Influences of B/Fe Composition and Substrate Temperature on the Structure of Fe-B Alloy Film Formed on MgO(001) Substrate

Mitsuru Ohtake, Tetsuroh Kawai, Yugo Asai, Masaaki Futamoto, and Nobuyuki Inaba*

Faculty of Science and Engineering, Chuo University, 1-13-27 Kasuga, Bunkyo-ku, Tokyo 112-8551, Japan
Faculty of Engineering, Yamagata University, 4-3-16 Jonan, Yonezawa, Yamagata 992-8510, Japan

Fe-B alloy films of 40 nm thickness are prepared on MgO(001) single-crystal substrates using alloy targets of Fe\textsubscript{100-x}B\textsubscript{x} (x = 0, 8, 13, 18, 25 at. %) by varying the substrate temperature from room temperature to 600 °C. The structure is systematically investigated by reflection high-energy electron diffraction, X-ray diffraction, and cross-sectional high-resolution transmission electron microscopy. (001) single-crystal films with bcc-based disordered \textit{A2} structure epitaxially grow at the investigated substrate temperatures for the B contents less than 8 at. %. On the contrary, the Fe\textsubscript{87}B\textsubscript{13}, the Fe\textsubscript{82}B\textsubscript{18}, and the Fe\textsubscript{75}B\textsubscript{25} films respectively deposited at temperatures lower than 200, 400, and 600 °C involve amorphous. The crystallization temperature increases with increasing the B content. The surface roughness increases with increasing the substrate temperature. Island-like surfaces are formed for the films deposited at elevated temperatures. The (001) films show four-fold symmetries in in-plane magnetic anisotropy, whereas the amorphous films show isotropic magnetic properties. The coercivity increases with increasing the substrate temperature due to that domain wall motion is suppressed by the crevasses in the films existing between large islands.

**Key words:** Fe-B alloy, epitaxial thin film growth, crystal structure, bcc-based disordered \textit{A2} structure, amorphous, microstructure, MgO(001) single-crystal substrate

1. **Introduction**

Soft magnetic materials with high permeability and high saturation magnetization have been investigated for applications like magnetic sensors, transformers, motors, etc. Alloys consisting of Fe and light element such as Fe-B, Fe-C, Fe-N, etc. are the typical soft magnetic materials.

Fe-B alloys prepared by using liquid quenching\textsuperscript{1-9} and film growth\textsuperscript{10-18} techniques tend to involve amorphous. In order to investigate the basic structural and magnetic properties, it is useful to employ a film deposited on a single-crystal substrate, since the crystallographic orientation can be controlled by the substrate orientation. However, there are few reports on the preparation of Fe-B films on single-crystal substrates\textsuperscript{19-21}. The relationships of B content and film growth temperature to the structure have not been made clear.

Electron diffraction is more sensitive in distinguishing the amorphous and the crystal than X-ray diffraction (XRD). On the other hand, an XRD system has better measurement precision of lattice spacing when compared with that obtainable with an electron diffraction system. A combination of electron diffraction and XRD is thus useful for the structural characterization. In the present study, Fe-B films are prepared on MgO(001) single-crystal substrates by employing an ultra-high vacuum sputtering system equipped with a reflection high-energy electron diffraction (RHEED). The influences of B content and substrate temperature on the structure are systematically investigated by RHEED, XRD, and high-resolution transmission electron microscopy (HR-TEM). The magnetic properties are also measured by vibrating sample magnetometry (VSM).

2. **Experimental Procedure**

A radio-frequency (RF) magnetron sputtering system was used for film preparation. The base pressures were lower than $4 \times 10^{-7}$ Pa. Polished MgO(001) substrates were heated at 600 °C for 1 hour before film formation to obtain clean surfaces. The cleanliness was checked by RHEED and the arithmetical mean surface roughness value, $R_\alpha$, estimated by atomic force microscopy (AFM) was lower than 0.1 nm.\textsuperscript{22}

Fe\textsubscript{100-x}B\textsubscript{x} (x = 0, 8, 13, 18, 25 at. %) alloy targets of 3 inch diameter were employed, and, in the present study, the film compositions were regarded to be similar to the target compositions. The distance between target and substrate and the Ar gas pressure were respectively kept constant at 150 mm and 0.67 Pa. Fe-B films of 40 nm thickness were deposited on the MgO(001) substrates at temperatures ranging from room temperature (RT) to 600 °C.

The crystal structure was studied by RHEED. The structural properties were investigated by 2θ-ω scan out-of-plane and 2θ-θ ϕ χ scan in-plane XRDs with Cu-Kα radiation (λ = 0.14518 nm). The cross-sectional microstructure and the surface morphology were respectively observed by HR-TEM and AFM. The magnetization curves were measured by VSM.
Fig. 1 RHEED patterns observed for (a) Fe, (b) Fe₉₂B₈, (c) Fe₇₇B₁₃, (d) Fe₆₃B₁₈, and (e) Fe₇₅B₂₅ films deposited on MgO(001) substrates at (a-1)–(e-1) RT, (a-2)–(e-2) 200 °C, (a-3)–(e-3) 400 °C, and (a-4)–(e-4) 600 °C. The incident electron beam is parallel to MgO[100].

Fig. 2 Schematic diagrams of RHEED patterns of (a) A₂-(Fe,B)(001) single-crystal, (b) Fe-B amorphous, (c)–(f) A₂-(Fe,B)(c) (001)-, (d) (011)-, (e) (111)-, and (f) (112)-oriented polycrystals, (g) C₁₆-Fe₂B(100) crystals, and (h) C₁₆-Fe₂B(001) single-crystal simulated by using the lattice constants of [(a), (c)–(f)] aₐ = 0.2866 nm and [(g), (h)] (a, c)ₐ = (0.5112 nm, 0.4248 nm). The incident electron beam is parallel to [(a), (h)] [100], (g-2) [001], or (g-3) [010]. Schematic diagram of (g-1) is drawn by overlapping (g-2) and (g-3).

3. Results and Discussion

3.1 Crystal structure

Figures 1(a) and (b), respectively, show the RHEED patterns of Fe and Fe₉₂B₈ films deposited on MgO(001) substrates at different temperatures observed by making the incident electron beam parallel to MgO[100]. Figure 2(a) shows the schematic diagram of diffraction pattern calculated for (001) crystal with bcc-based disordered A₂ structure formed in the orientation relationship of A₂-(Fe,B)(001)[110] || MgO(001)[100]. The observed RHEED patterns are in agreement with the simulated pattern. Fe and Fe₉₂B₈(001) single-crystal films with A₂ structure are obtained. In this configuration, the lattice mismatch between film and substrate is calculated to be

\[
\left(\frac{a_{A2(Fe,B)} - a_{MgO}}{a_{MgO}}\right) \times 100 = -4\%,
\]

3. Results and Discussion
Fig. 3  Cross-sectional HR-TEM image of an Fe$_{87}$B$_{13}$ film deposited on MgO(001) substrate at RT observed along MgO[100].

Fig. 4  Cross-sectional HR-TEM image of an Fe$_{87}$B$_{13}$ film deposited on MgO(001) substrate at 200 °C observed along MgO[100].
Fig. 5  (a) Cross-sectional HR-TEM image of an Fe$_{87}$B$_{13}$ film deposited on MgO(001) substrate at 400 °C observed along MgO[100]. (b) Enlarged view of the area surrounded by white square lines in (a). (c) FFT pattern of (a). (d) Inverse FFT image of the areas reproduced by employing the FFT spots surrounded by white circles in (c). \( \parallel \) and \( \perp \) marks show the dislocation positions.
where the lattice constants of bulk $A2$-Fe ($a_{A2-Fe} = 0.2866$ nm$^{20}$) and MgO ($a_{MgO} = 0.4217$ nm$^{24}$) crystals are used.

Figure 1(c-1) shows the RHEED pattern observed for an Fe$_{87}$B$_{13}$ film deposited at RT. A diffuse diffraction pattern typical for amorphous [Fig. 2(b)] is recognized. TEM observation was carried out to investigate the cross-sectional microstructure. Figure 3 shows the HR-TEM image of the Fe$_{87}$B$_{13}$ film deposited at RT observed along MgO[100]. Although the film possesses $A2$ structure in a region close to the MgO substrate, the structure varies to amorphous away from the substrate. The growth behavior of Fe$_{87}$B$_{13}$ film at RT will be explained as follows by considering the TEM result. First, $A2$[001] crystal nucleates on the MgO[001] substrate, where the crystallization is promoted through heteroepitaxial growth. As the thickness increases from 1 to 2 nm, amorphous starts to coexist with the crystal and the volume ratio of amorphous to crystal increases. With further increasing the thickness, the amorphous covers the surface. As the result, a diffuse RHEED pattern is considered to be observed for the 40-nm-thick film [Fig. 1(c-1)], since the RHEED detects the crystallographic information from the surface.

Figure 1(c-2) shows the RHEED pattern of an Fe$_{87}$B$_{13}$ film deposited at 200 °C. A ring-like diffraction pattern is overlapped with a diffuse pattern, indicating that the film is composed of polycrystal and amorphous. Figures 2(c–d)–(f) show the schematic diagrams of RHEED patterns of (001)-, (011)-, (111)-, and (112)-oriented $A2$ polycrystals simulated by using the lattice constant of $a = 0.2866$ nm$^{20}$. The ellipses in the calculated patterns show the positions where strong reflections are expected to appear. The observed ring-like pattern almost agrees with the pattern calculated for (001)-oriented polycrystal. The preferred orientation of Fe–B polycrystal is (001), though the surface free energy of (110) is the lowest for the $A2$ crystal. Figure 4 shows the TEM image of the film deposited at 200 °C. In the early stage of film growth, $A2$(001) crystal is expected to grow epitaxially on the substrate, similar to the case of film growth at RT [Fig. 3]. As the thickness increases beyond 1 nm, amorphous starts to appear, as shown by the areas surrounded by blue dotted lines. However when the thickness is further increased, the volume ratio of amorphous to crystal does not change much, indicating that the film around the surface also consists of a mixture of crystal and amorphous. The TEM result is consistent with the RHEED result obtained during the film deposition process. In order to investigate the mechanism of phase separation, a chemical analysis like electron energy-loss spectroscopy seems necessary. It is also noted in the TEM micrograph that the lattice lines are bending and are not continuous in some regions within an Fe–B crystal. The crystallographic quality causes an increase in the orientation dispersion of $A2$(001) crystal. As the result, the structure is considered to vary from $A2$(001) single-crystal to $A2$(001)-oriented polycrystal when the thickness is increased.

Figures 1(c-3) and (c-4) show the RHEED patterns of Fe$_{87}$B$_{13}$ films deposited at 400 and 600 °C, respectively. Clear patterns from $A2$(001) single-crystals are observed, similar to the cases of Fe and Fe$_{86}$B$_{14}$ films. Figure 5(a) shows the TEM image of the film deposited at 400 °C. Dislocations are introduced as shown, for example, in the enlarged view of Fig. 5(b). In order to investigate the dislocation distribution, fast Fourier transformation (FFT) was carried out for the TEM image. Figure 5(c) shows the FFT pattern of Fig. 5(a). Figure 5(d) shows the inverse FFT image of the areas reproduced by employing the FFT spots surrounded by the circles in Fig. 5(c), where $\parallel$ and $\perp$ marks show the dislocation positions. Many dislocations are recognized near the film/substrate interface as shown in Fig. 5(d). It is known that misfit dislocations are introduced around the film/substrate interface to reduce the lattice mismatch when a weak binding force is working between the deposited atoms and the substrate atoms. This type of epitaxial growth is reported for $A2$-Cr$^{26}$, $A2$-Fe$_{50}$Co$_{50}$$^{27}$, $A1$-Ni$_{80}$Fe$_{20}$$^{28}$), and $A3$-Co$^{29}$ films deposited on MgO[001] substrates. The Fe–B films prepared in the present study also seem to follow the growth mode.

Figures 1(d) and (e) show the RHEED patterns of Fe$_{86}$B$_{18}$ and Fe$_{75}$B$_{25}$ films, respectively. Diffuse patterns are recognized for the Fe$_{86}$B$_{18}$ films deposited below 200 °C [Figs. 1(d-1), (d-2)] and the Fe$_{75}$B$_{25}$ films deposited below 400 °C [Figs. 1(e-1)–(e-3)]. Diffuse patterns are overlapped with patterns from $A2$(001) crystals for the Fe$_{86}$B$_{18}$ film deposited at 400 °C [Fig. 1(d-3)] and the Fe$_{75}$B$_{25}$ film deposited at 600 °C [Fig. 1(e-4)]. A clear pattern from $A2$(001) crystal is observed.
XRD patterns measured for (a) Fe, (b) Fe$_{92}$B$_{8}$, (c) Fe$_{87}$B$_{13}$, (d) Fe$_{82}$B$_{18}$, and (e) Fe$_{75}$B$_{25}$ films deposited on MgO(001) substrates at 600 °C. The scattering vector of in-plane XRD is parallel to MgO[110]. The intensity is shown in logarithmic scale.

Figure 7 shows the out-of-plane and in-plane XRD patterns measured for (a) Fe, (b) Fe$_{92}$B$_{8}$, (c) Fe$_{87}$B$_{13}$, (d) Fe$_{82}$B$_{18}$, and (e) Fe$_{75}$B$_{25}$ films deposited on MgO(001) substrate. The film involves a small volume of C16(100) crystal shown in Fig. 2(g). The reason is possibly due to the formation of tetragonal ordered C16 structure (i.e. FeB$_2$ phase) is not included in the reflections from C16(100) crystal. The RHEED pattern observed for the film [Fig. 6(a)]. Weak out-of-plane $A_2(002)$ reflection is recognized for the film deposited at 200 °C [Fig. 6(b)]. Strong out-of-plane $A_2(002)$ and in-plane $A_2(200)$ reflections are observed for the film deposited at 400 °C [Fig. 6(c)].

Figure 8 shows the phase diagram of Fe-B alloy film deposited on MgO(001) substrate. The phase diagram shows the crystallization temperatures of Fe$_{92}$B$_{8}$ films are respectively around 400 and 600 °C, Amorphous formation is enhanced with increasing the B content.

Figure 6 shows the out-of-plane and in-plane XRD patterns measured for Fe$_{92}$B$_{8}$ films deposited at RT, 200 °C, and 400 °C which are respectively typical for films consisting of amorphous, a mixture of amorphous and polycrystal, and $A_2(001)$ single-crystal. Here, the in-plane XRD patterns are measured by making the scattering vector parallel to MgO[110]. There are only the reflections from substrate in the out-of-plane and in-plane patterns measured for the film deposited at RT [Fig. 6(a)]. Weak out-of-plane $A_2(002)$ reflection is recognized for the film deposited at 200 °C [Fig. 6(b)]. Strong out-of-plane $A_2(002)$ and in-plane $A_2(200)$ reflections are observed for the film deposited at 400 °C [Fig. 6(c)].

Figure 7 shows the XRD patterns measured for films with different B contents deposited at 600 °C. It is noted that weak reflections from (100) crystal with tetragonal ordered C16 structure (i.e. FeB$_2$ phase) is appearing around the diffraction angles, 2θ of 35° and 74° in the out-of-plane pattern measured for Fe$_{75}$B$_{25}$ film. The film involves a small volume of C16(100) crystal. The RHEED pattern observed for the film [Fig. 1(e-4)] dose not include the reflections from C16(100) crystal shown in Fig. 2(g). The reason is possibly due to...
that the volume of C16(100) crystal is so small to be detected by RHEED. By considering the RHEED and the XRD data, the phase diagram of 40-nm-thick Fe-B film deposited on MgO(001) substrate is determined as shown in Fig. 8.

3.2 Lattice strain

Figure 9 shows the lattice parameters, \(a\), \(c\), and \(c/a\), of films consisting of only the \(A2(001)\) crystals. Here, these values are calculated from the XRD data by using the relations of

\[
a = 2 \lambda_1 / [2 \sin(\theta_2)_{A2(000)}],
\]

\[
c = 2 \lambda_1 / [2 \sin(\theta_2)_{A2(000)}].
\]

The \(a\) and the \(c\) values of Fe and Fe-B films are in agreement with the value of bulk \(\gamma\)-Fe crystal \((d_{\gamma-Fe} = 0.2866 \text{ nm})\) with small errors less than \(\pm1.1\%\), though there exist the mismatches of about \(-4\%\) at the film/substrate interfaces. The dislocations introduced around the film/substrate interfaces are considered to reduce the strains caused by the lattice mismatches, as shown in the TEM image of Fig. 5.

The \(a\) and the \(c\) values of Fe films are respectively larger and smaller than those of the bulk. The in-plane and the out-of-plane lattices are respectively expanded and shrunk in accommodation of the lattice mismatch with minus sign. With increasing the substrate temperature from RT to 600 °C, the \(c/a\) value increases from 0.979 to 0.998, indicating that the strain decreases with increasing the substrate temperature. Many dislocations seem to be introduced in an earlier stage of film growth, as the substrate temperature increases. On the contrary, both \(a\) and \(c\) values of Fe-B films are larger than those of the bulk Fe. The crystal lattice in Fe-B film is apparently expanding by addition of B atoms. Furthermore, the \(c/a\) values are nearly 1. The lattice deformations of Fe-B films are not so large.

Figure 10 shows the out-of-plane and in-plane orientation dispersions, \(\Delta \theta_0\) and \(\Delta \theta_{\chi\theta_0}\), which are respectively the full widths at half maximum of \(\phi\) and \(\theta\) scan rocking curves measured by fixing the diffraction angles of \(\theta\) and \(\chi\) at the peak angles of out-of-plane \(A2(002)\) and in-plane \(A2(200)\) XRD reflections. The \(\Delta \theta_0\) and the \(\Delta \theta_{\chi\theta_0}\) values decrease with increasing the substrate temperature, whereas those increase with increasing the B content. B atom addition into Fe material enhances the orientation dispersion. The result seems to be related to the structural variation from \(A2(001)\) single-crystal to \(A2(001)\)-oriented polycrystal observed for the Fe82B18 film deposited at 200 °C.

3.3 Surface morphology and magnetic properties

Figure 11 shows the AFM images observed for Fe, Fe82B18, and Fe75B25 films. Figure 12 shows the substrate temperature dependences of \(R_s\) values of Fe and Fe-B films. The growth of Fe and Fe-B films seems...
to follow the Volmer-Weber\textsuperscript{(10)} (island-growth) mode. As the substrate temperature increases, the $R_a$ value increases for all the B contents. The films deposited at 600 °C are composed of large islands with diameters of 200–1000 nm. Migration and clustering of deposited atoms are promoted by employing a higher substrate temperature.

Figure 13(a) shows the magnetization curves of Fe films measured by applying the magnetic field along MgO[100] (\textit{||} A2-Fe[110]) or MgO[110] (\textit{||} A2-Fe[100]). The films deposited at temperatures lower than 400 °C are easily magnetized when the magnetic field is applied along $A_2[100]$, while the magnetization curves measured along $A_2[110]$ saturate at higher fields [Figs. 13(a-1)–(a-3)]. There were no clear differences in the hysteresis curves measured along MgO[100] (\textit{||} A2-Fe[110]) and MgO[010] (\textit{||} A2-Fe[110]) and measured along MgO[110] (\textit{||} A2-Fe[100]) and MgO[110] (\textit{||} A2-Fe[010]) (not shown here). Therefore, these films show four-fold symmetries in in-plane magnetic anisotropy, reflecting the magnetocrystalline anisotropy of bulk $A_2$-Fe crystal with the easy magnetization axes of <100>. The film deposited at 600 °C shows almost isotropic in-plane magnetic property [Fig. 13(a-4)]. The magnetic anisotropy is considered to be influenced by the shape anisotropy caused by the surface undulation which is more enhanced by using a higher substrate temperature. The coercivity increases from 25 to 75 Oe.

![Fig. 12](image-url) Substrate temperatures dependences on the $R_a$ values of Fe, Fe$_{87}$B$_{13}$, Fe$_{82}$B$_{18}$, and Fe$_{75}$B$_{25}$ films.

![Fig. 13](image-url) Magnetization curves measured for (a) Fe, (b) Fe$_{82}$B$_{18}$, (c) Fe$_{87}$B$_{13}$, (d) Fe$_{82}$B$_{18}$, and (e) Fe$_{75}$B$_{25}$ films deposited on MgO(001) substrates at (a-1)–(e-1) RT, (a-2)–(e-2) 200 °C, (a-3)–(e-3) 400 °C, and (a-4)–(e-4) 600 °C. The coercivities are measured by applying the magnetic field along MgO[110].
with increasing the substrate temperature from RT to 600 °C. Domain wall motion seems to be suppressed by the crevasses in the film existing between large islands, as shown in the AFM image of Fig. 11(a-4).

Figures 13(b)–(e) show the $M-H$ curves of Fe-B films. Isotropic magnetization curves are observed for the Fe-B films consisting of amorphous [Figs. 13(b-1), (d-1)–(d-2), (e-1)–(e-3)], Easy magnetization direction is observed along MgO[110] (|| A2-(Fe,B)(100)) for the Fe-B films consisting of A2(001) crystal formed at low substrate temperatures [Figs. 13(b-1)–(b-3), (c-2)–(c-3), (d-3)], whereas the A2-(Fe,B)(001) films formed at high substrate temperatures show four-fold symmetries in in-plane magnetic anisotropy, whereas the amorphous films and the A2 films deposited at elevated temperatures show isotropic magnetic properties [Figs. 13(b-4)–(e-4)]. These behaviors are similar to the case of Fe films. The magnetic anisotropy is influenced by the magnetocrystalline anisotropy and the shape anisotropy.

4. Conclusion

Fe-B alloy films are prepared on MgO(001) substrates by varying the B content from 0 to 25 at. % and by varying the substrate temperature from RT to 600 °C. The detailed structure is investigated by RHEED, XRD, and HR-TEM. A2-Fe and A2-Fe$_8$B$_8$ single-crystal films of (001) orientation are obtained at the investigated substrate temperatures. The Fe$_{87}$B$_{13}$, the Fe$_{75}$B$_{25}$, and the Fe$_{78}$B$_{22}$ films respectively deposited at temperatures lower than 200, 400, and 600 °C involve amorphous. The crystallization temperature increases at temperatures lower than 200, 400, and 600 °C involve amorphous. The crystallization temperature increases at temperatures lower than 200, 400, and 600 °C involve amorphous. The crystallization temperature increases at temperatures lower than 200, 400, and 600 °C involve amorphous. The crystallization temperature increases at temperatures lower than 200, 400, and 600 °C involve amorphous. The crystallization temperature increases at temperatures lower than 200, 400, and 600 °C involve amorphous. The crystallization temperature increases at temperatures lower than 200, 400, and 600 °C involve amorphous. The magnetic anisotropy is influenced by the magnetocrystalline anisotropy and the shape anisotropy.

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