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Quadrupole Distributions of the New Amorphous System a-Sc_3Fe*

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Abstract

Amorphous Sc-rich FeSc alloys have been prepared by quenching from the melt. The quadrupole splitting distributions for amorphous Sc_3Fe were determined from Mössbauer spectra: the zero-field distribution has a Gaussian form. The analysis of Mössbauer spectra taken in applied magnetic field show that both positive and negative electrical field gradients (EFG) are present. The crystallization process has also been investigated. Heat treatment of the amorphous alloys causes crystallization first into a metastable, and then into stable phases.

Introduction

Recently the formation of amorphous (a-) Fe-rich alloys $Fe_{1-x}Sc_x$ over the range x = 0.09 to 0.11 by single roller quenching has been reported /1/. Also for Sc-rich FeSc, the major requirments of glass formation i.e. low lying melting point and differing atomic radii are satisfied. (The phase

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diagram of FeSc shows a low lying eutectic at about x = 0.8. /2/) Over the composition range x = 0.75 to 0.80, it was found possible to produce amorphous alloys by rapid quenching from the melt. In the present work we report on Mössbauer quadrupole splitting (QS) measurements on amorphous and crystallized Sc_3Fe alloys.

Experimental

a-Sc_3Fe samples were prepared by splat-cooling with levitation melting using a vaccum better than $5*10^{-5}$ Torr. The resulting splats of ≈ 45 μm thickness and 3 cm diameter were amorphous as indicated by the lack of crystalline peaks in x-ray diffraction. The Fe and Sc used to make the alloys were of 99.99 % purity. In order to investigate the crystallization behaviour, the samples were heated in sealed quartz tubes ($< 10^{-4}$ Torr). Mössbauer measurements were performed with a 10 mCi ^{57}Co in Rh source.

Results and Discussion

Fig.1 shows (a) the Mössbauer spectrum and (b) corresponding quadrupole splitting distribution P(QS) for a-Sc_3Fe at T = 4.2 K in zero field (I). The P(QS) was determined using the LeCaër and Dubois /3/ program. Good fits to the asymmetrical spectrum were obtained by assuming a linear relation between QS and the isomer shift (IS) as suggested from the results for Fe-rich FeSc alloys /1/:

$$IS = -0.06\ QS - 0.1.$$

The average quadrupole splitting ($\langle QS \rangle$ = 0.6 mm/s) ist not too different from that found for amorphous Zr-rich ZrFe (0.52 mm/s) /4/ and ZrNi (0.51 mm/s) /5/ alloys, but the form of P(QS) is different. The P(QS) found from the histogram can be described as a normal distribution of the form:

$$P(QS) = (1/(2\pi)^{\frac{1}{2}}\sigma)\exp-((QS-\langle QS \rangle)/(2\sigma^2))$$

with σ = 0.25 mm/s and $\langle QS \rangle$ = 0.60 mm/s. Deviations from the

normal distribution were found for small QS. Information about the sign of EFG and thus about the local symmetry of the Fe sites in Sc_3Fe can be obtained by Mössbauer measurements in external magnetic field. For the analysis of such spectra we assumed P(QS) to be a superposition of two normal distributions of the form:

$$P(QS) = (1-P)(1/(2\pi)^{\frac{1}{2}}\sigma)\exp-((QS+\langle QS\rangle)/(2\sigma^2)) + P(1/(2\pi)^{\frac{1}{2}}\sigma)\exp-((QS-\langle QS\rangle)/(2\sigma^2))$$

with $0 \leq P \leq 1$, and a single value of the asymmetry parameter η. Spectra have been simulated using this form with the Blaes et al. /6/ program for the line shape with mixed magnetic dipole and electric quadrupole interactions for an isotropic EFG texture. A first-approximation fit was found for $P = 0.4$ and $\eta = 0$, shown as (II) in Fig. 1(a) and (b). Thus both positive and negative EFG are present. Presently we are working on improving these results to obtain more detailed information.

The crystallization temperature determined from DSC (differential scanning calorimetry) measurements with a heating rate of 20 K/min was found to be T = 782 K. Crystallization takes place first by the formation of a metastable phase whose structure is sensitive to the condition of heat treatment. Fig. 2(a) shows a typical spectrum (I) from a crystallized sample together with the distribution P(QS), Fig. 2(b), where we find $\langle QS\rangle = 0.28$ mm/s. An analysis of the x-ray diffraction pattern shows several phases, but allows only a clear identification of hexagonal Sc precipitates. In the stable crystalline state, two precipitates with the following lattice parameters were obtained: (1) hexagonal Sc with a = 0.33 nm, c = 0.52 nm, and (2) the intermetallic compound Fe_2Sc (C14 type) with a = 0.49 nm, b = 0.78 nm. Usually the spectrum of Fe_2Sc consists of the superposition of two sextets with relative intensity 3:1 /7/, but that of the Fe_2Sc obtained from crystallizing Sc_3Fe

is much different: Fig. 2(a), spectrum (II). Such differences can be expected from deviations from stoichiometric composition as is indicated by the difference in lattice parameter b from that reported in /7/.

Conclusion

We have shown that the Sc-rich ScFe alloys can be prepared in an amorphous state by quenching from the melt. Amorphous Sc_3Fe has been studied by ^{57}Fe-Mössbauer spectroscopy and it was found that the form of P(QS) ist different from that found in other similar amorphous metal-metal alloys (ZrNi, ZrFe). The distribution P(QS) was found to be Gaussian, and from in-field measurements, both signs of QS were found to be present. The crystallization process was followed by DSC, Mössbauer and x-ray studies. An intermediate metastable phase and stable crystalline phases were found. The end-product of crystallization consists of hexagonal Sc phase and a second intermetallic compound.

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References

/1/ Day R.K., Dunlop J.B., Foley C.P., Ghafari M., Pask H., Solid State Commun. 56, 843 (1985).

/2/ Kubaschenski O., *Iron Phase Diagrams*, Berlin, Springer Verlag, 1982.

/3/ LeCaër G., Dubois J.M., J. Phys E 12, 1083 (1979).

/4/ Ghafari M., Gonser U., Wagner H.G., Naka M., Nucl. Instrum. Methods 199, 197 (1982).

/5/ Ghafari M., Thesis Universität Saarbrücken 1982.

/6/ Blaes N., Fischer H., Gonser U., Nucl. Instrum. Methods, B9, 201 (1985).

/7/ Smit P.H., Buschow K.H.J., Phys. Rev. B 21, 3839 (1980).

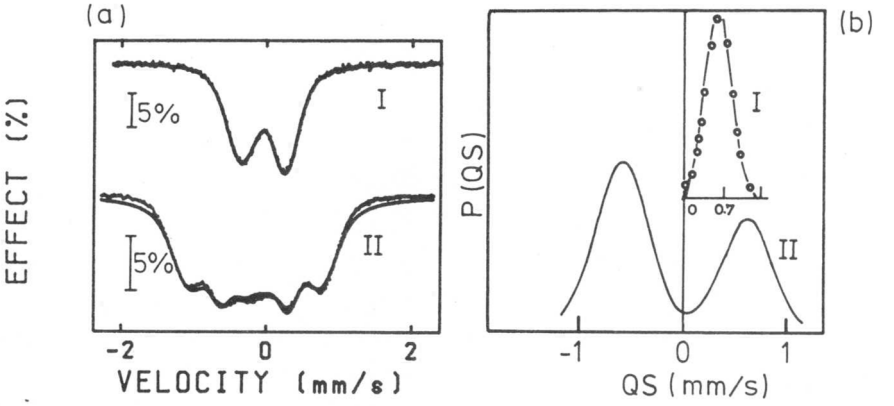

Figure 1. (a) Spectra and (b) Quadrupole distributions for a-Sc_3Fe at T = 4.2 K. (I): Zero field and (II): H_{ext} = 5 T, perpendicular to gamma ray. In (b-I), the results (solid line) is compaired to the normal distribution (shown as circles).

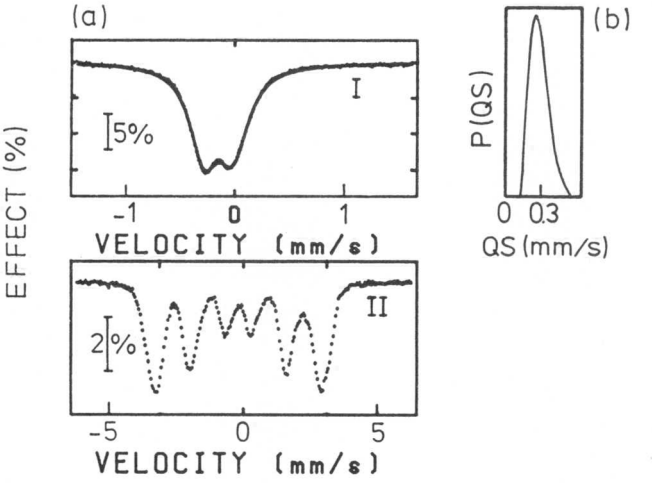

Figure 2. (a) Spectra and (b) Quadrupole distribution for crystallized Sc_3Fe heated to (I): 550 C for ca. 1 hour; (metastable state) measured at RT, and (II): 650 C for ca. 1 hour; (stable state) measured at T = 4.2 K (magnetic).