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## The effect of 40 keV helium ion irradiation on Fe–B amorphous alloys

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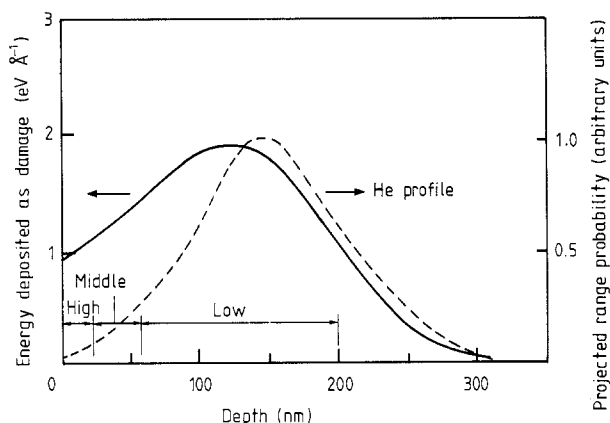
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**Abstract.** The phase conditions of amorphous  $\text{Fe}_{100-x}\text{B}_x$  alloys ( $x \leq 25$ ) irradiated with 40 keV  $\text{He}^+$  ions were studied by depth-selective conversion electron Mössbauer spectroscopy (DCEMS). A gas-flow-type proportional counter was used in the DCEMS measurements and the method is shown to be useful to detect the phase changes in the near-surface regions of the alloys. It is found that the crystalline phase of  $\alpha$ -iron is induced on the irradiated surface of amorphous  $\text{Fe}_{80}\text{B}_{20}$  and  $\text{Fe}_{85}\text{B}_{15}$  but not on  $\text{Fe}_{75}\text{B}_{25}$  and that the extent of the amorphous-to-crystalline transformation depends on the depth from the surface, decreasing towards the interior of the sample. It is likely that the irradiation leads to separation of the amorphous structure into boron-depleted and boron-enriched regions. It is suggested that an amorphous structure exists which is stable against irradiation after the formation of  $\alpha$ -iron.

### 1. Introduction

The behaviour of amorphous metal alloys in an irradiation environment has been the subject of considerable interest from the viewpoint of irradiation-induced relaxation and order–disorder effects in the amorphous state (Brimhall 1985). A number of experiments have been performed on the effect of irradiation of amorphous alloys using various methods such as scanning and transmission electron microscopy (Tyagi *et al* 1983), but the changes in microstructures due to irradiation do not seem to be fully understood as yet. Conversion electron Mössbauer spectroscopy (CEMS) is a powerful method for studying the crystallisation of amorphous iron alloys. Two of the present authors (Hayashi and Sakamoto 1982) showed that 40 keV  $\text{He}^+$  ion irradiation in the fluence range  $(0.5\text{--}3) \times 10^{18}$  ion  $\text{cm}^{-2}$  induces partial crystallisation, i.e. precipitation of  $\alpha$ -iron in amorphous  $\text{Fe}_{80}\text{B}_{20}$ . Furthermore, Yonekura *et al* (1983) demonstrated depth-selective measurements of conversion electron Mössbauer (DCEMS) with a gas proportional counter are particularly useful to study depth-dependent changes in microstructure of iron-based alloys near the surface; the depth-dependent effect of irradiation has been made clear in Fe–Si multilayer systems ion mixed with energetic particles by Hayashi *et al* (1986).

It was reported that crystallisation of the amorphous Fe–B alloys starts at the surface when alloys were thermally annealed in high vacuum (Ok and Morrish 1981, Wagner *et al* 1985). Therefore, it is quite reasonable to consider that the DCEMS method can



**Figure 1.** Range and energy deposition distributions as a function of penetration depth for 40 keV He<sup>+</sup> ions in amorphous Fe-B alloy. High, Middle and Low in the figure indicate the depth ranges in DCEMS measurements.

be applied to investigate the phase condition in the near-surface region of irradiated Fe<sub>100-x</sub>B<sub>x</sub> alloys. This paper shows the effect of He<sup>+</sup> ions on Fe<sub>85</sub>B<sub>15</sub>, Fe<sub>80</sub>B<sub>20</sub> and Fe<sub>75</sub>B<sub>25</sub> alloys, including preliminary results from H<sup>+</sup> ion irradiation. The analysis of DCEMS measurements shows clearly that the partial crystallisation is dependent on the depth from the surface and also on the relative concentration of elemental iron to boron in the alloys.

## 2. Experiments

The amorphous samples<sup>†</sup> were supplied as thin ribbons about 2 mm wide and 45 μm thick. The shiny side of the ribbon was used in the experiments. He<sup>+</sup> ion bombardment was carried out at normal incidence to the target surface, using a beam energy of 40 keV and a current density of about 5 μA cm<sup>-2</sup>. The vacuum in the target chamber was in the range of 10<sup>-5</sup> Pa during the irradiation. The sample temperature was estimated to be less than 160 °C by monitoring with a thermocouple attached to the sample holder.

Mössbauer measurements were performed at room temperature on the irradiated samples in scattering geometry, employing <sup>57</sup>Co in a rhodium matrix radioactive source (10 mCi). Conversion (7.3 keV) and Auger (5.6 keV) electrons emitted in <sup>57</sup>Fe were detected with a gas flow proportional counter, which employed a 90% He-10% CH<sub>4</sub> mixture. For DCEMS measurements the energies of the emitted electrons were selected by determining the pulse height spectrum. The selected ranges correspond mainly to three depth ranges, i.e. the high-energy (11-16 keV) range corresponds to the depth range from the surface to 20 nm depth, the middle-energy (6-9 keV) range corresponds to 20-60 nm and the low-energy (2-5 keV) range corresponds to deeper than 60 nm

<sup>†</sup> The samples were obtained by courtesy of Dr R Hasegawa of Allied Chemical Corporation.

from the surface. The high-energy electrons are contributed mainly from the sum<sup>†</sup> of *K* conversion and Auger electrons (7.3 + 5.6 keV).

We cannot straightforwardly convert a spectrum measured with a specified energy to a depth-selective one. For the conversion it is necessary to use a weight function (the probability of the electrons' escape) as described by Keune (1986). Then the weight function can be constructed by the use of a transmission function of energetic electrons through iron metals, which was defined by Liljequist (1979). In the present report the function was empirically determined from an experiment using a <sup>57</sup>Fe thin film covered with <sup>56</sup>Fe films of various thicknesses in the way described by Yonekura *et al* (1983). Figure 1 shows a comparison of the determined depth ranges and damage distributions of 40 keV He<sup>+</sup> ions in an amorphous Fe–B alloy, together with the depth ranges in our DCEMS measurements. The curves were calculated by Lindhard–Scharff–Schjøtt theory with the table from Northcliff and Schilling (1970).

### 3. Results

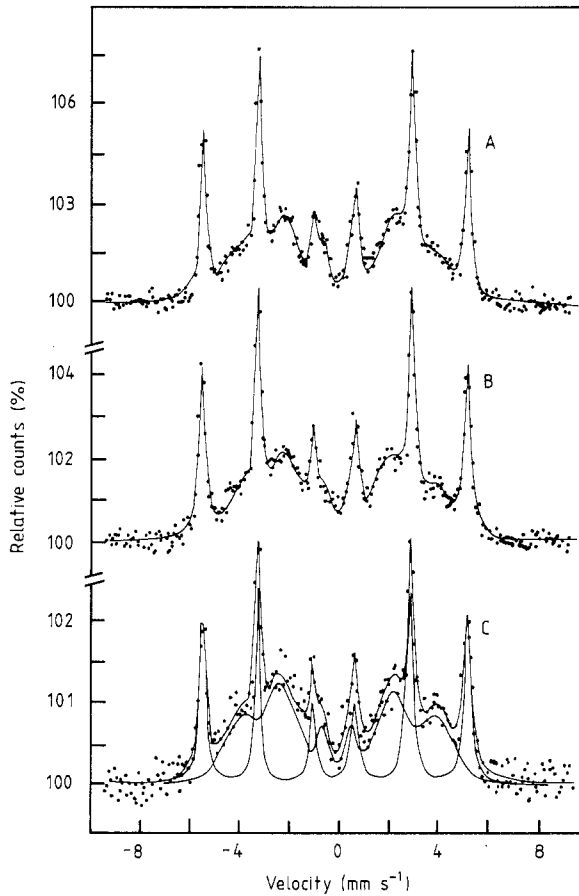
The DCEMS spectra taken from amorphous Fe<sub>85</sub>B<sub>15</sub> irradiated to a fluence of  $1 \times 10^{18}$  ions cm<sup>-2</sup> are shown in Figure 2. The three spectra A, B and C correspond to spectra from the electrons of high-, middle- and low-energy ranges, respectively. The spectra are characterised by the superposition of two sets of six peaks. It is noted that, while one set of six lines has a broad linewidth, typical of amorphous materials, the other has a sharp linewidth of 0.28 mm s<sup>-1</sup>. For the latter set the internal field is obtained to be 330 kG. It is apparent that these peaks should be assigned to a crystalline  $\alpha$ -iron phase, which is induced by 40 keV He<sup>+</sup> ion irradiation (Hayashi and Sakamoto 1982). On the contrary, the broad peaks indicate that another phase is still present as the amorphous state. The measured spectra were fitted using a least-squares minimisation program with Lorentzian profiles. In the analysis it is assumed that the amorphous phase has an asymmetrical Gaussian distribution  $P(H)$  of internal fields, i.e.

$$P(H) = \exp[-(H - H_0)^2 / (2\Delta H_{1,2})]$$

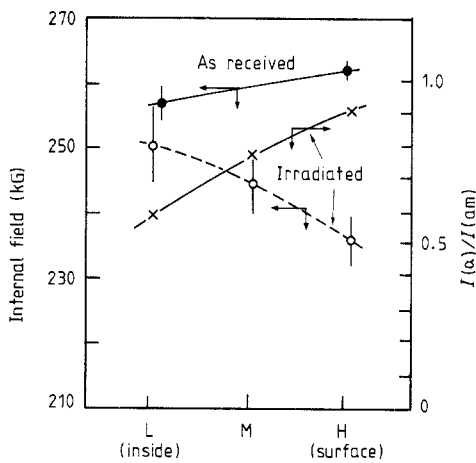
with  $\Delta H_1$  for  $H \leq H_0$  and  $\Delta H_2$  for  $H > H_0$ .

From figure 2 it is seen that the irradiation-induced crystallisation decreases towards the interior of the sample. Figure 3 shows the change in the ratio  $I(\alpha)/I(\text{am})$  of the intensity of  $\alpha$ -iron to the intensity of the amorphous phase, for the different depths of the high-, middle- and low-energy ranges, together with the change in the peak value of the internal field distribution  $H_0$  in the amorphous phase;  $I(\alpha)/I(\text{am})$  was calculated from the ratio of the spectral area of  $\alpha$ -iron to that of the amorphous state. While  $I(\alpha)/I(\text{am})$  decreases with increasing depth from the surface, the  $H_0$  curve increases from the surface to the interior. This result suggests that the precipitation of  $\alpha$ -iron leads to boron enrichment in the remaining amorphous state, since it was reported that the internal field in the alloy decreases with increasing boron concentration (Chien and Unruh 1981). The result shown in figure 3, therefore, is reasonable from the standpoint of the conservation of boron and iron atoms in the system and also is consistent with the observation in irradiation experiments on Fe<sub>80</sub>B<sub>20</sub> (Hayashi and Sakamoto 1982); the CEMS spectra demonstrate the fact that  $H_0$  in the amorphous state of the irradiated

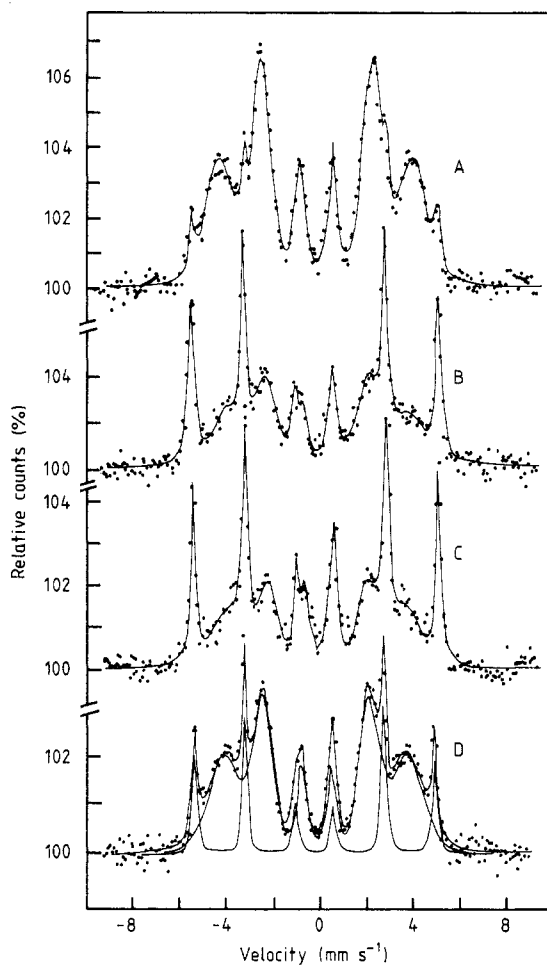
<sup>†</sup> This sum of electron counts arises because the proportional counter which has a resolving time of the order of a microsecond cannot discriminate between the conversion and Auger electrons emitted sequentially from the outermost surface.



**Figure 2.** DCEMS spectra from  $\text{Fe}_{85}\text{B}_{15}$  alloys irradiated with 40 keV  $\text{He}^+$  ions to a fluence of  $1 \times 10^{18}$  ions  $\text{cm}^{-2}$ . Spectrum A is recorded with high-energy (11–16 keV) electrons, spectrum B with middle-energy (6–9 keV) electrons and spectrum C with low-energy (2–5 keV) electrons.



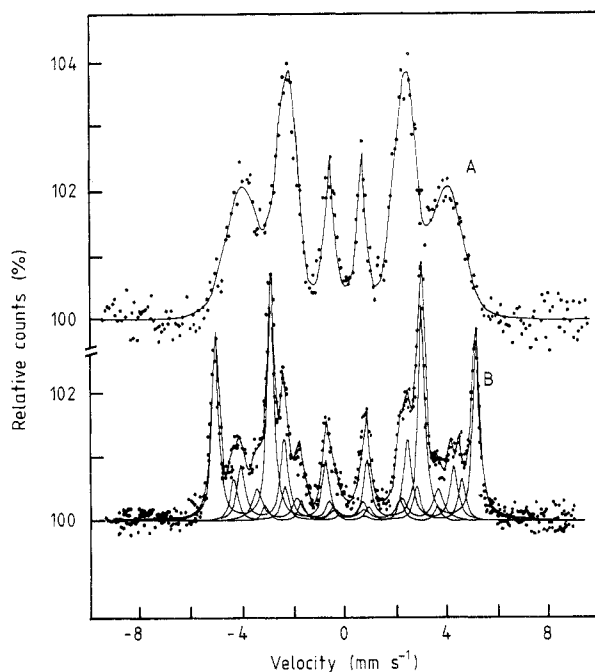
**Figure 3.** Variation in internal field  $H_0$  (○) and intensity ratio  $I(\alpha)/I(\text{am})$  of  $\alpha$ -iron to amorphous phase (×) are plotted against depth range in  $\text{He}^+$  ion-irradiated  $\text{Fe}_{85}\text{B}_{15}$  alloy.



**Figure 4.** CEMS spectra of Fe<sub>80</sub>B<sub>20</sub> alloys irradiated with 40 keV He<sup>+</sup> ions to fluences of  $5 \times 10^{17}$  ions cm<sup>-2</sup> (spectrum A),  $1 \times 10^{18}$  ions cm<sup>-2</sup> (spectrum B),  $3 \times 10^{18}$  ions cm<sup>-2</sup> (spectrum C) and with 40 keV H<sup>+</sup> ions to a fluence of  $3 \times 10^{18}$  ions cm<sup>-2</sup> (spectrum D).

sample is less than  $H_0$  for the as-received samples. It is worth noting that similar decomposition of metal-metalloid elements, i.e. separation into regions with depleted and enriched boron, was assumed in amorphous Fe<sub>40</sub>Ni<sub>40</sub><sup>10</sup>B<sub>20</sub> irradiated with reactor neutrons (Schimansky *et al* 1985).

The DCEMS spectra in figure 4 were measured in the high-energy range of Fe<sub>80</sub>B<sub>20</sub> irradiated in the fluence range  $(0.5-3) \times 10^{18}$  ions cm<sup>-2</sup>. The figure clarifies the fact that the formation of  $\alpha$ -iron is dependent on the helium fluence, becoming well defined beyond a fluence of  $5 \times 10^{17}$  ions cm<sup>-2</sup> and saturating at around a fluence of  $3 \times 10^{18}$  ions cm<sup>-2</sup>; the intensity ratio  $I(\alpha)/I(\text{am})$  in the surface region was calculated to be 7.5 for  $1 \times 10^{18}$  ions cm<sup>-2</sup> and 8.1 for  $3 \times 10^{18}$  ions cm<sup>-2</sup> irradiation. It was confirmed from further DCEMS measurements that the depth dependence of induced  $\alpha$ -iron for-



**Figure 5.** CEMS spectra taken from  $\text{Fe}_{75}\text{B}_{25}$  alloy irradiated with 40 keV  $\text{He}^+$  ions to a fluence of  $1 \times 10^{18}$  ions  $\text{cm}^{-2}$  (spectrum A) and  $\text{Fe}_{75}\text{B}_{25}$  alloy heated in vacuum at 350 °C for 4 h (spectrum B).

mation in  $\text{Fe}_{80}\text{B}_{20}$  is similar to that in  $\text{Fe}_{85}\text{B}_{15}$  shown in figure 2. The results from the DCEMS measurements are summarised in figure 6 later.

For comparison, figure 4, spectrum D, is obtained by irradiation with 40 keV  $\text{H}^+$  ions irradiated to a fluence of  $3 \times 10^{18}$  ions  $\text{cm}^{-2}$ . The precipitation of  $\alpha$ -iron appears to be small in  $\text{H}^+$ -ion-irradiated  $\text{Fe}_{80}\text{B}_{20}$  and  $I(\alpha)/I(\text{am}) = 0.26$  is obtained. This result is consistent with the lower rate of damage formation for  $\text{H}^+$  ion irradiation compared with  $\text{He}^+$  ion irradiation; the production of defects in the surface region is estimated to be about 0.2 displacements per atom (dpa) for  $\text{H}^+$  ion irradiation, one order less than for  $\text{He}^+$  ions. The result is also consistent with the fluence dependence of  $\alpha$ -iron formation mentioned above. This also means that the effect of irradiation is not caused by thermal effects due to beam heating, because the hydrogen and helium bombardments were performed under the same ion energies and fluxes.

Figure 5, spectrum A, is from an  $\text{Fe}_{75}\text{B}_{25}$  alloy irradiated with  $\text{He}^+$  ions up to a fluence of  $1 \times 10^{18}$  ions  $\text{cm}^{-2}$ . There is no indication of  $\alpha$ -iron precipitation in the spectrum. It is assumed that the alloy is highly resistant to irradiation-induced crystallisation. For comparison, a spectrum obtained from the unirradiated but heat-treated amorphous alloy is shown in figure 5, spectrum B; the heat treatment was carried out in high vacuum at a temperature of 350 °C for 4 h. The structural relaxation of the amorphous alloy is apparent in figure 5, spectrum B. In figure 5 the spectra are characterised by a superposition of five sets of six lines, whose internal fields were found to be 330 kG from the  $\alpha$ -iron phase, 288, 269 and 229 kG from the metastable crystalline  $\text{Fe}_3\text{B}$  phase and 217 kG from the  $\text{Fe}_2\text{B}$  phase. The crystallisation and the appearance of the quoted phases due to thermal treatment are in good agreement with results published already

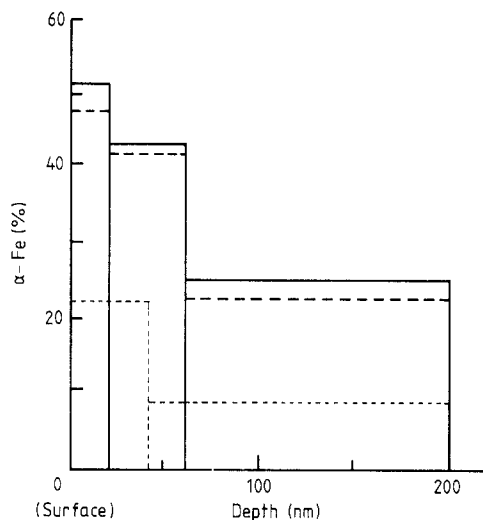


Figure 6. Variation in  $I(\alpha)/I(\text{am})$  with depth for Fe<sub>85</sub>B<sub>15</sub> He<sup>+</sup> ion irradiated to a fluence of  $1 \times 10^{18}$  ions cm<sup>-2</sup> (—), for Fe<sub>80</sub>B<sub>20</sub> He<sup>+</sup> ion irradiated to a fluence of  $3 \times 10^{18}$  ions cm<sup>-2</sup> (---) and for Fe<sub>80</sub>B<sub>20</sub> H<sup>+</sup> ion irradiated to a fluence of  $1 \times 10^{18}$  ions cm<sup>-2</sup> (-·-·-).

(Frank *et al* 1978). From a comparison of the two spectra in figure 5 it seems reasonable to conclude that ion irradiation leads to a structural relaxation different from thermal treatment. We have in fact discussed this issue in a previous paper (Hayashi and Sakamoto 1982), where comparisons of CEMS and transmission spectra from both He<sup>+</sup> ion-irradiated and heat-treated Fe<sub>80</sub>B<sub>20</sub> alloys were given.

#### 4. Discussion

In order to obtain a quantitative presentation of depth profiling, the spectra measured at specified energies were converted to spectra for the corresponding depth ranges. In figure 6 is shown the variation in amount of  $\alpha$ -iron formation at a depth of 200 nm from the surface. The fraction of  $\alpha$ -iron in the irradiated alloys decreases with increasing depth, in contradiction with the He<sup>+</sup> ion and damage distribution shown in figure 1; the damage distribution has its peak at a depth of 125 nm. On the contrary, the increase in  $\alpha$ -iron formation with increasing fluence seems to be reasonable from a standpoint of increasing damage (see figure 4). It is, therefore, considered that the surface properties of amorphous Fe–B alloys are different from the bulk properties, as is the crystallisation process.

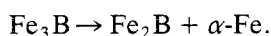
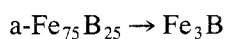
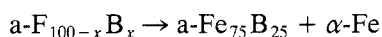
The formation of helium bubbles after irradiation at a fluence of about  $10^{17}$  ions cm<sup>-2</sup> has been observed in amorphous alloys such as Fe<sub>80</sub>B<sub>20</sub> and Fe<sub>40</sub>Ni<sub>40</sub>B<sub>20</sub>, although the helium retention seems to be lower than in crystalline materials (Tyagi *et al* 1983). Thus the partial crystallisation might be affected by displacement damage and/or gas precipitation into bubbles. However, it is observed that not only He<sup>+</sup> ions but also H<sup>+</sup> ions, which are implanted more uniformly at a lower density, induce crystallisation in amorphous Fe–B alloys. Furthermore, Schimansky *et al* (1985) found a compositional separation in neutron-irradiated Fe<sub>40</sub>Ni<sub>40</sub>B<sub>20</sub>. It is, therefore, supposed that the observed



partial crystallisation under  $\text{He}^+$  ion irradiation may not be affected by bubble formation nor by its distribution under the surface.

It was reported as a result of the observation of CEMS and transmission spectra that in thermally annealed amorphous  $\text{Fe}_{100-x}\text{B}_x$  alloys the crystallisation starts at a lower temperature and develops to a larger extent in the near-surface region than in the bulk (Ok and Morrish 1981, Wagner *et al* 1985). It has been assumed that the stoichiometry of the alloys in the surface region may deviate from the bulk constitution, i.e. the iron concentration at the surface is higher than in the bulk material. The internal field  $H_0$  in as-received  $\text{Fe}_{85}\text{B}_{15}$  is higher on the surface side as shown in figure 3, which supports our assumption. Our observations on  $\text{Fe}_{100-x}\text{B}_x$  alloys with  $x = 15$  and 20 indicate that the crystallisation of  $\alpha$ -iron is affected by the relative concentrations of iron and boron in the irradiated zone and seem to be in agreement with the DCEMS measurements where a larger extent of partial crystallisation has been observed on the surface side, suggesting a compositional change in the near-surface region. Although our results indicate that the off-stoichiometry from the composition near  $\text{Fe}_{75}\text{B}_{25}$  is the primary driving force for the partial crystallisation, it appears from figure 3 that the crystallisation proceeds more effectively on the surface; the depth dependence of the internal fields has been reversed after the irradiation. It is not possible to give a definite explanation for the surface effect, but it is likely that the number of crystalline embryos, as discussed later, may be increased much more than expected from the off-stoichiometry and that the amorphous-to-crystalline transformation is promoted in the near-surface region.

The structure and stability of  $\text{Fe}_{100-x}\text{B}_x$  amorphous alloys have been investigated by means of various methods such as differential scanning calorimetry and magnetisation measurements. Kemeny *et al* (1979) have claimed that in thermally annealed Fe–B alloys the crystallisation sequence can be described as

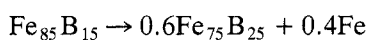


Our experiments clearly indicate that irradiation-induced crystallisation corresponds to the first step of thermally induced crystallisation. However, it is likely that no further crystallisation is induced in the case of irradiation, because the present experiment shows that the  $\alpha$ -iron formation is saturated at a certain  $\text{He}^+$  ion fluence, and also amorphous  $\text{Fe}_{75}\text{B}_{25}$  is shown to be resistant to partial crystallisation.

It has been indicated from an analysis of Mössbauer spectra that the amorphous structure should be based on a non-stoichiometric  $\text{Fe}_3\text{B}$  quasi-crystalline unit with locally distorted structure (Kemeny *et al* 1979). From an analysis of the interference function in x-ray diffraction, Hamada and Fujita (1982) have proposed a structural model of amorphous alloys based on the concept of quasi-crystalline BCC-like embryos. They predicted chemical compound clusters of  $\text{Fe}_3\text{B}$  type as the basic structural unit in amorphous Fe–B alloys with a high boron concentration. Our results seem to be in good agreement with the amorphous models mentioned above; that is, the quasi-crystalline embryo might be responsible for  $\alpha$ -iron formation, condensing boron atoms in the remaining amorphous phase. The transport of boron atoms for decomposition can be enhanced by an irradiation effect such as radiation-assisted diffusion. It is quite natural to assume that the fraction of crystalline embryos in amorphous  $\text{Fe}_{100-x}\text{B}_x$  alloys decreases with increasing  $x$  and leads to a smaller extent of  $\alpha$ -iron formation. On the contrary, it seems that chemical disorder can be introduced in the alloys with  $x$ -values

near 25 during irradiation and thus the amorphous structure will be stable under irradiation.

Berkowitz *et al* (1982) observed the crystalline-to-amorphous transformation in recrystallised Fe<sub>75</sub>B<sub>25</sub> ribbon which was bombarded with 4 MeV Ar<sup>+</sup> ions. The samples with the phases Fe<sub>3</sub>B, Fe<sub>2</sub>B and  $\alpha$ -iron were crystallised after annealing at 773 K. While crystalline Fe<sub>3</sub>B was completely converted to an amorphous state at a damage level of 0.15 dpa, the  $\alpha$ -iron phase was most resistant to such amorphisation. A similar crystalline-to-amorphous transition was also observed after irradiation with ions such as He<sup>+</sup>, B<sup>+</sup> and Ne<sup>+</sup> on iron pre-implanted with boron, where amorphous covalent metal–metalloid clusters are assumed to stabilise disorder due to irradiation (Rauschenbach and Heera 1987). The results on re-amorphisation are extremely consistent with the present results, supporting the above conclusion on the effect of He<sup>+</sup> ion irradiation on amorphous Fe<sub>100-x</sub>B<sub>x</sub> alloys. The extent of crystallisation can be roughly estimated by assuming an irradiation-induced reaction as follows:



where the conservation of boron was taken into account. Then the result  $I(\alpha)/I(\text{am}) = 0.4/(0.6 \times 0.75) = 0.9$  is obtained, in fair agreement with the experimental result for the near-surface region.

## 5. Conclusion

DCEMS has been successfully used to study the effects of irradiation in the near-surface region of amorphous Fe–B alloys. The method, which is non-destructive by nature, has proved to be useful in determining the phase conditions in the surface region of ion-irradiated iron-based alloys. Although the measurement with a gas proportional counter is poor with respect to energy resolution, it is very effective in studying natural (non-<sup>57</sup>Fe-enriched) iron alloys because of its high counting efficiency and simultaneous measurements with a multi-energy window.

In amorphous Fe–B alloys the precipitation of crystalline  $\alpha$ -iron has been observed on irradiation with 40 keV He<sup>+</sup> ions. The amorphous-to-crystalline transformation has been observed to be dependent on the irradiation fluence and also on the depth from the sample surface. The latter fact suggests that the iron-to-boron concentration ratio near the surface deviates from that in the bulk. DCEMS measurements show that the amount of  $\alpha$ -iron formation decreases with increasing depth from the top surface for amorphous Fe<sub>85</sub>B<sub>15</sub> and Fe<sub>80</sub>B<sub>20</sub>. On the contrary, Fe<sub>75</sub>B<sub>25</sub> is found to be resistant to irradiation-induced crystallisation. It is assumed that the effect of helium bombardment results in decomposition into boron-depleted and boron-enriched regions.

## Acknowledgments

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