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Microwave power absorption analysis in the devitrification process of Co-based amorphous ribbons

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ABSTRACT

Melt-spun $Co_{66}Fe_4B_{12}Si_{13}Nb_4Cu$ soft magnetic ribbons were devitrified at low annealing temperatures (623 K), for times 5–20 min. Microwave power absorption measurements at 9.4 GHz (X-band) were carried out in two geometries. In geometry 1, the ribbon's plane was oriented parallel to AC magnetic field. For the orientation 2, the ribbon's plane was normal to the AC magnetic field. In both cases, the ribbon's axis was parallel to the DC magnetic field. For both orientations, two absorptions were observed: the first corresponds to a low field microwave absorption (LFA) centered in zero dc magnetic field, and a higher field absorption corresponding to the ferromagnetic resonance (FMR). In the geometry 1, a single FMR spectrum was observed for all the samples, with a shift in resonant field as annealing increased. For geometry 2, evidence of the superposition of two FMR signals was observed. FMR spectra are therefore due to a combination of two different magnetic phases corresponding to the amorphous matrix and nanocrystallites. Deconvolution calculations were carried out on FMR spectra to separate the contributions. Their behavior as a function of annealing time was in good agreement with the magnetic softening, also obtained with LFA results. The differences in microwave absorption, for both geometries, can be explained by differences in the electromagnetic wave propagation volume.

1. Introduction

Nanocrystallized ribbons obtained by a controlled crystallization of amorphous alloys are an important group of soft magnetic materials [1]. These materials typically consist of nanocrystallites homogeneously distributed in the soft amorphous matrix. Due to the significant magnetic softening associated with the process, these materials are useful in a wide range of applications [2].

Both resonant and non-resonant microwave power absorption (MPA) measurements [3] are becoming useful techniques to gain insight into the structure of many magnetic materials. In this paper, we present MPA obtained on Co-rich amorphous ribbons in different nanocrystallization states. By using two different measuring geometries, evidence of the superposition of two ferromagnetic resonance (FMR) spectra were detected and resolved by a deconvolution calculation. It is shown that these spectra can be attributed to the two components of the nanocrystallized ribbons: nanocrystals and amorphous matrix. Non-resonant low field absorption (LFA) results exhibited clear evidence of the magnetic softening process as a function of the annealing time.

2. Experimental

$Co_{66}Fe_4B_{12}Si_{13}Nb_4Cu$ ribbons obtained at a tangential roll speed of 40 m/s were devitrified at a low annealing temperature (623 K) for times of 5, 10, 15 and 20 min. Their initial amorphous state was checked by X-ray diffraction. Transmission electron microscopy (TEM) studies were carried out with a JEOL JSM-5600LV microscope. FMR measurements were made on samples 3 mm wide and 22 μm thick, using a Jeol JES-RES3X spectrometer operating at 9.4 GHz (X-band). The power of the ac signal was 1 mW. The MPA spectra were obtained by the dc magnetic field modulation technique, with a modulation frequency of 100 kHz. LFA measurements were obtained by means of a JEOL ES-ZCS2 zero-cross sweep unit, which allows the detection of small fields close to zero, and the compensation of any electromagnet remanence. All spectra were taken at room temperature with a standard deviation of the measured field of less than 2×10^{-5} T. Measurements of magnetization were carried out in a LDJ 9600 vibrating sample

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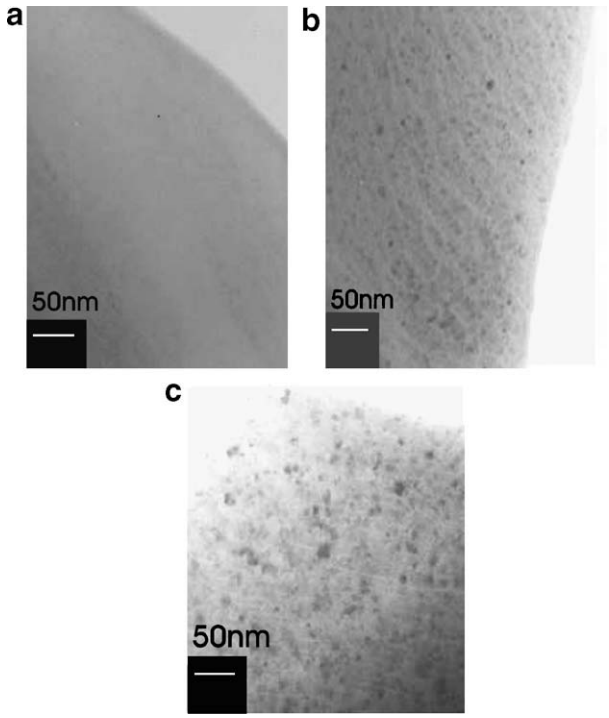

Fig. 1. TEM micrograph of $Co_{66}Fe_4B_{12}Si_{13}Nb_4Cu$ ribbons for (a) as-cast and annealed at 623 K for (b) 10 min and (c) 15 min.

magnetometer (VSM) at room temperature. As explained in the following section, two different measuring geometries were used.

3. Results and discussion

The nanocrystallization process was followed by TEM for each annealing time. Fig. 1(a) shows the as-cast alloy, (b) and (c) show the TEM images of 10 and 15 min of annealing, respectively. The presence of nanocrystals is clear in the last two micrographs.

Two geometries were used for MPA measurements. For geometry '1', the ribbon's plane was parallel to the AC field, with the ribbon's axis parallel to the DC field. For geometry '2', the ribbon's plane was perpendicular to the AC field, with the ribbon's axis parallel to the DC field. For both orientations, two absorptions were observed: the first corresponds to a low field microwave absorption (LFA) centered in zero DC magnetic field [4,5] and the second exhibited a high DC field absorption corresponding to ferromagnetic resonance (FMR).

For geometry 1, all the samples (both as-cast and annealed) exhibited a single FMR spectrum with a small shift in resonant field as a function of annealing time, t_{ann}, see Figs. 2(a) and (b). LFA spectra, expanded in Fig. 2(c) and plotted as a function of t_{ann} in Fig. 2(d) showed significant variations in the separation between maximum and minimum, which we call ΔH_{LFA}, for each annealing. Since this parameter is directly associated with the anisotropy field [6], it can be observed that there is initially a small hardening (t_{ann} = 5 min), followed by a magnetic softening that is maximum for about t_{ann} = 10 min. This minimum is in good agreement with the minimum observed in H_{res}, Fig. 2(b).

Fig. 3 shows the MPA results with geometry 2. In this geometry, the ribbon's plane was normal to the AC magnetic field and the ribbon axis was parallel to the DC magnetic field. For all the samples, both annealed and as-cast, FMR spectra showed an additional wide absorption at resonance fields lower than the one corresponding to

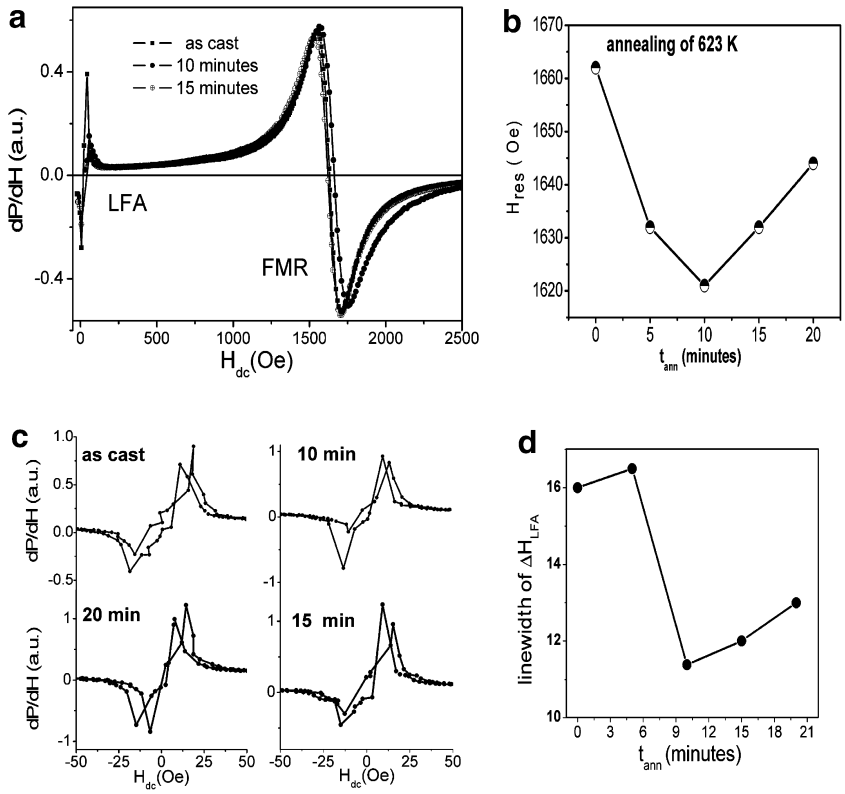

Fig. 2. (a) FMR spectra of Co-rich amorphous ribbons with different nanocrystallization stages. (b) The effects of annealing time on H_{res}, (c) and (d) Effects of annealing time on LFA signal.

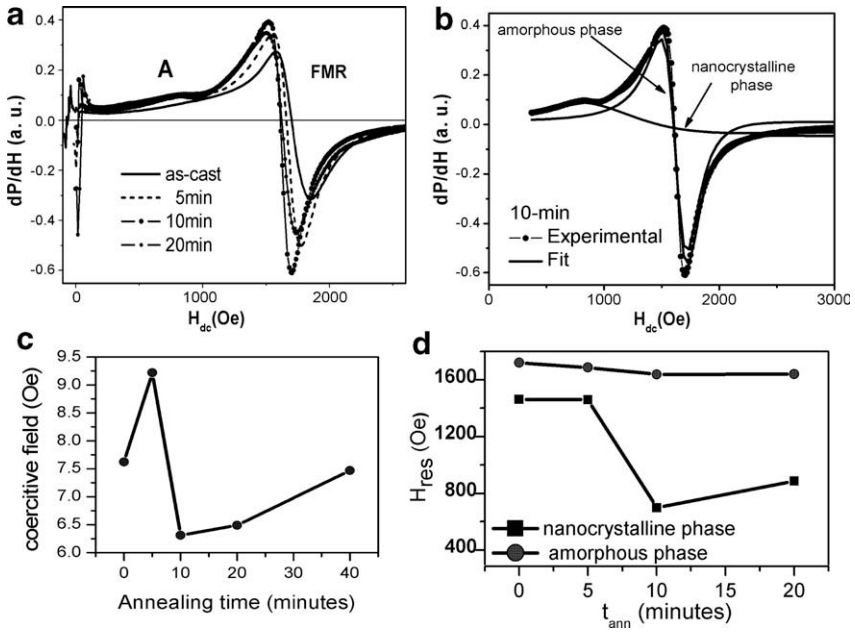

Fig. 3. (a) FMR spectra of amorphous ribbons with different nanocrystallization stages. (b)Shows deconvolution of FMR spectra at 10 min of annealing, The effects of annealing time on (c) coercivity field (H_c) and (d) H_{res}.

the main signal, marked by 'A' in Fig. 3(a). It can be observed that this absorption tends to increase as t_{ann} increases, and can obviously be associated with the appearance of the nanocrystallized phase. The FMR signals were resolved by a deconvolution calculation. We show the results obtained for t_{ann} = 10 min in Fig. 3(b). By means of the fitting, we can follow the dynamics of nanocrystallization for the two phases, as shown in Fig. 3(d). The resonant field for the nanocrystallites decreases as the annealing time increases reaching a minimum for t_{ann} = 10 min. For the amorphous matrix, H_{res} shows a small, monotonous decrease as t_{ann} increases. The coercive field, as obtained from VSM experiments, is in good agreement as shown in Fig. 3(c).

The presence of an additional signal for the as-cast sample seems odd, since no crystals were detected by X-ray analysis. However, it can be explained by the high sensitivity of FMR experiments, capable of detect a short range arrangement which constitutes the nanocrystallite precursor. In view of our results, such sensitivity appears to be modulated by the different experimental geometries.

4. Conclusions

By using a particular measuring geometry (geometry 2) which maximizes the volume in contact with the microwave propagation, it was possible to obtain evidence of the superposition of signals from the components of the nanocrystallized samples. A deconvolution process allowed the study of the separate spectra and the influence of annealing time on both, in good agreement with results obtained by LFA experiments on both geometries.

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