

Microstructure and mechanical properties of Al-1050 during incremental ECAP

M Salamati¹, S Tamimi¹, S Moturu¹, G Sivaswamy¹, M Qarni¹, A Rosochowski²

¹ Advanced Forming Research Centre, University of Strathclyde,
85 Inchinnan Drive, Inchinnan, Renfrewshire, PA4 9LJ, United Kingdom

² Design, Manufacturing and Engineering Management, University of Strathclyde,
James Weir Building, 75 Montrose Street, Glasgow, G1 1XJ, United Kingdom

E-mail: mohammad.salamati@strath.ac.uk

Abstract. Incremental ECAP is a new method of ECAP process where the severe shear deformation is incrementally applied on the sample resulting in grain refining and new texture developing. The fundamental objective of the present work is an observation of effect of different passes of I-ECAP on microstructure and mechanical properties of AA1050 billet. To that end, 8 pass of I-ECAP have been carried out using Bc route and microstructure evolution and mechanical properties of the I-ECAPed samples have been studied. The EBSD and TEM analyses indicate that I-ECAP is as capable as conventional ECAP to grain refinements and a UFG structure is resulted after I-ECAP cycles. Tensile testing and hardness measurements indicate that mechanical properties of the Al-1050 billets increase dramatically by increasing the I-ECAP passes.

1. Introduction

Severe plastic deformation (SPD) is one of the metal forming processes used to obtain ultrafine grained (UFG) structure (below 1 micron) [1-3]. UFG metals exhibit enhanced physical and mechanical properties compared to that of coarse grained counterparts. Various SPD processes exist but the equal channel angular pressing (ECAP) is one of the few methods that can produce UFG billets for industrial scale applications [4-6]. Segal and his co-workers [7, 8] were the first researchers to study this method. This method induces large shear strain in the billet material by passing it repeatedly through the two channel intersect at an angle $\phi=90^\circ$, and have equal cross-sections. Typically at each pass an equivalent plastic strain of approximately 1.15 is produced in the billets. This imposes large cumulative strains eventually leads to UFG structure in the billet. To achieve varying microstructural characteristics, different routes such as billet rotation between consecutive passes can be used. One of the main drawbacks of ECAP is the length of the billets processed, due to the limitation of the stroke length of the actuator. The incremental equal channel angular pressing (I-ECAP) being an extension of the ECAP process, does not have this limitation therefore can process very long billets. This paper systematically examines the mechanical properties, microstructure of multiple passes I-ECAP samples, with the channel intersection angle $\phi=90^\circ$.

2. Materials and methods



The billet material comprised of 99.5% Aluminium with the following traces elements Cu, Mg, Si, Fe, Mn, Zn and Ti. The billets (10×10×60mm) samples were extracted from round bars in the extrusion direction using electrical discharge machining (EDM). The billet surface was prepared using sandblasting, conversion coating with calcium aluminate and finally applying a thin layer of Loctite 8009 (a graphite based anti-seize lubricant) from Henkel technologies. Room temperature I-ECAP was carried out with a cycle frequency of 0.5 Hz and with a punch amplitude of 1.6 mm. A feeding stroke of 0.2 mm per cycle was used. The details of I-ECAP have been reported in previous studies [10, 11]. The I-ECAP process was performed on a custom built 1000KN servo-hydraulic press with Cubus software. The punch is attached to the press actuator and follows a sinusoidal cyclic command during processing. The material feeding is carried out by a screw jack which is driven by a servo-motor with the help of a dedicated LabVIEW application controls and synchronises the material feeding by monitoring the punch oscillation.

3. Results and discussion

3.1. Mechanical properties

The Zwick ZHV μ micro hardness tester was used to produce the hardness maps on as-received, 1,2,3,4,6 and 8 pass I-ECAP samples as shown in Figure 1. The billets were cut in half, along the flow plane direction and the cut surface was ground and polished to 1 μ m. In total of 500 indents with 1mm spacing between each indents and 5kg load was employed on each samples. With the number of IECAP pass the hardness of the sample increased proportionally.

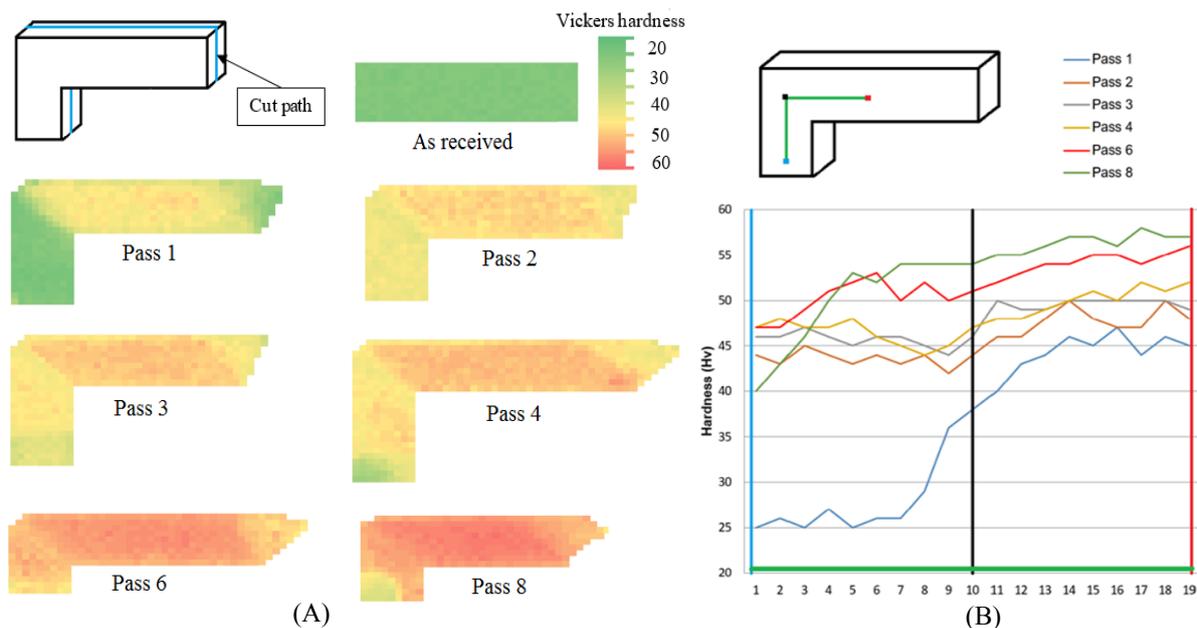


Figure 1: (A) Micro hardness measurement along the flow plain for partially processed billets after IECAP passes of $\phi=90^\circ$ at 1, 2, 3, 4, 6 and 8. (B): Hardening results of billets after passing through the plastic shear zone for passes 1, 2, 3, 4, 6, 8. The green line represents the centre of the billet and the location of the hardness measurements. The blue dot (starting point of hardness results shown in graph), is located 10mm from the centre of the shear zone in the vertical axis and the red dot (ending point of hardness results shown in graph), is located 10mm from the centre of the shear zone in the horizontal axis.

In comparison to other I-ECAP samples, the first pass sample experienced significant hardening, the hardness increased from 22HV to 45HV. The maximum hardness measured after eight passes is 59HV. The subsequent passes also show a more uniform hardness being achieved within the billets. It is also possible to see that the head section of the billet is non-uniformly hardened from the rest of the

billet. The first 10 to 15mm of the billet remains different from the rest of the billet even after 8 passes. It is also interesting to note that the hardness in the subsequent passes at the bottom of the tail section changes compared to the rest of the billet. The side towards the output channels appear to be hardening, whereas the side away from the output channel appears to be softening. It is clear to notice the changes in hardness measurement in all passes of the billets as they pass through the plastic shear zone. An area 10mm either side of the centre line of the shear zone was examined closer and the results can be seen in Fig. 1b.

Most of the hardness changes seem to occur 5mm before and after the centre of the plastic shear point shown in by the black dot in the schematic and the black line in the centre of the graph. Before and after this 5mm zone the hardness of the billet appears to be relatively consistent. This is true for all cases apart from the 8th pass where the partial processed billet was processed further than intended. Partially processing billets can be challenging as it is very difficult to see the billet during processing and determining the processing progress.

To investigate the effects of these changes, tensile testing was conducted on I-ECAP passed material. The processed I-ECAP samples and extracted tensile samples are stored in a freezer to minimize the dynamic recovery and recrystallization. Before tensile testing the specimens were brought to the room temperature. The results from the tensile testing are shown in Fig. 2A.

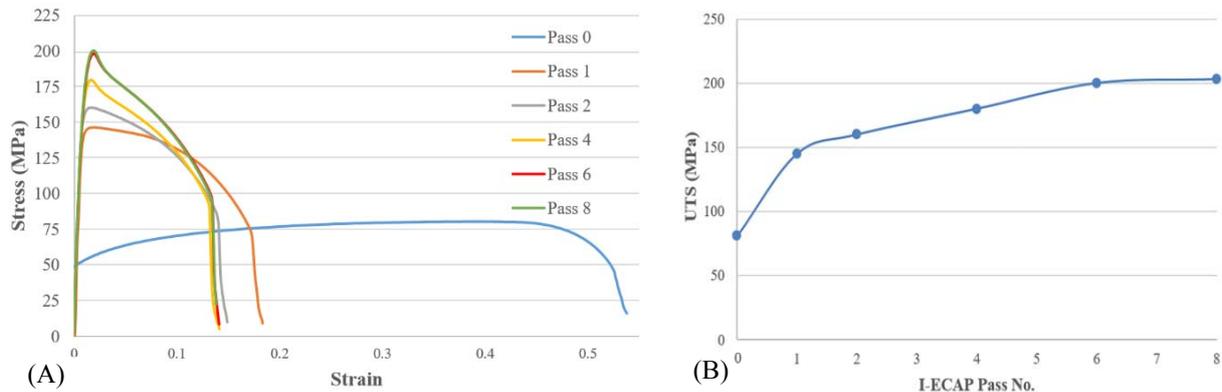


Figure 2: (A) Tensile testing results for I-ECAPed Al1050 specimens. Specimens were created from pass 0, 1, 2, 4, 6 and 8 billets. (B) The Ultimate Tensile Strength of I-ECAPed Al1050 specimens. The UTS of pass 0, 1, 2, 4, 6 and 8 have been plotted.

Due to the relative small size of billets produced in this I-ECAP process only micro tensile specimens could be extracted using EDM. The tensile strength of the Al1050 significantly increases with the number of I-ECAP passes. The ultimate tensile strength (UTS) was about 80MPa in the 'as received' condition, after 8 pass the UTS increased to 200MPa. The elongation before fracture is significantly higher in the as-received materials, After first pass the elongation of the material decreases less than 50%. While the ultimate tensile strength of material after first pass increases by about 75%. This trend continues with subsequent passes until the 6th pass but after that, the UTS only increase slightly. This pattern of stepped increase in mechanical properties with the initial passes was also seen in hardness measurements.

3.2. Microstructure analysis

In order to explain mechanical properties variation of the I-ECAP samples, the microstructure analysis of the samples after each pass was examined. The scanning electron microscope Quanta FEG 650, equipped with a field emission gun (FE-SEM) and with a NordlysF EBSD detector, was used. The AZtec acquisition software was utilized for data acquisition and Channel 5 software to analyse the data. All EBSD measurements were performed with a step size of 2 μ m, using the beam-scanning mode under dynamic focus conditions and with a SEM magnification of 200X. For EBSD analysis,

samples were grinded and finally electro polished using Struers lectroPol 5 and electrolyte A2. Figure 3, shows the microstructure of the as-received and different stages of I-ECAP samples. The observed microstructures at different stages of the I-ECAP clearly indicates the shear deformation of grains. The density of the high angle grain boundaries (greater than 15°) and refinement of grain structure in I-ECAP samples increases significantly in comparison to the as-received sample. The average grain size of the material decreased significantly with increasing ECAP pass. The rate of grain refinement through the initial passes of I-ECAP is higher than the rate of grain refinement through the higher passes.

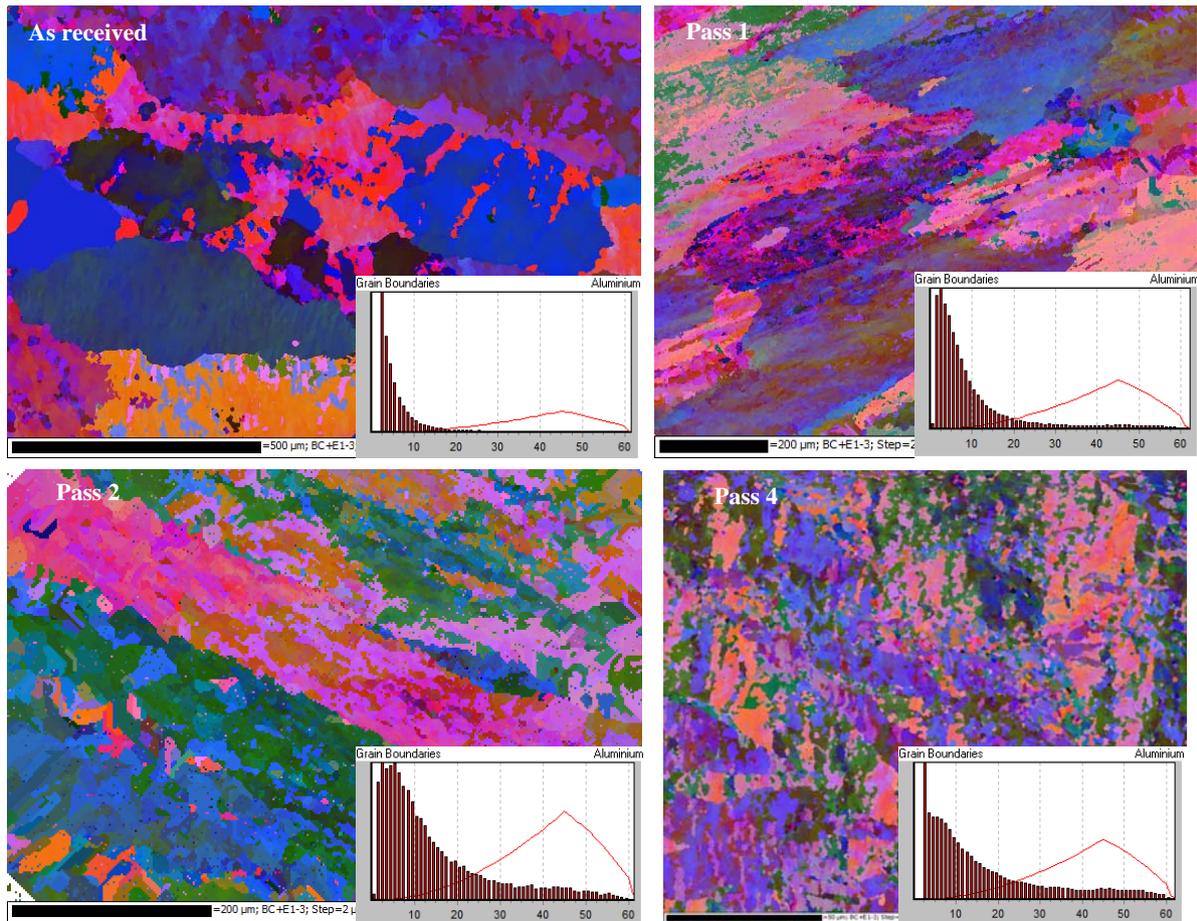


Figure 3: EBSD analyses of Al1050 billets: as received sample and after 1, 2 and 4 passes of I-ECAP.

For transmission electron microscopy (TEM) observations, the samples were sectioned to produce longitudinal sections (flow plane) using a slow speed abrasive saw to obtain slices of thickness less than $300\mu\text{m}$. The slices were mechanically polished to reduce the thickness. Discs of 3mm diameter were spark eroded. The twin-jet electro-polishing with 25% HNO_3 and 75% CH_3OH (by volume) mixture as electrolyte was done as a final polishing. Bright field micrographs recorded from the flow plane of the I-ECAP process samples using TEM are presented and discussed in this section (Fig. 4). Evidence for the formation of banded structure in the flow plane of billet after first is shown in Fig. 4(a). This is primarily due to the intense shear experienced by the billet when it passes through the shear deformation existing between input and output channel. The orientation of these bands are at angle 25° . The dislocations got accumulated with in those deformation bands are clearly shown by the regions having better contrast in micrograph (Fig. 4(a)). Dislocations stored in the banded structure started to undergo recovery process called dynamic recovery, when the billet subjected to second pass. Micrograph of second pass samples given in Fig. 4(b) revealed the formation of dislocations sub-grain

structure developed as a result of dynamic recovery process. Dislocations annihilating the sub-grain boundaries are noticed.

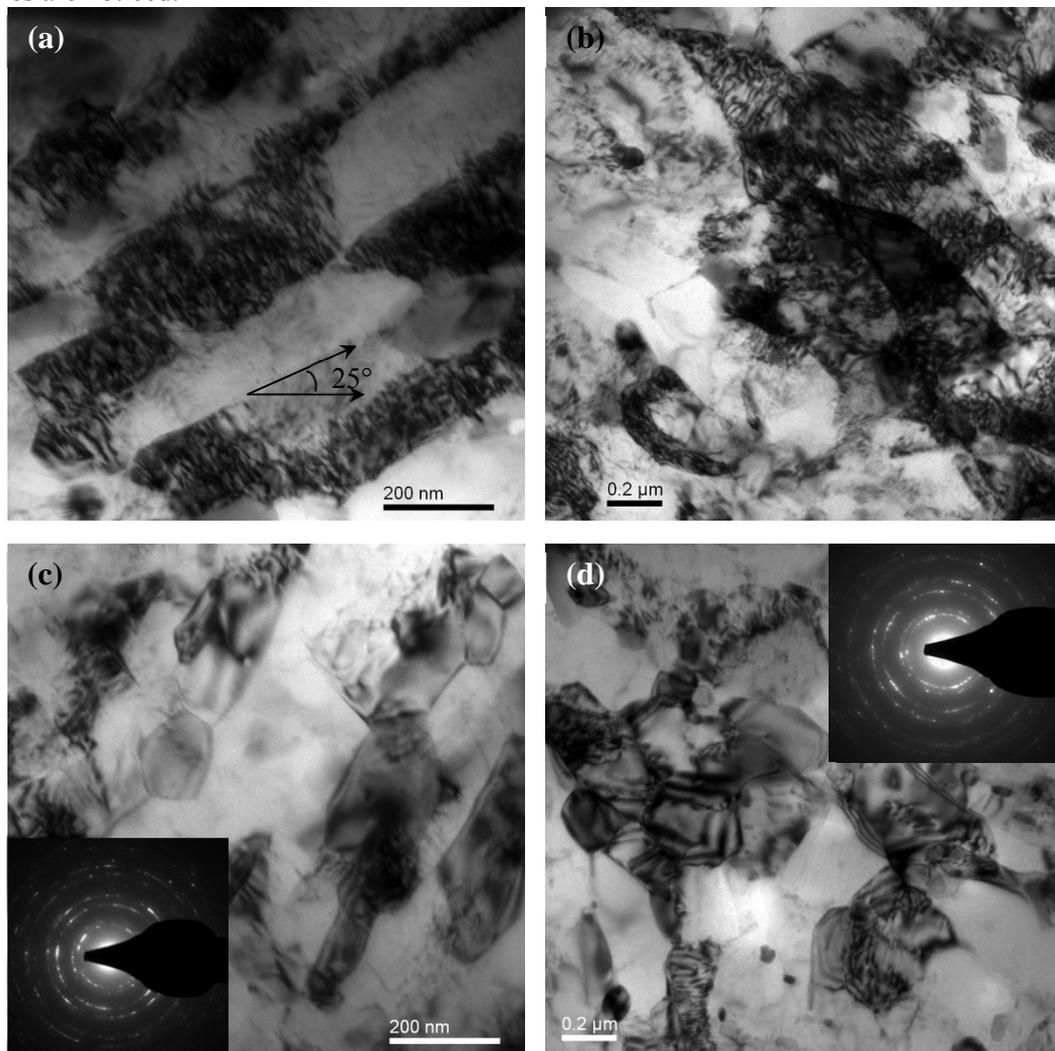


Figure 4: Bright field TEM micrographs showing the deformation characteristics of aluminium matrix in AA1050 alloy billet subjected I-ECAP process after (a) one pass, (b) two passes, (c) six passes and (d) eight passes. Corresponding SADP are given as inserts.

As amount of strain imposed on the billets during subsequent passes increases dynamic recovery assisted recrystallization started to dominate in the matrix leads to the formation of fine grains in the matrix (Fig. 4(c)-(d)). Temperature rise in the billet while crossing the shear deformation zone also influences the process of dynamic recrystallization to some extent. Dynamically recrystallized grains formed in the matrix after 6th and 8th passes are shown in Fig. 4(c) and Fig. 4(d), respectively. Dynamic recovery and dynamic recrystallisation at the 6-8 passes of I-ECAP can explain why the rate of increasing hardness and tensile strength of the samples at the final passes of I-ECAP reduces in compared to the initial stage of the process (see Figures 1 and 2).

Continuous ring patterns shown by the selected area diffraction pattern (SADP) confirming the formations of fine grains in the regions he shown in bright field micrographs of 6th and 8th passes are provided as inserts.

4. Conclusions

In this study we examined the next natural evolution of the classical ECAP method for grain refinement and improved mechanical properties in billets. I-ECAP improves some of the limitations that are often associated with ECAP. The results of this study showed that the first pass leads to the greatest increase in mechanical properties and the following passes had a more gradual increase in these effect. The microstructure results indicate that grain size in the billet decreases with the increase in the number of passes of I-ECAP. In addition, dynamic recovery and recrystallization take place which influence the mechanical properties of the material at the later passes of I-ECAP.

References

1. R. Valiev, R. Mulyukov, V. Ovchinnikov, and V. Shabashov, 1991: "Mossbauer analysis of submicrometer grained iron", *Scripta Metallurgica et Materialia (USA)*, 25, pp. 2717-2722.
2. N.A. Akhmadeev, R.Z. Valiev, V.I. Kopylov, and R.R. Mulyukov, 1992: "Formation of submicro grain structure in copper and nickel by extensive shear deformation", *Russian Metallurgy*, 2, pp. 96-101.
3. J. Wang, Z. Horita, M. Furukawa, M. Nemoto, N.K. Tsenev, R.Z. Valiev, Y. Ma, and T.G. Langdon, 1993: "An investigation of ductility and microstructural evolution in an Al- 3% Mg alloy with submicron grain size", *Journal of Materials Research*, 8, pp. 2810-2818.
4. R.Z. Valiev, 1997: "Structure and mechanical properties of ultrafine-grained metals", *Materials Science and Engineering A*, 234-236, pp. 59-66.
5. I.V. Alexandrov, Y.T. Zhu, T.C. Lowe, R.K. Islamgaliev, and R.Z. Valiev, 1998: "Microstructures and properties of nanocomposites obtained through SPTS consolidation of powders", *Metallurgical and Materials Transactions A*, 29A, pp. 2253 - 2260.
6. V.V. Stolyarov, Y.T. Zhu, T.C. Lowe, R.K. Islamgaliev, and R.Z. Valiev, 1999: "A two-step SPD processing of ultrafine-grained titanium", *NanoStructured Materials*, 11, pp. 947-954.
7. V.M. Segal, 1977: "The method of material preparation for subsequent working", Patent of the USSR No. 575892.
8. V.M. Segal, V.I. Reznikov, A.E. Drobyshvskiy, and V.I. Kopylov, 1981: "Plastic metal working by simple shear", *Russian Metallurgy*, 1, pp. 99-104.
9. M. Furukawa, Z. Horita, and T.G. Langdon, 2002: "Factors influencing the shearing patterns in equal-channel angular pressing", *Materials Science and Engineering A*, 332, pp. 97-109.
10. A. Rosochowski, and L. Olejnik, 2011: "Incremental Equal Channel Angular Pressing for Grain Refinement", *Materials Science Forum*, 674, pp. 19-28.
11. M.R. Salamati, M. J. Qarni, S. Tamimi, and A Rosochowski, 2016: "Effect of channel angle on the material flow, hardness distribution and process forces during incremental ECAP of Al-1050 billets", *ESAFORM 2016 AIP Conf. Proc.*, 1769, 090004-1–090004-6