Amorphous alloy formation by mechanical alloying and consecutive heat treatment in Fe₅₀B₅₀ powder mixture

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(Received 24 October 1994; accepted for publication 31 January 1995)

Equiatomic composition powder mixture of pure Fe and B was mechanically alloyed in a controlled atmosphere vibration mill. Amorphous alloy phase formation was established by Mössbauer spectroscopy and by transmission electron microscopy. The variation of the average boron concentration and the homogeneity of the amorphous phase during the alloying process was followed by Mössbauer spectroscopy. At the early stage of the milling process a broad concentration distribution (from 20 to 50 at. % B) was observed. Upon further milling the average B concentration of the amorphous component was gradually enhanced and the concentration heterogeneity decreased. The B concentration of the amorphous phase could be further enhanced by heat treating the milled powder mixture. The highest boron content alloy attained in this study is Fe₅₀B₅₀. © 1995 American Institute of Physics.

I. INTRODUCTION

The Fe-B system is suitable for a detailed study of the solid state amorphization process since the amorphous phase is found in a wide concentration range when prepared by melt quenching or vacuum evaporation.¹ The amorphous Fe-B alloys are ferromagnetic below approximately 60 at. % B concentration and the composition of the amorphous phase can be estimated from changes of the iron magnetic moment and Curie temperature.

It is widely assumed that a solid state amorphization reaction (SSAR) is possible if the heat of mixing is negative and the components have widely different diffusion rates (mobilities) in the relevant structures.² Alloy formation in multilayered thin films and in mechanically alloyed powders seems to be related since it was established³ that after a short attrition time a structure consisting of alternating layers is usually formed in the milled powder. (For reviews see Refs. 4 and 5.) This concept explains rather successfully the formation of amorphous transition metal-transition metal (TM-TM) alloys.⁴ It is generally assumed that SSAR is not very effective for the formation of transition metal-metalloid (TM-M) alloys, negative results have been found specifically for the FeB system.⁵,⁶ This is usually interpreted⁷ as due to the similar diffusivity of the two components.⁸

The above scheme, however, has no general relevance. The V-Zr system amorphizes on mechanical alloying,¹¹ although it is a non-fast-diffusing system. There are recent reports of amorphous alloy formation in systems with near zero or even positive heat of mixing (Zn-Nb;¹² Cu-Ta;¹³) and in Fe-B system.¹⁴–¹⁷

While the formation of the amorphous Fe-B alloy by mechanical alloying is unambiguously demonstrated, this fact is far from being understood, and even the details of the process are not experimentally well established. The maximum rate of amorphization has been reported¹⁵ to occur at 60 at. % B, while a recent work¹⁶ found it near 20 at. % B. The enhanced amorphization rate of 20 at. % B alloy in comparison with 38 at. % B sample is also reported.¹⁷ An amorphous phase with a steady state composition near 30 at. % B is reported in the whole range of nominal concentrations 10–90 at. % B.¹⁷ All these findings are in clear disagreement with the thermodynamic calculations,¹⁸ which predict a glass formation range for 32–47 at. % B.

Details of the amorphous phase formation are not discussed in earlier works. The intermixing of B with Fe on the atomic level, the possibility of the formation of crystalline solid solution phases are not yet investigated. No result is available on the possible heterogeneity of the amorphous phase or even on changes of the average concentration during the milling process. It is reported,¹⁶ that enrichment of the amorphous phase in B is possible via heat treatment in the early stage of milling when the amount of the amorphous component is small. It is not known whether the B diffusion proceeds when the crystalline Fe phase is completely absent. We have used transmission Mössbauer spectroscopy, transmission electron microscopy, and calorimetry to investigate these questions. Besides the room temperature Mössbauer studies, investigations in the 4–700 K temperature range were also carried out. Special care was taken to control the ambient atmosphere, since recent reports indicate its dominant influence.¹⁹,²²

J. Appl. Phys. 77 (10), 15 May 1995 0021-8979/95/77(10)/4997/7/$6.00 © 1995 American Institute of Physics 4997
II. EXPERIMENT

The mechanical milling was carried out in a vibrating frame single ball vessel continuously pumped during milling by a conventional diffusion pump system. The typical pressure was $10^{-4}$ mbar, but some tests (made with rotary pump only) indicated that any pressure below $10^{-2}$ mbar yield qualitatively the same trend. The technical construction of the vacuum vial corresponds to the description in Ref. 23: a hardened steel ball (60 mm diam, 870 g) oscillates on the top of a 65-mm-diam, 5-mm-thick tungsten carbide bottom plate. The oscillation frequency is 50 Hz, the amplitude of the ball movement is 15 mm. For this arrangement 1 g powder mixture charge was used to get high enough alloying rate and low impurity concentration. Aldrich Fe powder (99.9% purity, 44 μm average diameter) and Cerac B powder (99.9% purity, below 1 μm average size) was used. Some control tests made with 99.9% purity carbonyl Fe and Johnson Matthey B gave practically identical results. The photometric measurement of the size distribution of the as-milled powder mixture yields the following tentative results: besides approximately 50% of grains smaller than 1 μm, a further 36% is found in the 1–2.5 μm range and the remaining 14% is in the 2.5–10 μm interval. This size distribution is remarkably similar to that of the B powder (70% below 1 μm) and quite different from that of the Fe powder (only 3% below 1.5 μm and 50% above 6.5 μm).

Possible contamination was investigated by proton-induced x-ray emission. The as-milled powder contains some traces of Cr (less than 1 wt. %, also observed in the steel ball used for the milling). The other expected contaminants (Ni, W, Co, and C) were below the detection limit, less than 0.5 wt. %.

Beyond the standard x-ray diffraction monitoring of the powder mixture, transmission electron microscopy (TEM), Mössbauer spectroscopy (MS), and differential scanning calorimetry (DSC) experiments were carried out.

The TEM study of the samples was made with a Philips CM20 electron microscope with TWIN objective lenses. Bright field and dark field imaging techniques as well as selected area electron diffraction (SAED) patterns were used to detect various amorphous and crystalline phases in the samples. For the specimen preparation a special method based on ion milling was used. The powder grains mixed with araldite powder were filled into the slot of a Ti disk together with a small piece of silicon wafer. The epoxy was then hardened by polymerization in a furnace at 440 K for 1 h. After mechanical grinding to about 50 μm thickness the final thinning was performed by ion milling. It should be mentioned that though the ion milling procedure can raise the temperature of the specimen up to about 500 K, in the present study no effect of this extra heat treatment was observed.

The 57Fe Mössbauer spectra were recorded by a standard constant acceleration spectrometer using 50 mCi 57CoRh source. The low temperature measurements were performed in a homebuilt liquid He cryostat, the high temperature investigations were carried out in a vacuum furnace ($10^{-5}$ mbar). Standard procedures were used for the evaluation of the spectra: the crystalline component ($^{\alpha}$Fe) is fitted with the usual six-line pattern and after subtracting this fitted curve from the measured spectra, the remaining amorphous part is described by fitting binomial distributions. All the spectra were fitted by a double binomial distribution to achieve an appropriate fit. The typical error of the $^{\alpha}$-Fe fraction is below 2%. The accuracy of the average hyperfine parameters of the amorphous phase is slightly dependent on the amount of the subtracted $^{\alpha}$Fe spectra and on the shape of the distribution to be fitted. Standard errors never exceeded 0.3 T for the average hyperfine field, 0.5 T for the width of the hyperfine field distribution, and 0.01 mm/s for the average isomer shift.

The thermal behavior of the mechanically alloyed probes was investigated with a Perkin–Elmer DSC-2 differential scanning calorimeter. Standard procedures were used for data evaluation and it was established, that the typical accuracy of the indicated crystallization temperature was 2 K, while the error of the enthalpy measurement for the unusually broad transformation could even reach 10%. The data are presented as the rate of transformation curve, since it needs no mass normalization and thus it is independent from the amount of the remaining nonalloyed B.

III. RESULTS

A few representative Mössbauer spectra of samples milled for different times are shown in Fig. 1. Besides bcc-Fe, the spectrum of a ferromagnetic component with a very broad distribution of hyperfine fields evolves gradually as the milling time increases. By comparison with melt quenched and evaporated Fe-B amorphous alloys, it is identified as an amorphous phase, which will be discussed later. The $^{\alpha}$-Fe
spectrum is fully eliminated for 170 h milling and the amorphous phase is maintained by further attrition. However, at very long milling times traces of crystalline Fe₃B appear. Since this time limit is not very reproducible and it definitely occurs even before the full elimination of α-Fe when the vacuum is deteriorated, this finding is attributed to the disturbing effect of boron oxidization, as it is found for the mechanical milling of crystalline FeB.

Transmission electron micrographs as well as SAED patterns characterizing the microstructure of the early stage (24 h milling) and the fully amorphous sample (240 h milling) are displayed in Figs. 2 and 3.

In the early stage the sample consists of multiple phases. One of them is crystalline α-Fe with a typical grain size between 10 and 50 nm. An amorphous phase in globules of 100–300 nm is also observed. This latter phase gives a bright contrast in Fig. 2(b) but dark contrast in Fig. 2(c) indicating that its scattering is highest inside the first diffraction ring of α-Fe [see also Fig. 7(d)]. It is thus established to be a boron-rich phase. Since in the Mössbauer spectra shown in Fig. 1 no paramagnetic compound is found, this phase should be identified with essentially pure amorphous boron without any measurable Fe content. The Fe-containing amorphous phase observed in the Mössbauer spectra cannot be separately identified by electron microscopy at this early stage of milling. The presence of another crystalline phase (different from α-Fe) is suggested by a few sharp spots on the dark field electron micrograph of Fig. 2(b) taken with the objective aperture positioned well inside the first α-Fe ring. Due to its small volume fraction this third phase could not be identified by SAED.

After 240 h of milling (Fig. 3) the amorphous Fe-B alloy becomes the majority phase. In bright field electron micrographs this amorphous phase shows an irregular, striped, wavy contrast with a typical stripe width of a few times 10 nm, similar to other mechanically alloyed powders. The amorphous boron-rich phase (identified as essentially pure amorphous boron in the previous sample) was found to be the second most abundant phase in the form of 50–100-nm-diam globules. On the dark field micrographs taken with the objective aperture on the most intense diffraction ring [Fig. 3(c)] a few crystalline grains could also be observed, but not identified because of the coincidence on the SAED pattern of the weak crystalline reflections with the strongest amorphous ring.

Mössbauer spectra of the amorphous phase obtained after different milling times are shown in Fig. 4 after the subtraction of the α-Fe contribution. The corresponding hyperfine field distributions determined from these spectra are displayed in Fig. 5. The average hyperfine field and isomer shift belonging to different milling times and the standard width of the hyperfine field distributions are presented in Table I. Since all the spectra were fitted by two binomials the position of the main peak and its standard width is given too.

Table I contains the fraction of α-Fe subtracted before fitting the hyperfine field distribution of the amorphous part. For comparison, the parameters of a melt quenched Fe₄₅B₃₀ sample are also shown.

The composition dependence of the hyperfine parameters of amorphous Fe-B alloys is known both in melt quenched and in sputtered alloys. The average isomer shift of amorphous Fe-B alloys is known to increase with B content below about 50 at. % B. The B composition dependence of the average hyperfine field measured at room...
FIG. 4. Mössbauer spectra of the amorphous component after different milling times and after a consecutive heat treatment. Spectra belonging to 31 and 90 h milling times were obtained from the measured spectra of Fig 1 by subtracting the α-Fe subspectra.

The Mössbauer parameters of Table I, and the hyperfine field distributions of Fig. 5 show an obvious trend. For short milling times one can observe a very broad distribution of hyperfine fields which has a peak around 25 T and a low field tail at around 10 T. As the milling time increases, the main peak of the distribution gradually shifts to lower values and the width of the whole distribution as well as that of the main peak is reduced. As compared to melt quenched amorphous alloys, the width of the hyperfine field distribution is more than twice the usual value for short milling times and is at least 10% larger than the appropriate value even in case of

amorphous Fe-B phase

amorphous B-rich phase

FIG. 3. Bright field (a) and dark field [(b) and (c)] electron micrographs and diffraction pattern (d) of the mechanically alloyed powder mixture after 240 h milling. The position of the objective aperture for dark field imaging is indicated in (d).

FIG. 5. Hyperfine field distributions belonging to the fitted curves of Fig. 5. (31 h—small dashes, 90 h—medium dashes, 170 h—large dashes, 240 h—full line, after DSC at 750 K—thick dots).

temperature is found\textsuperscript{23} to decrease with the B concentration above about 20 at. % B. The composition of the amorphous phase formed by mechanical alloying will be estimated in the following on the basis of these data.
the longest milling time. It is plausible to assign the excess width of the hyperfine field distributions to concentration inhomogeneities. Therefore we can say that for short milling times (24, 31 h) the dominant fraction of the amorphous phase contains 20±5 at. % B, but a smaller amount of a larger (about 50 at. %) B content phase is also present, as reflected in the low field tail of the hyperfine field distribution. Since the intensity of this component never exceeds 10% in the whole milling process, it is difficult to tell if this is an amorphous or a crystalline phase. We could not find clear evidence of the possible presence of crystalline FeB either by MS or by TEM. As the milling time increases, the amorphous phase is gradually homogenized and its average B concentration is shifted to higher values. This is also reflected in the increase of the average isomer shift. However, even in case of the longest milling time, the width of the distribution is at least 10% larger than the value for melt quenched or evaporated samples with similar average hyperfine field. For milling times after α-Fe is fully eliminated (170 h), the main component can be estimated as a 32 at. % B content phase and the width of the distribution reaches its steady state value, which cannot be reduced either by further milling or by heat treatment. After the longest milling time examined (240 h), the average B concentration is about 35 at. % with less than 2 at. % inhomogeneity.

The thermal stability of the probe with the longest milling time (240 h) was investigated by DSC. A characteristic curve is displayed in Fig. 6: the rate of transformation, \( \frac{dC}{dt} = \frac{1}{SH} \frac{dh}{dt} \), where \( dh/dt \) is the power measured by DSC and \( \delta H \) is its integrated value (i.e., the total heat of the transformation) is plotted vs T. The measured value for \( \delta H \) is 300 J/g, which is surprisingly high. It is more than 50% above the highest value reported for the heat of crystallization of different melt quenched amorphous alloys.\(^9\)\(^,\)\(^14\)

For mechanically alloyed probes, the previously reported\(^17\) heat of transformation is also higher than that of melt quenched alloys (it is on the other hand significantly below the value found here). It is reasonable to assume that besides the crystallization some other exothermic process is also manifested in the calorimetric measurement.

In order to verify the above assumption, a portion of the probe was heat treated in the calorimeter just to the minimum between the two peaks in Fig. 6. This way it is possible to investigate the structure which is formed in the broad heat evolution.

First the TEM results of the as-milled and the heat treated probes are compared in Figs. 3 and 7. The similarity of the two pictures is striking. There is no visible change in

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### TABLE I

<table>
<thead>
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<th>t (h)</th>
<th>( c_{Fe} ) (%)</th>
<th>( H_{Fe} ) (T)</th>
<th>( \sigma_{Fe} ) (T)</th>
<th>( H_{max} ) (T)</th>
<th>( \sigma ) (T)</th>
<th>IS (mm/s)</th>
<th>T(K)</th>
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<td>3.8</td>
<td>0.14</td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
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<td>0</td>
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<td>4.7</td>
<td>21.0</td>
<td>3.4</td>
<td>0.15</td>
<td>293</td>
</tr>
<tr>
<td>Fe(<em>6)B(</em>{42})</td>
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the microstructure or in the diffraction pattern, i.e., the same phases can be found in the heat treated and in the as-milled sample. Besides, Figs. 7(e) and 7(f) demonstrate that the amorphous Fe-B phase formed by solid state reaction can be distinguished from the amorphous B phase by means of SAED technique. This way it is verified, that in the broad transformation observed between 550 and 750 K by calorimetry, no new crystalline phase is formed. The joint results of the DSC and TEM measurements show that the broad transformation is the solid state reaction between the amorphous Fe-B alloy and the unalloyed (also amorphous) B.

The room-temperature Mössbauer spectrum of the heat treated sample is shown at the bottom of Fig. 4. The hyperfine field distribution belonging to this spectrum is drawn with a thick full line in Fig. 5 and the parameters can be found in Table I. A significant decrease of the average hyperfine field and an increase of the isomer shift is observed. The width of the hyperfine field distribution remains unchanged as compared to the as-milled sample. This also supports the idea that the broad heat evolution peak in the DSC curve is due to the reaction of the unalloyed B with the amorphous phase. More precise verification of the composition change is made possible by measuring the Mössbauer spectra of the as-milled and the heat treated alloy at helium temperature and above the Curie temperature.

Mössbauer spectra of these probes were taken at 4.2 K. Parameters of the hyperfine field distribution at this temperature are given in Table I. Since these data are not influenced by the Curie point differences, the measured hyperfine fields are characteristic of the iron magnetic moments, that is of the B concentration of the amorphous phase. The Curie temperature, which can only be measured in case of the heat treated sample, is a further check of the boron concentration. The heat treated probe becomes paramagnetic at 693 K. The quadrupole splitting (0.55 mm/s) and the isomer shift (−0.14 mm/s at this temperature) is also in good agreement with figures for amorphous Fe_{50}B_{40} alloys. On the basis of the 4.2 K hyperfine field values and the measured Curie temperature, we estimate the B concentration of the as milled and the heat treated probe as 35±2 at. % and 40±2 at. %, respectively. The relative change of the concentration as determined from the decrease of the average hyperfine field at 4.2 K is 5±1 at. %.

IV. DISCUSSION

It is a remarkable feature of the Fe-B system that the mechanical milling results neither in the dissolution of B in bcc-Fe nor in the dissolution of Fe in the B structure. This is quite evident in Fig. 1 where the room-temperature Mössbauer spectra of the probes from different stages of milling are displayed. Dissolution of B in bcc-Fe is expected to show up in a low field satellite of the characteristic six-line pattern of pure bcc-Fe, but within the experimental error no change of the linewidth, the hyperfine field, or the isomer shift is observed. On the other hand the appearance of a paramagnetic peak is expected to indicate the solution of Fe in B, which is also not the case. The well-known fact that the mutual equilibrium solubility of B and Fe is negligible does not reduce this observation to a trivial result. Mechanical grinding is known to decrease the grain size significantly. In this way the fraction of atoms which are located so near to the grain boundary, that the solubility is enhanced by several orders of magnitude, can be increased. For a similar system with mutual insolubility, Fe-Y, the alloying near to grain boundaries has already been reported. Although the reduction of the grain size is evident from the present TEM results no dissolution of either Fe or B in the other crystalline phase is observed. This fact is especially remarkable at low α-Fe fractions and it indicates that atomic intermixing of Fe and B takes place exclusively at the nanocrystalline grain boundaries. This way, the B diffusion results in nanocrystalline Fe_{50}B_{40} are highly relevant for the understanding of SSAR in Fe-B powder mixtures.

The absence of crystalline solid solution is possibly connected to the size difference of Fe and B, which makes both interstitial and substitutional solution very unstable thermodynamically. This way, the intermixing of Fe and B is only possible by the formation of a separate amorphous phase. The hyperfine parameters of Table I clearly indicate that the alloy formation starts in a broad concentration range, i.e., from 20 to 50 at. % B for the shortest milling time when reliable parameters can be obtained after subtracting the majority α-Fe spectrum. However, one has to notice that in this stage more than 40% of the Fe atoms is already in the amorphous phase. Due to experimental limitations, no reliable Mössbauer parameters can be determined for the amorphous phase below this limit, i.e., the evolution of the amorphous phase cannot be followed from the very beginning of its formation. With prolonged milling not only does the alloying proceed, but the amorphous phase is gradually homogenized and the average boron content of the amorphous phase is increased. These changes of the B concentration are easily understood if B is the component with the higher mobility: in this case a low B content amorphous phase is formed first.

It is also evident from the data of Table I that the disappearance of α-Fe does not indicate the end of the alloying process, the B enrichment of the amorphous phase continues to higher milling times. No amorphous phase with a steady-state B content is achieved. In several independent milling runs, the composition of the amorphous phase when α-Fe is eliminated is around 32 at. % B, and it can be enhanced to 35 at. % B by further milling. However, these figures might depend on the milling intensity, which was not examined. Even in this stage, the width of the hyperfine field distribution is at least 10% larger than that of the melt quenched sample which can result from a few percent boron content inhomogeneity in the sample.

The boron content of the amorphous phase can be effectively increased by heat treatment. Approximately 5 at. % B content increase was found when the as-milled sample was heat treated in the DSC up to 750 K with a 20 K/min heating rate. Heat treatment for one day at 570 K in a vacuum furnace resulted in the same composition change. According to both the TEM results and the stoichiometry considerations, unalloyed B should remain in the sample even after these heat treatments. This might indicate that the Fe_{50}B_{40} composition is around the thermodynamic limit where the amorphous phase can be formed by pure solid state diffusion.
All these results suggest the special role of the B diffusion. Although Fe-B is not a fast diffusing system (in the sense established, for example, for Ni-Zr), the relevant B mobility might exceed that of the Fe atoms. The work of Höfler et al. makes it clear, that in a nanograin structure the diffusion of B is determined by the grain boundary contribution. Our results together with the findings of Ref. 38 indicate, that while the averaged diffusion of Fe and B might be comparable, the component of B diffusion which determines solid state amorphization should be significantly higher than the relevant value for Fe. This fact might explain why the Fe-B system shows mechanical amorphization without the asymmetric (bulk) diffusivity, which cannot be further assumed to be a necessary requirement for mechanical amorphization.

V. CONCLUSION

It is shown in this study, that mechanical alloying of Fe and B powder yields an amorphous structure. Despite the significant enhancement of the grain boundary fraction, crystalline solid solution phase is not formed. The B concentration of the alloy phase is shown to be very inhomogeneous at the early stage of milling, stretching up to 50 at. % when the average is definitely below 25 at. %. With the increase of the milling time, the amorphous fraction increases, its heterogeneity decreases, and the average boron content of the major alloy phase gradually increases. The end product is an amorphous alloy containing 35 at. % B with a composition heterogeneity of about 2 at. %. Heat treatment at relatively low temperature enhances the B content to 40 at. % without significantly affecting the composition heterogeneity. The observed mechanical amorphization in the non-fast-diffusing Fe-B system indicates that the asymmetry of the bulk diffusion values is not a necessary requirement for the amorphization in a nanograin structure.

ACKNOWLEDGMENTS

The financial support of OTKA-T4469 is gratefully acknowledged. We wish to thank Dr. L. Gránásy for providing the melt quenched Fe\textsubscript{66}D\textsubscript{40} sample and the Soros Foundation for covering the publication fee.

24. H. Rauch, 1993 (private communication).