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Original Research Article

Influence of Equal Channel Angular Extrusion on the Behavior of Lead Alloy

Olumoroti, I. A[1], Adebayo, A[2*]

[1]Department of Mechanical Engineering, Federal University, Oye-Ekiti, Nigeria
[2]Department of Mechanical Engineering, Ekiti State University, Ado-Ekiti, Nigeria

***Corresponding Author:** Adebayo, A
Department of Mechanical Engineering, Ekiti State University, Ado-Ekiti, Nigeria

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Abstract: Equal Channel Angular Pressing (ECAP) is the most promising material processing technique involving severe plastic deformation, and has been extensively employed and analysed. The aim of this work is to examine the influence of ECAP on the behaviour of Lead alloy. The technique was applied to Lead alloy at room temperature using route C, at channel angles of 45^0, 60^0, 75^0, 90^0 and 105^0. The materials were processed up to five ECAP passes. Hardness test, impact test, and microstructural changes of the processed materials were examined. Results show that extrusion force reduces as the strain level increases and that dynamic recrystallization and structural changes reduce the material hardness for all angles of ECAP. All the angles absorbed their least amount of energy at their 5th pass. Analysis of microstructure images also revealed that increasing the strain level leads to break down and dissolution of Antimony rich precipitate.

Keywords: Equal channel, Angular pressing, Lead alloy, channel angle, dynamic recrystallization.

1.0 INTRODUCTION

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. Severe Plastic Deformation (SPD) is developed with the aim of improving the microstructure and consequently the production of metals and alloys with proper microstructure and high strength and ductility. In recent decade, research has been focused on materials processing by Severe Plastic Deformation (SPD) due to the unique physical and mechanical properties obtainable by SPD processing and capability of producing bulk products free of porosity and inclusion [1].

Grain refinement has 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**.** In order to overcome the limitations of conventional processing techniques, Severe Plastic Deformation (SPD) techniques were evolved [6].

The equal channel angular pressing (ECAP) is regarded as the most commonly used technique 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. 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.

The demand for high-strength metals and alloys has been on the increase in today's emerging markets. ECAP products find applications in aerospace, automobile, transportation, food and chemical processing, electronics, and conventional defence industries. 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 [5].

For engineers, the fundamental interests in nanometals is their ability to decrease the dimensions of mechanical devices. In fact, the developments in all important fields of micro and nanotechnology depend on availability of suitable materials. Also successful shaping methods of engineering nanocomponents are required for building MEMS and nanodevices.

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 [6]. The application of the ECAP procedure is currently under investigation for many different materials ranging from aluminum, copper, magnesium, lead alloy and nickel alloys and composites materials.

The objective of this study is to examine the influence of ECAP on the behaviour of lead alloy using varying channel angles of 45°, 60°, 75°, 90° and 105°. Mechanical and microstructural properties of the processed materials were examined and compared to the control sample. Hardness and impacts tests were carried out on the processed samples. Microstructures of the processed materials at second and fifth passes were also taken.

2.0 METHODOLOGY

2.1 DESIGN AND CONSTRUCTION OF DIES AND PUNCH

Material selection for the die and the punch caused a bit of delay for this project as tool steel material was set out to be used initially, but the cost of tool steel in the Nigerian market was tool exorbitant. Since the material to be pressed was lead alloy (less aggressive material as compared to Aluminium), high carbon steel material was later used for the production of the die and the punch

Each die is made up of two halves of the same dimensions, otherwise called split die. A Split die was also used which enable 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 112mm × 153mm as a whole .The figure 2.1 below shows the typical pictorial view of the one of the coupled die used. There are also two locating pins in the die set to avoid misalignment of the die during die opening and closing and during extrusion process. Four bolts were also provided to fasten the die set.

Channels of dimensions 12.7mm × 6.35mm were machined into each half of the dies. The channels were machined in all the different extrusion angles of 45°, 60°, 75°, 90° and 105°. The punch has dimensions of 12.65mm × 12.65mm × 120mm which are slightly lesser than that of the channel for it to properly fit into the channel. Several punches were made for the purpose of this experiment.

Fig 1: A coupled die

2.2 Material Selected for the Work Pieces (Billets)

The material used for the work was a Multiple Lead billets with dimensions of 12.65 mm X 12.65 mm X 100 mm. The billets were machined out of a homogeneous block of Lead. The dimensions were used for the billet to fit into the channel of the die with a tolerance dimension of 0.05 mm.

2.3 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. The samples were rotated about the extrusion axis by 180° between each pass (route C) up to a total of 5 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 proven to be good in reducing friction and also less expensive.

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 2 below:

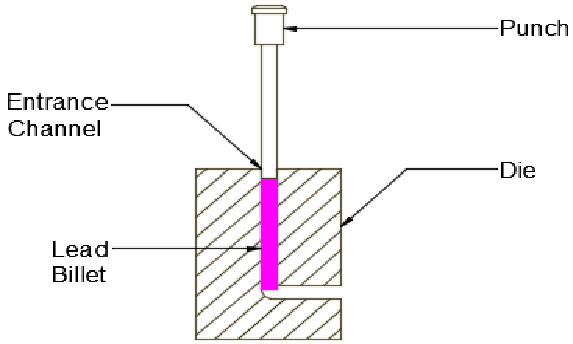


Fig 2: Experimental set up showing the punch, the billet and the die

A flat cover plate was placed on the punch head and the assembly was placed on the bed of the axial loading machine, ensuring the exit of the die pointing towards the operator for easy observation as billet comes out during pressing.

The Axial Loading Machine is 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 is done, the extrusion of the lead billet is 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 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 is started, the load values with corresponding displacement moved was taken and recorded. Load values for every 4mm movement were taken for this experiment. When the extrusion is done and all values taken, the die is de-coupled and the extruded lead specimen taken out and then reshaped in order for it to fit back into the entrance channel. The ends of the billets are usually eliminated after pressings to avoid the propagation of end-effects, leading to a decrease of the billet during multiple pressings.

Each specimen was clearly labelled after every successful pass to avoid mixed up of passes. The above steps are then repeated for every subsequent pass of extrusion and for all channel angles.

In this study, specimens with 2 passes up to five passes were produced for each channel angles of 45°, 60°, 75°, 90°and 105°. However, for the 45° die, 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.

2.4 Testing

After all extrusions were carried out, extrudites (processed samples) were prepared for testing by machining to dimensions 10mm × 10mm × 55mm, 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.

2.4.1 Impact and Hardness Test

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 10mm × 10mm × 55mm with a 2mm deep V-notch at the middle for the impact test to be carried out. Vickers Test as per ASTM E384 was carried out on all the specimens.

2.4.2 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 fifth 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.

2.4.3 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. The equipment used for the PMI test is Olympus Delta Professional PMI Equipment.

3.0 RESULTS AND DISCUSSION

3.1 POSITIVE MATERIAL IDENTIFICATION (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 displaced in the Table 1 below

Table 1: Results from PMI test

Elements	Al	P	Si	TI	Fe	Sn	Cu	Zr	Pb	Sb
%Specimen	0.310	0.269	3.740	0.078	0.128	3.096	0.08	0.007	**83.100**	9.190

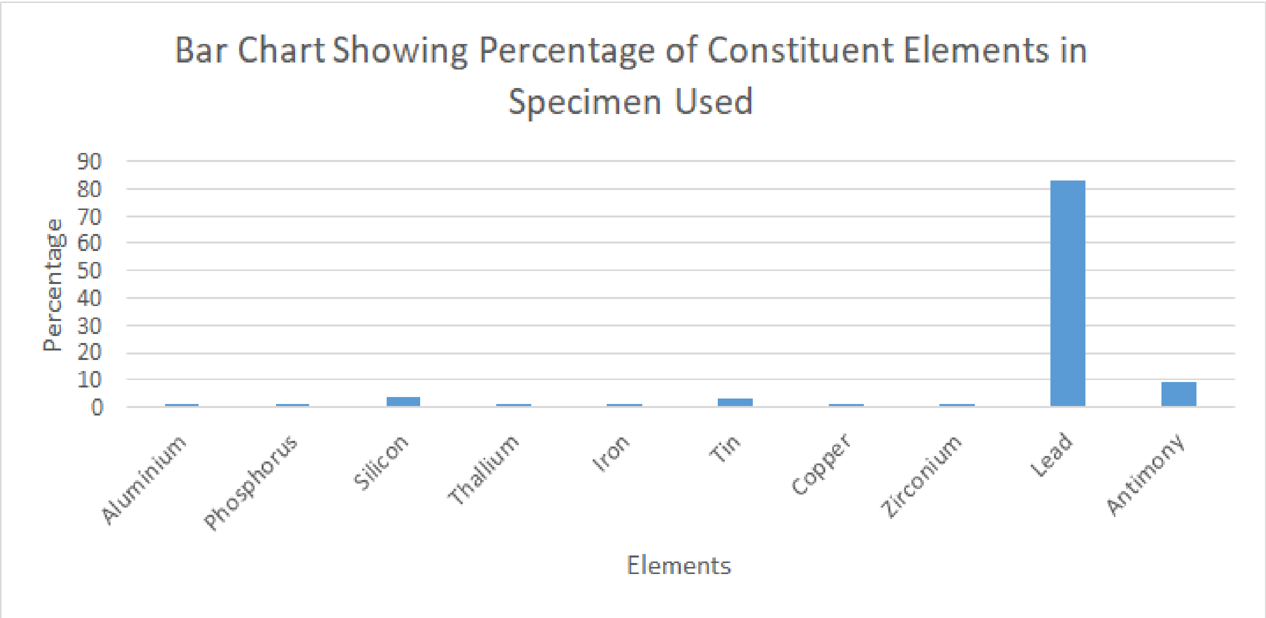


Fig 3: Bar Chart Showing the Results Obtained From PMI

It can be seen from the chart in Fig 3 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

The graph below shows the combined load – displacement behaviour of all the angles at second pass of extrusion:

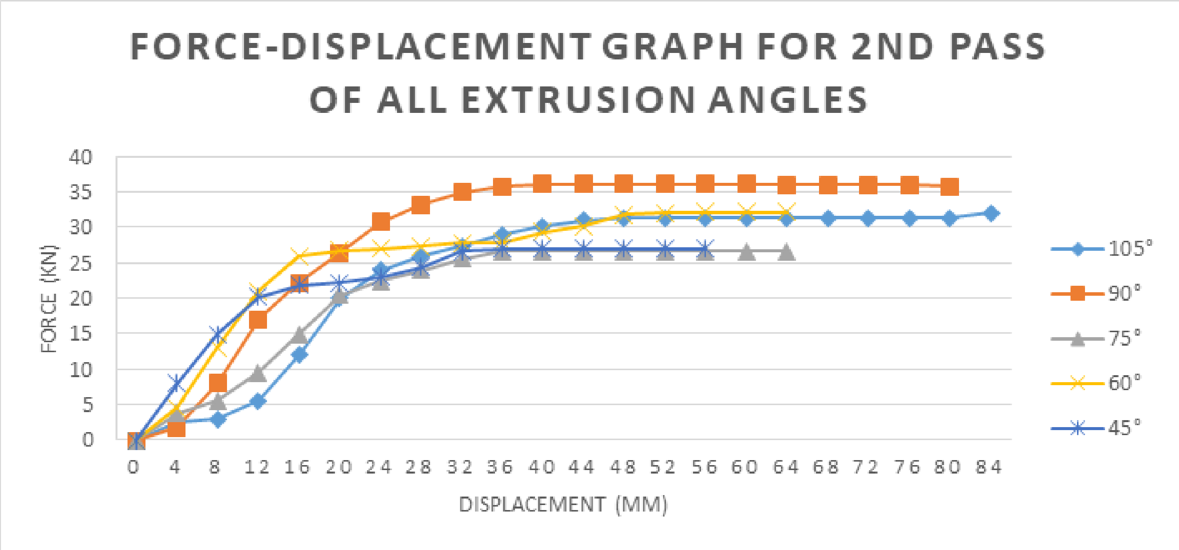


Fig 4: Combined Load- Displacement at 2nd Pass

It can be seen from the graph in Fig.4 above that it took a higher amount of force to extrude out the specimen from the 90° die than from the rest of the dies. It also took the least amount of force to extrude out from the 75° Die. Arranging the dies in order of descending amount of force to extrude the specimen in the second pass of extrusion is as below:

90°→ 60°→ 105° → 45° → 75°

3.2.2 LOAD-DISPLACEMENT FOR 3RD PASS

The graph below shows the combined load – displacement behaviour of all the angles at third pass of extrusion:

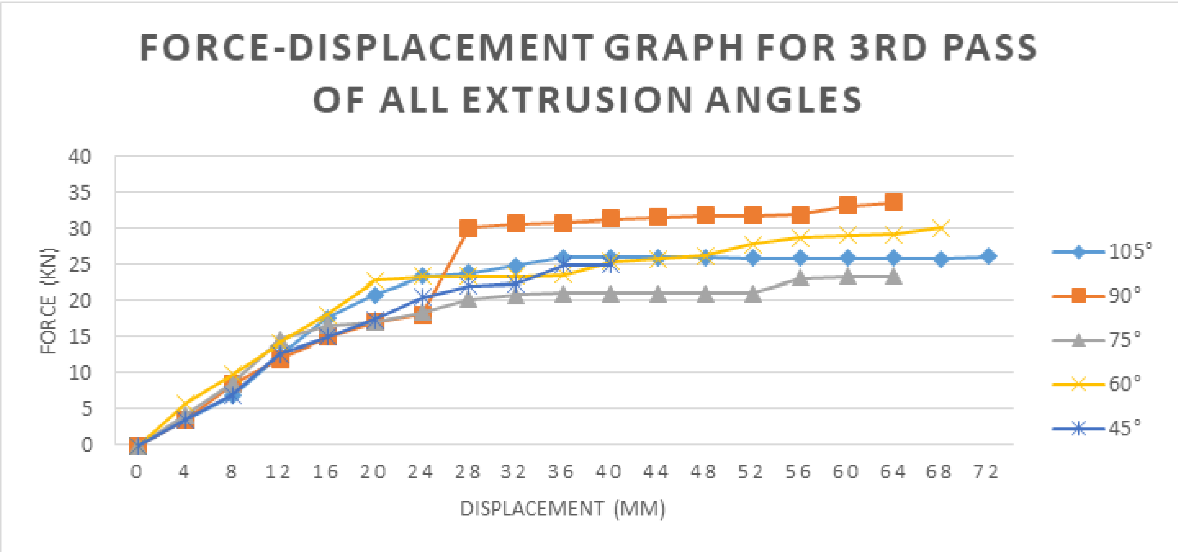


Fig 5: Combined Load- Displacement at 3rd Pass

It can be seen from the graph in Fig 5 above that it took a higher amount of force to extrude the specimen from the 90° die than from the rest of the dies. It also took the least amount of force to extrude out from the 75° die. Arranging the dies in order of descending amount of force to extrude the specimen in the third pass of extrusion is as below:

90° → 60° → 105° → 45° → 75°

The behaviour is the same with that of the second pass of the specimens.

3.2.3 LOAD-DISPLACEMENT FOR 4TH PASS

The graph below shows the combined load–displacement behaviour of all the angles at fourth pass of extrusion:

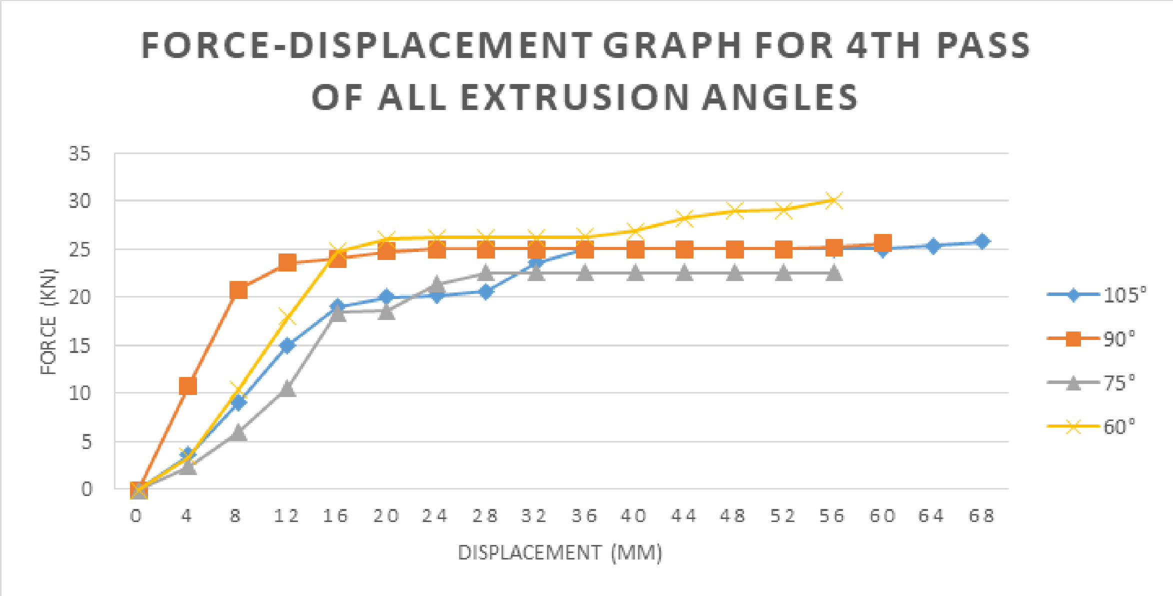


Fig 6: Combined Load-Displacement at 4th Pass

It can be seen from the graph in Fig. 6 above that it took a higher amount of force to extrude the specimen from the 60° die than from the rest of the dies. It also took the least amount of force to extrude from the 75° die. Arranging the dies in order of descending amount of force to extrude the specimen in the fourth pass of extrusion is as below:
60° → 90° → 105° → 75°

It took higher amount of force to extrude from the 60° die than the 90° die during the fourth pass of the specimens. All other dies exhibit the same behaviour as the second and third pass with the exception of 45° die which could not be extruded for the fourth pass as explained in the previous chapter.

3.2.4 LOAD-DISPLACEMENT FOR 5TH PASS

The graph below shows the combined load–displacement behaviour of all the angles at fifth pass of extrusion.

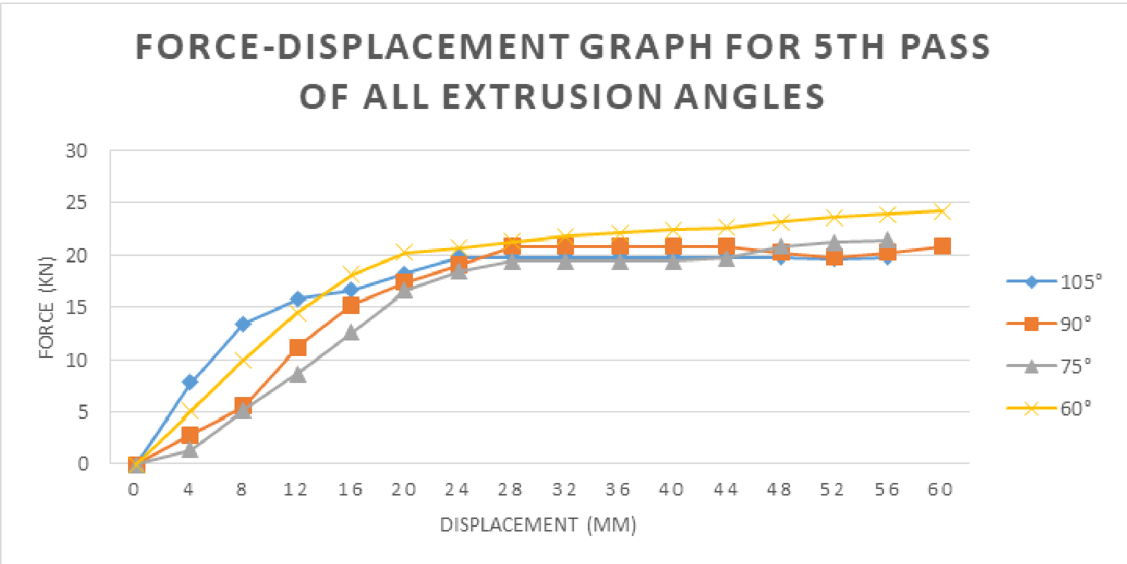


Fig 7: Combined Load-Displacement at 5th Pass

It can be seen from the graph in Fig 7 above that it took a higher amount of force to extrude the specimen from the 60° die than from the rest of the dies. It also took the least amount of force to extrude from the 105° die. Arranging the dies in order of descending amount of force to extrude the specimen in the fifth pass of extrusion is as below:
60° → 90° → 75° → 105°

The fifth pass of extrusion of specimens through the 60° die and the 90° has similar behaviours to fourth pass of the specimens, that is, they have the first and second highest load respectively. It took the least amount of force to extrude out of the 105° die.

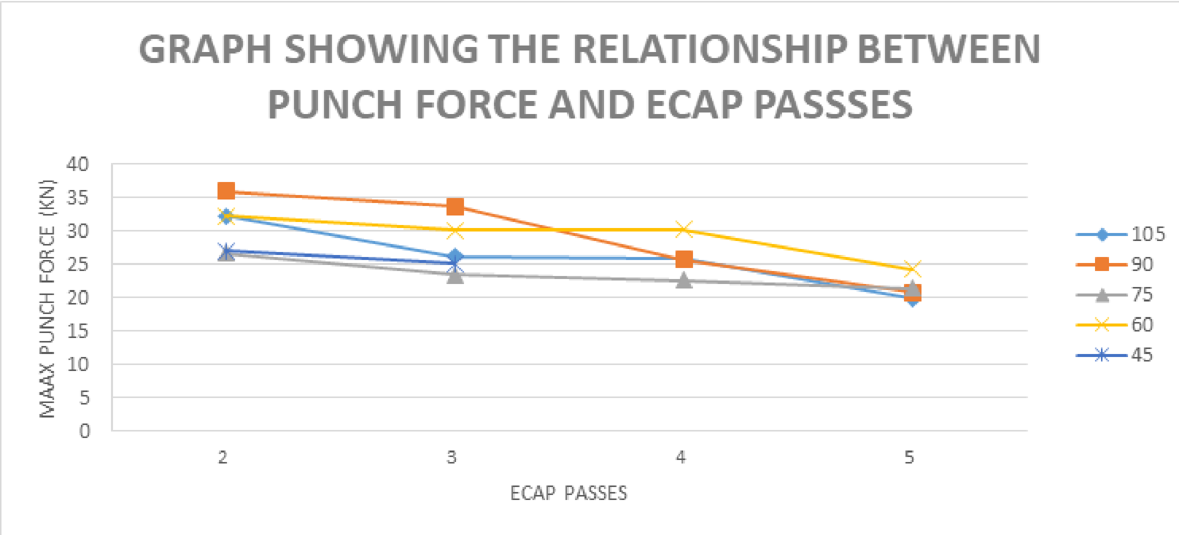


Fig 8: Max Punch Force versus ECAP Passes

The minimum number of passes was set to be two and extrusion was done for 2nd, 3rd, 4th and 5th pass for each angle. Each graph above exhibits different trends with one angle overtaking the other, in terms of extrusion force, at different number of passes, but generally the extrusion force progressively increased with steady increase in displacement and reaches a point where the force remains constant at an increase in displacement for all extrusion angles and at each pass. Looking at the graphs from 2nd pass to 5th pass for each angle, it can be deduced that the amount of extrusion force required extruding the specimens decreased with each pass. That is, the force used for the second pass is greater than the force used for the rest of the passes with the fifth pass requiring the least amount of force. This behaviour holds true for all extrusion angles as it can be seen in Fig 8.

3.3 HARDNESS BEHAVIOUR

Table 2 below shows the results gotten from the Hardness test carried out on the specimens while Table 3 displays the percentage reduction of hardness of control specimen for each angle at the end of 5th passes except for 45° that could not go beyond 3rd pass due to reasons advanced in the previous chapter. Fig 9 shows the graphical display of hardness at each pass for all angles.

Table 2: Hardness Results

	2nd Pass Hardness (HV)	3rd Pass Hardness (HV)	4th Pass Hardness (HV)	5th Pass Hardness (HV)
Control	16.77	16.77	16.77	16.77
105°	12.50	10.75	9.85	9.20
90°	9.80	10.55	8.95	8.20
75°	10.10	9.88	9.80	8.00
60°	9.95	9.90	9.85	8.35
45°	8.60	8.95		

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

Angles	Control Sample Hardness Value	3rd Pass Hardness Value	5th Pass Hardness Value	Percentage Reduction
105^0	16.77		9.20	45.10 (5th Pass)
90^0	16.77		8.20	51.10 (5th Pass)
75^0	16.77		8.00	52.29 (5th Pass)
60^0	16.77		8.35	50.20 (5th Pass)
45^0	16.77	8.95	-	46.63 (3rd Pass)

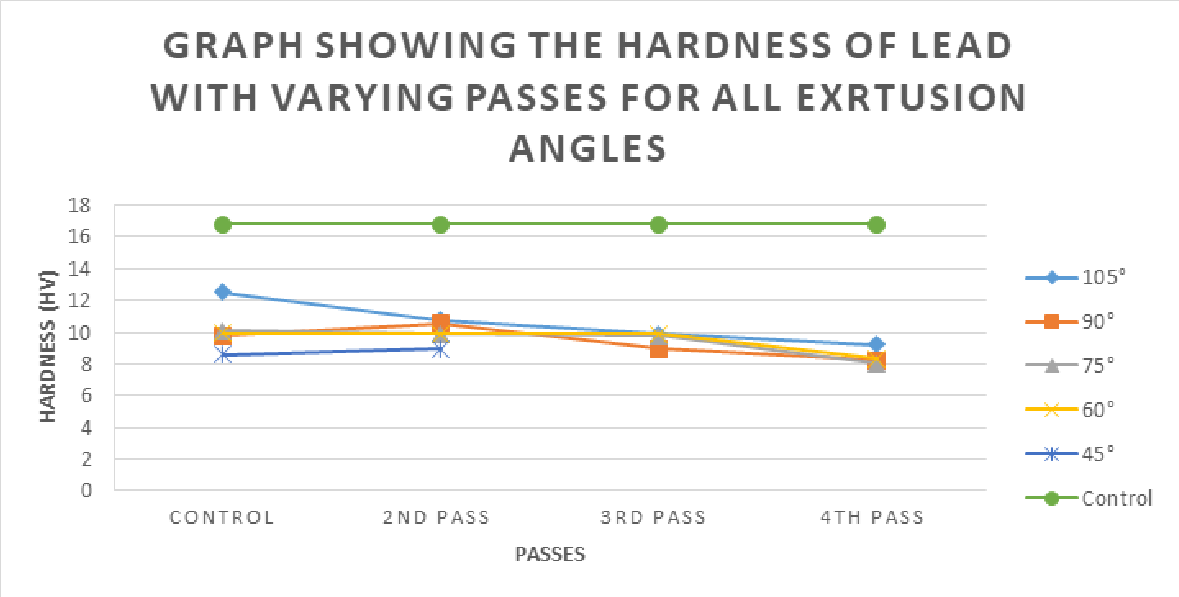


Fig 9: Graph Showing the Results Obtained From Hardness Test

It can be seen from the table 2 and the graph in Fig. 9 above that specimens behave differently at various passes for each angle. Table 3 shows the percentage reduction in hardness. This shows that the material extruded through the 105° die has the highest hardness for most of the passes. Specimens that were extruded through the 60° die had the second highest hardness for most of the passes followed by the 90° and 75° die. Specimens extruded through the 45^o die shows tendency to have the least hardness if extruded further because at 3rd pass its hardness had reduced to almost 46%. It also shows increased in hardness from 2nd pass to 3rd pass, but generally decreased**.**

It can also be seen from the graph that hardness decreases with increase in the number of extrusion passes. Also, it can be seen 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 while Fig 3.7 shows the graphical display of the results.

Table 4: Results from Impact Test

Angles	2nd Pass Energy Absorbed (J)	3rd Pass Energy Absorbed (J)	4th Pass Energy Absorbed (J)	5th Pass Energy Absorbed (J)	Average Energy Absorbed (J)
105^0	7	6	7	4	6
90^0	3	5	4	2	3.5
75^0	4	6	5	4	4.75
60^0	4	4	5	3	4
45^0	4	4			2

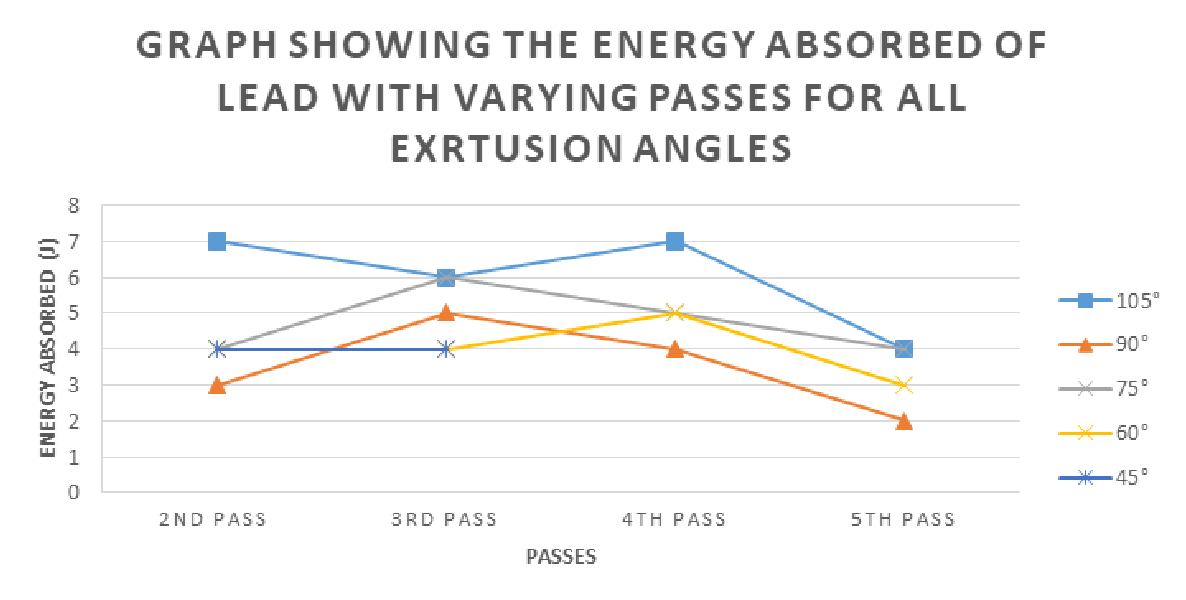
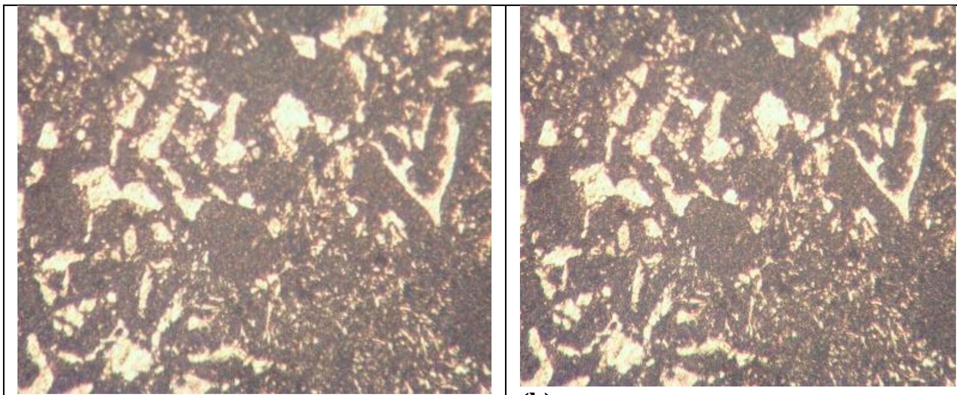


Fig 10: Graph Showing the Results Obtained From Impact Test

Generally speaking, it can be seen from the graph in Fig 10 above, specimen extruded through the 105° die absorbed the most average energy with 90° being the least. All angles absorbed their least amount of energy at their 5th pass.

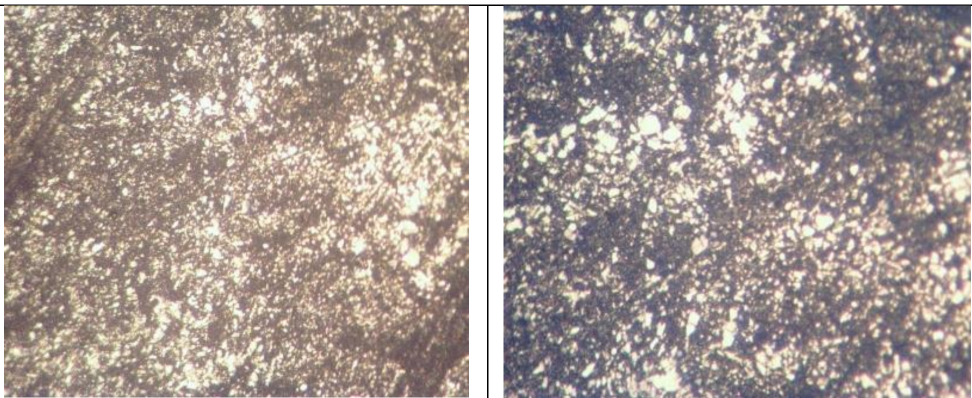
3.6 MICROSTRUCTURE

Images of the specimens viewed at × 100 and × 200 magnifications at 2nd and 5th passes for each angle under an optical microscope are shown in the figures below.



(a) (b)

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



(a) (b)

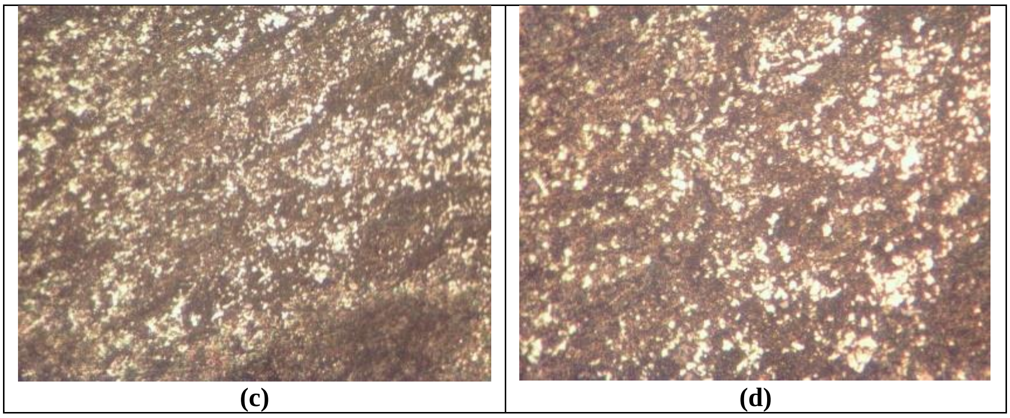


Fig 12 (a-d): Angle 75^{o} at 2nd Pass × 100, 2nd Pass × 200, 5th Pass ×100 and 5th Pass × 200 Respectively

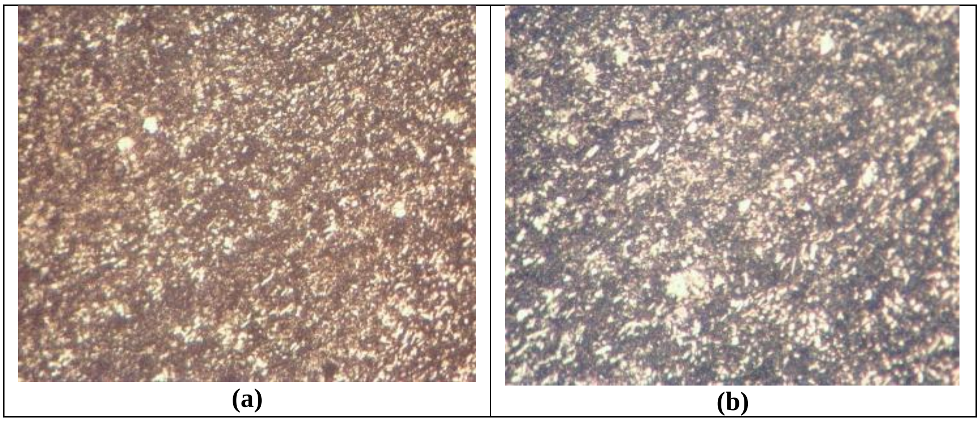


Fig 13 (a-b): Angle 60^{o} At 2nd Pass ×100, 2nd Pass ×200 Respectively

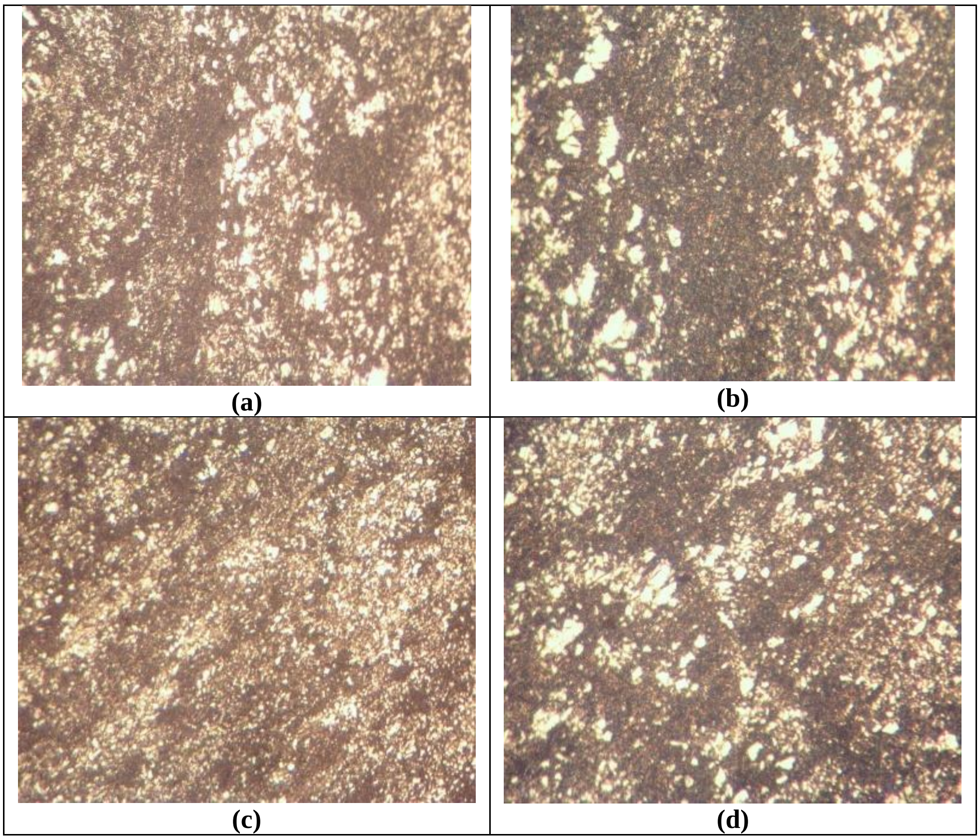


Fig 14 (a-d): Angle 90^{o} at 2nd Pass ×100, 2nd Pass ×200, 5th Pass ×100, and 5th Pass ×200 Respectively

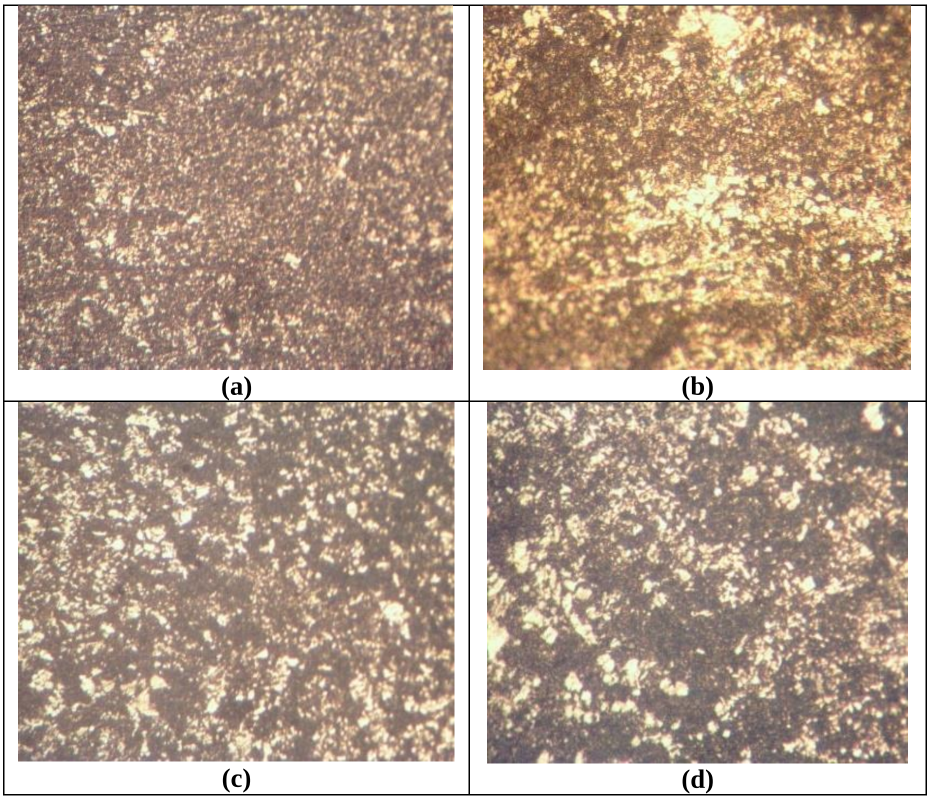


Fig 15 (a-d): Angle 105^o at 2nd Pass ×100, 2nd Pass ×200, 5th Pass ×100, and 5th Pass ×200 Respectively

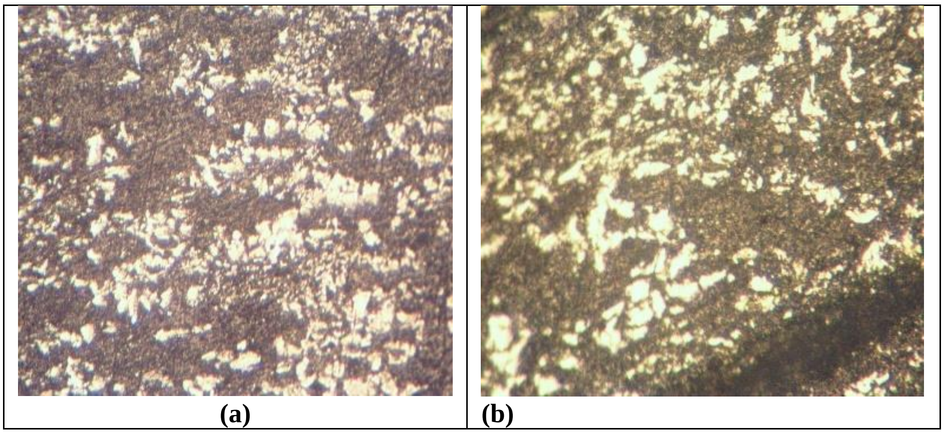


Fig 16 (a-b): Angle 45^o at 2nd Pass ×100 and 2nd Pass ×200 Respectively

The decrease in the punch force as number of passes increase, shown in the figures 8, as expected, were undoubtedly associated with lower hardness as passes increases, although the degree of decrease is approximately not the same. The hardness of the processed materials are connected basically to the internal changes in mechanical properties of the material, and the abrupt fall in this property after two ECAP pass, up to 5th pass, is probably caused by dynamic recrystallization in the material during the pass. The Pb recrystallization temperature is below 0°C 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 [3].

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. Dynamic recrystallization, as opposed to static recrystallization, the nucleation and growth of new grains occurs during deformation rather than afterwards as part of a separate heat treatment [8].

On the other hand, the punch force reflects not only the local strength changes, but also the overall strengthening effect associated with the second phase distribution in the material, as well as the dynamic recrystallization kinetics of the material.

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. Figure 12-16 shows the evolution of the precipitate distribution of Antimony alloy at 2nd and 5th ECAP passes for all angles.

One common feature of the microstructures is that in second pass broke some Antimony precipitates, as seen in all 2nd passes for all angles, but the degree of dissolution for angle 45^o is less when compared to others. 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 for all angles at 2nd pass.

Further straining seems to reduce the size of the Antimony particles, at fifth pass for angles 60^o, 75^o, 90^o and 105^o, precipitates were broken further into smaller ones. It is here important to note that the complete breakup of the control sample is possible with further straining. That is, a certain number of ECAP passes is necessary to completely redistribute the second phase Antimony structure. This indicates that ECAP caused some dissolution of the Antimony-rich phase similarly to the reports in the literature for precipitates in an Al-Fe alloy [5].

4.0 CONCLUSION

Lead alloy processed by Equal Channel Angular Pressing, up to 5 passes using route C, with different dies at varying extrusion angles gave the following behaviours:

The extrusion force reduced with increased strain level (number of pass) for all angles. Hardness of the control sample reduced with increased number of extrusion passes for all angles as a result of dynamic recrystallization of the material. However, Impact test showed that all angles absorbed their least amount of energy at 5th pass.

The Antimony-rich precipitates in the control sample undergo 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 5th passes suggests more strains are required for the complete break-up of the original microstructure.

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