Abstract
The microstructure evolution under elevated plastic deformations was investigated by means of transmission electron microscopy (TEM) in different aluminium alloys processed by the equal channel angular pressing (ECAP) technique in route Bc to strain of 8. The alloys was characterized by submicron-scale grains. The AA1200 high-angle boundaries (misorientation higher than 15°) accounted for ~70% of all boundaries. In the AA5754 measurements indicated average grain sizes of about 0.3–0.4 µm in the as-pressed condition, thereby demonstrating that ECAP is an especially effective procedure for attaining an ultrafine grain size. It appeared that many of the grain boundaries were ill-defined and it is reasonable to assume that, as in an earlier report on ECAP of commercial Al alloys, these boundaries are in high-energy non-equilibrium configurations. For the AA6082 different kinds of alloys have been investigated, the first alloy (commercial AA6082) has a microstructure evolution depending on the presence of Mg2Si and Si particles, the others alloys (with Zr and Sc+Zr) have a microstructure stabilized by the dispersoids.

Riassunto
L’evoluzione microstrutturale di alcune leghe che hanno subito severe deformazioni plastiche ad opera del processo ECAP è stata investigata attraverso indagini al microscopio a scansione elettronica (TEM). La deformazione impartita è arrivata ad un livello pari ad μ=8 seguendo la route Bc. Le leghe sono caratterizzate da una dimensione dei grani di scala sub-micrometrica. Nella lega AA1200 i bordi ad alto angolo arrivano al 70% dei bordi visualizzati, nella AA5754 la dimensione media dei grani dopo la deformazione arriva a 0.3-0.4 µm, questo e altri risultati che vedremo in seguito dimostrano come l’ECAP sia un processo effettivamente efficace per l’affinamento microstrutturale con la possibilità di ottenere materiali con attitudini superplastiche. Molti dei bordi di grano sembrano mal definiti ed è ragionevole supporre che, come riscontrabile nella bibliografia riguardante l’ECAP di leghe commerciali di Al, questi bordi sono in configurazioni ad alta energia di non-equilibrio. Per la lega AA6082 oltre a quella commerciale sono state studiate, delle leghe modificate con l’aggiunta di alliganti particolari. La prima lega (AA6082 commerciale) ha uno sviluppo della microstruttura determinato dalla presenza delle particelle del silicio e di Mg2Si, nelle altre in cui vediamo aggiunti alla lega Zr e Sc+Zr la microstruttura è determinata e stabilizzata dalla presenza di dispersoidi.
INTRODUCTION

Different techniques for producing ultrafine-grained (UFG) materials for structural applications have been introduced and patented, especially in the last decade [1]. The advantages of fabricating materials with sub-micron size grained microstructure as structural components lie in their improved mechanical properties such as strength, hardness, ductility, fatigue resistance and low-temperature superplasticity [1-10]. Equal channel angular pressing (ECAP), introduced and developed by Segal et al. and Segal [1,3], is a promising technique that uses severe plastic deformation (SPD) to refine microstructure. ECAP has the important advantage to maintain billet shape. A typical ECAP die, Fig. 1, consists of two intersecting channels of identical cross-section. A billet of material is introduced in the vertical channel and forced by a plunger into the horizontal one. Shear strain per pass through the die is determined by the angles of channel intersection and curvature [6,11,12]. Many processing parameters have dramatic effects on the resulted microstructure [12]: die angles (determining the strain introduced into the material), the number of passes (accumulation of strain), deformation route (critical parameter for texture and microstructure evolution with strain), and also the extrusion speed, temperature, friction. Langdon and co-workers [6,10] found the angles $\Phi = 90^\circ$ and $\Psi = 20^\circ$, Fig. 1 and eq.1, to be the most efficient, while the extrusion speed and specimen-die channel friction have minor influence on the refining process. A number of theories have been proposed to explain the effect of processing routes on the microstructure. Iwahashi et al. [11] and Furukawa et al. [12,13] proposed that route Bc ($90^\circ$ rotation of the billet at each pass) is most favorable for producing a microstructure consisting of essentially uniform and equiaxed grains separated by high-angle boundaries (HABs). This was suggested to be due to crossing shear planes, and to a regular restoration into equiaxed structure during consecutive pressing. Sun et al. [14,15] studied the different routes: A, Bc, Bc, and C, where the route A refers to repetitively pressing the sample without any rotation, the route Bc refers to a rotation of $90^\circ$ back and forth between each pass, route Bc refers to a rotation of $90^\circ$ between each pass and route C refers to a rotation by $180^\circ$ between each pass as a function of different microstructure parameters. They found that the effectiveness in terms of formation of HABs was $A>Bc>C$, in terms of reducing grain size was $Bc>A>C$ and in terms of generating equiaxed grains was $Bc>C>A$. Studies on cell and grain evolution have been performed only in recent years [4,16-21,23,24]. Several investigations [12-15,22-32] have shown that, during deformation [23,24], grains subdivide into many small crystallites, each having a crystal rotated orientation. Thus, during straining, dislocations generally arrange into a mosaic-like pattern. The mosaic-like configuration is basically composed of boundaries surrounding regions with relatively low dislocation density. Studies on metals established that grains were fragmented into domains of different slip systems, called cell blocks, during deformation [24,27]. Concerning the Zr and Sc+Zr dispersoids, several studies have shown that aluminium alloys, alloyed with scandium, or zirconium, have excellent mechanical properties at room temperature, due to the presence of coherent, nanometre size $\text{Al}_3\text{Sc(Zr)}$ precipitates effectively dispersed either.

Fig. 1: Open solid ECAP die used in this study. The intersection and curvature angle values are $\Phi = 90^\circ$ and $\Psi = 20^\circ$ respectively (see eq. 1).
in the grain boundaries and within grains, thus blocking mobile dislocations and stabilizing a fine-grained structure [33,34]. These \( \text{Al}_3\text{Sc}(\text{Zr}) \) precipitates are stable up to the typical recrystallisation temperatures for aluminium alloys [35]. Combined additions of Sc and Zr were shown to be more effective in refining as-cast material microstructures; in particular, Zr is able to replace some of the Sc in \( \text{Al}_3\text{Sc} \), giving rise to \( \text{Al}_3(\text{Sc}_{1-x}\cdot\text{Zr}_x) \) dispersoids having an L12 crystal structure like the \( \text{Al}_3\text{Sc} \) [33-35].

**EXPERIMENTAL DETAILS**

The chemical composition of the tested alloys is reported in Table 1. The alloys were cast into a rod-shaped bars 10 mm in diameter. Before ECAP processing, the AA 6000 alloys were full annealed and a T8 treatment was carried out in the Alloy 1 and Alloy 2. The die was placed in a dedicated pressing machine with a maximum load of 80-100 kN and a pressing speed of 4 mm min\(^{-1}\), operating at room temperature. The die was constituted of two blocks of SK3 tool steel (Fe-1.1%C), which were bolted together to give a single internal L-channel 10 mm in diameter (Fig. 1). The two cylindrical channels, intersected at an angle \( \Phi = 90^\circ \) and a curvature \( \Psi = 20^\circ \). Samples and channels were coated with a spray lubricant containing MoS\(_2\).

Based on Eq. 1, this configuration allowed to introduce a true strain of \( \varepsilon = 1.055 \) at each pass (N) [3,18,37].

\[
\varepsilon = \frac{N}{\sqrt{3}} \left[ 2 \cot \left( \frac{\Phi}{2} + \frac{\Psi}{2} \right) + \Psi \cos \varepsilon \left( \frac{\Phi}{2} + \frac{\Psi}{2} \right) \right]
\]

Table 1 - Chemical composition (wt%).

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Mg</th>
<th>Mn</th>
<th>Cr</th>
<th>Ti</th>
<th>Cu</th>
<th>Si</th>
<th>Fe</th>
<th>Zn</th>
<th>Zr</th>
<th>Sc</th>
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<tr>
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<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
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<td>0.05</td>
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<tr>
<td>AA5754</td>
<td>2.5</td>
<td>0.3</td>
<td>0.4</td>
<td>---</td>
<td>0.1</td>
<td>0.4</td>
<td>0.4</td>
<td>0.2</td>
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<td>AA6082</td>
<td>1.193</td>
<td>0.650</td>
<td>0.010</td>
<td>0.015</td>
<td>0.005</td>
<td>1.019</td>
<td>0.267</td>
<td>---</td>
<td>---</td>
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<td>---</td>
</tr>
<tr>
<td>Alloy 1</td>
<td>0.34</td>
<td>0.014</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>0.51</td>
<td>0.16</td>
<td>---</td>
<td>0.10</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>Alloy 2</td>
<td>0.34</td>
<td>0.014</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>0.51</td>
<td>0.16</td>
<td>---</td>
<td>0.10</td>
<td>0.117</td>
<td>---</td>
</tr>
</tbody>
</table>

Three orthogonal planes (X,Y) were used to define the orientations of the billet deformed by ECAP, where X is perpendicular to the pressing direction, Y is the flow plane, i.e., the one containing the pressing and the transverse direction [10-13,19].

TEM samples were sectioned along the Y plane. Thin foils were prepared by mechanical grinding 1-mm thick slices down to a thickness of 70-90 \( \mu \)m, followed by chemical polishing (1/3 HNO\(_3\) in methanol) in order to minimize the grinding damage. Disks 3 mm in diameter were subsequently thinned with a double-jet electro-polisher using a solution of 20% HClO\(_4\) and 80% methyl alcohol at \(-15^\circ\)C and 24 V and examined in a Philips CM200 TEM operating at 200 kV, equipped with a double-tilt stage.

Microstructure examination was focused on the cell, grain size distribution and boundary misorientation. Concerning the boundary misorientation, three independent Kikuchi patterns were taken into account, typically either incidental dislocation boundaries (IDBs) and geometrically necessary boundaries (GNBs). As the strain increases the fraction of boundaries that are low-angle boundaries (LABs) decreases steadily. The misorientation angle is defined as minimum rotation angle that can cause the two crystals to coincide each other by selecting an arbitrary rotation axis. Moiré fringes were evaluated, instead of Kikuchi analysis. The misorientation measurement was carried out by counting the number of dark-bright pairs directly on TEM plates and, subsequently, using Eq. (2a) [36,38].

\[
D = \frac{d_1 d_2}{d_1^2 + d_2^2 - 2 d_1 d_2 \cos \phi}
\]

where \( d_1 \) and \( d_2 \) are the two superimposed crystal lattice spacing, \( \phi \) is the rotational angle and \( D \) is the overall width of the resulting Moiré fringes. In the case of simple matrix rotation, Moiré fringes are characterized by an angle \( \phi \) and \( d_1 = d_2 \), thus Eq. (2a) reduces, for small angles, to Eq. (2b):

\[
D = \frac{d}{\phi}
\]
RESULTS AND DISCUSSION

Fig. 2 (a) to (d) shows some representative micrographs of the AA1200 illustrating the boundary spacing evolution with strain of either LABs and HABs. Boundary misorientation angles are also reported. Fig. 3 shows the comparison of HAB fraction among the three different routes to which the 1200 alloy has been subjected: route A, C and Bc; the fraction of HABs after 8 passes are 81, 62, 58 %, respectively.

Initially at low strains very coarse deformation bands are formed. As the strain increases block walls develop within the coarse bands. The majority of cells, formed after the first and second passes, were nearly free of dislocations in the interior. After one ECAP pass, cells within the bands are elongated as well as the shearing bands along the shear direction. Mean linear intercept measurements showed that the block walls are 2.68, 1.26, 1.75 mm, in length, and 880, 640, 815 nm, wide, for the sample A-2, C-2 and Bc-2, respectively. In sample A-2 and Bc-2, the dominant features are the parallel bands of elongated block walls inclined at an angle of 45–50° with the pressing direction, depending on the specific strain path, equiaxed cells can also be found in some areas, in sample C-2. For sample Bc-2, the

![Fig. 2: TEM-BF of the AA1200 showing the deformation process of different straining levels, after 1 (a), 4 (b), 5 (c), 8 passes (d). Some of the boundary misorientation values are also reported. The extrusion direction (ED), i.e. ECA-pressing direction, is indicated.](image-url)
microstructure consisted of parallel bands of elongated block walls as well as large number of tangled dislocations, although the banded structure is less evident. Fig. 4 is a plot of the LABs and HABs misorientatoin as a function of strain. The average misorientation angles of LABs and HABs are very close, i.e., 4.2° for LABs and 34° for HABs, among the three strain paths. In terms of the production of HABs, route A is more effective than route Bc, which, in turns, is far more effective than the route C. In route C, the shear plane does not change during repeated pressing and the strain of the first pass can be reversed in the following pass. This route contributes to a redundant strain process and less HABs are generated. Then, the effectiveness in creating HABs is essentially in the sequence: A > Bc > C.

Fig. 5 (a) to (c) shows the misorientation distribution as a function of strain for routes A, C, Bc, respectively. The boundary misorientation distributions have been divided into 15° ranges: Low Angle Boundaries (LABs), misorientation less than 15°, Low High Angle Boundaries (LHABs), from 15–30°, Medium High Angle Boundaries (MHABs), from 30–45°, and Very High Angle Boundaries (VHABs), with misorientation beyond 45°. Boundary misorientation angles shows nearly constant population evolution toward high mean values, from low to high angles, in the three strain paths. Yet, a bimodal distribution, with a large fraction of low-angle boundary, is found for A and C samples, and an almost constant distribution, from LAB to VHAB, for route Bc (Fig. 5 (a) to (c)). TEM inspections indicate that the billets deformed to large strains by ECAP (8 passes) still have a significant fraction of low-angle boundaries as compared with the boundary misorientation of grains with random Mackenzie distribution. This is attributed to the continuous generation of low-angle boundaries during deformation. This process is more evident in route C and, to some extent, in route Bc, but less pronounced in route A.

On average, the misorientation between adjacent block walls increases with strain and some of them may eventually evolve into new high angle boundaries. LABs retained at very high strains, are typically transverse to the HABs. The HABs often appears as lamellar structures prevalently aligned ~45° to the pressing direction. As the strain increases (up to the maximum strain of e = 8), new HABs progressively evolve at the block walls within the primary deformation bands. These block walls account for the build up of the LHAB fraction, and, at the same time, some of the MHABs may march upwards to VHABs. The strain direction of
Fig. 5: AA1200: cumulative misorientation distribution of LABs, LHABs, MHABs and VHABs, corresponding to misorientation ranges of (0.1°-15°), (15.1°-30°), (30.1°-45°) and above 45.1°, respectively; route A (a), route C (b), route Bc (c).
route Bc changes every pass therefore contributes intersections, which develop a near spatially uniform network in the material, and consequently leads to a higher efficiency in HAB generation. Microstructural examinations of billets pressed through 2 to 6 passes, of the alloy AA5754 revealed an array of reasonably equiaxed grains having average sizes of <1 µm (see also [39]). Selected area electron diffraction patterns after 6 passes showed diffuse rings and evidence for the presence of an array of grains with high disoriented angles. Measurements indicated average grain sizes of about 0.3–0.4 µm in the as-pressed samples. It appeared that many of the grain boundaries were ill-defined and it is reasonable to assume that, as in an earlier report on ECAP of AA1200 [38], these boundaries are in high-energy, non-equilibrium configurations. The effect of severe plastic deformation on the secondary phase particles (Si and Mg₂Si) studied by means of TEM inspections, showed a much higher volume fraction of very fine b” metastable particles (with a mean equivalent diameter in the range: 130 - 95 nm after 6 passes) than prior to deformation. Now, these particles have a slightly smaller volume fraction and a greater mean spacing. This means that both fragmentation of the Mg₂Si particles and partial dissolution and redistribution of Mg and Si in the Al-matrix took place (see also [40]). Representative TEM and SEM-EDS analyses of the material after 6 passes, Fig 6 (a),(b), shows a partial dissolution, of Mg and Si previously forming long, needle-like Mg₂Si particles, fragmented by glide dislocations created by the severe plastic deformation. It is interesting to note that the first ECAP pass is responsible for the Mg₂Si fragmentation and for partial dissolution of the Si particles. Thus, the Mg₂Si refinement into finer particles effectively contributed to the hardening by pinning dislocations.

In that case a strain of about 12 have been obtained. The cells, which were found to contain high levels of interior dislocations, had a mean size of ~2 θm after the first pass in presence of Sc, and of ~5 θm in the other material; cell boundary misorientation was mainly of 4-5¡, and 1-2¡, respectively. Cell size shrank with strain more effectively in Alloy 2, reaching a mean size of ~330 nm, after 8 passes, and ~250 nm after a strain of 12, while it was considerably less pronounced in Alloy 1, reaching the value of ~720 nm, after 8 passes, and ~450 nm at a strain of 12. The cell boundary misorientation increased much more in the presence of the Al₃(Sc₁ₓ,Zrₓ) to a mean value of ~8¡, compared with ~5¡ in Alloy 1. In the Alloy 1, the Al₃Zr particles are mostly coherent and heterogeneously distributed throughout the matrix; in Alloy 2, the Sc-containing particles are much more homogeneously distributed. The deformation and microstructure refining mechanism is believed to be greatly influenced by the spatial distribution and size of the fine dispersoids. In a sense, they act as preferential deforming paths for the deformation bands, which control the refining process. Dispersoids pin not only free dislocations, but, if possible, even more effectively, also cell boundaries and the newly introduced deformation bands.
which tend to form block walls. Thus, either block walls and cell boundaries, formed by increasing severe plastic deformation, flow and slide within the microstructure, primarily by the effect of the specific die geometry \[18, 27, 28, 37-43\]. They also adhere to the row of fine dispersoids encountered in their path. This may also explain the markedly smaller fraction of high-angle boundaries (essentially block walls) formed during straining in Alloy 2, compared to Alloy 1. Particles pin the new formed boundaries and, inducing them to follow the previously formed boundaries, contribute to their much pronounced misorientation gradient and, as a consequence, their smaller fraction with respect the material containing a smaller fraction of Al\(_3\)Zr dispersoids (Fig. 8).

In a previous manuscript (Cabibbo et al. \[40\]), a breaking-up of Mg\(_2\)Si and shrinking of Si particles in a similar 6082-T8 subjected to ECAP was documented. The same investigations were carried out on the Alloy 1 and Alloy 2; results are reported in Table 1, where for a sake of convenience, the data published in \[44\], and related to the parent Al-Mg-Si T8 alloy, are also reported.

The major aspect is by far the remarkable Mg\(_2\)Si size reduction with strain (especially after the first pass), whilst Si particles reduction effect is rather limited. This appear also in the case of Al Alloy 1 than Alloy 2, revealing a Sc-containing dispersoids effect on the microstructural deforming aspects. The very interesting aspects in the present case are the reduced scale of the former phenomenon following severe plastic deformation, especially in the material containing Al\(_3\)(Sc\(_{1-x}\),Zr\(_x\)) dispersoids, compared to the case presented in [44]. Dispersoids containing particles are believed to be able to inhibit the effect of dislocations in cutting and promoting re-solution of Mg\(_2\)Si and Si particles, by effectively pinning them. The Al\(_3\)(Sc\(_{1-x}\),Zr\(_x\)) has by far a predominant role in the material hardening mechanism over all the other particles decorating the microstructure. The rather low volume fraction of both Si and Mg\(_2\)Si fine secondary phase particles is basically due to the overaging treatment at 463K/8h, which induces the particle growing to a micrometer scale. These were not taken into account in our analyses due to their far less effectiveness in alloy hardening.
Table 2 - Equivalent diameter ($d_{eq}$), volume fraction ($N_v$), subgrain size ($\lambda$) as a function of strain (number of ECAP passes) for AA6082-Zr (alloy-1), AA6082-Sc-Zr (alloy-2) and commercial AA6082.

<table>
<thead>
<tr>
<th>Alloy 1</th>
<th>$d_{eq}$ [nm]**</th>
<th>$N_v$ [$10^{19}$ / m$^3$]**</th>
<th>$\lambda$ [nm]**</th>
</tr>
</thead>
<tbody>
<tr>
<td>as-extruded + T8</td>
<td>S 130</td>
<td>1.05</td>
<td>1090</td>
</tr>
<tr>
<td></td>
<td>Mg$_2$Si 185</td>
<td>1.30</td>
<td>810</td>
</tr>
<tr>
<td>ECAP 1 pass</td>
<td>Si 118</td>
<td>0.90</td>
<td>1420</td>
</tr>
<tr>
<td></td>
<td>Mg$_2$Si 168</td>
<td>0.85</td>
<td>1160</td>
</tr>
<tr>
<td>ECAP 8 passes</td>
<td>Si 116</td>
<td>0.60</td>
<td>1530</td>
</tr>
<tr>
<td></td>
<td>Mg$_2$Si 125</td>
<td>0.80</td>
<td>1220</td>
</tr>
<tr>
<td>ECAP 12 passes</td>
<td>Si 102</td>
<td>0.45</td>
<td>1810</td>
</tr>
<tr>
<td></td>
<td>Mg$_2$Si 115</td>
<td>0.50</td>
<td>1620</td>
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<table>
<thead>
<tr>
<th>Alloy 2</th>
<th>$d_{eq}$ [nm]**</th>
<th>$N_v$ [$10^{19}$ / m$^3$]**</th>
<th>$\lambda$ [nm]**</th>
</tr>
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<tr>
<td>as-extruded + T8</td>
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<td>1.00</td>
<td>1050</td>
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<tr>
<td></td>
<td>Mg$_2$Si 190</td>
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<td>780</td>
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<tr>
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<td>0.90</td>
<td>1140</td>
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<tr>
<td></td>
<td>Mg$_2$Si 200</td>
<td>0.95</td>
<td>900</td>
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<tr>
<td>ECAP 8 passes</td>
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<td>0.80</td>
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<td>Mg$_2$Si 120/65</td>
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<tr>
<td></td>
<td>Mg$_2$Si 160</td>
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<tr>
<td>T8</td>
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<td>490</td>
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<td></td>
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<tr>
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<td>Mg$_2$Si 150</td>
<td>0.75</td>
<td>3030</td>
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<tr>
<td>ECAP 4 passes</td>
<td>Si 110</td>
<td>0.65</td>
<td>4540</td>
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<td>Mg$_2$Si 135</td>
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<td>2640</td>
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<td>4560</td>
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<tr>
<td></td>
<td>Mg$_2$Si 110</td>
<td>1.40</td>
<td>2660</td>
</tr>
</tbody>
</table>

Only the nanometre-scale particles are here considered.

*: Associated error: 5%.

CONCLUSIONS

AA1200. Despite the redundant nature of route Bc, up to a total strain of 8, this processing route still gave rise to marked grain refinement. After the maximum strain, the billet contained a bimodal structure of grains fairly larger 1-1.2 mm and submicron grains concentrated in bands. Among the three different processing routes, the material processed via route Bc contained the lowest density of high angle boundaries with a high angle grain boundary fraction of only 30 pct, after a strain of $e \approx 3$, and still 25 pct of LABs, after the maximum strain ($e \approx 8$).

AA5754. TEM investigation reveals that ECAP is an effective tool for achieving substantial reduction in the grain size. The initial grain size of 70 µm was reduced to $\approx 0.3-0.4$ µm by ECAP after 8 passes at room temperature.

AA6082. Moreover, in the full annealed material the strengthening effect of Si appeared to be predominant over Mg$_2$Si, whereas in the severely deformed material, the metastable b” particles (precursors of the stable Mg$_2$Si) revealed a widespread tendency to be fragmented and the finer particles were more effective in pinning the dislocations. By contrast, Si tended to dissolve under SPD and its strengthening effect was drastically reduced.
AA6106 with Zr and Zr+Sc. The major results for these two alloys can be highlighted as follows:

a. The elongated grains of full annealed and overaged, pressed (Sc-Zr)-containing material were thinner respect the ones of the Zr-containing material, the mean transverse grain spacing being ~12 mm and ~20 mm, respectively.

b. The presence of the very fine Al₃(Sc₁₋ₓₜₓₓ, Zrₓ) dispersoids, in the (Al-Mg-Si)-Zr alloy has highlighted some complex and important effects. The particles effectively reduce the block walls spacing and the cell size, thereby increasing the critical strain required to acquire a sufficiently large fraction of high angle boundaries (essentially block walls) in the microstructure. At the same time, boundary misorientation continuously increased up to a strain of 12, especially for the (Sc-Zr)-containing alloy.

c. An interesting aspect is the reduction of breaking-up of Mg₂Si particles and Si shrinkage, generated by the severe plastic deformation, especially in the material containing Al₃(Sc₁₋ₓₜₓₓ, Zrₓ), compared with the parent AA6082.

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REFERENCES

39. Langdon
Experimental Investigations of the Hydroforming Processes Performed on Seamless Tube of AA 7003

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Abstract
The hydroforming process is a promising technique for the production of components in complex shapes which may increase stiffness and momentum of inertia. This study has been focused on a complete experimental characterization of hydroformed aluminium seamless tubes. The Al seamless tubes have been marked with circle grids produced by a specifically developed chemical etching based on CuCl₂ reactant. During forming, the circle patterns are deformed with the Al alloys and so they permit to measure the local deformation. Moreover, under the hypothesis of volume conservation during plastic deformation, the measurements of the deformed markers allow the evaluation of the thickness variations also as a function of the imposed curvature radius. Finally, the texture analysis has been performed by SEM-EBSD and the development of particular crystallographic textures, which revealed possible residual formability, have been identified. The results represent a preliminary step for further implementation of a simulation activity which will take into account also the forming behaviour of the materials under friction forces produced on the tube during expansion in the die.

Riassunto
Il processo di idroformatura è una tecnologia promettente in grado di realizzare geometrie complesse, allo scopo di incrementare rigidità e momento di inerzia di alcuni componenti. Questo studio consiste nella caratterizzazione sperimentale dello stato di deformazione di tubi senza saldatura in alluminio idroformati. Sui tubi è stata ricavata una opportuna marcatura, attraverso un attacco chimico a base di CuCl₂. Durante il processo, la marcatura subisce la stessa deformazione del tubo, permettendo così di verificare localmente lo stato di deformazione. Inoltre, sfruttando l’ipotesi di costanza del volume durante la deformazione plastica, la misura dei marcatori deformati ha permesso di valutare la variazione dello spessore, in funzione del raggio di curvatura imposto. Infine, è stata analizzata la tessitura cristallografica con la tecnica SEM-EBSD, portando alla individuazione delle componenti di tessitura sviluppate.

Lo scopo di questo tipo di attività è quello di fornire un supporto alla simulazione dei processi, tenendo in considerazione anche il comportamento del materiale sottoposto alle forze di attrito sviluppate durante l’espansione nella cavità dello stampo, attraverso il contatto con le pareti.
INTRODUCTION

Tube hydroforming (THF) is a metal forming process through which the tubes are formed into complex shapes within a die cavity by the simultaneous application of internal pressure and axial compressive forces (Figure 1) [1,2,3]. An increasing acceptance and application of this technology demands a full understanding of interaction among the processing variables for a sound, and defect-free component. An overall review of the hydroforming process can be found in several publications [1-8] when the general conclusion that for steel tube the maximum strain which can be imposed is in the range of 0.1 - 0.7, has been reached.

In hydroforming of the tubes the ultimate goal is to form a blank tube of uniform cross-section into complex shape with varying cross-sections without causing forming instability like bursting, necking, wrinkling or buckling. The overall success of hydroforming heavily depends on the incoming tubular material properties [1,8,9,10,11,12]: yield and tensile strength, ductility and anisotropy. The effect of the strain-hardening exponent (n-value) and of the plastic anisotropy (r-value) depends on the crystallographic orientation and can affect the developed internal pressure, the wall thickness distribution and the maximum expansion [13]. The control of tube production and of the annealing process needs to be carefully managed to produce a tube with desired properties.

In the work an experimental evaluation of strains in complex shape sections of hydroformed tubes was conducted by studying the influence of two different internal pressures on the crystallographic textures affecting the formability performances of the materials.

Fig. 1: Elements of a typical THF process. (Fq) Axial Force, (Fr) Counter Force, (Pi) internal pressure, (Rc) corner radius, (Re) fillet radius [1].

The aim of this study is the experimental determination of the local strain of the thinning realized on seamless tube of AA7003 as a function of the imposed curvature radius and of the friction developed by the contact between the tube and the die wall. The determination of the textures performed by SEM-EBSD analysis on the hydroformed aluminium tube has allowed to associate the texture evolution with the strain imposed by the hydroforming system and by the applied operating parameters. This step can represent a fundamental issue for the implementation and validation of a reliable computational model taking into account the macroscopic stress strain relations, the interaction among the tube and the die walls and the evolution of the involved crystallographic textures.

EXPERIMENTAL PROCEDURE

The hydroforming experiments have been performed on AA7003 seamless tubes whose chemical composition [14] and the related mechanical properties are reported in tables 1 and 2, respectively. Before the hydroforming operation the wiredrawn tube underwent an annealing treatment at 390 °C for 2h and a successive controlled cooling in furnace to a temperature of 250 °C and then the tube has been cooled in quiet air to room temperature. The hydroforming of the tube has been performed 35h after the end of the annealing treatment.

<table>
<thead>
<tr>
<th>%wt</th>
<th>Si</th>
<th>Fe</th>
<th>Cu</th>
<th>Mn</th>
<th>Mg</th>
<th>Cr</th>
<th>Zn</th>
<th>Ti</th>
<th>Pb</th>
<th>Zr</th>
<th>Bi</th>
<th>Sn</th>
<th>Ni</th>
</tr>
</thead>
<tbody>
<tr>
<td>Contents</td>
<td>0.153</td>
<td>0.257</td>
<td>0.031</td>
<td>0.176</td>
<td>0.838</td>
<td>0.120</td>
<td>5.505</td>
<td>0.029</td>
<td>0.003</td>
<td>0.1505</td>
<td>0.0001</td>
<td>0.0020</td>
<td>0.0040</td>
</tr>
</tbody>
</table>

Table 1 - Measured chemical composition of AA7003 (wt%)
The textures of the tube have been measured before and after the hydroforming procedure through the SEM-EBSD technique, and the polar diagram, the inverse polar diagram and the ODF (Orientation Distribution Function) have been determined.

The tube surface have been drawn by a chemical etching producing a round shape grid. The grid should resist to the high friction produced by the contact between the tube and the die allowing a correct measurement of the resulting grid circles. The round shape of the markers was chosen because it allowed a rapid evaluation of the strain along different directions. The circles of the grid were 4 mm diameter and the distance between the centres of two adjacent circles was 6 mm.

The tube external surface was pickled through a solution of NaOH (30 g/dm³), at 35–40 °C for 180–300 s and then was dipped into a solution 33% HNO₃, at room temperature for 30–45 s and dried. The tube was covered by adhesive polyethilene film with a sequence of circles representing the grid dipped in a solution of CuCl₂·2H₂O (10 g/dm³), H₂SO₄ (90 g/dm³), at room temperature for 105–135 s and finally washed by immersion in tap water for 300 s and dried by forced air at 45°C (Figure 2). Each circular array of round shape markers (at a certain distance from the tube end) is composed of 22 elements. The circle grid analysis have been operated along four different directions: 0°, 45°, 90°, 135° rotated directions from the tube axis (Figure 3, Figure 4).

<table>
<thead>
<tr>
<th>Mechanical properties</th>
<th>Rₘ [N/mm²]</th>
<th>Rₚ₀₂ [N/mm²]</th>
<th>A₅ [%]</th>
<th>A₂₀ [%]</th>
<th>HB</th>
</tr>
</thead>
<tbody>
<tr>
<td>186</td>
<td>94</td>
<td>21</td>
<td>17.5</td>
<td>52.4</td>
<td></td>
</tr>
</tbody>
</table>

Table 2 - Mechanical properties before the hydroforming operation

The tube external surface was pickled through a solution of NaOH (30 g/dm³), at 35–40 °C for 180–300 s and then was dipped into a solution 33% HNO₃, at room temperature for 30–45 s and dried. The tube was covered by adhesive polyethilene film with a sequence of circles representing the grid dipped in a solution of CuCl₂·2H₂O (10 g/dm³), H₂SO₄ (90 g/dm³), at room temperature for 105–135 s and finally washed by immersion in tap water for 300 s and dried by forced air at 45°C (Figure 2). Each circular array of round shape markers (at a certain distance from the tube end) is composed of 22 elements. The circle grid analysis have been operated along four different directions: 0°, 45°, 90°, 135° rotated directions from the tube axis (Figure 3, Figure 4).

Fig. 2: An example of the grid produced on the tube before the hydroforming procedure after the extraction from the etching bath (a), after drying (b) and scrubbing (c).

Fig. 3: An example of the grid produced on the tube before the hydroforming procedure after the extraction from the etching bath (a), after drying (b) and scrubbing (c).
The shape of the deformed markers, can be easily revealed through an image analyser which permits the successive computation of strain by the well known formula:

$$\varepsilon_x = \ln \frac{d}{d_0}$$  \hspace{1cm} (1)

A precise determination of the local deformation produced on the tube wall under the hypothesis of the volume conservation during the plastic deformation allows the computation of the local thinning.

The SEM-EBSD analysis has been performed with the following operative parameters: magnification 150X, pixel resolution 20 µm², accelerating voltage 20 kV.

In each analysis an area of 2 mm² has been investigated to grant the statistical reliability of the crystallographic data. The reference system has been set so that the RD direction has been set parallel to the tube one and the ND is always perpendicular to the external profile of the investigated tube section. The textures have been identified before and after the hydroforming. The hydroforming experiments have been performed on a tube: 780 mm long, 1.5mm thick and with an external diameter of 46 mm. The length of the tube extremities forbidden to expansion was 55 mm.

After lubricant deposition the experiments have been realized at two different levels of the maximum internal pressure: 36 MPa and 81 MPa. The deformation pattern of the formed tube has been analysed in correspondence of four section taken at 188, 442, 592, 706 mm from the reference extremity (Figure 5).
RESULTS

The results related to the revealed state of deformation have been associated to each measured strained marker and its identification has been performed through a correct numbering of the 22 round shape markers corresponding to a specific section. The markers composing the array belonging to a particular section have been ordered through a clockwise rotation from the upper point of the section taken as the reference position (Figure 6). The maximum strains belong to the sections located at 442 mm, 592 mm and at 706 mm featured by the smallest imposed curvature radius.

Fig. 7: Circle grid analysis in correspondence of the two applied levels of internal pressure.
The high deformations reached in the section at 442 mm from the extremity can be expected without particular surprise, because this is the section in which the down part of the hydroformed tube underwent the largest expansions and the largest displacement in correspondence of the markers 10 and 11, but it is interesting that the highest applied internal pressures

Fig. 8: Principal deformations computed on the basis of the sizes of the circle grid analysis of the two applied levels of internal pressure.
seem to increase the homogeneity of the deformation induced in the tube wall.

On the other hand, the presence of changes of concavity and the imposition of small curvature radius, as the ones realized in the sections 592 mm and 706 mm from the reference extremity, implies some incoming critical situations in the deformation states as well.

The evolution of the textures from the crystallographic situation before the hydroforming is clear (Table 3).

The revealed textures are consistent with the revealed deformation state. In particular, a sharp increase of the texture components featured by planes \{001\} tangent the contour profile of the wall (i.e. \{010\} \times 001 > \{-101\} \times 101 > \{-001\} \times \{120\} > ) characterizes the regions interested by the largest deformations. These texture components have been revealed in correspondence of the points which underwent the most significant deformations and, using a logarithmic scale, their statistical intensities, if compared with a random distribution, assume the value of 8 which is characteristic of very sharp texture components. This phenomenon is related to a progressive decreasing of the favourable texture components characterized by \{011\} and \{111\} planes and an intensification of the ones featured by \{123\} and \{001\} (Table 4).

### Table 3 - Texture components revealed in the AA7003 before the hydroforming

<table>
<thead>
<tr>
<th>Textures components</th>
<th>Intensity (log scale)</th>
</tr>
</thead>
<tbody>
<tr>
<td>{10} \times 001 &gt;</td>
<td>(8)</td>
</tr>
<tr>
<td>{11} \times 111 &gt;</td>
<td>(8)</td>
</tr>
<tr>
<td>{14} \times 132 &gt;</td>
<td>(7)</td>
</tr>
</tbody>
</table>

### Table 4 - Texture components revealed in the AA7003 at different deformation condition.

<table>
<thead>
<tr>
<th>Applied internal pressure</th>
<th>Region</th>
<th>Region</th>
<th>Region</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Under marker 3 of each section</td>
<td>Under marker 13 of the section at 442, 592, 706mm</td>
<td>Under marker 12 of the section at 592, 706mm</td>
</tr>
<tr>
<td></td>
<td>Intensity (log scale)</td>
<td>Intensity (log scale)</td>
<td>Intensity (log scale)</td>
</tr>
<tr>
<td>36MPa</td>
<td>{010} \times 001 &gt;</td>
<td>(8)</td>
<td>{012} \times 010 &gt;</td>
</tr>
<tr>
<td></td>
<td>{24} \times 231 &gt;</td>
<td>(8)</td>
<td>{24} \times 231 &gt;</td>
</tr>
<tr>
<td></td>
<td>{10} \times 101 &gt;</td>
<td>(7)</td>
<td>{001} \times 100 &gt;</td>
</tr>
<tr>
<td></td>
<td>{001} \times 120 &gt;</td>
<td>(7)</td>
<td>{011} \times 322 &gt;</td>
</tr>
<tr>
<td></td>
<td>{11} \times {121} &gt;</td>
<td>(6-7)</td>
<td>{011} \times {121} &gt;</td>
</tr>
<tr>
<td></td>
<td>{11} \times {121} &gt;</td>
<td>(7)</td>
<td>{11} \times {121} &gt;</td>
</tr>
<tr>
<td>81MPa</td>
<td>{11} \times 011 &gt;</td>
<td>(8)</td>
<td>{25} \times {121} &gt;</td>
</tr>
<tr>
<td></td>
<td>{001} \times {121} &gt;</td>
<td>(8)</td>
<td>{23} \times {221} &gt;</td>
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<tr>
<td></td>
<td>{14} \times 261 &gt;</td>
<td>(8)</td>
<td>{001} \times {121} &gt;</td>
</tr>
<tr>
<td></td>
<td>{11} \times 261 &gt;</td>
<td>(8)</td>
<td>{001} \times {121} &gt;</td>
</tr>
<tr>
<td></td>
<td>{011} \times {011} &gt;</td>
<td>(8)</td>
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<td>(8)</td>
<td>{011} \times {011} &gt;</td>
</tr>
<tr>
<td></td>
<td>{11} \times {011} &gt;</td>
<td>(7)</td>
<td>{11} \times {011} &gt;</td>
</tr>
<tr>
<td></td>
<td>{32} \times {121} &gt;</td>
<td>(7)</td>
<td>{11} \times {011} &gt;</td>
</tr>
<tr>
<td></td>
<td>{24} \times {210} &gt;</td>
<td>(7)</td>
<td>{11} \times {011} &gt;</td>
</tr>
</tbody>
</table>
**DISCUSSION**

A good fitting has been found among the deformations measured through the thickness and the ones computed under the hypothesis of volume conservation on the basis of the values of the deformation revealed along the direction parallel and perpendicular to the tube axis. So, the measured deformations along the direction perpendicular and parallel to the tube axis ($e_{\perp}, e_{\parallel}$) can be assimilated to the principal deformations ($e_1, e_2$). This result implies that the principal deformation directions correspond to the direction parallel and perpendicular to the tube axis, although after a first approximate sight the overall deformation patterns can appear as very complex in order to realize such a complex shape. Thus, the deformation through the thickness can be considered the third principal component of deformation ($e_3$).

The increase of the internal pressure can permit the decreasing of the localized wall thinning. This is extremely advantageous because such a situation grant a lower thinning of the tube wall avoiding the plastic instability which can take place in the presence of an excessive reduction of the tube thickness. The evaluation of the experimental results clearly indicates that the application of the highest internal pressure can favour a more homogeneous plastic behaviour. This situation should be confirmed also by a successive dynamic structural simulation, but it seems to be due to the fact that, in presence of a low internal pressure, the points characterized by a change of concavity or by a restriction of the section profile can constitute an obstacle to the plastic flow of the material which enters in contact with the forming die. So, those points represent the constraint points for the material to be formed, due to the frictions developed by the relative motions among the die walls and the material during its deformation. The imposition of the highest pressure levels can certainly produce an increase of the friction forces, but can also contribute to increase the plastic flow of the material on those critical points, favouring the thinning and the general plastic flow of the material towards the other regions of the material, which have not already reached the final point of their displacement on the die surface. In presence of the highest pressure levels in the constraints points the tube wall is subject to a greater thinning, but this reduces the thinning experienced by the other material regions which are still under deformation to reach the die surface. Thus, the application of the highest internal pressure makes the wall thickness more homogeneous and decrease the possibilities to meet the conditions of the plastic instability.

The induced textures point out a satisfactory consistency with the defined measured deformation states and, provided a specific point, they do not present significant variations between the inner and the outer region of the wall thickness. Actually, the regions featured by the largest deformations show the texture components with the lowest formability and there is a well defined pattern of crystallographic organization which leads from the isotropy to the well formable textures featured by the $\{111\}$ and $\{011\}$ planes perpendicular to the normal of the profile section and from the weakening of these last ones to a progressive increasing of the textures featured by $\{001\}$ and by the textures near the $\beta$-fiber (i.e. $\{123\}$-$\{124\}$) which is associated to the decrease of the intensity of the textures belonging to the $\alpha$-fiber and $\gamma$-fiber. On the other hand, the application of the highest internal pressure allows to realize also a more homogeneous distribution of the crystallographic orientation and to slower the reaching of $\{001\}$-$\{001\}$ (Cube texture) preserving the formability and the thoughness of the material [16].

**CONCLUSION**

This preliminary study about the experimental evaluation of the local deformations and about the development of the crystallographic textures induced within the hydroformed tube allows to state that:

- the procedure based on the chemical etching and designed to produce a grid suitable for the experimental identification of the local deformation hydroformed tube has been tested and it can be considered as extremely reliable;
- the most critical sections are those interested by a significant thinning and by the largest expansions, by the smallest imposed curvature radius and by a possible change of the profile concavity;
- the increase of the imposed internal pressure allows the tube to obtain a more homogeneous state of deformation and of crystallographic textures. The highest internal pressure can be balanced by the constraint effects produced on the tube displacement by the change of concavity or by the restriction of the profile section. The larger thinning takes place in the points of the section constrained by the highest friction and this phenomenon seems to improve the plastic flow of the materials in these regions, so that they can provide a larger quantity of material to the regions which have not already reached the die surface;
- the strongly deformed regions are featured by the decreasing of $\{111\}$ and $\{011\}$ planes perpendicular to the normal section with a simultaneous increasing of the textures featured by $\{001\}$ and by the textures near the $\beta$-fiber, (i.e. $\{123\}$-$\{124\}$) and the decreasing of the intensity of the textures belonging to the $\alpha$-fiber and $\gamma$-fiber: this degeneration of the crystallographic texture points out a reduction of the formability and toughness resources associated to the hydroformed alloy.
REFERENCES


LIST OF SYMBOLS

- $e_X$: deformation measured along the generic direction $X$.
- $d_X$: measured final diameter of the round shape marker along the generic direction $X$.
- $d_0$: initial diameter of the round shape marker.
Abstract
The aim of the present research was to study the effect of the Friction Stir Welding process on the microstructure and impact toughness of the composites W6A20A (AA6061 reinforced with 20vol.% of Al₂O₃ particles) and W7A10A (AA7005 reinforced with 10vol.% of Al₂O₃ particles).
FSW, because of the concurrent effect of severe plastic deformation and frictional heating during welding, had effects both on the reinforcing particles and the aluminium matrix. It induced a significant reduction in the reinforcement particles size and their better distribution in the welded zone as well as a grain refinement of the aluminium alloy matrix in the nugget due to dynamic recrystallization. The frictional heating, moreover, had effects on the growth, dissolution and re-precipitation of hardening precipitates. The impact tests showed that the total impact energies increased in the FSW composites, respect to the corresponding base materials.

Riassunto
In questa ricerca è stato studiato l’effetto della Friction Stir Welding sulla microstruttura e sulla resilienza dei compositi Duralcan W6A20A e W7A10A, aventi come matrici le leghe AA6061 e AA7005 rinforzate con il 20% e 10% in volume, rispettivamente, di particelle di Al₂O₃. I risultati della caratterizzazione microstrutturale hanno evidenziato come il processo FSW, in conseguenza dei campi termici e di deformazione plastica indotti, abbia effetti sia sulla matrice, che sulle particelle di rinforzo. In entrambi i compositi l’utensile ha indotto una frammentazione delle particelle di maggiori dimensioni, un arrotondamento degli spigoli ed una migliore distribuzione delle stesse nel cordone, rispetto al materiale base. A seguito di fenomeni di ricristallizzazione dinamica, si è osservato un affinamento dei grani della matrice nel nugget; mentre per quanto riguarda i precipitati indurenti, i campi termici hanno indotto fenomeni di ingrossamento, dissoluzione e riprecipitazione di diversa entità nelle varie zone del cordone. Le modificazioni microstrutturali hanno portato ad un incremento significativo dei valori di resilienza dei campioni sottoposti a FSW rispetto ai valori rilevati nei compositi non saldati.
INTRODUCTION

Aluminium matrix composites, reinforced with ceramic particles, can be welded by fusion processes and solid state joint processes [1]. However, the traditional fusion welding techniques should be critical when applied to these materials. Some difficulties are associated with the typical welding problems of aluminium alloys, such as: high thermal expansion and conductivity, high solubility of gases in the molten state, solidification shrinkages and cracking presence of oxide inclusions. Moreover, the presence of the ceramic reinforcement can cause other problems, making welding difficult, such as: high viscosity of the melted composites respect to the unreinforced alloys, that leads to an extensive presence of solidification shrinkages and porosity; undesired interfacial chemical reactions between the ceramic reinforcement and the molten matrix alloy; different thermal expansion coefficients between the matrix and the ceramic reinforcement, which cause thermal stresses during welding; segregation of particles during solidification, with a consequent reduction in the mechanical properties.

Friction Stir Welding (FSW) is a relatively new joining process, developed at The Welding Institute (TWI) in 1991 for aluminium alloys, and is presently attracting considerable interest [2-5]. In this solid state welding technique (schematically showed in Fig. 1 [6]) a rotating tool, cylindrical in shape with a pin of smaller diameter extending from the tool shoulder, is translated along the joint line and produces, during its path, frictional heating and also plastic deformation of the material, due to a stirring effect around the pin. The material along the joint line is heated to a softened condition, transferred around the periphery of the tool, and subsequently solid state welded. Important process parameters include the tool geometry, the rpm and travel speed, as well as the downward force on the tool.

Friction Stir Welding has been initially developed for welding aluminium alloys, but several recent studies show that it should be also successfully applied to other materials, such as particles reinforced aluminium based composites [7-9]. In this case, wear damage of the tool occurs during welding, due to the abrasive action of the ceramic reinforcement [10]. However, since joining occurs in the solid state, FSW, respect to the classic fusion welding techniques, avoids the formation of shrinkages, porosity as well as the aggregation of the ceramic reinforcement in the welded zone and significantly reduces the thermal stresses.

The aim of the present research was to study the effect of the FSW process on the microstructure and impact toughness of two aluminium matrix composites, based on the AA6061 reinforced with 20vol.% of Al₂O₃ particles (W6A20A) and on the AA7005 reinforced with 10vol.% of Al₂O₃ particles (W7A10A). The impact behaviour was studied using an instrumented Charpy impact pendulum and the results were related to the microstructure modifications induced by the FSW.

EXPERIMENTAL

The W6A20A and W7A10A composites were produced by Duralcan (USA) using a proprietary molten metal processing. The as-cast composites were extruded at 480 °C to a rectangular plate (cross section of 100x7 mm²) and then were heat treated at the T6 state, including: solubilization at 540 °C for 1 h, water quenching and ageing at 145°C for 16 h, for the W6A20A; solubilization at 465 °C for 1 h, water quenching and aging at 95 °C for 1h and 145 °C for 16 h, for the W7A10A.

The extruded and T6 treated plates (7 mm in thickness) were Friction Stir Welded at the GKSS Research Institute (Geesthacht, Germany), by using a NeoTriceps 805, CN controlled, five-axis robot [8]. The FSW tool (showed in Fig.2), with 20 mm diameter shoulder and 8 mm diameter pin, was made with a highly wear resistant steel (an age-hardenable martensite reinforced with 30vol.% TiC), having a hardness of about 63 HRC.

The microstructural characterization of welded composites, was carried out by means of optical (OM) and scanning electron microscopy (SEM) equipped with an energy dispersive spectroscopy (EDS). Image analyses, performed with the Image Pro-Plus software, were carried out on the optical micrographs, in order to evaluate the effect of the FSW process on the reinforcement particles (size, shape and local volume fraction) and on the aluminium matrix grain size. The effect of the FSW process on the particles distribution was also...
studied by means of the Voronoi tessellation method [11-12]. According to this statistic method, given a set of points (called generators) and a distance function, Voronoi tessellations are subdivisions of another set of points into subsets such that the points in each subset are closest, with respect to the given distance function, to one of the generators than to any of the other generators. Voronoi tessellation is useful in very different applications: simulation of grain growth damage and shear banding in polycrystals, characterisation of composites and simulation of microcrack nucleation or intergranular stress corrosion.

For metallographic investigations the specimens were mechanically ground on coarse emery papers, then polished with 9, 6 and 1 µm diamond paste and finally etched with Keller’s reagent. Microhardness profiles were taken across the welded joint. A 20 g load was used for the Vickers indentation (HV$_{0.02}$), in order to evaluate only the matrix interparticles microhardness.

The impact tests were carried out on welded and base composites (five specimens for each material), using an instrumented pendulum machine (CEAST, Resil Impactor 50 J), according to ASTM E 23. Sub-size Charpy V specimens (10x5x55 mm$^3$, with 2 mm deep V-notch) were used for the tests, due to the reduced thickness of the plates; the specimens were electro-discharge machined with the notch perpendicular to the welded line (Fig.3). In order to investigate the influence of the FSW process on the mechanisms of failure of the tested composites, SEM analyses were carried out on the fracture surfaces.

**RESULTS AND DISCUSSION**

**Microstructural characterization**

The Fig. 4 shows a friction stir welded composite plates. The surface in contact with the tool shoulder (Fig.4-a) is characterized by the presence of semicircular features, similar to those induced by a conventional milling process. The average surface roughness Ra on this side, evaluated by a stylus profilometer along the y-direction (according to the scheme in Fig.5), was equal to 3.5 µm. The opposite surface (Fig.4-b) doesn’t show evident surface modification induced by the FSW process and so the average surface roughness was the same of the base material (as received) (Ra=0.7 µm).

The microstructural characterization was carried out on samples cut from the transverse cross section of the welded plate, at different y-values (according to Fig.5), that is from the base material to the nugget zone (welded zone), and at different z-values, that is at different distances from the shoulder tool. Figure 6 is a typical low magnification optical image of the welded zone, showing the “onion ring” structure characteristic of the FSW. None of the typical defects, generally observed in the welded zone of MMCs joined using conventional melting processes, such as porosity or reinforcement segregation, was detected. Optical micrographs showing the transition from the base material (left side) to the nugget (right side) are shown in Fig.7 (a-b) for the W6A20A and W7A10A, respectively. In both materials, a different distribution of particles and a reduction in their size, due to the abrasive action of the hard tool, are evident. Fragmentation of the large alumina particles, induced by the FSW, was confirmed by the image analyses, carried out on optical micrographs of the base and welded materials. The results, reported in Table I, show that the particles refinement was higher for the W6A20A, characterized by initial larger reinforcement particles (average particle area =135 µm$^2$), respect to those of the W7A10A (average particle area = 44 µm$^2$). The particle area, in the welded W6A20A, decreased of about 60% and 40%, in zones closer or farther from the shoulder respectively, probably due to different stresses induced by the
tool on the particles and to the highest contact area between the tool and the material, due to the tool geometry. In this composite, also the particle shape factor was reduced by the FSW process, from 2.1 in the base material to 1.9 in the FSW zone. It is worth noting that the variations in the reinforcement size and shape should reduce the stress intensification caused by the particles, enhancing the toughness of the welded composite. A reduction in the particles area (about 30%) and no variation in the particle shape factor were, instead, observed in the W7A10A composite, probably due to the smaller initial size of the reinforcement particles.

Several authors report that FSW also leads to dynamic recrystallization of aluminium alloys, due to severe plastic deformation producing large frictional heating [13-15]. This effect is enhanced by the reinforcement particles, stimulating nuclei for recrystallization [16]. A substantial grain refinement in the aluminium alloy matrix, in fact, was observed in the nugget of both welded composites, as one can see by comparing the microstructures of the base materials and nugget zones (Fig. 8). The average grain size of the aluminium alloy matrix, in the W6A20A, decreased from about 29 µm in the base material to 20 µm in the FSW zone. A superior grain refinement was observed in the W7A10A, that showed a decrease of the aluminium matrix grain size from 29 µm in the base material to 12 µm in the nugget zone.

It is well known that a homogeneous distribution of the reinforcement particles is one of the main requirements to achieve good mechanical properties in discontinuously reinforced composites. Therefore, it is important that the welding process doesn’t lead to particle clusters or particle denuded zones. Among the various methods for characterising second phase distributions, tessellation methods have attracted particular attention in their ability to uniquely characterise the surroundings of individual particles within a distribution. The Voronoi tessellation method, in particular, utilizes the centre of the particles to construct a network of polygonal cells, such that any point within a cell is closer to the centre of the particle than to any other centre [11-12]. In particles reinforced composites, with a perfect distribution of the reinforcement, the ratio between the particles area and its area of influence in the matrix, should be equal to the volume fraction of the reinforcement. Therefore the Voronoi tessellation should be a useful method to evaluate the degree of clustering in these composites [17]. In this work, the Voronoi tessellation was used to evaluate the effect of the Friction Stir Welding on the reinforcement particles distribution. The results of the statistical analyses, carried out on the W6A20A and
W7A10A, are reported in Figs. 9-11, respectively. It can be noted that, consequently to the abrasion and fragmentation of the reinforcement particles, caused by the tool and the following increase in their number, the polygons area decreased, then also decreased the area of influence of each reinforcement particle. The plots in Fig. 10 and Fig. 11 show that the FSW process also led to a decrease in the standard deviation of the local reinforcement volume fraction, respect to the base material, indicating a more uniform distribution of the particles. This reduction was equal to about 26% in the W6A20A composite and about 67% in the W7A10A, suggesting that the final microstructure of the latter composite extend to the random model. This result should be probably due to the lower reinforcement content, in the W7A10A, that permits a superior stirring effect of the pin into the 7005 aluminium alloy matrix.

Microhardness measurements

In order to investigate softening or hardening effects induced by the FSW process on the aluminium matrix alloys, microhardness measurements, with a very low load (HV$_{0.02}$), were made from the base material to the nugget zone, on cross-sections of the welded plates. The results are shown in the plots of Fig. 12(a) for the W6A20A, and Fig. 12(b) for the W7A10A. The microhardness profile of the W6A20A shows a decrease of the interparticles matrix microhardness from about 80 HV$_{0.02}$ in the base material up to about 50 HV$_{0.02}$ at the middle line of the FSW zone. This microhardness decrease in the aluminium alloy matrix, even with a reduction in its grain size, was also observed in FSW unreinforced AA6061 and should be probably related to coarsening or partial dissolution of the intermetallic compounds, induced by the frictional heating and severe plastic deformation [18,19]. The microhardness profile for the W7A10A, in Fig. 12(b), shows a minimum value of about 77 HV$_{0.02}$ at the middle line of the nugget zone, a maximum value of about 100 HV$_{0.02}$ in the thermomechanical affected (TMAZ) zone, a second minimum of about 75 HV$_{0.02}$ in the heat affected zone (HAZ); then, the interparticles matrix microhardness increased up to about 84 HV$_{0.02}$ in the base material. This trend can be also related to the microstructural changes induced by the friction stir welding process on the aluminium alloy matrix. In particular, the observed maximum in the TMAZ is probably due to the concurrent effects of strain-hardening and re-precipitation of the transition phases; the lower microhardness in the nugget should be related to
coarsening and/or dissolution of the precipitates and, finally, the minimum of microhardness in the HAZ may be caused by coarsening of the precipitates induced by the frictional heating [20-24]. The different interparticles microhardness profiles in the two FSW composites is therefore due both to the different aluminium matrix grain size, induced by dynamic recrystallization in the nugget, and to the different aging response of the matrix alloys during and after welding.

**Instrumented Charpy Impact Tests**

Impact tests were carried on the sub-size specimens shown in Fig. 3, using an instrumented Charpy pendulum, to investigate the effect of the FSW process. The load-time curve (Fig. 13) can be divided into an elastic zone, corresponding to the initial rise of the curve, a plastic zone starting at the change of the curve slope and a crack zone, where the load rapidly decreases, indicating the start of the crack propagation. The first fluctuation in the rising side of the curve is caused by the inertial loading of the hammerhead, as a result of the acceleration of the specimen from rest. The total energy adsorbed by the specimen under fracture, given by the area under the load curve, is the sum of the energy required for crack initiation, $E_i$, and the energy required for crack propagation, $E_p$, and therefore gives the impact toughness of the tested material.

The results of the Charpy impact tests for the base and welded composites are reported in Table II (average values on five tests). Representative load-time curves for the base and welded materials are shown in Fig. 13(a) for the W6A20A and Fig. 13(b) for the W7A10A. The impact energies increased in both the FSW composites, with respect to the corresponding base materials, from 0.7 J to 2.6 J for the W6A20A composite, and from 1.2 J to 2.9 J for the W7A10A composite. This significant increase in the total adsorbed impact energies can be related to the microstructural modifications induced by the FSW process, such as: refinement and roundness of the reinforcement particles, homogeneous distribution of the reinforcement and reduction of the matrix grain size. In fact, it has been shown that ductility of particle reinforced aluminium alloys is reduced by the presence of large particles [25], but the particle size has little effect on ductility when particles are small [26]. Particle clusters in the matrix can also decrease ductility [27-28]. Also the effect of particle shape on fracture properties of the AA6061 matrix composites has been studied.
and it was found that, since stress concentration in the matrix increases around angular particles, tensile ductility of the composite will be reduced owing to the severe plastic deformation around the particle corners [30]. It is reasonable to suppose that reduction in size and blunting of the reinforcement particles should improve the impact toughness. In fact, it is reported that large and angular particles act as stress concentration sites and easy crack propagation, resulting in low impact toughness [31].

The increase in the crack initiation energy Ei and dynamic yield strength Tab.II) are, probably, mainly related to the grain refinement of the matrix, that was of higher entity for the W7A10A (about 60%) respect to the W6A20A (about 30%). The histograms in Fig.14(a-b) show the contribution of the initiation (Ei) and propagation (Ep) energies to the total impact values, for the base and welded composites. The increase in the propagation energy was greater for the W6A20A, than for the W7A10A, and it is probably related to the higher reduction in the particles size (up to 60%) and particle shape-factor induced by the FSW in this composite, that mainly influence the crack propagation stage.

Fracture surfaces

Micrographs (Fig. 15) of the fractured Charpy specimen surfaces, show a greater amount of plastic deformation in the friction stir welded composites (Fig. 15 b-d), respect to the base materials (Fig. 15 a-c), clearly related to the increase in the total adsorbed energy.

This behaviour was confirmed by SEM analyses, which also permit to evidence more details on the mechanisms of fracture. Fracture surfaces of the impact specimens, machined from base and FSW composites, were always characterized by a bimodal distribution of voids, associated with decohesion of the reinforcement particles, and small dimples associated with the ductile failure of the matrix (Figs. 16 and 17).

In the welded W6A20A composite it is possible to observe a higher volume fraction of small voids, caused by decohesion of the reinforcing particles, and minute dimples, due to the plastic deformation of the matrix, than in the base composite (Fig. 16).
Table 1 - Results of the image analyses measurements carried out on the reinforcement particles and matrix grain size of the W6A20A and W7A10A composite before and after FSW.

<table>
<thead>
<tr>
<th>Material</th>
<th>Zone of analysis</th>
<th>Area $\mu m^2$</th>
<th>Shape factor</th>
<th>Length $\mu m$</th>
<th>Width $\mu m$</th>
<th>Size $\mu m$</th>
</tr>
</thead>
<tbody>
<tr>
<td>W6A20A</td>
<td>Base Material</td>
<td>135</td>
<td>2.1</td>
<td>16</td>
<td>9</td>
<td>29</td>
</tr>
<tr>
<td>&quot;</td>
<td>FSW zone farther from the shoulder tool</td>
<td>82</td>
<td>2.0</td>
<td>12</td>
<td>7</td>
<td>5</td>
</tr>
<tr>
<td>&quot;</td>
<td>FSW zone closer to the shoulder tool</td>
<td>56</td>
<td>1.9</td>
<td>9</td>
<td>6</td>
<td>20</td>
</tr>
<tr>
<td>W7A10A</td>
<td>Base Material</td>
<td>44</td>
<td>2.2</td>
<td>9</td>
<td>5</td>
<td>29</td>
</tr>
<tr>
<td>&quot;</td>
<td>FSW zone</td>
<td>30</td>
<td>2.1</td>
<td>8</td>
<td>4</td>
<td>12</td>
</tr>
</tbody>
</table>

Table 2 - Results of the instrumented Charpy impact tests on the base and FSW composites

<table>
<thead>
<tr>
<th>Composite</th>
<th>Total Impact Energy J</th>
<th>Initiation Energy J</th>
<th>Propagation Energy J</th>
<th>Dynamic yield strength MPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>W6A20A BM</td>
<td>0.7</td>
<td>0.4</td>
<td>0.3</td>
<td>43</td>
</tr>
<tr>
<td>W6A20A FSW</td>
<td>2.6</td>
<td>1.0</td>
<td>1.6</td>
<td>48</td>
</tr>
<tr>
<td>W7A10A BM</td>
<td>1.2</td>
<td>0.8</td>
<td>0.4</td>
<td>38</td>
</tr>
<tr>
<td>W7A10A FSW</td>
<td>2.9</td>
<td>1.9</td>
<td>1.0</td>
<td>49</td>
</tr>
</tbody>
</table>
The higher presence of small voids and lower presence of cracked particles in the welded composite, can be explained with the reduction of local plastic constraints due to the reduction of particles size and better distribution of the reinforcement, induced by the welding process. The small dimensions of ductile dimples can be attributed to the constraints in plastic flow of the matrix, or to the reduction of strains and hence stresses, induced by the refined reinforcement. These differences are not so evident for the W7A10A composite (Fig. 17), since the reinforcement particles are smaller in the base composite and their refinement after welding was lower than in the W6A20A (Table I). The larger presence of dimples in the fracture surfaces of the welded composites, especially in W6A20A, is in agreement with the significant increase in the Charpy impact toughness.

**CONCLUSIONS**

In this work the effect of the FSW process on the microstructure and impact toughness of two particles reinforced aluminium matrix composites (W6A20A and W7A10A) was investigated.

a) The microstructural characterization didn’t show the typical defects generally observed in the welded zone of MMCs, joined using conventional arc welding processes. The main effects of the FSW was a significant reduction in the reinforcement particles size (greater in the W6A20A, due to the larger size of the particles in the base materials) and a more homogeneous distribution of the particles in the welded zone, as confirmed by the Voronoi tessellation analysis. A substantial grain refinement of the aluminium alloy matrix was also observed in the nugget of the welded zones, in both composites, due to dynamic recrystallization induced by the severe deformation and concurrent frictional heating during welding.

b) The interparticles microhardness profiles, on cross-sections of the FSW specimens, was related to the microstructural changes induced by the process on the aluminium alloy matrix. The differences between the two composites is due to the different aluminium matrix grain size, induced by dynamic recrystallization in the nugget, and to the different modifications of the intermetallic compounds.

c) The impact tests, carried out on an instrumented Charpy pendulum, showed that the total energy impact significantly increased in both the FSW composites, respect to the corresponding base materials. This increase can be related to the microstructural modifications induced by the FSW process, such as: refinement and blunting of the reinforcement particles, homogeneous distribution of the reinforcement and reduction in the matrix grain size.

d) Fracture surfaces of the impact specimens were always characterized by a bimodal distribution of voids, associated with decohesion of the reinforcement particles, and small dimples associated with the ductile failure of the matrix. The larger presence of dimples in the fracture surfaces of the welded composites, especially in W6A20A, is in agreement with the significant increase in the Charpy impact toughness.

**REFERENCES**


The microstructure and mechanical properties of a friction stir welded AA6056-T6 aluminum alloy plate were investigated by using polarized optical and transmission electron microscopy techniques. The microstructure revealed different grain morphologies in the thermomechanically affected zones, in proximity of the weld nugget; as the advancing side had fairly elongated, bent grains, whilst the broader retreating side had elongated grains whose width is narrower compared to the advancing side. On both sides, the thermomechanical affected zones showed a process of grain subdivision due to shear strain and temperature increase on approaching the nugget zone. Along with overaged precipitates in both thermomechanically affected zones, \( \beta'' \) and \( \beta' \) particles were detected only within the retreating side. Tensile tests showed yield and ultimate strength slightly lower across the weld compared to the parent material; this difference resulted in a reduction in ductility of the weld region.

**Riassunto**
Il lavoro di ricerca, oggetto del presente lavoro, presenta i risultati relativi alle indagini microstrutturali mediante microscopia ottica in luce polarizzata e microscopia elettronica in trasmissione su un giunto in AA6056-T6, realizzato mediante friction stir welding. La zona termo-mecanicamente alterata mostra una morfologia a grani ben diversa da quella evidenziata in corrispondenza del nugget. Inoltre è stata documentata una asimmetria microstrutturale tra il lato di avanzamento del pin, in rotazione rispetto ai laminati da saldare, e quello opposto. La regione di avanzamento mostra grani allungati ed estremamente curvati, mentre la zona opposta, rispetto al nugget, presenta grani allungati ma molto più sottili rispetto al lato di avanzamento (maggiore rapporto tra lunghezza ed ampiezza). In entrambi i lati i grani sono, comunque, soggetti a frammentazione il che è dovuto a processi di deformazione plastica ed escursione termica durante la saldatura, sebbene fenomeni di precipitazione di fasi indurenti quali \( \beta'' \) e \( \beta' \) siano stati rivelati solo all’interno della zona opposta a quella di avanzamento. Prove di trazione hanno mostrato una resistenza a snervamento e a rottura leggermente più basse in corrispondenza della zona saldata, rispetto a quelle del materiale base, questo indica valori di duttilità leggermente inferiori in corrispondenza del giunto di saldatura.
INTRODUCTION

The major advantages of friction stir welding (FSW) in aluminum alloys, when compared to conventional fusion welds, are primarily the elimination of cracking and evaporative loss of alloying elements. This is due to the solid-state joining and a weld zone with fine worked or recrystallized grain structure generated by stirring and forging during FSW [1]. During welding, temperatures remaining below the melting point, result in a low shrinkage phenomenon and excellent mechanical properties, together with reduction of residual stress within the welded zone [2-13]. Mechanical properties of the FSW joint are quite good and fatigue properties are practically the same as the parent metal [1]. Generally, tensile failures occur well away from the nugget [1,2].

To date, the major application fields of FSW are marine industries (ship sections, hulls, structures), aerospace industry (fuselages, wings, fuel tanks), railway industry (high speed trains, carriages), automotive industry (chassis, wheel rims, space frames, truck bodies) motorcycle frames and other sectors, such as electrical and refrigeration [1].

Due to the combined effect of the tooling, FSW produces five different microstructure zones: the nugget in the centre of the weld where the pin has passed, a shoulder contact zone on the top surface, thermo-mechanically affected zones (TMAZ) that are immediately on each side of the nugget, heat affected zones (HAZ) adjacent to the TMAZ that experiences a thermal cycle but no a mechanical shearing, and the unaffected parent material [4,5,7,14-18]. FSW produces an asymmetric microstructure in which the advancing and retreating sides experience different strain levels and thermal cycles [3,16]. Material on the advancing side (rotation opposed to plate motion) experiences more shear than the retreating does [3]. The closer the material to the tool, the higher is the deformation and temperature gradients to which it is subjected. This implies an effective deformation rate higher in the advancing side than in the retreating, resulting in different stress, flow and temperature cycles [16]. Moreover, almost all the metal form in front of the tools is sheared to the retreating side creating a much broader flow band than on the advancing. In addition to the grain and substructure evolution during the severe thermo-mechanical conditions imposed by FSW, the various thermal cycles in the different weld zones induce different precipitate distributions within each zone [12-14].

The present paper reports a microstructure and mechanical study on an AA6056 T6, FSW plate. The microstructure inspections focused on the asymmetric aspects within the TMAZ between the advancing and retreating side of the weld, especially in terms of grain morphology and secondary particle alteration. Tensile and hardness tests were carried out across the weld nugget and compared to the parent material.

EXPERIMENTAL METHODS

FSW has been carried out by Alenia Aeronautica (Torino) as a bead-on-plate weld. The welding was performed under pin load control at a rotational speed of 1800 rpm and an angle of 3º respect to the plate axis; plate translational speed was of 15 mm/s. The tool consisted of a 18 mm diameter flat, scrolled shoulder and truncated cone pin, whose tip size was of 1.8 mm. The 10-mm thick welded plate was AA6056 (1.1 Mg, 0.65 Si, 0.23 Cr, 0.15 Mn, Al bal., in wt.%) in the T6 condition.

Weld cross section and middle-transverse section of the weld were ground, polished and anodized (a solution of 4% fluoro-boridic acid in ethanol at 25V for 120s) for polarized optical microscopy (POM) inspections. The grain structure of the different welded zones (nugget, TMAZ, HAZ) and the parent material was characterized by POM. Statistical evaluation of grain mean size and aspect ratio was carried out by image analysis software.

Sheets for transmission electron microscopy (TEM), were extracted from the middle-longitudinal section along the welded zone. Various weld locations were selected in the attempt to follow the microstructure changes, from the parent material, to the nugget on both advancing and retreating side. Care was taken for the correspondence of locations to the hardness profile along the weld. Sheets were ground to 200 mm thickness, chemically polished to 70 mm, punched as 3-mm discs. A Tenupol-5 twin jet electro-polisher was used to produce electron transparent sections using a solution of 30% nitric acid in methanol at -35ºC and 18V. TEM inspections were carried out on a Philips CM200 operating at 200kV and equipped with a double tilt stage. Boundary misorientation has been measured by using the Kikuchi pattern method [19].

Vickers micro-hardness was carried out with a 100gf load and 15s dwell time; data were acquired along the longitudinal section at the middle thickness of the weld and each data point averages some 5 different measurements, the distance apart being 0.25 mm. Tensile tests were performed on a 250 kN servo-hydraulic machine operated in ram displacement control at room temperature and with a nominal strain rate of 2×10⁻¹s⁻¹. A computer-based data acquisition system was used to control machine operation and data recording. Two stets of specimens were extracted, one from across the welded region, another far away from the welded zone, within the parent material. Both sets of test-pieces were extracted along the transverse direction, in order to avoid textural inhomogeneities.
RESULTS

Fig. 1 shows panoramic views of the cross section and transversal middle-thickness section of the welded plate. Material moves upwards and the tool spins counter clockwise, thus the advancing side is on the left and retreating on the right. There are seven different regions across the weld, three on each side of the nugget (parent material, HAZ, TMAZ). From Fig. 1 (b), (d) it is clear that there is

Fig. 1: Coordinate system scheme (a); polarized optical micrographs (POM) of the cross section (b), and longitudinal section at middle thickness (d) across the friction stir weld. The major zones (HAZ, TMAZ, nugget) of which the weld is composed are labeled. A micro-hardness profile, of same scale as (a), is reported in (c), showing a double, but not symmetrical, minimum in the TMAZ.
not a smooth narrow interface between nugget and TMAZ on either side. This indicates material is bulged from advancing side and both swept and bulged from retreating side to fill in behind the pin. In Fig. 2 (a) and (b), POM documents the different microstructural zones from the parent material to the nugget zone, on the advancing and retreating sides. In both TMAZ, the grains are bent upwards indicating additional displacement, relative to parent plates, caused by the advancing pin somewhat similar to the flow around a punch, or hardness indenter. On the advancing side, grains in the TMAZ have bulged only a little into the region behind the pin, leaving a sharp interface between the TMAZ and nugget. On the retreating side, such flow is greater because the rotating tool shears metal from before it to that side. Moreover, the additional sweeping of material into the region behind the pin results in a very irregular interface between elongated grains and the equiaxed nugget. The TMAZ-nugget interface zone is very wide, having alternating layers of elongated grains with fine cells and of crystallites with larger diameter. The micro-hardness profile, along the middle section of the weld is reported in Fig. 1 (c). The frictional heat results in a minimum of the hardness because of the growth and coarsening of the T6 strengthening precipitates.

TEM micrographs of the nugget, at two different magnifications in (Fig. 3 (a,b)), exhibit the structure where the pin has passed. The fine equiaxed crystallite structure with average size of 12 mm in diameter is finer by a factor of roughly 40 than the 450 mm grains of the parent material. Kikuchi diffraction pattern analyses, performed by TEM, revealed these boundaries to be mostly high-angle, about 80% HAB. On both sides of the nugget in the TMAZ cores (Fig. 3 (c,f)), grains are elongated and bent similarly, both being 350 mm in equivalent diameter with an aspect ratio rising from 3 to 11 on approaching the nugget zone. Crystallites have grown by dynamic recovery (DRV) because of the higher temperature or lower strain rate than in the TMAZ core, with some static recovery (SRV) later. The alternating layers of crystallites and elongated grains with cells indicate there has been no discontinuous recrystallization. Moreover, there was no evidence of growth by boundary migration from lower density nugget layers to more deformed cell layers. In the region close to the pin, i.e. the sharp interface zone between the TMAZ and nugget, the grain structure appears as elongated bands of fine crystallites which are quite similar to the ones observed within the nugget region. The core of advancing side TMAZ, characterized by having a large fraction of high-angle boundaries (HABs), is the result of both shear strain and temperature.
increasing as the material approaches the pin. In comparison to the advancing side, the retreating side TMAZ is wider due to the tool rotation causing the material ahead of the pin to displace to the right-hand side. This process is accompanied by a continuous decrease in the strain of the rotated material, with distance from the rotating pin. Furthermore, the TMAZ band closest to the nugget appears as a mixture of fine crystallites, approaching the cell dimension in the elongated grains, as already noted for the advancing side TMAZ-nugget interface. From TMAZ-nugget interface, Fig. 4 shows the intersection of a

![Fig. 3: Transmission electron micrographs (TEM) showing the microstructure of the nugget at two different magnifications, 3800x (a), 6600x (b), and TMAZ in the advancing side (c,d), and retreating side (e,f) at different locations.](image-url)
low-angle boundary (LAB) and a strain induced high-angle boundary (HAB) that would not be possible with an original grain boundary. Diffuse dislocation interactions with fine particles and Frank network dislocations inside subgrains, are also observed at the TMAZ-nugget interface, on the retreating side. They are believed to form during the thermal quench from the high welding temperature to the room temperature.

An interesting aspect is the asymmetric precipitation sequence occurring on the retreating side with respect to the advancing side. The advancing side has solely large precipitates (> 400-500 nm) that might come from overaging during the passage of the pin (Fig. 3 (c)). Beside coarse particles, the retreating side is characterized by fine rounded particles (less than 300 nm) and finer needle-like precipitates (less than 200 nm long with one-tenth the width) that are G.P. zones and b’ (Fig. 3 (f)). These fine particles result in slightly higher hardness values in the retreating TMAZ-nugget interface (Fig. 1). In both sides the dislocation density within the grains is rather high. Quite a number of dislocations pinned by secondary phase particles were observed, especially in the retreating side. The grain structure of the HAZ is similar to that of the parent material. The mean grain size was 470 mm and 390 mm in the advancing and retreating side, respectively, with a common aspect ration of about 2.5 against 2.2 of the parent material. This slight increase in aspect ratio is quite little compared to change in the TMAZ core and confirms that the HAZ has deformed very slightly at the most.

Fig. 5 shows the tensile curves carried out on specimens from across the welding zone and from the parent material. Compared to the base alloy, the ductility of the joint is near 40%, but the ultimate strength is about 90% (280 MPa relative to 330 MPa) and the yield strength is about 66% (160 to 230 MPa).

**DISCUSSION**

The large scale pattern of grain flow is similar to that observed in other studies, being in agreement with some reports [3,6,7,10-15,20,21], and disagreement with others [5,22-27]. The flow in the retreating side seems to be much greater and broader than on the advancing side. If the material flow would have been induced by a non-rotating indenter, there would be equal flow on both sides, somewhat like the cross-section of a tubular impact extrusion; here grains are bent upwards from the parent plates that provide the die walls. The rotation of the tool induces a narrow shear...
strain gradient (Figs. 1, 2(a)), and thermal spike generated on the advancing side. The tool rotation carries much material in front of it, towards the retreating side, producing a higher flow rate (Figs. 1, 2). The heat in that transported metal helps to develop a much broader temperature gradient that facilitates the more massive plastic deformation. Despite difference in the plastic zone width, the degree of strain in the TMAZ cores is roughly the same based on the change in aspect ratio of the grains. With allowance for differences in parent metal and welding conditions, the present anisotropic microstructures are consistent with microscopic, thermal and flow studies and simulations reported for several aluminum alloys and stainless steels [3,5,6,8,10,12,13,16-18,21-26].

Cho et al. [3] found that the effective stress was highest in the stirred zone and higher on the advancing side than on the retreating side; the mean stress was compressive ahead of the tool and tensile behind the tool (Fig. 1). Reynolds and co-workers [28,29] documented for AA2024 a banded structure within the TMAZ and mixed banded and almost equiaxed crystallites at the TMAZ-nugget interface, in agreement with the present microstructures (Fig. 2) and changes in mechanical strength (Fig. 5).

The sharp drop, on the advancing side, in the hardness profile (Fig. 1 (c)), supports a narrow temperature spike as suggested above. The very low hardness zone is related to the disappearance of G.P. zones and formation of overaged precipitates (Fig. 3 (c)). The slightly higher hardness in the retreating TMAZ is due to a broader temperature gradient resulting in less dissolution and overaging (Fig. 3 (f)). The higher hardness in the nugget is related to complete solution and new precipitation in this slowest cooling zone; this is counteracted by the additional softening relative to the TMAZ due to substructure coarsening. As regard to the different precipitation sequences [5,12,16-18,27-29], Yang et al. [5] measured the peak temperatures for three different FSW tool speeds in two 2000 series alloys. In their paper, they reported a lower particle fraction, size and density in the advancing side TMAZ in similarity to behavior detected in our inspections. A study on a AA2024 by Norman et al. [5] reported temperature peaks of 450-500°C and 450-350°C for the nugget and TMAZ, respectively; on this basis they proposed only partial dissolution in the TMAZ and complete solution of particles within the nugget, possibly followed by precipitation during cooling [5,30-33].

The nugget is formed to a limited extent by the material from each side of the pin flowing into the region it is vacating as a result of lateral constraints by the parent plates. The region is mainly filled by the hottest metal in the broad retreating side flow zone, being swept in by the pin rotation. This is confirmed by the wide feathery interface of mixed elongated and equiaxed layers (Fig. 2 (b)). The strain rate declines gradually to zero as the pin moves further away, before cooling takes place. The substructure increases in dimensions by dynamic recovery (DRV), as confirmed by the 12 mm crystallite size compared to 2 mm subgrains in the TMAZ (Fig. 3 (d),(e)). Some static recovery (SRV) occurs after all straining has stopped, but it is much less effective than DRV.

The TMAZ can best be understood from the theory of geometric dynamic recrystallization (gDRX) [25,26,32-37]. During steady-state straining (in narrow bands) at constant temperature and strain rate, the subgrains remain equiaxed as the grains elongate, developing serrated boundaries [25,26]. Due to the greater grain boundary area, the density of HAB increases and many subgrains have several high-angle facets [26,32]. These small cellular regions are renamed crystallites, being intermediate between subgrains and grains. In the core of the TMAZ, the grains elongate but never become thinner than about two subgrain diameters [36,37]. Due to the mobility of grain boundaries as they absorb intersecting sub-boundaries, serrations in neighboring grain boundaries impinge. This pinches off the grain making it shorter but, at the same time, making the neighbors thicker. In POM, the elongated regions from an initial grain are still evident due to similar texture. As strain rate decreases in the nugget region, the crystallites grow larger by rearrangement of the sub-boundaries as occurs during creep tests when the strain rate is changed [38,39]. The time at high temperature after straining is too short for discontinuous static recrystallization (dSRX) to take place even at interfaces between the equiaxed and elongated regions with greater sub-structure density.

In similarity to this, Su et al. [11] have reported in AA7050 the transformation of elongated grains with 1-2 um cells in the TMAZ into 1-4 um equiaxed structure with many HAB in the nugget. They proposed that the growth and the creation of HAB occurred by some cell walls absorbing dislocations; they failed to consider rearrangement of the GB from the TMAZ through the gDRX mechanism. In one part of the nugget of AA2024, Norman et al. [5] observed 6.3 um crystallites (63% HAB) coming from about 3.1 um cells (73% HAB) in the TMAZ in similarity to the present; however in the nugget center, they suggest that the 2.4 um grains (85% HAB) were produced by discontinuous DRX from the TMAZ.

Prangnell and colleagues [40] have shown that the crystallite size in the nugget is consistent with structures developed in torsion to the same strain at equivalent temperatures and strain rates. Jata and Semiati [41] found nugget crystallite size consistent with torsion subgrains in an Al-Li alloy [42]. Because of the refined crystallite structure developed and despite the diminished precipitation hardening, the FSW weld quality is far higher than conventional fusion welding as confirmed by the results of the mechanical tests.
SUMMARY

The microstructure of a FSW AA6056-T6 plate was investigated by POM and TEM. The grain structure can be divided into several major regions: i) fine equiaxed crystallites in the nugget at the weld centre, ii) highly elongated grains with very small cells in the broad retreating side TMAZ and in the narrow advancing side, and iii) slightly elongated coarse grains in the HAZ and the parent material. Strain rate and temperature gradients are much steeper in the advancing side than in the retreating. The nugget is defined by large crystallites formed by sub-boundary rearrangement from fine subgrains swept in from the TMAZ due to high temperature and low strain rate. Precipitation distribution was quite different from the needle-like T6 particles in the parent material with some coarsening in the HAZ. Solely in the retreating side TMAZ a bimodal particle population was detected being composed of remaining aged precipitates together with coarse incoherent b-Mg2Si particles. The advancing side TMAZ shows a narrow zone of overaged precipitates only. The hardness profile is lowest in the advancing side TMAZ, slightly higher in the retreating side TMAZ and higher in the nugget where greater dissolution was accompanied by re-precipitation. Mechanical properties of the welded region were quite good, the ultimate being 90% and yield strength 66% of the values in the base alloy.

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REFERENCES

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