45 Degree Equal Channels Angular Pressing for Grains Refinement and Softening of Lead Alloy

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ORIGINAL RESEARCH

Abstract— Severe plastic deformation (SPD) has over the times been employed in the production of enhanced lightweight metals and generally to improve a specific property of material without introducing expensive alloying elements to the current materials or applying high energy demanding heat treatment technique. This paper examined the influence of severe plastic deformation of lead alloy using equal channel angular pressing (ECAP). The technique was applied to lead alloy at room temperature using channel angles of 45°. The materials were processed up to three ECAP passes and subjected to equivalent strains of 8.70. Hardness test, impact test, and microstructural changes of the processed materials were examined. Results showed that extrusion force reduced as the strain level increased and that dynamic recrystallization and structural changes reduced the hardness of lead processed through 45° ECAP. Hardness of 8.95 HV was recorded when the material was processed through third ECAP pass, while 9.05 HV was recorded for second ECAP pass. The analysis shows clear reduction in the material hardness as number of pass increases when compared to the control sample with hardness of 16.77 HV. The result of impact test showed no clear difference between the values obtained for the material when processed through second and third pass because the amount of energy absorbed remained unchanged from 4 J for the two passes. Analysis of microstructure images also revealed that increasing the strain level leads to break down and dissolution of antimony rich precipitate. The microstructural change caused by ECAP of the control sample softens the material, as a result of break-up of the original precipitate structure and the acceleration of the dynamic recrystallization of the material

Keywords— dynamic recrystallization, ECAP, lead alloy, SPD.

1 INTRODUCTION

Extrusion is a process used to create objects of a fixed cross-sectional profile. A material is pushed or drawn through a die of desired cross-section. The two main advantages of this process over other manufacturing processes are its ability to create very complex cross sections and work materials that are brittle, because the material only encounters compressive and shear stresses. It also forms finished parts with an excellent surface finish (Iwahashi et al., 1998).

Severe Plastic Deformation (SPD) is an innovative process capable of producing uniform plastic deformation in a variety of materials, without causing significant change in geometric shape of the work piece (Edalati, 2023). Severe Plastic Deformation (SPD) was developed with the aim of improving the microstructure and consequently the production of metals and alloys with proper microstructure and high strength and ductility.

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Grain size is considered to be a key microstructural factor affecting nearly all aspects of the physical, chemical and mechanical behaviours of polycrystalline metals. Based on the Hall-Petch relationship (Naik et al., 2020), strength of materials related to their grain size as shown in equation 1.

$$\sigma = \sigma_0 + k_y d^{-12} \qquad \text{equation (1)}$$

Where the σ is the friction stress, d is average grain size, σ_0 is the yield stress and K_y is the yield constant (Naik et al., 2020).

Grain refinement has therefore been employed as a method of improving mechanical properties of metals and alloys which can be accomplished traditionally through the use of processes such as rolling, forging, and extrusion, sometimes followed by a post-processing heat treatment. However, these traditional means are restricted in their ability to produce ultra-fine grains structures for two principal reasons: one, there is a limitation on the amount of strain that may be imparted using these processes because of the reduction in the geometry or cross-sectional dimensions of the work-piece. In rolling, for example, the strain levels required to attain the formation of ultra-fine grain structures are only reached in thin foils. Two, the strains imposed in traditional methods are therefore insufficient to produce large ultra-fine grains structures because of the generally low workability of metallic alloys at ambient and relatively low temperatures. Therefore, these problems pose significant limitations for production of larger parts or synthesis and processing of new materials with the objective of developing special microstructure and properties. Meanwhile, the concept of ultra-SPD, with the capacity to introduce the shear strains of over 1000 to reduce the thickness of sheared phases to levels comparable to atomic distances was recently utilized to synthesize novel 'superfunctional' materials (Edalati, 2023). In order to overcome the limitations of conventional processing techniques, Severe Plastic Deformation (SPD) techniques were evolved. With SPD, strain achieved was found to be sensitive to the die angle, friction conditions, and the application of a back-pressure, all of which can have a large effect on the microstructure and strain inhomogeneity within the processed billet. The distribution of strain is most uniform, and approximates most closely to a simple shear, if the deformation zone is constrained to be as narrow as possible. The best processing conditions appear to be obtained with a sharp die corner, low friction, and a constraining back-pressure (Bowen et al., 2000).

Equal-channel angular pressing (ECAP), otherwise known as Equal-channel angular extrusion (ECAE) process is a severe plastic deformation process capable of producing ultrafine grains with superior mechanical properties. The mechanical properties and grain refinement depend on the number of passes in the ECAP process (Gupta et al., 2021). ECAP has been the most commonly used technique in severe plastic deformation because it offers the potential for high strain rate super plasticity by effective grain refinement from macro-grain structures to the level of the nano-scale through a special die (Gupta et al., 2021). The procedure has been proven to be capable of producing significant grain refinement in materials that can greatly improve the tensile strength and hardness of a material while maintaining reasonable levels of ductility. The process of ECAP involves pressing a billet (material) through a die consisting of two channels of equal cross sections, intersecting at an angle (**Φ**) as shown in figure 1.

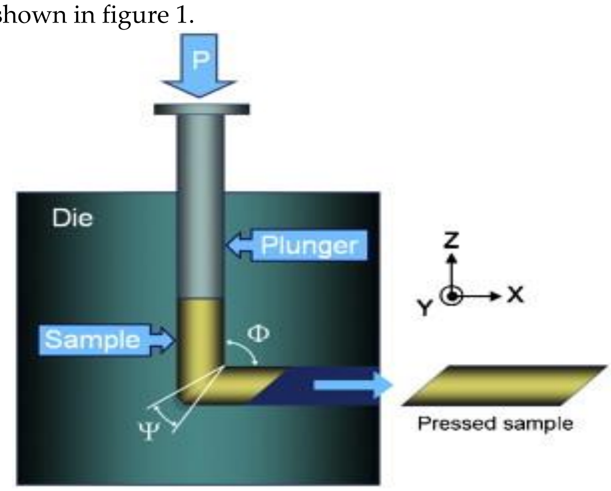

Figure 1: A schematic illustration of the equal channel angular pressing set-up, showing the die channel angle (**Φ**), angle of curvature (Ψ) and the plunger used to press the material (RAM) (Frint et al., 2016).

The growing interest in nanomaterials and especially nanoparticles has increased in the last decades primarily due to their novel or enhanced physical and chemical properties compared to bulk material. These extraordinary properties have resulted in multitude of innovative applications in the fields of medicine and pharma, electronics, agriculture, aerospace, automobile, transportation, food and chemical processing, conventional defence industries and many others (Joudeh et al, 2022). These exceptional material properties requirements in the industries have led to a considerable interest in the development of ultra-fine-grain/nanomaterials by severe plastic deformation.

The processing of materials by ECAP has undergone active development in several areas. These areas include the development of many different nano-scale metals and alloys and the commercial production of semi-finished products within ultra-fine grained structures using a wide range of metals and alloys (Iwahashi et al., 1998). Also, microstructure of a material, that shows the arrangement of its atoms and the associated material phases, has a significant impact on its macroscopic characteristics. Understanding microstructure is critical because it serves as the basis for predicting and modifying material behavior (Kumar, et al., 2023)

The objective of this study is to examine the influence of ECAP on the behaviour of lead alloy using channel angles of 45°. Mechanical and microstructural properties of the processed material were also examined and compared to the control sample.

2 METHODOLOGY

2.1 Material Selection, Design and Construction of Dies and Punches

The material used for this project was lead alloy while high carbon steel material was for the production of the die and the punches instead of harder tool steel material. This is because lead billet is soft and required lesser force to be extruded. Multiple Lead billets with dimensions of 12.65 mm x 12.65 mm x 100 mm were machined out of a homogeneous block of lead. This dimension was used for the billet to fit into the channel of the die with a tolerance of 0.05 mm.

The die is made up of two halves of the same dimensions, otherwise called split die as shown in Figure 2. The Split die enables an increased flexibility in providing required design features in same die-set. After all considerations, such as the dimensions of the billet to be used, the cross sectional dimensions of each die was 112 mm × 153 mm as a whole. Figure 3 shows the pictorial view of the die when coupled. There are also two locating pins in the die set to avoid misalignment of the die during die opening

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and closing and during extrusion process. Four Allen bolts were also provided to fasten the die set.

Channels of dimensions 12.7 mm × 6.35 mm were machined into each half of the dies. The punch has dimensions of 12.65 mm × 12.65 mm × 120 mm . The clearance of 0.025 mm between the channel and the punch allowed the punch to be properly fit into the channel and also create room for adequate lubrication. Several punches were made for the purpose of this experiment to allow easy replacement when the plunger got deformed during extrusion process. Figure 4 shows the picture of a punch.

Figure 2: Die with 45° Extrusion Angle

Figure 3: A coupled die

Figure 4: The Plunger

2.2 EXPERIMENTAL PROCEDURES

The extrusion was performed using a Universal Tensile Testing Machine of 600kN capacity. The machine has different force range graduations, but the 0kN - 120kN range was selected based on projected force required to carry out extrusion for lead billet. The samples were rotated about the extrusion axis by 180° between each pass (route C) up to a total of three passes. This processing route imparts the smallest end-effects on the samples from multiple ECAP pressings.

The experiment started off with de-coupling of the die for its channel to be lubricated and insertion of billet material into it. Both the lead billet to be extruded and the channel of the die were lubricated. The lubricant used for this experiment was palm oil. Palm oil has good viscosity, less expensive and had proven to be good in reducing friction (Yahaya et al., 2023).

The die was then coupled after the lubrication and the already lubricated billet was inserted into the channel of the die. The punch was placed into the channel entrance. The schematic representation of the arrangement is shown in figure 5 below:

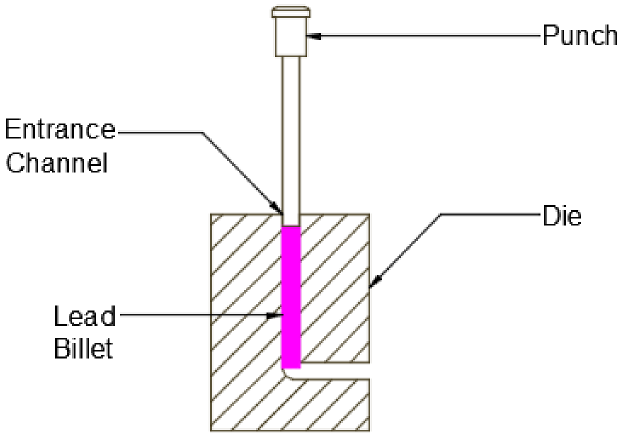

Figure 5: Experimental set up showing the punch, the billet and the die

A flat cover plate was placed on the punch head and the assembly was placed on the bed of the axial loading machine as can be seen in the picture below, ensuring the

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exit of the die pointing towards the operator for easy observation as billet comes out during pressing.

Figure 6: Die placed on Axial Loading Machine

The Axial Loading Machine (Figure 6) was turned on through the power button and the movable top half of the machine descended to the top of the punch and once the scale reading was just about to begin, (at a point, the punch makes contact with the billet), the machine was stopped. The machine was then calibrated for the load and displacement scales to read zero.

After the calibration was done, the extrusion of the lead billet was then carried out by applying load on the billet gradually, which then extrudes or pushes the lead billet through the exit channel. Care was taken not to push the punch too far in order for it not to follow the curvature of the channel which damages the punch and lead to incorrect load reading.

Immediately the extrusion started the load values with corresponding displacement moved were manually taken and recorded. Load values for every 4mm movement were taken for this experiment. When the extrusion was done and all values taken, the die was de-coupled and the extruded lead specimen taken out and then reshaped with hand file in order for it to fit back into the entrance channel. Reshaping was required because it was discovered that surfaces at both ends of the extrudate became uneven after each pass. Thus, ends of the billets were removed after pressings to avoid the propagation of end-effects. this lead to a decrease of the billet length during multiple pressings.

In this study with channel angle 45°, only specimens that have undergone 2 and 3 passes were produced as the specimen became too short after it has undergone the third pass for it to be extruded further.

2.3 Testing

At the end of extrusion process, extrudates (processed samples) were prepared for testing by machining to dimensions 10 mm × 10 mm × 55 mm, and subjected to hardness and impact tests. The properties were compared with the properties of the sample before passing it through the die, called control sample. Hardness test and Impact test were also carried out on a fresh billet to serve as a control. Positive Material Identification (PMI) test was also done on the lead billet as displaced in table 1 in order to determine the percentage composition of various elements of the billet.

2.3.1 IMPACT AND HARDNESS TESTS

Charpy Impact Test at ambient temperature of 25°C, in accordance with ASTM E23 using an Instron 406J (300 ft-lbs) machine was carried out on all the specimens. The specimens had to be machined to a dimension of 10 mm × 10 mm × 55 mm with a 2 mm deep V-notch at the middle for the impact test to be carried out. Vickers Test as per ASTM E384 was carried out also specimens.

2.3.2 MICROSTRUCTURAL ANALYSES

Samples of unprocessed and processed lead materials were prepared for microstructural analyses. Samples were taking from processed materials that have undergone through second and third passes. The microstructure was revealed after etching the samples with a solution of 3 percent nitric acid and 97 percent Ethanol – NITAL (3% HNO_3 plus 97% CH_3COOH). The specimens were observed by optical microscope. Square work pieces for compression tests were machined from the raw billet and the ECAP-processed material. They had 10 mm sides and 55 mm height.

2.3.3 POSITIVE MATERIAL IDENTIFICATION TEST (PMI)

PMI test as per ASTM E572 was carried out on one of the lead billet used for the extrusion to know the individual constituents of the billet and their percentage composition. Olympus Delta professional PMI equipment was used for this test.

3 RESULTS AND DISCUSSION

3.1 RESULT OF PMI

The following are the results obtained from the PMI test carried out on the specimen used for this experiment: The chemical composition of the lead alloy is displayed in the Table 1.

Table 1: Results from PMI test

Elements	Al	P	Si	Tl	Fe	Sn	Cu	Zr	Pb	Sb
Amount (%)	0.310	0.269	3.740	0.078	0.128	3.096	0.08	0.007	83.10	9.190

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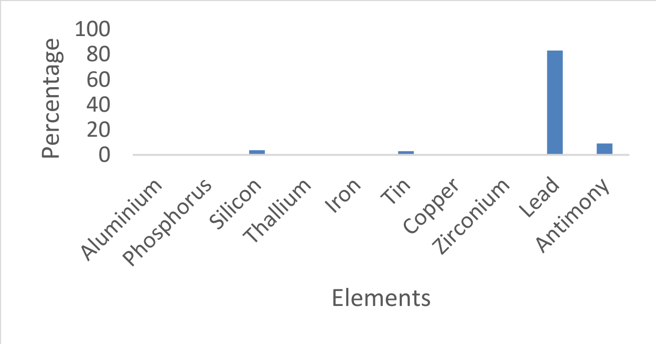

Figure 7: Bar Chart Showing the Results Obtained from PMI test on the lead billet used

It can be seen from the chart in Fig 7 that the specimen used is a lead alloy with greater percentages of antimony, silicon and tin.

3.2 LOAD-DISPLACEMENT BEHAVIOURS

3.2.1 LOAD - DISPLACEMENT FOR 2ND PASS

Data obtained from the load-displacement behaviour at second pass of the extrusion were recorded and presented graphically in Figure 8.

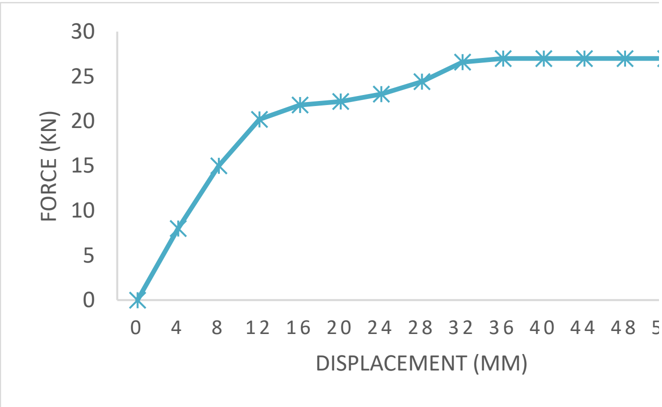

Figure 8 Load- Displacement Graph at 2rd Pass

3.2.2 LOAD-DISPLACEMENT FOR 3RD PASS

Data obtained from the load-displacement behaviour at third pass of the extrusion were recorded and presented graphically in Figure 9.

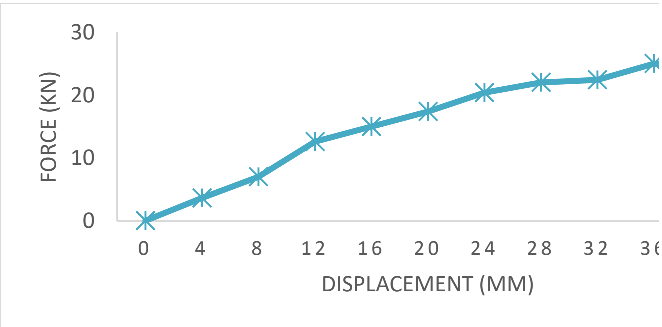

Figure 9 Load- Displacement Graph at 3rd Pass

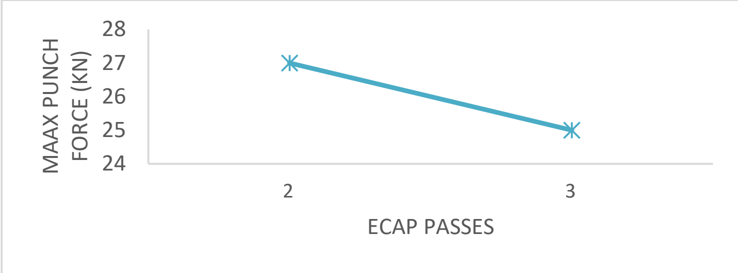

Figure 10: Maximum Punch Force versus ECAP Passes

The extrusion was done for only 2nd and 3rd passes. Extrusion force progressively increased with steady increase in displacement and reaches a point where the force remains constant at an increase in displacement for the two passes. Looking at the graphs from the two passes, it can be deduced that the amount of extrusion force required in extruding the specimen decreased with passes. That is, the force used for the second pass is greater than the force used for the third pass.

3.3 HARDNESS BEHAVIOUR

Table 2 shows the results gotten from the hardness test carried out on the specimens while Table 3 displays the percentage reduction of hardness with the control specimen.

Table 2: Hardness of the Specimens after 45° ECAP

	2nd Pass Hardness (HV)	3rd Pass Hardness (HV)
Control	16.77	16.77
45°	9.05	8.95

Table 3: Percentage Reduction of Hardness of Control Specimen for Each Angle

Angles	Control Sample Hardness Value	3rd Pass Hardness Value	Percentage Reduction
45^0	16.77	8.95	46.63

From the Table 2, it was observed that the hardness decreases from the second pass to the third pass. It can be deduced that the extrusion of the lead specimen results in a decrease in hardness as the hardness of the un-extruded control sample is higher than that of the extruded samples.

3.4 IMPACT BEHAVIOUR

Table 4 shows the results gotten from the impact test carried out on the specimens.

Table 4: Results from Impact Test

Angles	2nd Pass Energy Absorbed (J)	3rd Pass Energy Absorbed (J)	Average Energy Absorbed (J)
45^0	4	4	2

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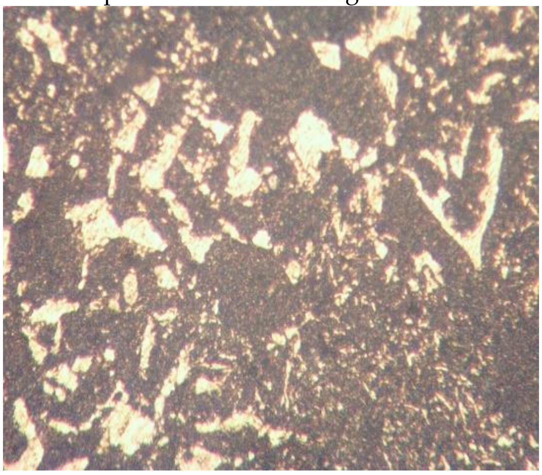
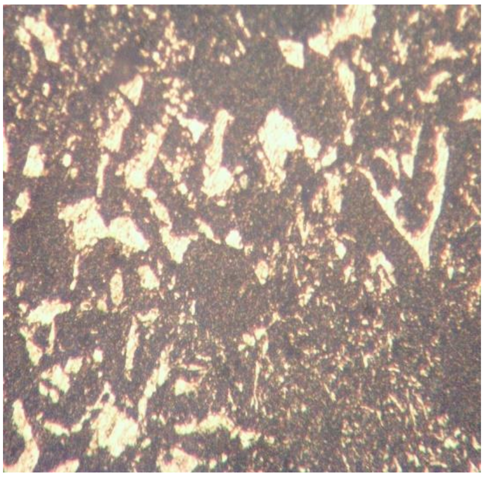
The data obtained shows no difference in the amount of energy absorbed for both second and the third passes

3.5 MICROSTRUCTURE

Images of the specimens viewed at × 100 and × 200 magnifications at 2nd pass for angle 45^0 under an optical microscope are shown in the figures below.

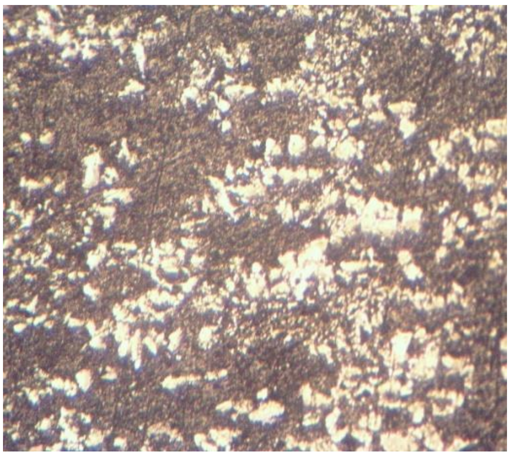


(a)

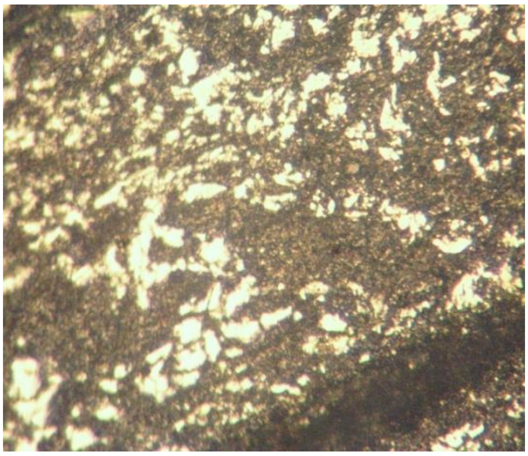


(b)

Figure 11: a-b: Control Sample at × 100 and × 200 Magnifications Respectively



(a)



(b)

Figure 12 (a-b): Angle 45^o at 2nd Pass ×100 and 2nd Pass ×200 Respectively.

The decrease in the punch force as number of passes increased, shown in the Figure 10, was undoubtedly associated with lower hardness as the number of passes increases. This abrupt fall of hardness after two ECAP pass is probably caused by dynamic recrystallization in the material during the pass. The Pb recrystallization temperature is below 0°C (Vander Voort, et al., 2004) and since the extrusion was carried out at room temperature, so it is expected that the material recrystallizes during the process, leading to no grain refinement effects caused by ECAP.

Recrystallization is a process by which deformed grains are replaced by a new set of defects-free grains that nucleate and grow until the original grains have been entirely consumed. Recrystallization is usually accompanied by a reduction in the strength and hardness of a material and a simultaneous increase in the ductility (Stolyarov et al., 2003). In dynamic recrystallization, as opposed to static recrystallization, the nucleation and growth of new grains occur during deformation rather than afterwards as part of a separate heat treatment.

The microstructure generally shows three phases. The dark phase is the lead-rich matrix; the bright phase corresponds to antimony-rich precipitates while the brownish phase must be tin phase. Figures 11-12 shows the evolution of the precipitate distribution of antimony alloy at 2nd ECAP passes for angle 45^o.

One common feature of the microstructures is that the second pass broke some antimony precipitates, as seen in Figure 12. Some unnatural lines or black spots were seen in the microstructure of 45^o at 2nd pass which is probably due to some cracks sustained during extrusions. However, there are still remaining some larger particles of antimony observed.

This indicates that ECAP caused some dissolution of the antimony-rich phase similarly to the reports in the

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literature for precipitates in an Al-Fe alloy (Chinna Maddaiah et al., 2023).

Finally, reports in literature had identified wastages as a major limitation to SPD due to cutting off of uneven surfaces at both end of the billet due to non-identical deformation (Chinna Maddaiah, et al., 2023). The has not been developed to meet large industrial production due to limited length of work piece.

4 Conclusion

Lead alloy processed by Equal Channel Angular Pressing up to three passes using 45° die angle gave the following behaviours:

The extrusion force reduced with increased strain level (number of pass). Hardness of the control sample reduced with increased number of extrusion passes as a result of dynamic recrystallization of the material.

Impact test showed no appreciable difference in the data obtained from the material processed through second and third pass as the energy absorbed remained unchanged. These results show a clear deviation from behavior of other metals processed through ECAP as observed by Gupta et al where impacts and hardnesses increased with number of passes. Hence ECAP can only be recommended for lead in applications where lesser impact and hardness are desired.

The antimony-rich precipitates in the control sample underwent dissolution, caused by the ECAP processing. The microstructural change caused by ECAP in the control sample softens the material, due to the break-up of the original precipitate structure and to the acceleration of the dynamic recrystallization of the material.

The original microstructure of the control sample is broken by the ECAP, but evidence from the microstructures at 2nd pass suggests more strains are required for the complete break-up of the original microstructure.

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