

Consolidation of Al-2024 Powder by Conventional P/M Route and ECAP – A Comparative Study

Nishtha Gupta , S.Ramesh Kumar , B.Ravisankar and S.Kumaran

Abstract—In this study, mechanically alloyed Al 2024 powder is densified by conventional sintering and by equal channel angular pressing (ECAP) with and without back pressure. The powder was encapsulated in an aluminium can for consolidation through ECAP. The properties obtained in the compacts by conventional sintering route and by ECAP are compared. The effect of conventional sintering and ECAP on consolidation behaviour of powder, microstructure, density and hardness is discussed. Room temperature back pressure aided ECAP results in nearly full denser (97% of its theoretical density) compact at room temperature. NanoIndentation technique was used to determine the modulus of the consolidated compacts.

Keywords—Al-2024, Back Pressure, ECAP, Nanoindentation

I. INTRODUCTION

THE ultra fine grained powders processed by “bottom-up” approaches such as inert gas condensation, electro deposition, ball milling, cryo milling, needs to be consolidated for large scale structural applications. Conventional pressing and sintering, Hot Isostatic Pressing, Direct extrusion are employed for consolidation of the powders. The finished products from these techniques invariably contain some degree of residual porosity and low level of contamination which is introduced during the fabrication procedure [1]-[11].

Industrial level, direct extrusion or compaction of even fine Al based particles is limited due to extreme pressing loads (Industrial feasible loads are up to 900 MPa) and with no room for further increase of extrusion temperature due to structural coarsening and grain growth. Utilisation of backward extrusion leads to significant decrease in pressing load, on the other hand, it often yield insufficient straining and improper consolidation¹². Due to high pressing loads (over 1.4 GPa) needed for breakthrough of the compacted material, direct extrusion was not possible below 350°C.

Nishtha Gupta¹ is with the National Institute of Technology, Trichy, Tamilnadu, India. (corresponding author to provide phone: 91-7829910731 e-mail: dec28nishtha@gmail.com).

Ramesh Kumar Subramanian² is with the Mechanical Engineering Department, Oxford Engineering College, Trichy, Tamilnadu, India (e-mail:srk1306@gmail.com)

Ravisankar B1 is with the Metallurgical and Materials Engineering Department, National Institute of Technology, Trichy, Tamilnadu, India (e-mail: brs@nitt.edu)

Kumaran S1 is with the Metallurgical and Materials Engineering Department, National Institute of Technology, Trichy. Tamilnadu, India(e-mail:kumara@nitt.edu)

Recent research has shown that large bulk solids, in an essentially fully-dense state, produced by the top down approach in particularly using Equal Channel Angular Pressing (ECAP) [1]- [10] though originally ECAP has been developed for the fabrication of ultra fine grained alloys. During ECAP, very high shear strain can be obtained by multi passes through a die without any change in the billet dimensions. Simple shear condition minimizes the redundant normal forces, thus yielding considerable reduction of pressing loads compared to conventional consolidation [12]. In addition, it has been shown that the shear mode of plastic deformation leads to a change in the spherical pore geometry to an elliptical shape aligned with the shear plane, which is favourable for closure of voids under hydrostatic pressure[7],[8]. ECAP consolidation of ultra fine grained Aluminium powders was carried out at a lower (atleast 3 times) pressing loads compared to direct extrusion [13].

Besides, consolidation of powders by normal ECAP, work had been in progress in consolidation of powders by applying back pressure during ECAP [6]-[11]. An important advantage of imposing back-pressure is that it leads to a considerable improvement in the workability of the processed bulk samples. Another important advantage of back-pressure is that visible enhancement has been introduced in the uniformity of the metal flow during the ECAP operation. As a result, the microstructural refinement becomes more uniform, especially in the vicinity of the bottom surface of the billet. In case of powder compacts, the back pressure increases the hydrostatic stress which can be utilised for compaction of powders.

Limited literatures have been available in back pressurised ECAP processes for consolidation of Al 2024 powders. In this paper, ball milled Al 2024 powder was densified by conventional sintering and by normal ECAP and ECAP with back pressure. Density, microstructure, hardness and Young's Modulus of the compacts had been investigated. This work is mainly focused to improve the density of the powder.

II. EXPERIMENTAL WORK

Powders used in this study were elemental powders of Al, Copper, Magnesium, Manganese, iron, Silicon, Zinc, Titanium, and Chromium. The composition of Al 2024 is given in Table I.

Al 2024 was prepared by mechanical alloying route. The powder mixture had been subjected to pot milling with tungsten balls. Tungsten balls with a diameter of 10mm had been used to mill the powder using a ball to powder ratio of 2:1. The milling time was up to 15 h. Milling has been carried out at 50 rpm using toluene medium in order to avoid oxidation and sticking of powders on the walls of the vials.

In this paper, Al-2024 powder was consolidated by conventional powder metallurgy route i.e. green compaction and sintering and by ECAP. In conventional route the green cylindrical pellets with aspect ratio 1:3 were compacted using Universal Testing Machine of capacity 60 tones by single end die compaction method. High carbon high chromium steel die with inner diameter 30 mm was used for compaction. The compaction was carried out with the aid of Lithium Stearate and Graphite as the lubricant and the load applied was 15 tones. After the compaction, the clean green compact were coated with aluminium paint in order to avoid oxidation during sintering. The compact were given two coatings were allowed to dry in air for 8 hours after each coating. The coated compacts were sintered in a muffle furnace at 660°C for a time period of 2 hours. After 2 hours the compacts were allowed to cool in the furnace itself.

A schematic of an ECAP die used in for compaction is shown in Fig. 1. The die angle Φ is 90° and the outer arc curvature ψ is 20° , and the diameter of the channels and diameter of the punch were 12 mm.

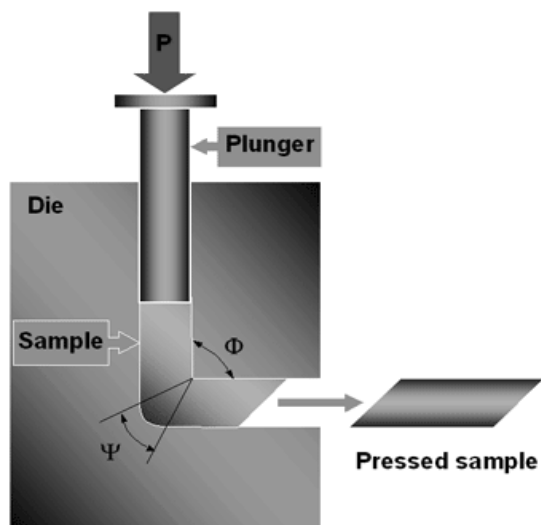


Fig. 1 Schematic representation of ECAP die (Channel angle $\Phi = 90^{\circ}$ and $\psi = 20^{\circ}$)

In order to consolidate the Al 2024 powder through ECAP, Al can was prepared. The length of the cap is 52 mm with outer diameter 11.9 mm. The wall thickness is 1 mm and bottom wall thickness is 3 mm.

The powder was encapsulated and ECAPed with and without back pressure. Route A is used for the densification. Technically, back-pressure can be imposed in several different ways. The conventional way is to apply pressure from the exit

channel. However, this needs a complicated die and experimental set up. Alternatively, in order to increase back

TABLE I
COMPOSITION (WT %) OF AL 2024

Element	Cu	Mg	Mn	Fe	Si	Zn	Ti	Cr	Al
Composition (wt %)	4.4	1.5	0.6	0.5	0.5	0.25	0.15	0.1	balance

pressure within ECAP channels, Al bulk front stoppers of length 50 mm were placed in front of the compacted materials as shown in Fig.2. The back pressure is induced due to obstacle in the exit side. This method is followed by Balog Martin et.al [13]

The draw back in this method is that the actual back pressure applied could not be quantified directly. However, it can be calculated indirectly from the load required to deform the material in the channels with and without Al front stopper. The methodology adopted to quantify the back pressure was discussed elsewhere by the same author [14]. Thus the back pressure applied was about 200 MPa

Density measurements were performed based on the Archimedes principle. Hardness was measured by the Micro Vickers hardness measurement which indicates the degree of grain refinement and densification. HMV-2000 Vickers micro hardness tester was used in this work. The applied load and the indentation time used were 200g and 15 seconds, respectively. The reported micro hardness value for each sample is the average of atleast six measurements. The Young's modulus was evaluated using Nanoindentation by calculating the slope of the unloading curve.

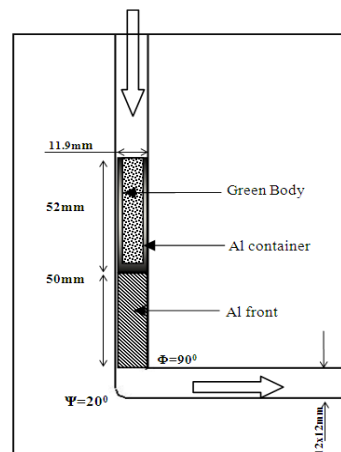


Fig. 2 Schematic representation of imposing back pressure in ECAP using Al front stopper

II. RESULTS AND DISCUSSION

Sintered density is the major factor influencing the mechanical properties of the material processed through powder metallurgy. Sinter densities of the compacts were determined by Archimedes principle and the values are summarized in Table II. The theoretical density of Al 2024 is 2.80 g/cc

The density obtained in ECAP even without back pressure is higher than the conventional compacted and sintered samples in both the materials. As the number of ECAP passes increases the relative density increases in both the materials.

TABLE II
DENSITY OF COMPACTS

Sl.No	Condition	Density (%) related to theoretical density
1	Compacted and Sintered	85
2	After I pass of ECAP without back pressure	88
3	After I pass of ECAP with back pressure	92
4	After II pass of ECAP (route A) without back pressure	95
5	After II pass of ECAP (route A) with back pressure	97

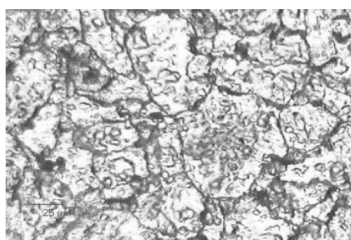


Fig. 3 Microstructures of sintered samples

The micrograph of sintered sample is shown in Fig. 3 for Al 2024. The particles are reasonably well distributed with in Al-2024 matrix. The microstructure of conventional sintered samples shows coarse grains. In the samples, compacted by ECAP, the grains become elongated and finer. The microstructure of powder ECAP (Fig. 4) reveal that grains are uniformly elongated and with each pass the grains gets fragmented. The grains are elongated since the second pass is done with route A which imposes monotonic strain in the same direction of the first pass. The average normalised grain size for conventionally sintered and ECAPed samples is shown in Table III.

TABLE III
GRAIN SIZE OF AL 2024 AND AL 2024 – ALUMINA COMPOSITE DENSIFIED BY CONVENTIONAL SINTERING AND ECAP

Sl.No	Condition	Average Grain size (micrometer)
		Al 2024
1	Conventional sintering	26
2	ECAP- I pass without back pressure	18
3	ECAP- I pass with back pressure	14
4	ECAP- II pass without back pressure	09
5	ECAP- II pass with back pressure	07

The easier densification due to ECAP may be attributed to the following reasons; Shearing exposed fresh and clean surface of the particles and compressive stress ensured good contact between them. Under these conditions bonding occurred spontaneously without needing high temperature and extremely high pressures in contrast to conventional sintering

which requires long time and temperature for bonding between particles to take place[14]. However, detailed TEM studies are necessary to suggest the mechanism of bonding which is in progress.

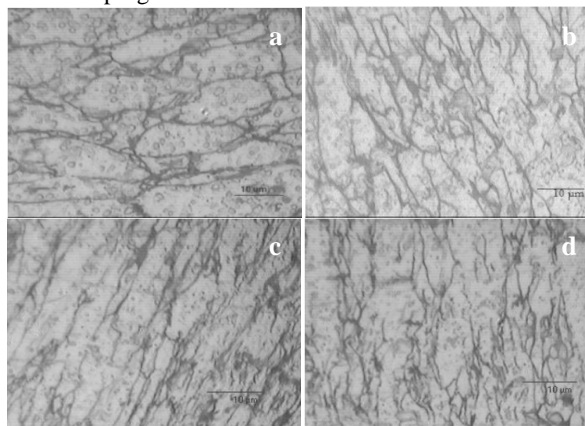


Fig. 4 Microstructures of alloy Al-2024 (a) I pass without back pressure (b) I pass with back pressure (c) II pass without back pressure (d) II pass with back pressure

TABLE IV
MICROHARDNESS OF DENSIFIED SAMPLES

Sl.No	Condition	Average Hardness HV _{1.0 kg} Al 2024
1	Conventional sintering	32
2	ECAP- I pass without back pressure	40
3	ECAP- I pass with back pressure	43
4	ECAP- II pass without back pressure	45
5	ECAP- II pass with back pressure	47

The hardness of the conventionally compacted and sintered and ECAPed samples are given in Table IV. A significant increase in hardness is attained in the ECAPed samples as compared to the conventional sintering. The higher hardness is obtained by imposing back pressure. The increase in hardness value is in line with the density obtained. Since the hardness recorded is based on the average of atleast six readings for each measurement, the results indicate that the specimen is hardened (densified) uniformly

The integrity of the consolidated powders was demonstrated not only by the almost full densities obtained, but also by examining the hardness and by optical metallograph. In Al 2024 compacted by conventional sintering and ECAP aided with and without back pressure, no defects such as cracks or blisters on the outside surface and internal parts were found. Almost full densification of composite powders (over 97% of relative density) without surface cracks has been obtained through ECA pressing method. In order to evaluate the Young's modulus of the compact, Nano Indentation technique was used. The load versus depth of indentation is plotted as shown in the Fig. 5.

TABLE V
YOUNG'S MODULUS FOR DENSIFIED SAMPLES

Sl.No	Condition	Young's Modulus, GPa Al 2024
1	Conventional sintering	40.44
2	ECAP- II pass with back pressure	85.7

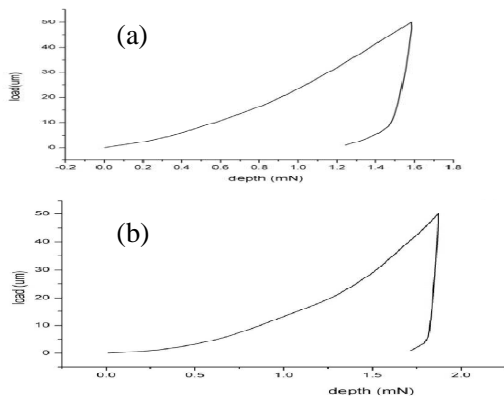


Fig. 5 Load versus depth graph of the compacted powder
(a) Al -2024 densified by conventional sintering
(b) Al -2024 densified by ECAP (After two passes with back pressure)

The Young's modulus calculated from the slope of the unloading curve is shown in Table V. The modulus of Al 2024 is 73 GPa. In the case of conventional sintering, the modulus for Al 2024 is 40.44 GPa and 55.56 for the composite. As we know that Young's modulus (E) is inversely related to the level of density achieved with respect to theoretical density (ρ , by the equation $E = E_0(\rho)^3$) where E_0 is the Young's modulus in fully densified condition. Thus the Young's modulus for the Al 2024 in conventional sintered condition ($\rho = 0.85$) is 44.83. The determined values are matching with the experimentally determined values. In the same way, the modulus for the compacts processed through ECAP should be 66.7 GPa for Al 2024 ($\rho = 0.97$). However, the experimentally determined values (Table V) are about 1.28 times higher than the calculated values. The higher value of modulus due to ECAP may be due to the strong texture formed during ECAP. However, a definite conclusion could not be arrived without texture measurements which are in progress. The optical micrograph (Fig. 3 and 4) of longitudinal sections and mechanical properties (Hardness) and the Young's modulus implies that a good bonding between powders is found after 2 pass ECAP with back pressure. The good bonding is attributable to the combined effect of hydrostatic pressure and shear stress.

IV. CONCLUSION

Bulk Al 2024 from powder mixtures were processed both by conventional sintering and room temperature ECAP aided by back pressure in two passes in route A to achieve densification and a near full density was achieved in ECAP.

The maximum density obtained, due to sintering (660^o C for a time period of 2 hours) of green compacts made by uni axial

compaction with load of 15 tons) was only about 85%. But room temperature ECAP results in dense compacts (about 97%). It was found by the micro hardness tests and density measurements that effective densification, homogeneous microstructure and high mechanical strength (Hardness and Young' modulus) could be achieved effectively as a result of the severe plastic deformation of ECAP and the well bonded powder contact surface during powder ECAP. Thus, ECAP is much economical and effective route in densification process as compared to conventional techniques. This high densification as well as good powder bonding represents the promising future of ECAP for powder processing. The main deformation mode in ECAP of solid (non-porous) materials is simple shear involving large plastic shear deformation in a very thin deforming layer of a work piece moving through a die. However, the volume of porous work piece can be changed according to the imposed hydrostatic stress, because the deformation mode in powder ECAP is not as simple as for non-porous materials. Finite element simulations for densification during ECAP based on the constitutive model of porous materials are now underway.

REFERENCES

- [1] Z.V.Ruslan and Terence G. Langdon " Principles of equal-channel angular pressing as a processing tool for grain refinement" *Progress in Materials Science*, vol.51,2006, pp 881-981
- [2] P.Quang, Y.G.Jeong, S.C.Yoon, S.H.Hong and H.S.Kim "Consolidation of 1 vol.% carbon nanotube reinforced metal matrix nanocomposites via equal channel angular pressing" *Journal of Materials Processing Technology*, **187-188**,2007, pp 318-320.
- [3] K.Morsi and S.Goyal "Equal channel angular pressing followed by combustion synthesis of titanium aluminides" *Journal of Alloys and Compound*, Vol. **429**, 2006, pp L1-L4.
- [4] Pham Quang, Young Gi Jeong, Seung Chae Yoon, Sun Ig Hong, Soon Hyung Hong and Hyoung Seop Kim, "Carbon Nanotube Reinforced Metal Matrix Nanocomposites Via Equal channel angular pressing" *Materials Science Forum*, Vols. **534-536**, 2007, pp 245-248
- [5] Seung Chae Yoon, Sun Ig Hong, Soon Hyung Hong and Hyoung Seop Kim "Densification and Consolidation of powders by Equal channel angular pressing" *Materials Science Forum*, Vols. 534-536, 2007, pp.253-256
- [6] S.Goussous, W.Xu, X.Wu and K.Xia " Al-C nanocomposites consolidated by back pressure equal channel angular pressing" *Composites Science and Technology*, Vol. 69, 2009, pp 1997-2001
- [7] R.Lopovok "Damage Evolution under Severe Plastic Deformation" *International Journal of fracture*, vol.115, 2002, pp 159-172
- [8] R.Lopovok, D.Tomus and C. Bettles " Shear Deformation with imposed hydrostatic pressure for enhanced compaction of powder" *Scripta Materialia*, Vol. 58, 2008, pp 898-901
- [9] K.Xia and X. Wu " Back pressure equal channel angular consolidation of pure Al particles" *Scripta Materialia*, vol.53, 2005, pp 1225-1229
- [10] W.Xu, X.WuX, T. Honma, S. P. Ringer and K. Xia " Nanostructured Al-Al₂O₃ composite formed in situ during consolidation of ultra fine Al particles by back pressure equal channel angular pressing" *Acta Materialia*, vol.57, 2009, pp 4321-4330
- [11] Tiancheng Zhang and D.Y.Li "Improvement in the resistance of aluminum with yttria particles to sliding wear in air and in a corrosive medium" *Wear*, Vol. 251, 2001, pp 1250-1256
- [12] V.M.Segal "Materials Processing by simple shear" *Materials Science and Engineering A*, Vol. 197, 1995, pp 157-164
- [13] Balog Martin, Simanick Frantisek, Bajana Otto, Guillermo Requena "ECAP vs direct extrusion Techniques for consolidation of ultra fine Al particles" *Materials Science and Engineering A*, vol.504,2009, pp 1-7
- [14] Ramu Yarra, P.Venkatachalam, S.Ramesh Kumar, B.Ravisankar, K.Jayasankar and P.S. Mukherjee " Densification of Al-Y₂O₃ composite powder by equal channel angular pressing" *Transactions of Indian Institute of Metals*, vol.63,2010, pp 813-817.