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Principles of ECAP–Conform as a continuous process for achieving grain refinement: Application to an aluminum alloy

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Abstract

Equal-channel angular pressing (ECAP) combined with the Conform process provides a solution for the continuous production of ultrafine-grained materials. Rods of a commercial Al-6061 alloy were processed by ECAP–Conform at room temperature for up to a total of four passes. Microstructural observations showed significant grain refinement but with elongated grains after four passes with average widths of ~150 nm and lengths of ~1.2 μm when viewed on the longitudinal planes. Microhardness measurements after a single pass revealed inhomogeneities both on the cross-sectional planes and along the rod. After processing through four passes there was reasonable homogeneity throughout the rod. Measurements of the shear strengths in two orthogonal directions perpendicular to the extrusion axis showed significant strengthening after ECAP–Conform and there was no evidence for any plastic anisotropy after processing through four passes.

Keywords: Conform; Equal-channel angular pressing; Hardness; Homogeneity; Plastic anisotropy

1. Introduction

The processing of metals through the application of severe plastic deformation (SPD) has attracted much attention because of the potential for achieving remarkable grain refinement to the submicrometer or even, under some conditions, to the nanometer range [1]. Several different SPD processing methods are now available [2] but the procedure receiving the most attention is equal-channel angular pressing (ECAP) where the sample, in the form of a relatively short rod or bar, is pressed through a die constrained within a channel bent through an abrupt angle [3].

Processing by ECAP has proven effective for a wide range of metals, intermetallics and composites and it has the advantage that it may be scaled up for the production of relatively large bulk samples [4,5]. Nevertheless, ECAP has three critical disadvantages. First, and most important, conventional ECAP is a discontinuous process involving a repetitive sequence in which the billet is inserted into the die, pressed through the die, removed and then reinserted to impose an even higher strain. This means that, although ECAP is an effective processing tool for laboratory research, it is labor-intensive and not easily adapted for use in industrial operations. Second, there is a limitation on the total lengths of the billets employed in ECAP because the length is restricted both in order to maintain a critical aspect ratio and as a consequence of the limitation on the total available displacement of the pressing ram. Third, there is a problem because very significant waste is incurred with ECAP due to the non-uniform deformation

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and gross distortions that are introduced at the front and back of every billet. For example, detailed hardness measurements were recorded on longitudinal sections of pressed billets of an aluminum alloy and they showed there was an effective length of uniform flow occurring over a distance of ∼38 mm for billets having initial lengths of 70 mm [6]. These measurements suggest that conventional ECAP may entail a potential material wastage of >40% and this means in practice that successful applications of the ECAP technology are probably most effective in developing high-cost products such as medical implants [7].

Several procedures have been proposed for the continuous processing of metals by ECAP where these processes generally involve incorporating an ECAP step, in the form of an abrupt bend, into the exit channel of a rolling facility. Examples of this approach include the continuous confined strip shearing (C2S2) process [8], equal-channel angular rolling [9,10], conshearing [11] and continuous frictional angular extrusion [12]. Nevertheless, these approaches have received only limited attention to date for the processing of large batches of materials.

An alternative approach is based on the Conform process, which was introduced many years ago for the continuous extrusion-forming of metal wires [13,14]. A pioneering study of the ECAP–Conform process, in which an ECAP step was introduced in the exit channel from a conform facility, showed this procedure was capable of producing significant grain refinement in commercial purity (CP) aluminum with grain sizes of ∼650 nm after processing [15]. Subsequently, the procedure was applied also to CP titanium and the resultant grain size was ∼200–300 nm [16]. These results suggest, therefore, that ECAP–Conform provides a unique opportunity for achieving excellent grain refinement in a continuous SPD processing procedure.

The present investigation was initiated to critically evaluate the ECAP–Conform process with a special emphasis on two areas where no information is at present available. First, detailed hardness measurements were undertaken to evaluate the extent of the homogeneity within the microstructure after processing by ECAP–Conform with separate measurements taken to determine both the homogeneity within the cross-sectional planes of processed rods and the variations in microstructural homogeneity along the lengths of the processed rods. Second, mechanical testing was undertaken to determine the extent of any plastic anisotropy that may be present within the rods after processing by ECAP–Conform.

2. Experimental material and procedures

The experiments were conducted using a commercial Al-6061 aluminum alloy having a chemical composition, in wt.%, of 1.01% Mg, 0.59% Si, 0.37% Fe, 0.29% Cu, 0.23% Cr and 0.20% Zn. This alloy was selected because it was used earlier for an extensive evaluation of the hardness distributions in billets processed by conventional ECAP [17,18]. The alloy was received in an as-cast condition and it was extruded into bars having cross-sectional diameters of ∼4.0 mm and lengths of ∼20–25 cm.

Processing by ECAP–Conform was conducted at room temperature using the facility shown schematically in Fig. 1. The principle of processing is that, as in conventional conform, there is a rotating inner shaft and an outer stationary die. The material enters the facility constrained within a groove having a circular cross-section, it is carried forward by frictional forces so that it rotates with the shaft and moves into a groove having a rectangular cross-section, and ultimately it is forced to exit from the facility at an abutment where it is turned abruptly through an angle, Φ, of 90°. This latter displacement through 90° is not generally present in conventional conform and it represents the introduction of an ECAP step in the processing procedure. Since the strain introduced in ECAP is ∼1 for a displacement through an angle of 90° [19], it is reasonable to anticipate the strain introduced in the present investigation is also ∼1 for each passage through the facility. In the vicinity of the point of exit, the groove had a square cross-section with dimensions of 3.5 × 3.5 mm^2. All rods were processed by ECAP–Conform at a constant speed of 0.2 mm min^{-1} up to a maximum of four separate passes through the facility. As in ECAP, the rods were rotated in the same sense by 90° along their longitudinal directions between each pass in the procedure known as route B_C [20].

Fig. 2 shows examples of the rods used in this investigation where the upper rod is untested and the remaining rods were processed by ECAP–Conform through one and four passes, respectively, where the two processed rods are oriented so that the front is on the left and the back is on the right. It is important to note that the rods processed by ECAP–Conform are in excellent condition with no evidence for any macroscopic cracking.

Following processing, disks having thicknesses of 3 mm were cut from the as-pressed rods perpendicular to their longitudinal axes at the representative positions labeled A–C in Fig. 2 corresponding to sections near the front, near the center and near the back of the rods, respectively.

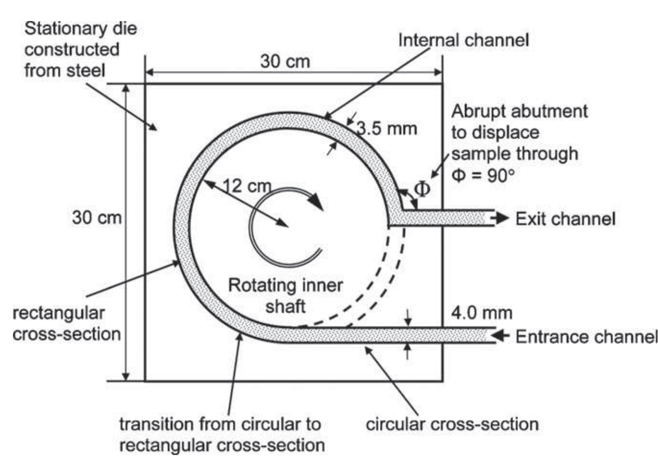

Fig. 1. Schematic illustration of the principle of the ECAP–Conform process.

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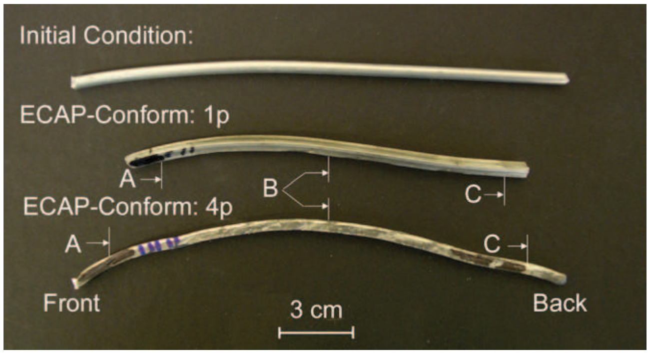

Fig. 2. Appearance of samples of an Al-6061 alloy in the initial unprocessed condition (upper) and after processing by ECAP–Conform (lower) through one and four passes (1p and 4p), respectively: the front and back of the rods are marked and the positions labeled A–C were used for detailed hardness measurements.

These disks were mounted and carefully polished to a mirror-like finish. Measurements of the Vickers microhardness, *Hv*, were recorded over the surface of each disk using an FM-1e microhardness tester equipped with a Vickers indenter. These measurements were taken following a rectilinear grid pattern with a spacing of 0.5 mm between each separate position. For comparison purposes, a similar set of measurements was also recorded on an unprocessed rod.

Samples were also prepared for microstructural examination by scanning electron microscopy (SEM) using an ion beam facility (JEOL SM-09010) at a temperature of 203 K. Samples were bonded to a mounting stub fixed on a moveable stage and aligned so that the bulk of the sample was protected by a shield and the remaining exposed material was milled away by the argon ion beam. All of the microstructural observations were performed using a JEOL JSM-7000F Field Emission scanning electron microscope operating at 20 kV. The structures were examined both on longitudinal planes parallel to the extrusion axis and on cross-sectional planes cut perpendicular to the extrusion axis.

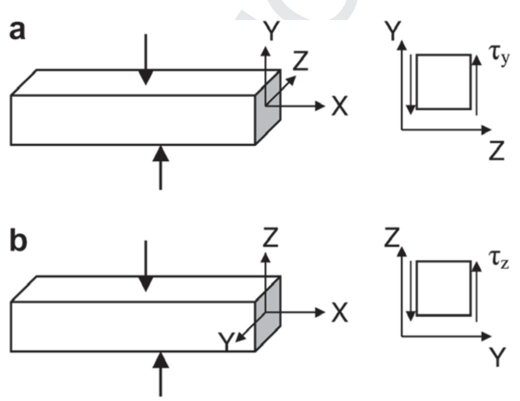

Fig. 3. Schematic illustration showing the two orientations of the samples used for shear testing: the *X* axis lies along the extrusion direction.

To evaluate the significance of any plastic anisotropy perpendicular to the extrusion axis after processing, samples were deformed to failure using an Instron Satec Model 5590 testing machine using a single shear test apparatus and following standard procedures [21–23]. The potential anisotropy was evaluated by testing samples cut from the rods in two different orientations. These orientations are shown in Fig. 3 where *X* denotes the extrusion direction and *Y* and *Z* are perpendicular to the side face and the upper face on exit from the ECAP–Conform facility, respectively. Thus, the samples were subjected to single shear to failure in the *Y* and *Z* directions, respectively, where τ_y and τ_z denote the relevant shear stresses. The mechanical testing was undertaken at room temperature (298 K) with the testing machine operating at a constant rate of cross-head displacement of 0.3 mm s^{-1}.

3. Experimental results

3.1. Microstructural observations

Examples of the microstructures are shown in Fig. 4 where (a) corresponds to the initial unprocessed condition and (b) and (c) denote representative microstructures recorded by SEM on longitudinal sections after processing through one and four passes, respectively. Prior to processing, the Al-6061 alloy exhibited a typical coarse-grained structure as shown in Fig. 4a with reasonably equiaxed grains and a measured average grain size of ~350 μm.

Following processing, it is apparent that the microstructures exhibit two important characteristics on the longitudinal sections. First, the grain size is significantly reduced after ECAP–Conform by comparison with the unprocessed condition. Second, the grains are elongated on these sections and close inspection showed the maximum lengths were aligned approximately along the extrusion axis. Measurements gave average grain sizes of ~700 nm in width and ~1.5 μm in length after one pass and ~150 nm in width and ~1.2 μm in length after four passes. These results show that both the grain width and the grain length decrease with additional passes through the ECAP–Conform facility. By contrast, the grain structures were reasonably equiaxed when viewed on the cross-sectional planes.

3.2. The distributions of microhardness values on cross-sectional planes

Following the procedures introduced earlier [17,24–28], the microhardness data may be plotted in the form of color-coded contour maps in which different colors are used to denote ranges of hardness values. The results are shown in Fig. 5 where the upper row represents one pass, the lower row represents four passes, and the images from left to right denote measurements taken at position A near the front of the extrusion, position B near the center of the extrusion and position C near the back of the extrusion, respectively, where these positions are shown in Fig. 2:

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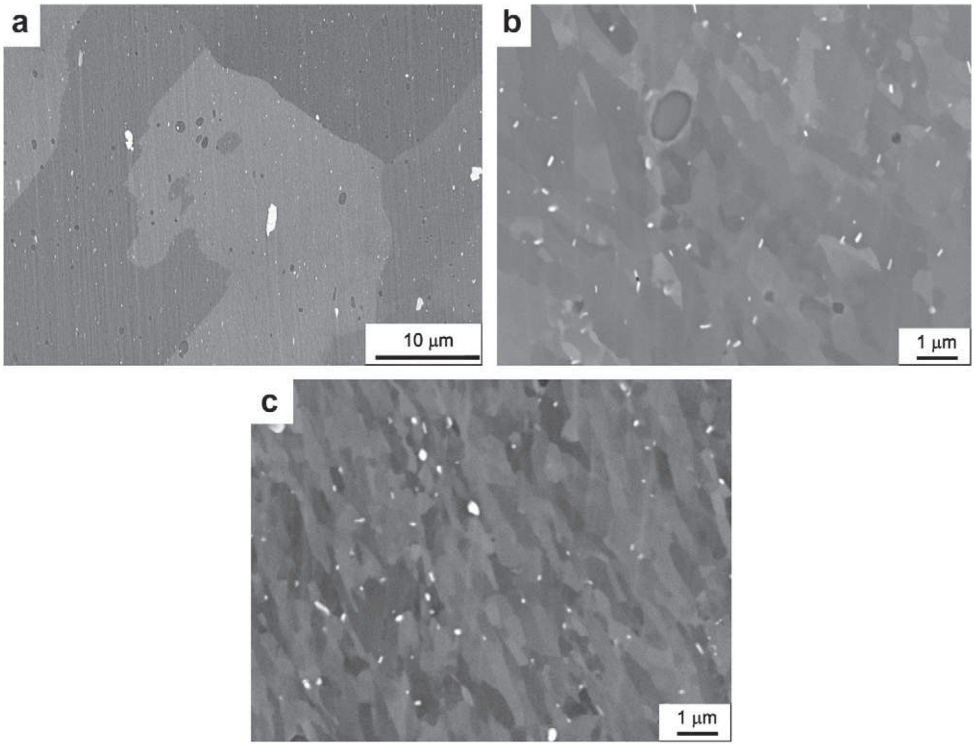

Fig. 4. Microstructures of the Al-6061 alloy: (a) in the as-received unprocessed condition and after processing by ECAP–Conform for (b) one pass and (c) four passes.

the horizontal axes in Fig. 5 correspond to the *Y* direction, the vertical axes correspond to the *Z* direction, the measurements were taken on cross-sectional planes looking along the extrusion axes towards the rear of each rod, and the significance of the colors is shown by the inset at upper right, which denotes color ranges through increments of $Hv = 10$ from 40 to 90. All microhardness values were plotted using the rectilinear coordinate system and then separate colors were superimposed to provide a simple visual representation of the data.

The maps depicted in Fig. 5 provide an excellent overview of the sample homogeneity after processing by ECAP–Conform. After one pass, the maps show there is inhomogeneity on the cross-sectional plane near the front and rear of the rod but the hardness values are more homogeneous in the central region. The presence of inhomogeneity after a single pass is similar to earlier measurements on the Al-6061 alloy showing an inhomogeneous distribution of hardness values after processing through one pass by conventional ECAP [17]. However, whereas billets processed by ECAP show inhomogeneity and lower hardness values adjacent to the bottom surfaces of the billets [17], there are no similar regions of lower hardness along the bottom surfaces at positions B and C in Fig. 5b and c although there is some evidence for lower values of *Hv* along the bottom surface at position A in Fig. 5a near the front of the rod. This suggests that the front of the rod behaves reasonably similarly to billets pressed through conventional ECAP dies whereas over much of the length, and probably as a consequence of the frictional forces acting on the rod during processing, the microhardness values are reasonably homogeneous both over the cross-sectional areas and along almost the total length of the rod.

The situation after four passes is shown in the lower row in Fig. 5 and in this condition the microhardness distributions become more homogeneous both over the cross-sectional planes and with respect to variations along the longitudinal axis. Thus, it is apparent from Fig. 6d–f that the rod processed by four passes exhibits an excellent homogeneous hardness distribution.

An alternative presentation was developed recently in which the hardness values are plotted in a three-dimensional format in order to provide an improved visual image of the variations in hardness across individual planes [29,30]. This form of representation is shown in Fig. 6 for (a) the as-received unprocessed alloy and after processing through (b) one pass and (c) four passes, respectively, where the *Y* and *Z* directions remain as defined in Fig. 5 and the plots show the cross-sectional planes cut near the centers of the rods. It is apparent that the microhardness values are distributed homogeneously over the whole cross-sectional plane before processing with an average value of $Hv \approx 40$, there is a significant increase after a single pass by a factor of ~ 2 and a small but measurable additional increase when the processing is continued to four passes. Although there is some variation in *Hv* across the cross-sectional plane after one pass in Fig. 6b, these variations are

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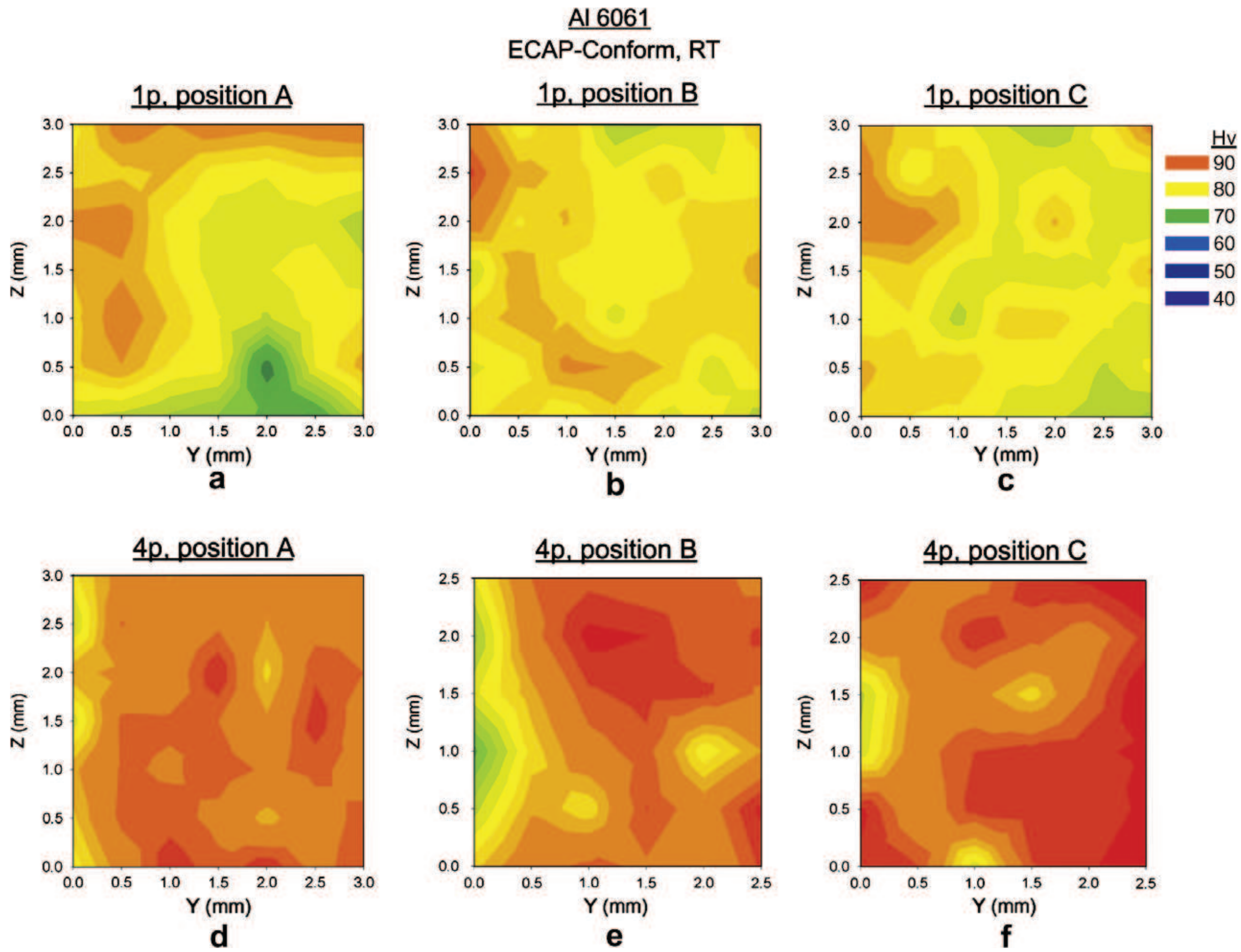

Fig. 5. Color-coded contour maps showing the distribution of the Vickers microhardness over cross-sectional surfaces of the Al-6061 alloy after processing by ECAP–Conform at room temperature for: (a) one pass at position A, (b) one pass at position B, (c) one pass at position C, (d) four passes at position A, (e) four passes at position B and (f) four passes at position C. The positions A–C are shown in Fig. 2 and the significance of the colors is given in the inset at upper right. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

reduced and the hardness distribution becomes more homogeneous after four passes in Fig. 6c.

Considering all of the measurements recorded on the cross-sectional planes at the three different positions of A–C along the longitudinal axes after ECAP–Conform, Table 1 shows the average values and the associated error bars calculated at the 95% confidence level. Also included in Table 1 is the average initial hardness before processing of $Hv \approx 43.7 \pm 0.6$. Inspection of these values shows that the average hardness values increase with increasing numbers of passes and in general there is excellent homogeneity along the lengths of the rods.

3.3. An evaluation of plastic anisotropy using single shear testing

The hardness values attained on the cross-sectional planes reveal excellent homogeneity. Furthermore, earlier experiments conducted on an Al-6061 alloy using conventional ECAP demonstrated a general correlation between the measured values of the yield strength and the microhardness values [31]. Nevertheless, the microstructural observations reveal an elongated grain structure which is different from the equiaxed grains generally attained in conventional ECAP. It is important, therefore, to perform mechanical testing in order to determine whether there is any plastic anisotropy associated with the testing of specimens in directions perpendicular to the extrusion axis. This was accomplished using samples tested to failure in single shear with the two orthogonal orientations shown schematically in Fig. 3 and labeled the Y and Z directions, respectively.

The results are shown in Fig. 7 where the measured ultimate shear strength is plotted against the number of passes in ECAP–Conform using the Y and Z shear directions. These results demonstrate that the shear strengths increase significantly after one pass and then continue to increase but at a slightly lower rate up to four passes. This behavior is very similar to detailed measurements of the ultimate tensile strength in a series of commercial aluminum alloys processed by conventional ECAP for up to eight passes [32]. Fig. 7 reveals a small difference between the Y and

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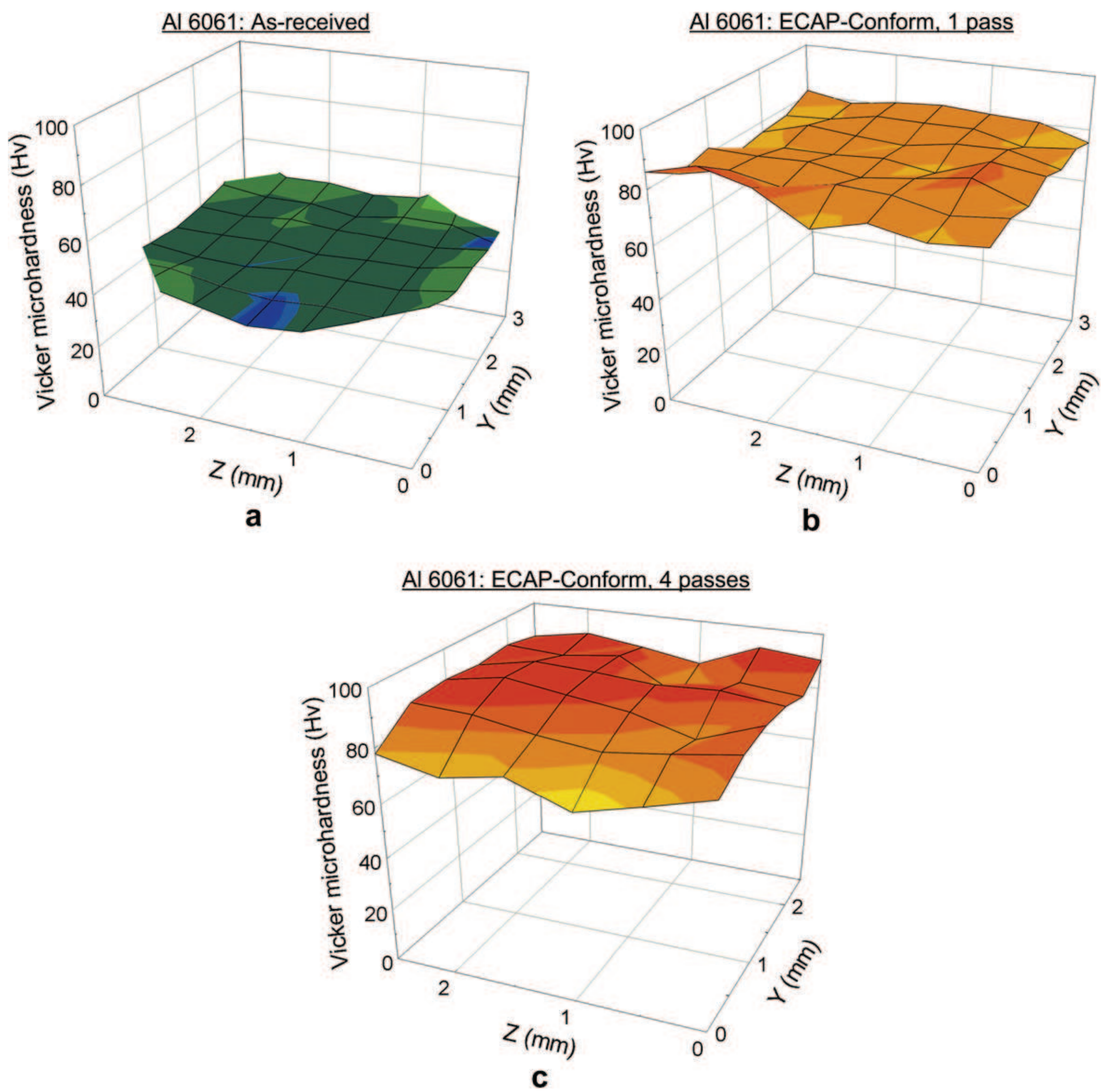

Fig. 6. Three-dimensional representations of the hardness distributions over cross-sectional planes near the centers of the rods for the Al-6061 alloy: (a) in the as-received condition and after processing by ECAP–Conform for (b) one pass and (c) four passes.

Table 1
Average values recorded for the Vickers microhardness, *Hv*.

Number of passes, *N*	Position on rod		
	A (front)	B (middle)	C (back)
0		43.7 ± 0.6	
1	79.3 ± 1.4	80.4 ± 0.9	79.3 ± 0.9
4	86.2 ± 1.0	85.5 ± 1.9	86.6 ± 1.7

Z directions after two and three passes but this difference lies within the experimental scatter. After four passes, when there is excellent hardness homogeneity within the processed rod, the measured shear stresses for the *Y* and *Z* directions are essentially coincident. It should be noted that processing through four passes is sufficient to attain a reasonably equilibrated microstructure when pressing aluminum with an ECAP die having a channel angle of 90° [33]. The present results confirm there is no plastic anisotropy on the cross-sectional planes after ECAP–Conform and this is similar to conventional ECAP where compression testing in three orthogonal directions has shown an absence of any plastic anisotropy in the Al-6061 alloy after processing by ECAP [31].

4. Discussion

There are significant disadvantages in conventional processing by ECAP because of the discontinuous and labor-intensive nature of the procedure and the limitation imposed on the aspect ratio and therefore on the total length of any billet. These problems may be avoided by using the established conform processing method, introduced many years ago for the extrusion-forming of metal wires [13,14], and additionally incorporating an ECAP step to introduce grain refinement. The present investigation demonstrates the success of this approach and confirms

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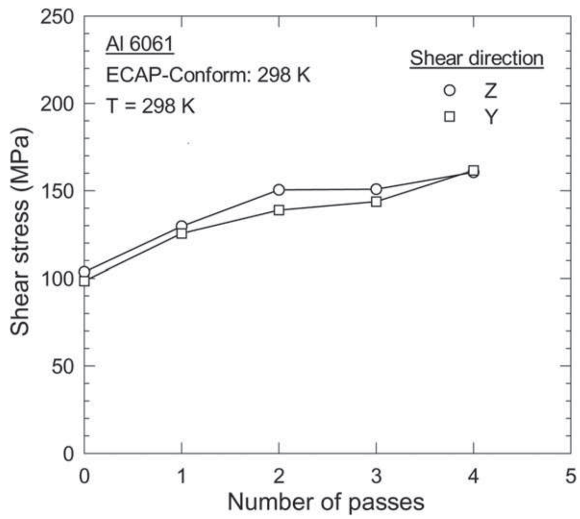

Fig. 7. The variation of the ultimate shear stress at room temperature in two different orthogonal testing directions perpendicular to the extrusion axis vs. the number of passes for the Al-6061 alloy processed by ECAP–Conform.

the potential for both introducing significant grain refinement by ECAP–Conform and increasing the strength of the material.

Table 2 summarizes the results obtained to date for materials processed using a combination of ECAP and Conform. The earlier results on CP Al [15] and CP Ti [16] demonstrate the potential for using ECAP–Conform with long rods, up to >1 m, and for all three materials processed to date by ECAP–Conform the average grain sizes measured on the cross-sectional planes are within the submicrometer range. In the present investigation, the grain size of the Al-6061 alloy was reduced from an initial equiaxed value of ~350 μm to an elongated structure with a width of ~150 nm and length of ~1.2 μm when viewed on the longitudinal plane after four passes. The measured width of ~150 nm is comparable to the equiaxed grain size of ~250 nm reported in an Al-6061 alloy after processing by conventional ECAP through six passes [31]. However, the present microstructures are different from conventional ECAP because of the elongated nature of the grains as shown in Fig. 4b and c. A similar grain elongation on longitudinal sections was reported earlier for the CP Ti [16] and this means in practice that the microstructures produced by ECAP–Conform have similarities to those achieved in accumulative roll-bonding [34,35]. Nevertheless, the lack of any plastic anisotropy when testing in shear perpendicular to the extrusion axis confirms the potential for employing the ECAP–Conform process for the production of materials for use in industrial applications.

The ECAP–Conform process is attractive for the production of wires or relatively thin rods. Furthermore, the lack of any significant wastage is especially attractive by comparison with conventional ECAP. Dental implants of CP Ti with diameters of 2.4 mm are currently in production by ECAP [36,37] and this small size appears to be ideal for production using the ECAP–Conform process. The current process also has an advantage over other procedures such as the proposed down-scaling of ECAP for the production of net-shaped products for micro-electro-mechanical systems (MEMS) [38,39] and the use of ECAP and microextrusion for the production of miniature gears [40].

The present results demonstrate the overall viability of the ECAP–Conform process, they confirm that homogeneity is attained over long sections within the rods processed by this procedure, and this means in practice that any use of this process will entail only minor wastage at either end of the rod. In addition, the results demonstrate the excellent microstructural refinement produced in the Al-6061 alloy by ECAP–Conform and mechanical testing shows an absence of any plastic anisotropy when shear tests are conducted perpendicular to the extrusion axis.

5. Summary and conclusions

1. Rods of an Al-6061 alloy were processed by ECAP–Conform at room temperature for up to a total of four passes. Significant grain refinement was introduced with the grains reduced from an equiaxed configuration of ~350 μm initially to elongated grains having widths of ~150 nm and lengths of ~1.2 μm when viewed on the longitudinal planes after processing through four passes.
2. Measurements of the Vickers microhardness, *Hv*, were recorded over cross-sectional surfaces of selected samples following a regular rectilinear grid pattern and these measurements were used to construct color-coded contour maps to provide a visual representation of the hardness distributions.
3. After a single pass of ECAP–Conform, the hardness distributions were inhomogeneous both on the cross-sectional planes and in the longitudinal direction along the rod. After four passes, the inhomogeneity was significantly reduced and the hardness distributions were reasonably homogeneous.

Table 2
Summary of results using ECAP–Conform.

Material	Initial dimensions		Number of passes, *N*	Grain size		References
	Diameter (mm)	Length (m)		Initial (μm)	Final (on cross-section) (nm)	
CP Al	3.4	>1	1–4	5–7	~650	Raab et al. [15]
CP Ti	8	>1	6	25–30	~200–300	Raab et al. [16]
Al-6061	3.8	0.20–0.25	1–4	~350	~150	This investigation

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4. Measurements of the shear strengths in two orthogonal directions perpendicular to the extrusion axis showed significant strengthening introduced by ECAP–Conform but with no evidence for any plastic anisotropy after processing through four passes.

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