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Solid-State Recycling of Aluminum Alloy (AA-6061) Chips via Hot Extrusion Followed by Equal Channel Angular Pressing (ECAP)

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A B S T R A C T

Aluminum alloy (AA-6061) chips were recycled using hot extrusion followed by equal channel angular pressing (ECAP) process at room temperature. AA-6061chips were cold compacted into billets, then extruded into rods under extrusion ratio of 5.2 at different extrusion temperatures (ET). Finally, the rods were processed through ECAP die with inner angle Φ of 90°, and outer arc angle Ψ of 32.8°, which impose strain ε of 1 per each pass up to different number of passes. The effects of the ECAP number of passes and extrusion temperature on the microstructure and mechanical properties were fully investigated. Grain refinement were noted after the ECAP process. Moreover, the ECAPed samples revealed higher mechanical properties than those of the extruded samples. The extrusion temperature (ET) and the number of the ECAP passes have an obvious effect on both the microstructure and mechanical properties of the solid state recycled chips samples.

1- Introduction

Aluminium alloy 6061 is one of the most extensively used of the 6000 series aluminium alloys due to its preferred properties, for example, medium to high strength, good toughness ,excellent corrosion resistance, and good workability. When aluminium products are manufactured a considerable amounts of scrap (chips and discards) are produced [1]. Recycling of scrap become a very economical method for producing materials because of the low cost of recycled materials [2] .There are two primary methods of scrap recycling:- conventional and solid state recycling [3]. The conventional technique requires melting of the scrap to be recycled. It is characterized by high energy consumption, high operating cost, and a large number of operations [4]. The solid state recycling is the recycling of scrap without re-melting to avoid the troubles of conventional method. Compared with conventional recycling, the solid state recycling of aluminium scrap may result in 40% material, 26–31% energy and16–60% labour savings [5]. In case of chips produced from machining of semi-finished aluminium products, it is very difficult to be recycled by conventional methods due to their elongated spiral shape, small size, surface contamination with oxides and machining oil [6].

Stern [7] recycled aluminium chips by solid state recycling through hot extrusion. Gronostajski et al. [8-9] applied hot extrusion for the production of composites based on Al and AlCu4 alloy chips and tungsten powder. A major advantage of this process is that up to 95% of the primary material can be used by avoiding metal loss during the re-melting process.

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Furthermore, Gronostajski et al. [1] proposed a list of factors contributing the mechanical properties of the chip-based extruded profiles involving the size of chip, the cold pressing parameters, extrusion temperature, extrusion ratio and rate of extrusion. The critical issues in the process of consolidation of aluminium chips are sound bonding between chips and elimination of porosity, which directly influence the mechanical properties of the extruded profiles. From the metallurgical point of view a certain level of strain is needed in order to break oxide layers on the chips' surface and to achieve sound bonding between chips. Fogagnolo et al. [6] investigated the feasibility of recycling of AA-6061chips by cold and hot compaction followed by hot extrusion. In case of hot compaction, there was sufficient chip bonding at extrusion ratio (ER) = 6.25. While, in case of cold compaction the extrusion ratio must higher than 25 in order to obtain sound bonding.

Suzuki et al. [10] compacted the AA-6061 chips and followed by hot extrusion at various extrusion ratios and temperatures. Nakanishi et al. [11] examined the relationship between the extrusion ratio and mechanical properties of hot extruded machining chips of AZ91 magnesium alloy.

In order to meet the increasing demand of Al alloys with high mechanical properties, it is necessary to enhance the properties of recycled materials. Recently, several studies on solid-state recycling of machining chips using severe plastic deformation (SPD) have been conducted. SPD processes introduce ultra large plastic strain into bulk metals which results in the formation of an ultra-fine grained microstructure with grain sizes below 1µm and grain boundaries of high misorientation angles [12] .SPD processing methods that can introduce large strain such as equal-channel angular pressing (ECAP) [13], high pressure torsion (HPT) [14], cyclic extrusion compression (CEC) [15] and friction extrusion [16] have been reported. ECAP is a novel technique able to introduce heavy shear strain into the processed material without changing the cross-section of the workpiece during the multi-passes of deformation through a special die [17]. It was introduced and patented by Segal in 1977 [18].

It is capable of refining bulk materials with coarse grain structures, so ECAP can be applied for solid state recycling of Al machining chips to get materials with high mechanical properties.

However, up to now, few studies have been performed on improving the properties of recycled products using ECAP. Ying et al. [19] investigated the solid state recycling of AZ91 Mg alloy machining chips through the process of cold compaction, hot compaction, extrusion and single pass ECAP. The average grain size was proven to be much smaller compared to cast AZ91 alloy processed by extrusion and ECAP. Results were attributed to the dispersion of oxide contaminants. After hot extrusion, the extruded cast workpiece showed superior strength and ductility compared to the recycled ones. After applying one pass ECAP, the recycled workpiece showed higher strength but lower ductility compared to the cast billet processed under the same conditions. The objectives of this research are first, investigate the feasibility of solid state recycling of AA-6061 chips through hot extrusion followed by cold ECAP. Second, investigate the capability of ECAP process in improvement of recycled material properties. Third, investigate the influence of extrusion temperature and number of ECAP passes on the microstructures and mechanical properties of the AA-6061 chips solid state recycled.

2- Experimental work

The material used in this work (as received) was aluminium alloy AA6061 rod, provided by ALU Misr Company. Table 1 presents the chemical composition of the AA6061 rod analysed by optical emission spectroscopy using a Thermo Scientific ARL 3460 Metals Analyzer. The machined chips with average dimensions L x W x T of 37 mm×2 mm×1 mm were prepared by dry turning of the as-received rod under cutting conditions shown in Table 2. The chips were placed in a die with a diameter of 25 mm, cold compacted at room temperature, and then hot extruded at three different temperatures (350, 425 and 500 ^{0}C) with extrusion ratio of 5.2:1. Cylindrical billets with diameter 11 mm and 90 mm length were cut from the extruded rods for ECAP process.

Table 1.Chemical composition of the AA6061 aluminum alloy.

Si	Mg	Fe	Cu	Mn	others	Al
0.8	0.85	0.48	0.26	0.051	0.329	Balance

Table 2. The cutting conditions used in chips production.

Cutting speed	Feed	Depth of cut
88 m/min	1mm/rev.	1mm

The ECAP die had a channel angle of 90° and a curvature angle of 32.8°. ECAP was carried out at room temperature for multiple passes until the specimen was cracked or broken. The solid state recycling process stages are shown in Fig. 1 .The extrusion and ECAP dies are shown in Fig. 2 and 3 respectively.

Fig. 1. Solid state recycling stages (a) initial AA-6061 chips (b) cold compacted sample (c) hot extruded sample (d) ECAPed samples.

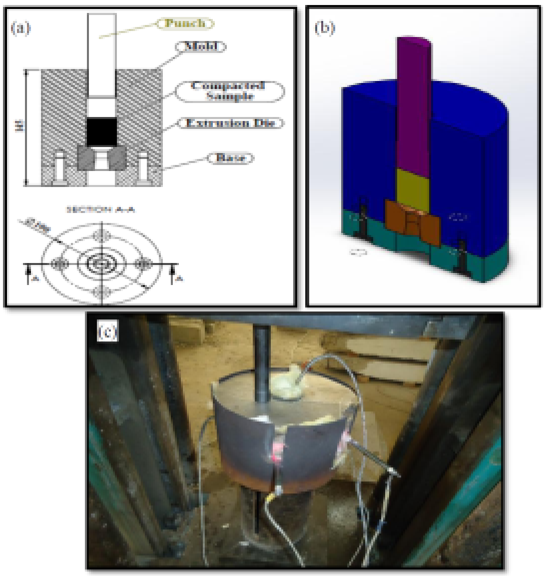

Fig. 2. Hot extrusion die (a) complete assembly drawing (b) half section assembly (c) out fitted.

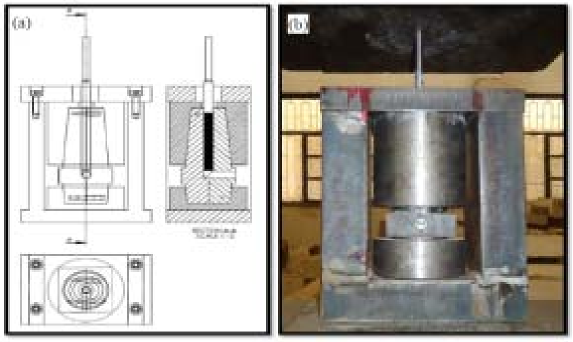

Fig. 3. ECAP die (a) complete assembly drawing (b) out fitted.

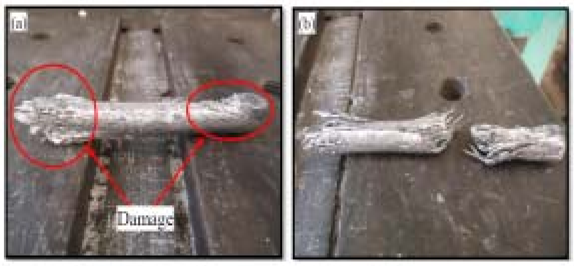

Fig. 4. ECAPed sample at extrusion temp 350 ^{0}C (a) After 1 pass (b) After 2 passes.

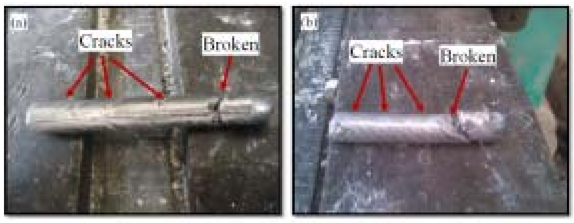

Fig. 5. ECAPed sample (a) Extruded at 450 ^{0}C and ECAPed up to 5 passes (b) Extruded at 500 ^{0}C and ECAPed up to 7 passes.

The billets extruded at 350 ^{0}C were hardly damaged after 1 ECAP pass and totally broken after 2 passes as shown in Fig.4.b. On the other side, the billets extruded at 425 ^{0}C and 500 ^{0}C were cracked and broken after 5 passes and 7 passes respectively as shown in Fig. 5.

For experimental tests, samples with a length of 20 mm were cut off from the middle part of the extruded and ECAPed rods. The samples were further mechanically ground using SiC emery papers (grit 500, 1000, and 2400 for 180 sec each) and finally polished with Al_2O_3 suspensions.

The relative density of processed samples at different conditions was measured experimentally using Archimedes' principle. The density measurement was repeated 3 times for each condition and the average density was calculated at each condition.

The microstructures of extruded samples was examined before ECAP using light optical microscopy on Zeiss Axio Imager. While after ECAP, it was examined using FEI INSPECT S50 scanning electron microscope (SEM). Specimens were etched using killer's reagent.

Vickers microhardness was measured under an applied loading of 100 gf and a dwell time of 15 sec at room temperature using Buehler Micromet 5100 device microhardness tester equipped with a Vickers indenter.

The tensile test was carried out up to failure at room temperature using LR300K (300 kN) universal testing machine. Tensile tests were performed at an initial strain rate of 8.33×10^{-4} s^{-1}at room temperature. Tensile test specimens were fabricated parallel to extrusion direction by turning in accordance with ASTM-B557- 06. All tensile tests were repeated three times for each condition and the average of three measurements was reported.

3- Results and Discussion

3.1 Relative density measurements

The relative density of the compacted sample was 2.511 gm /cm3 which represents 93% of the theoretical density (2.7 gm/cm3) and 7% are porosities. Furthermore, the bonding between individual chips is weak as it will be seen from microstructure investigation. However, this value of average density is higher as compared with the work of other researchers [1,7-8, 20-21], specially, the compaction process is conducted at room temperature. High compacting pressure of 2000 MPa used in this work is higher compared with previous work. Similar observations were obtained by Fogagnolo [6], in which maximum relative density achieved after cold compaction is ~ 92% of the theoretical density. The relative density of extruded and ECAPed samples after different number of passes are shown in Fig. 6. For hot extruded specimens, the relative density increased with increasing extrusion temperature (ET). The relative density of the extruded samples increased to 96, 96.4, 99.8% at ET of 350,425 and 500 ºC respectively. High extrusion temperature enables plastic flow of material to fill voids and make the sample fully dense [9].

Also, the results show that the relative density increased on the initial ECAP passes until reaching a maximum value and after that there isn't any improvement in relative density. For the sample extruded at 500 ºC, the maximum relative density (99.9%) was achieved after one pass. On the other hand, the sample extruded at 425 ºC, the maximum relative density (99.8%) was achieved after 2 passes. In fact, large shear strain and hydrostatic pressure imposed during ECAP improve the consolidation process and increase the relative density [22]. After the first ECAP pass, the size of porosities is decreased to a great extent. More ECAP passes will decrease the amount of porosities but, extensive nucleation of voids is expected, which will limit the improvement in relative density [23,24].

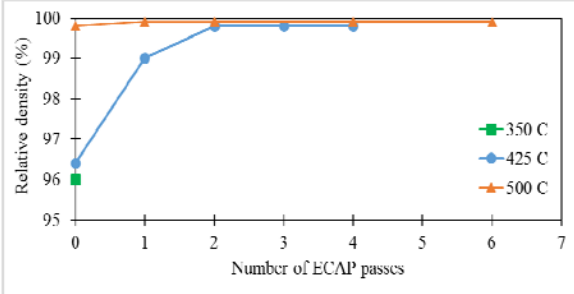

Fig. 6. Relative density of recycled samples.

3.2 Bonding quality of recycled samples

Fig. 7. shows the optical microscopic photomicrograph of the cold compacted chips sample. The machined chips were consolidated after compaction at room temperature. The surface of the chips exhibited some breaking and bonding. The boundaries between chips are distinguished from each other indicating that sound bonding of the chips is not yet complete, and there is a need for an additional consolidation process. Furthermore, voids can be observed between the boundaries of the chips. These observations are in a good agreement with the low relative density of the compacted sample. Moreover, similar observations of voids and visible chips boundaries in the case of compacted chips of alumimium or magnesium chips were obtained by Selmy et al. [25-29].

Aluminium has the capability of forming an oxide layer on the surface. Two criteria must be fulfilled to guarantee sound bonding between chips [29]. The first, there must be a high plastic strain to break down the oxide layer on the surface of the chips to obtain the clean metal-to-metal contact. The second, suitable conditions of high pressure and temperature to achieve complete welding of the chips together. If the oxide layer is partially broken, it will prevent the chips from perfect welding [29].Hot extrusion, involving high compressive and shear forces at high temperature, therefore it was applied to obtain complete consolidation of the recycled AA-6061 compacted chips [26].

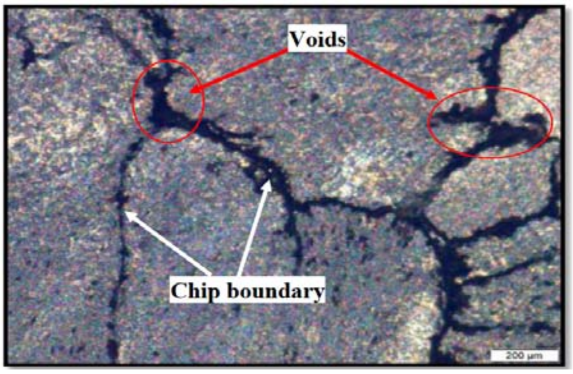

Fig. 7. Optical microscopic photomicrograph of cold compacted sample (white arrows indicate chip boundaries and red arrows indicate void between chips).

Figure.8 shows the optical microscopic photomicrographs of the chip extruded and ECAPed samples. It was noted that the extruded samples at ET of 350 ^{0}C contain unbounded chip boundaries and micro size voids as shown in Fig. 8 (a). The plastic strain and conditions of temperature and pressure were not sufficient to break down the oxide layer between chips. Therefore, the oxide layers prevented sound bonding between chips [26]. With the increase of the ET up to 500 ^{0}C the quality of bonding between the chips was improved and the micro voids were completely disappeared as shown in Fig. 8 (b).

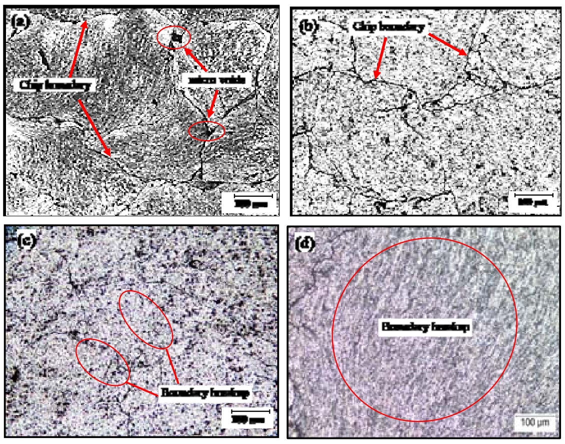

Fig. 8. Optical microscopic photomicrograph of recycled samples (a) ET = 350 ºC, 0 passes, (b) ET = 500 ºC, 0 passes (c) ET = 425ºC, 4 passes and (d) ET = 500 ºC, 4 passes.

The bonding quality increased with increasing temperature which confirms the results obtained by Hasse et al. [20] and Ceretti et al. [30]. However, the plastic strain was not enough to completely break down the oxide layer and chips boundaries are still observed. The observations of non-sufficient bonding between chips at extrusion ratio lower than 25 confirms the results obtained in previous work [3,6].

During the ECAP process, the sample is subjected to severe plastic deformation resulting in severe shear strains when passing repeatedly through the die corner. Therefore, more of chips boundaries were broken and the bonding between chips was improved as shown in Fig. 8(c) and (d). Similar observations was obtained by P. Luo et al. [31].

3.3 Microstructure evaluation

The microstructure of the as-received sample is composed of coarse-grained structure with grain size of 48.2 µm. On the other hand, the recycled samples consisted of finer grains as compared to as-received specimen as shown in Fig. 9 (b), (c) . The grain size is 10.9, 13.7 and 15.8 µm for samples extruded at 350, 425 and 500 ºC respectively as shown in Table. 3. The fine-grained microstructure is caused by severe plastic strain imposed during machining, compaction and extrusion processes [32]. In addition, chip boundaries act as natural barriers between grains and prevent grain growth so fine grains is obtained [20]. Furthermore, it is noticed that grain size was increased with increasing ET. These results are in agree with other work [20].

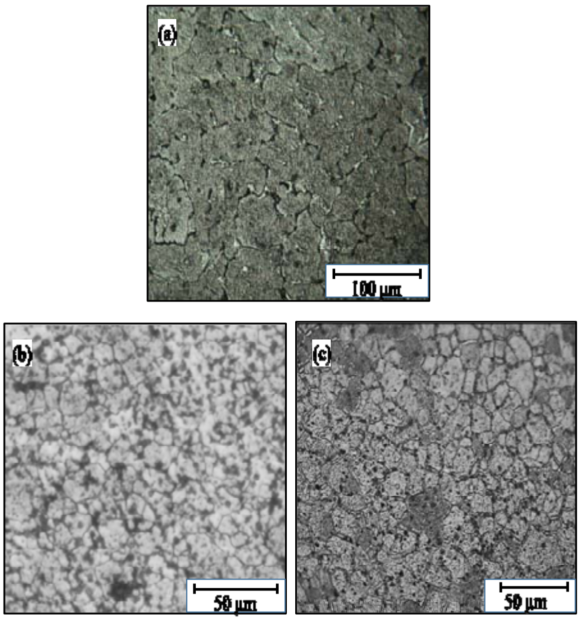

Fig. 9. Optical microscopic photomicrographs of (a) as-received and extruded samples (b) ET= 350ºC, (c) ET=500 ºC.

To study the effect of ECAP passes on the microstructure of the extruded samples at 425 and 500 ºC, SEM micrographs of ECAPed samples after 2, 4 and 6 passes are shown in Fig. 10. It is noticed that there is more grain refinement as compared to extruded sample. Additionally, it is noticed that grain size decreased with increasing the number of ECAP passes. The average grain size measurements are shown in Table 3.The average grain size of samples extruded at 425 ºC is always smaller than samples extruded at 500 ºC at the same number of passes. During the ECAP process, the sample is subjected to severe shear strains.

Table 3. Effect of ECAP process on the grain size and mechanical properties of recycled samples.

Properties	ET (ºC)	Number of ECAP passes			
		0 (Hot extrusion)	2	4	6
Quality of bonding	350	Weak bonding	Improvement of chips welding		
	425				
	500				
Grain size (µm)	350	10.9	----	----	----
	425	13.7	4.02	2.66	----
	500	15.8	5.1	3.28	2.46
Microhardness (HV)	350	49	----	----	----
	425	43	80.75	74.75	----
	500	41	94.5	103.5	110
UTS (MPa)	350	85	----	----	----
	425	116	230.8	347.8	----
	500	133	288.5	349	403
Elongation to failure%	350	4.4	----	----	----
	425	9.4	11.3	13.2	----
	500	13.6	11.9	13.7	14.6

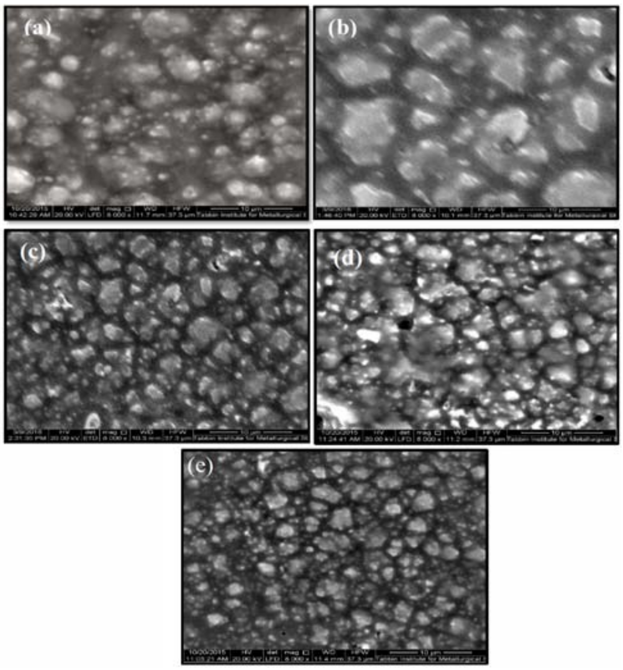

Fig. 10. SEM photomicrographs of ECAPed samples (a) ET=425 ºC, after 2 passes (b) ET=500 ºC, after 2 passes (c) ET=425 ºC, after 4 passes (d) ET=500 ºC, after 4 passes and (e) ET=500 ºC, after 6 passes.

The amount of plastic strain introduced depends on the number of ECAP passes. When applying more ECAP passes, more plastic strain is imposed so the sub-grains are generally divided into equiaxed fine grains with high angle boundary, therefore grain refinement is obtained [33-34]. It is found that the initial grain size has significant effect on the final grain size after ECAP processing, similar observation was obtained with other works [33, 35-36].

3.4 Microhardness

The influence of ECAP number of passes and ET on the microhardness of recycled samples were evaluated and compared with Hasse et al. [20] as shown in Fig. 11. The microhardness of the as received sample and cold compacted chip was 59 HV and 98.9 HV respectively. The higher hardness of compacted chips relative to that of the as received sample is due to imposed plastic strain during turning and compaction processes [37].It is noticed that as the ET increased from 350 to 500 ºC, the microhardness of the extruded samples decreased from 49 HV to 41 HV. This can explained by the increase of the grain size and recovery due to increase of ET. Similar trend of decreasing microhardness with increasing ET was obtained in other work [20].

For the samples extruded at 425 ºC, the microhardness increased from 43 HV before ECAP to 76.25, 80.75 and 82.75 HV after 1, 2 and 3 passes respectively. Then decreased to 74.75 HV after 4 passes. The drastic increase of microhardness after ECAP up to 3 passes is due to the work hardening that is caused by the formation of sub-micrometer ordered grains and the increase of dislocation density occurring with the shear deformation in the initial grain interior [36] .However, the decrease in microhardness can be related to any voids in the investigated specimen. For the samples extruded at 500 ºC, the microhardness increased from 41 HV before ECAP to 82, 94.5, 103.5 and 110 HV after 1, 2, 4 and 6 passes respectively. In previous work by Haase [20], AA-6060 chips was recycled through hot extrusion with ER of 8.7 followed by hot ECAP in the same die. The process was conducted at temperatures of 450 and 550 ºC. In contrast with the trend observed in this work, the microhardness of AA-6060 recycled samples was decreased with increasing number of passes. Also in future work by Haase et al. [38], hot ECAP process decreased the microhardness of recycled samples through hot extrusion from 42 to 41HV. The microhardness of recycled samples through hot extrusion followed by cold ECAP is significantly higher than samples recycled through hot extrusion followed by hot ECAP.

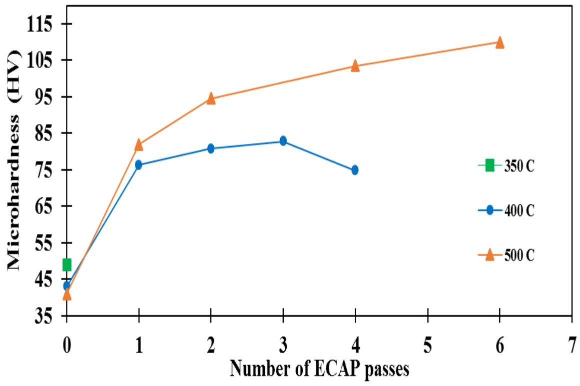

Fig. 11. Influence of ET and ECAP number of passes on microhardness of recycled samples.

3.5 Tensile test

The variation of ultimate tensile strength (UTS) and elongation to failure % of recycled samples with number of ECAP passes are shown in Figs. 12. (a) ,(b). UTS and elongation to failure of the extruded chip samples were significantly increased with increasing ET. UTS increases from 85 to 133 MPa with increasing ET from 350 to 500 °C.

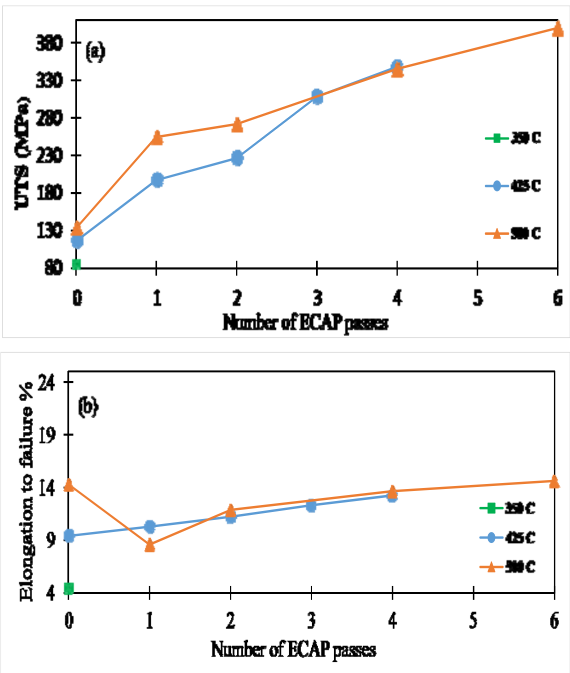

Fig. 12. Influence of ET and number of passes on (a) UTS (b) elongation to failure.

Similar observation were also noted in the case of elongation to failure increases from 4.4 % to 13.6 % with increasing extrusion temperature (ET) from 350 to 500 °C. Increasing ER, minimize the porosities in recycled material as proved previously, and improve the quality of bonding between individual chips and consequently ultimate tensile strength and elongation of the recycled specimen increases. Similar observation were reached by other authors in previous work [3,20, 25].

The UTS is found to increase with the increase of the number of passes for both samples extruded at 425 and 500 °C as shown in Fig.12 (a). The UTS values are observed to increase from 116 MPa before the ECAP process to 198, 230.8 and 347.8 MPa after 1, 2 and 4 passes respectively for sample extruded at 425 °C. In case of sample extruded at 500 °C, the UTS values are observed to increase from 133 MPa to 263.9, 288.5, 349 and 403 MPa after 1, 2, 4 and 6 passes respectively. The increase of the UTS can be attributed to the grain refinement and the work hardening during the ECAP process. Same behaviour of the increase of the UTS with the increase of the number of passes was observed in the case of processing AA-6061alloy through ECAP [33]. The increase in UTS is more significant after the first pass, reaches 71% and 98.4% for samples extruded at 425 and 500 °C respectively. It has been established that the strain imposed by the first ECAP pass has a strong effect on the breakdown and refinement of the initial microstructure [24]. In addition, the formation of sub-grains during the first pass of the ECAP process has an effect on increasing the strength [39].

It can be noted that the elongation to failure increased from 9.4 % before the ECAP to 10.4 %, 11.3 % 12.4% and 13.2 % after 1, 2, 3 and 4 passes respectively in the case of sample extruded at 425 °C. While in case of sample extruded at 500 °C, the elongation to failure is observed to decrease from 14.3 % before the ECAP to 8.6 % after 1 pass. Then increased to 11.9%, 13.7% and 14.6% after 2, 4 and 6 passes.

The increase of elongation to failure can be related to the improvement of bonding between the chips during the ECAP process in which the extensive shear deformation breaks up the oxide films on the chips surfaces. So that it yields fresh and clean surface of metal and ensures good contact and bonding between individual chips [22]. Besides that, it has a close relation to the annihilation kinetics of extrinsic grain boundary dislocation introduced by severe plastic deformation and the number of dislocations necessary to deform the ultra-fine grain [40- 43].

Up to 3 passes, it is noticed that the UTS of samples extruded at 500 ^oC is always higher compared with those of samples extruded at 425 ^oC. While after 4 passes, the UTS is nearly the same. The difference between the UTS values is 57.7 MPa after two passes. Then, the difference is found to decreases with the increase of the number of passes. Similar trend was observed in case of elongation to failure. Also after 4 passes, elongation to failure % of samples extruded at 425 and 500 ^oC is nearly the same.
As compared with work of Hasse et al. [20], the UTS values were observed to increase from 162.5 MPa before the ECAP process to 173.6 MPa (6.8%) and from 166.7 MPa to 194.2 MPa (16.5%) after 4 passes for sample extruded at 450 and 550 ^oC respectively. Furthermore, elongation to failure increased from 14 % before the ECAP process to 23.8 % (70%) and from 14.4 % to 24 % (40%) after 4 passes for sample extruded at 450 and 550 ^oC respectively. In the present work, the UTS increased from 116 to 347.8 MPa (~200%) and 133 to 349 MPa (162.4%) at the same number of passes for samples extruded at 425 and 500 ^oC respectively. Furthermore, elongation to failure increased from 9.4 % before the ECAP process to 13.2 % (40.4 %) and decreased from 14.3 % to 13.7 % (- 4.2 %) after 4 passes for samples extruded at 425 and 500 ^oC respectively. the UTS of recycled samples through cold ECAP is significantly higher than those of recycled through hot ECAP.
According to these observations, Cold ECAP is more efficient in recycling and producing high strength materials. Cold ECAP is conducted at room temperature, therefore the grain growth due to high temperature applied in hot ECAP is eliminated and fine microstructure is obtained. Furthermore, it saves the energy required for heating. However, the bonding quality achieved through hot ECAP is significantly better than cold ECAP and the results of elongation to failure confirmed this conclusion.

4- Conclusions

The investigations carried out on solid state recycling of AA-6061 through hot extrusion followed by cold ECAP, allowed to obtain the following conclusions.

(1) Hot extrusion is effective techniques to produce recycled samples with high relative density. While cold ECAP process slightly increased the relative density.

(2) For the extrusion ratio ER = 5.2, the plastic stain was not sufficient to break down the oxide layers between individual chips. While the plastic strain imposed through ECAP was sufficient to break the oxide layers and guarantee sound bonding. Furthermore, the quality of bonding was increased with increasing number of ECAP passes.

(3) Recycling of aluminium machining chips through hot extrusion followed by ECAP process instead of using hot extrusion only, leads to improvement in strength, ductility and hardness.

(4) The superior mechanical properties of the samples extruded through the ECAP die were in accordance with the microstructure characterization showing fine uniform grains and improvement of chips welding.

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