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Experimental and FEM Investigation of ECAE on the Mechanical Properties, Extrusion Pressur and Microhardness of Pure Lead

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Abstract: The main aim of this study is to investigate the effect of Equal Channel Angular Extrusion (ECAE) process on the mechanical characteristics, microhardness and the extrusion pressure. Equal Channel Angular Extrusion (ECAE) process is a technique used for imparting a large plastic deformation to materials without a resultant decrease in cross sectional area. The pressures required for extrusion were measured, and the mechanical properties, microhardness and extrusion pressure for lead were evaluated. ECAE die was designed and manufactured in order to carry out this study, ANSYS software was used to investigate stress, strain, strain energy and displacement based on FEM. It was found that the extrusion pressure is decreased as the number of passes increased, where the microhardness is also decreased as the number of passes increased. But the mechanical characteristics have been enhanced by 33.5% after the second pass. The lead material condensed and become rigid as the number of passes increased. This mentioned that the ECAE could be used to have the super plastic behavior in materials, so the max force was reduced by 60% after the third pass of extruding pure lead.

Key words: ECAE, maximum extrusion pressure, microhardness, lead

INTRODUCTION

Extrusion is one of the most important metals forming processes due to its high productivity, lower cost and increased physical properties. There has been a considerable interest in the investigation of the effects of die geometry and other extrusion parameters on the structure, flow pattern, extrusion pressure and mechanical properties of shaped sections (Goswami *et al*., 1999; Pacanowski and Zasadzinski, 1998). Severe Plastic Deformation (SPD) processes have been receiving increasing attention as methods to develop fine-grained microstructures. The most attractive Is Equal Channel Angular Pressing (ECAP) or extrusion (ECAE). This technique provides large cumulative strains in material without changing the cross sectional area of the workpiece. This is available after extruding the specimen many times through a specially designed die having two equally sized channels connected at a finite angle (Segal, 1995; Aida *et al*., 2001; Nakashima *et al*., 1998; Nakashima *et al*., 2000). The grain sizes obtained by ECAE are generally in the sub-micrometer range (Chang *et al*., 2000). Therefore, ECAE processed materials may be attractive for different applications, mainly those needing super-plastic behavior and/or enhanced mechanical properties, i.e., fine-grained microstructure (Vinogradov *et al*., 2005; Urrutia *et al*., 2005). In ECAE, it is possible to rotate the specimen around its longitudinal axis between successive passes, creating then different processing routes (Stolyarov *et al*., 2001; Iwahashi *et al*., 1997; Zhu and Lowe, 2000). There are four basic routes involving different slip systems during the pressing operation so that they lead to significant differences in the microstructures produced by ECAE (Segal, 1995; Nakashima *et al*., 1998; Stolyarov *et al*., 2001). In route A, the sample is pressed many times without rotation; in route BA the sample is rotated by 90º in alternate directions between two consecutive passes, in route BC (simply designated by B) the sample is rotated by 90º in the same sense (either clockwise or counter clockwise) and in route C the sample is rotated by 180º. Various combinations of these routes are also possible, such as combining routes BC and C by alternating 90º and 180º rotations, but in practice the experimental evidence obtained to date suggests that these more complex combinations don't lead to additional improvement in the mechanical properties of the as-pressed materials (Nakashima *et al*., 2000). Although the accumulated strain, i.e. the number of passages, is the main factor which determines the strength level achieved,

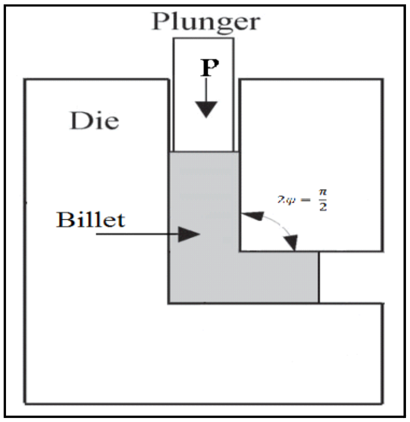


Fig. 1: ECAE process

few works seem to reveal a non-negligible effect of the processing route on the obtained properties. (Muñoz-Morris *et al*., 2006) have shown in Al-TiAl composites produced by sintering and ECA-extruded via routes A and C that the increase of the hardness and of the flow stress is faster in the material processed via route A, where finer grain sizes are achieved after two and four passes. Materials deformed by route C reached higher hardness even though the grain sizes achieved were somewhat larger than those of route A. Furthermore, they found that route A lead to high angle boundaries, whereas route C lead to many lower angle boundaries arranged as dislocations walls. On the other hand, the threshold value of the flow stress is reached at N = 2 in route A and at N = 4 in route C indicating probably a higher efficiency of deformation accumulation in route A. The authors (Muñoz-Morris *et al*., 2006) suggested that grain size is not the only parameter controlling the material strength. Although the microstructure obtained after ECAE is supposed to be maintained before heating, few works showed that recovery may take place even at low temperatures due either to the high dislocation density introduced which enhances annihilation processes, or to the non-equilibrium state of fine grained microstructures (Van *et al*., 1999). Extensive researches have been carried out on the microstructure development and on the mechanical property evolution during ECAE in several materials. Aluminum and light alloys are usually of particular interest due to their low density. Their strength is improved by ECAE but usually with a dramatic loss in ductility. It has been shown (Iwahashi *et al*., 1996) in aluminum containing a very low volume fraction of Al8Fe2Si precipitates (which form even at low Fe and Si contents), that the microstructure depends on the observation planes with an important increase in the yield stress and in Vickers hardness. The deformation of the workpiece during each ECAE pass can be reasonably approximated as simple shear on the intersection plane of the entry and the exit channels and hence depends mainly on the die angle (2ö) between the two channels as shown in Fig. 1.

Little work has been done on lead extrusion using ECAE, Therefore the main objective of this study is to design and manufacturing of ECAE die, then it will be used to investigate the effect of ECAE on the mechanical properties, hardness and extrusion pressure.

MATERIALS AND EQUIPMENT

Material: The study was conducted at Tafila Technical University during the study year 2010. different materials were used, namely AISI D2 tool steel, brass, and pure lead.

AISI D2 tool steel: AISI D2 tool steel is an air hardening, high-carbon, high-chromium tool steel possessing extremely high wear resisting properties. It is very deep hardening and is practically free from size change after proper treatment and this tool steel is characterized by High wear resistance, High compressive strength, Good through-hardening properties and High stability in hardening. The chemical co m position is shown in Table 1.

Mechanical properties: High-carbon, high-chromium steels such as D2 tool steel achieve their excellent wear resistance due to a chemical balance which renders them notch sensitive and low in ductility. Meaningful tensile data are unavailable. The practical experience indicates that compressive loads in excess of 2758 MPa can be withstood if evenly applied at low rates of loading.

Heat treatment process:

Hardening: D2 tool steel is extremely sensitive to overheating during hardening. It is therefore imperative

Table 1: Chemical composition of AISI D2 tool steel

AISI D_2 steel	C	Si	Mn	Cr	Mo	V	Fe
wt %	1.55	0.3	0.4	11.8	0.8	0.8	Bal.

that care be taken to insure that the hardening temperature is within the recommended range of 982/1024ºC. If overheated, D2 tool steel, like other high-carbon, high-chrome tool steels, will not reach its maximum obtainable hardness and will shrink badly. Don't overheat it. Without preheating, place the tool right in the hot furnace and let it heat naturally until its color uniformly matches the color of the thermocouple in the furnace. Tools should be soaked at temperature 20 min plus 5 min for each inch of thickness, then quenched in air. Control of decarburization can be achieved by using any one of the several modern heat-treating furnaces designed for this purpose. If endothermic atmospheres are used a dew point between -6.7 to +4.4ºC is suggested. In older type, manually operated exothermic atmosphere furnaces, an oxidizing atmosphere is required. Excess oxygen of about 4 to 6% is preferred. If no atmosphere is available, the tool should be pack hardened or wrapped in stainless steel to protect its surface.

Tempering: D2 tool steel has two toughness peaks, one at 232ºC and the other at 371ºC. For the best combination of toughness and hardness, temper at 232ºC. While this is the best tempering temperature for practically all applications, greater ductility can be obtained by tempering at 371ºC, although there will be some sacrifice of hardness. Double tempering is desired with the second temper 13.8ºC below the first temper. The hardness curve for D2 tool steel shows the same "kickback" or secondary hardening found in high-speed steels. In this material, it occurs at 538ºC and, if by accident tools have been overheated in hardening, causing shrinkage and loss of hardness, they might be salvaged by tempering them at 538ºC. They will regain some of their lost hardness and will expand close to their former size. The hardness values given in the chart at 538ºC are based on 1 h soaking time. Longer soaking time will result in somewhat lower Rockwell C hardness. Since D2 tool steel maintains a high hardness after a 538ºC temper, it lends itself well to gas nitriding or liquid cyaniding. This provides added wear resistance for forming tools. D2 steel is recommended for tools requiring very high wear resistance, combined with moderate toughness (shock-resistance), and may be used in Blanking dies, Forming dies, Coining dies, Extrusion and Drawing dies.

Lead: Is a main-group element with symbol Pb and atomic number 82, Lead is bright and silvery when freshly cut but the surface rapidly tarnishes in air to produce the more commonly observed dull luster normally associated with lead. It is a dense, ductile, very soft, highly malleable, bluish-white metal that has poor electrical conductivity. This true metal is highly resistant to corrosion, and because of this property, it is used to contain corrosive liquids (e.g., sulfuric acid).

Fig. 2: Brass mold

Equipments: In order to carry out the experiment procedure, the following machines and tools are used:

- EDM wire cutting type (EDGE 12)
- CNC milling machine type (MORI SEIKI, mv-55)
- CNC lathe machine type (MORI SEIKI, L200)
- Brass molds were manufactured to be used in the preparation of test specimen as shown in Fig. 2

TESTING AND EXPERMANTAL PROCEDURES

Testing:

Microhardness test: Micro hardness is the property of a material that enables it to resist plastic deformation, usually by penetration. However, the term hardness may also refer to resistance to bending, scratching, abrasion or cutting. The microhardness test was carried out on microhardness Vickers tester type (model HWDM-3) at 50 gf.

Extrusion test: The manufactured ECAE die was used to perform the extrusion test using instron testing machine of 100 KN capacity at $1\times10^{-3}s^{-1}$ strain rate as shown in Fig. 3.

Compression test: The prepared specimens were subjected to compression test using Quasar 100 Universal Testing Machine with 100 KN capacity at $1\times10^{-3}s^{-1}$ strain rate. The load-deflection curve was obtained for each extruded specimen from which the true stress-true strain curve was determined. Four tests were carried out on each specimen (original, one pass, two pass and three pass).

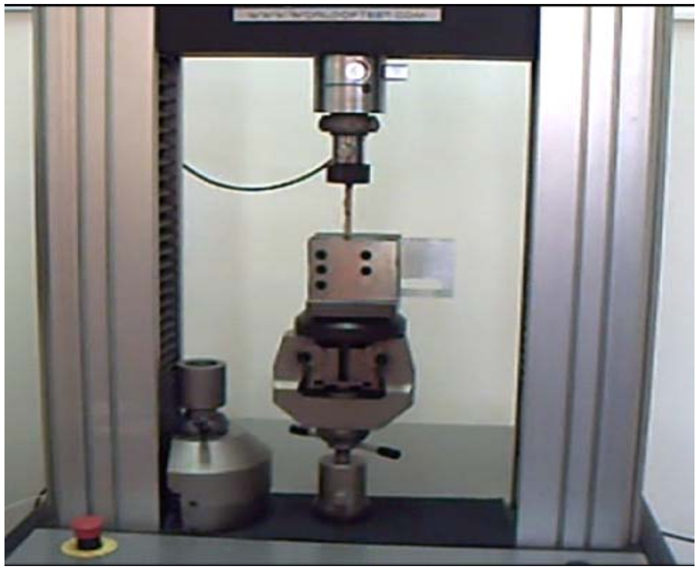


Fig. 3: Instron testing machine (Quasar 100)

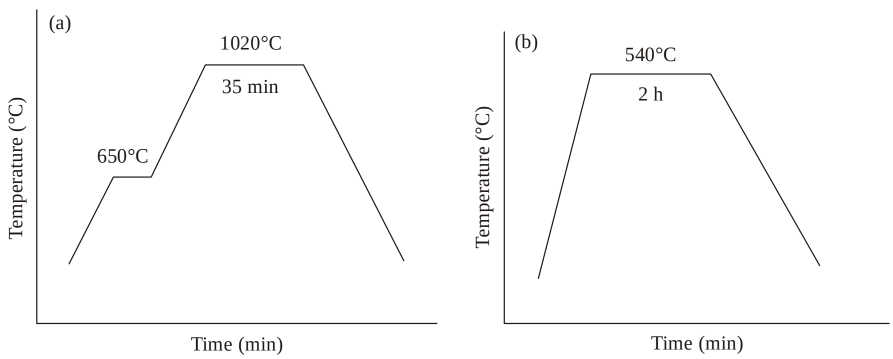


Fig. 4: (a) Hardening diagram (b) Tempering diagram

Experimental procedures:

Design stage: The lead was used in this study to investigate the ECAE on its properties, where the properties of lead are: Yielding stress (σy) = 10.5 MPa, Fracture stress (σf) = 16 MPa, and the Modulus of elasticity (E) = 16 GPa Force analysis was carried out to determine the needed pressure to perform the extrusion test (Pacanowski and Zasadzinski, 1998). The effect of selected parameters of aluminum extrusion on temperature changes in the die system, Arch. Metall., 43 (4): 389-398.The material of die was chosen carefully to sustain the high pressure during this process, D2 steel is recommended for this purpose. Then the appropriate heat treatment was carried out. To ensure the design of die ANSYS package was used based on FEM analysis.

Manufacturing stage: The die was produced at (Japanese Institute of manufacturing molds-Jordan, 2010), the process of manufacturing die can be seen in appendix A. but the main issue in die manufacturing is the heat treatment that contain two main steps the hardening one followed by tempering

- **Hardening of parts:** The remaining time of the parts in the furnace was 35 min as shown in Fig. 8, according to the following equation:

  Remaining time for electrical furnace = (1.5-2) min/min of the smallest dimension parts
  The smallest dimension was 20 mm, so; (1.5-2) × 20 mm = 35 min

The hardening and tempering regimes are show in Fig. 4. Finally, by using the coolant oil of (50 -70)ºC, to get 60 HRC

- **Tempering of parts:** After tempering process the parts were remaining to cool in air, and the hardness was 55 HRC. All the above temperatures used in hardening and tempering processes were adapted from the material brochure:
  - Coincident with manufacturing of mold, the material of lead was melt at temperature of 400ºC and was cut into piece of mass 100 g and put in crucible in order entering the furnace for 30 min. Then the molted lead was casted in mold of brass shown in Fig. 2, after cooling the molten of metal in air media (about 15 min), the lead metal was removed from the mold of cast.
  - CNC milling machine at speed 1500 rpm and feed 100 was used to prepare the lead specimens to (6.35x6.35x60) mm.

Test stage:
Micro hardness test:

- By applying the tests on the casted lead, and record the results
- As the mold was ready, the angular extrusion test on three lead samples was applied (A grease lubricant and a strain rate 1×10^{-3} s^{-1} were used) as the following:
  - First sample was extruded one pass
  - Second sample was extruded two passes
  - Third sample was extruded three passes
- By taking the extruded samples and applying the tests on them (tensile test and micro hardness)
- Finally, getting the results and comparison the differences between the samples before and after angular extrusion test

THEORETICAL ASPECTS AND CALCULATIONS

Force calculation:

$$F_{tot} = F_{shear} + F_{fw} \qquad \text{(Dieter, 1986)} \quad (1)$$

where, F_{shear}: force required for shear deformation, F_{fw}: force due to friction between die wall and outer surfaces of specimen (before entering deformation zone)
But:

$$F_{shear} = A\, \sigma_m\, \varepsilon \qquad (2)$$

$$F_{fw} = \mu\, \sigma_m\, A_o L \qquad (3)$$

where,
A: Area of shear, σ_m: mean flow stress, ϵ: strain, μ: coefficient of friction
A_oL_o: surface area before deformation zone

Hence, $A = 6.35 \times (6.35^2 + 6.35^2)^{0.5} = 57.02\ mm^2$
$A_o L_o = 6.35 \times 4 \times 55 = 1397\ mm^2$

$$\sigma_m = \frac{1}{s_a - s_b}\int_{s_b}^{s_a} \sigma d\varepsilon \qquad (4)$$

where,
ε_a = Prior plastic strain given to specimen
ε_b = Total plastic strain experimental by the specimen of the end of the pass
$\varepsilon_a - \varepsilon_b$ = Plastic strain important to specimen as a result of one extrusion pass

The true stress- true strain of pure lead is shown in Fig. 5 So:

$$\sigma_m = \frac{1}{0.03 - 0.2177}\int_{0.2177}^{0.03} \sigma d\varepsilon \qquad (5)$$

$$\sigma_m = 14.183$$

and

$$\varepsilon = \frac{2\cot\left(\varphi + \frac{\psi}{2}\right) + \psi \csc\left(\varphi + \frac{\psi}{2}\right)}{\sqrt{3}} \qquad (6)$$

(Segal *et al*., 1995)

where, $\varphi = \pi/4$, $\psi = 0$

So One ECAP-passing results only in the deformation of $\varepsilon = 1.15$ using a 90^o tool [16].

$$\varepsilon = \frac{2\cot\left(\frac{\pi}{4} + 0\right) + 0\csc\left(\frac{\pi}{4} + 0\right)}{\sqrt{3}} = 1.15$$

Hence,

$$F_{shear} = A\, \sigma_m\, \epsilon = 57.02 * 14.18 * 1.15 = 929.8\ N$$

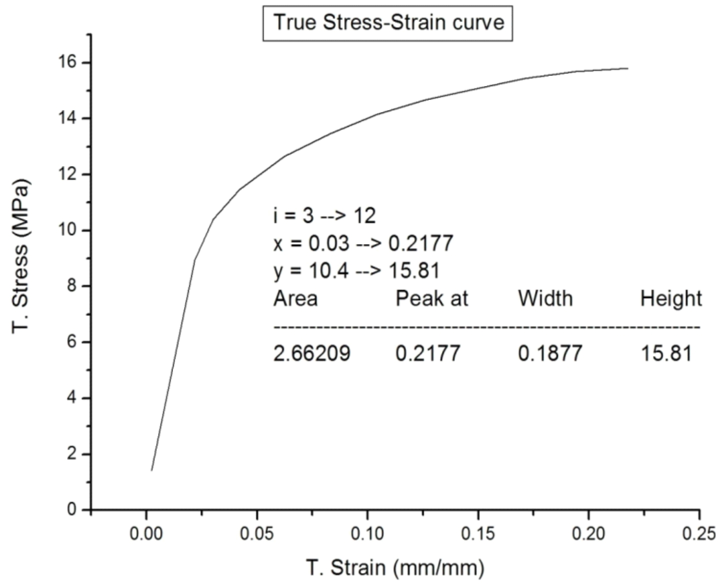
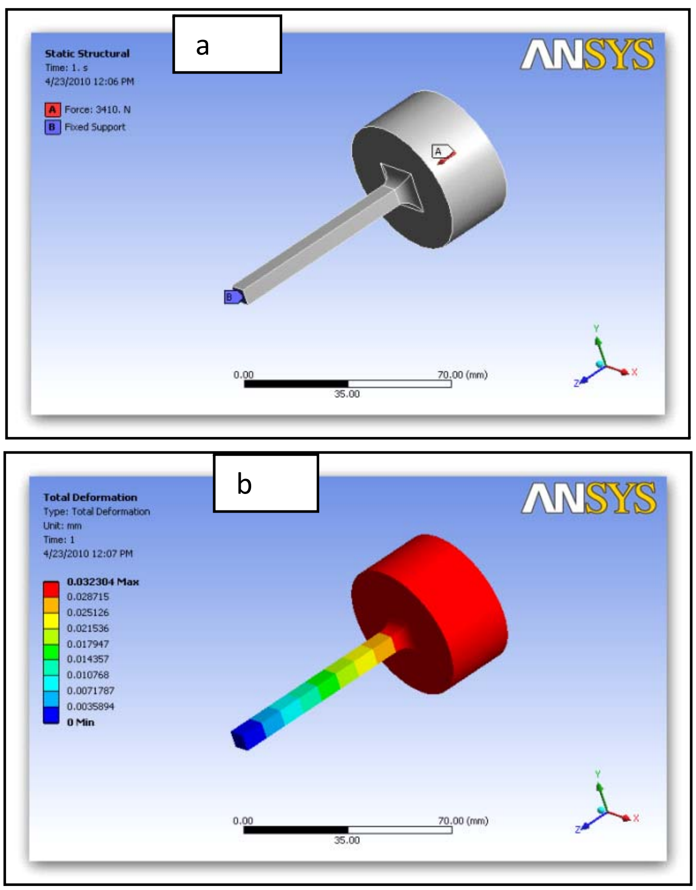
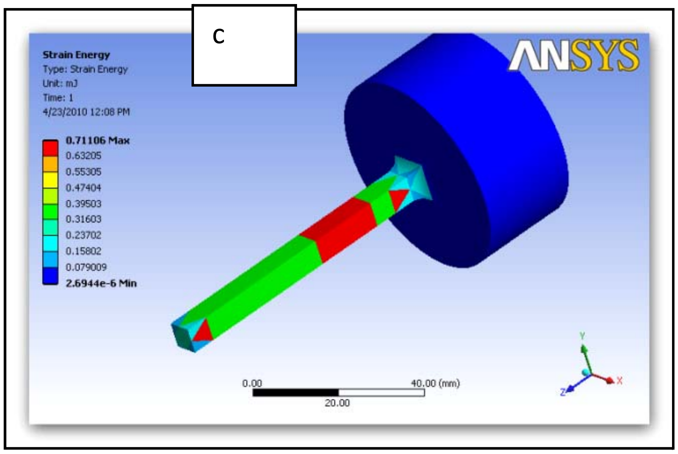


Fig. 5: True stress-true strain curve of lead





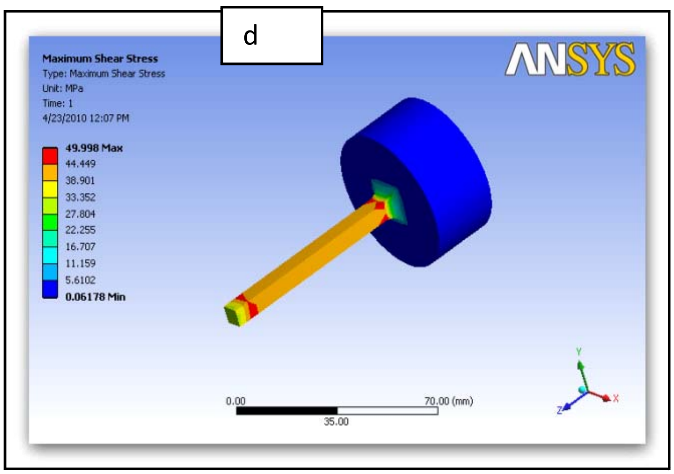
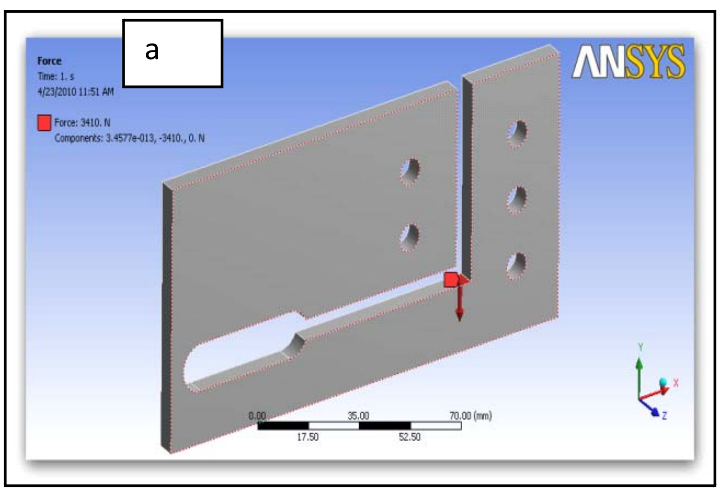


Fig. 6: (a) Static structural analysis, (b) total deformation analysis, (c) strain energy analysis, (d) maximum shear stress analysis



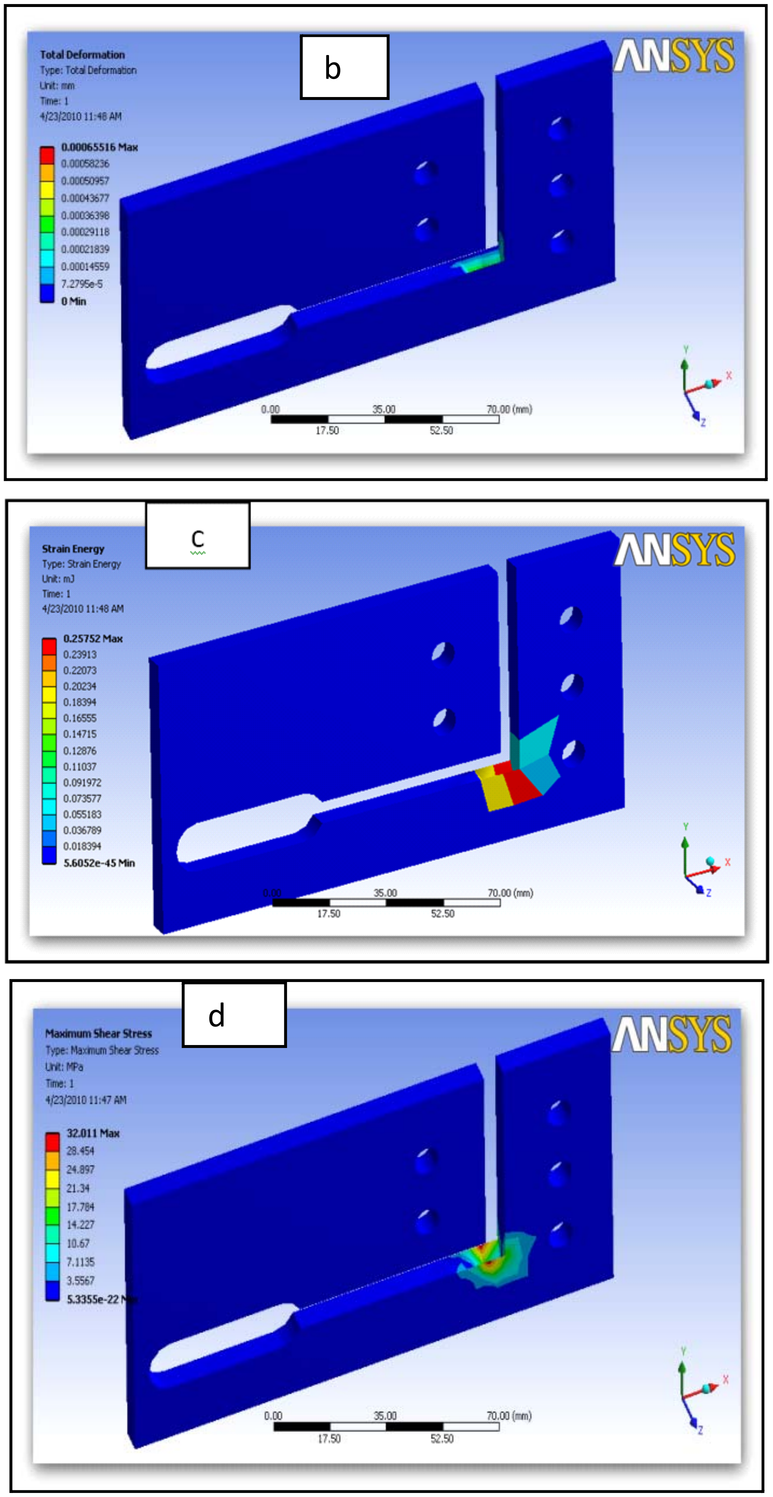
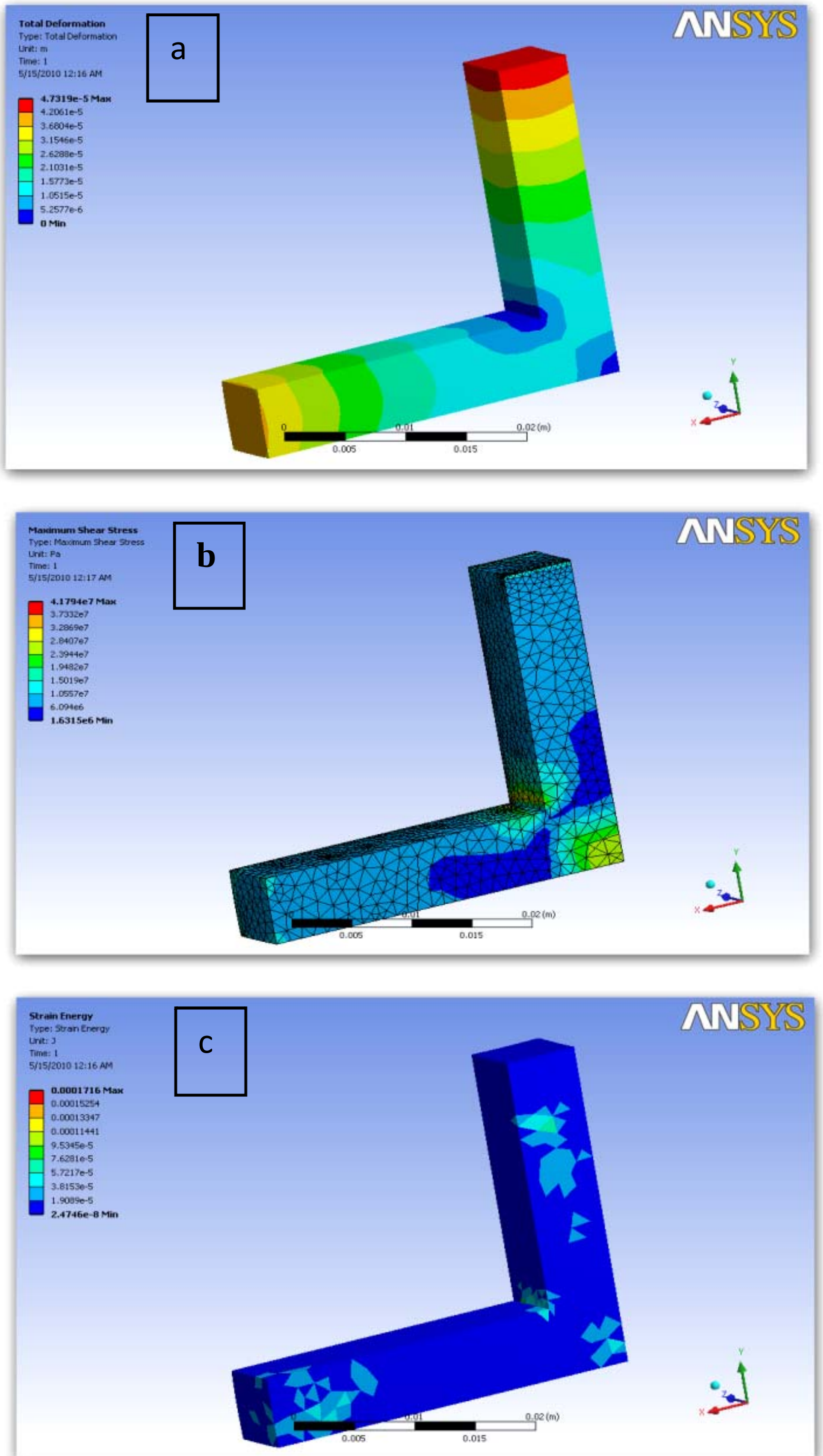


Fig. 7: (a) Force analysis, (b) total deformation analysis, (c) strain energy analysis, (d) maximum shear stress analysis


Total Deformation
Type: Total Deformation
Unit: m
Time: 1
5/15/2010 12:16 AM
4.7319e-5 Max
4.2061e-5
3.6804e-5
3.1546e-5
2.6288e-5
2.1031e-5
1.5773e-5
1.0515e-5
5.2577e-6
0 Min
a
ANSYS
0.005
0.01
0.015
0.02 (m)
Maximum Shear Stress
Type: Maximum Shear Stress
Unit: Pa
Time: 1
5/15/2010 12:17 AM
4.1794e7 Max
3.7332e7
3.2869e7
2.8407e7
2.3944e7
1.9482e7
1.5019e7
1.0557e7
6.094e6
1.6315e6 Min
b
ANSYS
Strain Energy
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0.0001716 Max
0.00015254
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c
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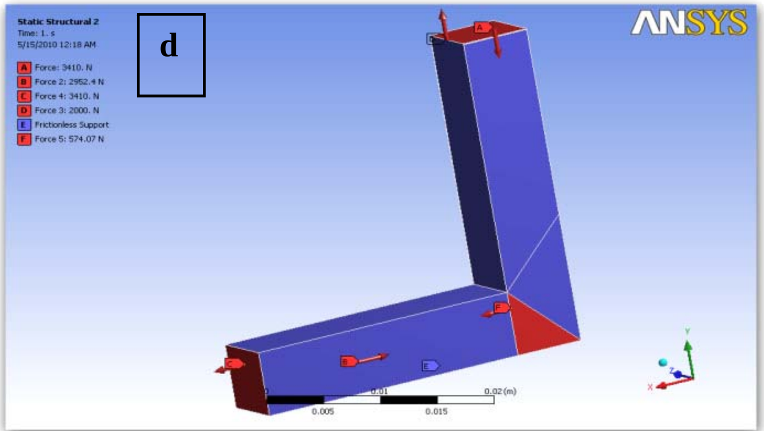
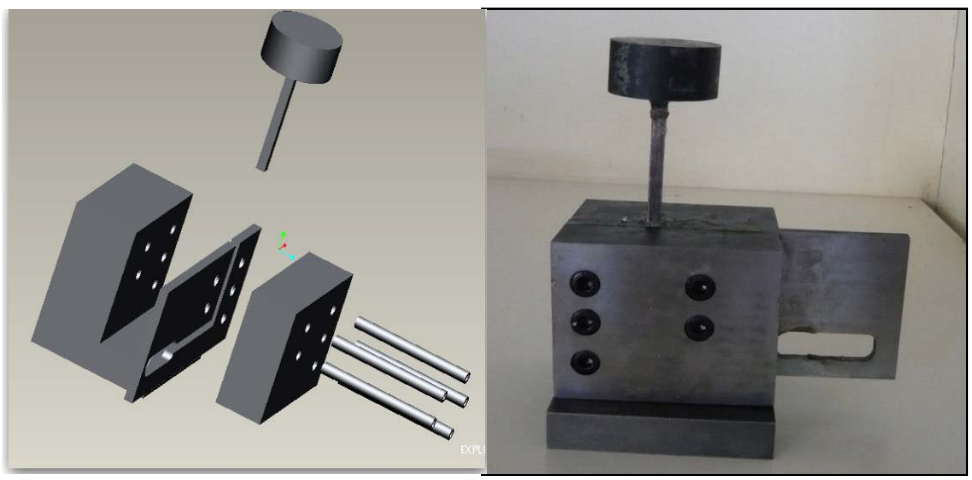


Fig. 8: (a) Total deformation analysis, (b) maximum shear stress analysis, (c) strain energy analysis, (d) static structural analysis



(a) (b)

Fig. 9: (a) Disassembly of ECAE die, (b) Assembly of ECAE die

$$F_{fw} = \mu\ \sigma_m\ A_o\ L = 0.25 * 14.18 * 1397 = 4952.37\ \text{N}$$
$$F_{tot} = F_{shear} + F_{fw} = 574.07 + 5403.7 = 5882.17\ \text{N}$$

FEM analysis: The finite element analysis of equal channel angular extrusion die was studied using ANSYS software. Different values were investigated such as: total deformation, strain energy, equivalent stress and maximum shear stress. The analysis was carried on plunger, die, and workpiece.

Plunger analysis: It was obvious from Fig. 6 that the maximum shear stress occurred at the beginning and at the end of the plunger. This will be considered during the manufacturing process.

Central die analysis: The FEM analysis for the central die is shown in Fig.7.

Sample analysis: The sample FEM analyses are shown in Fig. 8. It clear that maximum stress occurred at corner.

CAM of the designed die: Both the assemble and disassemble of the extrusion die are shown in Fig. 9.

RESULTS AND DISCUSSION

Effect of ECAE on the microhardness of pure lead: From Fig. 10 it can be seen that the microhardness decreased as the number of passes increased. This can be

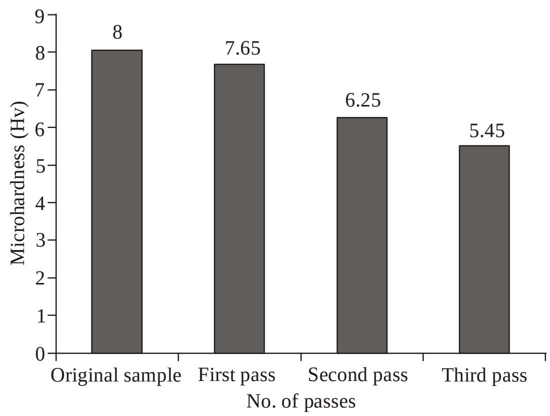


Fig. 10: Microhardness of pure lead

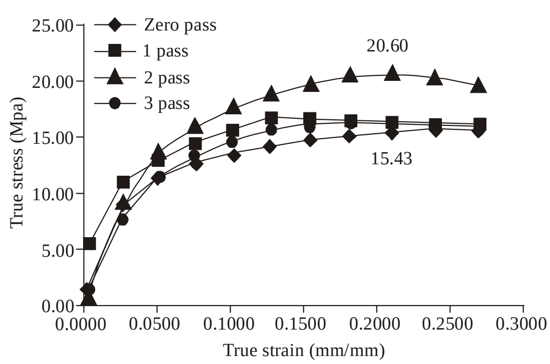


Fig. 11: True stress - true strain diagram

attributed to hot extrusion forming of pure lead. The maximum reduction in hardness is 31.8 % that achieved after the third pass.

Effect of ECAE on the mechanical properties of pure lead: It was obvious from Fig. 11 That there is an enhancement in mechanical characteristics, however the maximum enhancement was 33.5% of flow stress at 0.2 strain that achieved after pass 2.

Effect of ECAE multi passes on final length of pure lead: Figure 12 shows the final length after each pass, the length after pass is sharply decreased where the decreasing in length restricted after pass2. The maximum reduction is 95 % that achieved after the third pass. This is due to high vacancies and defect in casted lead materials. EACE may recommend having a condensed and rigid structure.

Fig. 12: Length of extruded sample

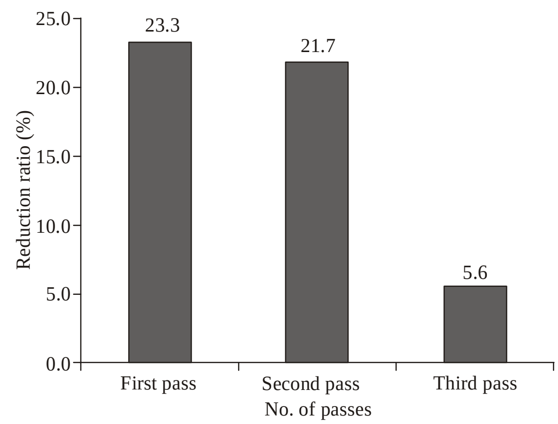


Fig. 13: Length reduction ratio of the specimens

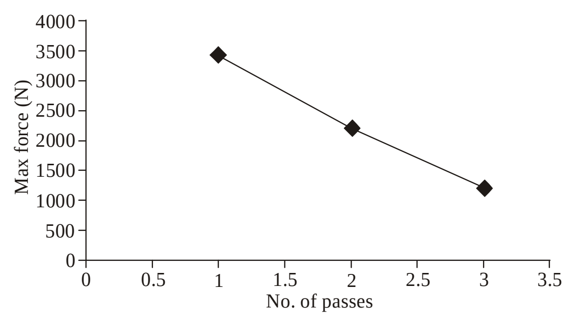


Fig. 14: Maximum extrusion force vs. number of passes

Effect of the number of passes on length reduction ratio: From Fig. 13 that the length reduction ratio is a simple indication about the amount of length reduction between different passes. It obviously shown that the length ratio is decreased as the number of pass increased, the maximum ratio was 23.3% that achieved after the first pass.

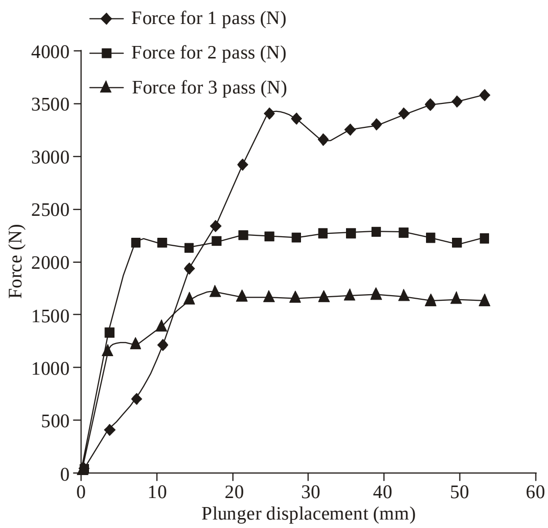


Fig. 15: Plunger force - plunger displacement

The effect of the number of passes on the maximum extrusion force: Figure 14 illustrates the relationship between the maximum extrusion force and the number of passes, the maximum force decreased as the number of passes increased; the maximum reduction is 60 % that achieved after pass 3, where the real autographic record is shown in Fig. 15. Also it was observed that the maximum extrusion pressure decreased as the number of passes increase, this indication that the material has a super plastic behavior.

Where the mechanical properties are decreased as the number of passes increased as shown in Fig. 15.

CONCLUSION

The following can be concluded:

- The maximum force decreased as the number of passes increased.
- The final length after pass 1 is sharply decreased where the decreasing in length restricted after pass2. This due to high vacancies and defect in casted lead materials.
- The microhardness decreased as the number of passes increased. This can be attributed to hot extrusion forming of pure lead. Also it was observed that the maximum extrusion pressure decreased as the number of passes increase, the material become a super plastic material.
- There is an enhancement in mechanical characteristics; however the maximum enhancement was 33.5 % of flow stress at 0.2% strain that achieved after pass 2.

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