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Effect of back pressure and temperature on the densification behaviour of Al-Mg alloy

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Abstract

The current research has been aimed to study densification of Al-Mg alloy which was made with optimum sized Nanopowders through Equal Channel Angular Pressing (ECAP) technique. Al-Mg alloy nanopowder was synthesized through high energy ball milling process in the optimised condition. XRD was used to analyze the crystallite sizes of powders prepared at 10, 20, 30, 40 and 50 hrs in ball mill and the minimum crystallite size of 20.388nm achieved at 30hrs was found to be the best milling time. Consolidated specimens were prepared at three working conditions; without back pressure, with back pressure and with back pressure at high temperature (250°C). At each working condition, two passes were made to get better densification in the specimen. The specimens were analyzed for hardness, density, and microstructure. It was found that 92.11% of dense material was formed with a hardness of 64HR$_B$.

Keywords : Consolidation, Pressure, Milling, Crystallite, Channel, Temperature, Powder, Density, Hardness

I. Introduction

Equal Channel Angular Pressing (ECAP) method was introduced by Segal *et al.* for the processing of metals where the high shear strain is imposed to the sample without altering the dimensions [XVI], [IX]. Materials processed through ECAP have ultrafine grains with good ductility. ECAP was used for conventional strengthening and can also be used for powder consolidation. Consolidation with the ECAP for copper as well as aluminium powders has been carried out by many researchers [XVI]. Recently, it was also used to consolidate amorphous powders [VII],[XIII]. During the ECAP process, the specimens are forced through the metal die by a plunger, which was having two equal channel at an intersecting angle of 90°. The induced strain during the process was equal to one at each pass [XVI],[II]. From the recent studies, it has been confirmed that the mechanical properties of the sample showing better when it was prepared with a combination of conventional forms with the ECAP process for solid materials [XVI],[XIV]. However, the fewer attempts made by researchers for the consolidation of powder through ECAP [XVI],[II]. Conventional compaction and sintering process will take longer durations at higher temperatures that lead an increase in the grain size [III], whereas ECAP was an effective technique for consolidation because of its lesser time intervals and at room temperatures [XVII]. Magnesium alloys are also being extensively deformed to improve the mechanical properties by ECAP [XV],[IX]. ECAP was also be used to fabricate the bimetallic tube of different materials like Cu and Al [V]. Recent investigations show that bimetallic rods of Al and steel can be successfully made using this process [XII],[XIX]. Effect of lubrication during ECAP of Al-2024 alloy was carried and suggested that Zinc stearate lowers the friction coefficient that reduces power consumption and produces less distorted parts [VIII]. Experimental studies [X] revealed that heat treated specimens for ECAP shown better results.

Ball milling also called as mechanical alloying was an effective way to prepare alloys, composites and also to reduce the size of particles [XXII]. Nanocrystalline materials prepared by using ball milling shows an increase in strength compared to the coarse-grained materials [XXI].

In this present study, Al-5083 alloy was chosen because of its good formability, corrosion resistance, weldability, good tensile strength and non-heat treatable [III]. Al-5083 alloy has been extensively used in Rail cars, vehicle bodies, tip truck bodies [I][VI].

The present paper aims to prepare the Al-5083 powder through high energy ball milling from elemental powders and consolidate through the ECAP process in a die with and without back pressure and at room temperature and higher temperatures. The ball milled Al-5083 alloy powder particles are characterized through XRD analysis and SEM to calculate the particle size. The prepared samples were examined for density with the help of Archimedes principle and also the mechanical properties of consolidated samples.

II. Materials and Methods

Composition of Al-Mg alloy was represented in Table 1.

The optimised process parameters for high energy ball milling for mechanical alloying of Al-5083 alloy powdered particles were given below.

- B/P ratio: 10:1
- Speed of milling: 250rpm
- Medium during milling: Toluene
- Time of milling: 10, 20, 30, 40, 50hrs

Table 1: Composition of Al-5083 alloy

Elemental Powder	Wt. % Present
Cr	0.05
Fe	0.4
Zn	0.25
Ti	0.15
Si	0.4
Mn	0.4
Cu	0.1
Mg	4
Al	Balance

Initially, the elemental powders of Al-5083 were mixed thoroughly as mentioned in Table 1 and were milled keeping milling parameters in consideration.

The powders are consolidated through equal channel angular processing die, which was shown in Fig.1 **[XX]**. Shear angle (ϕ) maintained as 90° with corner angle (Ψ) of 20^0. Punch diameter was 11.9mm, and the channel diameter was 12mm.

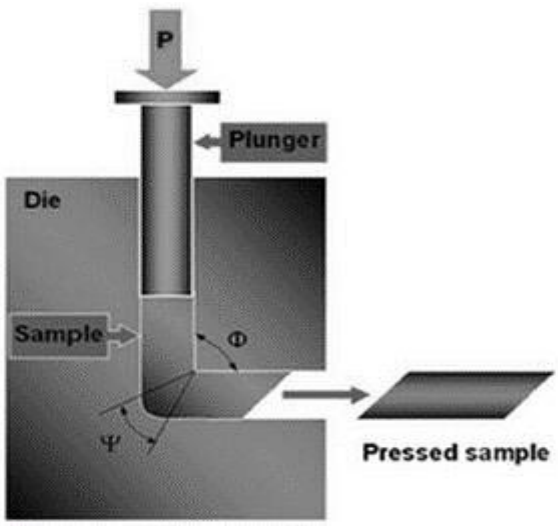


Fig 1. Schematic representation of ECAP die [XX]

The synthesized Al-5083 alloy powder was encapsulated into a commercially pure Al can of length 50mm and diameter 11.9mm. The process was done with and without back pressure. Several techniques can be used for imposing it. To create and increase the back pressure within the die, an aluminium stopper was used. The aluminium bulk materials are having the length of 20mm in front of the can. This back pressure created and induced at the exit side due to the obstacle.

There are four routes that the process of ECAP can be done which were highlighted in Fig 2 [XXI]. In this work route A was used to process the samples.

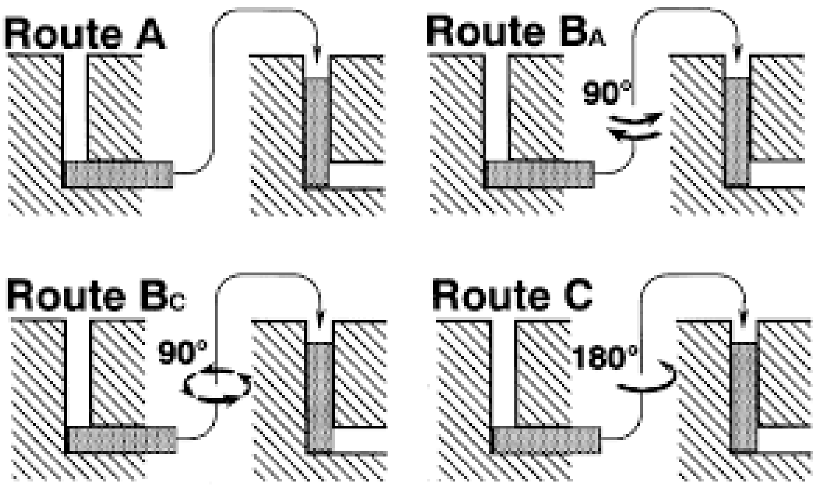


Fig 2. Schematic representation of routes in ECAP process [XVIII]

The composition of the powder samples was examined using X-ray diffractometer (Ultima IV, Rigaku, Japan). To examine the particle size Scanning Electron Microscopy (SEM, Hitachi-S3000N, Japan) was used. Mechanical alloying of elemental powders was done using high energy Ball mill (Insmart PBM07). Density values were calculated using the Archimedes principle and hardness values of the specimens were measured by Rockwell hardness (B scale) with 100kgf and 10sec dwell time.

III. Results and Discussions

XRD Analysis

The XRD graph of the mechanically milled aluminium alloy Al-5083 powders is shown in Fig 3 milled with different intervals from 0 to 50 hrs.

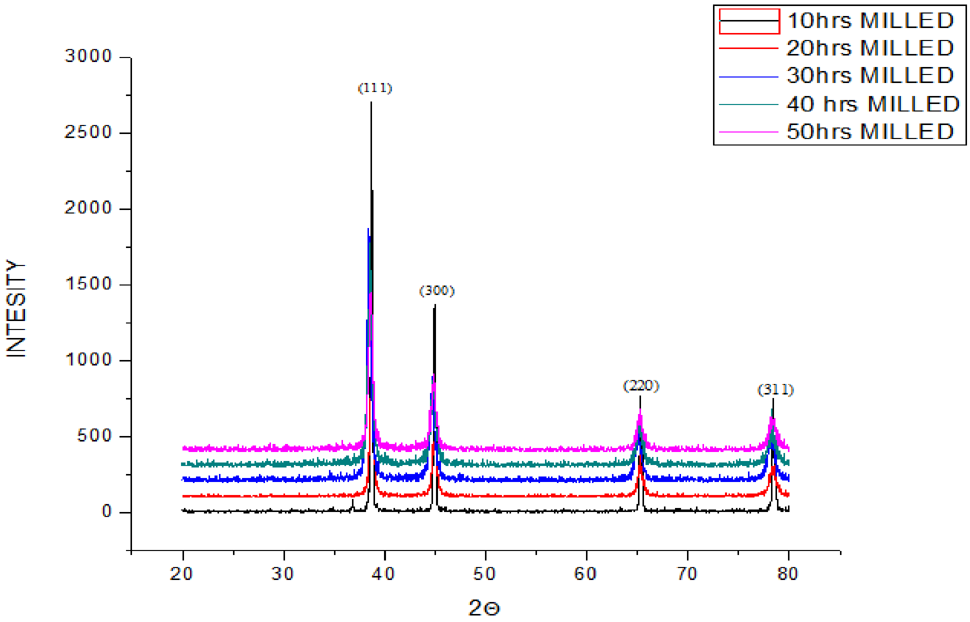


Fig 3. XRD patterns for 10, 20, 30, 40 and 50hrs milled powders

From the XRD graph, peaks of Mg in aluminium as a solid solution and Al alone was visible, and since the other elements are less than 2%, it could not be seen in the XRD. Formation of Al-5083 alloy was indicated from the XRD pattern represented in the peaks observed in the XRD pattern of the powders (Fig 3) are indexed and given in Table 2 based on the JCPDS files. The purpose was to indicate that the Mg was in solid solution with Al during ball milling and no other separate phases had been formed due to ball milling.

Table 2: XRD results of various sample powders milled (a) 10hrs (b) 20hrs (c) 30hrs (d) 40hrs and (e) 50hrs

S.No	Angle	Material	Plane
1	38.76	Al	(111)
2	**44.92**	**Al Mg**	**(300)**
3	65.32	Al	(220)
4	78.52	Al	(311)

(a)

S.No	Angle	Material	Plane
1	38.4	Al	(111)
2	**44.74**	**Al Mg**	**(300)**
3	65.08	Al	(220)
4	78.2	Al	(311)

(b)

S.No	Angle	Material	Plane
1	38.52	Al	(111)
2	**44.80**	**Al Mg**	**(300)**
3	65.06	Al	(220)
4	78.26	Al	(311)

(c)

S.No	Angle	Material	Plane
1	38.45	Al	(111)
2	**44.76**	**Al Mg**	**(300)**
3	65.1	Al	(220)
4	78.26	Al	(311)

(d)

S.No	Angle	Material	Plane
1	38.58	Al	(111)
2	**44.79**	**Al Mg**	**(300)**
3	65.12	Al	(220)
4	78.27	Al	(311)

(e)

Crystallite Size Measurement

Crystallite size has been estimated using Equation 1 in accordance with Williamson-Hall relation [IV] as given below:

$$\frac{\beta_{hkl}\cos\theta_{hkl}}{\lambda} = \frac{K}{D} + \frac{4\varepsilon\sin\theta_{hkl}}{\lambda} \quad (1)$$

Where, β_{hkl} –Width @ half maximum,
λ - Wavelength,
ε- Strain,
D- Crystallite size
θ - Diffraction angle.

The measured crystal size with respect to milling time was furnished in Table 3. The percentage decrease in crystal size increases rapidly at 10hrs, and the rate decreases with respect to time till 30hrs. It was stabilized at 30hrs and hence decided to use powders milled for 30hrs for further experimentation.

Table 3: Al-5083 Crystallite size vs milling time

S.No	Milling time in hrs	Crystallite size in nm	Percentage (%) decrease in crystal size
1	0	77.885	0
2	10	34.063	56
3	20	24.712	68
4	30	20.388	74
5	40	22.456	71
6	50	24.348	69

Size Measurements of Milled Powders

Average particle size of milled powders at 30hrs of milling has been represented in Table 4. Particle size has been estimated by intercept method using SEM images (refer Fig 4).

Table 4: Particle size of Al-5083 alloy powder

S.No	Milling Time in hrs	Particle Size in µm
1	30	7.524 ± 5

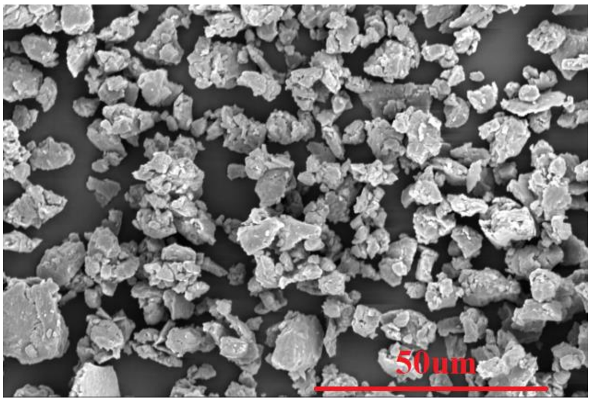


Fig 4. Milled powders SEM images for 30hrs

Densification of Milled Powders

The images of the samples are shown in Fig 5. The first image indicated the commercially pure aluminium was used for encapsulation. The powders are filled into the can and processing carried out through the ECAP die.

The second image represents the sample after the first pass at 250°C. The can was filled with powder and heated in the furnace for 30min and then it was kept in the die and pressed by using route A. The third image represents the sample after the second pass.

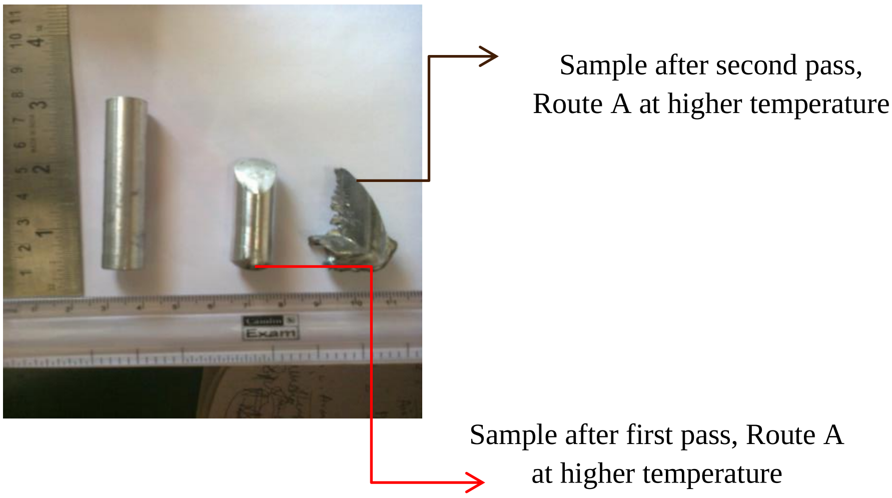


Fig 5. Consolidated ECAP Sample after first and second pass at a higher temperature

Density Measurements

The consolidated sample densities were calculated by the Archimedes principle. The weight of the samples was measured before and after immersion in distilled water. The formula for the Archimedes principle was given in Equation 2.

$$\rho = \frac{W_a}{W_a - W_w} \tag{2}$$

Where, W_a represents the weight in air and W_w represents the weight in water. Measured values for the density with and without back pressure at room and higher temperatures for the single and double pass are tabulated in Table 5. The density obtained for a single pass without back pressure is 37% only, but the relative density was increased to 92.11% at a higher temperature when the back pressure was applied. Further increasing number of passes or increasing the processing temperature will fetch better densification of the powder.

Table 5: Density of consolidated samples using ECAP

S.No	Sample	Density (g/cc)	Relative Density (%)
1	Al-5083 ECAP first pass(No back pressure)	1	37.59
2	Al-5083 ECAP with back pressure first pass	2.2705	85.35
3	Al-5083 ECAP with back pressure second pass, Route A	2.39	90

4	Al-5083 ECAP with back pressure first pass at 250^0C	2.41	90.66
5	Al-5083 ECAP with back pressure second pass at 250^0C	2.45	92.11

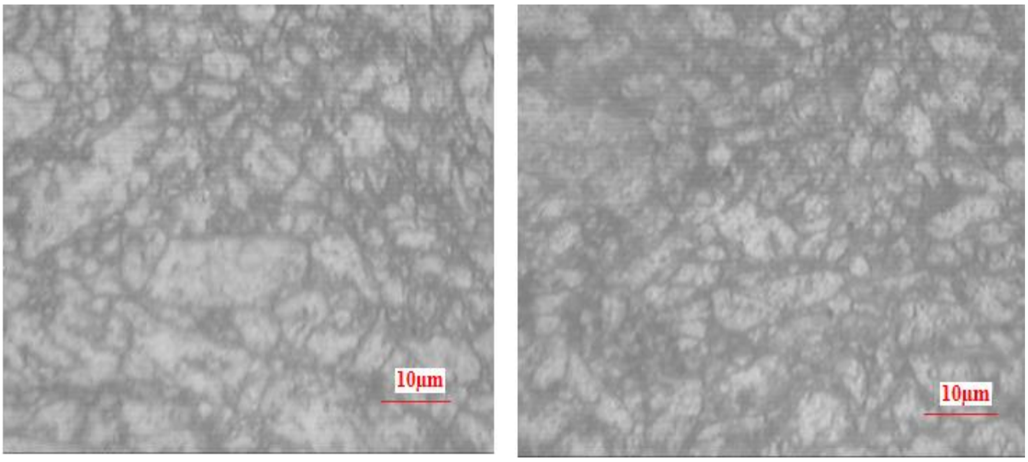
Hardness & Grain Size Measurements

Rockwell Hardness values for consolidated Al-5083 through ECAP process accompanied by back pressure have been tabulated in Table 6. The hardness of the compact increased as the number of passes increases and the processing temperature. Two main factors are responsible for the increase in hardness; (i) grain size and (ii) density of the compacts. The density increases as the number of passes and temperature of processing increases. The grains are also get refined as the number of passes increases, and hence the hardness increases as the number of passes and temperature increased as expected. Maximum hardness of 64HR_B was observed in samples processed at 250^0C in two passes, though the density achieved is only 92.11% to its theoretical density. The hardness at this condition is higher than the 100% dense wrought Al-5083. The higher hardness in the compacted samples indicates clearly that the improvement in hardness was due to grain refinement as mentioned in literature [VI].

Table 6: Hardness of consolidated sample with backpressure using ECAP

S.No	Process	Hardness Rockwell-B Scale
1	Al-5083 Wrought Alloy	54
2	Al-5083 ECAP sample first pass	45
3	Al-5083 ECAP sample second pass, Route A	50
4	Al-5083 ECAP with back pressure first pass at 250^0C	58
5	Al-5083 ECAP with back pressure second pass at 250^0C	64

The microstructure of the samples was taken after the first pass (refer Fig 6a) and after second pass (refer Fig 6b) with the application of back pressure at higher temperatures. The images represent that the alloy was consolidated properly and there was a reduction in grain size and increase in the density due to the application of severe deformation by ECAP process. The grain size has been estimated through linear intercept method. It has been found that the average grain diameter has been reduced from 3.1333µm to 2.6333µm.



(a) **(b)**

Fig 6. Microstructure of Al-5083 ECAP sample processed at 250°C after (a) First pass (b) Second pass.

IV. Conclusions

The current research was aimed to study densification of Al-Mg alloy which was made with optimum sized Nanopowders through ECAP technique. The following conclusions were drawn from this work:

- From the results of XRD, it was proved that the Al-Mg alloy was formed successfully in a high energy ball mill.
- The size of crystallite has been reduced from 77.885nm to 20.388nm at 30hrs of milling time.
- From SEM images, it was clear that the size of the particle at 30hrs of milling was (7.524 ± 5)µm.
- The powders prepared were consolidated using the ECAP process by the application of back pressure at high temperatures.
- The relative density of 92.11% and hardness of 64HR_B was achieved after the second pass at 250^{0}C with the application of back pressure.

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