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Characterisation of Conform^{TM} and conventionally extruded Al–4Mg–1Zr. Effect of extrusion route on superplasticity

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Abstract Continuous extrusion (Conform^{TM}) is a well-established technique for the production of profiles from both solid and particulate feed stock. To the first approximation it is considered to be analogous to conventional extrusion, although there are significant differences in the metal flow during both processes. Metal flow during conventional extrusion is characterised by relatively low redundant work, whereas the Conform^{TM} process requires significant redundant work to be successful. Most of the available scientific literature to date is concerned with the simulation of the Conform^{TM} process and not its effect on the resultant microstructure of the product. In this paper, a detailed comparison of the microstructure, texture and superplastic properties developed during Conform^{TM} and conventional extrusion for a particulate Al–4Mg–1Zr alloy are presented.

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Introduction

It has been demonstrated at the pilot scale that an Al–4Mg–1Zr alloy [1] produced via a commercial particulate casting route [2] can produce sheet that is capable of achieving superplastic ductilities of the order of 600%, at a strain rate of 10^{-2} s^{-1}. The alloy yields greater service strength than conventional SPF 5083, while forming pressures are lower. In this development work the particulate feedstock was cold compacted into billets prior to consolidation into bar by hot extrusion. For commercial exploitation of this technology a more economic means of converting the cast particulate starting material into sheet is required. One such possibility would be continuous extrusion utilising the Conform^{TM} process.

Conform^{TM} is a well-established processing route for the continuous production of a wide range of extruded profiles. The method was conceived in the early 1970s at the UKAEA and was first used for the extrusion of wire from continuous rod feed [3]. The most commonly processed metals are various aluminium and copper alloys, although other materials have also been processed successfully. The principle of the process is summarised in Fig. 1a. The feedstock is fed into the profiled groove of the Conform^{TM} wheel by means of a coining roll and the groove is blocked by a close fitting shoe. The material is prevented from continuing its passage along the wheel by means of an abutment. As the feedstock progresses, its temperature rises progressively so that the frictional force generated between the wheel and the feedstock is sufficient to cause the latter to yield and deform plastically over a short length ahead of the abutment. The temperature rises further due to friction and plastic deformation and the compressive stresses build up, finally causing the material to exit through the extrusion die.

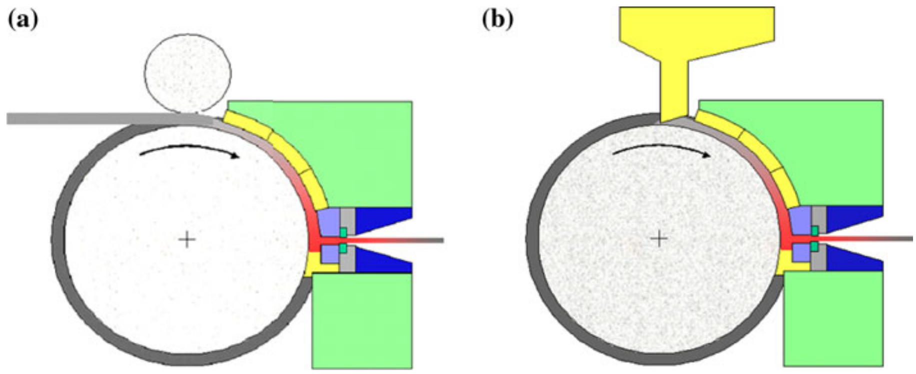


Fig. 1 Schematic of the Conform^{TM} process **a** for continuous rod extrusion and **b** adjusted for particulate feedstock (courtesy of Outokumpu Holton Ltd, UK)

Recently, another group of feedstock materials have generated significant interest. These can be described generically as 'particulates', although the variety is large, ranging from fairly coarse items which can be several centimetres long to extremely fine powders [4]. The Conform^{TM} process can be adapted for particulate feedstock via the use of a vibrating hopper, from which the particulate is fed into the groove of the rotating wheel (Fig. 1b). The flow rate is metered and controlled to provide a constant flow into the groove.

In many respects the process resembles both conventional extrusion and equal channel angular extrusion (ECAE). Compared to conventional extrusion, no external heat source is used. Nevertheless, although the material is fed into the wheel at room temperature, temperatures as high as 350–500 °C are reported [5, 6] especially in the area where new material is mixing with material from the abutment. Therefore, it is probable that the combination of high temperature and redundant work, together with the rather complex turbulent flow in the area of the abutment, will result in significant microstructural changes.

To the authors' knowledge no reports are available on the subject of texture development during the Conform^{TM} process. In this study, the microstructures and textures resulting from Conform^{TM} extrusion of an aluminium alloy in particulate form are compared with those from conventional hot extrusion of the same material. The effect of the resultant microstructure from each extrusion technique on the superplastic properties of the Al–4Mg–1Zr is also discussed.

Experimental

An experimental alloy with a nominal composition of Al–4Mg–1Zr was Conform^{TM} extruded using the particulate feedstock approach shown in Fig. 1b. The needle-shaped particulates (produced via a proprietary casting route [2]) were fed into the groove through a vibrating hopper onto a conveyor and extruded into a fully dense rectangular bar (40 mm × 18 mm cross-section).

For comparison, a 75 mm diameter, cold compacted billet of Al–4Mg–1Zr particulates was conventionally hot extruded using a 5 MN ENEFCO hydraulic press at 525 °C, with an extrusion ratio of ~10:1 to produce also a rectangular bar with a 40 mm × 18 mm cross-section.

The microstructure and the corresponding texture from different areas were characterised with electron backscattered diffraction (EBSD) analysis in a Field Emission Scanning Electron Microscope (LEO Gemini 1525). In the EBSD maps, white lines represent grain boundaries and grey lines subgrain boundaries.

After extrusion, both materials received a typical heat treatment for zirconium containing aluminium alloys (100 h at 360 °C) to allow precipitation of zirconium from supersaturated solid solution and were subsequently cold rolled to a thickness of 1.5 mm, representing a reduction of 91.6%. Tensile test pieces (with a gauge length of 12.7 mm) were taken from both the extruded bar and rolled sheet. To assess the superplastic performance for each processing condition, hot uniaxial tensile tests were carried out over a temperature range of 300–550 °C. The optimum ductilities reported in this paper correspond to the maximum value obtained from this procedure for each processing condition. The samples were tested at a constant cross-head velocity equivalent to an initial strain rate of 10^{-2} s^{-1}.

Results and discussion

Figure 2 shows cross-sections (surface normal to the extrusion direction) from the two processes after chemical macroetching. Examination of the surfaces indicates that the structure in the conventional extrusion (Fig. 2a) is relatively homogeneous and fully consolidated. For the Conform^{TM} extrusion (Fig. 2b) macroetching revealed concentric rings around the centre with an ovular shape. The rings were observed to be of a higher density towards the periphery of the extrusion. This feature, otherwise known as "onion skin" is a recognised phenomenon of

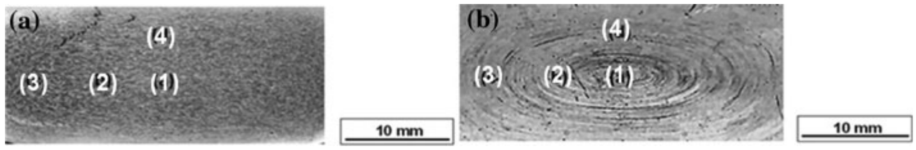


Fig. 2 Macroetch of cross-sections taken from material **a** conventionally extruded and **b** extruded via the Conform^{TM} process, showing locations for further analysis

continuous extrusion and according to the developers of the process is not believed to be detrimental to the subsequent service properties of the material.

A detailed microstructural and textural analysis was conducted on the surface normal to the extrusion direction for both products. In particular, four different regions (labelled 1–4 in Fig. 2) were examined at varying distances from the central extrusion axis in the short and long transverse directions. For each extrusion process and location, EBSD analysis was performed and the corresponding texture data were calculated (for areas 1, 3 and 4 only). Given that the feedstock material was in the form of cast particulates, it is assumed that they possessed random orientation and any texture measured after extrusion will be solely the result of the process.

Conventional extrusion

At the centre of the extrusion axis (Fig. 3a) coarse flattened grains (~20 μm) coexisted with submicron (possibly dynamically recrystallised) ones, resulting in an average grain size of 1.1 μm (as provided by the EBSD software). There is evidence that some of the original grains in the as-cast particulates survive during hot extrusion and are present in the as-extruded microstructure [7] with sizes similar to the coarse grains observed in area 1. At an intermediate distance between the centre of the extrusion and the long transverse surface the grain variation was less (Fig. 3b) and the average grain size was 1.6 μm. In the area close to the long transverse surface (Fig. 3c) a higher fraction of fine grains existed resulting in further grain refinement (0.9 μm). Finally, at the short transverse surface the grain size was 1.2 μm (Fig. 3d). In terms of texture, at the centre of the extrusion (Fig. 4a) there is a weak β-fibre component with the majority of the crystallites aligned along ⟨111⟩ and to a lesser extent to ⟨100⟩. Moving away from the centre, towards the long and the short transverse positions (Fig. 4b, c, respectively), a stronger fibre texture develops. This is also shown both in the Orientation Distribution Factors (ODF) (Fig. 5) and the representation of the volume of crystallographic components (Fig. 6). It should be mentioned here that the pole figures measured in positions 3 and 4 are similar to those observed by other researchers with extruded Al–5Mg–1.2Cr powder (when the Euler angle is rotated 90°) [8] and Al–Mg–Si–Cu rods [9]. Generally, the Goss {011}⟨100⟩ and Cube {001}⟨100⟩ components are considered to represent recrystallised grains (oriented nucleation will also increase Cube [10, 11]),

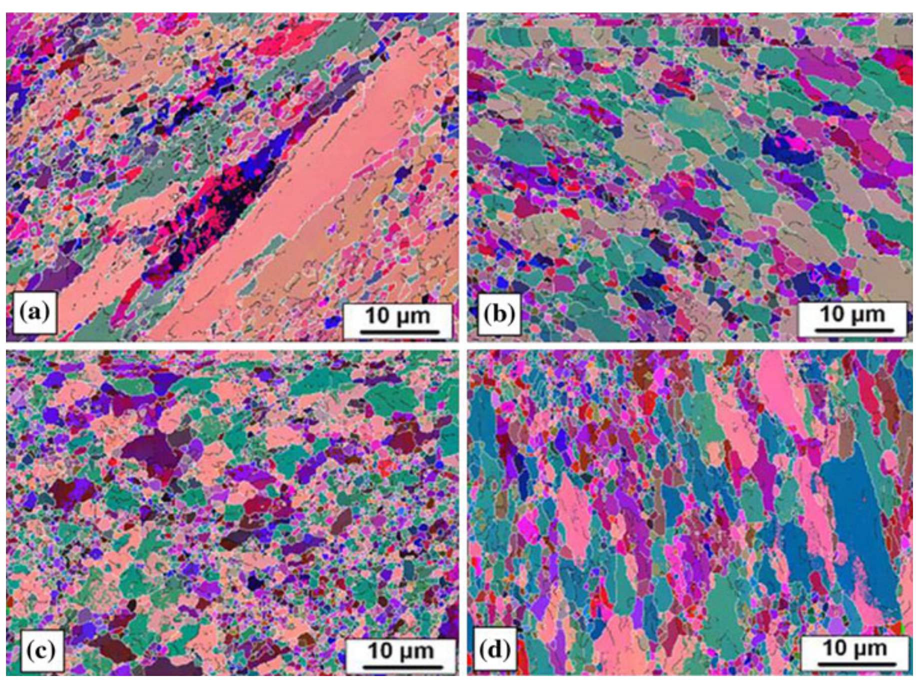


Fig. 3 EBSD micrographs (*white lines* grain boundaries, *dark lines* subgrain boundaries) of areas (*1*)–(*4*) of the rectangular, conventionally extruded, Al–4Mg–1Zr (Fig. 2a)

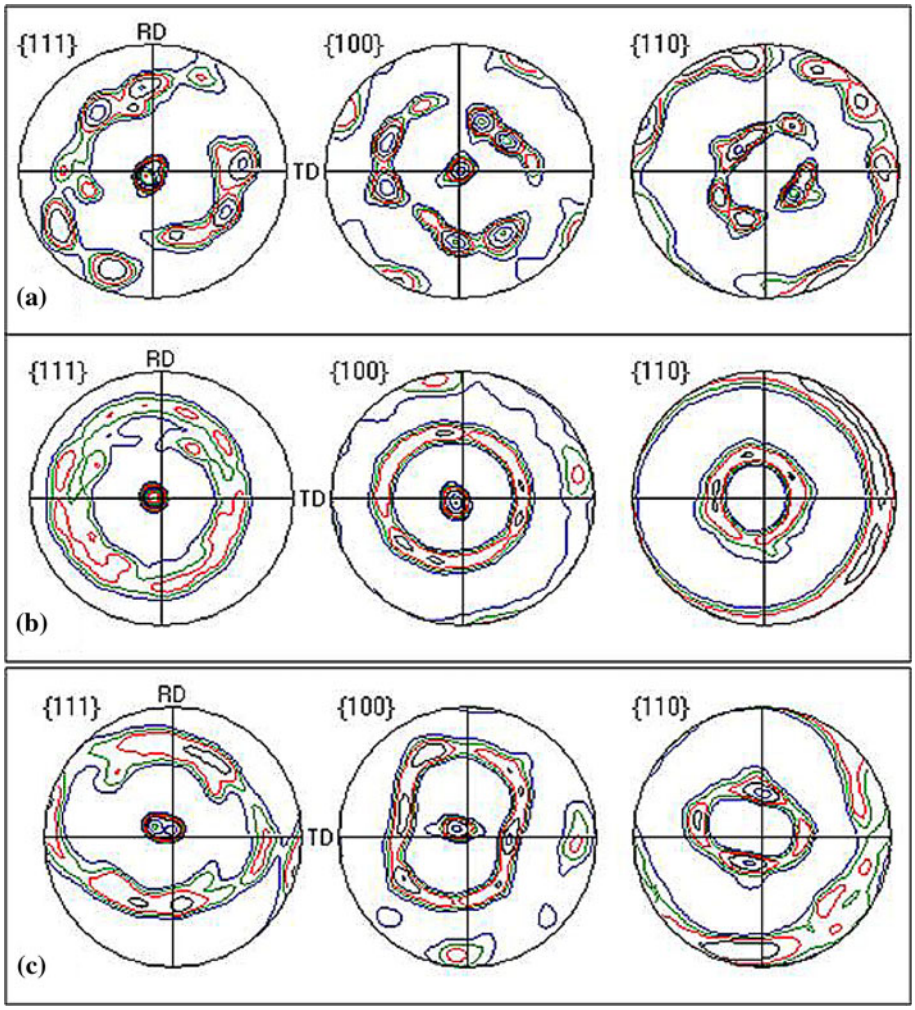


Fig. 4 {111}, {110}, {100} pole figures (contour levels: 1, 1.5, 2, 3, 5, 7, 10) of areas *1*, *3* and *4* shown in Fig. 2a

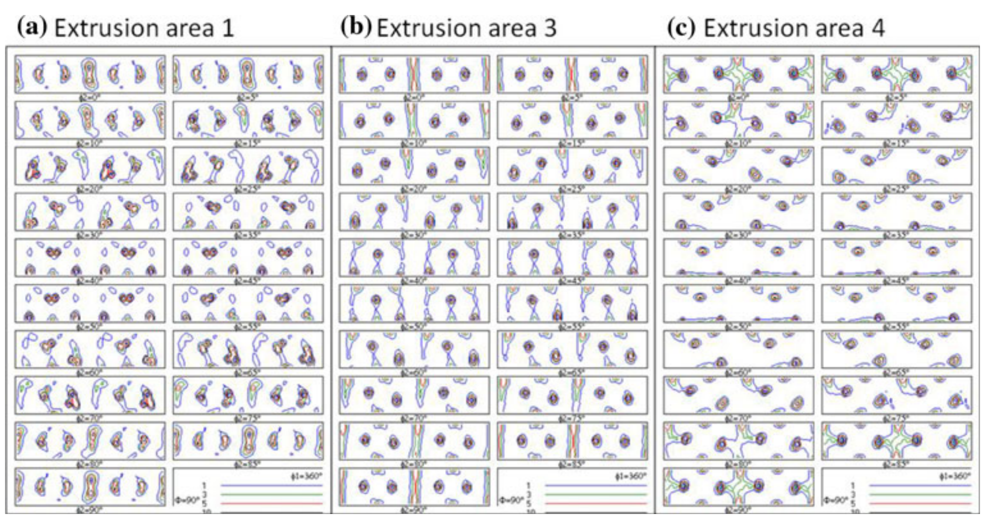


Fig. 5 Orientation Distribution Factors (ODF) of areas *1*, *3* and *4* shown in Fig. 2a

whereas the Brass {011}⟨211⟩, P {011}⟨111⟩, Copper {112}⟨111⟩ and S {123}⟨634⟩ deformed grains (e.g. shear). These observations also agree with the grain refinement observed closer to the surface (Fig. 3), implying that the degree of recrystallisation, most probably dynamic due to further breaking to smaller grains when compared to the centre, is higher there. In Fig. 7 the percentage of high angle grain boundaries (HAGB) versus low angle grain boundaries

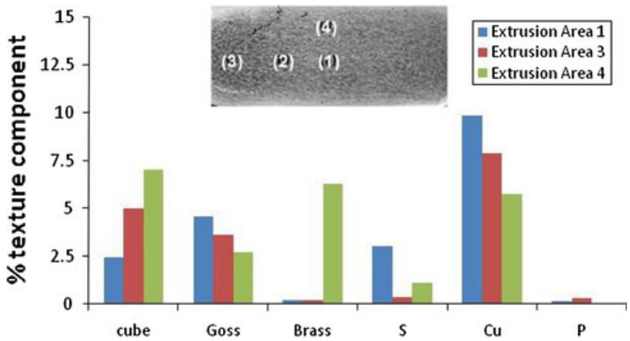


Fig. 6 % Texture crystallographic components for areas *1*, *3* and *4* shown in Fig. 2a

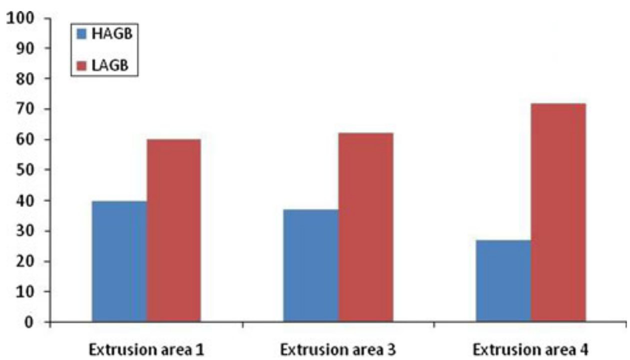


Fig. 7 % of HAGB and LAGB for areas *1*, *3* and *4* shown in Fig. 2a

(LAGB) is presented. Usually, a higher percentage of LAGB indicates a wrought structure, while a higher percentage of HAGB indicates a more recrystallised structure as the subgrains are consumed by the new recrystallised ones. Despite the observed refinement described above, a relatively high volume of substructure (LAGB) is present in all positions.

Conform^{TM} extrusion

At the central axis of the Conform^{TM} extruded metal (Fig. 8a) recrystallised grains with an average size of 1.9 ± 1.7 μm were present. Further away from the centre, the microstructure consisted of a mixture of coarse and finer grains with an average size of 1.6 μm (Fig. 8b). Finally, close to the surface (both long and short transverse) the structure was, as with the conventional extrusion, refined compared to the centre, with an average grain size of 1.2 ± 0.7 μm (Fig. 8c) and 1.1 ± 1.0 μm (Fig. 8d). In Fig. 8 apart from some directionality, interchanging between bands of coarse and finer grains is observed, which is probably the origin of the 'onion skin' phenomenon described earlier.

In terms of texture, at the centre of the extrusion (Fig. 9a and 10a) grain orientation is virtually random as opposed to the weak fibre observed in the case of conventional extrusion. This may be explained by the fact that in conventional extrusion, grains at the centre deform uniaxially and at much lower strains, whereas in Conform^{TM} grains travel/deform uniaxially around the wheel radius but are forced to change direction by 90° to exit the die or even travel to the bottom of the abutment and return before exiting the die. For similar but no so complex movement in ECAE, it has been reported that neighbouring grains will

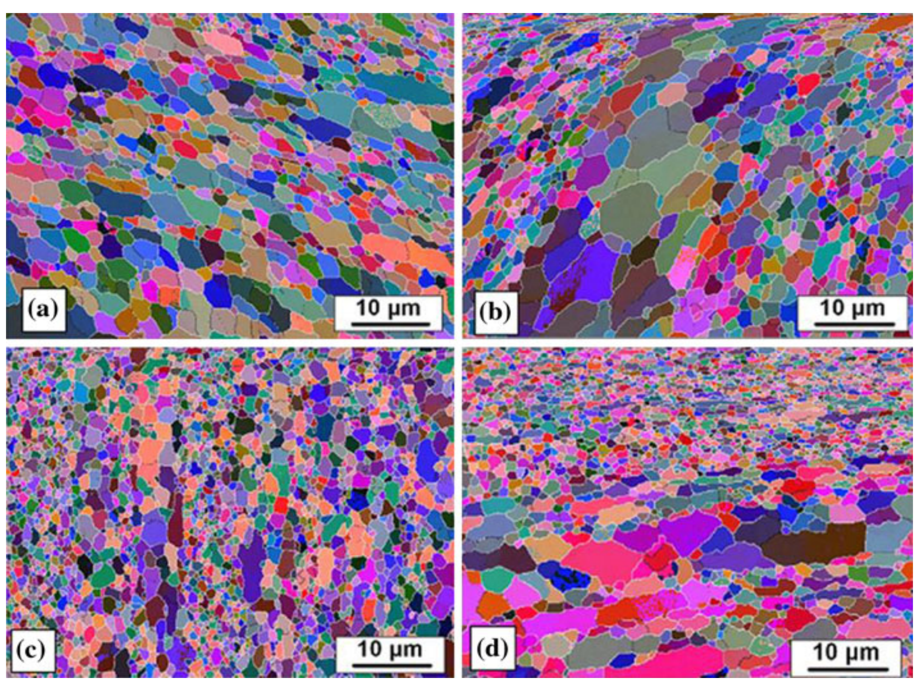


Fig. 8 EBSD micrographs (*white lines* grain boundaries, *dark lines* subgrain boundaries) of areas (*1*)–(*4*) of the rectangular, Conform^{TM} extruded, Al–4Mg–1Zr (Fig. 2b)

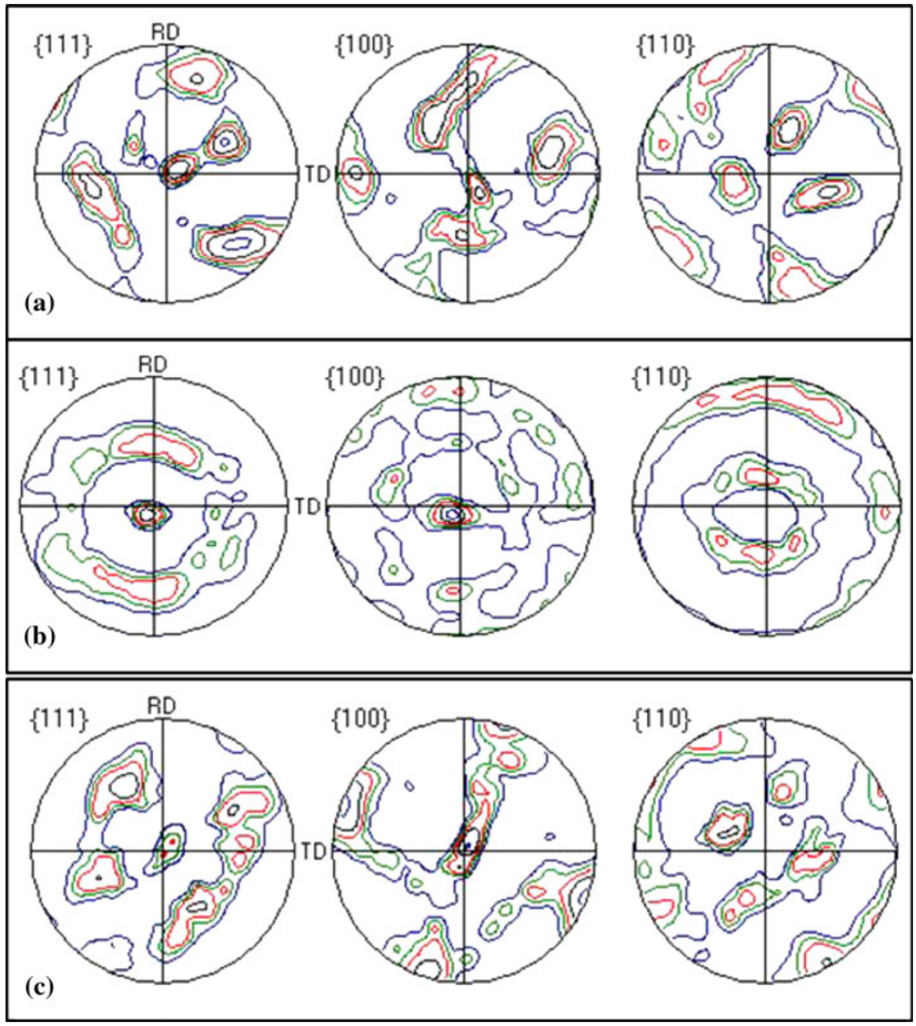


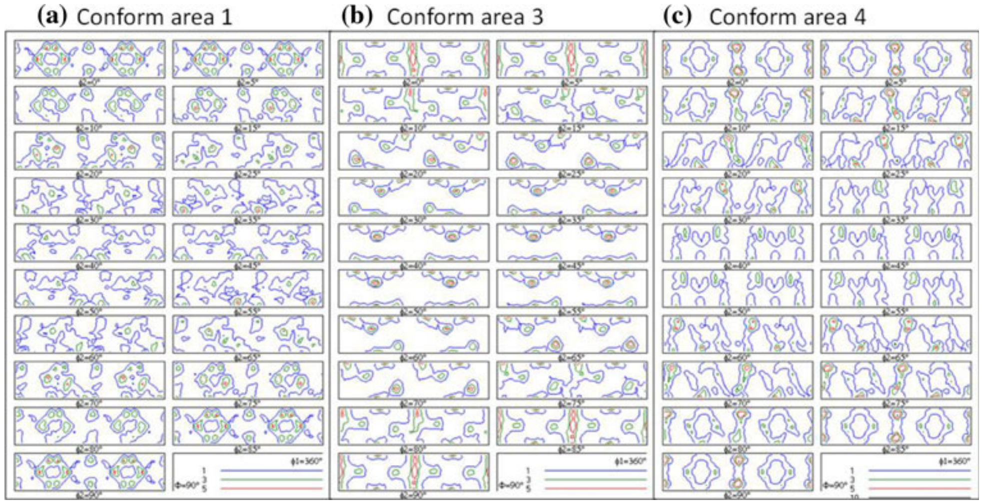
Fig. 9 {111), {110}, {100} pole figures (contour levels: 1, 1.5, 2, 3, 5, 7, 10) of areas *1*, *3* and *4* shown in Fig. 2b

develop different orientations during the 90° direction change, resulting in random texture [12]. Finite element analysis [6, 13, 14] provides insight into the metal flow and thermomechanical conditions associated with the Conform^{TM} process and can support further claims that plastic deformation at the abutment can result in a random texture. It is accepted that the workpiece moves along the circumferential path and that frictional heating by the time it reaches the abutment enables plastic flow of the material through the die. The exit temperature is generally comparable to that experienced in conventional extrusion. Unlike conventional extrusion, the metal flow in the region of the abutment and die is very complex and asymmetric. While the apparent extrusion ratios (i.e. the ratio between the cross-sectional areas of container and die) are low and can indeed be less than one, the complexity of the metal flow leads to high redundant work. Furthermore, the reported strain rates associated with the process are at least one order of magnitude higher than those reported for conventional extrusion of the same geometry. Nevertheless, the high strains involved in the short distance between the abutment and the die exit appear to be sufficient for a weak fibre texture, similar to conventional extrusion, to start developing close to the surface (Figs. 9b, c; 10b, c). However, no sharp components are present as shown in the summary of crystallographic components in Fig. 11. In agreement with the references on finite element analysis modelling, a comparison of the grains (HAGB)/subgrains (LAGB) ratio between Conform^{TM} and conventional extrusion indicates that the degree of recrystallisation is higher in the former process (Fig. 12).

Superplastic performance

Figure 13 shows the effect of post extrusion processing on the hot ductility of Al–4Mg–1Zr. In the as-extruded condition the superplastic performance of the conventional and Conform^{TM} product are of a high and comparable level. However, unlike the conventionally extruded material, the Conform^{TM} product loses nearly all its high temperature ductility when cold rolled. Similar findings are reported by other researchers who compared ECAE of aluminium

Fig. 10 Orientation Distribution Factors (ODF) of areas *1*, *3* and *4* shown in Fig. 2b



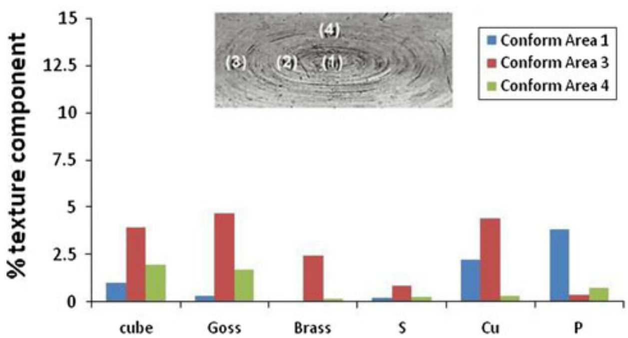


Fig. 11 % Texture crystallographic components for areas *1*, *3* and *4* shown in Fig. 2b

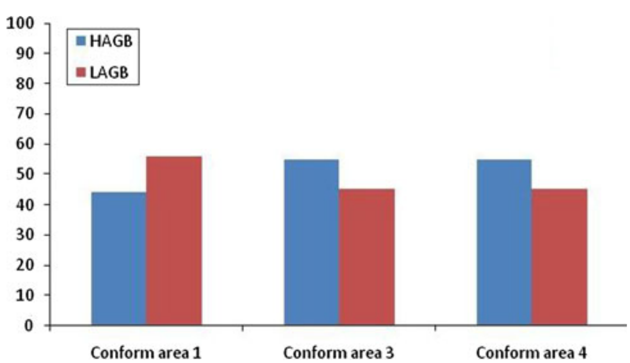


Fig. 12 % of HAGB and LAGB for areas *1*, *3* and *4* shown in Fig. 2b

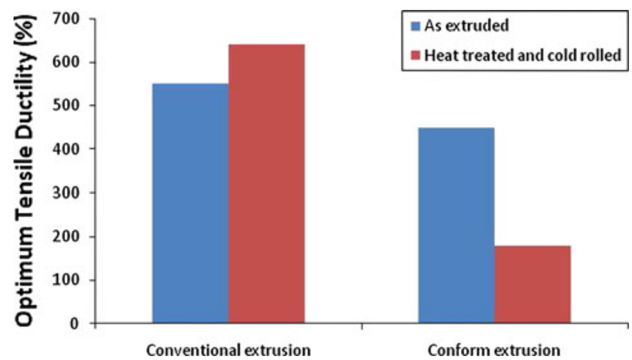


Fig. 13 Effect of post-processing on the hot tensile ductility of Al–4Mg–1Zr extruded conventionally and via the Conform^{TM} process

powder vs conventional extrusion and observed that the initial fine microstructure was very unstable during high temperature processing [15].

Key to the success of the Al–4Mg–1Zr alloy is the Zener pinning ability of the fine dispersion of metastable Al_3Zr precipitates to resist grain growth during superplastic forming. Work conducted on the same alloy using a combination of severe plastic deformation techniques such as friction stir welding in conjunction with cold rolling [16] and multiple extrusion [17] has shown similar behaviour. In these studies it was concluded that combinations of severe plastic deformation processes (i) coarsened the Al_3Zr particles present in the alloy leading to reduction of their pinning action and (ii) significantly increased the stored energy. The combination of those two phenomena rendered the alloy less temperature resistant. Therefore, it can be concluded that Conform^{TM} extrusion has a similar effect on the microstructure of the Al–4Mg–1Zr alloy as other severe plastic deformation processes.

Conclusions

A comparison between Conform^{TM} and conventionally extruded Al–4Mg–1Zr was conducted. In the case of conventional extrusion the structure varied significantly from the centre to the surface of the extrusion. The centre consisted of a bimodal distribution of coarse deformed grains and fine equiaxed ones. Moving away from the centre to the surface, this transformed to a standard distribution of fine equiaxed grains.

In the Conform™ extruded condition, despite what appeared to be an inhomogeneous macrostructure, a near to uniform distribution of fine grains was observed throughout the cross-section. It is proposed that the complexity of the metal flow and high redundant work associated with Conform™ extrusion leads to a recrystallisation occurring in the abutment region prior to extrusion. The central region is then extruded without further recrystallisation events taking place, whilst in the surface regions where the strain and strain rate are higher secondary recrystallisation events take place. This leads to further grain refinement near to the surface of the extrusion.

Although the billet from which the conventional extrusion was produced was in the form of cold compacted particulate, a reasonably uniform β-fibre texture was measured throughout the section (weaker at the centre, stronger at the surface). In this respect it differed very little from that expected from extrusion of a monolithic billet.

In the Conform™ extruded material a random texture was observed in the central region and a weak fibre towards the surface together with a high degree of high angle grain boundaries (recrystallised grains) indicating a much stronger influence of recrystallisation in this process. The stronger tendency towards recrystallisation in the Conform™ extrusion is believed to be a direct result of the higher redundant work.

The Conform™ extrusion demonstrated that a fully consolidated product could be produced from a particulate feedstock with a uniform refined grain structure that could exhibit good superplastic properties. However, the high level of redundant work resulted in a product whose microstructure and properties were sensitive to subsequent processing.

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