



## Stress annealed CoFeNbB alloy : A structural study

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### ABSTRACT

Article presents the influence of Nb content on structural properties of stress annealed  $\text{Co}_{21}\text{Fe}_{64-x}\text{Nb}_x\text{B}_{15}$  ( $x = 3, 5, 7$ ) alloys has been studied using differential scanning calorimetry, in-situ length change measurements during annealing and x-ray diffraction. Results show that, increase of Nb content in the alloy increases the stability of the alloy against crystallization and is also responsible for higher elongation of the specimens. Crystallization leads to the formation of bcc Fe-Co nano-granular phase containing Co up to 38 % and affects the spin texture.

### 1. INTRODUCTION

Stress annealing of magnetic materials is known to induce significant uniaxial magnetic anisotropy. In materials consisting of  $\alpha$ -Fe type nanoparticles dispersed in an amorphous matrix, this treatment can result in a strong anisotropy. In FINEMET type alloys (FeSiBNbCu), the stress induced anisotropy is perpendicular ( $K_U < 0$ ) to tensile stress direction [1] and strong ( $\sim 10 \text{ J.m}^{-3}\text{MPa}^{-1}$ ) whereas Co substitution turns it to longitudinal direction ( $K_U > 0$ ) for 20 to 50% Co substituted to Fe [8]. The sign of uniaxial anisotropy coefficient is generally the same as that of the magnetostriction coefficient of the nanograins. However, for some alloys (FeZrB NANOPERM or 80% Co substituted FINEMET) the direction of the anisotropy is opposite to expectations [2]. It seems that this phenomenon is linked to the viscous behaviour of the amorphous phase in the supercooled liquid region, i.e., between the glass transition and crystallisation temperatures ( $T_g$  and resp.  $T_X$ ). To gain insight into this problem, it is necessary to study alloys with different  $T_X - T_g$  values. The influence of Nb

content on structural properties of stress annealed  $\text{Co}_{21}\text{Fe}_{64-x}\text{Nb}_x\text{B}_{15}$  ( $x = 3, 5, 7$ ) alloys has been studied using differential scanning calorimetry (DSC), in-situ length change measurements during annealing and x-ray diffraction (XRD).

### 2. EXPERIMENTAL DETAILS

Ribbons of nominal composition  $\text{Co}_{21}\text{Fe}_{64-x}\text{Nb}_x\text{B}_{15}$  with  $x = 3, 5, 7$  (about 20  $\mu\text{m}$  thick and 10 mm wide) were prepared using a planar flow casting technique on copper wheel. First crystallization peak temperature ( $T_{X1}$ ) was determined using DSC measurements performed at a heating rate of 20  $^\circ\text{C}/\text{min}$ . Specimens were annealed at 450, 500 and 550  $^\circ\text{C}$  with applied stress of 200 MPa for 1 h in the protective atmosphere of flowing Ar. The elongation ( $\Delta L$ ) of the 1 m long ribbons was measured during annealing by means of a Linear Variable Differential Transformer. Recording of  $\Delta L$  and temperature was performed using a PC linked GPIB standard card as a function of time at a rate of 1 point/s during the whole thermal treatment (heating at 10 K/min, plateau at  $T_A$ , cooling at 10 K/min). Cu- $K_\alpha$  XRD measurements were done at room temperature, and were analyzed using pseudo-Voigt line profile to obtain lattice parameter ( $a$ ) and average grain size ( $D$ ) and the volume fraction of the nanograins ( $V_x$ ). Transmission Mössbauer spectra were recorded at room temperature, using  $^{57}\text{Co}:\text{Rh}$  source.

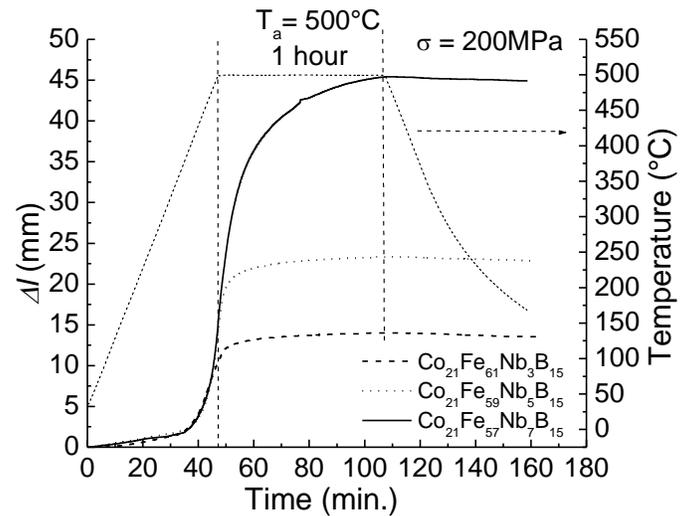
### 3. RESULT AND DISCUSSION

$T_{X1}$  for samples with  $x = 3, 5$  and  $7$  are respectively  $408, 442$  and  $475$  °C, showing that increase of Nb content increases the stability of the alloy against crystallization. Figure 4 shows the elongation ( $\Delta L$ ) for  $\text{Co}_{21}\text{Fe}_{64-x}\text{Nb}_x\text{B}_{15}$  during the same annealing cycle. Perusal of figure 4 shows that, higher the Nb content in the alloy higher is the elongation of the alloy.

$T_{X1}$  for samples with  $x = 3, 5,$  and  $7$  are respectively  $408, 442$  and  $475$  °C, showing that increase of Nb content increases the stability of the alloy against crystallization. Fig. 1 shows the elongation ( $\Delta L$ ) for  $\text{Co}_{21}\text{Fe}_{64-x}\text{Nb}_x\text{B}_{15}$  during the annealing cycle (mentioned above). Perusal of Fig. 4 shows that, higher the Nb content in the alloy higher is the elongation of the alloy.  $T_g$  is deduced from the plot of  $dL/dt$ , which is the viscous flow speed. Below  $T_g$ , this speed is small and equal for all samples (0.5 mm/min). After correction of the thermal dilatation ( $\sim 5 \times 10^{-6} \text{ K}^{-1}$ ), the constant viscosity ( $\sim 0.5 \times 10^{-12} \text{ Pa.s}$ ) is attributed to short range ordering [3].

Maximum flow speed was 60 mm/min for  $x=7\%$  and the viscosity felt down to  $4 \cdot 10^{-9} \text{ Pa.s}$  at  $500$  °C. The higher elongation is observed for the same sample due to the increase of crystallization temperature, enhancing the super cooled liquid region. It is important to note that application of stress modifies the total free energy of the metastable amorphous phase resulting in a lower  $T_g$  compared to measurements performed by DSC.

XRD data shows that the crystallization of the specimens starts after annealing at  $450$  °C with  $200$  MPa stress (except for the specimen with  $x=7$ , where crystallization starts after annealing of the specimen after  $500$  °C) showing a co-existence of the crystalline phase and the residual amorphous matrix.



**Figure 1: Elongation ( $\Delta L$ ) for  $\text{Co}_{21}\text{Fe}_{64-x}\text{Nb}_x\text{B}_{15}$  alloys after annealing at  $500$  °C**

Table 1 depicts the Scherrer's crystallite size ( $D$ ), lattice parameter ( $a$ ) and volume fraction of the nano-grains ( $V_x$ ) obtained by fitting the XRD data. In the early stage of crystallisation ( $450$  °C), lattice parameters are rather high which suggests the precipitation of bcc Fe nano-granular phase with rather low Co content (15%) and a substantial lattice expansion that can be due to the presence of Nb solute atoms. As the annealing temperature increases, lattice parameters become consistent with bulk values and indicate a Co content of 15 - 20%, 30%, and 35% for  $x=3, 5,$  and  $7$  respectively. As Nb content in the specimen increases, for same annealing temperature ' $D$ ' is systematically smaller for the sample containing higher Nb content.  $V_x$  shows similar behavior for the studied samples. Behavior of  $D$  and  $V_x$  can be understood in terms of increase of  $T_{X1}$  values with increase of Nb content in the alloy.

**Table 1: Thermodynamical ( $T_x$ ,  $T_g$ ) and structural ( $a$ ,  $D$  and  $V_x$ ) parameters for stress annealed  $\text{Co}_{21}\text{Fe}_{64-x}\text{Nb}_x\text{B}_{15}$  specimens.**

Nb (at.%)	$T_x/T_g$ ( $^{\circ}\text{C}$ )	Ann. Temp ( $^{\circ}\text{C}$ )	$\Delta L$ (mm)	$a$ (nm) $\pm 0.0001$	$D$ (nm) $\pm 2$	$V_x$ (%) $\pm 2$
3	408 342	450	10.4	0.2869	30	45
		500	14.1	0.2867	35	55
		550	17.9	0.2867	38	55
5	442 384	450	12.9	0.2870	15	15
		500	13.4	0.2869	16	45
		550	27.2	0.2864	15	60
7	475 397	450	10.7	-	-	-
		500	45.4	0.2862	10	25
		550	>50	0.2862	15	55

#### 4. CONCLUSION

In conclusion, higher Nb content in the alloy increases the stability of the alloy against crystallization and is also responsible for the obtained higher elongation of the alloy through the enhancement of the super cooled liquid region. Crystallization of the studied alloys affects their spin texture in the amorphous matrix showing that higher the elongation, the more spin texture is out of plane.

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