Enhancement the Microstructure and Mechanical Properties for Pb-Sn-Sb Alloys by Using Equal Channel Angular Extrusion

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Abstract
This study aims to enhance the compression strength in one type of Pb-Sn-Sb alloys which wellknown by (Babbitt-ASTM B23 Alloy 13). The processing doing via equal channel angular extrusion technique. Three casting were implemented to manufacture the alloy; Chill Casting (CC), New Rheocasting (NRC) and Gravity Die Casting (GDC). The microscope examination shows that the microstructures contain two phases, α-Pb and cubic shaped intermetallic compound (β-SbSn) in a matrix of ternary phases. CC was fine equiaxed as well as NRC, while in GDC was a dendrite α-Pb phase with remaining β-SbSn phase as a cubic shape. Higher mechanical properties in compression were recorded for Gravity die casting (12.7 %) while the NRC registered the highest value in yield strength (11.7 %). On the other hand, the casting techniques had a slight difference in Young's modulus. The other resulted data like hardness showed that NRC is the first reading (12.55 %) and then gravity casting recorded as second score comparing with other (11.79 %), The results showed also that increasing forming temperature during angular extrusion has an adverse effect on compression strength. The study concluded that microstructural change caused by ECAP softens the material due to the break-up the original precipitate and accelerate from the dynamic recrystallization.

Keywords: Babbitt, ASTM B23, Lead alloys, Equal Channel Angular Extrusion

1- Introduction
Equal Channel Angular Extrusion considered as one of newly distinguished forming techniques used for producing ultra-fine grain materials. Obtaining fine grain means improving mechanical and physical properties that destine them for a commercial use [1],[2]. Principally, it can be defined as simple shear deformation achieved by pressing the workpiece through a die containing two channels symmetrical in cross-section and meet at a predetermined angle. The deformation occurs near the plane lying at the intersection of two channels; the processing will do by using particular tool geometries (set of dies) prevent the free flow of material and thereby produce a significant hydrostatic pressure. The presence of high hydrostatic pressure in combination with large shear strains is essential for creating dislocations. Mainly, high densities of a crystal lattice that can provide a significant refining of the grains; the grain refinement according to the Hall-Petch relationship is considered an effective method to attain both the preferred strength and ductility at ambient temperature [3]. The white metal or babbitt has sizable advantages as a bearing material, and it's suggested as the first choice for most bearing applications. The bearing is usually designed to support the heaviness of the shaft while providing a non-damaging surface for the high rotating shaft, in the event of failure or contamination of over time, the softer Babbitt alloy consume before the journal or shaft [4]. The Babbitt or bearing materials is a part of term Pb-Sn-Sb series of alloys which involve tin as well as lead-based alloys [5]. Lead-based white metals show a lower friction coefficient, better bonding to the shells, and better properties for casting and extrusion than the tin-based alloys. These results marked it in high status to manufacturing the bearings since its shearing stress of the soft matrix is relatively low [6].

2- Experimental Producer.
Material Preparation
The materials used in this experiment was sold from Roto Metals. The ingots of Babbitt (ASTM B23 Alloy 13) produced later by Three casting techniques Gravity Die Casting (GDC) and New Rheocasting (NRC) and Chill casting (CC). Gravity die was made of AISI H13 steel 60x60x120mm with the thickness of 20mm. Two thermocouples K type with 3mm in diameter stainless steel sheath covered were employed. The first thermocouple was used to measure the pouring temperature, and other was fixed in the mould wall through the tip to ensure confident contact between the sensor and the molten metal to determine cooling rate through solidification. The molten metal, in this case, was poured at a temperature of 370°C then left to...
solidify at room temperature [7]. New Rheocasting were produced in mold made of AISI 304 austenitic stainless steel of 60x60x120mm. The die was inclined at an angle of 75° for the purposes increasing the contact area among melted and die walls and to give a good circumstance for crystals to creating on the wall and increase the distance of metal stream which helps in increasing the chance of extrication the freezing crystals from the die wall [8]. The molten metal poured at temperature of 265°C on the wall of the inclined mould. When the temperature got approximately 230°C (this temperature at liquid-solid interface) the mould was water cooled (quenched), which promotes the formation of casts with a dense, fine-grained structure and, therefore, good air tightness and superior physicochemical properties [9]. The Chill casting techniques used Water-Cooling (WC) for Oxygen Free High Conductivity (OFHC) copper (99.95%C) [10], the thickness of the mould walls was 20mm evenly from each direction, contain groves open head for propuse of flow. the produced billet (65 mm length * 15 mm²). In the water-cooling technology, the molten metal was poured into direct water-cooled mould at temperature 370 ºC then left to solidify to room temperature.

Hardness Readings
The microhardness test was carried out using Vickers hardness tester. The test load and dwelling time were 100g and 10 Sec. respectively according to ASTM -E384. Microhardness measurement selected at four positions from the outer to inner. Brinell Hardness was implemented at each position with a 2.5mm in diameter and load of 6.25kgf, Three impressions were made then middling for eadings was register, the test caring at room temperature (20-30°C).

Microstructure Characterization
Samples from as castings and extruded billet cutting in vertically section (Figure 1). Four regions were chosen to be investigated, at 1, 2, 3, and 4mm in a direction from outer edge to inner center. Each position has a distance 2.5 mm in X and Y plans from a previous point edge.

Billet Perparation
The leading end of the billet was ground into a hemispherical shape using coarse grinding on a grinding table with water as a lubricant. This grinding method was chosen in order to prevent heating by the friction of the specimen. This hemispherical shape was necessary to reduce stress concentrations as the billet entered the bend in the ECAE die. The entire billet was polished with 1200 grit SiC paper and then cleaned with a non-abrasive soap and lubricated by MoS₂ before being subjected to deformation in the ECAE die. It was necessary to reshape and re-polish the billets after each pass because of the spring-back associated with the processing, which makes the processed billet diameter to be slightly larger than the original one. This reshaping and polishing of the billet were also done using water to eliminate the chance of friction heating of the specimen.

Equal Channel Angular Extrusion:
Figures (2) shows the set of dies that used in extrusion. The extrusion process was performed using a hydraulic press (maximum force: 120 KN, maximum stroke: 150 mm). One “K” type thermocouples were inserted to monitor the temperature evolution of extrusion dies. It prepositioned into the die at 5 mm to insure it nearest as much as possible from the internal edge of channel. The heating system consisted in 4 Industrial pin heater at 650-watt power to ensure a uniform temperature in along the channel path. The temperature was measured using a specially calibrated chrome-alumel (K-type) thermocouple connected to a portable thermometer.

Production Operation
In this study, the preformed samples were reheated from room temperature to temperatures 100°C in an external electrical resisting furnace for 15 minutes to reach the targeting processing temperature of extrusion and to allow the temperature in the sample to became fully homogeneous. The punches were made 14.5 mm square cross section area with constant contact ratio of value 0.428, the purpose from above dimensions is to give some clearance. The punches were also made in it front fillet with radius 8mm to avoid any contact with the curvature of the channel in the walls of dies. The channels bend remained a constant 15 mm,
the parts of the die held together by sex 1/2" steel bolts. The ECAE process involves pressing a lubricated billet into six different dies vary between 75°, 90° I/II (die 90 II Inner Corner Radius Ri equal to Zero mm while in 90° I the inner corner 5 mm ), 105°, 120°, 135°. Several variables influence ECAE process, the primary ones being in this experiment was: feed rate, applied load, and temperature. In this research, the feed rate was fixed at 0.1 mm/sec. The applied load was varied as necessary by the hydraulic press to maintain the constant feed rate. Extrusion took place at constant temperature 100°C, the billet and die lubricated with graphite coating "high-temperature anti-seize compound MoS₂" to facilitate the extrusion and to lower as much as possible of friction during processing. The push-rod was also lubricated with MoS₂ compound and positioned in the die above the sample. The die halves were then fitted together and bolted into position. The rod was driven through its stroke by a hydraulic pressing machine. The Flash and galling still occurred sometimes in a limited amount, but were both easily removed by cleaning and re-polishing.

The billet was oriented in the die following the Bc route (This means that the billet was rotated in the same direction 90º between each pass), the selection for this route because this route has been shown to provide the most homogenous shearing of the microstructure.

Figure 2: Set of Dies used in Extrusion

Die 75°  Die 90° R 0  Die 90° R
Die 105°  Die 120°  Die 135°

Figure 3: Front View for Assembly of Extrusion

Die Holders  Heaters  Active Die Plate  Housing Plate

Processing up to 4-pass was attempted with each sample. Then specimens were water-quenched immediately after each finish the cycle of pressing, thus eliminating any chance of recovery at such elevated processing temperature.

3- Results and Discussion

In general, without additives of any element, the microstructure of lead-based Babbitt alloy 13 consists of two phases. The white is a phase of β-SbSn which like cubic shape in a ternary eutectic soft matrix formed from the dark phase α-Pb, white β-SbSn and Sn-rich solid solution which was also white. The microstructure obtained from chill cast was fine equiaxed grain for α-Pb phase rather than dendritic structure. The finest microstructure obtained in permanent mold cooling by water is due to the higher cooling rate for molten metal which was undergoing through casting. The average grain size near the walls rather than in the center of specimen is not much-appreciated difference, The average grain sizes of α-Pb are recognized around 14µm; Furthermore, the average cubic β-SbSn grain size was approximately 23µm to 38µm. While the microstructure in Gravity Die Casting showing different DAS sizes in the microstructure that obtained from this technique, The measurement of average DAS indicates that there is difference from the wall to center. from the wall, where they are 16, 25, 28 and 37 µm respectively. This increasing toward the center is due to the decrease in cooling rate with increasing the distance from the wall, The microstructure examination in NewRheoCasting showed fine cluster equiaxed grain from α-Pb phase rather than a dendritic structure with different grain
size. The average grain size at 2mm from the wall is roughly 13μm due to the high thermal gradient in cooling by water. Moreover, a low size fraction of β-SbSn with average size equal 12μm was detected in fine eutectic. When it moved toward 7mm from the wall were recognized 19μm and 14μm grain size of α and β. The Average grain size sizes of α and β became between 21μm and 16μm respectively with the eutectic coarseness increases at distance of 15mm from the wall was shown. Table (1) shows the mechanical properties of alloy 13 produced by four casting techniques. The figures token at deformation 25% reduction from the total length of test specimens (25±1mm). Higher mechanical properties in compression are recorded for Gravity die casting (12.7%) while the NRC registered the highest value in yield strength (11.7%), on the other hand, the casting techniques have a slight difference in Young's modulus. The other resulted data like hardness showing that NRC is the first reading (12.55%) and then gravity casting recorded as second score comparing with other (11.79%), while registered s received as cast the minimum one. Between GDC and NRC curves located chilling curve but with not appreciated difference on NRC. Young moduls which the relationship between stress (force per unit area) and strain token under above constrains (within proportional deformation). It is so clear from the statistics that the technique of equal channel could successfully to refine the grain of β-phase to average 3.24μm, 7.91μm near the walls and centers respectively, this significant of refinement is due to hardness ship of these grains rather than other phases. It is good to know here that this phase has grain size before the processing reach to 23.84μm in average. Also, it could notice that angle 105° give up the most equilibrium distribution for this phase above the cross-section of specimens, this may be justifying to us why that this angle has the highest value in compression strength if we compare with others angles. In the same time, it could notify that grains near the walls are always smaller than the then center, this is because of undergoing higher cooling rate when the processing is finish. However, the differentiation in the sizes is not exceeded 4.67μm which is normally considered if we take into our consideration the size and the shape of the billet. Related to phase α-Pb, which is varying its shape between fine equiaxed and clustering in the course and fine dendritic shape. The common characteristic that distinguishes this phase in all conditions that the grains of lead have the ability to growing up to different sizes depend on its distance from the walls, and it seems there no significant effect for the equal channel on refining its structure. This is due to low recrystallization temperature of Pb which consider within room temperature. This gives us clear answer why we got down in results of compression strength. In general, the possibility of elongate Pb grain with increase the temperature within the direction of extrusion seems to be effected negatively on the results. We conclude from the above, It successes in manage the defragment the beta phase, but at the same time we could not control on growth the alpha phase, so we now have two structures but un-equilibrium in grain sizes and entirely different comparing with it essential cases. Also, it concludes that the boundaries of using a die of 105° with route Bc and slow strain give us the best results because its ability to produce most equilibrated and smallest grain sizes possible among others dies.

Table (1): Mechanical properties of ASTM B23/13 At room temperature

<table>
<thead>
<tr>
<th>Materials</th>
<th>Properties</th>
<th>σc MPa</th>
<th>σy MPa</th>
<th>HB Kgf/mm2</th>
<th>E GPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>As Received ( Ingot )</td>
<td></td>
<td>84</td>
<td>59</td>
<td>16</td>
<td>30(±2.95)</td>
</tr>
<tr>
<td>GDC</td>
<td></td>
<td>107</td>
<td>63</td>
<td>19</td>
<td>30(±1.8)</td>
</tr>
<tr>
<td>NRC</td>
<td></td>
<td>94</td>
<td>69</td>
<td>20</td>
<td>30(±0.1)</td>
</tr>
<tr>
<td>CC</td>
<td></td>
<td>98</td>
<td>61</td>
<td>17</td>
<td>30(±0.2)</td>
</tr>
</tbody>
</table>

Vickers Micro Hardness Distribution for Chill Specimens

Vickers Microhardness measurement (MHV) at four positions was recorded from the outer edge to center for chill casting samples. From the figures (4) It conclude, That the Vickers Micro hardness registered higher readings nears the walls more than the center in all cases except the die of 75° which register limited reverse values, the reasons behind this fact due to refining the grains nears walls and the coursing towered the centers, while in the last case is due to long path of inner curve which mean more of heat exchange between the billet and the mould. The heat has negative effect and cause to coursing and dendrite the particle of α-Pb and then not homogenies the microstructure. This is connected by why the billets that extruded by this specific die have the lowest value among other set of dies. however, the differentiations between the readings in same group not exceed 4 HV0.1 which consider reasonable in Vickers Hardness.
scale, it concludes from the hardness study that the equal channel have a great impact on homogenies the hardness along cross section of extruded billet, this is the main advantage of this technique rather than refining the grains.

**Figure 4**: Vicker's Microhardness for Extruded chill casting Specimens

**Figure 5**: Evolution of the Force Required for Equal Channel Extrusion for chill casting technique

**Figure 6**: Universal Compression Flow Curves for Five Different Extrusion Angles for Chill Specimens
Evolution of the Force Required for Equal Channel Extrusion for chill

The results show that the force required for Equal Channel Extrusion Increase with decreasing the angle of extrusion for all extruded samples. It is clear that the maximum force is 29KN for chill Casting, at temperature 100°C, respectively at room temperature the forces.
Effect of Extrusion Angle on Compression Strength for Chill Casting Specimens.

To compare between compression properties of ARAC, GDC, NRC and CC as castings and the extruded samples, compression tests were performed at room temperature. The primary \( \alpha \)-Pb and \( \beta \)-SbSn phases in all cases are small at the surface and gradually increase toward the center region of the part. In the center, we can clearly see the grain size is bigger and thicker. It is also observed that at the surface, the primary \( \alpha \)-Pb is elongated in the direction of extrusion and the \( \beta \)-SbSn phase is fragmented to small parts. It obviously is seen that the particles near the wall underwent severe plastic deformation in all cases, but the fine grains indicate that recrystallization has occurred. These microstructure changes during the equal channel extrusion process are closely related to the angle of extrusion, the temperature of processing and strain rate. The extruded microstructures of conventional gravity die cast, the new cast and chill casting show that phases have semi-different distribution. However, modulate for at least three specimens were tested in each condition to provide a fitting curve. Stress- Displacement curves were re-drawing for compression test for chilled extruded specimens in five angles 135°, 120°, 105°, 90°, 75°. While it selected soft angle for others casting because strain hardening that accompanies to extrusion and it causes by cracking in early stages. All experiments for compression strength and hardness did at room temperature (20-30° C). Also, the study explained hardness ship of production by die 90 II ( Inner Corner Radius Ri equal to Zero mm ) as it produces elliptical billets that could not easy to re-preparing for next pass. The Variation between two angles could justify by that the metal flow in the second case was higher in the inner curve more than the outer curve that gives us this unique shape, it conclude there is must be found a proportional movement aspects between the inner and outer radius must be taken in our consideration of design.

The Effect of Extrusion Angle 135° Route Bc Low Strain Rate on Microstructure and Compression Strength for New Rheocasting

The microstructure examination showing that within the direction of extrusion on walls of billet surface that the microstructure was elongated \( \alpha \)-phase structures with average grain size of 24.28\( \mu \)m, while the \( \beta \)-phase was not clearly visible because of its refining to a low volume fraction of average size less than 4.77\( \mu \)m was observed in a fine eutectic. Average grain sizes of \( \alpha \) and \( \beta \)-phase increase Moreover within a layer of 2.5 mm deep from the wall surface of the specimen showing that of \( \alpha \) and \( \beta \) are 32.33 and 5.1\( \mu \)m, respectively; At a distance of 5 mm deep from the wall in a section perpendicular to the extrusion direction, \( \alpha \)-Pb has coarse dendritic; the DAS is 12.88\( \mu \)m. While the microstructure in the center of the specimen in showing that \( \alpha \)-Pb have a dendritic with DAS 14.6 \( \mu \)m whereas \( \beta \)-SbSn phase still appears in both locations as shapes of flakes instead its cubic shapes. The average grain size for \( \alpha \)-pb reach to 35.17 in the center of specimens. The \( \alpha \)-Pb remains elongated structure at all planes, directions, and distances that mean the effect of equal channel technique here some kind (low action) with new Rheocast if we are comparing with chill casting, the islands, as well as coarse elongation structures, lead us to negative results in mechanical properties of the alloy. The compression strength registers little lower reading rather than as casting, the amount of compression strength in limits of displacement 6.25 mm was 84.75 MPa. The hardness is also effected negatively after processed by the equal channel to register 17.3 HB. While the micro hardness gradient from 21.4 to 20.7 As Justification for the behavior of for the curve at it end stages , we can notice that the tail of the curve is still to raise-up and this because of behavior of this kind of soft metals that compressed; it does not eventually shear but continues to flatten out with increasing loads.
The Effect of Extrusion Angle 135° / Route Bc on Microstructure and Compression Strength for Gravity Die Cast

Figure (15) shows the change in microstructures with increasing the heating temperature of extrusion up to 100°C for GDC angle 135° and under condition 0.1 strain rate. The grain sizes of α-Pb were elongated up to the twice time approximately to register between grain size between 33-70 µm, while β-SbSn defragment to the shapes of white islands and flacks instead its original cubic shapes. It is good to worth the information here that grain size of GDC without extrusion was about 16-37 of α-Pb and 15-31µm of β-SbSn phase. The processing temperature plays an important role in the formation of coarsening grain microstructures. In fact, decreasing the deformation temperature results in a finer microstructure. This explains the fact that as the temperature increases, the size of dynamically recrystallized grain increases [6]. The compression strength registers reading 87.1 MPa. While the hardness is effected after processed by the equal channel to register 17.73 HB. While the micro hardness average registered 19.1, 18.15 nears the walls and centers respectively, the behavior at it end stages is similar in all cases, it notice that the tail of the curve is still to raising-up and this because of behavior of this kind of soft metals that compressed; it does not eventually shear but continues to flatten out with increasing load. The metal totally compliant for applied load and the readings represent the resistance of cokes on each others as the layer of metals being very thin.

Figure 13: Flow chart of compression strength for New Rheo specimen extruded at route Bc angle 135° low strain rate

Figure 14: Vicker's Microhardness for Extruded Gravity Die Castings Specimens

Figure 15: Grain Size for Extruded Gravity Die Castings Specimens
4- Conclusion

• The study pointed for microstructural change caused by ECAP softens the material due to the break-up of original precipitate and accelerate from the dynamic recrystallization.

• The materials that were processed at elevated temperatures possess a larger grain size than those that were processed at room temperature. Also, the disorientation of the newly formed grain boundaries tends to be lower at elevated temperatures than at room temperature.

• Increasing forming temperature during angular extrusion has a negative effect on compression, yield strength and hardness.

• With decreasing extrusion angle, the grain size is refined, and the mechanical properties at room temperature are improved effectively.

• Normally applied load will be increased with decreasing the angle of extrusion angle.

• There is no obvious relation between the relative magnitude of either the ultimate strength or yield point and the both type of hardness ( Brinel and Vickers). The hardness of B23-alloy 13 drops off very rapidly with increasing temperature.

5- Reference:


Pb-Sn-Sb using the microhardness and microstructural characteristics of the two alloys.

The study aims to improve the Pb-Sn-Sb solder characteristics by using a microhardness test and observing the microstructure of the solder. The study used three techniques to produce the solder: the conventional method (GDC), the liquid method (NRC), and the solid method (CC). The results showed that the microhardness test revealed the presence of two types of Pb-Sn-Sb phases in the alloy, while the conventional method showed a solid phase of Sn-Pb and a liquid phase of Sn-Sb. The study concluded that the solder manufactured using the conventional method has better microhardness properties, while the results from the other two methods are lower. The study also found that the temperature has a significant effect on the solder hardness, with higher temperatures leading to higher hardness due to the increased grain size of the solder. Consequently, the study recommends using lower temperatures to improve the solder microstructure and properties.