Trans Indian Inst Met (August 2012) 65(4):381–386
DOI 10.1007/s12666-012-0149-2

TECHNICAL PAPER

TP 2565

Densification of Al-2024 and Al-2024/Al_2O_3 Powders by Conventional P/M Route and ECAP: A Comparative Study

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Received: 21 November 2011 / Accepted: 30 May 2012 / Published online: 30 June 2012

Abstract In this study, mechanically alloyed Al-2024 and Al-2024/Al_2O_3 powders are 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. Nano Indentation technique was used to determine the modulus of the consolidated compacts.

Keywords Al-2024, Al-2024 composites · Back pressure ECAP · Nano indentation · Mechanical alloying

1 Introduction

The ultra fine grained powders processed by ''bottom-up'' approaches such as inert gas condensation, electrodeposition, ball milling, cryomilling, 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 pressure 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 yields insufficient straining and improper consolidation [12]. Due to high pressing pressures (over 1.4 GPa) needed for breakthrough of the compacted material, direct extrusion was not possible below 350 °C.

Recent research has shown that large bulk solids, can be produced in an essentially fully-dense state, by the top down approach 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 minimises 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 lower (at least three 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

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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 literature is available in back pressurised ECAP processes for consolidation of Al-2024 and Al-2024/Al_2O_3 composite powders. In this paper, ball milled Al-2024 and Al-2024/Al_2O_3 composite powders were 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 powders.

2 Experimental Work

Powders used in this study were elemental powders of Al, copper, magnesium, manganese, iron, silicon, zinc, titanium, and chromium. Alumina powder (10 wt%) was added for reinforcement. The composition of Al-2024 is given in Table 1.

Table 1 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	0.10	Balance

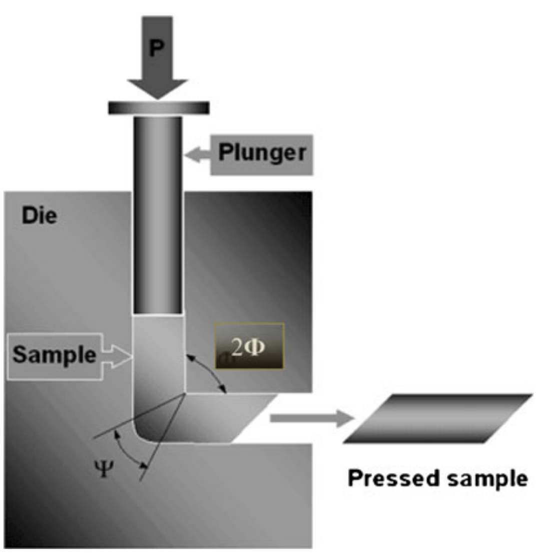

Fig. 1 Schematic representation of ECAP die (Channel angle $2\Phi = 90°$ and $\psi = 20°$)

Al-2024 and Al_2O_3 dispersion strengthened Al-2024 aluminum was prepared by mechanical alloying route. The powder mixture (Al-2024-10 wt% Al_2O_3) had been subjected to pot milling with tungsten balls. Tungsten balls with a diameter of 10 mm 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 and Al-2024/Al_2O_3 powders are consolidated by conventional powder metallurgy route i.e.

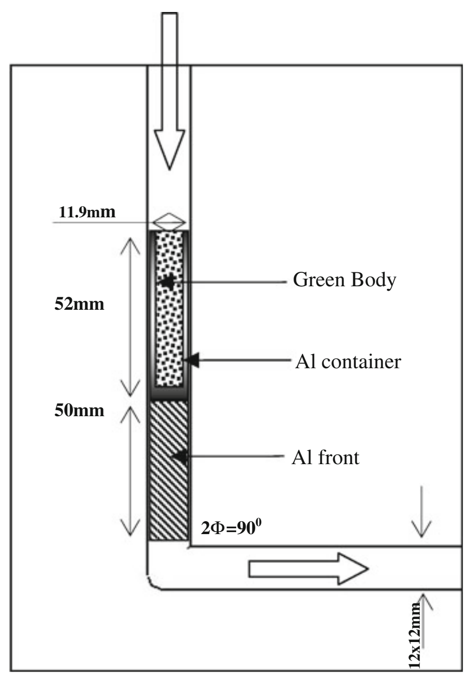

Fig. 2 Schematic representation of imposing back pressure in ECAP using Al front stopper

Table 2 Density of compacts

Sl. No	Condition	Theoretical density (%)	
		Al-2024	Al-2024/Al_2O_3
1	Compacted and sintered	85	84.4
2	After I pass of ECAP without back pressure	88	86.2
3	After I pass of ECAP with back pressure	92	90.3
4	After II pass of ECAP (route A) without back pressure	95	92.4
5	After II pass of ECAP (route A) with back pressure	97	95.2

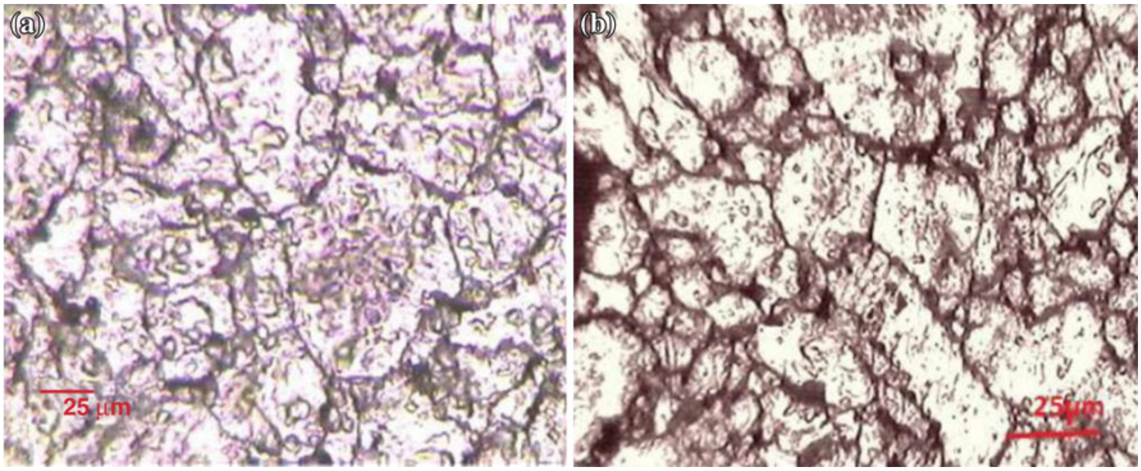

Fig. 3 Microstructures of sintered samples **a** Al-2024 alloy **b** Al-2024/Al_2O_3

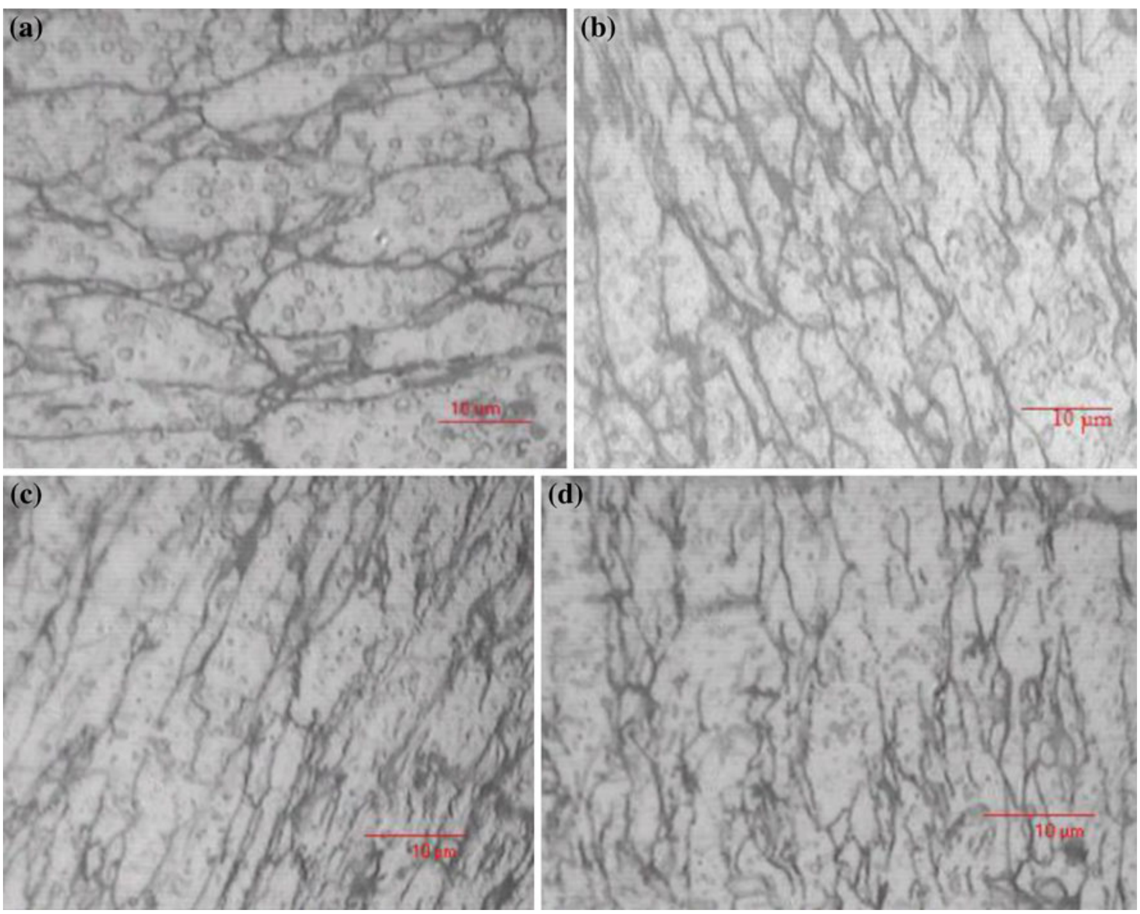

Fig. 4 Microstructures of alloy Al-2024 **a** I pass without back pressure **b** I pass with back pressure **c** II pass without back pressure **d** II pass with back pressure

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

Table 3 Grain size of Al-2024 and Al-2024-alumina composite densified by conventional sintering and ECAP

Sl.No	Condition	Average grain size (μm)	
		Al-2024	Al-2024-alumina composite
1	Conventional sintering	26	20
2	ECAP-I pass without back pressure	18	14
3	ECAP-I pass with back pressure	14	12
4	ECAP-II pass without back pressure	09	08
5	ECAP-II pass with back pressure	07	06

Table 4 Microhardness of densified samples

Sl.No	Condition	Average hardness $HV_{1.0\ kg}$	
		Al-2024	Al-2024-alumina composite
1	Conventional sintering	32	37
2	ECAP-I pass without back pressure	40	75
3	ECAP-I pass with back pressure	43	77
4	ECAP-II pass without back pressure	45	79
5	ECAP-II pass with back pressure	47	81

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 h after each coating. The coated compacts were sintered in a muffle furnace at 660 °C for a time period of 2 h. After 2 h 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.

In order to consolidate the Al-2024 and the composite 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 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 200 g and 15 s, respectively. The reported micro hardness value for each sample is the average of at least six measurements. The Young's modulus was evaluated using Nano Indentation by calculating the slope of the unloading curve.

3 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 2. The theoretical density of Al-2024 is 2.80 g/cc and the theoretical density of Al-2024/Al_2O_3 composite powder was calculated based on rule of mixture and found to be 2.90 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.

The micrographs of sintered samples are shown in Fig. 3 for both Al-2024 and Al-2024 alumina composite. The particles are reasonably well distributed with in Al-2024 matrix. The interface between matrix and reinforcement could not be detected using the optical metallograph that requires TEM studies which is in progress. 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) revel 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.

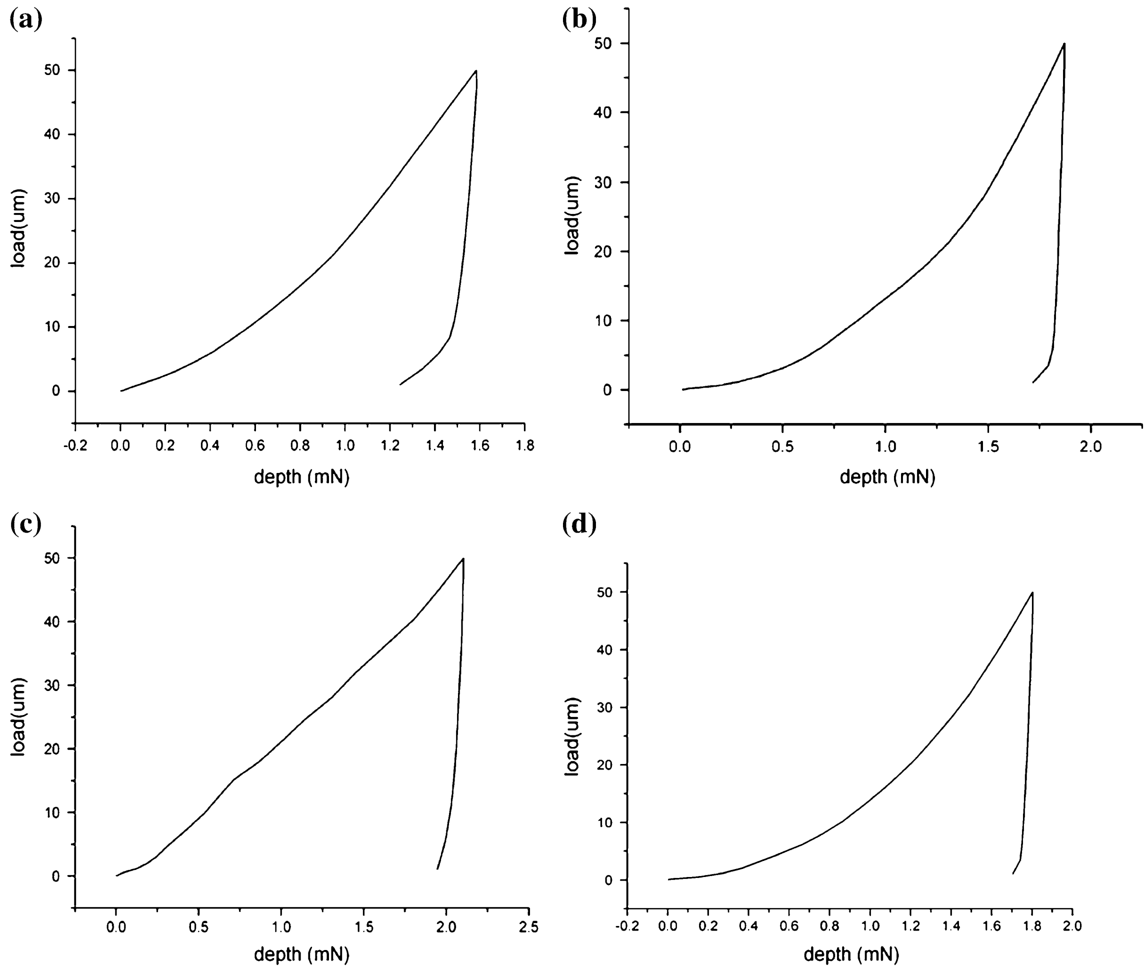

Fig. 5 Load verses depth graph of the compacted powder. **a** Al-2024 densified by conventional sintering **b** Al-2024 densified by ECAP (after two passes with back pressure) **c** Al-2024/alumina composite densified by conventional sintering **d** Al-2024/alumina composite densified by ECAP (after two passes with back pressure)

The average normalised grain size for conventionally sintered and ECAPed samples is shown in Table 3.

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. In the case of Al-2024/alumina composites the particles located inside the grains may have acted directly as the obstacle to dislocation movement, raising the yield strength by dispersion hardening because of Orowan's mechanism. The higher load applied for the movement of dislocation also used for the closure of voids. Modeling work has shown that Orowan mechanism plays major role in densification than strengthening through load transfer in composites [9, 14]. However, detailed TEM studies are necessary to suggest the mechanism of bonding which is in progress.

The hardness of the conventionally compacted and sintered and ECAPed samples are given in Table 4.

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. However, in the case of the powders densified by ECAP shows many fold increase in hardness though the increase in density especially in Al-2024-10 wt% alumina composite. Since the hardness recorded is based on the average of at least 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 and Al-2024/alumina composite compacted by

Table 5 Young's modulus for densified samples

Sl. No	Condition	Young's modulus (GPa)	
		Al-2024	Al-2024-alumina composite
1	Conventional sintering	40.44	55.56
2	ECAP-II pass with back pressure	85.7	95.02

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.

The Young's modulus calculated from the slope of the unloading curve is shown in Table 5.

The modulus of Al-2024 is 73 GPa and for the composite, based on the rule of mixture, is 95.7 GPa. In the case of conventional sintering, the modulus for Al-2024 is 40.44 and 55.56 GPa 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 and for composite ($\rho = 0.84$) 57.53 GPa. 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 ($\rho = 0.97$) and 82.6 GPa for composite ($\rho = 0.95$). However, the experimentally determined values (Table 5) are about 1.28 and 1.15 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 (Figs. 3, 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.

4 Conclusion

Bulk Al-2024 and Al-2024-alumina composites 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 °C for a time period of 2 h) of green compacts made by uniaxial 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 boding 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.

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