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Ultrafine-grained pure Ti recycled by equal channel angular pressing with high strength and good ductility

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ABSTRACT

Machining chips of commercially pure Ti (ASTM grade 2) were consolidated into full density by equal channel angular pressing (ECAP) with an average grain size as low as 0.8 μm, yield strength up to 650 MPa, and ductility of ~16%. Effect of recycling condition on the microstructure and mechanical properties were investigated in terms of ECAP temperature, number of passes and chemical composition. Using electron backscatter diffraction it is evident that continuous dynamic recrystallization (however, which is purely a phenomenological terminology) plays a significant role in grain (with misorientation $\geq 15^\circ$) formation, whilst benefitting from high stacking fault energy, this continuous conversion of subgrain ($<15^\circ$) into grain can be essentially considered as an extended recovery with a substantial presence of low angle grain boundaries in the recycled Ti. The Hall–Petch relationship is adapted to explain the strengthening of the recycled Ti. Additionally, using scanning electron microscopy fractography, the ductility was analyzed by a modified Griffith criterion. Last, superior energy efficiency of ECAP reduces environmental impact when comparing to conventional melting/casting. ECAP develops an innovative solid-state process for improving the recycling value of waste Ti.

1. Introduction

Of importance practically, Ti is extensively applied to biomedical engineering (primarily referring to pure Ti of excellent biocompatibility) and aerospace manufacturing (employing preferably Ti–6Al–4V alloy due to high strength and good ductility), leading to a large quantity of waste since up to 80% of the costive raw materials may be lost to valueless chips in order to machine the component with a complex profile in particular. To reduce waste recycling by melting is widely conducted in spite of the high energy consumption. A solid-state process is thus in urgent need. In the last decade, an extensive literature on equal channel angular pressing (ECAP) has indicated that as a commonly used severe plastic deformation process ECAP is effective to produce ultrafine grains. For example, aiming to tailor submicron grains Furukawa et al. (1998) studied ECAP shearing with sample rotation between passes whereby six different deformation routes were defined. It is recommended that route B_c (i.e. rotating the sample by 90° in the same direction after each pass) is optimal to grain refinement. Additionally, expressing a particular interest in the application of a back pressure, Xia and Wu (2005) demonstrated the simplicity and effectiveness of ECAP to consolidate pure Al particles into fully dense bulk material with excellent tensile strength and ductility. It is therefore reasonable to assume that ECAP may be adapted to consolidate machining chips with superior mechanical properties owing to grain refinement induced by severe plastic deformation, and significant energy savings compared to melting.

To the present, a variety of studies have been reported on solid-state recycling of nonferrous metals including Mg and Al, however, without resorting to severe plastic deformation consolidation. For example, AZ91 Mg alloy was recycled by combining ball milling, sintering (at 500 °C) and extrusion (at 350 °C), exhibiting an improved yield strength of 470 MPa (Lu et al., 2006). Similarly, an ultimate tensile strength of 340 MPa and an elongation to failure of ~10% were achieved in recycled AZ91D Mg alloy (Hu et al., 2008). Meanwhile, Chino et al. (2006) recycled AZ31 Mg alloy using an extrusion with a ratio as high as 1600:1. As a result, high yield strength up to 259 MPa was achieved due to grain refinement and oxide dispersion. Aizawa et al. (2002) developed an Al–12 wt% Si alloy recycled from Si particles and Al chips. With an original size ranging over 20–30 μm, the irregularly shaped Si particles were refined gradually (~5 μm) after 400 cycles of forging. Further, the solid-state process can recycle composites. For example, Gronostajski et al. (2001) developed a Fe–Cr–Al composite from a mixture of Al–Mg alloy chips and Fe–Cr powders via sintering (at 550 °C) and the subsequent hot extrusion (at 500–550 °C). Moreover, a bearing composite was consolidated by hot extrusion (at

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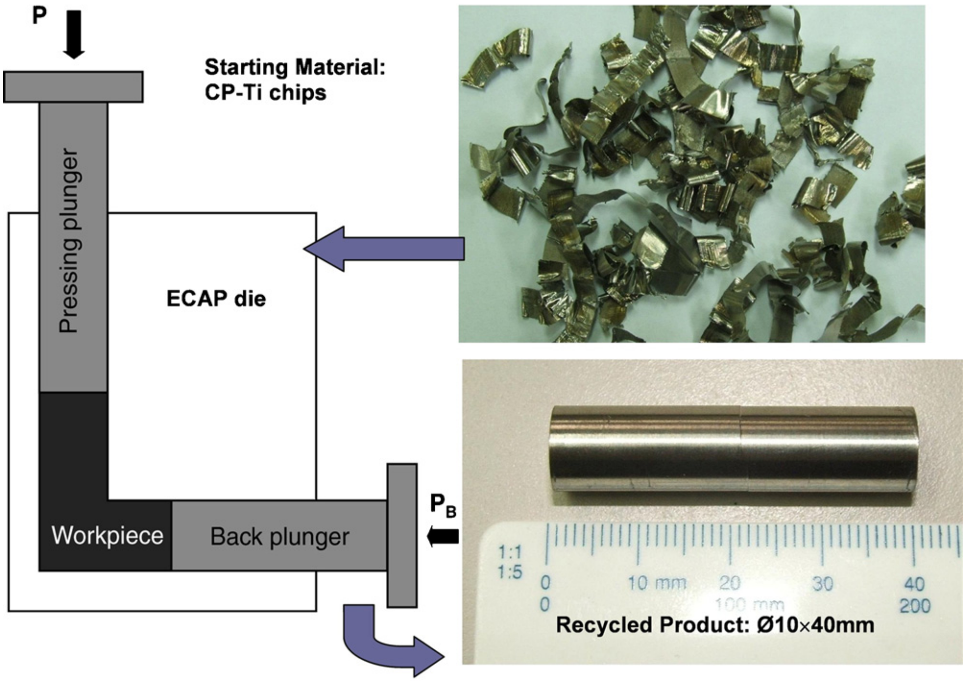

Fig. 1. Schematic illustration of ECAP recycling process for pure Ti chips.

500–525 °C) through mixing Al chips with such reinforcing phases as $CuAl_{12}Fe_4Ni_4$ and $CuAl_{14}Fe_4Ni_4$ powders (Gronostajski et al., 2002). Particularly, annealing (at 500–545 °C for 7 h) endowed the product with full density owing to the Al and Cu atomic diffusions.

In view of solid-state process, nevertheless, it is difficult to consolidate Ti due to its inferior formability at ambient temperature. As a promising structural material with high strength and good ductility, to date, little was done to recycle Ti machining chips particularly by novel severe plastic deformation consolidation in spite of the investigations conducted in recent years by the authors developing ECAP consolidation of Ti chips (Luo et al., 2010), not only analyzing microstructural evolution during dynamic restoration and the strength improved significantly by grain refinement and subgrain formation (Luo et al., 2012a,b), but also studying the strengthening mechanism in the recycled Ti on account of solid solutes, grains/subgrains with either large ($\geq 15^\circ$) or small angle ($<15^\circ$) misorientations, and the oxides on chip surfaces (Luo et al., 2012a,b). A further study in this paper therefore aims to reveal, from the industrial processing point of view, the effects of ECAP recycling parameters (e.g. temperature, number of passes, and composition) on microstructure, strength and ductility of the recycled Ti. Such contributory factors as grain refinement and oxide inclusion were taken into account complying with the Hall–Petch relationship and a modified Griffith criterion, respectively. In addition, the energy efficiency of ECAP was calculated in terms of thermal and mechanical works.

2. Experimental materials and procedure

The chips of commercially pure (CP) titanium (ASTM grade 2) with an average size of 5 mm × 3 mm × 0.15 mm were collected after face mill under dry (without coolant or lubricant in order for reduced contamination) with 75 m/min in feed rate, and 0.12 mm per tooth. Recycling was conducted by ECAP with two channels (11 mm in diameter) intersecting at 90°, as shown in Fig. 1. Converted from a pressure value of 80 pound-force per square inch (psi) in servo-hydraulic activator, a back pressure of 50 MPa was provided by a back plunger in exit channel, Besides, a pressure of up to 1.0 GPa (converted from up to 1600 psi) was provided by a pressing plunger to consolidate the chips into full density. After cleaning with ethanol (99.9%), the CP Ti chips wrapped in a piece of steel foil were put in entrance channel with graphite lubrication. Using a heating device enabling stabilization of temperature within ±1 °C, the die was heated to either 450 or 590 °C. Recycling took place at a speed of 5 mm/min following route C, namely by turning the sample about by 180° between passes. After consolidation, the density was measured by the Archimedes method. Metallographically prepared samples were electropolished in a solution of CH_3OH (600 ml), $C_4H_9OCH_2CH_2OH$ (360 ml) and $HClO_4$ (60 ml) at 30 V for 20 s at −10 °C. High resolution electron backscatter diffraction (EBSD) was carried out in a JEOL JSM FEGSEM using HKL Channel 5 acquisition system. Further analysis was conducted by HKL Channel 5 TANGO. Moreover, transmission electron microscopy (TEM, FEI Tecnai F20 at 200 kV) was used to study the microstructure of the recycled Ti using a sample prepared by twin-jet electro-polishing (Tenupol-3, Struer) with a solution containing H_2SO_4 (10%) in methanol at −30 °C and 20 V; further thinning was carried out using the Gatan precision ion polishing system (PIPS, model 691) with Ar-beam energy of 4 keV at an incident angle of ±4°. Optical microscopy (OM) was used to observe the oxide layer in the sample etched in a solution of H_2O (100 ml), HF (3 ml) and HNO_3 (5 ml) after mechanical polishing. To obtain the 0.2% proof stress, compressive tests were conducted at room temperature with the samples of sizes 4 mm × 4 mm × 6 mm at an initial strain rate of $1 \times 10^{-3}\ s^{-1}$. Besides, to investigate the ductility, at a strain rate of $1 \times 10^{-4}\ s^{-1}$ tensile tests were conducted at room temperature using samples of the recycled Ti with a gauge length of 10 mm. The fracture surface of the tensile specimen was inspected under scanning electron microscopy (SEM, FEI-Sirion 200 at 20 kV). For composition analysis, the oxygen and nitrogen contents in the recycled Ti were analyzed using a LECO TC600 inert gas fusion instrument.

3. Results and discussion

Densities of $\geq 4.50\ g/cm^3$ were obtained in all the consolidated CP Ti samples compared to the theoretical density of pure Ti: