Producing High-Strength Metals by I-ECAP**

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Incremental equal channel angular pressing (I-ECAP) is used in this work to produce ultrafine-grained (UFG) pure iron, aluminum alloy 5083, commercial purity titanium (grade 4), and magnesium alloy AZ31B. Pure iron is processed at room temperature, aluminum alloy at 200 °C, titanium at 320 °C, and magnesium alloy at 150 °C. Strength improvement, attributed to the grain refinement below 1 μm, is reported for all processed materials. The yield strength increase is the most apparent in pure iron, reaching almost 500 MPa after one pass of I-ECAP, comparing to 180 MPa in the as-forged conditions. UFG titanium, aluminum, and magnesium alloys obtained in this study reached yield stress of 800, 350, and 300 MPa, respectively, in each case exhibiting the yield strength increase by at least 30%, comparing to the alloys processed by conventional metal forming operations such as forging and rolling.

1. Introduction

Modern metallic materials with improved strength and good ductility are constantly sought by various industries in order to reduce weight of final components; especially aerospace and automotive sectors are looking for energy savings resulting from weight reduction. A possible approach to producing lightweight metallic components is to replace existing materials with new metals having lower density, e.g., magnesium, aluminum, and titanium. However, their relatively low strength limits the field of possible applications.

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Different strategies can be used for strength improvement, including addition of alloying elements, precipitation hardening, and grain refinement. Modification of microstructure by alloying is a commonly used practice for tailoring properties (strength, ductility, corrosion resistance) of metals. The research in this field has been conducted for several years and the currently available palette of lightweight alloys is very wide, giving a designer many options for material selection tailored for the particular application. Nevertheless, it seems that for most alloys it will be very difficult to go beyond the boundaries of the already achieved mechanical properties by addition of new elements. Alloying with more sophisticated additions, e.g., rare earth elements, vanadium, niobium, molybdenum, is attempted but they are relatively expensive, which increases the final material price.

Mechanical properties of metals and alloys can be also improved by proper thermo-mechanical treatment, including conventional forming operations, e.g., forging, extrusion, rolling, with carefully selected process parameters. More recently, processes of severe plastic deformation (SPD) have come into focus since they are capable of refining grain size of conventional metals and alloys to a submicron range, the so-called ultrafine-grained (UFG) materials, without changing geometry of the billet. SPD provides a possibility of tailoring mechanical properties of metal for a desired application by controlling the grain size within a range much wider than available by conventional metal forming processes. The main advantages of UFG metals are high strength, good formability, and improved fatigue resistance.[1–4] Very often, they also exhibit superplastic properties at temperatures lower than their conventional equivalents.[5–7]

Despite their extraordinary properties, UFG metals have not found many applications in industry due to a low productivity of conventional SPD processes such as equal

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channel angular pressing (ECAP),[8] high pressure torsion,[9] cyclic compression extrusion,[10] multiaxial forging,[11] and many others. Research works focused on developing a continuous SPD process have been conducted in many universities across the world. One of the breakthrough inventions in this field was ECAP-Conform,[12] which combined continuous extrusion (Conform) with ECAP. The process has opened a very promising perspective for the large-scale production of UFG bars and rods; however, it is not suitable for producing plates and sheets. Friction-related issues, such as galling, are also very likely to occur since friction plays a crucial role in feeding of the material into a deformation zone in ECAP-Conform.

More recently, the concept of incremental equal channel angular pressing (I-ECAP) was proposed by Rosochowski and Olejnik[13] as a result of research conducted at the University of Strathclyde, which was aimed at the development of cost-effective process for producing UFG materials. The main advantage of the I-ECAP over conventional ECAP is ability to produce long billets, including bars, plates, and sheets, as published in refs.[14–17] In the I-ECAP, the stages of material feeding and deformation are separated (Figure 1) which significantly reduces frictional force. The punch is moving in a reciprocating manner while the billet is fed into deformation zone in consecutive steps. As the first trials with pure aluminum and pure copper were successful, the work is now being continued with magnesium alloys[18–21] and other materials. The goal of the current work is to show the I-ECAP capability of improving strength of different alloys, which would extend the field of their possible applications.

2. Materials and Methods

Bars with cross-sectional dimensions $10 \times 10\,mm^2$ and length 140 mm were processed by I-ECAP with two billets as illustrated in Figure 1. Materials used in this study were as follows: 1) as-forged slugs of pure iron, having a composition of, in wt%, 0.004% C, 0.04% Mn, 0.007% S, 0.006% N, 0.004% P, and at least 99.8% Fe; 2) cold-rolled plate of aluminum alloy 5083-H12 (referred to as Al5083 in this paper), with the main alloying elements, in wt%, 4–4.9% Mg, 0.4–1% Mn, 0.4% Si, 0.4% Fe, 0.1% Cu, 0.25% Zn; 3) hot-rolled and annealed plate of commercial purity titanium grade 4 (CP-Ti), in wt%, 0.01% C, 0.01% N, 0.017% Fe, 0.34% O_2; 4) hot-rolled and annealed plate of AZ31B magnesium alloy, in wt%, 3% Al, 1% Zn, 0.5% Mn. Different strategies for strength improvement were proposed; steel was subjected to only one pass of I-ECAP at room temperature while aluminum, titanium, and magnesium alloys were processed at elevated temperatures to avoid fracture. Al5083 was heated up to 200 °C and subjected to eight passes of I-ECAP while CP-Ti was subjected to four passes of I-ECAP at 320 °C. Gradual decrease of temperature with consecutive passes was used for AZ31B; first pass was conducted at 200 °C, second one at 175 °C, and the last two passes at 150 °C. Additionally, I-ECAPed magnesium billets were subjected to heat treatment for 1 h at 150 °C, which aimed at increasing ductility without lowering strength. Billets were rotated by 90° between consecutive passes (route B_C) in the cases of Al5083 and CP-Ti, however, our previous results[18] showed that processing path without any rotation (route A) resulted in more efficient improvement of strength for AZ31B magnesium alloy than route B_C. The summary of experimental plan for different materials can be found in Table 1.

I-ECAP process with a die angle 90° was realized on a servo-controlled 1 MN hydraulic press. Billets were fed using a motor driven screw jack with a feeding stroke 0.2 mm. A punch was following a sine waveform signal with a peak-to-peak amplitude of 2 mm and frequency 0.5 Hz. Dies were preheated to the processing temperature and the billets were heated from the dies. Temperature was controlled using a thermocouple placed 15 mm from the deformation zone. The billets were coated with a graphite-based lubricant Durcol W1040-02 supplied by The James Durrans Group.

The microstructures of the as-processed steel, titanium, and aluminum alloys were characterized using transmission electron microscope (TEM) FEI Tecnai G2; magnesium alloy sample was observed in a high-resolution scanning electron microscope (HR-SEM) FEI Inspect F50 equipped with EBSD module for analysis of crystallographic orientation. Uniaxial
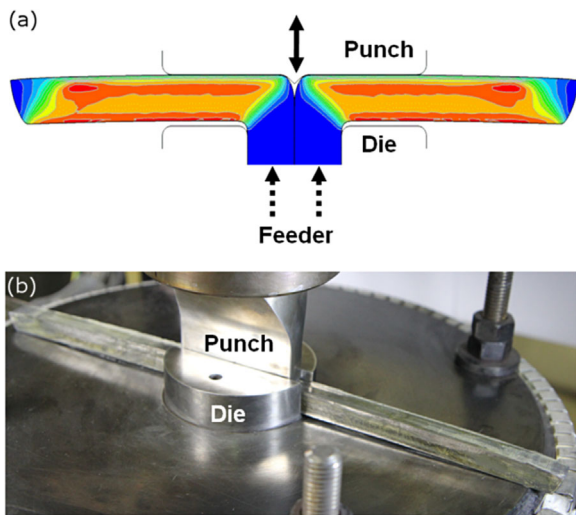

Fig. 1. Tool kinematics and strain distribution in I-ECAP of two billets (a) and the practical realization of this process (b).[14]

Table 1. Experimental plan.

Material	No. of passes	Route	Temperature [°C]	Comments
Pure iron	1	n/a	Room temperature	
Aluminum alloy 5083	8	B_C	200	
CP-Titanium (grade 4)	4	B_C	320	
AZ31B magnesium alloy	4	A	200 + 175 + 150 + 150	Followed by annealing at 150 °C for 1 h

tensile testing was conducted on the universal testing machine Instron 5969 with a load capacity 50 kN. Tests were carried out at room temperature with an initial strain rate $1 \times 10^{-3} s^{-1}$; true strain was measured using video-extensometer. Flat specimens with strain gauge dimensions $3 \times 12 \, mm^2$ and thickness 2 mm were used.

3. Results and Discussion

The results of mechanical testing of the as-received and modified materials are shown in Figure 2. It is apparent that yield strength of pure iron (Figure 2a) was increased from 180 to 490 MPa by only one pass of I-ECAP at room temperature. The increased strength was accompanied by reduction of ductility; true strain at fracture dropped from 0.5 to 0.18, however, the retained formability still allows further processing. Pronounced shear bands are visible in the microstructure (Figure 3a), which is attributed to only one pass of I-ECAP. Usually, the process is repeated to obtain equiaxed grains and homogenous structure; however, the occurrence of band structure does not seem to lower mechanical properties. On the contrary, the yield strength improvement from 180 to 490 MPa in only one pass shows a great potential of the processes for cost-effective production of steel with improved strength without addition of expensive alloying elements. The obtained strength of UFG steel enables reducing weight of the currently used components made of this material by approximately 50%.

Yield strength of Al5083 increased from 265 to 350 MPa after eight passes at 200 °C (Figure 2b). In contrast to pure iron, good hardening response was observed with maximum stress reaching 440 MPa. Additionally, ductility was also enhanced from 0.09 to 0.17 of true strain at fracture. The microstructural analysis showed that the I-ECAPed material owes its improved properties to the ultrafine-grained structure (Figure 3b). The observed improvement of ultimate tensile strength, accompanied by increase of ductility, gives opportunity for reduction of component weight by approximately 30% without lowering its performance. This opens up new possible fields of applications for this alloy family (5×××), which has very good corrosion resistance and weldability. The latter could be especially beneficial for replacing aluminum alloys 2×××, which normally possess higher strength than 5××× but are not weldable. Ultra-fine grained Al5083 combines both, good weldability and strength at the level of 2××× aluminum alloys.

Significant improvement of strength was obtained for CP-Ti grade 4; yield strength was raised from 470 to 805 MPa with fracture strain decreasing from 0.3 to 0.16 (Figure 2c). Despite uniform elongation being smaller than in the case of aluminum, an outstanding tensile strength of 900 MPa was obtained. A possible application of pure titanium is the medical sector,[22] where titanium alloy Ti–6Al–4V ELI is currently used for orthopedic implants, e.g., femoral stems for hip replacement. The addition of aluminum and vanadium to titanium makes it stronger so the implant can sustain
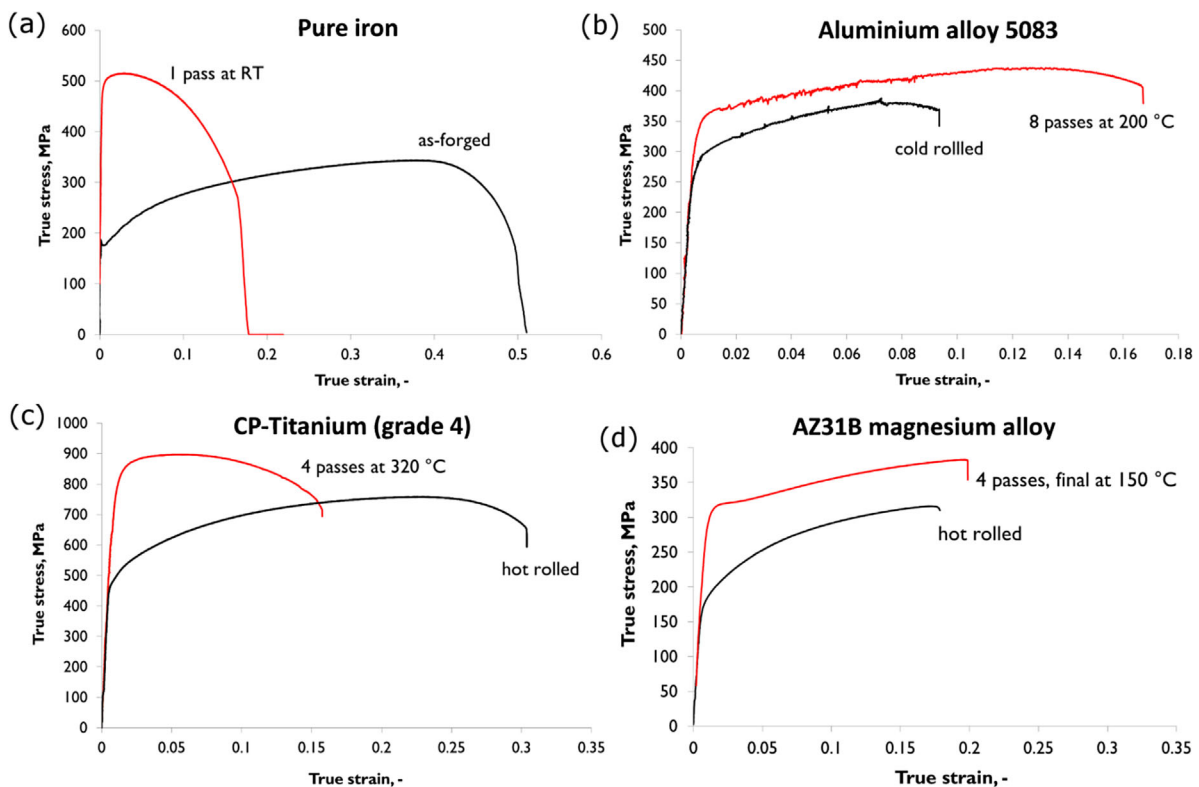

Fig. 2. Mechanical properties of UFG alloys obtained in this work compared with their conventional equivalents: (a) pure iron, (b) aluminum alloy 5083, (c) CP-titanium grade 4, (d) magnesium alloy AZ31B.
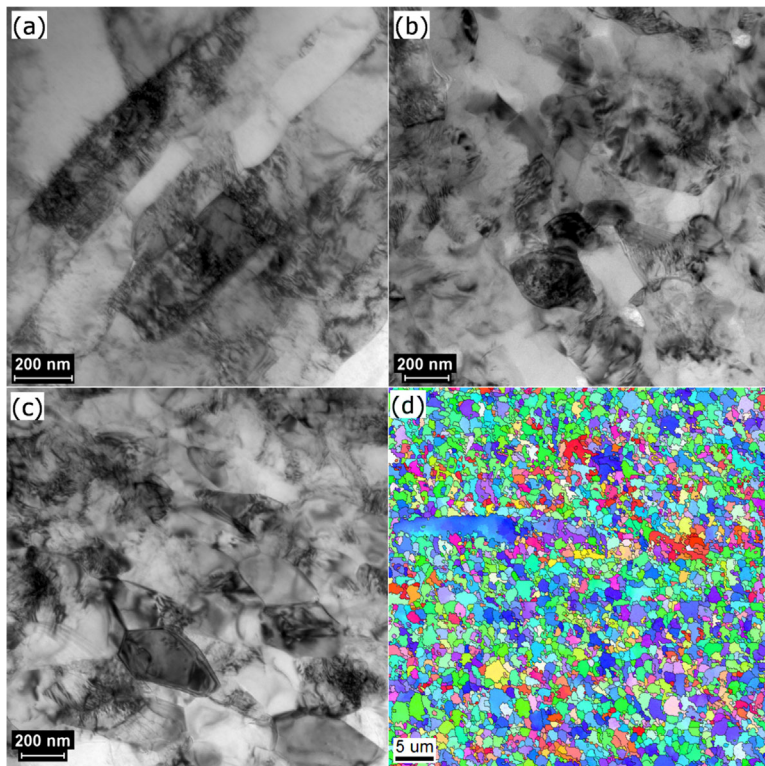
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Fig. 3. Microstructures of UFG alloys obtained in this work: (a) pure iron (TEM image), (b) aluminum alloy 5083 (TEM image), (c) CP-titanium grade 4 (TEM image), (d) magnesium alloy AZ31B (SEM-EBSD image).

the forces applied in the service conditions, however, aluminum and vanadium can be harmful to human organism due to possible release of toxic ions.[23] UFG structure (Figure 3c) developed in pure titanium grade 4 by I-ECAP raised the strength level beyond the minima required for Ti–6Al–4V ELI defined in ASTM-F136 as 795 MPa of yield strength, 860 MPa of ultimate tensile strength and 10% of elongation.

Four passes of I-ECAP, followed by annealing at 150 °C, resulted in a very good combination of strength and ductility in AZ31B magnesium alloy, yield stress was increased from 165 to 305 MPa with a simultaneous ductility enhancement to 0.2 (Figure 2d). EBSD map displayed in Figure 3d revealed that the average grain size was 0.7 μm, which explains significant strength increase. The obtained properties are even better than for cold-rolled plate of aluminum alloy 5083-H12, which exhibited yield strength ≈40 MPa lower than AZ31B. Moreover, usually poor room temperature formability of magnesium is kept at a reasonable level 0.2 of true strain at fracture, which enables further processing. Ultrafine-grained AZ31B can efficiently replace aluminum alloys for applications not requiring high corrosion resistance without lowering performance of the component but reducing its weight by at least 30% due to the lower density of magnesium. Finally, the strength of AZ31B achieved by I-ECAP is higher than in the commercially available magnesium alloys with additions of expensive rare-earth element, e.g., the yield strength of Elektron 43 (Mg–4Y–3RE)

is 195 MPa,[24] which is almost 100 MPa less than obtained in this study for commonly available AZ31B.

4. Conclusions

I-ECAP was shown to be successful in improving strength of metals and alloys by refining their grain structure to the sub-micrometer level. A significant weight saving (30–50%) can be obtained by applying this process to alloys currently used for structural applications. Other benefits include elimination of harmful or expensive alloying elements. While ductility is normally reduced, it remains reasonable or can be even improved, which makes UFG metals suitable for metal forming operations. Finally, the process has a great potential for scaling-up and implementation into industrial practice due to its incremental nature, which enables processing long billets.

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