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# Industrial Fe-Ni alloys for HTS coated conductor tapes

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**Abstract.** Best of the art of industrial rolling conditions have been developed to get a roughness inferior to 20 nm, improved further if necessary by electrolytic polishing (roughness of 6nm has been reached) or cold rolling. The effects of annealing conditions (temperature and atmosphere) on the cubique texture were analyzed. RX pole figures have been achieved on samples annealed up to 1100°C. Attention has been paid to the rolling texture and to the annealing processes. The rolling texture has been found to be mainly copper-type (C, S and B contributions). Beginning of recrystallization occurred around 500°C and stabilized between 1050°C to 1100°C, depending on the nickel content of the alloy, higher temperature annealing leading to overgrowth of grains. Finally, several samples, rolled and annealed under the appropriate conditions, have been characterized. Pole figure measurements gave the global in-plane and out-of-plane disorientations of our samples which are in-plane 5.5° and out-plane (RD) 4.5° for best samples. EBSD maps have shown the details of the distribution and show no twined {122}<21-2> orientations.

## 1. Introduction

Bi-textured substrate is one of the critical steps for the HTS coated conductor development. The RABiTS (Rolling Assisted Biaxially Textured Substrate) method is the common way to get such substrate. Most researches have focused on the nickel tape for its good texture properties and its cell parameter close to the YBCO one [1-6]. Cheaper substrates like copper rich Cu-Ni and Cu-Fe [7-12] and iron rich Fe-Ni alloys [13] have been proposed and developed. However only one composition in the Ni-Fe system has been studied (Fe<sub>50</sub>Ni<sub>50</sub>) and the effects of different compositions and of alloying elements on texture have not been studied. With the price of the nickel increasing rapidly, we are looking for alloys with less nickel as possible and with alloys used industrially for other applications in order to reduce their price. This study deals with Fe-Ni substrates used industrially as Permalloy but enhanced for a better bitexture and a better surface quality than usually used for magnetic alloys. In this study we present results obtained by RABiTS processing of iron-nickel alloys, recrystallized at various times and temperatures with textures comparable to the ones obtained on pure nickel.

## 2. Experimental

### 2.1. Sample preparation

Three iron-nickel alloys were studied in order to evaluate the possibility to use industrial alloys chosen among low nickel content alloys. We used alloys of composition Fe<sub>62</sub>Ni<sub>33</sub>Cr<sub>5</sub> (M1), Fe<sub>58</sub>Ni<sub>42</sub> (M2) and

Fe<sub>53</sub>Ni<sub>47</sub>(M3) from Imphy alloys. They were provided with a width of 25 mm and different thicknesses (250, 160 and 160 μm respectively) and different surface aspects, roughness of : Ra= 40, 20 and 10 nm respectively were measured by AFM. It is to note that the M1 alloy has the roughness of industrial rollers, the M2 alloy has been rolled with special rollers polished to a good precision in order to achieve this very good roughness. The M3 alloy was industrially electropolished on very long length by the Poligrat™ Company.

More cold rolling was realized on sample M1 in order to enhance the roughness on a Redex rolling mill with very good mirror rollers and the final thickness of this sample was 200 μm and Ra=11 nm, corresponding to final deformation rates of 98.5, 96.1 and 98.5% for M1, M2 and M3 alloys respectively.

## 2.2. Characterizations

X-ray pole figures were performed on a Siefert goniometer mounted on a standard RX generator using a copper tube (K<sub>α1</sub>, K<sub>α2</sub>).

The electron back scattering diffraction (EBSD) measurements were performed using a JEOL 840A SEM at 20kV, at a working distance of about 22 mm and a specimen tilt of 70°. Pictures were imaged on a phosphor screen, viewed by a HAMAMATSU camera and the ARGUS image processor. Channel 5 HKL software was used to index the grains' orientation. A final picture of 400\*400 μm<sup>2</sup> with a measurement step of 4 μm was obtained.

The surface topography and the roughness (in root mean-square, RMS) of samples were explored using a Dimension 3100 Atomic Force Microscopy, AFM (Veeco Inc, Santa Barbara, CA). Measurements were performed in air in tapping mode with tip ARROW-NC-SPL from Nanosensors. These Silicon tips have a curvature tip less than 10 nm, a thickness of 4.6 μm, a length of 160 μm, a width of 45 μm, a resonance frequency of 285 kHz (nominal value: 335) and a force constant of 45 N/m. Images were collected with a resolution of 512 points per line at a scan rate of 1 Hz. Most of them were processed by flattening in order to remove the background slope. Contrast and brightness were adjusted.

## 3. Results and discussion

After cold rolling, the samples were cut in pieces of 2 cm by 1 cm. An X-ray diffraction experiment in  $\Theta$ -2 $\Theta$  scan was performed on each sample. These scans show that there is some orientation of the cubic structure in the 002 direction for all samples before annealing.

Different thermal treatments were applied by heating at various temperatures the sample at a constant speed rate of 10°/min followed by a cooling down at the same rate. A constant gas flow of argon/5%hydrogen was applied all along the heating treatment. At the end of these thermal treatments, X-ray diffraction  $\Theta$ -2 $\Theta$  scans were performed and revealed that only the (111) peak were present in all the samples. The texture of the annealed tapes was realized at these different annealed temperatures together with  $\omega$  (200) and  $\phi$  (111) scans in order to measure the in- and out-of-plane disorientations.

The figure 1 shows some pole figures drawn with an intensity logarithm scale of the (111) reflection obtained after annealing at different temperatures. For the M1 alloy, the cubic (100){001} texture is predominant but a small twin component (122){21-2} still remained in the middle of the pole figure. By increasing annealing temperature, the quantity of the twin component diminishes and is very low for an annealing at 1100°C. The pole figure of the M2 alloy has been measured only for the sample annealed at 1050°C, but it presents a lot of twin components, even at this high temperature. The pole figure of the M3 alloy annealed at 1050°C is free of twin components and the sample annealed at 1100°C presents a very good texture, free of twins, but extra crystals have grown in bad directions and appear as black spots on the pole figure.

The table I gives the values of the disorientations angles (expressed as FWHM) obtained after annealing the three alloys at different temperatures by rotating the sample around the (200) reflection and by scanning the (111) reflection. By increasing the annealing temperature the FWHM values are reduced for both components, the best values have been obtained for the M1 sample annealed at

1100°C and for the M3 sample annealed at 1050°C as above this temperature it presents extra peaks on pole figures.

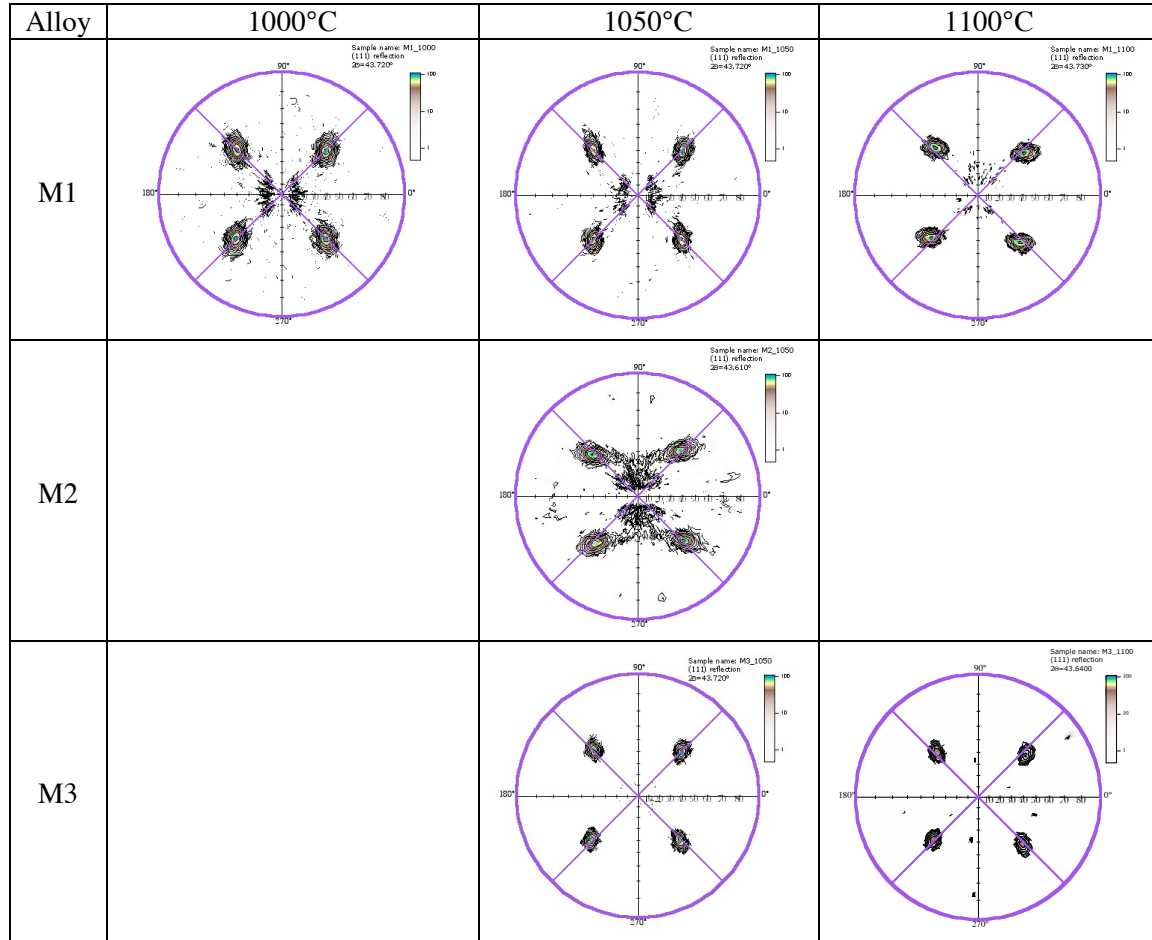


Figure 1: (111) reflection pole figures of the three alloys annealed at different temperatures.

Alloy		800°C	900°C	1000°C	1050°C	1100°C
M1	$\omega(\text{rot})200$			7.3	7.0	<b>5.7</b>
	$\phi(111)$			7.4	6.7	<b>5.5</b>
M2	$\omega(\text{rot})200$				7.1	
	$\phi(111)$				6.9	
M3	$\omega(\text{rot})200$	7.4	6.9	6.8	<b>6.5</b>	6.5
	$\phi(111)$	6.2	5.8	5.4	<b>5.7</b>	5.4

Table 1: FWHM (°) measured on the three alloys annealed at different temperatures.

The textures of the tapes were also characterized by means of the SEM-based EBSD technique. A  $\langle 001 \rangle [001]$  EBSD orientation map of the 3 alloys, annealed at different temperatures, is shown in figure 2. The red parts are the crystals disoriented along the  $\langle 111 \rangle$  direction by more than  $10^\circ$ , the crystals with the cubique texture orientation represent only 96.2% of the studied surface for the M2 alloy annealed at 1050°C. On the contrary the M1 and M3 alloys annealed respectively at 1100 and 1050°C present more than 99% of the crystals with the correct orientation confirming the measurements done by the R-ray pole figures.

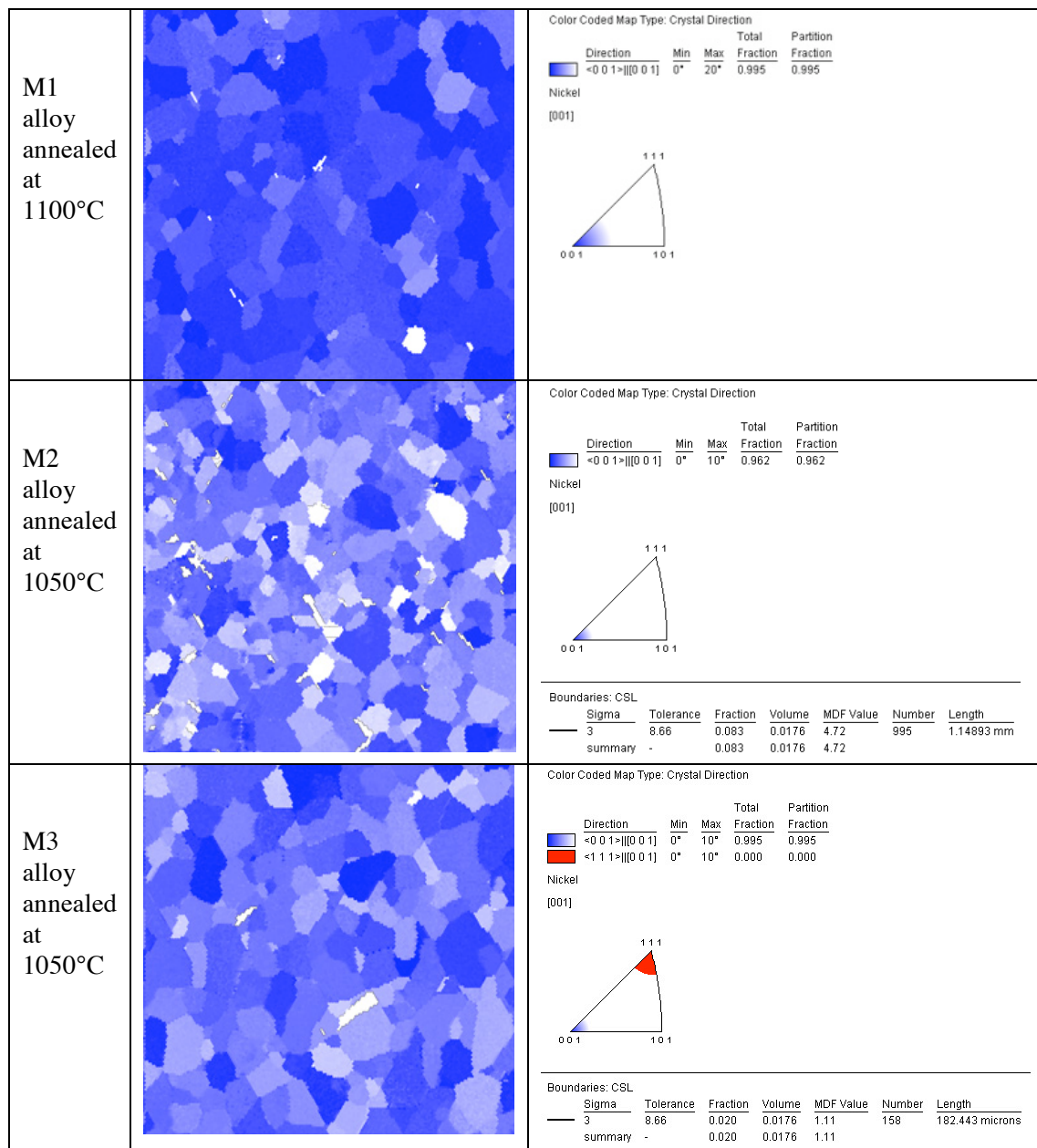


Fig.2:  $\langle 001 \rangle \parallel [001]$  EBSD maps of the three alloys annealed at different temperatures, maps are  $300 \times 300 \mu\text{m}^2$  with a  $2 \mu\text{m}$  step. On the right side are the color maps legends for disorientation angles lower than  $10^\circ$ .

The surface aspect measured by the AFM technique on the three alloys annealed at their best temperature are presented on figure 3. The roughness values don't change from their initial values, the M1 and M2 alloys show typical lines from the rolling process. On the other hand the M3 alloy, which has been electropolished, has a roughness of 6 nm with no lines from the rolling process. This demonstrates that the properties obtained by the electropolishing process performed before the annealing process are maintained during our thermal process. This is mainly due to the combination of the speed of the heating process and of the flowing atmosphere, which is sufficient to change the texture of the grains, but does not change too much the size of the grains and the matter located

between the grains. In a previous study [12], we have shown that the same process performed under vacuum was leading to marked grain' joints whose matter was removed and showed deep valleys between the grains and consequently the roughness was very high.

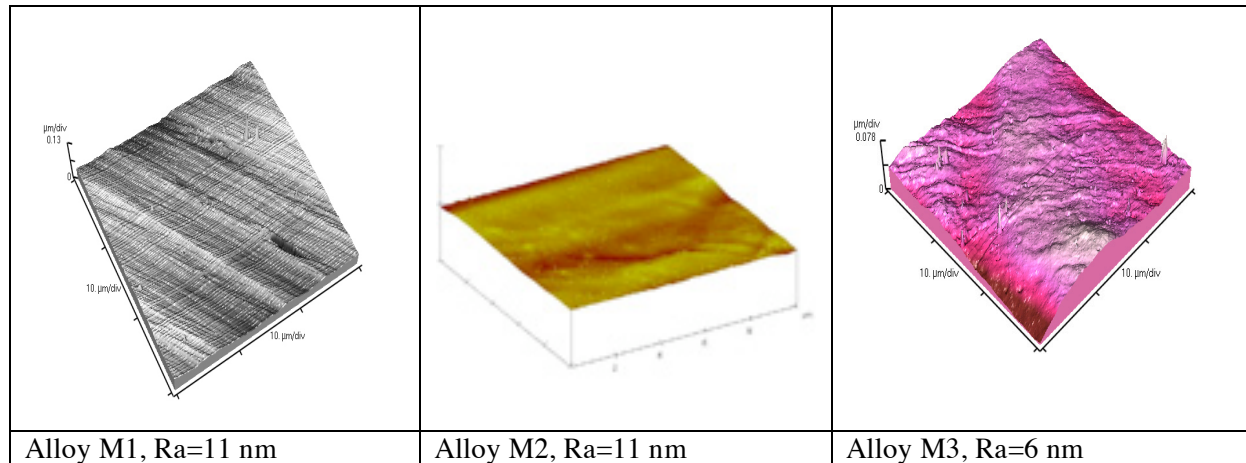


Figure 3: AFM images of the three annealed alloys at their best annealing temperature.

#### 4. Conclusion

By using commercially available Fe-Ni alloys with low nickel content laminated to present good ferromagnetic properties, it is possible to prepare well textured and smooth metallic ribbons in very long length. These substrates presenting magnetic properties can be used in devices such as current limiters and DC current conductors. A very good roughness of some nanometers can be obtained industrially by using good rollers followed by an electropolishing. In the present study, roughnesses of 4 to 6 nm have been measured on long length-laminated ribbons. We have tested three alloys composition,  $\text{Fe}_{62}\text{Ni}_{33}\text{Cr}_5$  (M1),  $\text{Fe}_{58}\text{Ni}_{42}$  (M2) and  $\text{Fe}_{53}\text{Ni}_{47}$  (M3) from Imphy alloys, but with different deformation rates of 98.5, 96.1 and 98.5% for M1, M2 and M3 alloys respectively. The best results have been obtained with the alloys presenting a deformation rate of 98.5%, the other one presenting a lot of twinned crystals was only deformed at 96.1%. The best temperature for annealing between samples M1 and M3 that correspond to different Fe/Ni percentages is 1050°C for the M3 alloy, but slightly above 1100°C for the M1 alloy that contains less nickel. The disorientation angles for the in-plane and out-of-plane orientations are 5.7 and 5.5° for the M1 sample annealed at 1100°C. These values are among the best ones measured on Ni:W substrates. Preliminary chemical depositions tests of  $\text{La}_2\text{Zr}_2\text{O}_7$  have been performed on these samples were successfully done.

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