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Abstract

The stored energy, which is the main driving energy of the primary recrystallization, was measured in two Fe-48%Ni cold-rolled samples using three different approaches: the neutron diffraction method based on the peak broadening, the Kernel Average misorientation (KAM) and the Dillamore methods both based on the misorientation and dislocation cell size estimation using EBSD (Electron Back Scatter Diffraction) data. The results were compared with each other and showed differences in stored energy values. In this paper, it is demonstrated that the stored energy calculated by both KAM and Dillamore approaches is underestimated compared to that one calculated from neutron diffraction peak broadening. This is because Dillamore approach considers only the GND (Geometrically Necessary Dislocations), blocked in the cell walls, the KAM method takes into account only the GND in all of the microstructure (cells and walls) and the neutron diffraction method takes into consideration all types of dislocations (SSD (Statistically Stored Dislocations) and GND) within the microstructure. The measurement principle and the energy gap observed between the different approaches were discussed.

Keywords: EBSD; Neutron diffraction; Dillamore; KAM; Stored energy.

1. Introduction

The stored energy during the plastic deformation is an important feature for the optimization of texture, microstructure and then the mechanical properties of a given material. During the cold rolling, the energy is accumulated in the material. A large amount of this energy is dissipated as a heat and the rest is stored in the material as dislocations [1-2]. In the literature, several techniques have been used to measure the stored energy as calorimetry, diffraction and microscopy techniques. These techniques estimate the stored energy as a function of crystallographic

orientation except from the calorimetry which measures the stored energy of a set of orientations. By calorimetry technique, Knudsen et al. [3] found value of 126J/mol in the Nickel 98% cold-rolled. Guiglionda et al. [4] studied the stored energy in the hot deformed Al-2.5%Mg by X-ray diffraction and shows that the S component has the highest stored energy compared to the Brass, Goss and Cube orientations. Furthermore, Branger et al. [5] showed that the stored of these components estimated by neutron diffraction in the Fe53%Ni 50% cold rolled vary as E_C (28J/mol) > E_S (18J/mol) = E_B (18J/mol) > E_{Cube} (10J/mol) while Theyssier et al. [6] found that the stored energy of the texture components vary as E_S (22.2J/mol) > E_C (18.4J/mol) > E_B (15.5J/mol) > E_{Cube} (10.4J/mol) in the pure aluminium bicristal using microscopy technique (Dillamore).

Values of stored energy depend on the measurement techniques which are based on different approaches. Thus, in the IF steel 40% cold-rolled, Samet-MEZIOU et al. [7] have measured the stored energy for different texture components using Dillamore and neutron diffraction methods. For the {111}<110> component, the authors reported values of 3J/mol and 5J/mol, respectively. In their investigations, Taheri et al. [8] calculated the stored energy in a commercial cold-rolled aluminum (AA1050) by Dillamore and calorimetry approaches, and found different values between the two methods.

In the present study, two cold rolled Fe-48%Ni alloys were used to measure the stored energy of the main deformation components, which are the Brass (B: {110}<112>), the Copper (C: {112}<111>) and the Aluminum (S: {123}<634>). Moreover, the stored energy of Cube ({100}<001>) component was also measured. The difference between both alloys is the sulfur content.. These two alloys have not the same texture and microstructure after recrystallization, thus, the effect of sulfur addition (0 and 40 ppm) on the driving force of recrystallization (stored energy) was studied.. The stored energy was calculated on one hand with the neutron diffraction method, which is based on the diffraction peak broadening measurement, and on the other hand by the KAM (Kernel Average Misorientation) and the Dillamore approaches both based on the use of EBSD data (misorientations, dislocation cell size). The results of the three measurement techniques were compared and discussed for the first time in the litterature.

2. **Experimental procedure**

The Fe-48%Ni studied samples were elaborated by Aperam alloys Imphy and exhibit the chemical compositions showed in table 1. After melting, casting (bar) and hot-rolling, samples underwent a cold rolling of 99% of thickness reduction. The obtained samples have the form of thin tapes of 50µm of thickness. The samples analyzed by EBSD were firstly mechanically polished and then electro-polished. For KAM and Dillamore approaches, the microstructure and texture parameters were analyzed by EBSD and OIM^{TM} software (Orientation Imaging Microscopy) in the rolling surface (RD, TD) where RD is the rolling direction and TD is the

transverse direction. The scans explored an area of 1500 x 800µm with 0.1µm step size. The scanning electron microscope FEG-SEM SUPRA 55 VP operating at 25 kV was used to collect results. For neutron diffraction measurements, the samples were stacked to form $1cm^3$ cube volume (200 thin tapes). The neutron measurements were performed at the Laboratoire Léon Brillouin (Saclay/France) on the four circles diffractometer 6T1.

Table 1: Chemical composition of the studied samples (%Wt).

Samples	Fe	Ni	Mn	S
S-0	51.7	48	0.3	0
S-40	51.696	48	0.3	0.004

2.1. Determination of stored energy by neutron diffraction

The stored energy determination by neutron diffraction is a based on the diffraction peak broadening measurement. Many studies on the stored energy estimation have been done by using either X-ray diffraction [9-11] or neutron diffraction [5,12-13]. The diffraction peak broadening of both X-ray and neutron diffractions is mainly due to the local modification of inter-reticular distance [14]. This modification may be related to the dislocations and the heterogeneous distribution of chemical elements in the material. In the present materials, 99% cold-rolled, the peak broadening is mainly due to the existence of dislocations. Thus, the broadening enables directly the measurement of the elastic deformation in the crystal lattice, which leads to the measurement of stored energy. Neutrons have a high capacity of penetration in a bulk material. This capacity allows the measurements to be non-sensitive to the effect of the surface and moreover, to be statistically representative of all of the material volume.

The orientation distribution function (ODF) was calculated from four complete pole figures {111}, {200}, {220} and {311} using the discrete ADC method [15]. In the present work, samples were cold-rolled and exhibit an orthotropic symmetry. For this purpose, stored energy measurements were performed on quarter of pole figures only. Many scans were achieved in each pole figure quarter to obtain stored energy for a large number of orientations and in particular to provide accurate values for the deformation texture orientations. The scans $(\theta - 2\theta)$ were measured for various values (α, β) on each quarter of pole figure, using a 5x5(°) grid, completed by the positions where the diffracted intensity is significant.

The neutron diffraction pattern of sample cubic terbium garnet was measured and refined by Rietvield profile program [16] to determine exactly the resolution curve of the diffractometer. Then the *u*,*v* and *w* parameters, which depend on the diffractometer geometry, were calculated and related to the Full Width at Half Maximum of the peaks [17]. The peak broadening was fitted

using a Gaussian distribution to determine the integral width of the peak. The integral method has been developed to separate the domain size and the strain effects from the diffraction peak broadening [18]. Thus, the normalized integral width $b_m(s)$of the measured diffracted peak is described as sum of the two gauss functions depending on the size domain D and on the strains $\langle \varepsilon^2 \rangle$.

$$b^2{}_m(s) = \frac{1}{D^2} + 2\pi \langle \varepsilon^2 \rangle s^2 \qquad (1)$$

D is the average size of the diffracting domains and s, which is equal to $(2\sin\theta)/\lambda$, is the diffusion vector.

Assuming that for a given reduction R, the term of deformation $\langle \varepsilon^2 \rangle$remains constant for the same family of planes and that $D$is constant whatever is the plan, the average size of diffracting domain deducts easily from two successive orders of the same family of planes, as {111} and {222}.

For a crystallographic orientation "j" diffracted in the position (α, β), where, α and β are the azimuthal and radial angles of a point from the pole figure, respectively, the stored energy is given by the following equation [13] for each position in the pole figure.

$$E_j(\alpha,\beta) = \frac{3}{2}\frac{Y_{hkl}}{(1+2\nu^2{}_{hkl})}\langle \varepsilon^2 \rangle_j(\alpha,\beta) \qquad (2)$$

Where, Y_{hkl} and ν_{hkl} are the Young modulus and Poisson coefficient of the material, respectively. $\langle \varepsilon^2 \rangle$ is the micro-deformation which is obtained from the width of the broadening peak $b_m(s)$. This parameter $b_m(s)$ is expressed by the equation (2).

2.2. Determination of stored energy by Dillamore approach

Calculation of stored energy by Dillamore approach is based on the fact that the deformation energy of a grain is accumulated into the dislocation cell walls [19]. The principle of this technique consists in measuring the cells size *d* and the average misorientation angle θ between them and then deduct the stored energy (Figure 1).

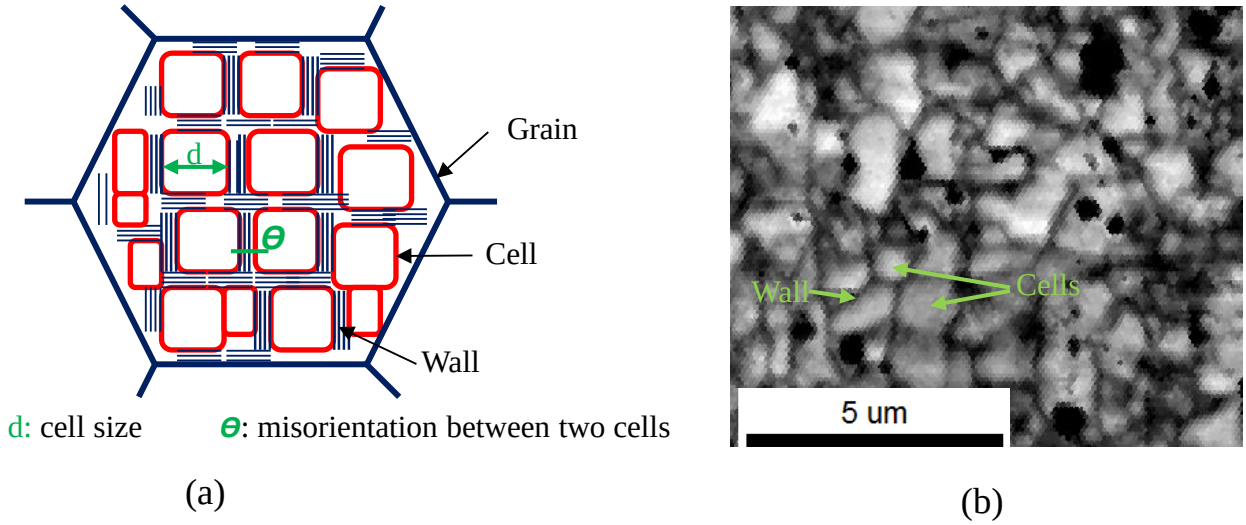


Fig. 1. (a) Dislocation cells and walls inside of a deformed grain. (b) Microstructure of the deformed state.

The stored energy according to Dillamore is given by the following equation 3 [5]:

$$E = \frac{KV\gamma_s}{d} \tag{3}$$

Where, K is a constant which depends on the cell shape (K = 3.31 for the equiaxed cells), V is the molar volume, γ_s is the boundary energy which is given by Read and Schockley relationship (equation 4) [20].

$$\gamma_s = \gamma_m \frac{\theta}{\theta_m}\left(1 - \ln\left(\frac{\theta}{\theta_m}\right)\right) \tag{4}$$

γ_m is the maximal energy of a grain boundary, which corresponds to the maximal misorientation value θ_m.

2.3. Determination of stored energy by the KAM approach

The local variations in misorientation is a good indicator of strain in crystalline materials and thus of the stored energy. Kernel Average misorientation (KAM) is an OIM analysis tool that characterizes the local misorientation. For a given point, the average misorientation θ_{KAM} of that point with all of its neighbors in the kernel is calculated with the priso that misorientations exceeding some tolerance value (maximum misorientation) are excluded from the calculation. In

this study, only the points at the perimeter of the kernel were used (Figure 2).The kernel size is defined according to the searched information.

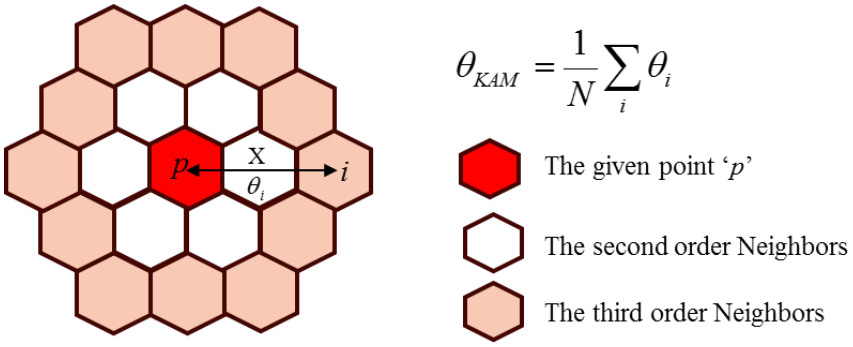


Fig. 2. Principal of KAM technique. θ_i is the misorientation between the first point (the central point) and a neighbor point "*i*", θ_{KAM} is the average misorientation between the first point and its "*N*" neighbors.

From misorientation, the dislocation density is estimated by the following equation [21-23].

$$\rho = \frac{\alpha \theta_{KAM}}{Xb} \tag{5}$$

Where, α is a parameter which depends on the grain boundary type and is equal to two for tilt boundaries, four for the twist ones and three for the mixt of the two types b is the burgers vector (b=0.253nm for Fe50%Ni alloy [24]) and $X = nd$ is the kernel size (n the defined nearest neighbor and d is the scan step) (Figure 2). Then, the stored energy can be formulated as:

$$E = \frac{1}{2}\rho \mu b^2 \tag{6}$$

Where, μ is the shear modulus.

3. **Results**

The stored energy of two Fe-48%Ni samples cold-rolled to 99% of thickness reduction, containing different sulfur contents (S-0 and S-40), has been measured by the three approaches described above.

3.1. Stored energy calculated from neutron diffraction measurements

The measurement of the stored energy was performed on the B, C, S and Cube orientations. As it can be observed in figure 3, the stored energy of the S and C orientations increases with the sulfur addition. However, the one of the Cube orientation remains quasi-stable and exhibits the lowest value. This low energy of the Cube component favors its development despite of the other orientations, during recrystallization [9].

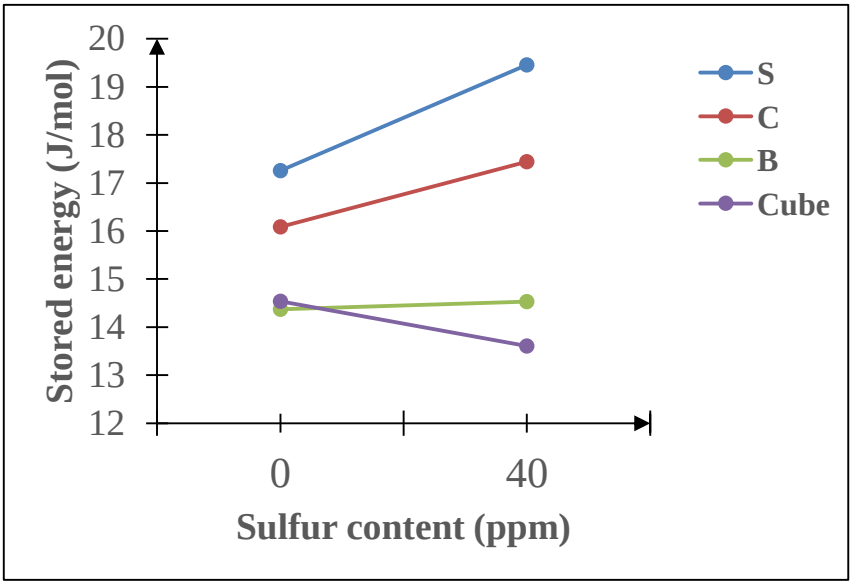


Fig. 3. Stored energy of S, C, B and Cube components in the deformed state of the two S-0 and S-40 samples, measured by neutron diffraction.

3.2. Stored energy calculated by the Dillamore approach

For each orientation (B, C, S and Cube of the deformed state), the average size of dislocation cells and the average misorientation between cells have been measured on EBSD scans by using OIM^{TM} software. In order to get more statistic results, many misorientation profiles (about 20 profiles by orientation) between cells were done on the whole EBSD scans and average results are shown in table 2.

Table 2: Average misorientation, cell size and stored energy of the different orientations for the S-0 and S-40 samples.

	S-0				S-40			
Orientation	S	C	B	Cube	S	C	B	Cube
θ (°)	3.60	4.70	2.80	2.78	3.85	3.67	2.57	2.74
d (µm)	0.80	1.01	0.78	0.91	0.80	0.82	0.73	0.91
Stored enegy (J/mol)	8.03	7.26	7.16	6.12	8.31	7.91	7.30	6.06

From the data of θ and *d*, the stored energy was calculated by the Dillamore method. It should be noted that cells were equiaxed (K = 3.31). The evolution of the stored energy of the different components (B, C, S) for the two sulfur contents is similar to the one obtained from neutron diffraction, the stored energy values obtained by the Dillamore approach being lower.

3.3. Stored energy calculated by the KAM method

In order to get a good precision of the stored energy values by the KAM method while the cell size is about [0.7-1µm], a scan step size of 0.1µm, a kernel size of 0.3µm (n=3) and a maximum misorientation of 15° have been chosen for the KAM calculation. Moreover, the boundaries were supposed to be of flexion type. Once again, the stored energy found by this method varies in the same way than the one measured by neutron diffraction depending on the orientation and on the sulfur content.

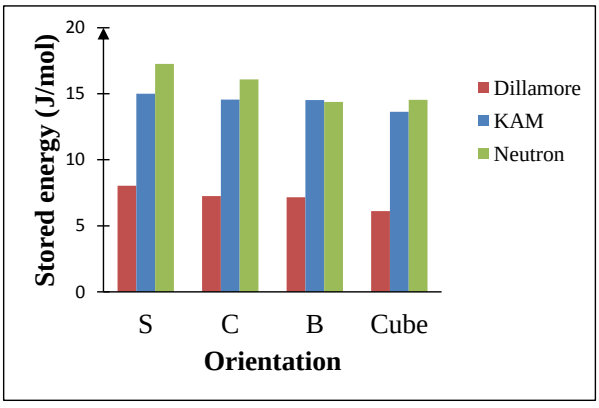
Table 3: Stored energy of the different orientations for the S-0 and S-40 samples estimated by KAM.

	S-0				S-40			
Orientation	S	C	B	Cube	S	C	B	Cube
Stored enegy (J/mol)	15.00	14.55	14.51	13.62	16.00	15.33	15.2	13.65

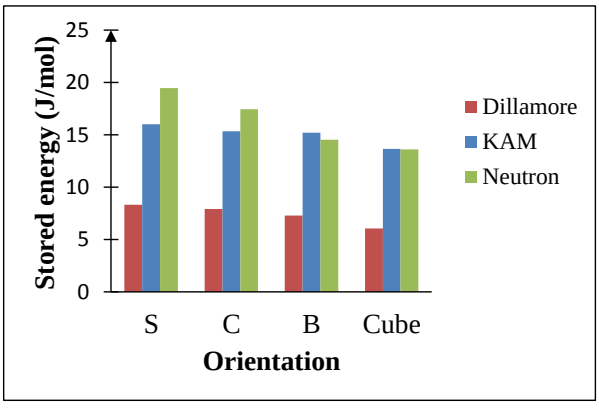
4. Discussion

In the literature, the calculation of the stored energy after plastic deformation is not unanimous. Experimental results were often dispersed, and even contradictories. The influence of the crystallographic orientation on the stored energy has been confirmed by different authors [26-28]. For copper single crystals deformed by tension, Steffen et al. [27] showed that the stored energy measured by calorimeter of the {111} orientations is higher than that one of the {110} orientations. While Nakada [28] showed that these two orientations have the same energy. Moreover, even for the same studies, results are difficult to be interpreted, thus, for aluminum single crystals deformed by compression, Nakada [28] found that the {100} exhibits lower stored energy than that of the {111} orientations, but in the case of silver single crystal, the author found inverted results. In their study, Jakani et al. showed that the stored energy measured by neutron diffraction for the copper alloys (38% deformed) inside the {111} orientations (3.6J/mol) is higher than inside the {001} orientations (1.8J/mol) [29].

The aim of the present study is to compare the values of stored energy obtained by the three approaches. For the two investigated samples, a comparison of the calculated stored energy of each orientation is shown in figure 4. The evolution of the stored energy follows the same trend: $E(S) > E(C) > E(B) > E(Cube)$ for the three techniques. Furthermore, this trend is reproducible in both samples with or without sulfur. These results are in agreement with those of Etter et al. [12] who found that, for Fe53%Ni alloy cold-rolled to 77% and 95% of thickness reduction, $E(S) \cong E(C) > E(B) > E(Cube)$.



(a)



(b)

Fig. 4. Comparison of stored energy of S, C, B and Cube components in the deformed state of the two S-0 and S-40 samples, calculated by the three approaches.

In spite of the similar evolution of the stored energy with the crystallographic orientation, the values of this energy are very different from a technique to another. The global analysis of the results shows that the stored energy calculated from neutron diffraction is higher than the one measured by KAM, which itself is higher than the one calculated by the Dillamore approach ($E_{Neutron} > E_{KAM} > E_{Dillamore}$). A significant difference between the stored energy calculated by the Dillamore and the other methods is found. This energy gap between methods can be induced by the ability for the different methods to measure the different types of dislocations (SSD (Statistically Stored Dislocations) and GND (Geometrically Necessary Dislocations)) and also to measure the dislocations located in the cell interiors and in the cell walls. In plasticity, dislocations can be separated into two different categories, geometrically necessary dislocations (GND) which appear in strain gradient due to geometrical constraints of the crystal lattice, and statistically stored dislocations (SSD) which evolve from random trapping processes during plastic deformation [30] (Figure 5). Figure 5 shows that, misorientation is principally due to GND dislocations.

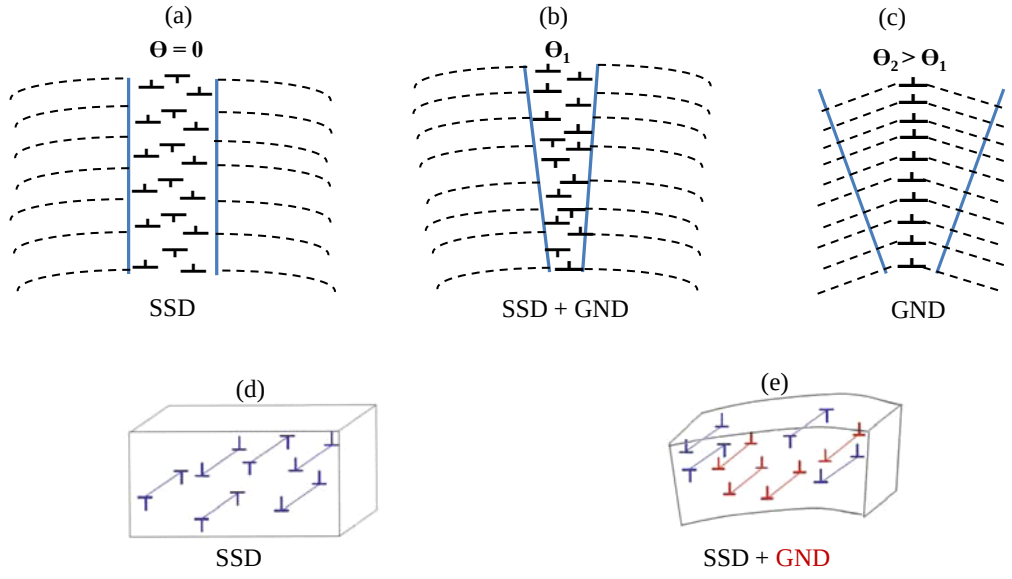


Fig. 5. Schematic process of GND dislocations accumulation in the crystal. In figures (a) to (c) GND accumulation increase misorientation between two crystal and in (d) to (e), GND accumulation lead to the crystal lattice distortion [30].

In the case of the neutron diffraction, the dislocation walls and cells both contribute to the diffraction peak broadening [31, 32] which is due to the distortion of the crystal lattice. The crystal lattice distortion is generated by all of the volume dislocations (SSD+GND), where the presence of GND polarizes the field distortion. As a conclusion, the measurement of stored energy by neutron diffraction takes into account dislocations in the entire microstructure (cells and walls) and all types of dislocation (GND and SSD).

Contrary to the neutron diffraction method, the Dillamore approach takes into account only the dislocations within cell walls [19] and only one type of dislocations (GND). Indeed, this last approach is based on the misorientation between dislocation cells which is the result of the presence of the GND. Then, this method under-estimates the stored energy. Both the interior of cells and the SSD are not taken into account in the calculations.

The measurement of stored energy by KAM method is also based on misorientation but between one point and its neighbors. As the Dillamore approach, the misorientation is due only to GND dislocations. The present calculation of the misorientation by KAM method takes into account dislocations within cells and walls since the KAM parameters have been chosen according to the dislocation sub-structure. Indeed, the kernel size of 0.3µm is smaller than the cell size (d<1µm) and the KAM calculation excludes misorientations greater than 15° to avoid the contribution of grain boundaries.

Contrary to the Dillamore approach, values of stored energy calculated by KAM method are closed to those calculated by neutron diffraction. Nevertheless, a slight underestimation is observed.

The energy gap observed between the neutron and KAM methods which is about 1.2J/mol, is due to the fact that the KAM approach does not take into account the SSD in both the cell interiors and walls.

The energy gap between KAM and Dillamore approaches is about 7J/mol. This gap is due to the fact that the Dillamore approach misses the dislocations in the cell interiors. As regards to the comparison between neutron and Dillamore methods, the energy gap is about 8.5 J/mol.

The comparison of stored energy measurements by the different methods are summarized in the following equations:

$$E_{Neutron} = E_{cells} + E_{walls} = E_{GND_{Cells}} + E_{SSD_{cells}} + E_{GND_{walls}} + E_{SSD_{walls}} \quad (7)$$

$$E_{KAM} = E_{cells} + E_{walls} = E_{GND\,cells} + E_{GND_{walls}} \quad (8)$$

$$E_{Dillamore} = E_{walls} = E_{GND_{walls}} \quad (9)$$

These results show that the SSD dislocations contribute slightly (~ 8%) to the global stored energy value (Equation 10).

$$E_{SSD} \cong E_{Neutrons} - E_{KAM} \quad (10)$$

At the same time, the effect of sulfur addition on the stored energy was also investigated. Results show that the stored energy increases by sulfur addition (Figures 3 and 4). This could be explained by the effect of alloying elements which increase the strain hardening of the material and then the stored energy. Moreover, it was observed that the sulfur element combines with Manganese element in the S-40 sample to form MnS precipitates of size 100-200µm. These precipitates lead to a hardening of the material and then increase the stored energy. Thus, the presence of sulfur in the Fe-48%Ni alloy implies an increase of the stored energy [33] which is clearly evidenced by the three techniques investigated in the present work.

5. Conclusion

This investigation consisted in studying two main subjects: a comparison between the measurements of stored energy by different approaches and the effect of sulfur addition on the stored energy, of two highly cold-rolled Fe-48%Ni (99% of thickness reduction) alloys containing different sulfur contents.

First, it was demonstrated that Dillamore approach highly underestimates the stored energy while the one calculated by KAM method is slightly underestimated. Nevertheless, the one calculated by neutron diffraction approach, based on the diffraction peak broadening, exhibits the best estimation. Dillamore approach considers only the GND dislocations, blocked in the cell walls. The KAM method takes into account only the GND in all of the microstructure

(cells and wall). However, the neutron diffraction method takes into consideration all of the dislocation types present in all of the microstructure features. As a conclusion, for the measurement of the stored energy in Fe-48%Ni alloys, the neutron diffraction remains the most complete technique. Furthermore, values of stored energy obtained by KAM approach are very close to the one obtained by neutron approach. This implies that the quantity of the SSD are not significant in the cold-rolled samples (equation 10). However, it is important to choose the correct parameters in the KAM calculation to consider dislocations in cells and in walls.

Second, whatever the different techniques, it was found that the addition of sulfur in the Fe-48%Ni alloys increased the stored energy. The sulfur combines with manganese to form precipitates (MnS). That implies a hardening of the material and then increases the stored energy.

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