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Mössbauer study of sputtered Fe-Ti alloys with wide composition range

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$\text{Fe}_x\text{Ti}_{100-x}$ alloys over a wide composition range has been made by high-rate sputtering and studied by ^{57}Fe Mössbauer spectroscopy. Samples with $30 \leq x \leq 80$ are amorphous whereas samples with $x \geq 85$ are metastable crystalline bcc alloys. Large differences in magnetic properties (e.g., T_C) and hyperfine interactions (H_{eff} , isomer shift) are found between the amorphous and bcc alloys. Comparison of FeTi and Fe_2Ti in amorphous and crystalline states show that the short-range order in the amorphous state has no resemblance to that of the crystalline compounds.

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I. INTRODUCTION

Under equilibrium conditions, solids containing Fe and Ti exist as crystalline intermetallic compounds (FeTi and Fe_2Ti) and low solubility (a few percent) alloys.¹⁻³ We show in this work that by using a vapor-quench method, $\text{Fe}_x\text{Ti}_{100-x}$ alloys can be fabricated over the wide composition range of $30 \leq x \leq 100$. Depending on the composition, the alloy appears either as a crystalline bcc structure or an amorphous solid.^{4,5} Very different magnetic properties and hyperfine interactions have been observed between the crystalline and amorphous alloys. Mössbauer results indicate that the local short range order in the amorphous state has no resemblance to that of crystalline FeTi and Fe_2Ti .

II. EXPERIMENT

$\text{Fe}_x\text{Ti}_{100-x}$ ($30 \leq x \leq 100$) films, 5–10 μm in thickness, were deposited by a high-rate sputtering device onto liquid nitrogen cooled Kapton or Cu substrates.⁶ The samples with $30 \leq x \leq 80$ are amorphous, whereas the samples with $x \geq 85$ are metastable crystalline bcc alloys. Using an energy dispersive x-ray diffraction method, the bcc structure of the samples with $x \geq 85$ is ascertained. The lattice parameters of these bcc alloys are very close to that of $\alpha\text{-Fe}$. The crystalline intermetallic compounds of FeTi and Fe_2Ti were made by arc melting in an argon atmosphere. A ^{57}Co in Rh source was used in a conventional Mössbauer spectrometer.

III. RESULTS AND DISCUSSIONS

At ambient temperatures, the solubility of Ti in bcc Fe is only about 4 at. %.³ Through vapor quenching, the solubility in the bcc state is substantially extended to at least 15 at. % ($x \geq 85$). At higher Ti contents ($30 \leq x < 80$), the sputtered samples are amorphous. As described in the following, the properties of the amorphous alloys and the bcc alloys are very different. Consequently, there are abrupt changes in properties at the boundary separating the amorphous state and the crystalline bcc state. The large composition range of the amorphous state covers the compositions corresponding to crystalline FeTi and Fe_2Ti . One can therefore directly compare FeTi and Fe_2Ti in both crystalline and amorphous state.

A. Magnetic ordering temperature (T_C)

The magnetic ordering temperatures of the amorphous samples ($30 \leq x \leq 80$) have been determined by the zero veloc-

ity thermal scan method. The measured values of T_C , all below room temperature, increase monotonically with x as shown in Fig. 1. Below the magnetic threshold of $x_c \simeq 43$, the low Fe concentration samples are nonmagnetic. On the other hand, the values of T_C for the metastable bcc alloys ($x \geq 85$) are very much higher than room temperature. Due to structural transformation of these metastable alloys at high temperatures, their T_C can not be directly measured. Nevertheless, the values of T_C are substantially higher than 850 K, above which the samples transform irreversibly.

B. Magnetic hyperfine interaction

Mössbauer spectra of amorphous $\text{Fe}_{75}\text{Ti}_{25}$ and $\text{Fe}_{80}\text{Ti}_{20}$ at 4.2 K are shown in Fig. 2. Despite relatively high Fe content, the effective magnetic hyperfine field (H_{eff}) and therefore the Fe magnetic moment are rather small.⁵ As shown in Fig. 3, the value of H_{eff} of amorphous Fe-Ti decreases monotonically as the Fe concentration is reduced, and is extrapolated to zero at about the magnetic threshold of $x_c \simeq 43$ observed in Fig. 1. This indicates, as already observed in many binary amorphous Fe-containing alloys, such Fe-B, Fe-Nb, Fe-Zr, etc., that the disappearance of Fe moment causes the samples with $x < x_c$ to be nonmagnetic.⁶⁻⁸

Completely different spectra are observed for the metastable bcc Fe-Ti alloys. The bottom figure of Fig. 2 shows the spectrum of the as-prepared bcc $\text{Fe}_{85}\text{Ti}_{15}$ alloy at 4.2 K. The splitting and correspondingly the value of H_{eff} are much larger than those of the amorphous Fe-Ti alloys. As shown

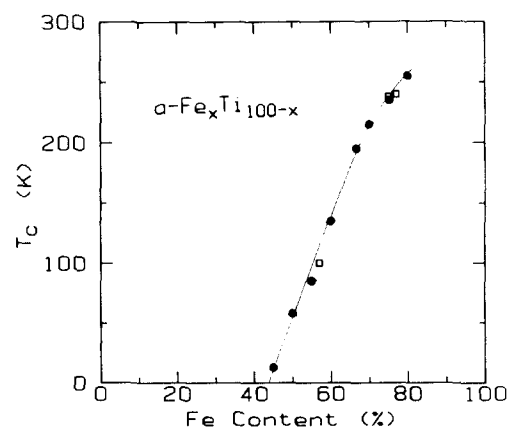


FIG. 1. Magnetic ordering temperatures of amorphous Fe-Ti alloys. The open squares are taken from Ref. 4.

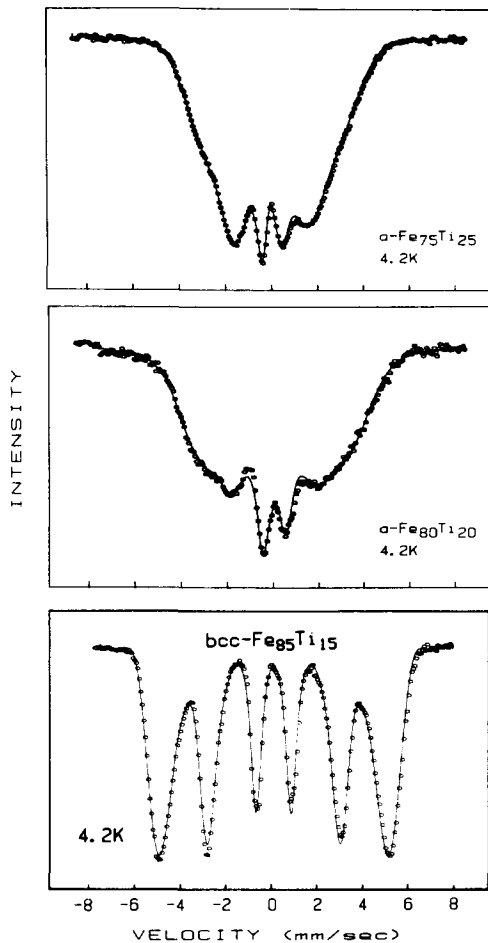


FIG. 2. Mössbauer spectra of amorphous $\text{Fe}_{75}\text{Ti}_{25}$, $\text{Fe}_{80}\text{Ti}_{20}$, and crystalline bcc $\text{Fe}_{85}\text{Ti}_{15}$ at 4.2 K.

in Fig. 3, from amorphous $\text{Fe}_{80}\text{Ti}_{20}$ to bcc $\text{Fe}_{85}\text{Ti}_{15}$, there is an abrupt increase in H_{eff} of about 50%. Sumiyama *et al.* have observed a similarly large increase in the Fe moment.⁵

At elevated temperatures, the metastable bcc Fe-Ti alloys transform irreversible into α -Fe and Fe_2Ti .

C. Quadrupole spectra

At room temperature, all the amorphous Fe-Ti samples exhibit quadrupole spectra, from which values of isomer

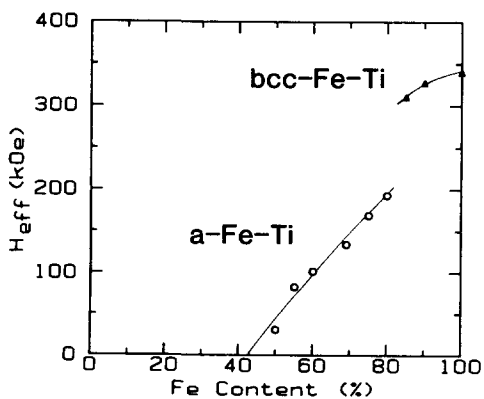


FIG. 3. Magnetic hyperfine field (H_{eff}) at 4.2 K of amorphous and bcc Fe-Ti alloys.

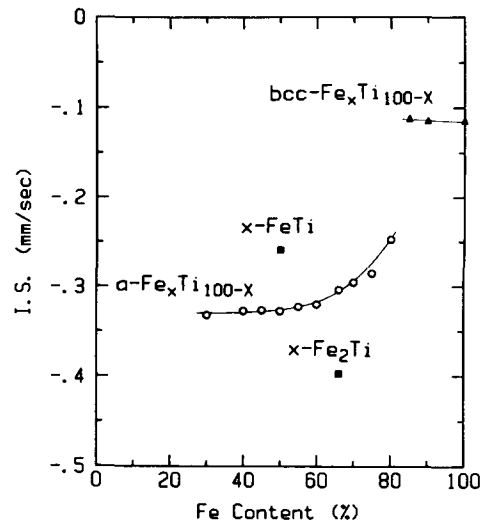


FIG. 4. Isomer shifts of amorphous and bcc Fe-Ti alloys and crystalline FeTi and Fe_2Ti at room temperature.

shift and quadrupole splitting can be determined. The isomer shift of amorphous and bcc Fe-Ti alloys are shown in Fig. 4. The isomer shifts of the amorphous alloys, like other amorphous Fe-transition metal alloys, are negative with respect to α -Fe.⁷⁻⁸ The isomer shifts of the bcc alloys are essentially the same as that of α -Fe. Most noticeably, there is a very large difference between the isomer shifts of amorphous and bcc Fe-Ti alloys.

The quadrupole splitting of the amorphous Fe-Ti alloys are shown in Fig. 5. These values, similar to other amorphous Fe-early transition metal alloys, decrease with Fe content and exhibit a shallow minimum at $x \approx 70$.

D. Comparison of crystalline and amorphous FeTi and Fe_2Ti

In the crystalline Fe-Ti system, there are only two intermetallic compounds (FeTi and Fe_2Ti), both with well-known properties and crystal structures. Crystalline FeTi (x -FeTi) has a simple (B2) CsCl structure with *cubic* symmetry. The Mössbauer spectrum consequently shows zero quadrupole splitting. Crystalline Fe_2Ti (x - Fe_2Ti) has a (C-14) MgZn_2 structure.¹ The Mössbauer spectrum shows a large quadrupole

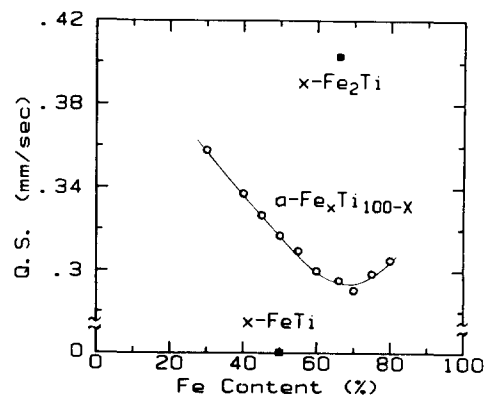


FIG. 5. Quadrupole splittings of amorphous Fe-Ti alloys and crystalline FeTi and Fe_2Ti at room temperature.

pole splitting of 0.40 mm/sec. These values, in good agreement with those in the literature, are shown in Figs. 4 and 5. It is clear that these values are distinctively different from those of α -FeTi and α -Fe₂Ti; in fact, amorphous Fe-Ti of *all* compositions.

IV. CONCLUSIONS

The results of amorphous and bcc Fe-Ti alloys illustrate that the magnetic properties and hyperfine interactions depend sensitively on the structure. Abrupt changes occur near the boundary separating the amorphous and the crystalline bcc states.

The comparison of FeTi and Fe₂Ti in amorphous and crystalline states provide insights of the local charge distributions and atomic arrangements. The isomer shifts and quadrupole splitting are so far apart that they provide evidences *against* the conjecture that similar local short-range

order may be found in both amorphous and crystalline states.

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