Effect of magnesium on the properties of ultrafine grained aluminum processing through equal channel angular pressing (ECAP)

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Abstract

Equal channel angular pressing (ECAP) was carried out at room temperature and at 100 °C to refine the grain size of two different aluminium alloys, AA-5050 and AA-5754. AA-5050 contains 1.5 % magnesium and AA-5754 contains 3.3 % magnesium. Optical light microscopy revealed that ultra-fine grain sizes are attained in both alloys. Tensile and compression tests at room temperature shows that the strength increases with an increase in the number of passes but the elongation to failure is decreased. Special attention was paid to the tension and compression asymmetry of the both alloys and it found that the yield strength in the case of tension is almost 25-30 % higher in both alloys. The maximum yield strength observed in AA-5050 after 4 passes during tensile test is 450 MPa at 100 °C while in compression is 356 MPa. In case of AA-5754 this value is 445 MPa in tension and 372 Mpa in compression.

Keywords: Al–Mg alloy; Equal-channel angular pressing (ECAP); Microstructure; Dislocations.

Introduction

In the last two decades a lot of attention was paid to different methods of obtaining ultra-fine grained materials. Especially in recent years a huge effort was made in this area to obtain materials with refined microstructure. This interest in decreasing the crystallite size, a material has a very reasonable basis. It was proved that small grain size and large fraction of grain boundaries very often lead to new qualities in a material or at least increase those well known, like strength of construction alloys of different metals.
However, techniques of grain refinement like powder metallurgy are very inefficient and in consequence expensive, not to mention other drawbacks. Therefore some other ways of decreasing grain size of poly crystals are attaining more and more attention.
As mentioned in the introduction, very high strains may lead to refinement of microstructure of metals. Because commonly used techniques like rolling or extrusion have the side-effect of following a continuous strain path and leading to a so-called ‘cellular’ or ‘fibrous’ substructure, some other method of plastic deformation is needed. One of interesting groups of techniques that allows decrease of grain size of the materials bases on Severe Plastic Deformation (SPD). Methods considered as SPD ones are Severe Plastic Torsion Straining (SPTS), Multiple Forging (MF), Equal Channel Angular Pressing (ECAP), also known as Equal Channel Angular Extrusion (ECAE). All of those techniques were proved to be able to decrease grain size in a material to sub micrometer level (SPTS, MF, and ECAP). The most promising way of deformation for future use on industrial scale seems to be ECAP as it fulfills a very important condition, that it does not change the dimensions of processed material. [i ii iii].

Equal Channel Angular Pressing (ECAP)

Equal Channel Angular Pressing is a relatively new technique of plastic deformation of materials. It was first described and then developed by V.M. Segal and co-workers in the beginning of 80s [v]. The most important characteristic of this method of deformation is the possibility of introducing very high strains into the material without change of its cross-section. Stability of dimensions of the processed billet allows repetitive pressing and in consequence the introduction of extremely high strains.
Since the beginning of 90’s, the method intensively investigated by many different research groups. Main efforts are going in two directions:

- Investigation of ECAP fundamentals
- Investigation how ECAP influences microstructures and mechanical properties of different materials (mostly commercial alloys of different metals) [iv v]

ECAP mechanics, which is the way of introducing strain into the sample and describing the path of deformation, have been analyzed very carefully by different scientists in the last 10 years. A relatively good understanding of the principles of the process enabled scientists to explain the aspects of its influence on microstructure of materials. The problem is very complex though and demands further attention. Influence of ECAP on properties of the materials will be discussed later [vi vii viii].

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Principles

During ECAP, strain is introduced into the material by pressing the billet through two channels of identical diameter, intersecting each other at an angle of 90 – 135 degrees Figure 1b. The sample, which is put in the vertical channel, is pressed by the plunger of a pressing machine to the horizontal channel as shown in Figure 1a. When the sample is bent through the corner a large shear strain is introduced into the material. The strain introduced to the sample in one pressing depends on the die geometry. The whole process of deformation is quite complicated and will not be discussed here. Detailed theoretical study of the sample deformation on the intersection of two channels can be found in [vi vii viii]. The theoretical total strain induced into the sample can be estimated by the following equation [vii]:

$$\varepsilon N = (N/\sqrt{3}) * (2 \cot(\phi/2 + \psi/2) + \psi \operatorname{cosec}(\phi/2 + \psi/2))$$

εN – strain introduced into the material,
N – number of passes through a die,
ϕ - angle of channels intersection,
ψ - angle describing an outer corner.

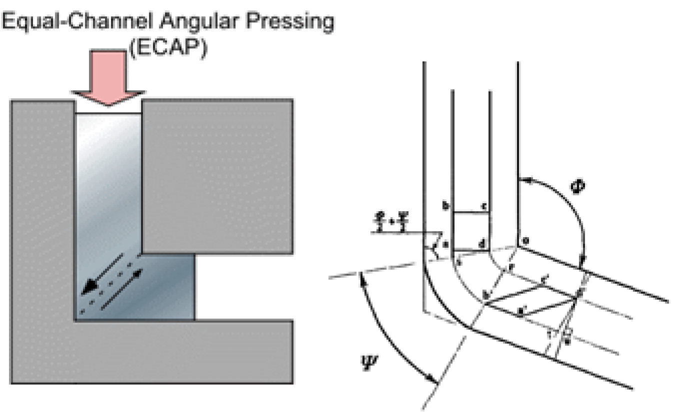

Figure 1: Principle of ECAP; a) sharp corner die with deformation plane marked (taken from [ix]); b) round corner die explaining basics of deformation (taken from [Error! Bookmark not defined.])

The actual strain is always lower due to friction effects between billet and walls of the die. The importance of that phenomenon have analyzed [x].

When a billet is pressed through a channel, it is deformed in a specific way. Most commonly one of four routes of deformation is used (Figure 2):

- A – no rotation between consecutive press ings,
- BC – rotation by +90° (successive) after each pressing,
- BA – rotation by +90° (alternating) after each pressing,
- C – rotation by +180° after each pressing.

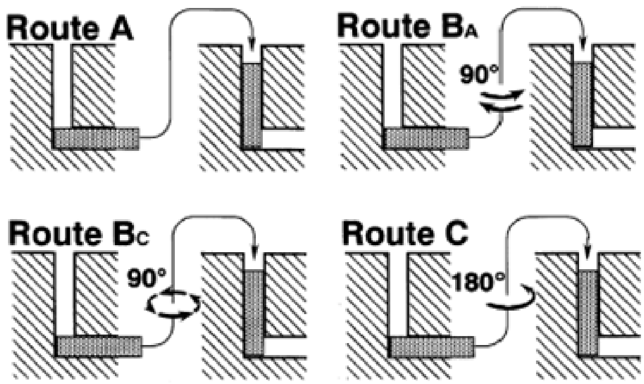

Figure 2: Four basic routes of deformation in ECAP [xi].

Experimental Procedures

During ECAP process, the material is subjected to a large shear strain. All ECAP samples have been machined with the extrusion direction (ED) parallel to the former rolling direction (RD). These samples has a diameter of 12mm and length of 60 mm and were processed by ECAP following route Bc for 2, 4 and 8 passes. The deformation was carried out at room temperature and at 100°C, with molybdenum disulfide (MoS_2) used as lubricant. The ECAP die had an intersection angle $\Phi = 90°$, with rounding radius $\psi = 0°$ In the used experimental setup the total equilibrium strain is 1.155 per pass [xii]. In some cases the samples are heat treated after one pass, at a temperature of 400°C by using a vertical tube furnace in an argon atmosphere for one hour. The purpose was the dissolution of precipitates and (partial) recovery of the microstructure, allowing the sample to be deformed to a higher number of passes.

Mechanical Characterisation

Tensile testing has been done for both the 5050 and 5754 specimens by using an Instron-5567 type-testing machine with a crosshead speed of 0.3 mm/min (constant during the test) and a load cell of 30 kN. Tensile specimens of both materials have been machined in such a way that only the homogeneous zone of the ECAP sample will be subjected to deformation. Tensile sample geometry was chosen as per figure 3, which made it possible to perform tests successfully. The test zone has a diameter of 5mm and length of 12 mm (approximately) [in fact the real gauge length will be 10=12-2x rounding radius 1] due to a slight difference in the internal radius. Due to small size of the samples, stainless steel custom made extension parts were screwed onto the sample ends and fixed in the clamps.

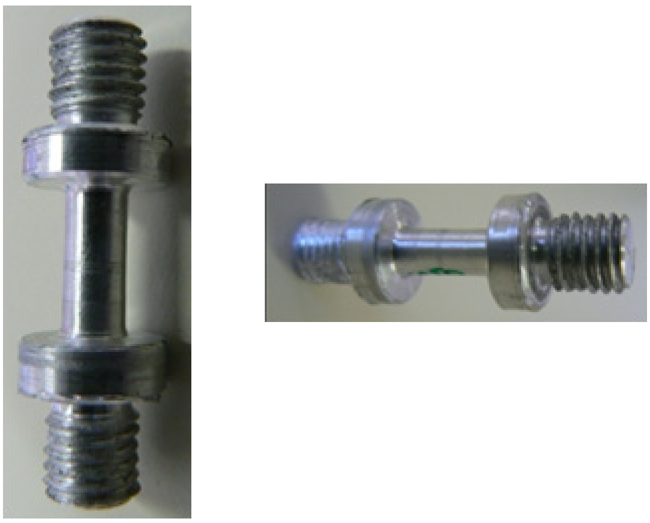

Figure 3: Geometry of the tensile specimen.

For compressive testing the cylindrical samples were cut out with a diameter of 6 mm and a height of 9 mm. Compressive tests performed on an Instron-5567 type testing machine with 30 kN load cell. For compressive tests, strains were limited to $\varepsilon = 0.7$ to avoid excessive inhomogeneous deformation due to barreling.

Microstructural Characterisation

To reveal the microstructure, the samples have been prepared by step-by-step grinding and polishing. Cross sections perpendicular to the tensile and compressive axis of ECAP material (mentioned in figure 4) were cut using a cutting disk and mounted by using a cold mounting technique (because no intentional heating was involved). Planer grinding of the surface undertook with different emery paper with the size range from 80, 120, 320, 800, 1200 and 4000. After cloth polishing has been done with a 3 and 1µm diamond paste. Final polishing was done on OPS disk using Alumina (0.1- 0.25 µm). Samples have been etched with an anodizing process using Barkers reagent (2.44 % HBF4 Solution). The solution was 40ml distilled water and 1ml Flourboric acid. A steel foil was used as a cathode. The parameters were 15 volts for 50 seconds. Between all steps water/ethanol were used for 'wash and dry'. optical microscope (LEICA, Light microscope) has been used. For the observation of shear bands various portions of the specimens were examined.

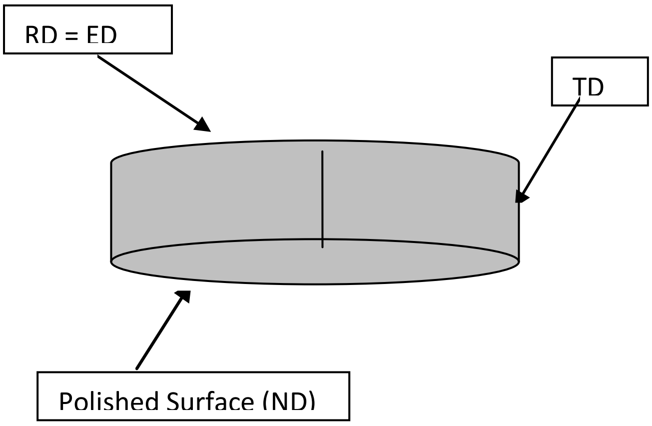

Figure 4: Schematic representation of the various direction of the specimen

VII. RESULTS AND DISCUSSIONS

Microscopic analysis revels that the grain size of the basic material in case of AA-5050 alloy is 80 to 100 µm in width and more than 100 µm in length, but in case of AA-5754 the grain size is 40 to 50 µm, and the grains are more or less equiaxed. The pictures were taken at the same magnification. The small black dots in case of 5050 alloy are supposed to be fine precipitates of Al3Mg but the large circular drops are due to improper etching. In Error! Reference source not found. the percentage composition of the magnesium and other alloying elements is given. The Si and Fe impurities can occur as despersoids, which have the ability to pin the grain boundaries and hinder the

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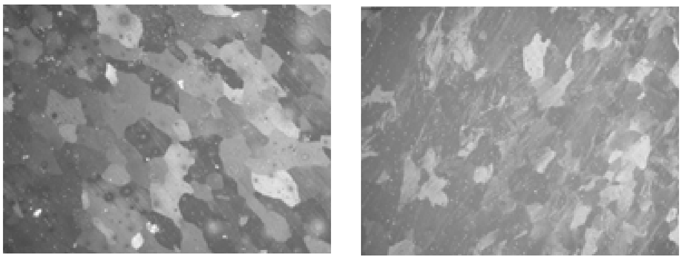
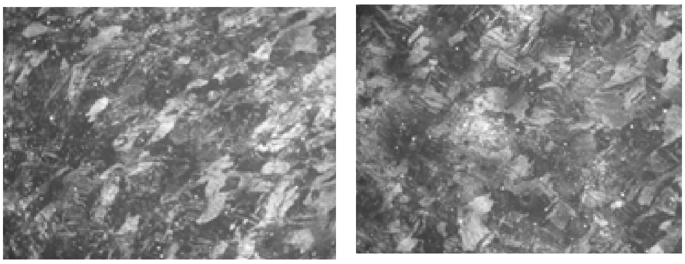
grain growth and recrystallization [xvi].

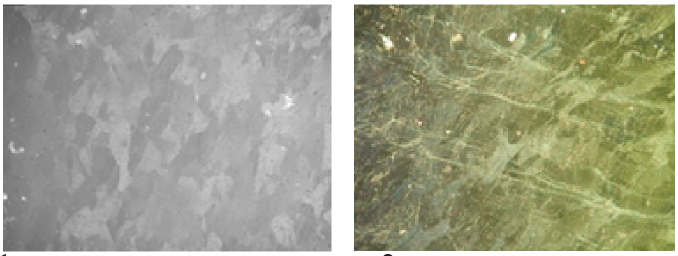


Basic 1 pass

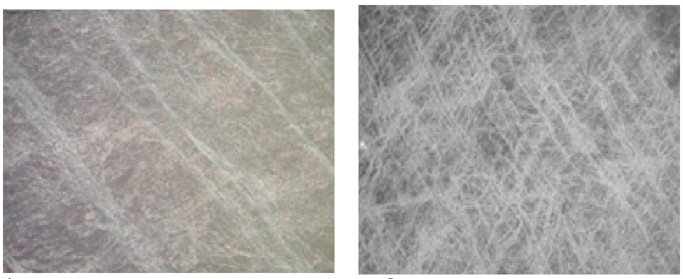


2 passes 4 passes

Figure 1: Microstructure of AA-5050 deformed at room temperature.

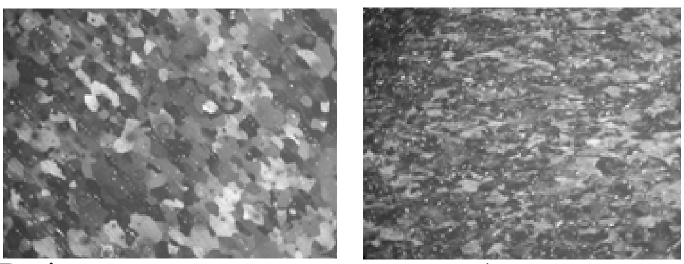


1 pass 2 passes

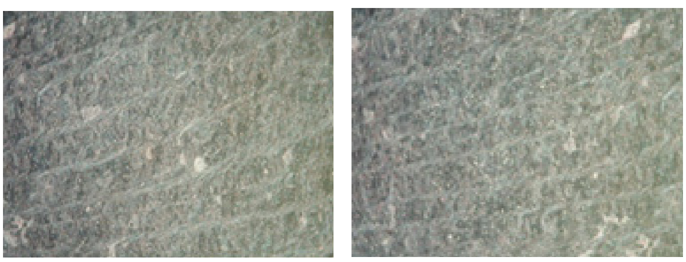


4 passes 8 passes

Figure 2: Microstructure of AA-5050 deformed at 100 °C.



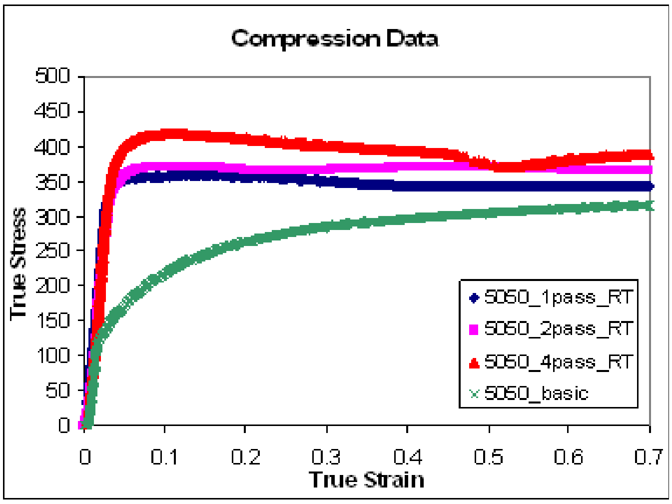
Basic 1 pass



2 passes 4 passes

Figure 3: Microstructure of AA-5754 deformed at 100 °C.

Tensile and compressive samples are machined in such a way that only central zone of the ECAP sample (homogeneous without wall friction) is subjected to deformation. The samples were deformed parallel to the rolling direction (RD) as mentioned in figure 4. The results for the various combinations are presented below



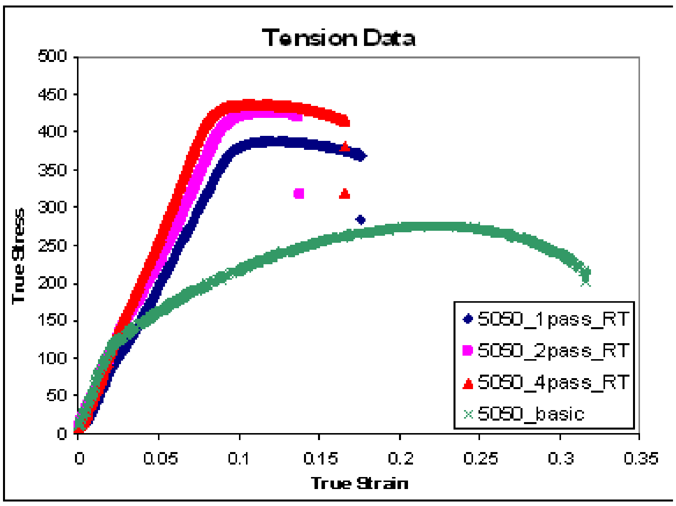


Figure 4: True stress-strain graph (AA-5050) for tension and compression at room temp

Figure 8 and 9 show that there is an evolution in yield stress which is visible in tension and compression with increasing number of passes, the yield strength increases. Yet the tendency is less pronounced than in compression or for material with ECAP at room temperature. The ‘decrease’ of yield strength in tension for 8pass is due to both experimental error and strength saturation. In case of 5754 alloy, it is impossible to deform this alloy at room temperature. It breaks even during the first pass, irrespective of route. It’s also cleared from data that yield strength is increasing as magnesium percentage is increasing in alloy.

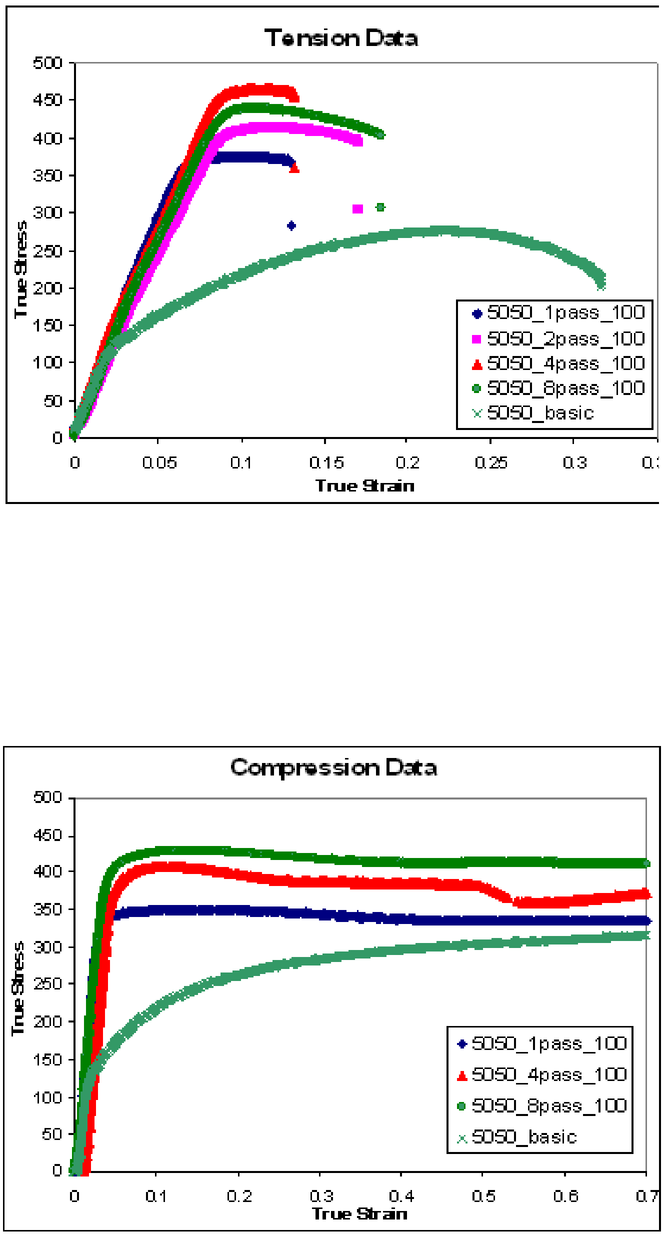


Figure 5: True stress-strain graph (AA-5050) for tension and compression at 100 °C.

It is supposed that the deviation in the elastic region is due to the deformation in the screw part of the sample which is made for clamping. It can also be observed from tension graphs that elongation is reduced after ECA pressing. The basic material is showing a facture strain of 30%, which is reduced to 15-20 % after ECA pressing. Both tension and compression graphs are showing the large and continuous strain hardening in case of basic material but it is almost negligible after ECA pressing, and some samples are also showing softening in case of compression testing. The sudden drop in compression graphs is due to the fracture along the shear band. It can also be noticed that the softening in case of 5754 alloy is more pronounced as compared to 5050 alloy in compression. It can also be noticed that there is a significant increase in yield stress after one pass, and followed by a gradual increase with a further increase in strain. The value of yield stress is highest in case of AA-5050 deformed at room temperature after 4 passes, and it saturates the material.

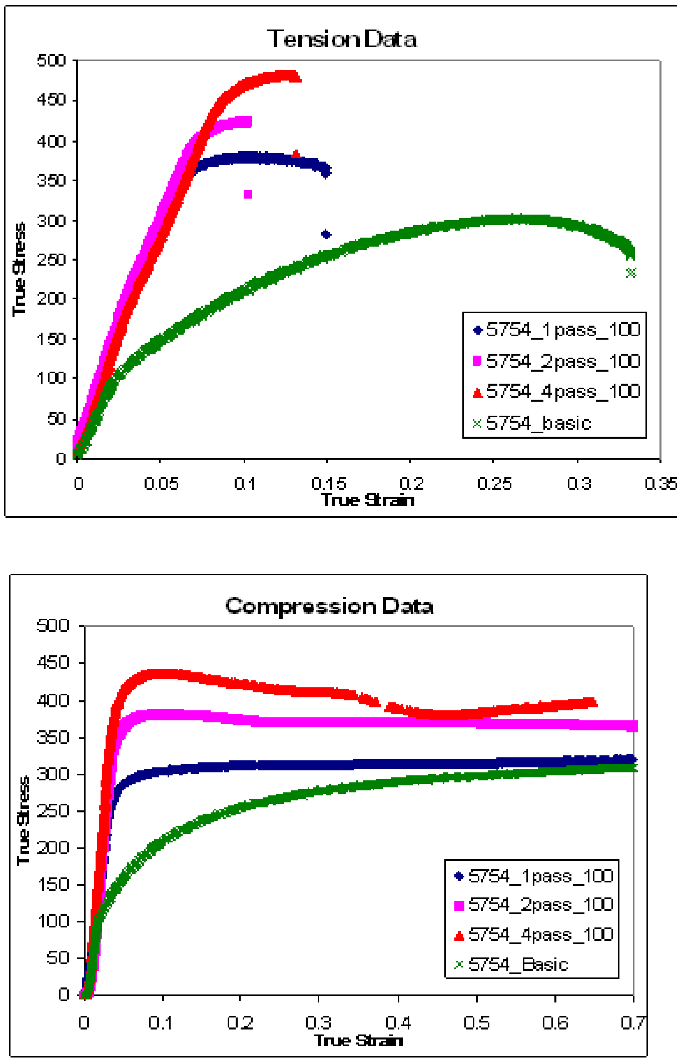


Figure 6: True stress-strain graph (AA-5754) for tension and compression at 100 °C

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The table below shows that the yield strength is increasing with the no. of passes, and it seems it saturates after 4 passes in route Bc in case of 5050. It is also cleared from the table that the magnesium increases the strength of the aluminium alloy. The asymmetry in yield strength for both alloys can be observed.

Table 1:

0.2 % offset yield stress for both alloys in tension and compression.

Material	No. of Passes	Route	Temp (Co)	0.2%Y.S Compression (MPa)	0.2%Y.S Tension (MPa)
5050	0		RT	117	114.33
5050-10	1	A	RT	315.5	367.5
5050-12	2	Bc	RT	314.5	409
5050-14	4	Bc	RT	349	452
5050-15	1	A	100	312	337
5050-17	2	Bc	100	315	393
5050-19	4	Bc	100	356	450.67
5050-21	8	Bc	100	357	432

Material	No. of Passes	Route	Temp (Co)	0.2%Y.S Compression (MPa)	0.2%Y.S Tension (MPa)
5754	0		RT	131	140
5754-16	1	A	100	292	358.33
5754-18	2	Bc	100	320.5	405.5
5754-20	4	Bc	100	372	445

Tension Compression Asymmetry

It can be noted in Table 2 that the yield strength in the case of tension is almost 25-30 % higher in both alloys than in compression after ECAP. Similar behavior has already been observed [xii,xiii] in case of AA-1050, where the yield strength in tension is 23% higher than in compression. In a phenomenological model, the asymmetry in yielding can be taken into account with the incorporation of a back stress [xiv, xv]. It is cleared from figure 11 that tension and compression asymmetry is increased up to 2 passes and then there is a steady decrease in both AA-5050 and AA-5754 alloy. The tension and compression asymmetry in case of AA-5754 after one pass is 23 % as compared to AA-5050 where it is 16% at room temperature and 8% at 100 °C. It shows that there is a significant effect of magnesium content on the initial deformation in case of AA-5754, but on further deformation this higher content of magnesium does not have a significant effect. This could be due to the pinning of dislocations and impeding their motion (solute–dislocation interaction) initially, and later less strength can be due to dislocation–dislocation interaction.

Table 2 :

Tension and compression asymmetry observed in different alloys..

Material	No. Of Pass	Temp (Co)	Yield Stress (MPa)		Asymmetry		
			Tension	Compression	Absolute	Relative	
						(T-C)/T	(T-C)/C
1050	0	RT	51	53	-2	-4	-4
1050	1	RT	147	130	17	12	13
1050	2	RT	156	135	21	13	16
1050	4	RT	173	157	16	9	10
1050	8	RT	195	161	34	17	21
5050	0	RT	114	117	-3	-2	-2
5050	1	RT	368	316	52	14	16
5050	2	RT	409	315	95	23	30
5050	4	RT	452	349	103	23	30
5050	1	100	337	312	25	7	8
5050	2	100	393	315	78	20	25
5050	4	100	451	356	95	21	27
5050	8	100	432	357	75	17	21
5754	0	RT	140	131	9	8	9
5754	1	100	358	292	66	19	23
5754	2	100	406	321	85	21	27
5754	4	100	445	372	73	16	20

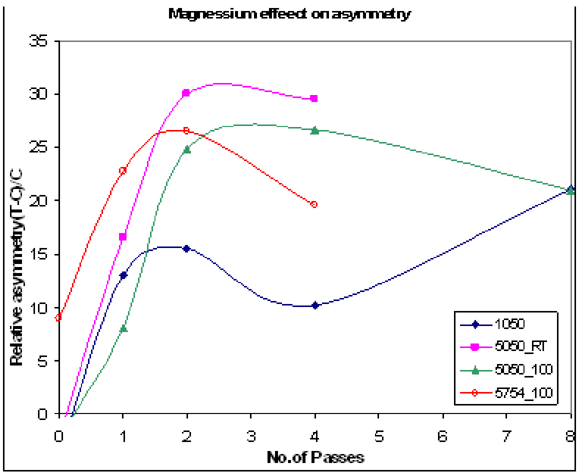

Figure 11: Effect of magnesium content on the tension compression asymmetry.

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Conclusion

Mechanical properties and microstructural evaluation of the AA-5050 and AA-5754 samples are different. The results suggest that the AA-5754 shows higher strengths in tension than AA-5050. In hot rolled condition the AA-5050 shows the higher yield strength in tension as well as in compression but after ECAP AA-5754 shows higher yield strength in tension compared to the other.

ECAP can indeed produce bulk materials with fine grain microstructure. However, it should be noted that this causes the reduction of ductility in tension. The yield strength in both alloys increased as ECA pressing increased. The yield strength in the case of AA-5754 (3.3 % magnesium) is higher in tension as compared to the AA-5050 (1.5 % magnesium) at a temperature of 100 °C. It seems that in case of AA-5050, the material saturates in strength and shows less yield strength beyond 4 passes at 100 °C.

Aluminium and magnesium alloys are showing a clear tension compression asymmetry, which is suggested to be due to the back stress.

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