CBPF-NF-051/94

NMR Study of the Nanocrystalline Alloy $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$

D. Aliaga-Guerra[a,b], *L. Iannarella*[a], *M.B. Fontes*[a], *I. Škorvánek*[c,d] and *A.P. Guimarães*[a,d]

[a]Centro Brasileiro de Pesquisas Físicas - CBPF
Rua Dr. Xavier Sigaud, 150
22290-180 Rio de Janeiro/RJ, Brasil

[b]Facultad de Ciencias, Universidad
Nacional de Ingenieria,
P.O. Box 31-139 Lima, Peru

[c]Institute of Experimental Physics
SAS, 04353 Kosice, Slovakia

[d]On leave at the Massachusetts
Institute of Technology,
Cambridge MA 02139, USA

Abstract

Nanocrystalline $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ alloys were studied with spin echo NMR at 4.2 K, from 15 to 100 MHz. Several lines are observed, with signals from domains and domain walls. Signals at 50-90 MHz appear to arise from ^{93}Nb nuclei in the amorphous matrix and in the interface of the crystallites.

Key-words: Soft magnetic amorphous systems; Controlled crystalization; Nanometric crystals; NMR spin echo.

Presented at the International Conference of Magnetism, Warsaw (1994); to be published in J. Mag. Mag. Mat.

Nanocrystalline FeCuNbSiB alloys prepared by controlled crystallization of initially amorphous ribbons [1] have attracted much attention in recent years. The alloy of standard composition $Fe_{73.5}\ Cu_1\ Nb_3\ Si_{13.5}\ B_9$ has been studied using a number of experimental techniques, in order to understand better the relationship between the soft magnetic properties and the microstructure of this material. In addition to the various methods already applied for this purpose, such as TEM, X-ray Diffration, and Mössbauer Spectroscopy, NMR seems also very appropriate because of its sensitivity to the changes in local atomic environments. NMR has in particular the potentiality of studying the environment of Nb atoms.

In this paper we report the first NMR results on this alloy system. The structure of the NMR lines identified with the ^{57}Fe and ^{93}Nb resonances gives us information on the distribution of these elements in the amorphous and nanocrystalline regions of the investigated alloy.

The samples were prepared by melt spinning and were heat treated under an Argon atmosphere.

The NMR spectra were obtained with an automated NMR spectrometer. The shape of the spectra is extremely dependent on the rf-power and on the related pulse width; typically 500 ns-wide pulses were used.

The present NMR results, obtained at 4.2K, show:

1) In the range 10-50 MHz (Fig. 1), the spectra for the as-cast amorphous alloy show four broad sets of lines around 21, 26, 36 and 40 MHz. Some signals are also present at 14 MHz.

The spectra for the sample submitted to the optimum heat treatment (OHT sample: 550^0C for 1 hour) is more complex, with many narrow lines as a consequence of the crystallization, and new lines in the range of 29-34 MHz. Also the lines around 14 and 40 MHz are enhanced.

According to the previous Mössbauer studies on the same system [2, 3] and the NMR on Fe_3Si and Fe-rich off-stoichiometric Fe_3Si [4], most of the lines in the range 20–44 MHz can be attributed to the different environments of ^{57}Fe in the nanocrystal and residual amorphous phases, with a broad hyperfine field distribution between 14.5 and 31.9 T (for ^{57}Fe $\gamma/2\pi = 0.13757$ MHz/kG). The lower frequency lines (14 – 16 MHz) may correspond to Fe environments with higher metalloid concentration (Si+B) or to B resonances.

2) In the high frequency range (50-100 MHz), we observe NMR resonances characteristic of well resolved quadrupolar interactions (Fig. 2). This fact excludes ^{57}Fe and ^{29}Si nuclei because both have I=1/2. Also Cu resonances are not possible because there is no evidence for the simultaneous occurrence of the lines for the two isotopoes ^{63}Cu and ^{65}Cu in a 2/1 proportion. ^{11}B resonances in Fe-borides are expected at lower frequencies. Therefore, we attribute these signals to ^{93}Nb with different environments.

The well defined structures observed in the spectrum for the as-cast alloy indicate that short range order around Nb sites is present even in the amorphous state. The OHT sample seems to preserve the dispersion which exists in the amorphous state, avoiding the tendency of clustering.

This conclusion is supported by a study in which the rf excitation conditions were changed, as is shown in Fig. 3. The main feature is that in fact each spectrum shown in Fig. 2 is composed of two different ^{93}Nb resonances; one of them (in the high frequency

side) disappears as the rf-power is strongly attenuated. The same phenomenon is also observed with the ^{57}Fe resonances, where lines in the range 30-44 MHz disappear as the rf is attenuated. As these ^{57}Fe resonances correspond to those observed in the Mössbauer spectra, which show predominantly nuclei in domains, we tentatively attribute these resonances to Fe and Nb atoms linked with the nanocrystallites, the Nb atoms being located at the interface with the amorphous part of the alloy. The remaining ^{93}Nb resonance around 75 MHz may come from this amorphous state with lower Fe concentration. More detailed studies are in progress. These includes the changes of the lineshape with the rf-attenuation which may allow to separate signals coming from domains from those corresponding to domain walls. We also intend to identify ^{63,65}Cu resonances and try to know how clusters of Cu atoms catalyze nucleation of DO_3 crystallites [5].

Figure Caption

Fig. 1. Spin-echo spectra (mainly from ^{57}Fe) of the as-cast (top) and annealed FeCuNbSiB samples.

Fig. 2. ^{93}Nb NMR spectra of the as-cast (top) and annealed FeCuNbSiB alloys.

Fig. 3. ^{93}Nb NMR spectra of the as-cast (left) and the OHT FeCuNbSiB samples obtained under two different excitation conditions.

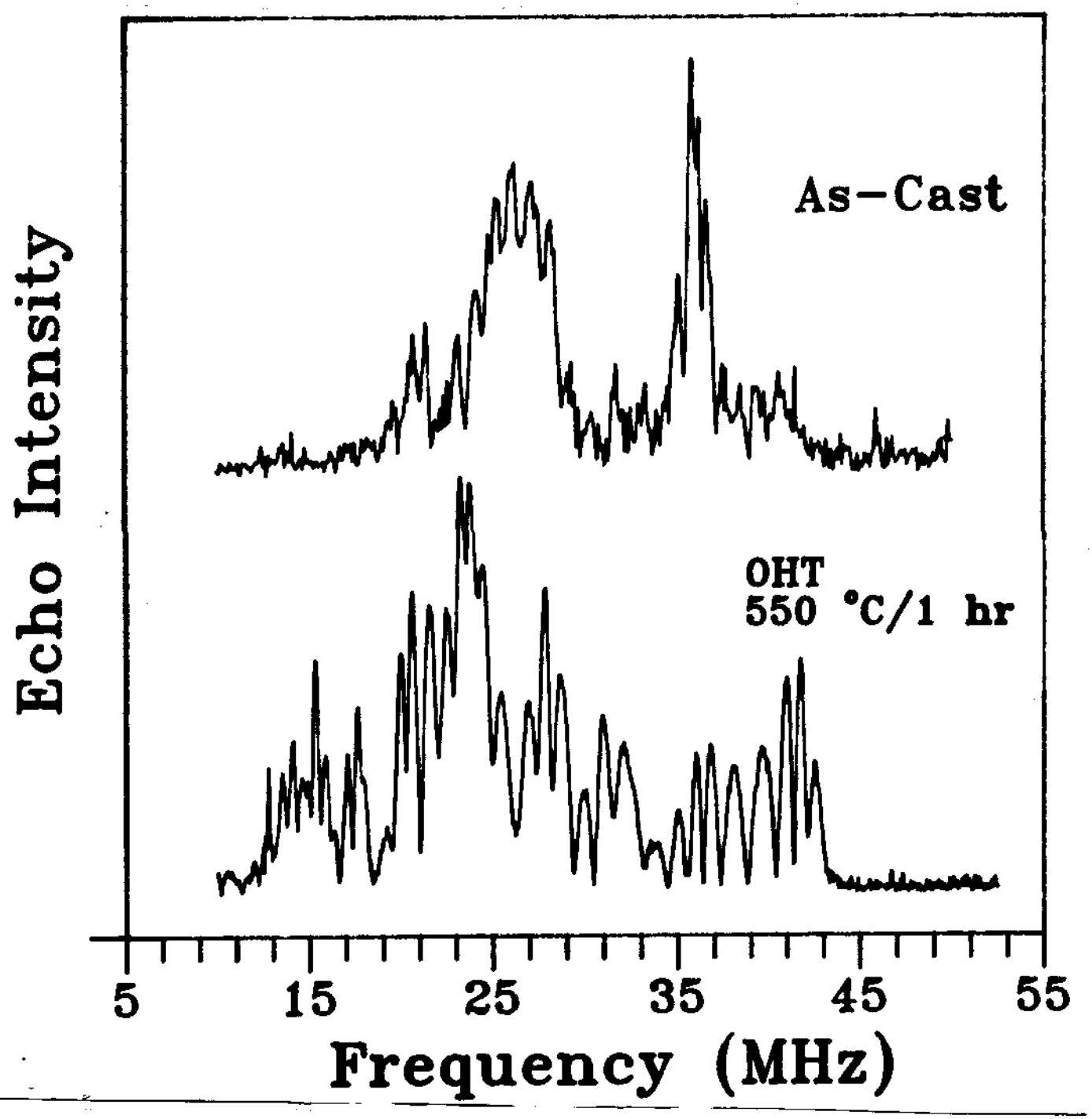
Echo Intensity
As–Cast
OHT
550 °C/1 hr
5
15
25
35
45
55
Frequency (MHz)

FIG. 1

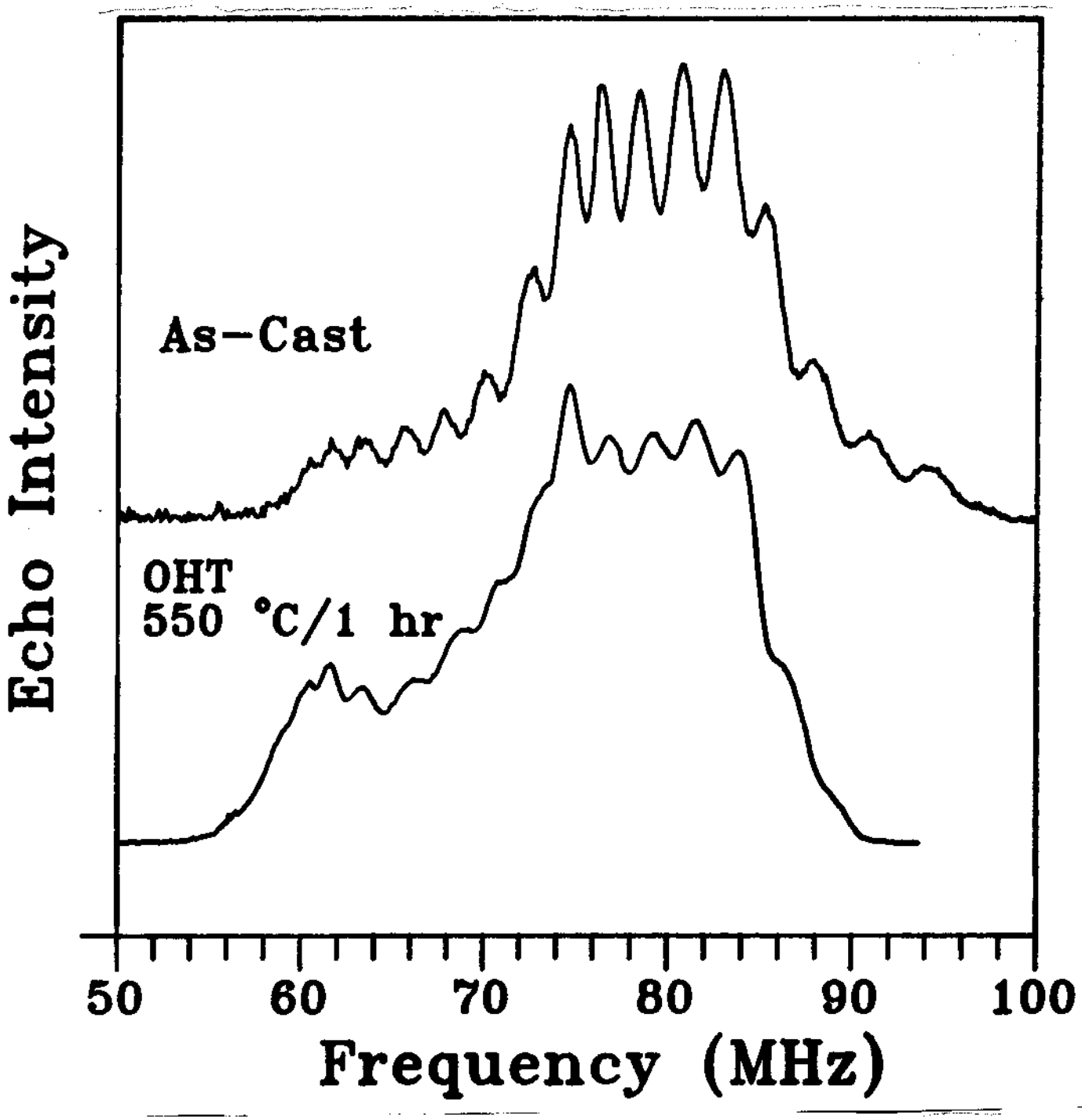
Echo Intensity
As–Cast
OHT
550 °C/1 hr
50
60
70
80
90
100
Frequency (MHz)

FIG. 2

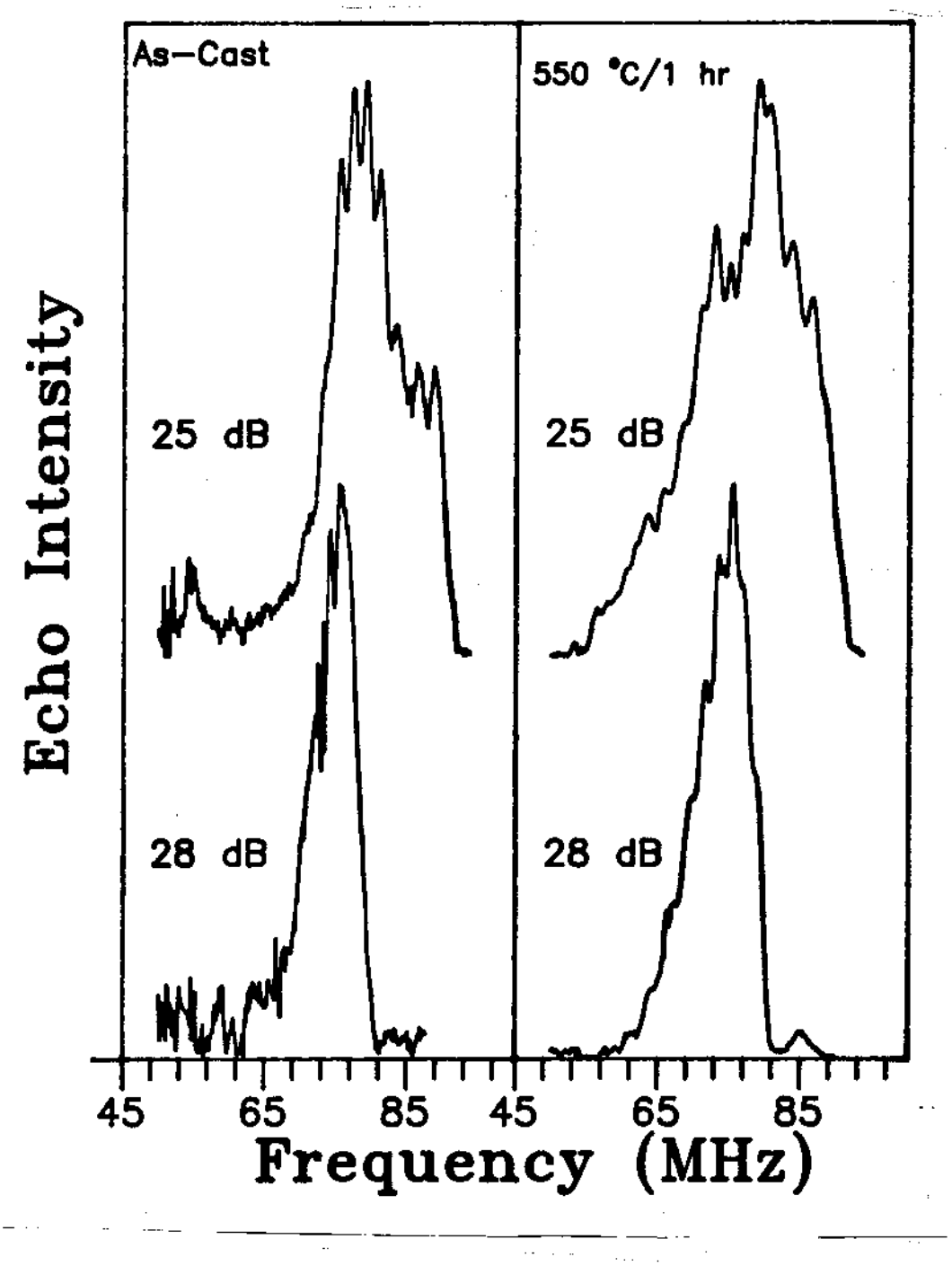
As–Cast
550 °C/1 hr
25 dB
25 dB
28 dB
28 dB
Echo Intensity
45
65
85
45
65
85
Frequency (MHz)

FIG. 3

References

[1] Y. Yoshizawa, S. Oguma and K. Yamauchi, J. Appl. Phys. **64** (1988) 6044.

[2] T. Zemcik, Y. Jirásková, K. Záveta, D. Eckert. J. Schneider, N. Mattern and D. Hesske, Materials Lett. **10** (1991) 313.

[3] X.Z. Zhou, A.H. Morrish, D.G. Naugle and R. Pan, J. Appl. Phys. **73** (1993) 6597.

[4] T.J. Burch, T. Litrenta and J.I. Budnick, Phys. Rev. Lett. **33** (1974) 421.

[5] J.D. Ayers, V.G. Harris, J.A. Sprague and W.T. Elam, IEE Trans. Magn. **29** (1993) 2664.