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Study of the microstructure and texture heterogeneities of Fe-48wt%Ni alloy severely deformed by equal channel angular pressing

Rabeb Lachhab^{a*}, Mohamed Ali Rekik^a, Hiba Azzeddine^{b,c}, Thierry Baudin^d, Anne-Laure Helbert^d, François Brisset^d, Mohamed Khitouni^a

^a Laboratoire de Chimie Inorganique Ur-11-Es-73, Faculté des Sciences of Sfax, BP 1171, 3018, Sfax Tunisie.

^b Faculté de Physique, Université des Sciences et de la Technologie Houari Boumediene, Bab Ezzouar, BP32, El Alia, Alger, Algérie.

^c Département de physique, université de Mohamed Boudiaf, M'sila, Algérie

^d ICMO, SP2M, Univ. Paris-Sud, Université Paris-Saclay, UMR CNRS 8182, 91405 Orsay Cedex, France

* Corresponding author. Tel.: +216 25252807 ; E-mail: lachhab_rabeb@hotmail.com

Abstract

A Fe-48wt%Ni alloy was processed by severe plastic deformation using equal channel angular pressing process. A stacking of 9 sheets was introduced and pressed up to two passes into die with an inner angles of $\Phi=90^\circ$ and outer arc of curvature $\psi=17^\circ$ at room temperature following route A. The same material in bulk form was also ECAPed up to one pass. The microstructure and the texture were investigated by means of electron backscattered diffraction and X-ray diffraction, respectively. To evaluate the mechanical response, Vickers microhardness was carried out. The given analyses concern the as-received sample, the peripheral and the central plates of the pressed stacks and the upper, the middle and the lower parts of the pressed bulk material. The deformation was heterogeneous and variations in texture and microstructure, resulting from different efficiencies in the shearing process, were locally noted. For the stacks samples, the microstructure evolved from equiaxed grains of 9 μm with high fraction of high-angle grain boundaries (around 90%) to a heterogeneous fine grain structure with an average grain size of 3 μm after two passes. On the contrary, for the bulk sample, the evolution was

to a banded structure after one pass. Results of mechanical property show that microhardness increased significantly from 147 Hv before deformation to mean values of 244 (after one pass) and 235 Hv (after two passes) for the bulk and stacked samples, respectively. The Hall–Petch effect and dislocation density were evaluated as the most responsible in material strengthening.

Keywords: Equal Channel Angular Pressing (ECAP); Fe-Ni alloy; X-ray diffraction (XRD); electron backscatter diffraction (EBSD); Microstructure; Texture.

Introduction

Equal Channel Angular Pressing (ECAP) is one of the severe plastic deformation (SPD) techniques available to achieve ultrafine-grained materials with higher mechanical properties [1-5]. Its principle is to shear the material by imposing it to pass several times through an angled channel with identical cross sections on entry and exit. To modify the strain path during ECAP process, different rotations of the billet between two successive passes are possible (90° in alternative directions in route B_A, 90° in the same direction in route B_C, and 180° in the same direction in route C). Lee and Cho [6] have shown that the deformation via route A (used in the present work) is the dominant route for the grain refinement and strengthening. In general, texture evolution after ECAP process following these four routes depends on three factors: the applied deformation (die configuration (Φ) and the number of passes (N)), the deformation mechanism (slip and twinning systems) and the initial texture [7].

As the cross-section of the billet remains constant after ECAP, the process can be repeated several times to increase the amounts of strain introduced into the material and ultimately to refine the grain size to the theoretical limit imposed by the characteristics of the used metal or alloy. The initial analytical treatments to estimate approximately the amount of strain per pass during ECAP have mostly supposed a deformation without friction and with a uniform strain in the cross-section of the extruded billet [8-10].

However, analytical modeling [11,12] and finite element studies [10-15] have shown that important factors leading to strain heterogeneities must be taken into consideration as the high friction along the bottom die wall, the presence of a dead zone on the outer die corner and back pressure. Other experimental and theoretical studies were realized on polycrystalline billets have confirmed variations in the grain size and local texture evolution along the billet height [16–19].

ECAP has been extensively applied over the last decade on a wide variety of metals and alloys [1, 20]. Numerous studies documenting the textural and microstructural evolution of ECAPed Ni [21] and the grain refinement of pure Ni after ECAP [22] have been published. There are also many reports on SPD of Ni based alloys as Ni-Ti alloys [23, 24]. At the present time, some reports are also available describing the SPD processing of various Fe–Ni alloys. To date, there are researches dealing with the microstructure, texture evolution of Fe–36%Ni alloy processed by Accumulative Roll Bonding (ARB) for ten cycles [25] and by Cross Accumulative Roll Bonding (CARB) up to six cycles [26]. Concerning the Fe–48%Ni alloy, previous texture and microstructure studies have been carried out after cold rolling [27] and after ARB up to 6 cycles [28] where the microstructure and mechanical properties of specimens of Fe–48%Ni and Fe–36%Ni prepared by ARB have been investigated. Results showed that both alloys underwent a different texture evolution and a substantial grain refinement but smaller grains and higher strength have been found in Fe-48%Ni. To the best of our knowledge, local texture evolution of Fe-48wt%Ni alloy during ECAP processing has never been reported in the literature.

Accordingly, the aim of this study is to process a Fe-48% Ni (wt.%) alloy, sensed for the manufacture of substrate for photovoltaic thin films, by ECAP at room temperature and to obtain a detailed understanding of the evolution of microstructure, texture and hardness properties using Electron Backscatter Diffraction (EBSD), X-ray diffraction (XRD) and

Vickers microhardness analysis. For that, different state of samples as the as-received sample, the peripheral plates and the central one of stacked sheets and three longitudinal sections from a bulk sample were used to understand the dependence of the strengthening behavior on the sample forms and on the ECAP process.

Materials and methods

The material selected for the present study was a commercial supra 50 alloy containing Fe–48%Ni (wt.%). It was provided by Aperam alloys Imphy (France). Table 1 gives the chemical composition of the present alloy.

Table 1 Chemical composition of the Fe-48%Ni (wt.%) alloy

Ni	Mn	Si	C	Fe
48	0,5	0,1	0,005	Bal

The alloy was received in the annealed condition as strips with dimensions of $300 \times 300 \times 1 \text{ mm}^3$ and $300 \times 45 \times 10 \text{ mm}^3$. On the one hand, stacked sheets (S.S) having rectangular shape with dimensions of $70 \times 10 \times 1 \text{ mm}^3$ were cut and deformed by ECAP using a die with intersecting channel angle $\Phi = 90^\circ$ and an outer arc of curvature $\psi = 17^\circ$ (Fig. 1a and Fig. 2) up to two passes. The processing was limited to two passes because of S.S's fracture. On the other hand, a bulk billet (B.B) ($70 \times 10 \times 10 \text{ mm}^3$) was also cut and processed by ECAP (Fig. 1b) in the same die up to one pass. Owing to the die's fracture, experiment was finished at the first pass. Fig.1.c presents the axis reference system XYZ, where "X" coincides with the extrusion direction (ED), "Y" is the normal direction (ND) and "Z" corresponds to the transversal direction (TD). In the present work, ECAP was conducted at room temperature, via route A, with a constant speed of 0.48 mm.s^{-1} . To reduce the friction, molybdenum disulphide was used as lubricant.

Figure 1 Schematic illustration showing details of the samples and the ECAP die geometry: (a) stacked sheets (S.S), (b) bulk billet (B.B) and (c) details of the ECAP die geometry.

The as-received materials, the peripherals and the central sheets (Fig. 1a) obtained after each pass through the die were characterized. For the bulk billet, three longitudinal sections (Fig. 1b) with 1mm of thickness were cut in the upper edge, in the center and in the lower edge along ND in order to compare these areas with the selected sheets.

Figure 2 Photographs of the die used showing ECAP processing of stacked sheets after one pass.

The microstructure of samples before and after ECAP was investigated by EBSD in the ED-ND plane after a mechanical and electropolishing at room temperature using the A2 Struers electrolyte at 30 V. The observation was carried out using a scanning electron microscope FEG-SEM SUPRA 55 VP operating at 20 kV. The EBSD step size was 0.4 μm and the grain size was determined by the intercepts method. EBSD data acquisition and analysis were conducted using the TSL Orientation Imaging Microscopy, OIMTM software.

The crystallographic texture was measured in the ED-TD plane by X-ray diffraction in a Siemens goniometer system. The X-ray radiation used was Co K α ($\lambda = 0.1789 \text{ nm}$). Data were analyzed with MTEX software [29]; the orientation distribution functions (ODF) were calculated using the harmonic method with expansion to L=22 from three pole figures ($\{111\}$, $\{200\}$, and $\{220\}$).

The micro-hardness of the specimens was measured with a Vickers microhardness tester SHIMADZU type HMV-2 using a load of 0.3 kg ($H_{v0.3}$). Five hardness indentations were arbitrarily performed in the central areas of each sample.

Results and discussion

Microstructure and texture before ECAP processing

The analysis of the as-received materials (sheet and bulk samples) showed the same results of microstructure, texture and microhardness. Fig.3.a illustrates the inverse pole figure (IPF) map ($\{hkl\} // (ED-ND)$) of the as-received alloy. The image shows a recrystallized microstructure composed by equiaxed grains having an average grain size of $\sim 9.3 \mu\text{m}$, and high fraction of twins. The distribution of the misorientation angles (Fig. 3.b) shows a majority of high-angle grain boundaries (HAGB) ($\sim 91\%$) with a large amount close to 60° , which corresponds to twin boundaries $\Sigma 3$ ($60^\circ <111>$). These twins, called “annealing twins”, are particularly observed in f.c.c alloys with low to medium stacking fault energy [30,31].

Figure 3 a) IPF map showing the microstructure (Scale bar: 100μm) and b) histogram of the misorientation angles of the as-received Fe-48wt%Ni alloy.

The initial texture of Fe-48(wt.%)Ni alloy is presented as ODF sections ($\varphi_2 = 0, 45$ and 65°) in Fig. 4. The details of the ideal rolling and annealing texture components identified in this study are also presented in the Fig. 4 and their descriptions are given in Table 2. The texture of the initial state is characterized by the presence of the Brass component (9.7%). Other components can also be found with lower intensities; Goss (7.6%), S (7.6%) and Cube (5.4%). Wasserman and Heye [32] reported that this type of texture is due to the occurrence of mechanical twinning as a deformation mechanism added to $\{111\}\langle 110 \rangle$ slip. Moreover, it has been shown that the texture development in f.c.c materials is strongly related to stacking fault energy. Indeed, a Brass type texture is very often found in materials with low stacking faults energy [33]. This is the case of Fe-48wt%Ni alloy with stacking fault energy of $\gamma_{\text{SFE}}=40 \text{ mJ.m}^{-2}$. [34].

Figure 4 ODF sections at $\phi_2 = 0, 45$ and 65° of as received Fe-48wt%Ni alloy obtained from XRD measurement in ED-TD plan.

Table 2 Miller indices and Euler angles of the f.c.c rolling and annealing orientations.

Component	{hkl}<uvw>	Euler angle ($^\circ$)		
		ϕ_1	Φ	ϕ_2
Brass	{011} <211>	35	45	0
Cube	{001} <100>	0	0	0
Goss	{011} <100>	0	45	0
Copper	{112} <111>	90	35	45
S	{123} <634>	59	37	65

Microstructure evolution after ECAP process

The microstructure evolution in the top, middle and bottom of stacked sheets and bulk billet of Fe-48wt%Ni alloy after ECAP processing are illustrated in Fig. 5.

Figure 5 IPF maps ($\{hkl\} // (ED-ND)$) showing the microstructure evolution of the Fe-48wt%Ni after ECAP processing. (Scale bar: 100 μ m)

As shown IPF maps depict various microstructures after processing by ECAP. One can see clearly that the refinement degree of the microstructure increases with passes number. The microstructure in the bottom and the central region tend to deform less so that there are larger and less elongated subgrains than in the top. After one pass, the microstructures in bulk billet consist of fine but elongated subgrains mostly in the top and the middle while the microstructure in stacked sheets was refined only in the top and some equiaxed grains can be observed in the middle and in the bottom. In this context, authors have assumed that equiaxed grains, already formed in f.c.c materials after a large deformation at low or at room temperatures, were probably formed by a continuous recrystallization process [35-37].

Fig. 6 shows the evolution of the average grain size as a function of the pass number for the different samples. The grains tend to be larger in the bottom and the center than in the top thereby indicating heterogeneity in the microstructures along the normal direction (ND). In parallel, grain size of the stack sample, after one pass, is smaller than the bulk billet mostly in the top and the middle. Generally, the grain size decreases slightly for all specimens. It diminishes from 9.3 μm to 2.75 and 3.45 μm , respectively, in the upper and lower sheets and to 4.38 and 7.5 μm in the upper and lower zones, respectively, of bulk billet.

Figure 6 Evolution of the average grain size of the stacked sheets and bulk billet of the Fe-48wt%Ni alloy after ECAP process.

Histograms of the angle misorientations measured at edges and at centers of the Fe-48wt%Ni alloy after ECAP process are shown in Fig. 7a-c. Fig. 7d presents the calculated fractions of HAGB along the ND after ECAP process.

After one pass in the die no twins $\Sigma 3$ ($60^\circ <111>$) were observed in the bulk billet while a low fraction is recorded in stacked sheets which disappears after two passes. For the stacks

sample, the HAGB fraction after one ECAP pass decreases until 40% in the top and 50% in the two other regions (Fig. 7). However, in the bulk material, this fraction decreases until 7.5, 9 and 6% in the top, the middle and the bottom regions, respectively. Whereas, after 2 passes, it diminishes until 20% and the microstructure is mainly formed by a large amount of LAGB (Fig.7b and d) as a result of dislocations generation.

Figure 7 Histograms of the misorientation angle of the Fe-48wt%Ni after ECAP processing (a) 1 pass and (b) 2 passes for the stacked sheets, (c) 1 pass for the bulk billet and (d) variation of the HAGB fraction along ND.

The obtained results indicate heterogeneity in the microstructures along ND. This may be related to the reduced number of passes. In this context, Hadj Larbi et al.[38] found a similar result, in the beginning of deformation of an ECAPed Cu-Ni-Si alloy while after 12 passes they found that the microstructure was continuously refined at both the upper

edge and the center of the samples and became more homogeneous. Since in the present study, the Fe-Ni alloy was extruded only twice via route A in the case of the stacks and once in the case of the bulk form, it may be difficult to acquire a microstructure completely homogeneous in the ND. Another explication of the heterogeneity in the case of the stacks has been proposed by Figueiredo et al. [39] in relation with the nature of route. In fact, the heterogeneous microstructure consists to the inhomogeneous distributions of effective deformation, the damage, the effective deformation rate and the maximum principal stress observed at a single shear plane after one pass in the die. This can lead to more effective deformation near the upper zone compared with the middle and the lower ones.

Texture evolution after ECAP process

The evolution of the texture after ECAP process for all samples is shown in Fig. 8. For convenience, the ideal ECAP orientations for f.c.c materials are summarized in Table 3 [40]. The texture did not change significantly after one pass in the edges of the stack sample compared with the initial texture. Nevertheless, one can notice the appearance of the Copper component (8.5%) in the top while Brass component disappeared in the center leaving the Cube (8%) as the dominant component.

Table 3 Ideal ECAP orientations for f.c.c materials using a channel angle of 90° [40].

Component and symbol	{hkl}<uvw>	Euler angle (°)		
		ϕ_1	ϕ	ϕ_2
◆ A	{111}< $\bar{1}10$ >	180	55	45
⊞ $\bar{A}$	{111}<1 $\bar{1}0$ >	0/36	55	45
■ A ₁	{111}<2 $\bar{1}\bar{1}$ >	30/210	55	45
▲ A ₂	{111}< $\bar{2}11$ >	150/330	55	45
○ B	{112}< $\bar{1}10$ >	180	35	45
● $\bar{B}$	{112}<1 $\bar{1}0$ >	0/360	35	45
▮ C	{001}<110>	45/135/225/315	0/90	0
		0/180/360	45	0
		0/180/360	0	45

The obtained textures, after one pass, for the bulk sample are different from those obtained for stacks one. In fact, the upper zone of bulk sample shows a typical shear texture. One can observe principally the A component $\{111\}\langle\bar{1}10\rangle$ with a fraction of (5,4%), while the other zones exhibit rolling type texture, mainly the Brass component with fractions of 6.5% and 5.9% in the middle and the bottom, respectively.

Figure 8 ODF sections ($\varphi_2= 0^\circ, 45^\circ$ and 65°) obtained from XRD measurements for Fe-48wt%Ni alloy after ECAP process (ED-TD plan) in sheet and bulk forms.

After two passes, the lower sheet shows only the existence of the Brass component (8.8%). The texture changes completely in the top and the middle regions and becomes typical of shear. Thus, the orientations are rather concentrated on A $\{111\}\langle\bar{1}10\rangle$ (9.1%) and $\bar{A}$ $\{111\}\langle 1\bar{1}0\rangle$ (8.8%) components in the center, and A_1 $\{111\}\langle 2\bar{1}\bar{1}\rangle$ (9.3%) and A_2 $\{111\}\langle\bar{2}11\rangle$ (8.8%) components in the top region. In this context, Beyerlein and Tóth [7] assumed that in ECAPed f.c.c materials with low stacking fault energy can develop A/ $\bar{A}$ and A_1 or A_2 components by increasing passes number. It is possible here to relate shear textures to rolling ones. It has been revealed somewhere else that a double rotation around

the transverse direction over approximately $+35^\circ$ and -35° transforms a shear texture in a nearly rolling texture [41]. Other studies [42,43] have argued that the Cube orientation is unstable during severe plastic deformation and leads to the formation of shear and deformation bands.

In order to explain quantitatively the texture changes as a function of the passes number, the texture index (I) was calculated. In fact, the texture index (I) of any ODF is described by an integration of the mean square of their intensity $f(g)$ over the entire Euler space G [44]:

$$I = \frac{1}{8\pi^2} \int_G f^2(g) \cdot dg \quad (1)$$

The value of I is equal to 1 for an isotropic state and it increases if the texture strengthens [45].

Table 4 shows the texture index for all specimens. This parameter was calculated from the XRD results. Calculated values are relatively low for all samples. However, after the first pass, this parameter increases slightly and then it decreases after the second pass for all sheets except for the central one. Its maximum value of 1.5 was observed in the center of the bulk sample after the first pass. Gholinia et al [46] reported that the texture index for ECAPed samples is relatively low in comparison with results obtained after rolling. This low can be related to the intensive introduction of grain boundaries via ECAP leading to a high fraction of HAGB [1,2,47,48].

Table 4: Texture index variation along ND for all samples before and after ECAP process

	Number of ECAP passes, N	Top	Middle	Bottom
As-received	0		1.18	
Stacked sheets	1	1.45	1.1	1.32
	2	1.32	1.21	1.16
Bulk sample	1	1.35	1.5	1.42

The number of factors that can cause texture heterogeneity is so large. Finite element simulations have shown that these factors include Φ and ψ angles, material behavior (hardening, initial yield, softening, rate sensitivity, thermal–mechanical properties, etc...), processing route, pre-form shape, friction, backpressure, and cross-sectional shape [7]. Grosdidier et al [49], in their study on the deformation heterogeneity in a Ni single crystal extruded once in a squared die section ($\Phi=90^\circ$ and $\psi=0^\circ$), referred the origin of the microstructure and texture heterogeneities observed along the vertical direction ND to the die geometry which can change the intensity and the distribution of the shear along ND. In other words, as the sample entered into the deformation zone of the die, the sides were always guided and the material could harden sufficiently to avoid a drastic localization of the stresses during the last step of ECAP process. This can generate a local variation in the deformation paths [49]. This heterogeneity in the texture has been also observed in polycrystalline crystals. As an example, Beyerlein et al. [50] observed in pure polycrystalline copper samples extruded once in a circular cross-sectioned die a difference between the texture of the bottom and the rest. Additionally, they related this difference to a large rigid body rotation, which is more energetically favorable than severe plastic shearing, and to the effects induced by the friction with the bottom wall of the die. Hence, in the present study, the heterogeneity observed along ND whatever in the stacks or even in the bulk sample, can adopt the same origin. In fact, it is the result of combined effects: (i) factors related to the geometry of the ECAP matrix play a key role. Indeed, the lower part is subjected to a complex mixture of two types of deformation: a shear more or less similar to that imposed in the central part and a shear given by the effects induced by the presence of the dead zone (presence of friction). (ii) The complexity of the type of deformation in this part of the sample is also given by the decrease in the intensity of the first type of shear.

Mechanical properties

Fig. 9 illustrates the evolution of microhardness along ND before and after ECAP process. It is clear that the first pass increased significantly the microhardness value from 147 Hv before deformation to 222 Hv in the upper sheet while in the case of the bulk sample the microhardness values reach 253, 244 and 236 Hv respectively in the top, in the center and in the bottom. After the second pass, the microhardness of the stacked samples becomes more important; 245, 228 and 234 Hv in the upper, in the middle and in the lower sheets respectively. Similar values and trends in the microhardness evolution have been reported in the invar Fe-36wt%Ni alloy deformed by ARB at 550°C up to 10 cycles [25]. The microhardness value of this alloy was 250 Hv after 2 cycles of ARB. Further studies about the SPD processing of pure Ni at room temperature by high-pressure torsion (HPT) [51] and ECAP [52] showed that the microhardness increased to 305 Hv after 2 HPT turns and to 265 Hv after 8 ECAP passes.

Figure 9 Variation of Vickers microhardness of Fe-48wt%Ni alloy, as stacks and bulk samples, along ND before and after ECAP process.

Generally, results show a significant heterogeneity in microstructure and texture. Further, one can notice that the hardening intensifies from bottom to top for the stacked samples as well as for the bulk sample. Moreover, it should be noted that one pass using a bulk sample was more effective than two passes using stacked ones in terms of hardening of the material (Fig. 9). This is due to sliding and multiple corner gap formation in the case of stacked sheets (Fig.2). That's why the passage of bulk sample into the die caused its fracture and the appearance of some cracks (red arrows) in the top of the sample (see Fig. 10).

Figure 10 Image quality (IQ) map on the top of bulk sample after one ECAP pass.

It is known that the total strength of materials is the contributions of different strengthening mechanisms such as the grain size strengthening, the precipitation strengthening, the dislocation strengthening and the solid solution hardening. In the present study the grain size and dislocation density are considered the most strengthening contributions responsible for increasing strain. Indeed, the solid solution hardening can be considered as constant and the precipitation strengthening practically inexistent because, for the present composition, Fe-48wt%Ni alloy is monophasic (Gamma phase) as shown in Fig. 11 which presents XRD pattern of the Fe-48wt%Ni alloy before ECAP process. The diffractogram revealed that the 2θ values of 51.26° , 60.12° and 89.90° are assigned to (111),

(200) and (220) crystallographic planes of the Ni, respectively. This confirms the absence of any other phase in the XRD pattern of the Fe-48wt%Ni alloy.

Figure 11 XRD pattern of Fe-48wt%Ni alloy before ECAP process

Conclusion

A stack of nine sheets and bulk samples of Fe-48wt%Ni alloy were deformed by ECAP twice and once, respectively. The present study has showed the heterogeneity, along ND, in texture, microstructure and mechanical properties developed locally after ECAP process. In fact, the top and bottom zones deformed differently than the central one. Shearing texture is typically enhanced in the top. On the other hand, the lower zone underwent significantly less deformation and the texture was identified close to the initial one. Moreover, the first pass, in the case of the stacked samples has not really led to a considerable change compared with the initial state and the bulk one. This later developed shear texture in the upper zone. Remarkably, the bulk sample showed cracks, while the

sheets had slid over each other. Finally, high hardness was achieved and the most strengthening contributions responsible for increasing strain are grain size and dislocation density (amount of LAGB) and no other phenomena such as precipitation. Remarkably, the bulk sample showed cracks, while the sheets had slid over each other. To conclude, the choice of stacked sheets is not advisable for ECAP elaboration, because of the sheets gliding which prevents to increase the strain hardening. Then, it will take more energy (2 passes) to reach the same level of plastic deformation as a massive sample (1 pass).

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Compliance with ethical standards

Conflict of interest: The authors declare no conflict of interest.

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