

Novel Drawing System Approach To Manufacture Performant Commercially Pure Aluminium Fine Wires

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Abstract

Due to its electro-mechanical properties, commercially pure aluminium wires have attracted the interest of automotive industry representing a functional and efficient economic solution to reduce vehicle's weight leading to the diminishing of energy consumption and emissions in today's society. However, to consolidate its use in this sector and in new market realities, it is necessary to increase the flexibility of the aluminium conductor wires, consenting their installation in very small spaces and with high curvatures, avoiding any failure and electrical conductivity decrease. Thus, the evolution of microstructure and service performance need to be investigated and controlled to improve the service safety. The present research shows a new approach to efficiently continuously manufacture long wires with smaller diameters and fine grains at room temperature. It is studied the strengthening effects (yield and tensile strength, plasticity, hardness), the electrical conductivity and the microstructural changes of commercial 1370 pure aluminium (99.7% Al) when traditional wire drawing process is combined with equal channel angular drawing (ECAD) technique. The results of this proposed procedure of deformation "drawing-ECAD-drawing" show an evident benefit, compared to the classic technology of production of aluminium wire, obtaining fine grain structure product with superior mechanical strength and not influenced electrical conductivity. The proposed manufacturing approach leads to fine wires enhancing the material mechanical properties by microstructural evolution (i.e. grain size reduction) avoiding the traditional post manufacturing thermal treatments requiring a high amount of energy and time and careful steps.

Introduction

Commercially pure aluminium wire (CPAW) is considered as an important conductor line for transmitting electric power due to its high corrosion resistance, good electrical conductivity and light weight [1–3]. Since the enhancement of its mechanical strength, without losing its properties as good electrical conductor, is an important property to be suitable for a wide field of applications of the electrical engineering, greater attention was attracted by new approaches of manufacturing processes for innovative mechanical and conductor material properties enhancement: the main task is to significantly increase the mechanical performance of the processed material avoiding any electrical conductivity drop. Different studies demonstrated that both the cited properties, usually mutually exclusive, are mainly controlled by material microstructure of which grain size is one of the most important parameters. In particular, finer grain size microstructure allows to obtain a material strength increase avoiding electrical conductivity negative variation [4]. For this reason, it is of main importance to investigate, analyse and control material microstructure also by providing new approaches to manufacture products characterized by finer grain size. According to Hall-Petch effect, the material flow stress behaviour is an inverse function of the square root of the grain size; since CPAW is manufactured by a standard cold-drawing process for different passes, it is interesting to analyse the influence of plastic deformation on grain fragmentation and to understand how microstructure could improve the material properties during the process of fine wires. Y. K. Zhu et al. [5] analysed the stress profile and the plastic deformation behaviour of the CPAW

during a multi-pass cold drawing by combining the numerical results with the microstructural observations. The results show the distribution of the plastic deformation mode, texture evolution and grain size on the wire cross section, clarifying the relationship between microstructure evolution and stress profile at each drawing pass. In particular, texture evolution and grain refinement induced an enhancement of both the yield strength and microhardness of the processed sample. X. M. Luo et al. [6] investigated the effects of microstructural changes on mechanical and electrical properties during cold drawing process of CPAWs. They found a great enhancement of the material flow stress behaviour with a slight variation of the electrical conductivity related to grain refinement. J. P. Hou et al. [7] studied the influence of microstructure evolution on material strengthening in the drawing process of CPAWs. The conclusions showed as grain size and dislocation density are essential factors to influence materials' performance in the drawing process.

These papers highlight the importance of the material microstructure and the necessity to study its fundamentals for a safe production development. For these reasons in the last years a great attention was paid on grain size and recrystallization phenomena as important factors to influence the CPAW behaviour. Y. Iwahashi et al [8] analysed the influence of equal-channel angular pressing (ECAP) on high purity aluminium with particular interest to the grain evolution. Final grain size in the range of 0.5–1.5 μm was found, explaining the results with the shearing patterns developed in the samples during the process. U. Chakkingal et al. [9] investigated the mechanical properties and microstructural evolution of pure aluminium 3004 when processed by equal channel angular drawing (ECAD) method at room temperature. The experimental results showed an increase in yield strength and hardness, suggesting severe plastic deformation (SPD) process as a valid industrial method to significantly increase mechanical properties of material through grain refinement. X. Chen et al. [10] studied the influence of a new accumulative extrusion bonding (AEB) method when processing CPA (Al 99.6 wt%). The proposed method showed the advantages in refining grains and enhancing mechanical properties (yield strength, ultimate tensile stress and hardness) of AA1060 samples compared with traditional accumulative roll bonding (ARB) process. A. A. Zisman et al. [11] investigated mechanical properties, textures evolution and microstructures of commercial pure aluminium processed by a new simple method of ECAD with adjustable die opening. Good enhancement in mechanical properties were found as a consequence of recrystallization phenomena. Nowadays, SPD methods are considered the most promising manufacturing techniques to produce materials with reduced grain size, and for this reason it should be useful to study and propose new strategies for microstructural design by combining the advantages of SPD with traditional manufacturing methods to allow novel and innovative processing routes for materials properties enhancement.

Hence, with the aim i) to give a further contribution in this direction investigating and analysing the beneficial influence of the microstructural changes to the enhancement of the material properties by recrystallization, ii) to extend the knowledge of the effectiveness of the SPD technique in controlling grain size evolution, iii) to promote aluminium as a valid alternative to copper production of electrical conductors (i.e. copper: more expensive, heavier and with a greater environmental impact) and iv) to analyse a new approach to the conventional drawing manufacturing system, in this paper a mechanical,

electrical and microstructural analysis was conducted on CPAWs by interposing ECAD method in the traditional wire drawing process at room temperature. A comparison between conventional cold-wire drawing process and the proposed new manufacturing procedure (drawing-ECAD-drawing) for the production of aluminium fine wires was done showing the enhancement of the analysed system on the material behaviour and its suitability for continuous processing of long wires with smaller diameters, finer grain microstructure and combined mechanical strength enhancement without a worsening of the electrical conductivity.

Material And Methods

Commercial 1370 pure aluminium rods (Table 1) with an initial diameter of $\phi = 9.5\text{mm}$ were analysed. Two manufacturing process procedures were performed, analysed and compared: procedure A - the proposed manufacturing approach in which the initial rods with a diameter of 9.50mm were reduced to a final diameter of 0.2mm by 15 initial cold-drawing passes, a successive ECAD process and 23 final cold-drawing passes; procedure B - the traditional cold-drawing technology in which the initial rods with a diameter of 9.50mm were reduced to a final diameter of 0.2mm through 38 cold-drawing passes.

Table 1 Material chemical composition

Al	Si	Fe	Cu	Mn	Mg
99.7	0.10	0.25	0.02	0.01	0.02
Cr	Ni	Zn	B	Ga	
0.01	-	0.04	0.02	0.03	

Both the compared procedures were based on a total of 38 cold-drawing passes each one, but in the proposed approach an ECAD process was performed after the fifteenth cold-drawing pass, Fig. 1 and Table 2.

In detail, sample 1 represents the initial rod with a diameter of 9.5mm, this is the same for both the two analysed process procedures. Sample 2, 3 and 4 represent the state of the wire during the several passes characterizing the proposed manufacturing approach (procedure A): sample 2 is the wire after the initial 15 cold-drawing passes: the wire diameter was reduced from 9.5 to 2mm, with a section reduction of 95.6% and a drawing strain of 3.11; sample 3 is the wire after the ECAD process, with an inner die angle $\Phi = 140^\circ$ and outer die angle $\psi = \pi - \Phi$ (Fig. 2), preserving the transversal dimensions (diameter of $\phi = 2\text{mm}$) and sample 4 is the wire after the final 23 cold-drawing passes: the wire diameter was reduced to 0.2mm, with a total section reduction of 99.95% and a total drawing strain of 7.72. Sample 5 represents the state of the wire after the 38 cold-drawing passes characterizing the traditional cold-drawing technology (procedure B), Fig. 1.

In order to make comparable the results of the two investigated procedures it was considered the same initial rod (sample 1), the same number of cold-drawing passes (38 passes) and the same drawing speed of 25m/sec.

Table 2
Samples for experimental analysis

Process procedure	Sample	Manufacturing step position	Diameter [mm]
Procedure A	1	Initial rod	9.5
	2	After 15 cold-drawn passes	2.0
	3	After ECAD process	2.0
	4	After 23 cold-drawn passes	0.2
Procedure B	1	Initial rod	9.5
	5	After 38 cold-drawn passes	0.2

In the proposed manufacturing approach (procedure A), an ECAD die (Fig. 2) is interposed between two reduction sections in multipass cold-drawing (i.e. between the exit of the fifteenth drawing pass and the entrance of the sixteenth drawing pass) causing a SPD on the wire. In this way, preserving the transversal dimension (wire diameter), an increase of the material strength is observed as a consequence of recrystallization phenomena. Uniaxial tensile tests were carried out at room temperature on an MTS Criterion Model 45 testing machine for wires until 2mm of diameter and on an MTS Criterion Model 42 testing machine for wires of 0.2mm of diameter. Tensile tests were performed on all the five samples to make a comparison with the traditional technology of production, that in this work consists of 38 cold-drawing passes (procedure B). In particular, were done 3 replications per sample (Table 2), for a total of 15 experiments, and the average value was considered. Then, a comparative tensile fatigue analysis was conducted to determine the influence of the investigated manufacturing techniques on the number of cycles to the failure. The investigation was done by utilizing the final wires obtained by the two analysed processes (sample 4 and sample 5), 10 specimens per sample were tested and the average value was considered. The stress amplitude for testing was determined as a percentage of 20% of the ultimate tensile stress, with a constant cyclic frequency of 15Hz and a load ratio (minimum load/maximum load) of 10%. Moreover, specimens of the five samples have been transversely cut and mounted into a resin holder to be mechanically polished and then etched using the Kroll's reagent (92ml of distilled water, 6ml of nitric acid, 2ml of hydrochloridric acid). The cross section of the samples has been studied under an

optical microscope for microstructural analysis while the micro-hardness ($HV_{0.01}$) was measured by an instrumented micro-nano indenter.

Micro-hardness was measured on 15 specimens (3 units per sample, Table 2), 10 indentations per specimen were performed, for a total of 150 tests, and the average value was considered. Finally, the change of electrical conductivity of the five samples (Table 2) was quantified by a measuring bridge to verify the influence of plastic deformation and material microstructure evolution on the electrical conductivity of the material. Electrical conductivity measurements were performed on 15 specimens (3 units per sample), five analysis per specimen were performed, for a total of 75 tests, and the average value was considered.

Results And Discussion

3.1 Tensile test and fatigue analysis

The tensile tests (Fig. 3) and the fatigue analysis (Fig. 4) show as the material behaviour is enhanced by both drawing and ECAD processes, but an evident incidence is registered when the last one is applied. In fact, considering the proposed approach (procedure A), after the initial 15 cold-drawing passes (sample 2) the material strength rises of about 2 times with a reduction of the plasticity from $\varepsilon = 0.11$ to $\varepsilon = 0.08$, according with X. M. Luo et al. [6] were it was demonstrated an increase of the yield strength and a decrease of the elongation when drawing a pure aluminium wire from an initial diameter of 9.5mm to 2.99mm with 9 passes. Then, after the ECAD process (sample 3) a further enhancement of 30MPa was observed in the material flow stress behaviour with a reduction of the true strain from $\varepsilon = 0.08$ to $\varepsilon = 0.04$. Finally, after the last 23 cold-drawing passes (sample 4), an evident increase of about 90MPa more was observed with a total true strain of $\varepsilon = 0.013$. Starting from the initial rods, the proposed combined process allowed to obtain final wires ($\phi = 0.2\text{mm}$) with a total strengthening of about 3.5 times and a corresponding plastic domain reduction of the 90%.

While, considering the current technology of production investigated (procedure B), consisting in 38 cold-drawing passes (sample 5), the material strength shows an improvement of about 2.9 times and a total strain decrease from $\varepsilon = 0.11$ to $\varepsilon = 0.02$, according with J. P. Hou et al. [7].

Comparing sample 4 (final wire of procedure A) and sample 5 (final wire of procedure B), it is evident the influence of the SPD imposed by ECAD method that induces a flow stress raise due to i) the grain size evolution (Fig. 5) related to the material strengthening for the Hall-Petch effect [12–15] and ii) the increase of the dislocation density (Table 4). In fact, considering the obtained experimental results (material flow stress and microstructural evolution) and the additive strengthening model (Eq. 1), proposed by Hansen [16], it is possible to predict the behaviour of the dislocation density and to study the incidence of the grain size and dislocations on the material strengthening. The material flow stress at a particular strain $\sigma(\varepsilon)$ can be predicted by the following empirical law in which the stress is divided into three parts:

$$\sigma(\varepsilon) = \sigma_0 + \sigma_{gs} + \sigma_{disloc} \quad (1)$$

Where σ_0 is the frictional stress: a constant which includes contributions from solutes and particles but not from dislocations, σ_{gs} is the contribution from the grain size-related strengthening and σ_{disloc} is the contribution from the dislocation-related strengthening.

σ_{gs} can be written:

$$\sigma_{gs} = k_1 d^{-1/2} \quad (2)$$

Where k_1 is a constant and d is the average grain size. It is evident that the material flow stress is influenced by the dynamic recrystallization that significantly modifies the microstructure of the material (i.e. grain refinement, Fig. 5) resulting in material strengthening: grain boundaries represent a real obstacle to the dislocations' movement.

σ_{disloc} can be written:

$$\sigma_{disloc} = M\alpha\mu b\sqrt{\rho} \quad (3)$$

Where M is the Taylor factor, α is a coefficient, μ is the elastic shear modulus, b is the length of the Burger vector and ρ is the dislocation density.

Replacing Eq. 2 and Eq. 3, the additive strengthening model can be written:

$$\sigma(\varepsilon) = \sigma_0 + k_1 d^{-1/2} + M\alpha\mu b\sqrt{\rho} \quad (4)$$

All the constants characterizing the material behaviour (Eq.4) are listed in Table 3.

Table 3
Material model parameters [16–19]

σ_0	k_1	M	α	μ	B
[MPa]	[MPa $\mu\text{m}^{1/2}$]			[GPa]	[nm]
11.4	40	3.06	1/3	26	0.286

Considering the experimental results related to material flow stress (Fig. 3) and grain size evolution (Fig. 5) it is possible to estimate the value of the dislocation density ρ by Eq. 4. In particular the initial rod (sample 1) and the final wires of the two investigated procedures were considered (sample 4 and sample 5) when a strain of 0.01 is reached, Table 4.

Table 4
Data for dislocation density estimation

Sample	$\sigma_{0.01}$ [MPa]	d [μm]	ρ [10^{14} m^{-2}]
1 - initial rod	77	58	0.64
4 - final wire (procedure A)	259	1.1	7.80
5 - final wire (procedure B)	238	2.3	7.12

Table 4 shows as both the two investigated procedures have a significant influence on the dislocation density change. However, comparing sample 4 and sample 5 it was registered an increase of about 10% of the dislocation density, when ECAD process is applied, resulting in a greater material hardening (Fig. 3). Moreover, considering Eq. 4, material parameters of Table 3 and the data of Table 4 it is possible to calculate the incidence of the grain size and dislocations on the material strengthening increase as reported in Table 5.

Table 5
Incidence of individual strengthening mechanism

Sample	$\sigma_{0.01}$ [MPa]	σ_0 [MPa]	σ_{gs} [MPa]	σ_{disloc} [MPa]
1 - initial rod	77	11.40 (15%)	5.25 (7%)	60.01 (78%)
4 - final wire (procedure A)	259	11.40 (4%)	38.13 (15%)	209.46 (81%)
5 - final wire (procedure B)	238	11.40 (5%)	26.37 (11%)	200.12 (84%)

From Table 5 it is evident as the contribution of the grain size-related strengthening is an inverse function of the square root of the grain size: the finer the grain size, the more resistance increases according to Hall-Petch effect. Moreover, Table 5 illustrates as the contribution of the dislocation-related strengthening plays the main role in improving the material strength with a percentage incidence of about 80% in all the investigated samples. In Fig. 4 is shown the influence of ECAD process that improves the number of cycles to failure of about 15% when the two procedures are analysed. This result is in accordance with Tanaka's observations [20]: the threshold stress, for crack growth and propagation, is an inverse function of grain size, Eq. 5.

$$\sigma_{th} = \sigma_{fr}^* + \frac{1.04K_c^m}{\sqrt{d}} \quad (5)$$

Where σ_{th} is the threshold stress, σ_{fr}^* is the frictional stress of dislocation motion, K_c^m is the critical value of microscopic stress intensity factor and d is the grain size.

In fact, the major grain refinement obtained through ECAD die (Fig. 5) leads to the presence of more grain boundaries and different micro orientation of grains that can block the crack propagation since they have a significant incidence for the path selection of the fatigue crack.

3.2 Microstructure, microhardness and electrical conductivity

Considering the proposed manufacturing approach (procedure A), the microstructure shows a grain fragmentation from 58 μm to 11 μm after the initial 15 cold-drawing passes (sample 2), then a successive reduction from 11 μm to 6 μm after ECAD process (sample 3), and a final reduction from 6 μm to 1.1 μm when wire diameter was reduced to $\phi = 0.2\text{mm}$ through the last 23 cold-drawing passes (sample 4). A total grain size refinement of 98% was reached. While, analysing the current technology of production (procedure B), after the 38 cold-drawing passes (sample 5), the effect of the total drawing strain $\epsilon = 7.72$ reduced the initial grain size from 58 μm to 2.3 μm (Fig. 5). These results agree with [21–24], in fact, the high plastic strain induced through the ECAD die produces in the material a high accumulation of dislocations. Hence, recrystallization occurs achieving a dislocation density increase and microstructural change that results in grain refinement, with the consequent development of a high strength of the material (Fig. 5). Figure 5a shows the microstructure of the initial rod with an average grain size of 58 μm . The microstructure was characterized by grains with slightly different shapes although the peculiar morphology was equiaxial. Subsequently, the grain size was drastically reduced to an average value of 11 μm (Fig. 5b) due to the metallurgical phenomena induced by the 15 cold-drawing passes and therefore the significant plastic deformation. The ECAD step allowed a further refinement of the grains with an average grain size equal to 6 μm (Fig. 5c) and afterward the last 23 cold-drawing passes permitted to achieve a final grain size of 1.1 μm (Fig. 5d) against the 2.3 μm (Fig. 5e) achieved with the 38 cold-drawing passes performed without the intermediate ECAD step.

As highlighted in Fig. 6a, the use of ECAD as intermediate step introduced a further grain refinement (sample 3) that positively affects the mechanical properties as shown in Fig. 6d. A significant increase, more than 65%, was observed in the initial material hardness (sample 1) for both the analysed wire processes productions (sample 4 and sample 5), with a difference of about 7HV when implementing the ECAD method (Fig. 6c). In both the investigated procedures the hardness evolution is mainly due to the high reached strain that induces microstructural changes: grain refinement (increase in the volume fraction of grain boundaries) for the dynamic recrystallization phenomena [5].

Figure 6b shows all the measurements of the electrical conductivity of the five samples (Table 2) showing a not sensitive influence of the mechanical and microstructural changes on this aspect. In fact, there is a not evident change of the electrical conductivity from initial rods (sample 1) to final wires (sample 4 and sample 5), but a slight electrical conductivity reduction of about 1% was registered when increasing mechanical deformation during material processing, in accordance with [6, 25]. The obtained

results showed in this case as the grain size reduction from 58 to 1 μm is not a critical aspect for electrical conductivity property.

Hence, the enhancement of the mechanical behaviour preserving its electrical conductivity promote aluminium as a valid economic alternative to copper for the production of electrical conductors.

Conclusions

In this paper it was studied the strengthening effects (yield and tensile strength, plasticity, hardness, etc.), the electrical conductivity and the microstructural changes of commercial 1370 pure aluminium (99.7% Al) by a novel drawing approach in which the advantages of SPD technique is combined with traditional manufacturing methods for longer and more resistant fine wire production.

In particular, the relation between microstructural changes and service performance (mechanical and electrical) was analysed. The results show as recrystallization, leading to microstructural evolution (i.e. grain refinement and dislocation density increase), is an important aspect to control material's behaviour during manufacturing. Concerning the mechanical properties, the increase of the material strength, subjected to high process strains, was mainly attributed to the dislocation density and to the grain size refinement both related to the material strengthening for the Hall-Petch (H-P) relation. Moreover, the comparative tensile fatigue analysis resulted in a higher number of cycles to failure for wires processed through the new manufacturing strategy (procedure A). In fact, due to grain refinement, the presence of more grain boundaries and different micro orientation of grains can influence the path selection of the fatigue crack. Finally, the high strain reached by ECAD process (procedure A) induced grain refinement (increase in the volume fraction of grain boundaries) for the dynamic recrystallization phenomena resulting in higher hardness when compared with the traditional investigated drawing wires production (procedure B). Concerning electrical conductivity, a not sensitive influence of the mechanical and microstructural variation was registered. In fact, only a slight decrease of about 1% was registered showing as the range of the obtained grain size is not a critical aspect for electrical conductivity.

Based on the obtained results and analysis it can be concluded that the proposed manufacturing strategy represents an interesting industrial procedure for pure aluminium fine wires production. In fact, the significant increase in yield and tensile stress, with a corresponding enhancement of hardness evolution and cycles to failure resistance and a not significant change in electrical conductivity

- i. Confirm the effectiveness of the proposed approach to increase the flexibility of fine drawn wires reducing failure risks and electrical conductivity evident decrease;
- ii. Allow to further reduce the final wire section avoiding to implement the expensive, time consuming and less sustainable thermal treatment cycles;
- iii. Demonstrate as in this case the grain refinement from 58 to 1 μm doesn't significantly change the electrical conductivity while increasing material strength and hardness;

- iv. Validate structural refinement (i.e. grain size-related strengthening and dislocation density related-strengthening) as a useful technique to enhance the mechanical behaviour of the component preserving its electrical conductivity;
- v. Promote pure aluminium as a valid alternative to copper production of electrical conductors (i.e. copper: more expensive, heavier and with a greater environmental impact) extending its use in aerospace, automotive and constructions fields;
- vi. Make the application of ECAD method, and generally the implementation of the SPD techniques, suitable to enhance the conventional manufacturing systems proposing new strategy for improving material properties and their applications.

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Figures

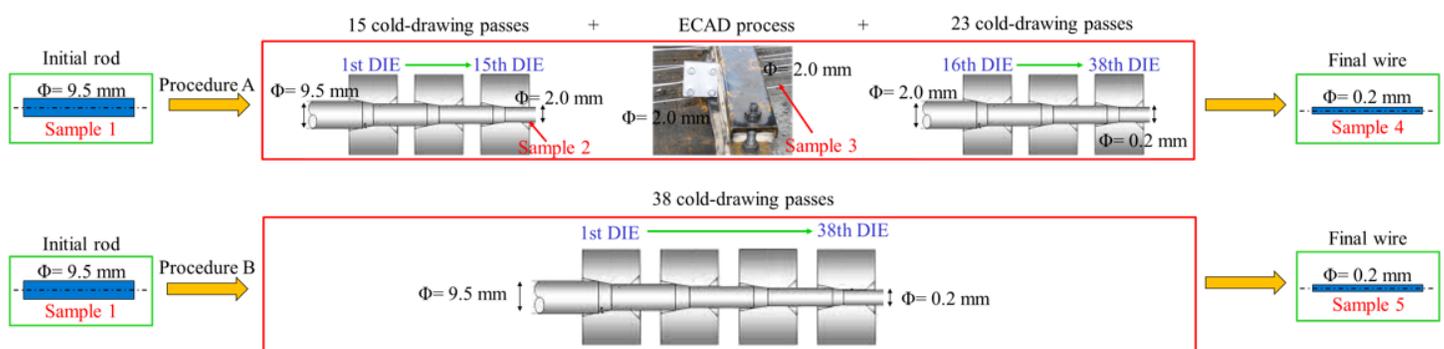


Figure 1

Experimental investigated procedures

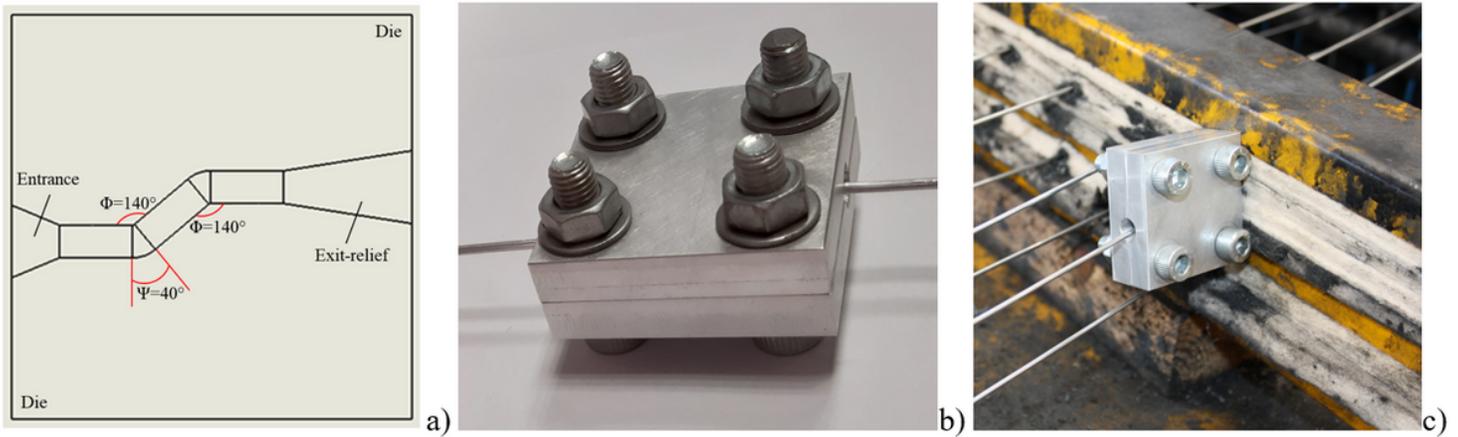


Figure 2

a-b) ECAD die and c) experimental set-up

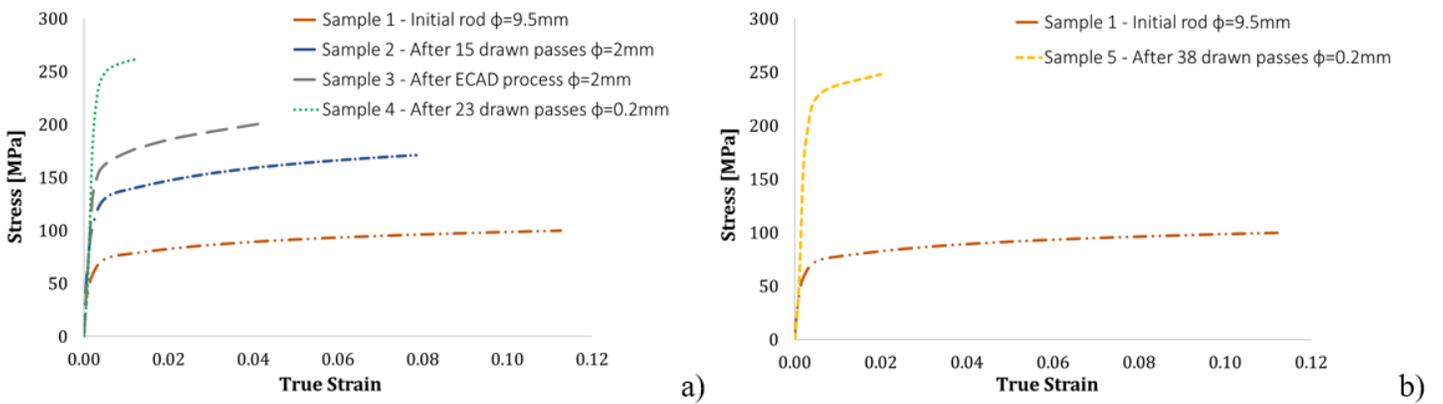


Figure 3

True stress-strain curve a) procedure A with interposed ECAD process and b) procedure B traditional technology

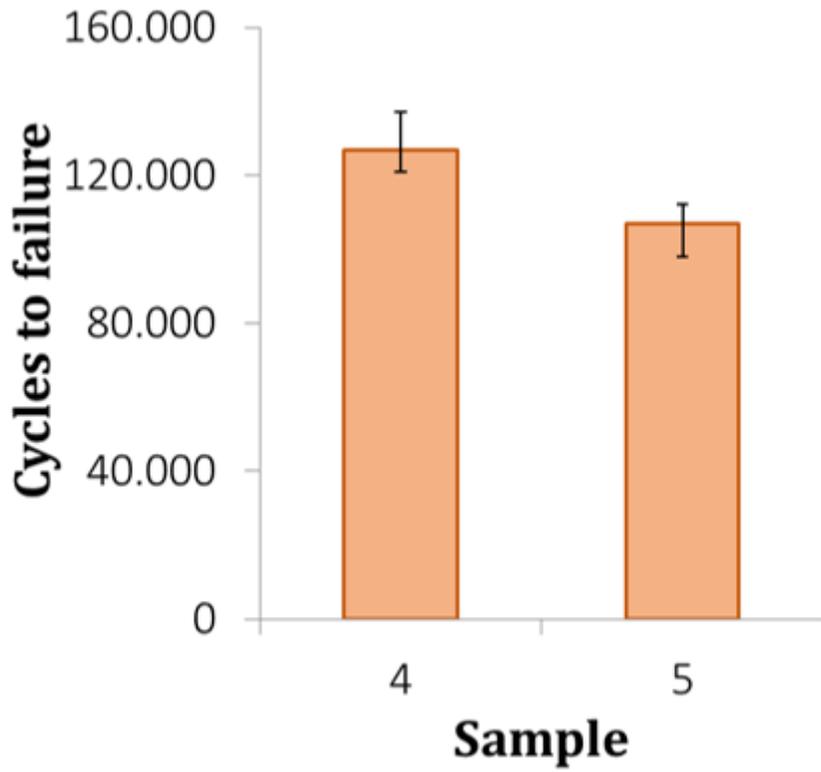


Figure 4

Tensile fatigue results

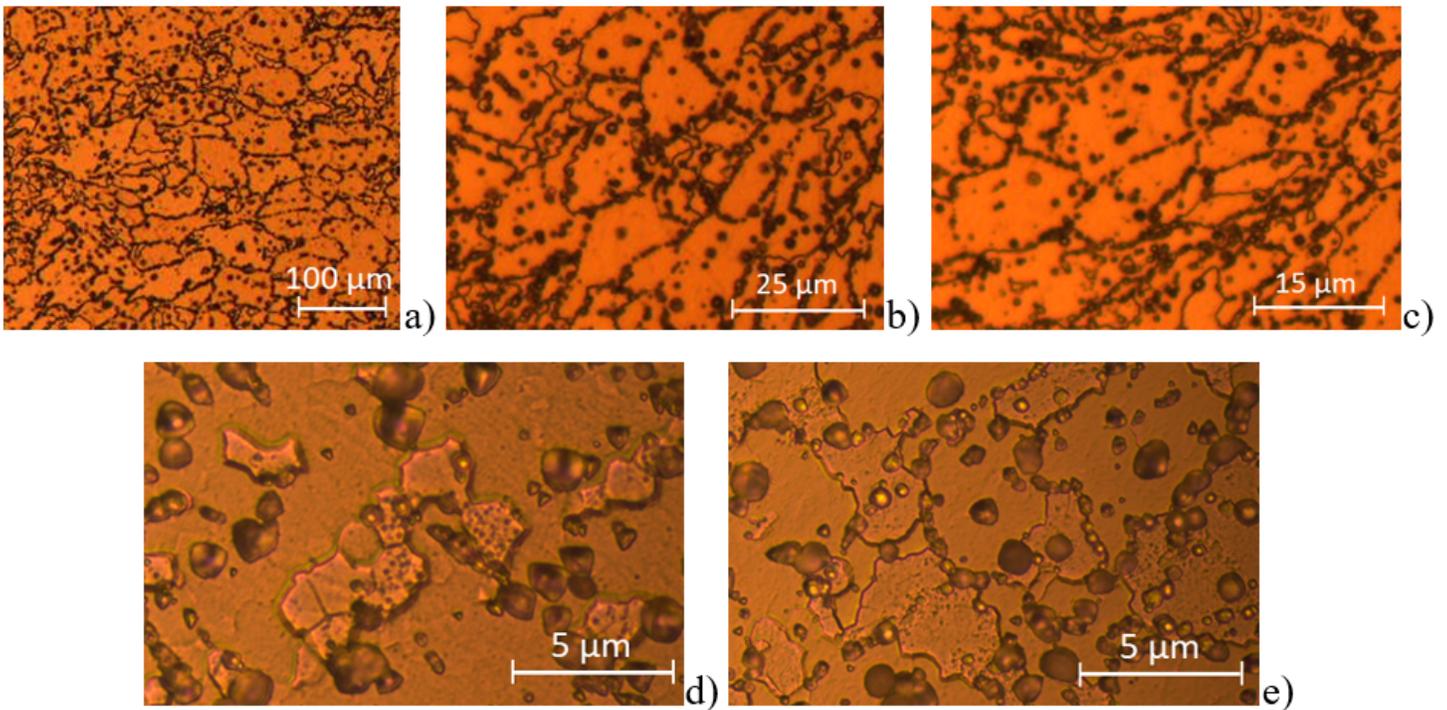


Figure 5

Transverse section microstructure a) sample 1, b) sample 2 c) sample 3, d) sample 4, e) sample 5

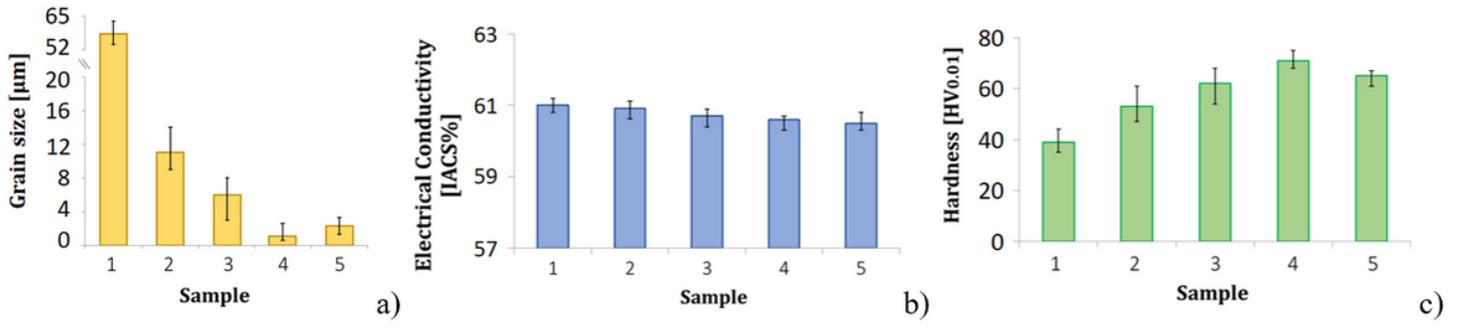


Figure 6

a) Average grain size measured on the transverse section, b) electrical conductivity and c) cross section hardness measurements