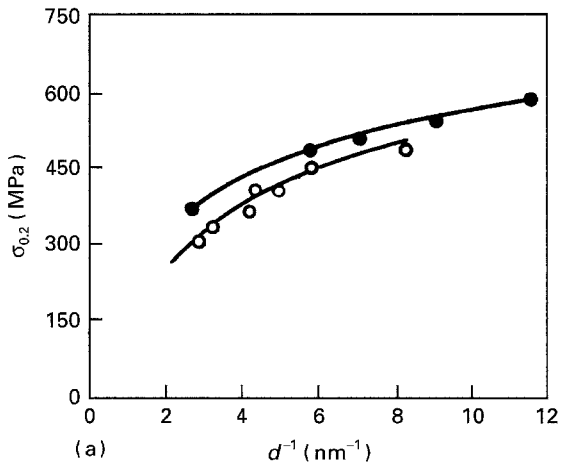


Figure 7 The cube of the mean dispersoid radius versus annealing time.



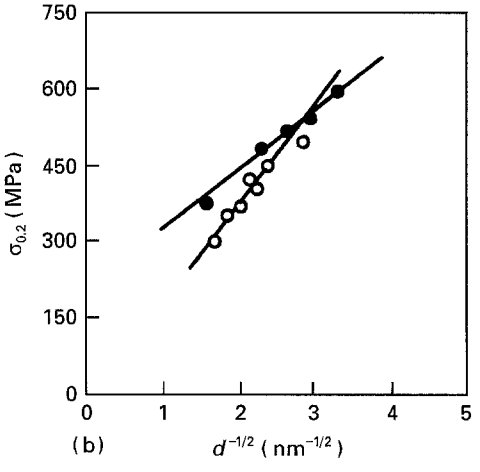


Figure 8 The 0.2% offset strength versus grain size (a) d^{-1}, and (b) $d^{-1/2}$: (●) as-extruded, (○) after annealing.

constant, $k = 216.2$ MPa $\mu m^{-1/2}$, was obtained, indicating that the yield strength increased rapidly with the reduction of grain size. For materials after heat treatment the intercept of the straight line of $\sigma_{0.2}$ versus $d^{-1/2}$ with the ordinate is negative, as shown in Fig. 8b, indicating that the Hall–Petch equation cannot be used.

As known, the Hall–Petch relation was established assuming that the grain boundary is the only obstacle for dislocation motion [7, 8]; the free path of dislocation movement is equal to the grain diameter. However, if there are many dispersoids in the interior of the grain, the movement of dislocation will be inhibited by these dispersoids.

TEM structures show that a very small fraction of dispersoids is located within the matrix grains of the as-extruded alloy, X-ray diffraction analyses also show that only a small amount of the Al_3Ti phase was formed during the extrusion process; thus the Hall–Petch relation holds good in this case. After annealing, the fraction of dispersoids increases; precipitation strengthening matches the fine grain size strengthening and thus the Hall–Petch equation ceases to be effective.

4. Conclusions

1. Mechanically alloyed Al–10Ti has high tensile strength at room and elevated temperatures, and is stable at 500 °C for moderate annealing times, since the high volume fraction of Al_3Ti dispersoids limits grain growth.

2. Coarsening of Al_3Ti dispersoids was observed after annealing at 500 °C up to 50 h, the coarsening rate follows the Lifshitz–Slyozov–Wager law.

3. The Hall–Petch equation holds good for extruded materials with few particles dispersed in the matrix grains. For annealed alloys with a high volume fraction of dispersoids precipitated in the aluminium matrix, the Hall–Petch equation cannot be used simply to depict the relationship of yield strength with grain size.

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