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Influence of twist extrusion process on consolidation of pure aluminum powder in tubes by equal channel angular pressing and torsion

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Abstract: In comparison with the conventional equal channel angular pressing (ECAP) process, a comprehensive study of influence of twist extrusion (TE) process on consolidating pure aluminum powder in tubes (PITs) by equal channel angular pressing and torsion (ECAPT) was conducted via three-dimensional (3D) finite element simulation, experimental investigation and theoretical analysis. Simulation results revealed that during the consolidation of aluminum powder particles by ECAPT, TE process played a significant role of back pressure. Due to the torsional shear and high hydrostatic pressure exerted by twist channel, both the magnitude and homogeneity of the effective strain were increased markedly. After one pass of ECAPT process using a square channel with an inner angle of 90° and a twist slope angle of 36.5° at 200 °C, commercial pure aluminum powder particles were successfully consolidated to nearly full density. Simulation and experimental results showed good agreement. In the microstructure observations, grains were greatly refined. At the same time, porosities were effectively eliminated by shrinking in size and breaking into small ones. Microhardness test indicated that strain distribution of ECAPT-processed billet was more homogeneous with respect to the ECAP-processed one. All these improvements may be attributed to the extreme intense shear strain induced during ECAPT and the increase in self-diffusion coefficient of aluminum due to the back pressure exerted by TE process.
Key words: aluminum powder; equal channel angular pressing and torsion; powder consolidation; back pressure

1 Introduction

Severe plastic deformation (SPD) is an attractive and effective method to fabricate bulk ultrafine-grained (UFG) materials. It has been extensively investigated by several groups [1−3] in the past two decades. Among these various SPD methods, equal channel angular pressing (ECAP) designed by SEGAL [4,5] is the most popular, cost-effective and prominent way due to the simplicity of process and tooling. However, to obtain well-defined and stable microstructure, it is generally necessary to repeat the ECAP process, which is time consuming, difficult to control and labor-intensive. Therefore, developing new SPD methods to increase process efficiency, in terms of imposing larger strain on the sample during individual pass to reduce the number of passes, is of great importance.

In recent years, many new SPD methods based on conventional ECAP process have been proposed for preparing bulk UFG materials, which include twist extrusion (TE) [6,7], twist channel angular pressing [8,9], half channel angular extrusion (HCAE) [10], equal channel angular pressing and torsion (ECAPT) [11], back pressure-equal channel angular pressing (BP-ECAP) [12], etc. In these processes, ECAPT is a novel, effective and promising method, which combines both ECAP and TE processes. Specifically, ECAPT process improves the conventional ECAP process by manufacturing a twist channel in the horizontal part of die channel, which can lead to higher values of the imposed strain for each pass as well as higher homogeneity. Currently, ECAPT process has attracted considerable attention due to its unique die design and enticing prospects, and several achievements have been obtained in the field of ECAPT research [13,14]. However, as it is mentioned

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above, ECAPT is known as one of rather newly developed SPD methods, the deformation mechanism of ECAPT process is very complicated, and still lacks a comprehensive and profound understanding. It has been shown that twist channel is a very important part in ECAPT die and has a significant influence on the density, microstructure and mechanical properties of the extruded billet [15].

In the present study, one pass of ECAPT process was conducted for consolidating commercial aluminum powder in tubes (PITs) at 200 °C. By comparing with the billet produced by conventional ECAP process, the influence of TE process on consolidating pure aluminum powder in tubes by ECAPT (PITs-ECAPT) was intensively studied using finite element simulation, experimental investigation and theoretical analysis. The correlation of grain refinement and mechanical property enhancement with the imposed effective strain was also explored. The aim of this study is to grasp a fully understanding and a detailed mapping of ECAPT deformation mechanism, and to provide a theoretical guidance for fabricating bulk UFG materials with homogeneous, porosity-free, fine microstructures and excellent properties from the metallic powder particles.

2 Principle of PITs-ECAPT process

The general principle of PITs-ECAPT process is schematically shown in Fig. 1.

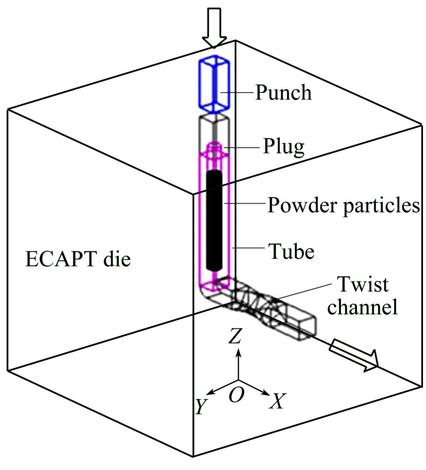

Fig. 1 Schematic diagram of PITs−ECAPT process

The most important feature of ECAPT process is that two processes named ECAP and TE are carried out subsequently in a single die and the need for additional equipment is eliminated. The powder may be pre-canned in tubes, with or without an isostatic compaction step, and then prepared by closing the tube with a plug after filling with the powder. Firstly, a billet is subjected into the vertical channel of ECAPT die and is forced around a corner between two channels of equal dimensions, then pressed through the twist channel situated in the horizontal part of the channel. Ultimately, the billet emerges from the die with no change of cross-sectional dimensions. During ECAPT process, the billet undergoes extensive shearing twice (i.e., ECAP and TE) in a single tool and is severely deformed, producing exceptional grain refinement and property enhancement. Moreover, the billet may be pressed repetitively through the die in order to attain a very high strain, which can effectively break up the original texture into ultrafine or even nanostructures and greatly improve mechanical properties. In addition, for consolidating powder particles, ECAPT process has other many potential advantages over conventional ECAP process，such as creating a back pressure during the consolidation of powder particles and thus preventing delaminating and surface cracking; a large potential for strengthening the metal from powder particles with homogeneous microstructures and good particles bonding.

3 Finite element model and experimental procedures

3.1 Finite element model

The commercial metal forming finite element code DEFORM−3D^{TM} V6.1 was used to analyze the deformation behavior of aluminum powder in tubes during ECAPT process. Compressible rigid viscoplastic thermal−mechnical coupling model was used to determine simulation factors. The ECAPT die was a set as follows: a cross-section of 10 mm × 10 mm, die channel angle Φ=90°, outer corner angle ψ=37°, transitional channel length L_1=15 mm, twist channel length L=15 mm (i.e., twist slope angle β=36.5°), angle of the twist rotation α=90°. Commercial pure aluminum (99.9%) was selected as a raw material. The pure aluminum cans (10 mm × 10 mm × 60 mm) used to contain aluminum powders had a inner chamber of 7 mm in diameter and 50 mm in length. The ECAPT process was carried out at 200 °C in order to improve the poor workability of aluminum powders at ambient temperature. The initial packing density of aluminum powder was 75% and the aluminum consolidate was assumed as porous material with 25000 initial tetrahedral elements. Doraivelu yield criterion was used and the influences of effective strain, strain rate, temperature, and relative density on the flow stress of material were considered, i.e., $\sigma=\sigma(\bar{\varepsilon},\ \dot{\bar{\varepsilon}},\ T,\ \rho)$. The punch and the ECAPT die were suggested as rigid ones. Constant shear friction coefficient of 0.05 was used for simulation, and the extrusion speed was 1 mm/s. Automatic re-meshing was applied to accommodating severe plastic deformation during simulation analysis. For comparison, ECAP process was also simulated to prove the efficiency of ECAPT process. Figure 2 shows the established finite element model of ECAPT and schematic illustration of the twist channel, respectively.

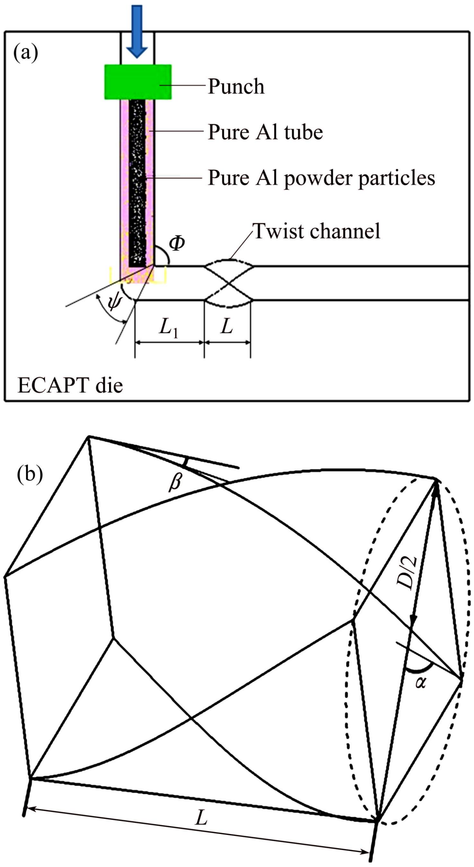

Fig. 2 Schematic illustration of ECAPT process: (a) Finite element model of ECAPT; (b) Schematic of twist channel

3.2 Experimental procedures

To verify the accuracy and reliability of simulation results, one pass of ECAPT process was performed in an appropriate ECAPT die which was designed and constructed (see Fig. 3(a)). The geometrical dimensions of ECAPT die and the extruded billet were identical to those used in finite element simulations. The specified composition of the as-received pure aluminum powder was as follows: *w*(Al)>99.0%, *w*(Fe)<0.60%, *w*(Cu) <0.05%, *w*(Si)<0.3%. Initial aluminum powder particles were of near spherical shape as shown in Fig. 3(b) and had an average diameter of 40.6 μm. MoS_2 was used as a lubricant. Pure aluminum cans were filled with aluminum powder particles (initial packing density of 75%) and then inserted into the vertical channel of lubricated ECAPT die. ECAPT process was carried out at 200 °C by a punch at a speed of 1 mm/s. The temperature of the set-up was raised by inductive heating. For comparison, one pass of ECAP process was also conducted under the same condition as that of ECAPT process.

After extrusion, aluminum consolidates were extracted from cans by machining. Different samples

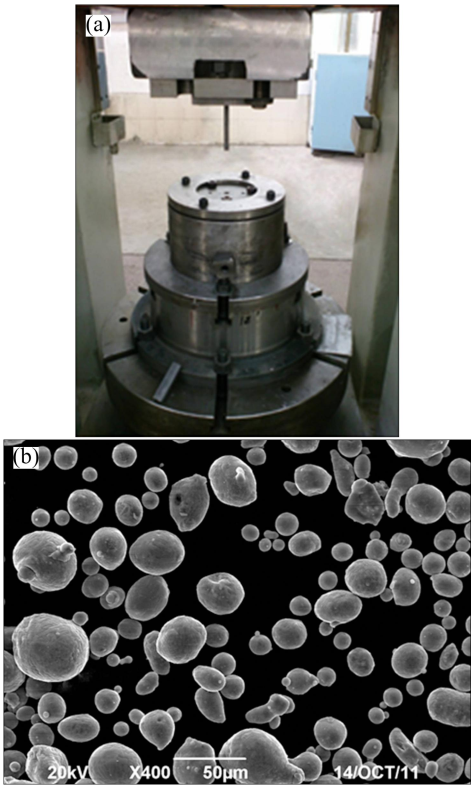

Fig. 3 Experimental die and morphology of as-received material: (a) Set-up of fabricated ECAPT die; (b) SEM image of initial aluminum powder particles

were carefully cut by wire electro-discharge machining (EDM) from homogeneously strained areas of aluminum consolidates. Samples for microstructure observation were cut from the central transverse plane (TP) and prepared by fine grinding and mechanical polishing, followed by etching in a solution of 3 mL HF + 97 mL H_2O for 15 s. An optical microscope (4XB−TV) was employed to examine the shape, size and distribution of the porosities and grains in the samples fabricated by ECAPT and ECAP processes. Densities of the samples with a volume of about 2 cm^3 were measured based on the Archimedes principle by using distilled water. In addition, to evaluate the effectiveness of the consolidation process, Vickers microhardness (HV) was measured at the central TP of the samples after applying each pass of ECAPT and ECAP processes at a load of 25 g and a dwell time of 10 s. To obtain an accurate reading, at least 10 points were selected for each sample, and all selected points were measured 3 times and then the average value was taken.

4 Results and discussion

4.1 Leading end shape of processed billets

Figure 4 compares the leading end shape of ECAP-processed billet with that of ECAPT-processed one by simulation and experiment.

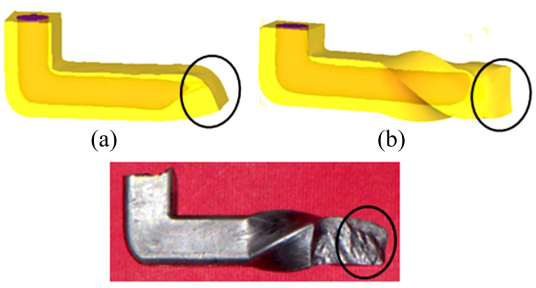

Fig. 4 Comparison of leading end shape of billets extruded by ECAP and ECAPT: (a) Simulation of ECAP; (b) Simulation of ECAPT; (c) Macrograph of billet during ECAPT process

It is obviously shown that TE process has a significant influence on the shape of the processed billet, and can effectively correct the phenomena of tilting and warping in the billets' leading end. At the initial stage of ECAPT process, the billet is extruded into the bend of the channel and suffers severe shear deformation by ECAP process first. As the leading end of billet enters the horizontal channel directly, no shear strain imposes on it. Moreover, due to the differences in flow velocity of metal particles on the upper and lower surfaces of the billet, tilting and warping are formed at the leading end. Subsequently, after a short rigid translation in the transitional channel, the leading end of the billet enters the twist channel situated in the horizontal part of the channel, and TE process starts. At this stage, deformation becomes steady, and the billet is passed through twist channel continuously until its tail leaves. Owning to the back pressure created by TE process, tilting and warping in the leading end of the billet disappear after completing a pass of ECAPT process. Therefore, a longer billet could be obtained by ECAPT process, which is suitable for mechanical testing and also perhaps for industrial application. Figure 4 also reveals that simulation findings (Fig. 4(b)) are in good agreement with the experimental results (Fig. 4(c)).

4.2 Load−stroke curve of simulation

Figure 5 presents the simulational load−stroke curves obtained during ECAPT and ECAP processes. It should be noted that five stages of the ECAPT load can be distinguished. Specifically, in the stage I region (i.e., during ECAP process), two curves of ECAPT and ECAP are almost overlapped. The load increases dramatically due to the intense shear strain accumulated in the bend of channels and reaches a local maximum, which is the maximum load needed for ECAP processing. However, the changing trend of two curves becomes completely different afterwards. For ECAP process, as the billet passes through the bend of channel continuously, it then enters the horizontal channel gradually, so the load is approximately steady and shows a slight decrease due to the friction between the billet and die channel. While for ECAPT process, after the first shearing in the bend of channels, the leading end of the billet enters twist channel subsequently, where extra torsional shear strain and large hydrostatic pressure imposed by TE process further make the billet deformed, causing work hardening. Therefore, larger force is required to continue the process, so an abrupt increase appears in the load−stroke curve of ECAPT process (stage II). In the twist channel, after achieving a local maximum of about 11.7 kN required for next TE process (stage III), the load becomes almost steady again. When the billet emerges from the twist channel, the load increases again with the maximum of about 13.7 kN (stage IV), which is the total force needed for ECAPT process under the conditions of our investigation. Afterwards, there is a sharp drop in load−stroke curve of ECAPT process (stage V), because the billet exits the twist channel at this moment and a single pass of ECAPT process completes.

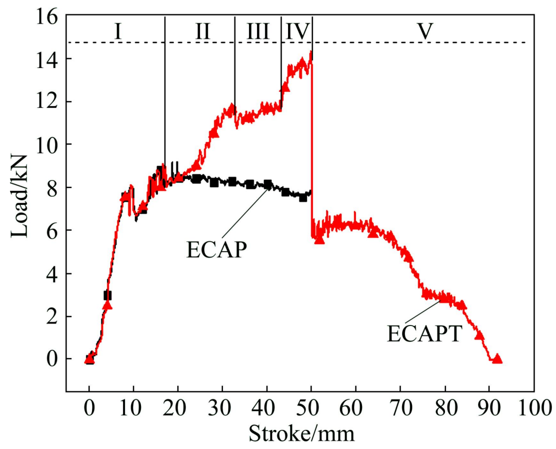

Fig. 5 Comparison of simulational load−stroke curves of ECAPT and ECAP processes

4.3 Simulational effective strain

It is well known that for the bulk UFG materials, the final obtainable microstructure and properties are mainly related to the magnitudes of strain imposed during SPD process [16]. Figure 6 shows the values and distribution of effective strain in the central TP of the processed billets during ECAPT and ECAP processes, respectively. Moreover, after one pass of ECAP and ECAPT, five tracking points located in the central TP of the processed billets are selected to reveal effective strain distribution.

As shown in Fig. 6, during ECAPT process, due to the additional torsional shear imposed on the billet exerted by twist channel, as well as the shear imposed by the shear plane of ECAP process, the values of effective strain are much higher. Moreover, it should also be noted

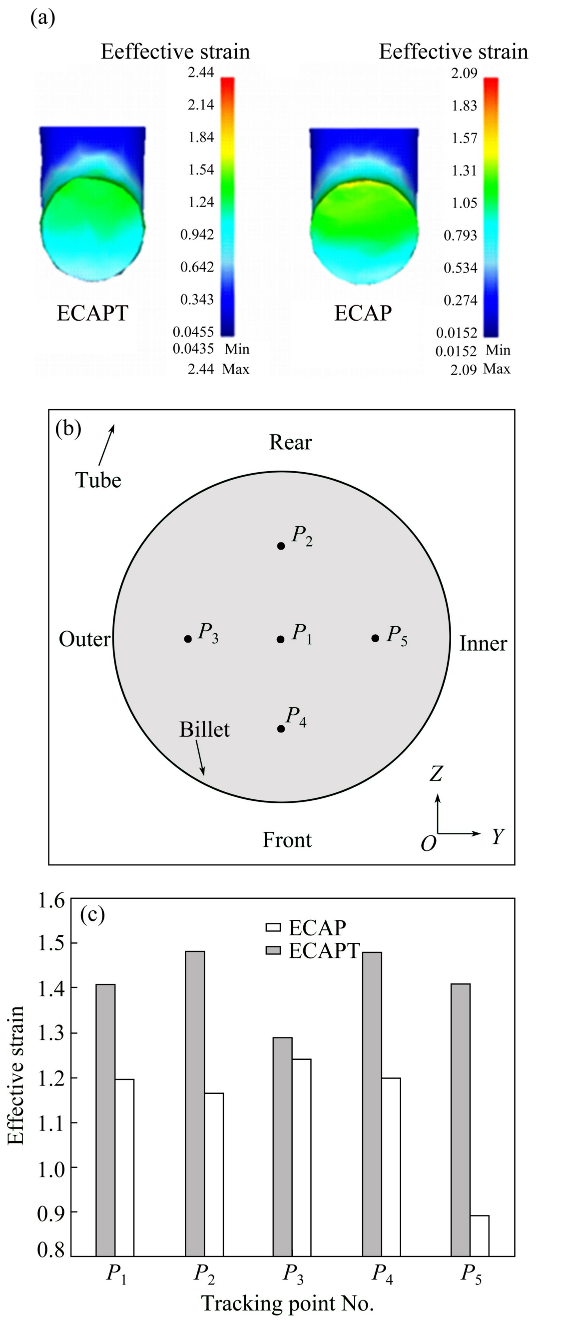

Fig. 6 Effective strain at central TP of billets processed by ECAPT and ECAP: (a) Effective strain contours; (b) Location of selected points; (c) Values of effective strain

that this strain increase is also accompanied by the simultaneous improvement of the strain homogeneity. Taking the central point P_1 for example, after a single pass of ECAP process, the value of effective strain is only 1.20 while it is as high as 1.41 after ECAPT process, with an increase of about 17.6%. Considering the strain homogeneity [17] at central TP further, the deformation inhomogeneity index of ECAP-processed billet is 0.31, while during ECAPT process, it drops to only 0.14, indicating that ECAPT process leads to a more homogeneous strain distribution. This is possible due to the severe torsional shear deformation in the region of twist channel, which forces the material particles into an intense vortex-like flow, causing the alternation of shear planes and shear directions. Consequently, the strain variation between the periphery and the center decreases and the strain homogeneity increases. Most importantly, the construction of twist channel in TE process allows the deformation to be applied under back pressure, which provides a sufficient resistance against material flow, and thus effectively prevents the porosities and cracks of the consolidated billets.

4.4 Microstructure observations

As seen from the simulation results, during ECAPT process, twist channel situated in TE process plays a role of back pressure. It is also clear that TE process increases the imposed shear strain as well as the hydrostatic pressure in the billet, decreases the free flow velocity of metal particles on the top surface of the billet, which are all beneficial for improving the deformation homogeneity and densification of consolidated material.

Figure 7 shows typical optical micrographs taken from the central TP in the billets produced after applying one pass of the ECAP and ECAPT processes at 200 °C. As shown in Fig. 7, in comparison with the conventional ECAP process, ECAPT process is much more effective in refining grains and eliminating porosities for the consolidated material. Referring to this figure, the optical observation of the material after ECAP reveals some level of porosity (Fig. 7(a)), while after ECAPT almost

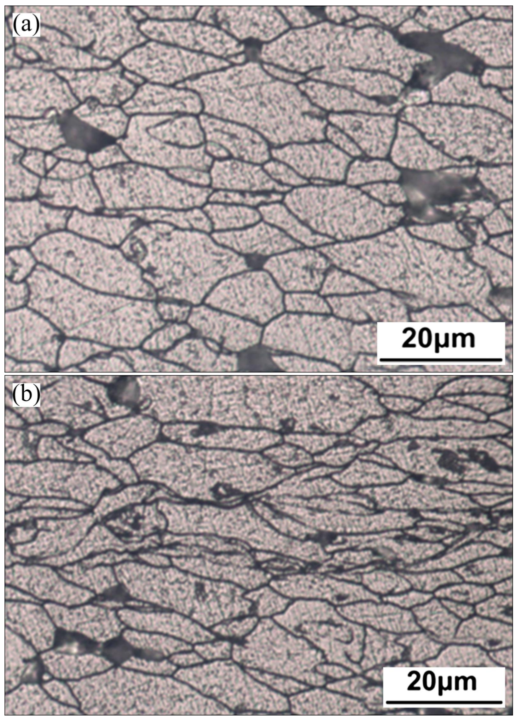

Fig. 7 Optical microstructures at central TP of billets processed by two processes: (a) ECAP; (b) ECAPT

no porosity can be observed (Fig. 7(b)). The density of the bulk material after ECAPT consolidation was measured as 2.67 g/cm^3 (i.e., the relative density increases from 0.75 to 0.99, with an increase of about 31.9%), showing that nearly full density is achieved at a relative low temperature (200 °C) after a single pass of ECAPT. Moreover, grains of the billet subjected to twist extrusion in succession after conventional ECAP process become more elongated, which leads to a significant refinement in the microstructure.

4.5 Microhardness test

Figure 8 represents the average microhardness values for each billet after one pass of extrusion.

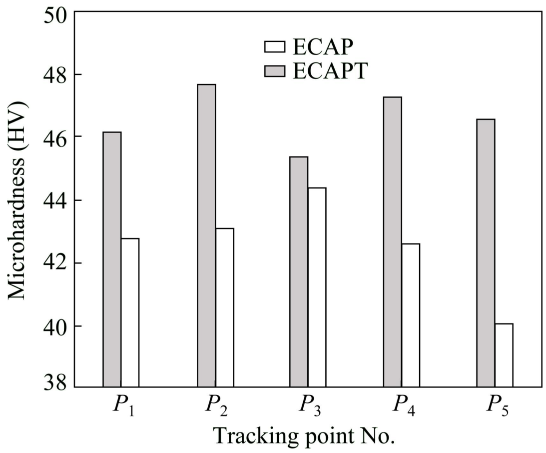

Fig. 8 Microhardness at central TP of billets processed by ECAPT and ECAP

As expected, for ECAPT-processed billet, the magnitude of hardness values and the homogeneity of value distribution are higher than those of ECAP-processed one. The difference in the microhardness average value can be an evidence of stronger bonding between the particles in ECAPT process with respect to that of ECAP process. In fact, the extra torsional shear strain, high hydrostatic pressure [18] and back pressure exerted by TE process which subsequently takes place after conventional ECAP process, improve the consolidation process of aluminum powder particles and significantly reduce the population of residual porosities as well as average grain size. Moreover, it is necessary to mention that for the extruded billets, the microstructure is in accordance with the predicted effective strain distribution, which also validates the reliability of simulation results shown in Fig. 6.

LAPOVOK et al [19] think that the increase in strength and relative density achieved by ECAP with back pressure at low temperatures may be attributed to the improved self-diffusion at the particles interface due to three factors, which are contact area between the neighboring particles, temperature and hydrostatic pressure. Therefore, in order to further investigate the influence of back pressure exerted by TE process on consolidating aluminum powder particles, self-diffusion coefficient for aluminum during ECAPT process is calculated on the basis of the previous simulation results.

In general, the self-diffusion coefficient for aluminum is calculated using the Arrhenius equation of the form as follows [20]:

$$D = D_0 \exp\left(\frac{-Q}{RT}\right) \tag{1}$$

where D_0 represents the frequency factor, Q is the activation energy, R refers to the mole gas constant and T is the thermodynamic temperature. It is seen that the self-diffusion coefficient increases with the decrease of activation energy. Using D_0=0.1 cm^2/s and Q=134.6 kJ/mol for aluminum and T=473 K yields the self-diffusion of aluminum as D_1=1.36×10^{-16} cm^2/s.

As shown in the load−stroke curves in Fig. 5, the load reaches a local maximum of 11.7 kN as the billet enters the twist channel after passing through the well-known shear plane of ECAP, and then the load becomes almost steady. This phenomenon arises from the fact that TE process designed in the horizontal channel creates a back pressure for the consolidation of powder particles. In ECAPT process, the pressure needed for extrusion of the billet in the exit channel is simply determined by dividing the extrusion force (11.7 kN) by the cross-section area of the billet (10 mm × 10 mm). This yields an extrusion pressure of about 117 MPa, which acts a back pressure for ECAP process.

The decrease in activation energy due to the back pressure can be estimated by the following equation [21]:

$$Q_{\mathrm{HP}} = \left(P + \frac{\sigma_{\mathrm{y}}}{\sqrt{3}}\right)\cot\left(\frac{\Phi}{2}\right)\Omega \tag{2}$$

where P is the applied back pressure during ECAP, σ_{y} is the yield strength of the material at a given temperature T, Φ is the die channel angle of ECAP die, and Ω is the mole volume. Inserting P=117 MPa, σ_{y}=60 MPa, Φ=90° and Ω=1.66×10^{-29} m^3 into Eq. (2) results in a 1.515 kJ/mol decrease in activation energy. As a result, the decrease in activation energy causes the self-diffusion coefficient of aluminum increases to D_2=2.05×10^{-16} cm^2/s.

Likewise, for the conventional ECAP process without an applied back pressure (i.e., P=0), the self-diffusion coefficient for aluminum is obtained as D_3=1.52×10^{-16} cm^2/s at 200 °C. By comparing D_1, D_2 and D_3, it should be noticed that the self-diffusion coefficient of aluminum in ECAPT process effectively increases by a factor of 34.9 % and 50.7% relative to its value during conventional ECAP process and in a condition that no load is applied, respectively.

The calculation analysis above is similar to the results of ECAP−FE process reported in Ref. [20] and is also in good agreement with our simulation and experimental results, thus showing the significant advantages of back pressure exerted by TE process in porosities elimination, grain refinement and mechanical property enhancement.

5 Conclusions

1) The TE process designed in the horizontal channel of conventional ECAP die creates a back pressure for consolidation of aluminum powder particles in ECAPT process. Finite element simulation results show that the extrusion load increases significantly, and reaches a local maximum of 11.7 kN when the billet enters twist channel due to the work hardening and high hydrostatic pressure.

2) During ECAPT process, both ECAP and TE processes are carried out subsequently in a single tool, so the billet undergoes severe shear deformation twice. The combination of the shear strain in the shear plane of ECAP and the shear torsion strain in the twist channel of TE leads to the changes in metal flow and shear pattern, causing an increase in effective strain values and strain distribution homogeneity.

3) Due to the high strain imposed during ECAPT and the back pressure exerted by TE process, a nearly full dense bulk aluminum with an excellent particles bonding is successfully produced by one pass of ECAPT process at 200 °C. The microstructure and microhardness of the consolidated billet improve significantly with respect to the conventional ECAP process. The results also show that the back pressure created by TE process effectively increases the self-diffusion coefficient of aluminum.

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挤扭成形对等径角挤扭工艺固结纯铝粉末−包套过程的影响

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摘 要：采用 3D 有限元模拟、实验研究和理论分析，并在与传统等径角挤压工艺对比基础上，系统研究挤扭成形对等径角挤扭工艺固结纯铝粉末−包套过程的影响。模拟结果表明，在等径角挤扭法固结纯铝粉末−包套过程中，挤扭成形起反向背压作用，螺旋通道所提供的旋转剪切变形和高静水压力可大幅增加材料内部的塑性剪切应变，显著改善变形坯料的变形均匀性。在内角为 90°、螺旋角为 36.5°的方形截面通道模具上，经 200 °C 下 1 道次等径角挤扭变形实验，成功将纯铝粉末颗粒固结为近致密的块体材料。有限元模拟与实验结果具有较好的一致性。显微组织观察和硬度测试实验结果表明，等径角挤扭法固结的块体材料晶粒更加细小，孔隙得到有效收缩焊合，组织性能均匀性更好。这是由于在等径角挤扭变形过程中剧烈剪切应变大大增加，同时挤扭成形所起的反向背压作用有效提高了 Al 原子的自扩散系数。

关键词：铝粉；等径角挤扭；粉末固结；背压

(Edited by Wei-ping CHEN)