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Nanocrystallization studies in Co-rich amorphous alloys

I. Betancourt [a], M. Jiménez [b], S. Aburto [b], V. Marquina [b], R. Gómez [b], M.L. Marquina [b], R. Ridaura [b], M. Miki [a], R. Valenzuela [a,*]

[a] *Instituto de Investigaciones en Materiales, National University of Mexico, Mexico*
[b] *Facultad de Ciencias, National University of Mexico, Mexico*

Abstract

Nanocrystallization from Vitrovac® amorphous ribbons was studied by Mössbauer spectroscopy, transmission electron microscopy and differential scanning calorimetry. Results are interpreted in terms of a crystallization of Co, Co_2B, and Co_3B, leading to an increase in Fe content in the residual amorphous phase, which in turn results in an increase in hyperfine field and Curie temperature.

Nanocrystallization in Fe–Nb–Cu–B–Si amorphous alloys (crystallization of grains smaller than 15 nm) leads to a considerable softening in magnetic properties [1]. Co-rich amorphous alloys have also shown a five-fold increase in initial magnetic permeability upon controlled thermal annealing at temperatures considerably lower than the crystallization temperature [2]. In this paper, a study of nanocrystallized Co-rich alloys is presented as a contribution to the understanding of this phenomenon, by using Mössbauer spectroscopy (MS), transmission electron microscopy (TEM) and differential scanning calorimetry (DSC).

Vitrovac® 6025 amorphous ribbons of nominal composition $Co_{66}Fe_4Mo_2B_{16}Si_{12}$, kindly supplied by Vacuumschmelze GmbH, Germany, were annealed at 400°C in an inert (Ar) atmosphere, for various times in the 5–60 min range. They were analyzed in a standard Mössbauer apparatus at room temperature. Small disks (3 mm diameter) of the alloy in the as-quenched state were thinned by electropolishing in an acid solution, then submitted to the same annealings in a Thermoanalyst 2000 system, with a standard DSC cell. DSC investigation was carried out in this apparatus.

Mössbauer spectra showed a distribution in the hyperfine field h for the as-quenched sample as well as for the annealed alloys. A reasonable fit to a single six-line pattern was obtained by assuming a continuous distribution of h, leading to average values. Average hyperfine fields showed a small increase for short annealing times t, followed by a stabilization for longer times, Fig. 1.

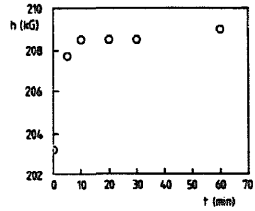

Fig. 1. Average hyperfine field as a function of annealing time at 400°C.

TEM observations confirmed the crystallization of small grains in the 12 nm range for annealings at $t = 10$ min. The intensity of diffracted rings in electron diffraction patterns was sufficient to allow an identification of the present phases only for $t > 10$ min, Fig. 2. The phases associated with the measured interplanar distances appear in Table 1.

DSC measurements on samples annealed at various temperatures exhibited an increase in Curie temperature

Table 1
Interplanar distances in observed diffraction patterns

d_{ob} (Å)	(hkl)	Phase	d (Å) [a]
3.25	101	Co_2B	3.23
1.95	211	Co_3B	1.94
1.67	004	Co_3B	1.66
1.46	102	α-Co	1.48
1.38	112	β-Co	1.37
1.28	312	Co_3B	1.30
1.05	311	β-Co	1.06

[a] JCPDS files.

* Corresponding author. Fax: +52-5-622 457.

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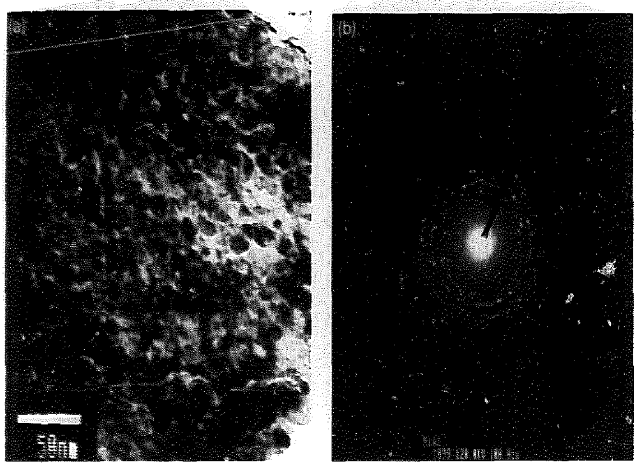

Fig. 2. TEM micrographs of alloys annealed 20 min at 400°C. (a) Image, (b) diffraction pattern.

T_C. Such an increase in T_C with annealing time could be explained by structural relaxation [3]. This phenomenon is characterized by a ln t dependence of the change in a given property [4]; plotting DSC results in the form T_C vs. ln t, Fig. 3, shows that for $T < 350$°C, the main contribution to the change in T_C can effectively be explained by structural relaxation. However, for $T > 350$°C, there is an additional increase in T_C, and this relationship is not followed any longer. TEM results (Table 1) can explain these results, since at this crystallization stage, all identified crystals contain no Fe. During Co-rich crystallization phases, the Fe relative content in the residual amorphous phase increases and by comparison with CoFeBSi system [5], the Curie point should increase. The absence of Fe crystallized phases in CoFeB systems with Fe contents lower than 8% has been previously reported [6].

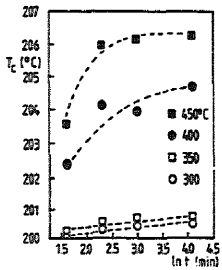

Fig. 3. Curie temperature measured by DSC, as a function of ln t.

Mössbauer results are consistent with this interpretation since the increase in Fe content in the residual amorphous phase during crystallization leads to an increase in the number of Fe–Fe interactions, and therefore to an increase in hyperfine fields.

These results are therefore congruent with the current interpretation of magnetic softening [7], which assumes that the presence of magnetic nanocrystals with size smaller than the exchange length results in a significant decrease of macroscopic anisotropy.

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