

Manuscript Number: JMAD-D-15-01798R3

Title: Microstructure and mechanical properties of friction stir welded joints made from ultrafine grained aluminium 1050

Article Type: Research Paper

Keywords: Aluminium, Incremental Equal Channel Angular Pressing, Friction stir welding, Microstructure, Mechanical properties

Corresponding Author: Mrs. Marta Lipinska, M.Sc.

Corresponding Author's Institution: Warsaw University of Technology

First Author: Marta Lipinska, M.Sc.

Order of Authors: Marta Lipinska, M.Sc.; Lech Olejnik; Adam Pietras; Andrzej Rosochowski; Piotr Bazarnik; Jacek Goliński; Tomasz Brynk; Małgorzata Lewandowska

Abstract: In order to obtain ultrafine grained structure, commercially pure aluminium (Al 1050) plates were subjected up to 8 passes of Incremental Equal Channel Angular Pressing (IECAP) following route C. Plates in different stages of IECAP were joined using Friction Stir Welding (FSW). All welded samples were investigated to determine their mechanical properties and structure evolution in the joint zone. The joining process reduced mechanical strength of material in the nugget zone, which was explained by the grain growth resulting from temperature rise during FSW. Nevertheless, the obtained results are promising in comparison to other methods of joining aluminium.

Warsaw, 24 August 2015

Dear Editor,

We are pleased to submit the revised version of the manuscript Ref. No. JMAD-D-15-01798R2 entitled „Microstructure and mechanical properties of friction stir welded joints made from ultrafine grained aluminium 1050” by M. Lipinska, L. Olejnik, A. Pietras, A. Rosochowski, P. Bazarnik, J. Golinski, T. Brynk and M. Lewandowska, which has been corrected according to the editorial comments. We have found the review instructive and it helped us to improve quality of the text to the level, which might hopefully meet the standards of your prestigious journal.

Below you can find the answers to editorial comments.

(1) Flow chart is not allowed in the graphic abstract. It has to be a single figure with no caption or title. Explanatory words have to be embedded into the figure.

Answer: Graphic abstract has been changed in accordance with editorial recommendation.

(2) Broken sentences are generally discouraged in the highlight. i.e. Plates were joined using friction stir welding -> so what? what is author's conclusion and novelty of that? Differences in the microstructure and mechanical properties of welds were observed -> then what are the differences?!?

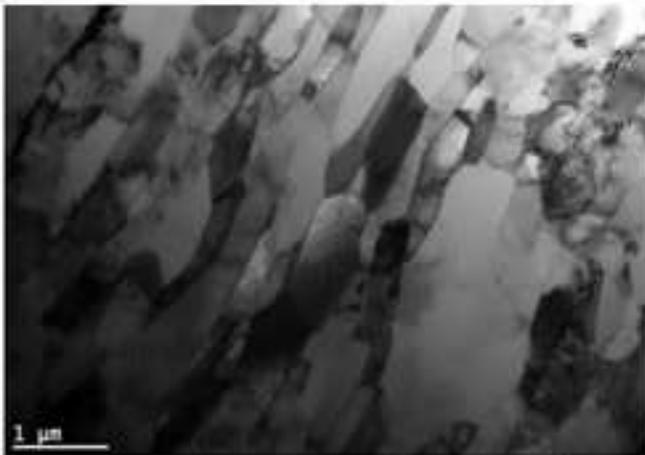
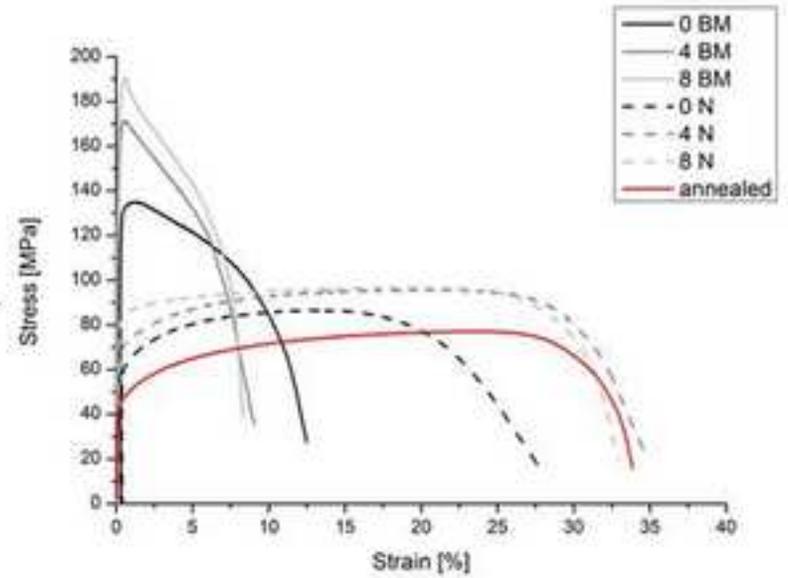
Answer: The highlights have been rewritten. Broken sentences are no longer present.

I sincerely apologize for such mistakes.

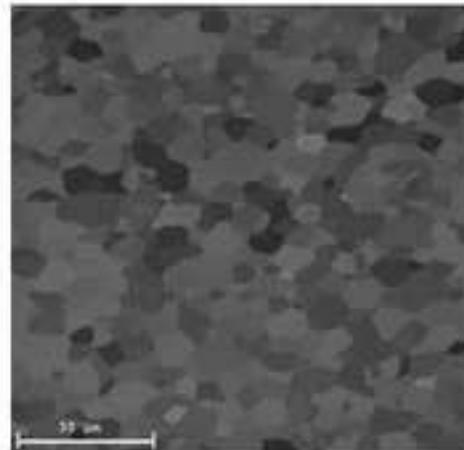
Sincerely yours,

Marta Lipinska
on behalf of all the authors

Good quality joint obtained by FSW of ultrafine grained plates produced by incremental ECAP, route C



UFG microstructure in a base material after I-ECAP



Micro grained microstructure in a nugget zone after FSW

Highlights

- Ultrafine grained Al 1050 plates with enhanced mechanical properties were produced using incremental ECAP, route C.
- The plates were successfully joined using friction stir welding and good quality joints were obtained.
- The welding process caused grain growth in stir zone, however, the mechanical strength of welds is higher than that of annealed Al 1050.

Microstructure and mechanical properties of friction stir welded joints made from ultrafine grained aluminium 1050

Marta Lipińska^{1*}, Lech Olejnik², Adam Pietras³, Andrzej Rosochowski⁴, Piotr Bazarnik¹, Jacek Goliński², Tomasz Brynk¹ and Małgorzata Lewandowska¹

¹ Faculty of Materials Science and Engineering, Warsaw University of Technology, Woloska 141, 02 507 Warsaw, Poland

² Institute of Manufacturing Processes, Warsaw University of Technology, Narbutta 85, 02-524 Warsaw, Poland

³ Department of Friction and Resistance Welding and Environmental Engineering, Institute of Welding, Czesława 16/18, 44-100 Gliwice, Poland

⁴ Design, Manufacture and Engineering Management, University of Strathclyde, 75 Montrose Street, Glasgow G1 1XJ, United Kingdom

Corresponding author. Tel.: +48 222348740, e-mail address: mlipinska.inmat@gmail.com

Highlights

- Ultrafine grained Al 1050 plates with enhanced mechanical properties were produced using incremental ECAP, route C.
- The plates were successfully joined using friction stir welding and good quality joints were obtained.
- The welding process caused grain growth in stir zone, however, the mechanical strength of welds is higher than that of annealed Al 1050.

Abstract

In order to obtain ultrafine grained structure, commercially pure aluminium (Al 1050) plates were subjected up to 8 passes of Incremental Equal Channel Angular Pressing (IECAP) following route C. Plates in different stages of IECAP were joined using Friction Stir Welding (FSW). All welded samples were investigated to determine their mechanical properties and structure evolution in the joint zone. The joining process reduced mechanical strength of material in the nugget zone, which was explained by the grain growth resulting from temperature rise during FSW. Nevertheless, the obtained results are promising in comparison to other methods of joining aluminium.

Keywords

Incremental Equal Channel Angular Pressing, Friction stir welding, Aluminium, Microstructure, Mechanical properties

1. Introduction

Nowadays, materials with ultrafine grained (UFG) structure are becoming more and more attractive for many industrial applications. The major advantage of these materials is their enhanced mechanical strength at ambient temperature due to increased amount of grain boundaries acting as obstacles for moving dislocations [1]. At elevated temperatures, the creep resistance decreases as documented for pure aluminium [2,3]. However, creep resistance is strongly influenced by the

character of grain boundaries, i.e. creep ability increases for higher amount of high angle grain boundaries. The creep resistance can be even improved for the grain structure containing the majority of low-angle grain boundaries.

The extraordinary mechanical strength of UFG metals stimulated the development of their production methods. Today, UFG metals can be efficiently produced by different variants of the severe plastic deformation (SPD) method. However, the major restriction in wide industrial application of UFG metals is the lack of a reliable welding process, which unlike riveting would give material continuity in the joint. There is also a requirement to weld UFG materials without losing their properties governed by the nanoscale structure. In this context, traditional welding processes based on melting, such as brazing or arc welding, are not applicable because they occur at high temperature causing a total change of structure leading to structure discontinuity and considerable decrease in mechanical strength along the joining line. In this work, Friction Stir Welding (FSW) [4] was chosen as an innovative bonding technology to weld aluminium. FSW takes place in the solid state without reaching the melting temperature of the base material [5]. Stable connection is obtained by mixing the friction-heated, plasticized and deformed metal along the contact line of welded elements. It can be performed by moving a rotating tool (a pin with a shoulder) along a joining line. The key factor for obtaining a consistent joint is large plastic deformation at elevated temperature. It results in bringing-up atoms to a distance which allows creation of a metallic bond. Accompanied by increased density of lattice defects, the final microstructure in different joint areas is mainly dependent on dynamic recrystallization and/or recovery [6] what is common for materials such as aluminium, which is characterized by high stacking fault energy.

Compared to other joining techniques, FSW exhibits a number of advantages. The rise in temperature cannot be higher than a melting point. On account of that, problems related with resolidification, such as porosity, embrittlement and formation of second phase are not present. Additionally, distortion and residual stresses, which are seen in FSW joints, are lower than those observed for other methods, based on melting of the base material and a filler [5]. Parameters of the FSW process are crucial for quality and properties of obtained joints. For example, many papers were devoted to shoulder diameter [7,8], which appears to be one of the most influential factors. Other parameters such as pin profile [9,10], tool travel and rotation speeds [11,12], depth of pin's penetration were investigated too. Structural features, e.g. initial grain size, were also taken into consideration [13,14].

Joining UFG aluminium and its alloys was also a subject of numerous investigations. Plates with refined structure, obtained during accumulated roll bonding (ARB) [14] or constrained groove pressing (CGP) [15,16] were joined using FSW. In each case, authors attempted maximizing mechanical properties and preventing grain growth in the joint zone. Relying on the improved properties in coarse grained materials, materials with refined structure were taken into similar investigation. When the initial material is annealed and characterized by coarse grained structure, subsequent FSW leads to the creation of new, equiaxial grains with much smaller size than the initial one. As a result the improvement in mechanical properties is observed [13]. In the case of materials with already refined structure, the FSW process leads to grain growth in the joint zone, which seems to be extremely difficult to overcome. As was shown for aluminium 1050 [16], which is the same material as in this study, annealed samples subjected to FSW exhibited a very fine grain structure in the joint, which was formed by dynamic recrystallization. In the same investigation, materials with the initial refined structure revealed an intense reduction of microhardness in the stir zone, which was explained by the grain growth. It was caused by thermal instability resulting from high stored

energy during severe plastic deformation. The samples after ARB were also annealed [13], which enabled achieving the best compromise between grain size and hardness. Unfortunately, joining immediately after ARB caused a decrease in mechanical properties of the stir zone. The conclusion from this work was that regardless of the initial grain size or shape, samples revealed equiaxial grains in the stir zone. Nevertheless, for base materials with UFG structure, there was a grain growth observed in the joints.

The aim of the present work was to join technically pure aluminium plates with ultrafine grained structure produced by Incremental Equal Channel Angular Pressing (IECAP) using FSW process and then determine the relationship between microstructure and mechanical properties in the base materials and joints.

2. Material and methods

The investigated material was technically pure aluminium (99,5 wt%) EN AW-1050A-H24 supplied by the Konin Aluminium plant in the form of 3 mm thick rolled sheets. The exact chemical composition is presented in Table 1.

Table 1. Chemical composition of aluminium 1050

	Si	Fe	Cu	Mn	Mg	Zn	Al
Content [%]	0.25	0.40	0.05	0.05	0.05	0.07	balance

The samples with dimensions of 3x62x105 mm were processed using IECAP [17] to obtain UFG structure. The main idea of this method is to apply plastic strain in a series of small deformation increments [18], which are based on simple shear. Separation of feeding and deformation steps reduces friction during feeding, which enables processing of very long or continuous billets. As conventional ECAP, this method has the ability to pass the ingot via different routes. With square plates, there is a possibility of employing deformation route based on rotation about the normal to the plate (Z axis), which in the case of aluminium led to a homogenous UFG structure [19] with a large number of high angle boundaries [20]. In this study the applied deformation route was based on the rotation of plates about their longitudinal axis by 180° between passes (so called route C). The plates were investigated in an initial state (0 passes), after 4 and 8 passes of IECAP, which corresponds to true strains of 0, 4.6 and 9.2, respectively. To simplify the sample description, they were described as sample '0', '4' and '8', depending on the number of applied passes.

The plates of the same type (i.e. subjected to the same processing path) were joined together using FSW, with the tool shoulder diameter of 17 mm and pin diameter of 5.5 mm. Samples were butt welded along Y plane, which means that joints were made along a longer edge of the rectangular plate. Rotational and linear speeds (Table 2) were different for materials with different number of IECAP passes, which comes from their structural differences.

Table 2. Values of rotational and linear speed used in FSW process

Sample	Rotational speed n [rpm]	Linear speed V _z [mm/min]
0	450	224
4	560	355
8	560	224

To reveal the microstructure of the joints, different techniques were implemented. Anodized metallographic specimens were observed in a light microscope using a polarized light beam. For detailed microstructure investigations, Focused Ion Beam (FIB) and Transmission Electron Microscope (TEM) JEOL JEM 1200 with accelerating voltage of 120 kV were used. Samples for FIB and TEM observations were cut out in the X plane, which is perpendicular to the welding line and also perpendicular to IECAP direction. Thin foils were prepared using a wire saw, ground down to 150 μm and electropolished using Struers Tenupol-5 system operating at a voltage of 35 V at a temperature of 278K. The solution containing ethanol, perchloric acid, butyl glycol and distilled water was used. Microstructure observations were taken together with the Selected Area Electron Diffraction (SAED) patterns with an aperture of effective diameter about 4 μm . Grain size was determined by calculating the equivalent diameter (d_2), which means the diameter of a circle with equal area as the investigated grain [21].

In order to characterize mechanical properties, tensile tests and microhardness measurements were carried out. In the case of tensile test, flat mini samples with a thickness of 0.6 mm (Fig. 1) were used. Tensile samples were perpendicular to the direction of the welding line marked also as the joining direction. Digital Image Correlation (DIC) has been used for accurate strain determination. Stir zone and base material were investigated separately in order to reveal differences between these two areas. For each zone 5 specimens were investigated. Microhardness measurements were carried out in the X plane of samples under a load of 200 g for 15 s. For each sample, 120 measurements were done in 3 parallel lines with 1 mm spacing along width and 0.5 mm along thickness of the butt welded rectangular plate.

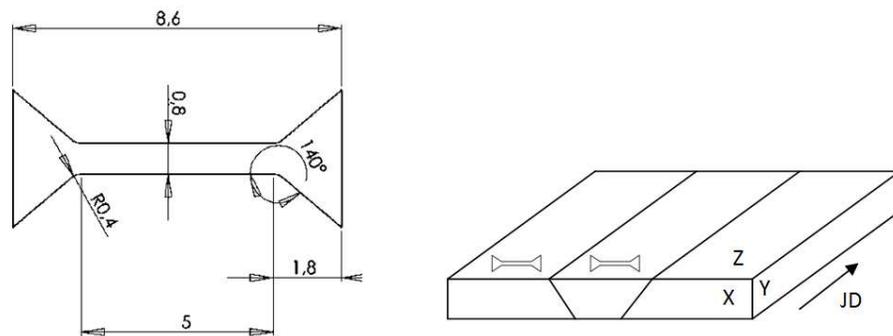


Fig. 1. Schematic illustration of tensile specimens together with their locations in butt welded plate (JD – joining direction along length of the plate)

3. Results

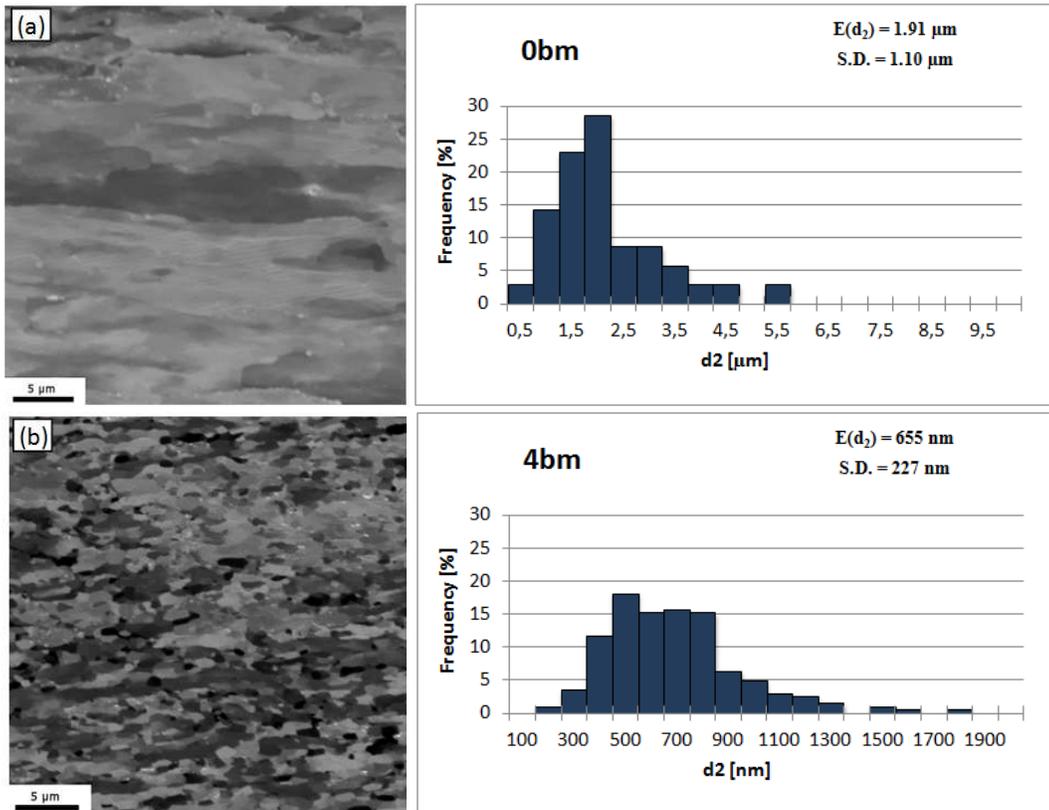
3.1. Microstructure

Microstructures of base materials together with histograms of grain size distribution are presented in Fig. 2. Sample '0' (in initial state) features a typical cold rolled structure with elongated grains of several microns in length. The average grain/subgrain size was about 2 μm . IECAP brought about significant microstructure refinement. After 4 passes, the grain size was reduced to about 655 nm. Further processing (up to 8 passes) only slightly affected the grain size which average value decreased to 630 nm. Histograms look comparable which is an evidence for similar structure of these two materials. The quantitative data regarding the microstructures is summarized in Table 3. Apart from equivalent diameter (d_2) and standard deviation (S.D.), grain elongation factor (d_{max}/d_2) and grain boundary development factor ($p/d_2 \cdot \square$), where p is perimeter) were determined. The sample '0'

exhibits the highest value of grain elongation factor which is a clear evidence for elongated grains. With increasing number of IECAP passes, the grains become more equiaxed, which is reflected in lower value of this factor. Grain boundary development factor is slightly higher for the sample '0' when compared to the samples '4' and '8', which is due to higher elongation of the grains.

Table 3. Quantitative parameters describing the microstructure in base materials (BM) and the nuggets (N): d_2 – average grain diameter, S.D. – standard deviation, d_{max}/d_2 – grain elongation factor, $p/d_2 \cdot \square$ – grain boundary development factor (p – grain perimeter)

	d_2 [μm]	S.D. [μm]	d_{max}/d_2	$p/d_2 \cdot \square$
0 BM	1.91	1.10	1.89	14.20
4 BM	0.655	0.23	1.63	12.92
8 BM	0.630	0.28	1.45	13.07
0 N	4.30	1.49	1.34	11.67
4 N	4.35	1.93	1.36	12.07
8 N	4.56	2.17	1.36	11.95



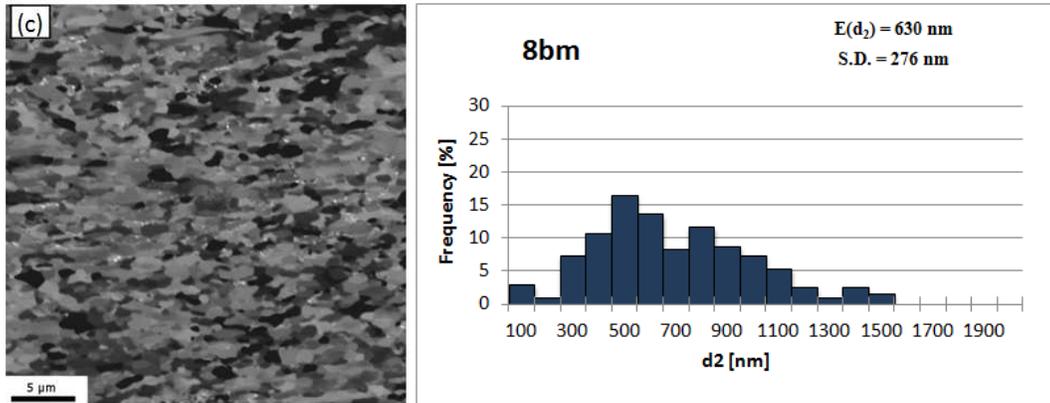
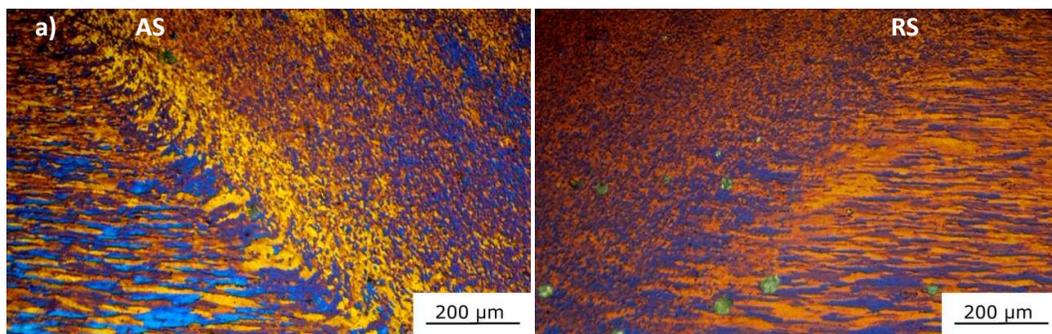


Fig. 2. Representative microstructures of base materials: (a) initial state, (b) after 4 and (c) after 8 passes of IECAP (X plane perpendicular to welding line) together with histograms of grain sizes (d_2)

The images of weld zones for welds are shown in Fig. 3. In each case the picture was taken from both the advancing side (AS) and retreating side (RS) of the weld. These two zones differ in the relative directions of deformation induced by tool rotation and tool advancement. They are parallel but in advancing side they proceed in the same direction while in retreating side in opposite directions. As a consequence, the deformation degree is higher on advancing side. On the images, one can distinguish heat affected zone (HAZ), thermomechanically affected zone (TMAZ), which is a transition zone, and nugget zone (NZ), which is characterized by complete recrystallization. The region outside the weld zone, which is unaffected by heat or deformation is the parent material, called here base material (BM). TMAZ is the most distinct in '0' joint. Also the width of this zone in this joint is the biggest. In '8' joint, TMAZ is very gentle. The transition between HAZ and stir zone is very smooth. On the other hand, in '8' joint the difference in grain colors is the biggest. It indicates the highest differences in grain orientation. BM in initial state reveals very strong morphological texture due to rolling process. As can be seen in Fig. 3, the greater degree of deformation, the structure in base material becomes more equiaxed.



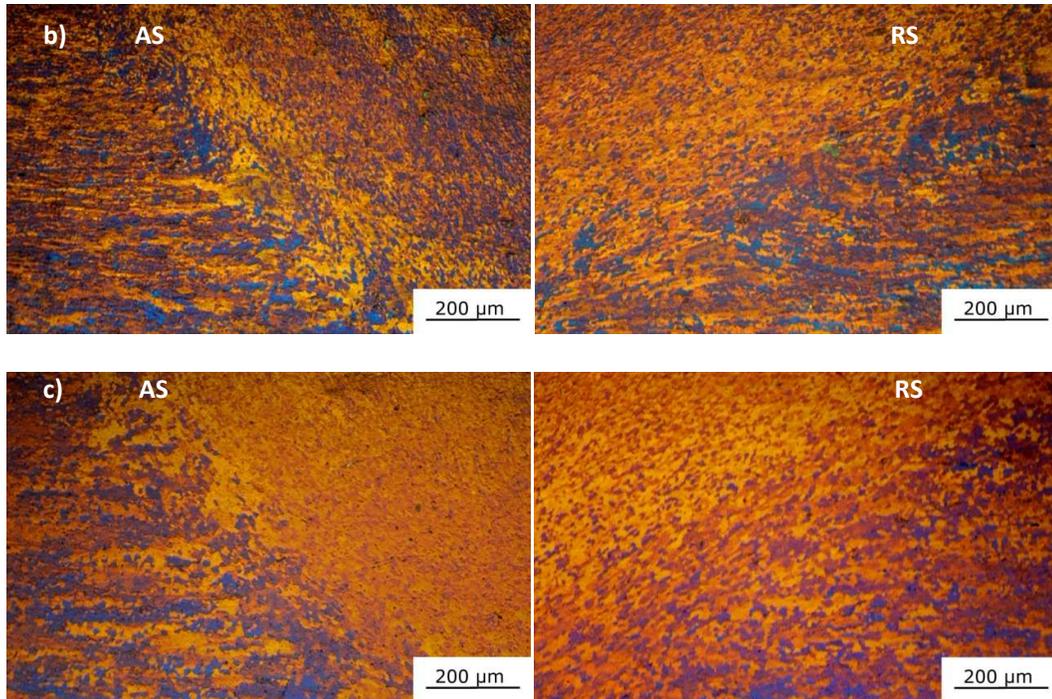
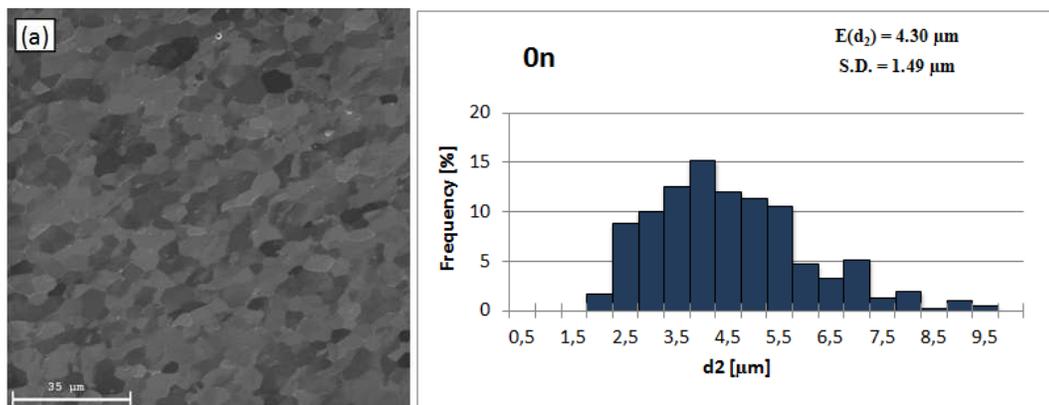


Fig. 3. Weld zones for samples: (a) '0', (b) '4' and (c) '8'

Fig. 4 shows the microstructures of the nuggets for '0', '4' and '8' samples together with the histograms of grain size distribution. Generally, the joint zone exhibits greater grain size than the base material in each case. For all samples, the structure in the nugget is very similar and consists of equiaxial grains with the similar average size of about 4 μm (Table 3), regardless of base material structure, which is consistent with the results reported in [14]. The histograms reveal that the most homogenous grain size distribution in the nugget was obtained for '0' sample (0n). Nuggets of '4' and '8' samples exhibit a greater scatter of grain size, which is reflected in more flat histograms. The grain elongation factor (d_{max}/d_2) for nuggets is at the same level. Its value (≈ 1.36) is typical for equiaxial grains (see data in Table 3). Grain boundary development factor also shows comparable values.



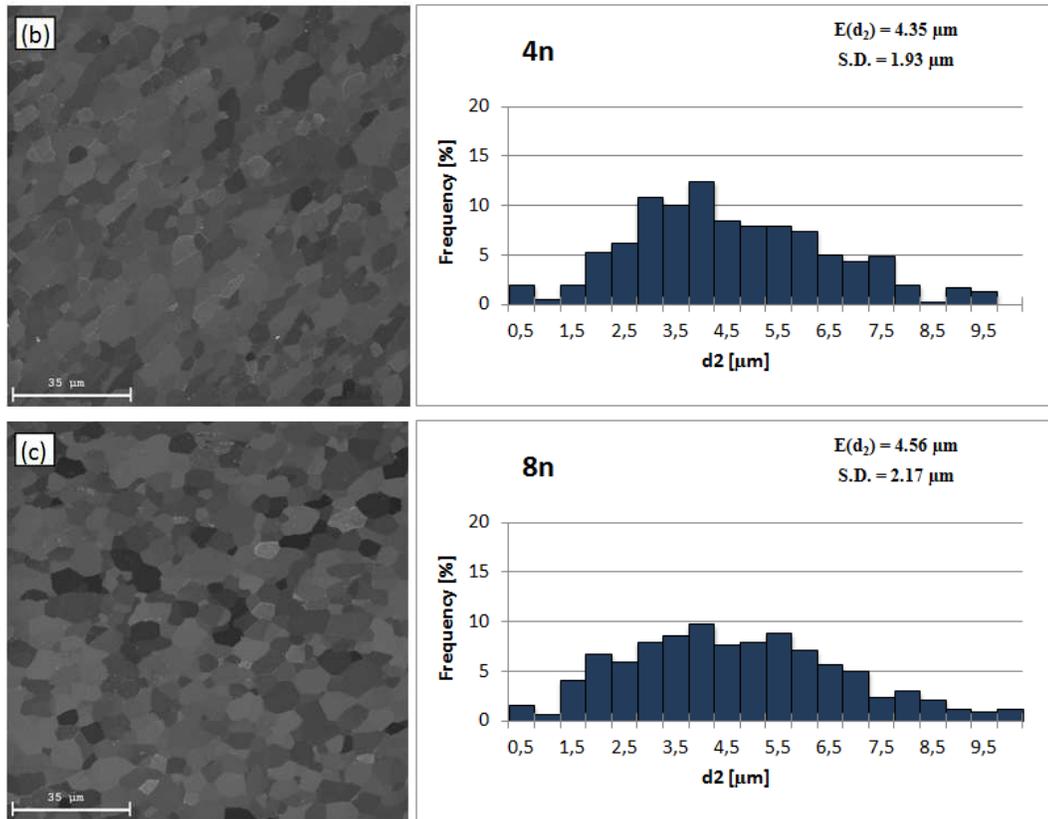


Fig. 4. Representative microstructures of nuggets in: (a) '0', (b) '4' and (c) '8' samples together with grain size (d_2) histograms

3.2. Microhardness

Microhardness distribution across the joints on the transverse cross-section (plane X) is presented in Fig. 5. Average values of microhardness for the base materials are 45, 54 and 58 Hv0.2 for the initial state and after 4 and 8 passes, respectively. This is an evidence of the improvement in mechanical strength caused by IECAP, which can be attributed to grain boundary strengthening effect described quantitatively by the Hall-Petch type equation predicting linear dependence between the strength or hardness and inverse square root of the grain size [22,23].

All the hardness curves are almost symmetrical about the center of the welds. The welding process softens the materials significantly. The hardness values in the weld zone of all the joints dropped to about 32 Hv0.2. Such a behavior is opposite to coarse grained materials, for which the average hardness of the nugget zone is higher than that of the TMAZ and HAZ, as presented in [24]. The minimum was reached at the TMAZ/HAZ boundary. In this study differences in microhardness between nugget zone and TMAZ are barely visible. Therefore, the main emphasis will be put on the relationship between nuggets and HAZ.

For nugget zones, the plateau in microhardness can be observed. However, some microhardness variation can be seen. Nevertheless, all profiles are comparable – the width of nugget zone is similar. From the profiles presented in Fig. 5, the width of HAZ on advancing and retreating sides was determined, as illustrated in Table 4. The width of HAZ was expressed as a percentage of nugget size (which was assumed as a size of shoulder diameter). HAZ was defined as a zone of reduced microhardness compared to base material, but situated beyond the nugget zone. From

these results, one can observe some general trends. The width of retreating side is greater than the advancing side width in all specimens. Furthermore, with increasing plastic deformation of the base material, HAZ also increases, regardless of the weld side.

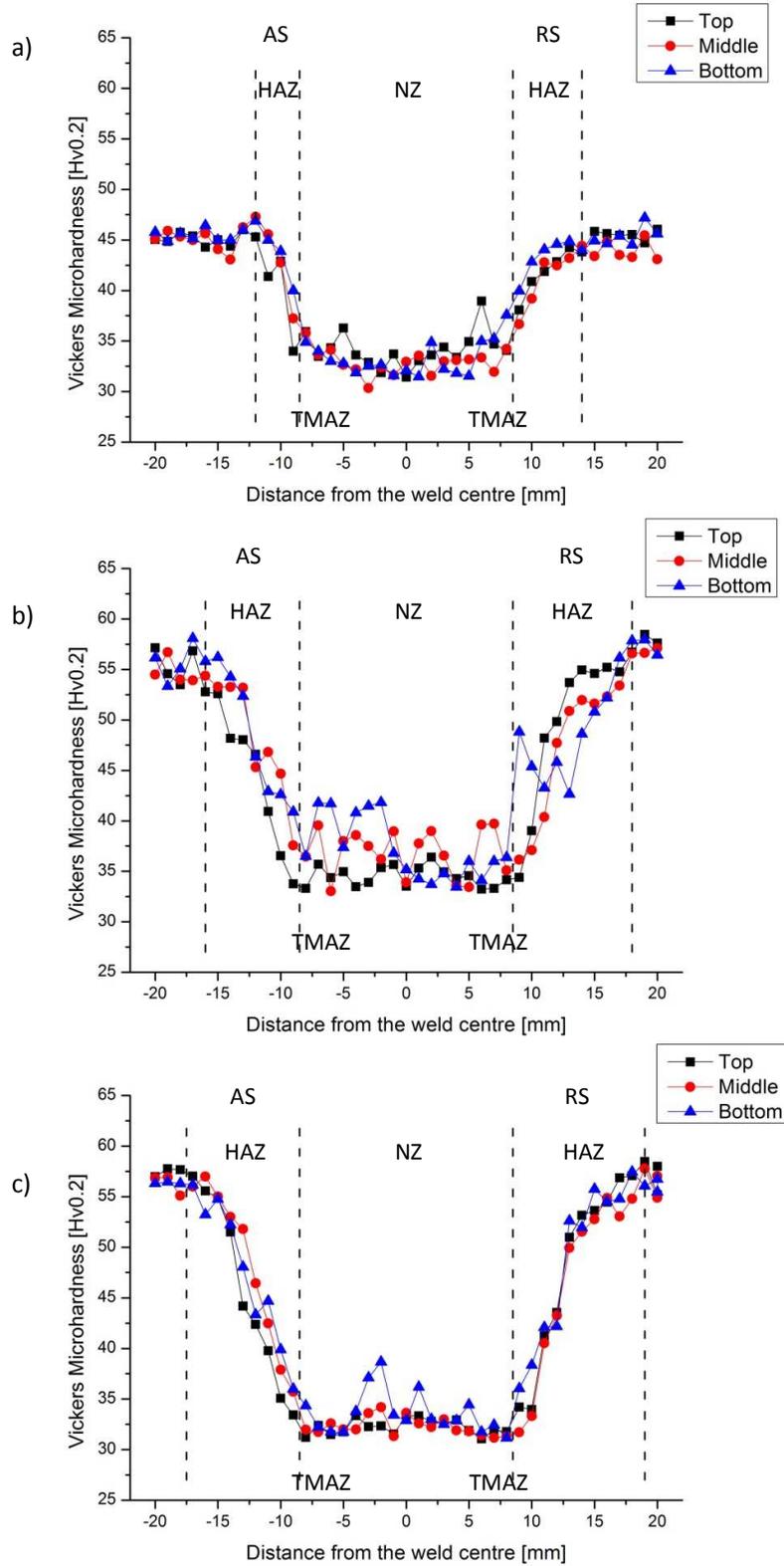


Fig. 5. Microhardness profiles for (a) '0' joint, (b) '4' joint, (c) '8' joint

Table 4. The width of HAZ on AS and RS as a percentage of nugget width

	'0'	'4'	'8'
Advancing side	20%	44%	53%
Retreating side	32%	56%	62%

3.3. Tensile properties

The representative tensile stress-strain curves for micro-specimens cut out from base materials and the nuggets are presented in Fig. 6. Additionally, a curve for the annealed Al 1050 is shown for comparative purposes. In general, the strength of base materials determined as ultimate tensile strength (UTS) and yield strength (YS) is significantly higher than that of nuggets. Values of UTS, YS, elongation to break (A) and uniform elongation (A_g) are summarized in Table 5, as an average value from 5 measurements. It should be noted that the values of YS and UTS for IECAP processed samples (base materials) are very similar (no significant work hardening is observed during tensile test), which implies that UTS and YS can even overlap when the standard deviation is considered. Rapid necking is typical for ultrafine grained materials processed by severe plastic deformation [25,26] and further promoted by the small dimensions of tensile samples used in the present study. The variation in tensile properties of the samples of the same type may be due to the incomplete homogeneity of the tested materials, resulting from the not fully refined structures in materials after plastic deformation. YS for base materials varies from 130 to 176 MPa for sample '0' and '8', respectively. It should be noted that such values of YS are typical for pure aluminium in work hardened and/or ultrafine grained state. The flow stress quickly achieves a maximum value and then decreases gradually, which indicates that necking occurs early and further deformation is accompanied by a deepening neck. As a consequence, the elongation to failure is limited and below 10% for UFG samples. Additionally uniform elongation for these materials is very low and does not exceed 1%. The tensile curves for samples cut from nuggets are different. The values of YS and UTS are significantly lower – below 100 MPa. However, joint '8' exhibits higher YS than the other joints. In addition, the nugget samples have considerable higher values of elongation (26-36%). It should also be noted that the nuggets of SPD processed samples exhibit better combination of strength and ductility, i.e. higher YS and UTS, as well as higher elongation. The curve for annealed sample, marked in red, reveals the lowest values of YS and UTS among all materials. Elongation value is comparable to the results achieved for 4N and 8N. Obtained results are entirely consistent with the aforementioned Hall-Petch's equation for the relationship between grain size and yield stress.

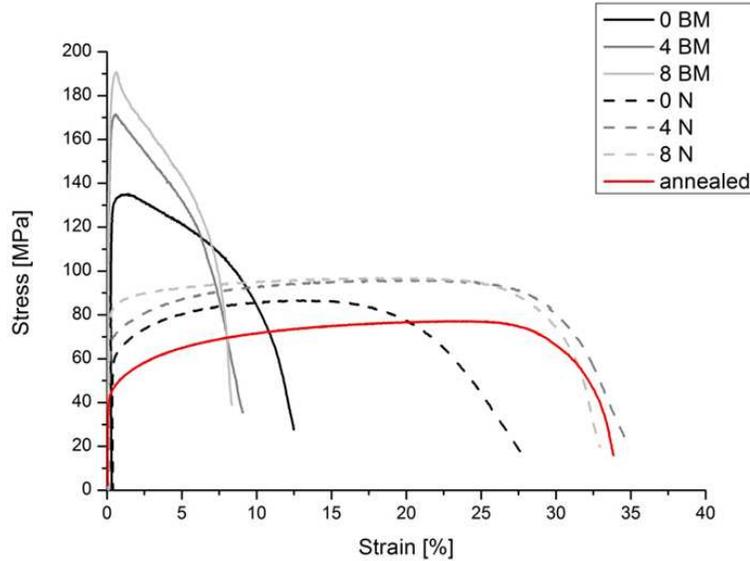


Fig. 6. Stress-strain curves for tensile specimens cut from base material (BM - solid lines) and nugget (N- dashed lines) for 0, 4 and 8 IECAP passes; red line – annealed material

Table 5. Values of Ultimate Tensile Strength (UTS), Yield Strength (YS), elongation to break (A) and uniform elongation (A_g) of base materials (BM) and nuggets (N) for all micro-specimens

Specimen	UTS (MPa)	YS (MPa)	A (%)	A_g (%)
0 BM	135 ± 5	130 ± 5	12.7 ± 0.7	1.5 ± 0.1
4 BM	180 ± 8	172 ± 4	9.7 ± 1.2	0.8 ± 0.2
8 BM	184 ± 7	176 ± 5	7.5 ± 0.8	0.6 ± 0.1
0 N	88 ± 5	63 ± 2	26.2 ± 1.5	14.2 ± 1.2
4 N	94 ± 2	66 ± 4	33.2 ± 3.2	21.4 ± 0.9
8 N	95 ± 2	79 ± 5	36.5 ± 3.7	22.4 ± 1.1
annealed	77 ± 2	45 ± 2	34.7 ± 3.0	25.3 ± 1.2

4. Discussion

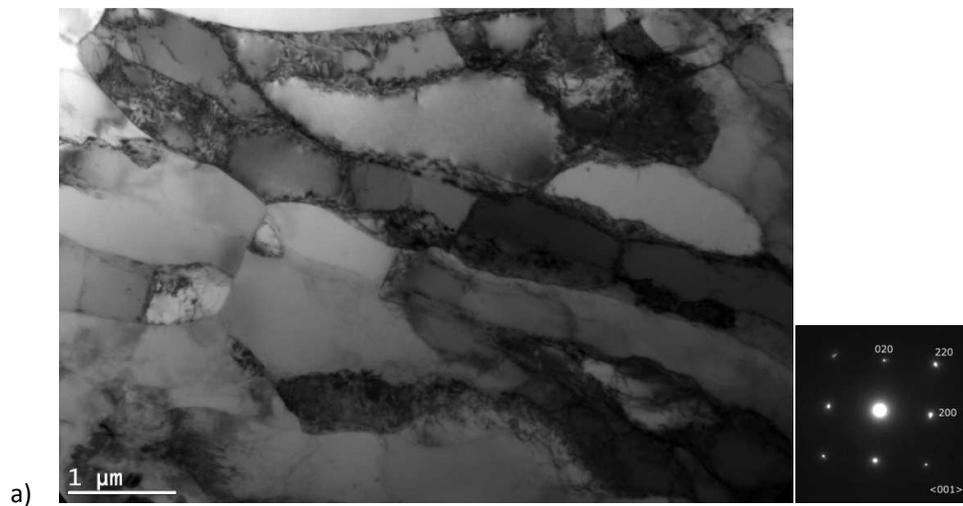
4.1. Mechanism of grain refinement during IECAP processing

Microstructure observations gave a clear evidence of grain size refinement during IECAP processing. However, to determine its mechanism more detailed investigations are needed. Careful inspection using TEM (Fig. 7) revealed more details in the structure development. The sample '0' (Fig. 7 (a)) exhibits a strong morphological texture and dislocation substructure within the grains. Samples after IECAP feature refined grain structure. Inside the grains, one can see free dislocations. The sample after 4 passes exhibits the inhomogeneous structure, which contains both – elongated and equiaxial grains, as shown in Fig. 7 (b). Further deformation did not cause successive reduction of the thickness of elongated grains but resulted in more equiaxial grains (Fig. 7 (c)). It indicates that grain refinement in this stage proceeds through subdivision of elongated grains by transverse boundaries. Such a microstructure evolution is typical for ECAP based processes. It is well established that for small applied strains (usually up to 4 passes), the microstructure consists of elongated deformation bands which at a closer look reveal to be transversely divided to create a dislocation cell structure

[27-30]. The width of the bands decreases with imposed strain until they reach the smallest possible size. Further microstructure evolution relies on an increase in misorientation angles of grain and subgrain boundaries [31,32] leading to a reasonably equiaxial grain structure with the majority of high angle grain boundaries (usually at the level of 70%) [28].

In the context of microstructure evolution, one should also consider the role of sample rotation between consecutive passes. From this point of view, four distinct processing routes have been established: route A, in which the sample is not rotated between passes, route B_A and B_C where the sample is rotated by 90° either in alternative directions or in the same direction and route C, in which the sample is rotated by 180°. It was reported that the most efficient in terms of creation of equiaxial grains is the route B_C due to a large angular range of shearing planes [27]. However, the highest efficiency in terms of high angle grain boundary formation was documented for the route A due to the lack of redundant strain [29]. The route C investigated here is known to be the most practical but the least efficient. This is why the microstructure is not fully equiaxial after 8 passes. The same number of IECAP with rotation about Z axis (instead of X) led to the formation of fully equiaxial microstructure with a very high fraction of high angle grain boundaries of about 80% [20], which was attributed to the activation of different slip systems in consecutive passes (thanks to sample rotation) and the lack of redundant strain which results in early establishment of equiaxial grain structure.

However, the advantage of the route C in IECAP used here for grain size refinement is that both surfaces of the plates are alternatively die and punch sides, which may lead to more homogenous microstructure across the plate's thickness. This phenomenon will be the subject of a separate study.



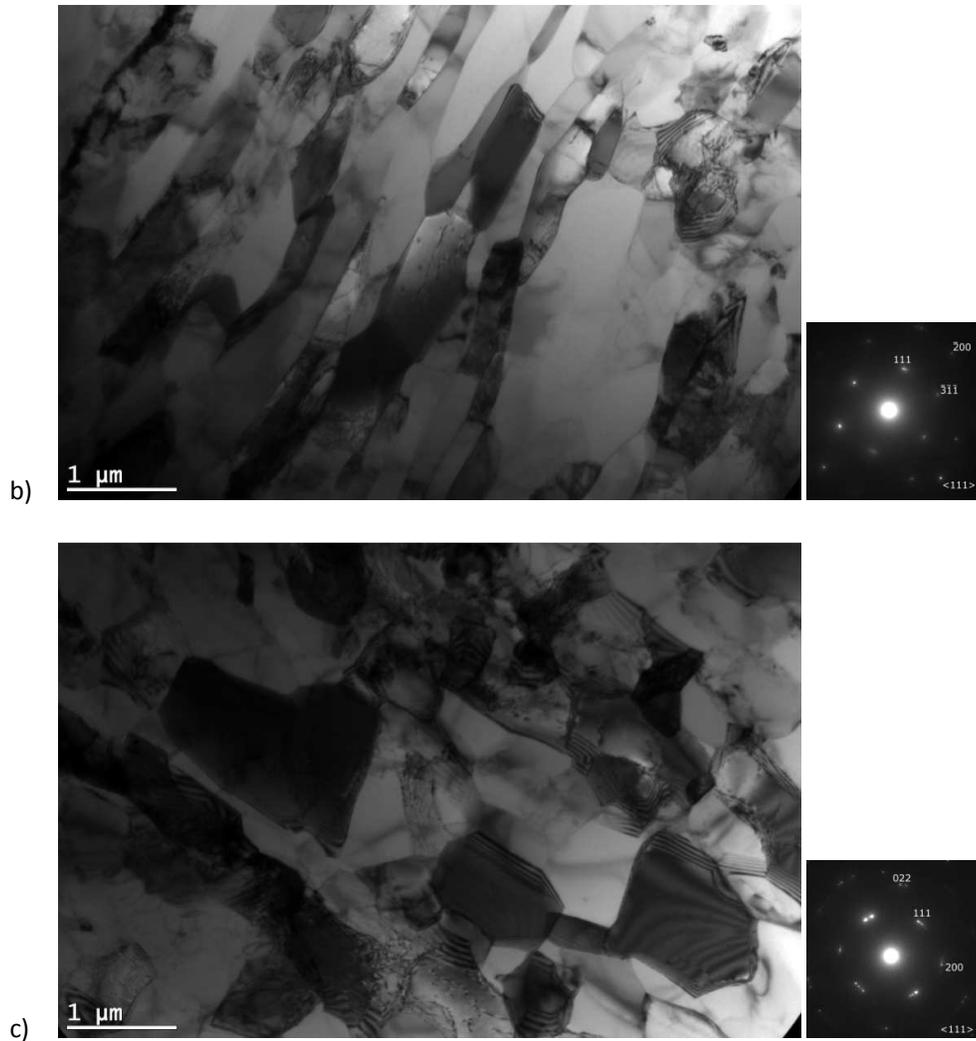


Fig. 7. Representative microstructures of Al 1050 together with SAED patterns: (a) initial state, (b) 4 passes and (c) 8 passes of IECAP (X plane perpendicular to welding line)

4.2. Factors influencing microstructural changes in weld zones

Microstructure observations (see section 3.1) gave a clear evidence of microstructural changes within weld zones. They are caused by the heat generated by friction of the tool shoulder and deformation induced by the tool pin during FSW. TMAZ is characterized by highly deformed and elongated grains. As can be seen in Fig. 3, the interface between zones is different on the advancing and the retreating side. TMAZ on the advancing side is well defined, which is an evidence of the distinct difference in grain sizes between zones. The retreating side is more diffused. This observation is in agreement with other works [9,33] and is explained by the differences in plastic flow state on both sides of a weld during FSW. On AS, the directions of deformation and tool translation are the same, which results in higher shear force and higher plastic strain imposed compared to RS, where the plastic strain is smaller [24].

The grains in nuggets form as a result of dynamic recrystallization during plastic deformation and heating caused by friction between the welding tool and the workpiece [34]. During FSW process, the temperature rise is high enough to induce recrystallization. The peak temperature can

be in the range from 0.6 to even 0.95 T_{melt} , and depends on the material, tool arrangement and process conditions [35]. Also, the shoulder diameter plays an important role [8], i.e. an increase in shoulder diameter increases the peak weld temperature. The maximum predicted temperature is located under the rim of the shoulder. For the shoulder diameter of 40 mm, temperature can rise up to 400 °C, whereas in the case of 20 mm diameter (our study – 17 mm), the peak temperature reached 200°C, which was consistent with predictions.

The temperature rise is also influenced by the properties of joined materials. For high strength materials, one may expect higher heat input. This is one of the reasons for different behavior of coarse grained and deformed materials [13-16]. In former ones, stir zone (SZ) exhibits higher strength and hardness whereas for the latter dynamic recrystallization and SZ softening is observed. In our case, the base materials are all in the deformed state with relatively small grain size varying from 2 to 0.6 μm . They all undergo dynamic recrystallization during FSW, which is also promoted by the high density of defects (mainly grain boundaries). Although the most pronounced grain growth has been observed for sample '8' (as illustrated in Table 3), the final microstructure in the nuggets is very similar in all samples. The average grain size is only slightly higher for sample '8' when compared to sample '0' (4.56 versus 4.30 μm). It is believed that the differences in the microstructure and the properties of based materials are small enough to not influence the final microstructure in the nuggets, which is mainly governed by welding parameters.

During welding, grain coarsening occurs not only in the nugget zone but also in adjacent areas due to heat dissipation. The higher the plastic deformation imposed in the IECAP process, the wider HAZ around the nugget. This may be attributed to different amount of heat generated for softer and stronger materials. Although this difference does not cause changes in the nuggets of investigated joints, it is sufficient to affect HAZ. In addition, samples '4' and '8' feature smaller grain size and are more prone to grain growth. These two factors bring about different width of HAZ in samples subjected to different IECAP processing.

Microhardness measurements also revealed differences between advancing and retreating sides. For each sample, the HAZ is greater by about ten percentage points in retreating side. It means that in advancing side the heat input is smaller. It has to be correlated with material flow and heat generated during FSW, which leads to a larger area of the deformed grains on retreating side. This is in agreement with the microstructural investigation, where transition zone in retreating side was more diffused and smoother. Advancing side, on the other hand, was steeper.

4.3. Comparison to other joining techniques

Generally, FSW welding of aluminium plates after SPD causes a relatively high reduction in hardness in the stir zone, which is consistent with results obtained in [13,16]. In this zone, grain coarsening is expected to occur due to temperature rise during FSW. Contrary to previous reports showing enhanced mechanical strength after FSW [8,13], the stir zone in '0' sample exhibits lower values of microhardness than the base material. However, it should be noted that '0' sample in our case was cold rolled and thus work hardened (H24 hardening state) whereas in the cited reports the joined materials had coarse grained structure after annealing. The possibility of improving mechanical properties of the joint zone is a significant advantage of the FSW method. However, in the case of material with UFG structure, it is more challenging because of the unstable nature of the UFG material and temperature rise during FSW.

However, it should be noted that although grain growth occurred in the welded zone of UFG aluminium, the grain size (about 4 μm) is relatively small and resulting mechanical strength ($YS=80$

MPa) relatively high as for technically pure aluminium. It should be noted that annealed Al 1050 exhibits lower value of the yield strength (45 MPa) than the nugget samples, with similar elongation. FSW process decreased enhanced mechanical strength resulting from IECAP processing, but the strength in nuggets is still improved compared to commercially available annealed material.

The mechanical properties of the FSW joints are also higher than compared to other joining techniques. For instance, a decrease in YS between an Al-Mg-Sc alloy base material and joint is 20% in plates joined by FSW, and 50% between BM and TIG joint [36]. In case of Al 6xxx [37], yield strength is slightly smaller for FSW joints than for MIG joints, but FSW specimens present higher values of rupture stress and elongation. Also detailed hardness examination revealed lower values in the MIG welded specimens. It can be thus concluded that FSW process is one of the most promising methods of joining materials with UFG structure. Therefore, it is worth investigating solid bonding by FSW using different UFG aluminium alloys.

5. Conclusions

Technically pure aluminium samples were subjected to severe plastic deformation by Incremental ECAP and then joined by FSW. The following conclusions based on the present study can be formulated.

1. The plates subjected to IECAP revealed the refined microstructure with average grain size of about 600 nm and improved mechanical strength and microhardness.
2. The ultrafine grained structure obtained by severe plastic deformation is unstable because of temperature rise during FSW.
3. Microstructure in the nugget zone for all joints consists of equiaxial grains with average value of 4.30-4.56 microns.
4. For all materials significant reduction in the microhardness of stir zone had occurred.
5. Despite the deterioration of mechanical properties and grain growth in joints compared to hardened base material, good quality butt joints were produced using FSW for UFG aluminium plates.
6. FSW is one of the most attractive methods of joining UFG aluminium, especially in comparison to other techniques such as TIG or MIG.

Acknowledgement

This work was financed by National Centre for Research and Development (Contract no. WPN/6/2013).

References:

- [1] Meyers MA, Mishra A, Benson DJ. Mechanical Properties of Nanostructured Materials, *Progress in Material Science* 2006;51;427-556.
- [2] Sklenicka V, Dvorak J, Svoboda M. Creep in ultrafine grained aluminium, *Materials Science and Engineering A* 2004;387-389;696-701.
- [3] Saxl I, Sklenicka V, Ilucova L, Svoboda M, Dvorak J, Kral P. The link between microstructure and creep in aluminum processed by equal-channel angular pressing, *Materials Science and Engineering A* 2009;503;82-85.
- [4] Thomas WM, Nicholas ED, Needham JC, Murch MG, Templesmith P, Dawes CJ. *Friction Stir Welding* 1991.

- [5] Gibson BT, Lammlein DH, Prater TJ, Longhurst WR, Cox CD, Ballun MC, Dharmaraj KJ, Cook GE, Strauss AM. Friction stir welding: Process, automation and control, *Journal of Manufacturing Processes* 2014;16;56-73.
- [6] McNelley TR, Swaminatham S, Su JQ. Recrystallization mechanisms during friction stir welding/processing of aluminum alloys, *Scripta Materialia* 2008;58;349-354.
- [7] Arora A, De A, DebRoy T. Toward optimum friction stir welding tool shoulder diameter, *Scripta Materialia* 2011;64;9-12.
- [8] Uygur I. Influence of shoulder diameter on mechanical response and microstructure of FSW welded 1050 Al-alloy, *Archives of Metallurgy and Materials* 2012;57;53-60.
- [9] Xu W, Liu J, Zhu H, Fu L. Influence of welding parameters and tool pin profile on microstructure and mechanical properties along the thickness in a friction stir welded aluminum alloy, *Materials and Design* 2013;47;599-606.
- [10] Aval HJ. Influences of pin profile on the mechanical and microstructural behaviors in dissimilar friction stir welded AA6082-AA7075 butt Joint, *Materials and Design* 2015;67;413-421.
- [11] Movahedi M, Kokabi AH, Seyed Reihani SM, Najafi H. Mechanical and Microstructural Characterization of Al-5083/St-12 lap joints made by friction stir welding, *Procedia Engineering* 2011;10;3297-3303.
- [12] Movahedi M, Kokabi AH, Seyed Reihani SM, Najafi H. Effect of tool travel and rotation speeds on weld zone defects and joint strength of aluminium steel lap joints made by friction stir welding, *Science and Technology of Welding and Joining* 2012;17(2);162-167.
- [13] Sun Y, Fujii H, Takada Y, Tsuji N, Nakata K, Nogi K. Effect of initial grain size on the joint properties of friction stir welded aluminium, *Materials Science and Engineering A* 2009;527;317-321.
- [14] Topic I, Hoppel HW, Goken M. Friction stir welding of accumulative roll-bonded commercial-purity aluminium AA1050 and aluminium alloy AA6016, *Materials Science and Engineering A* 2009;503;163-166.
- [15] Khorrami MS, Kazeminezhad M, Kokabi AH. Microstructure evolutions after friction stir welding of severely deformed aluminum sheets, *Materials and Design* 2012;40;364-372.
- [16] Khorrami MS, Kazeminezhad M, Kokabi AH. Mechanical properties of severely plastic deformed aluminum sheets joined by friction stir welding, *Materials Science and Engineering A* 2012;543;243-248.
- [17] Olejnik L, Rosochowski A, Richert M. Incremental ECAP of plates, *Materials Science Forum* 2008;584-586;108-113.
- [18] Rosochowski A, Olejnik L. FEM Simulation of Incremental Shear. *American Institute of Physics, Conf. Proceedings* edited by E. Cueto and F. Chinesta Vol. 907, 2007, 653 -658.
- [19] Olejnik L, Chrominski W, Rosochowski A, Lipinska M, Lewandowska M. Incremental ECAP as a novel tool for producing ultrafine grained aluminium plates, 2014 IOP, *Conf. Ser.: Mater. Sci. Eng.* 63 012004 doi:10.1088/1757-899X/63/1/012004.
- [20] Chrominski W, Olejnik L, Rosochowski A, Lewandowska M. Grain refinement in technically pure aluminium plates using incremental ECAP processing, *Materials Science and Engineering A* 2015;636; 172-180.
- [21] Wejrzanowski T, Pielaszek R, Opalińska A, Matysiak H, Łojkowski W, Kurzydłowski KJ. Quantitative methods for nanopowders characterization, *Applied Surface Science* 2006;253;204-208.
- [22] Hall EO. The deformation and ageing of mild steel: III. Discussion of results, *Proceedings of the Physical Society of London Sect B* 1951;64(9)189-200.

- [23] Sato YS, Kokawa H, Enomoto M, Ikeda K. Hall-Petch relationship in friction stir welds of equal channel angular-pressed aluminum alloys, *Materials Science and Engineering A* 2004;354(1-2);298-305.
- [24] Zhang F, Xuekuan S, Chen Z, Nie Z. Effect of welding parameters on microstructure and mechanical properties of friction stir welded joints of a super high strength Al-Zn-Mg-Cu aluminum alloy, *Materials and Design* 2015;67;483-491.
- [25] Kamachi M, Furukawa M, Horita Z, Langdon TG. Equal-channel angular pressing using plate samples, *Materials Science and Engineering A* 2003;361;258-266.
- [26] Horita Z, Fujinami T, Langdon TG. The potential for scaling ECAP: effect of sample size on grain refinement and mechanical properties, *Materials Science and Engineering A* 2001;318(1-2);34-41.
- [27] Iwahashi Y, Horita Z, Nemoto M, Langdon TG. The process of grain refinement in equal-channel angular pressing, *Acta Materialia* 1998;46(9);3317-3331.
- [28] Prangnell PB, Bowen JR, Apps PJ. Ultra-fine grain structures in aluminium alloys by severe deformation processing, *Materials Science and Engineering A* 2004;375-377;178-185.
- [29] Gholinia A, Prangnell PB, Markushev MV. The effect of strain path on the development of deformation structures in severely deformed aluminium alloys processed by ECAE, *Acta Materialia* 2000;48;1115-1130.
- [30] Cao WQ, Godfrey A, Liu Q. EBSD investigation of microstructure and texture evolution during equal channel angular pressing of aluminium, *Materials Science and Engineering A* 2009;361;9-14.
- [31] Cabibbo M, Blum W, Evangelista E, Kasser ME, Meyers MA. Transmission Electron Microscopy Study of Strain-Induced Low- and High-Angle Boundary Development in Equal-Channel Angular-Pressed Commercially Pure Aluminum, *Metallurgical and Materials Transactions A* 2008;39;181-189.
- [32] Chowdhury SG, Mondal A, Gubicza J, Krallics G, Fodor A. Evolution of microstructure and texture in an ultrafine-grained Al6082 alloy during severe plastic deformation, *Materials Science and Engineering A* 2008;490;335-342.
- [33] Ji SD, Meng XC, Liu JG, Zhang LG, Gao SS. Formation and mechanical properties of stationary shoulder friction stir welded 6005A-T6 aluminum alloy, *Materials and Design* 2014;62;113-117.
- [34] Singh RKR, Sharma C, Dwivedi DK. The microstructure and mechanical properties of friction stir welded Al-Zn-Mg alloys in as welded and heat treated conditions, *Materials and Design* 2011;32;682-687.
- [35] Askari A, Silling S, London B, Mahoney M. *Friction Stir Welding Processing*, TMS, Warrendale, PA, 2001;43-50.
- [36] Munoz AC, Ruckert G, Huneau B, Sauvage X, Marya S. Comparison of TIG welded and friction stir welded Al-4.5Mg-0.26Sc alloy, *Journal of Materials Processing Technology* 2008;197(1-2);337-343.
- [37] Moreira PMGP, Figueiredo MAV, Castro PMST. Fatigue behavior of FSW and MIG weldments for two aluminium alloys, *Theoretical and Applied Fracture Mechanics* 2007;48(2);169-177.