

Effect of microalloying elements on the Cube texture formation of Fe48%Ni alloy tapes

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Abstract. The effect of microalloying elements sulfur and niobium was studied in Fe48%Ni sheets on the first stages of nucleation and on recrystallization. It was shown that the addition of sulfur promotes the formation of Cube grains while the addition of niobium prevents the Cube grains formation. Regarding sulfur, it combines with manganese which is an inhibitor of recrystallization to form the MnS precipitates. The stored energy difference between Cube component and others components $\Delta E_{\text{Cube/other}}$ increases when sulfur is added. This stored energy difference which is the main driving energy for Cube grains formation during recrystallization explains the sharpness of the Cube texture when sulfur is added. On the contrary the niobium microalloying element addition prevents the formation of Cube grains. This could be explained by the fact that stored energy of cold-rolled components decreases with the addition of niobium and thus decreases Cube grains fraction when niobium is added. In order to explain these results, the development of Cube texture during recrystallization has been investigated in detail by EBSD, furthermore, the effect of stored energy has been studied by carrying out neutron diffraction measurements on the deformed states.

1. Introduction

One way to develop the thin-film silicon photovoltaic cells with a high conversion efficiency is to make oriented silicon grains on a thin substrate. This substrate must have the Cube texture $\{100\}\langle 001\rangle$ which promotes the silicon growing by epitaxy. Furthermore, the substrate must be non-fragile, flexible and must have a high melting temperature. The Fe48%Ni which develops the Cube texture after recrystallization [1-4] is suitable for this application [5]. In the present study, the effect of microalloying elements, sulfur and niobium on the development of the cube texture was studied.

The development of Cube texture in the FeNi alloys have been studied by some authors [1-2, 6-10]. But the effect of microalloying elements on the recrystallization of these alloys has not been yet studied. Nevertheless, this effect was studied in others alloys. Thus, Eickemeyer et al. [11] showed that sulfur (7 wt ppm content) can interfere in the formation of Cube grains during the recrystallization of Ni5%W alloys. In their study, Jakani et al. [12] demonstrated that sulfur can decrease the dislocation cell size after cold wire drawing and then decreases the kinetic of recrystallization in the copper alloys. For niobium microalloying element, most of the previous works showed that, this element in solid solution or as precipitates, delays the recrystallization process and increases the recrystallization temperature [13-15].

To obtain a highly textured Cube substrate, the material must be strongly cold-rolled and annealed at high temperatures [1-2]. After strong deformation by cold-rolling, the main components are formed: the aluminum component S $\{123\}\langle 634\rangle$, the copper component C $\{112\}\langle 111\rangle$ and the brass component B $\{110\}\langle 112\rangle$. At the deformed state, the Cube orientation fraction is very low (about 1%). During recrystallization, it increases at the expense of cold-rolled orientations. Then the Cube orientation becomes the main orientation after recrystallization.

2. Experimental procedure

After bar casting, the alloys were hot-rolled to 5mm thick tapes at Aperam alloys Imphy. The tapes were subsequently cold-rolled to 50 μ m (99% thickness reduction). The samples were then annealed in vacuum atmosphere at 1000°C. To obtain flat surface, the specimens were polished. The microstructure and texture were analyzed by EBSD and OIMTM software (Orientation Imaging Microscopy). The results were collected using a scanning electron microscope FEG-SEM SUPRA 55VP operating at 25kV. The texture and microstructure measurements were done in the rolling plane (RD, TD) where RD is the rolling direction and



TD the transverse direction. In order to obtain statistical results, the scans explored an area of $1500\mu\text{m} \times 800\mu\text{m}$ with $0.1\mu\text{m}$ step size in the deformed samples and $1\mu\text{m}$ step size in the recrystallized samples. The stored energy of the deformed components in the cold-rolled samples were estimated from neutron diffraction measurement at the Laboratoire Léon Brillouin (CEA Saclay/France) on the four circles diffractometer 6T1. A pile up of samples was realized to obtain 1cm^3 cubic specimen. Moreover, the kinetic of recrystallization were followed by Vickers Micro-hardness measurement along the rolling plane. In the table 1, the chemical composition of the samples is shown.

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

Samples	S-0	S-20	S-40	S-60	Nb-0	Nb-350	Nb-500
Ni	48	48	48	48	48	48	48
Mn	0.3	0.3	0.3	0.3	0.3	0.3	0.3
S	0	0.002	0.004	0.006	0.004	0.004	0.004
Nb	0	0	0	0	0	0.035	0.050

3. Results

3.1. Texture of the deformed state

After deformation, the microstructure and texture of sample were characterized by EBSD. It was shown the similar texture and microstructure for “S” and “Nb” samples. The texture was mainly composed of B, S and C components, the Cube component fraction is very low in these cold-rolled states.

3.2. Texture and microstructure after recrystallization

The corresponding volume fraction of the Cube orientation after recrystallization was calculated by OIMTM as well as misorientation of Cube grains around the ideal Cube orientation. When sulfur is added in the sample, the fraction of Cube grain increases and the misorientation around the ideal Cube orientation decreases (Fig.1). This result shows that the sulfur element favors the formation of Cube orientation. On the contrary, the fraction of Cube grains decreases and the misorientation increases with the niobium addition. Thus, contrary to the sulfur element, the niobium element prevents the Cube texture formation.

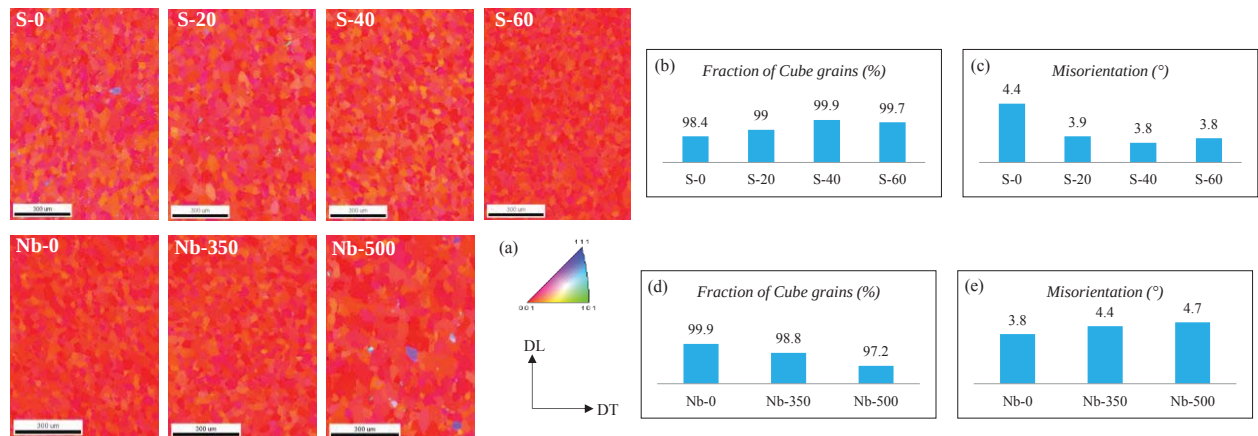


Figure 1: Orientation maps of samples after full recrystallization (a), fraction of Cube grains (b, d), average misorientation of Cube grains with 10° dispersion around the ideal Cube orientation (c, e). All samples were annealed at 1000°C during 60 minutes.

3.3. Kinetic of recrystallization and nucleation of cube grains

Fig.2 shows the effect of sulfur (a) and niobium (b) on the kinetic of recrystallization of Fe48%Ni alloy tapes. The extent of recrystallization was calculated from micro-hardness measurements, by using the following equation:

$$X_v = \frac{HV(\text{cold-rolled state}) - HV(t)}{HV(\text{cold-rolled state}) - HV(\text{fully annealed})} \quad (1)$$

X_V is the extent of recrystallization and HV is the Vickers Hardness.

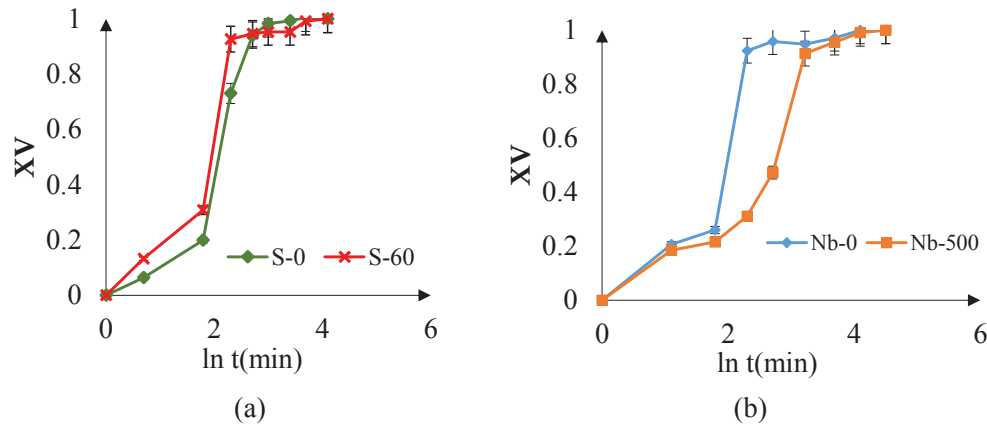


Figure 2: Evolution of the calculated recrystallized fraction determined from Vickers microhardness measurements as a function of annealing time at 600°C: (a) for S-0 and S-60 and (b) for Nb-0 and Nb-500 samples.

It is shown that recrystallization rate of S-0 samples is lower than the recrystallization rate of S-60 sample (Fig.2(a)) and that the recrystallization rate of Nb-0 is greater than the recrystallization rate of Nb-500 (Fig.2(b)). Thus, sulfur accelerates the recrystallization while niobium inhibits the recrystallization.

To complete the above results, an annealing treatment has been stopped at the beginning of recrystallization in order to study the first steps of nucleation in the extreme samples (S-0, S-60, Nb-0, and Nb-500). It was shown that the fraction of Cube nuclei increases with the sulfur content and decreases with the niobium content. The sulfur element favors the Cube grain nucleation, the main component after recrystallization, while the niobium element prevents this nucleation and these results are consistent with the recrystallization kinetic.

4. Discussion

The stored energy of cold-rolled samples was estimated from neutron diffraction measurement and Kernel Average Misorientation measurement (Fig. 3a and 3c). In the “S” samples, the stored energy of B, C and S components increases when sulfur content increases, the stored energy of the Cube component remains quasi-constant. In the “Nb” samples, the stored energy of B and S increases with the niobium content and that of C decreases with the niobium content. The stored energy of the Cube orientation is the lowest. By the SIBM (Strain Induced Boundary Migration) mechanism [10], the Cube orientation develops during the recrystallization to the detriment of B, C and S cold-rolling orientations. The increase of stored energy in the “S” sample can be attributed to the MnS precipitates formed in the Fe48%Ni in the presence of sulfur [1]. Furthermore, as reported in the literature, Mn element can hinder the recrystallization process by decreasing the grain boundary mobility [16]. Thus, in the present study, the precipitation of Mn into MnS reduces the detriment effect of Mn on the mobility of grain boundaries and could explain the speed up of the recrystallization kinetic (Fig.2). The variation of stored energy with niobium content can also be explained by the interaction between niobium and other elements in the microstructure and/or its presence in the grain boundary leading to decrease of grain boundary mobility. Consequently the nucleation is prevented by the niobium.

The stored energy difference ($\Delta E_{\text{Cube/other}}$) between the Cube orientation and the main B, C and S cold-rolling components (other) was calculated (Fig. 3b and 3d) to explain the fraction of Cube orientation evolution with sulfur and niobium content after recrystallization. This energy difference is the driving energy for the development of Cube grains. It is shown that the $\Delta E_{\text{Cube/other}}$ increases in the “S” samples when sulfur content increases and decreases in the “Nb” samples when niobium content increases. The increase of $\Delta E_{\text{Cube/other}}$ in the “S” samples promotes the Cube grain formation and growth during the recrystallization,

while the diminution of $\Delta E_{\text{Cube/other}}$ in the “Nb” samples decreases the fraction of Cube grains with the niobium content.

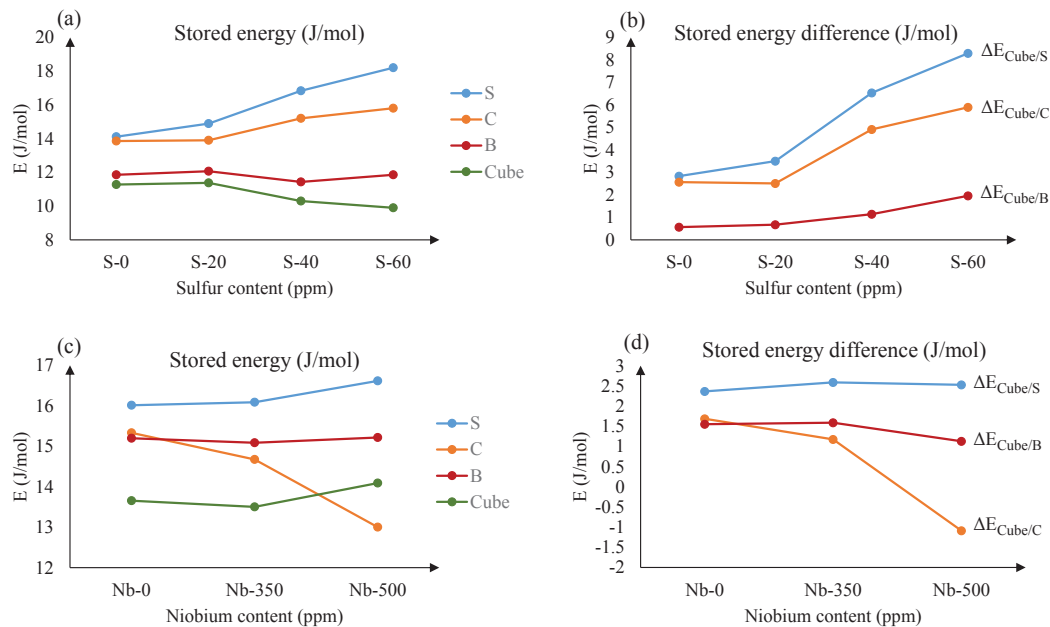


Figure3: (a), (c) Stored energy (J/mol) associated to B, C, S and Cube orientations as a function of sulfur and niobium content, respectively. (b), (d) stored energy difference between Cube orientation and B, C and S orientations and Cube orientation as a function of sulfur and niobium content, respectively.

5. Conclusions

The effect of microalloying elements sulfur and niobium on the formation of the Cube texture in the Fe-48%Ni has been investigated. It was shown that sulfur and niobium microalloying elements favors and inhibits the Cube orientation formation, respectively. It appears that the stored energy difference between the Cube orientation and the B, C and S cold-rolling orientations is the main factor for the development of the Cube orientation. In addition, sulfur accelerates the nucleation and the development of Cube grains while niobium inhibits theses process.

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