Electron Irradiation Induced Phase Transformation in Nd₂Fe₁₄B Alloy

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Transformation and change in the microstructure of Nd₂Fe₁₄B compound under electron irradiation were examined. The Nd₂Fe₁₄B compound did not maintain its original structure under electron irradiation and was transformed to an amorphous phase through solid-state amorphization. With further electron irradiation, crystallization of the amorphous phase occurred forming a nano-crystalline structure. The phase selection of electron irradiation induced crystallization depended strongly on the irradiation temperature; nanocrystalline α -Fe phase precipitated in the amorphous phase at 104 K, while a nanoduplex structure composed of α -Fe, compounds and residual amorphous phase was formed at 298 K. Electron irradiation induced phase transformation is a very effective method to control the nanocrystalline structure in Fe-Nd-B alloy. [doi:10.2320/matertrans.47.1762]

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1. Introduction

Fe-Nd-B alloys are known to be superior hard magnetic materials.¹⁻⁴ Their nanocrystalline structure is effective in effecting remarkable improvement in the mechanical and functional properties of metallic materials. To improve the hard magnetic property of these alloys, numerous systematic studies have been made to obtain the optimum nanocrystalline structure by controlling the annealing condition and/or solidification process.⁵⁻⁸ Recently, electron irradiation induced crystallization and formation of the nanocrystalline structure were reported in several alloys. Nanocrystallization of an amorphous phase has been found in Fe_{77.5}Nd_{4.5}B_{18.5} and Fe₈₆Nd₉B₅ under high energy electron irradiation to date.⁹⁻¹² On the other hand, constituent atoms in a crystalline lattice are elastically replaced by electron knock-on effect and the crystalline structure is destroyed resulting in amorphization. The solid-state amorphization was confirmed in more than 30 crystalline alloys under electron irradiation.¹³ This phenomenon was observed in Fe₈₆Nd₉B₅ alloy with multi-phase crystalline structure.^{13,14} More recently, the phenomena of both amorphization and crystallization were found during electron irradiation in Zr-based metallic glasses: crystal-to-amorphous-to-crystal (C-A-C) transition in Zr_{66.7}Cu_{33.3}¹⁴ and quasicrystal-to-amorphous-to-crystal (Q-A-C) transition in Zr_{66.7}Pd_{33.3}¹⁵ metallic glasses. A unique nanocrystalline structure which could not be obtained by the thermal process was formed through C-A-C type phase transformation in Zr-based alloys, although this C-A-C transition in Fe-based alloys has never been reported.

In the present study, electron irradiation induced phase transformation behavior on Nd₂Fe₁₄B compound was investigated to evaluate the possibility of realizing nanocrystalline structure formation in a Fe-Nd-B hard magnetic alloy system by a non-thermal process.

2. Experimental Procedure

A master ingot of Fe_{82.3}Nd_{11.8}B_{5.9} (at%) alloy (*i.e.*, stoichiometric Nd₂Fe₁₄B) was prepared by arc melting under highly purified Ar atmosphere using raw materials of 99.9 mass%Fe, 99.9 mass%Nd and Fe-18.9 mass%B mother alloy. Rapidly quenched ribbon was produced from the ingot by a single roller melt-spinning method at a roll surface velocity of 42 ms⁻¹ in an Ar atmosphere. The structure of the ribbon was examined by X-ray diffractometry (XRD) using Cu-K α radiation, transmission electron microscopy (TEM) using a JEM-3010 microscope operated at 300 kV and high resolution transmission electron microscopy (HREM) using a JEM-2010 microscope operated at 200 kV. The chemical composition for unirradiated and irradiated area in the specimen was examined by the energy-dispersive X-ray analysis (EDX) technique in TEM. Thin foils for TEM and HREM observation were ion-thinned by Ar⁺ whose accelerating voltage and angle of incidence were 5 kV and 11 degrees, respectively. Electron irradiation on the melt-spun ribbon was performed by an ultra-high voltage electron microscope (UHVEM) of H-3000 operated at 2 MV in Osaka University. The irradiation temperature was in the range of 104 and 298 K. The applied dose rate was chosen to be between 6.3×10^{24} and 1.0×10^{25} em⁻² s⁻¹. Change in the microstructure during electron irradiation was observed *in situ* by bright field images (BFIs) and selected area diffraction patterns (SADPs) in the UHVEM.

3. Results

3.1 Formation of Nd₂Fe₁₄B compound by rapid solidification

The microstructure of rapidly-solidified ribbon was investigated by XRD and conventional TEM using a JEM-3010 operated at 300 kV. Figure 1(a) shows the XRD pattern of a rapidly solidified sample; many sharp diffraction peaks can be seen. From the peaks, the constituent phase was determined to be tetragonal Nd₂Fe₁₄B compound with

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Fig. 1 X-ray diffraction pattern (a) and conventional TEM observation (b) of melt-spun $\text{Nd}_2\text{Fe}_{14}\text{B}$ ribbon.

lattice parameters of $a = 0.8805 \text{ nm}$ and $c = 1.2205 \text{ nm}$ (JCPDS:39-473). Since the $\text{Nd}_2\text{Fe}_{14}\text{B}$ compound shows many reflection peaks due to the extremely complex crystalline structure, main high intensity peaks are indexed in Fig. 1(a). No significant phases except $\text{Nd}_2\text{Fe}_{14}\text{B}$ were identified.

The BFI and SADP of melt-spun specimen (b) show an equiaxed grain structure of single phase. Dendrite and/or lamellae structure were not formed. The melt-spun specimen is composed almost entirely of $\text{Nd}_2\text{Fe}_{14}\text{B}$ compound. The average grain size of $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase was about 100 nm order and thus a nanocrystalline structure was not formed in the present work by rapid solidification.

3.2 Phase transformation behavior during electron irradiation in $\text{Nd}_2\text{Fe}_{14}\text{B}$ compound

Change in the microstructures of the $\text{Nd}_2\text{Fe}_{14}\text{B}$ compound during electron irradiation was examined *in situ* by UHVEM at 2.0 MV. Electron irradiation was carried out at a low temperature of 104 K to suppress the thermal recovery effect. Figure 2 shows changes in BFIs and corresponding SADPs of the melt-spun specimen during electron irradiation at 104 K with a dose rate of $6.3 \times 10^{24} \text{ em}^{-2} \text{ s}^{-1}$. Figures 2(a) and (d) show BFI and SADP of polycrystalline $\text{Nd}_2\text{Fe}_{14}\text{B}$ compound

Fig. 2 *In situ* TEM observation of changes in microstructures and selected area diffraction patterns of $\text{Nd}_2\text{Fe}_{14}\text{B}$ compound during electron irradiation at 104 K. (a), (d): as melt-spun; (b), (e): irradiated at $3.8 \times 10^{26} \text{ em}^{-2}$; (c), (f): irradiated at $1.7 \times 10^{28} \text{ em}^{-2}$.

before electron irradiation, respectively. After irradiation for 60 s, the BFI (b) shows a featureless contrast corresponding to an amorphous phase in the central region of electron irradiation. In the SAD pattern (e), crystalline spots which were observed in Fig. 2(d) were annihilated and halo rings indicated by arrows newly appeared. This suggests that electron irradiation induced amorphization of $\text{Nd}_2\text{Fe}_{14}\text{B}$ occurred. The $\text{Nd}_2\text{Fe}_{14}\text{B}$ compound cannot maintain its original crystalline structure under 2 MV electron irradiation and electron irradiation induced amorphization occurs. With further electron irradiation, the subsequent phase transformation of the amorphous phase obtained by solid-state amorphization is observed. In the specimen electron irradiated for $2.7 \times 10^3 \text{ s}$, an area of granular contrast smaller in size than that in melt-spun specimen appears as shown in Fig. 2(c). The mean grain size of crystalline precipitates was on the order of 10 nm. In the SADP (f), new Debye rings corresponding to nanocrystalline precipitates appear accompanied by halo rings. Although the $\text{Nd}_2\text{Fe}_{14}\text{B}$ crystalline structure is destroyed by electron knock-on effect and an amorphous phase is formed through electron irradiation induced amorphization, a crystalline phase is again formed through electron irradiation induced crystallization after solid-state amorphization. Such a unique phase transformation is called the crystal-to-amorphous-to-crystal (C-A-C)

Fig. 3 *In situ* TEM observation of changes in microstructures and selected area diffraction patterns of $\text{Nd}_2\text{Fe}_{14}\text{B}$ compound during electron irradiation at 298 K. (a), (d): as melt-spun; (b), (e): irradiated at $6.0 \times 10^{26} \text{ em}^{-2}$; (c), (f): irradiated at $2.7 \times 10^{28} \text{ em}^{-2}$.

transition. This is the first finding of C-A-C transition in Fe-based metallic materials. The nanocrystalline structure was formed through C-A-C transition in $\text{Nd}_2\text{Fe}_{14}\text{B}$ compound by electron irradiation at 104 K. No significant grain coarsening of the crystalline precipitates was observed during electron irradiation. This indicates that electron irradiation induced phase transformation is an effective method to obtain a nanocrystalline structure in a Fe-Nd-B hard magnetic alloy system.

Electron irradiation induced amorphization is known to be very sensitive to the irradiation temperature.^{13,16} The electron irradiation induced crystallization behavior is also influenced by the irradiation temperature. The difference in phase selection and kinetics of electron irradiation induced crystallization of melt-spun amorphous phase between 103 and 298 K was reported in Fe-Nd-B alloys⁹⁻¹² and Fe-Zr-B alloys.¹⁷⁻¹⁹ In order to investigate the temperature dependence of C-A-C transition in $\text{Nd}_2\text{Fe}_{14}\text{B}$ compound, electron irradiation was performed at 298 K; the obtained result is shown in Fig. 3. The typical polycrystalline contrast in BFI of melt-spun specimen (a) changes to a featureless contrast (b) after electron irradiation for 60 s. The electron diffraction spots corresponding to $\text{Nd}_2\text{Fe}_{14}\text{B}$ compound in SADP of melt-spun specimen (d) disappear with electron irradiation for 60 s as shown in Fig. 3(e); only broad halo rings can be

Fig. 4 Electron diffraction intensity profiles of nanocrystalline structure obtained by electron irradiation induced crystal-to-amorphous-to-crystal (C-A-C) transition of $\text{Nd}_2\text{Fe}_{14}\text{B}$ compound. (a) irradiated at 104 K with total dose of $1.7 \times 10^{28} \text{ em}^{-2}$, (b) irradiated at 298 K with total dose of $2.7 \times 10^{28} \text{ em}^{-2}$.

observed. After irradiation for $2.7 \times 10^3 \text{ s}$, the featureless contrast (b) in the BFI changes to a very fine granular contrast (c). In the SADP (f), Debye rings together with a weak halo ring are observed. The nanocrystalline structure was formed by electron irradiation induced crystallization after amorphization of $\text{Nd}_2\text{Fe}_{14}\text{B}$ compound at 298 K as well as at 104 K. No significant coarsening of grain size in the irradiated specimen was confirmed at 298 K. One should notice that the position and number of Debye rings obtained by electron irradiation induced C-A-C transition at 298 K are different from those at 104 K. The Debye rings indicated by the arrows in the SAD pattern shown in Fig. 3(f) cannot be seen in the pattern Fig. 2(f).

To examine the difference in constituent crystalline phases with nanocrystalline structure obtained by electron irradiation induced C-A-C transition, the electron diffraction intensity profiles were analyzed using a PICTRO STAT DIGITAL 400 image analyzer. Figure 4 shows the results of a nanocrystalline specimen obtained by electron irradiation at 104 K for $1.7 \times 10^{28} \text{ em}^{-2}$ (a) and 298 K for $2.7 \times 10^{28} \text{ em}^{-2}$ (b). Three sharp diffraction peaks marked by open squares can be observed in the spectrum (a). The positions of the three peaks correspond to the $\alpha\text{-Fe}$ crystalline phase. The $\alpha\text{-Fe}$ solid solution precipitates from an amorphous phase under electron irradiation at 104 K. The origin of the shoulder at the left side of the diffraction peak of $\alpha\text{-Fe}$ solid solution (110) is not clear and is of concern; it may be caused by the residual

Fig. 5 HREM images of the specimen electron irradiated at 298 K. (a) total dose of $6.0 \times 10^{26} \text{ em}^{-2}$ whose irradiation condition is corresponding to that just after amorphization, (b) total dose of $2.7 \times 10^{28} \text{ em}^{-2}$ whose condition is corresponding to that just after nanocrystallization.

amorphous phase and will be discussed in the later. The formation of α -Fe solid solution is also detected in the specimen electron irradiated at 298 K. The extra broad peaks at around $1/d = 3.2, 3.8, 6.2$ and 6.6 nm^{-1} corresponding the reflections indicated by arrows in Fig. 3(f) are seen in Fig. 4(b). Although the identification of crystalline phases is very difficult due to many candidates and few diffraction peaks, Fe₁₇Nd₂, Fe₇Nd and/or Nd₂Fe₁₄B crystalline phases with a large quantity of lattice defects may be formed under electron irradiation at 298 K. The constituent crystalline phases obtained by C-A-C transition are very sensitive to the irradiation temperature in Fe-Nd-B alloy, indicating that control of the nanocrystalline structure can be achieved by change in the irradiation temperature.

The amorphous phase and nanocrystalline phase were formed through electron irradiation induced phase transformation of Nd₂Fe₁₄B compound, which was examined by HREM. Figure 5 shows an HREM image of the specimen electron irradiated at 298 K with the total dose of $6.0 \times 10^{26} \text{ em}^{-2}$ (a) and $2.7 \times 10^{28} \text{ em}^{-2}$ (b), as an example of an amorphous specimen and nanocrystalline specimen, respectively. These irradiation conditions create the structures just after the primary amorphization and subsequent crystallization. In Fig. 5(a), a high magnification image with no atomic periodicity corresponding to the amorphous phase can be seen although fine crystalline fringes exists in some local

areas. Although the exact crystalline structure was not determined, the collapse of Nd₂Fe₁₄B crystals by electron induced amorphization was confirmed by HREM observation. The nanocrystalline specimen shows the cross-fringe image seen in Fig. 5(b). The nano-scale grains are randomly oriented. From the spacing of fringes and angle between fringes, formation of the nanocrystalline α -Fe precipitates was identified. In addition, the periodic fringe contrasts with spacing of 0.28 and 0.25 nm correspond to Fe₁₇Nd₂, Fe₇Nd and/or Nd₂Fe₁₄B. Not only α -Fe solid solution but also other crystalline phases may precipitate from an amorphous phase. The HREM image in Fig. 5(b) also shows a salt and pepper contrast corresponding to residual amorphous phase between each nanocrystalline precipitate. The nanocrystalline structure composed of nanocrystalline precipitates and residual amorphous phase is formed by electron irradiation induced C-A-C transition of Nd₂Fe₁₄B compound, while such nanocrystalline structure is rarely realized by the thermal process of rapid solidification from the melt.

4. Discussion

4.1 The origin of C-A-C transition in Nd₂Fe₁₄B compound

A unique phase transformation behavior of C-A-C transition in Nd₂Fe₁₄B compound was observed during electron irradiation. This phase transformation behavior during electron irradiation is discussed from the viewpoint of (1) change in chemical composition, (2) temperature increase, (3) oxidation and (4) mechanical atomic displacement by electron knock-on effect.

Energy dispersive spectroscopy (EDS) analysis was performed using an electron beam probe size of 50 nm to examine change in chemical composition during electron irradiation. The TEM-EDS spectra for unirradiated and irradiated areas at 298 K with a total dose of $2.7 \times 10^{28} \text{ em}^{-2}$ are shown in Fig. 6. The spectra intensity ratios of Nd to Fe for the irradiated and unirradiated areas were 5.5 and 5.7, respectively. No significant change in the spectra intensity ratio resulted from electron irradiation. This indicates that C-A-C transition during electron irradiation is not driven by change in chemical composition.

Kiritani *et al.*²⁰⁾ reported the expected distribution of the temperature rise in a specimen under electron irradiation. The temperature rise (ΔT) under electron irradiation is given by

$$\Delta T = \alpha \frac{Jr_0^2}{2K} \left\{ \frac{1}{2} \left(1 - \frac{r^2}{r_0^2} \right) + \ln \frac{R}{r_0} \right\} \quad \text{for } r < r_0$$

$$\Delta T = \alpha \frac{Jr_0^2}{2K} \ln \frac{R}{r} \quad \text{for } r > r_0$$

where R , r , r_0 , J , K and α are the specimen radius, the distance from the center of electron beam, the radius of the beam, current density, thermal conductivity of the specimen and a constant, respectively. The constant α was $0.4 \times 10^8 \text{ eV/cm}$ for iron at 2 MV electron irradiation.²⁰⁾ Figure 7 shows a calculated temperature distribution under electron irradiation in the present study. The calculated temperature rise was less than 10 K. The effect of temperature rise on phase transformation behavior is negligible under 2 MV electron irradiation because of high thermal conductivity. In

Fig. 6 TEM-EDS spectra for unirradiated (a) and irradiated areas (b) in the specimen electron irradiated at 298 K at total dose of $2.7 \times 10^{28} \text{ cm}^{-2}$.

Fig. 7 Calculated temperature distribution in $\text{Nd}_2\text{Fe}_{14}\text{B}$ alloy under electron irradiation.

the present study, electron irradiation was performed under a high vacuum of over 10^{-5} Pa by H-3000 UHVEM in Osaka University. The formation of iron oxide of Fe_3O_4 on Fe_4N under electron irradiation under similar high vacuum condition in TEM was reported.²¹⁾ However, it should be noticed that main constituent crystalline phase formed by the

Fig. 8 Schematic illustrations of crystal-to-amorphous-to-crystal (C-A-C) transition process under electron irradiation at 104 K (a) and 298 K (b).

electron irradiation induced phase transformation is not oxide but α -Fe phase in the present study. Oxidation is not a dominant factor for the C-A-C transition during electron irradiation. Previous systematic research work on electron irradiation induced amorphization of metallic materials concluded that electron irradiation features: no change in chemical composition, no temperature increase and no oxidation.^{13,16)} The above-described three factors do not affect the electron irradiation induced C-A-C transition in $\text{Nd}_2\text{Fe}_{14}\text{B}$ compound.

The mechanical atomic displacements in a lattice generally destroy the original structure of crystalline phases, resulting in amorphization of many of these phases.^{13,16)} If a crystalline structure were more stable than an amorphous structure under electron irradiation, the crystalline phase would precipitate from an amorphous phase. Once the crystalline structure with high phase stability against electron knock-on effect is formed, this phase does not change to an amorphous phase or other crystalline phases. The crystalline phase remains under electron irradiation.

Figure 8 shows a schematic illustration of the electron irradiation induced C-A-C transition observed in the present study. The phase transformation behavior is summarized as follows;

Under electron irradiation at 104 K and 298 K, solid-state amorphization of the $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase preferentially occurs as shown in Figs. 8(b) and (e), because the $\text{Nd}_2\text{Fe}_{14}\text{B}$

crystalline phase cannot retain its original crystalline structure under electron irradiation. With further electron irradiation, α -Fe solid solution precipitates from an amorphous phase obtained by electron irradiation at 104 K as shown in Fig. 8(c). At 298 K (f), not only α -Fe solid solution but also compound precipitate from the amorphous phase through electron irradiation induced crystallization. The C-A-C transition can be viewed as the combination of electron irradiation induced amorphization of Nd₂Fe₁₄B compound and subsequent electron irradiation induced crystallization of an amorphous phase. Therefore, the mechanical atomic displacement by electron knock-on effect may induce the C-A-C transition in Nd₂Fe₁₄B compound.

4.2 Nanocrystalline structure formation by electron irradiation

Crystal grain size in as melt-spun specimen was on the order of 100 nm, while that in the electron irradiated specimen was approximately 10 nm. Under electron irradiation, lattice defects are introduced in nanocrystalline grains by electron knock-on effect. The density of lattice defects in crystalline phase under electron irradiation is related to the degree of electron knock-on effect and thermal recovery effect. The thermal recovery effect is very sensitive to the annihilation site of lattice defects such as the interface between crystalline grain and residual amorphous phase. The greater the size of crystalline precipitates becomes, the more difficult is the annihilation of electron irradiation induced lattice defects at the interface. It is possible that only nanocrystalline precipitates maintain their original structure under electron irradiation because of the large number of the annihilation site of the lattice defects.

The phase selection in C-A-C transition of Nd₂Fe₁₄B compound at 104 K was different from that at 298 K. Only α -Fe solid solution was formed at 104 K, while at 298 K not only it but also other compounds precipitated from an amorphous phase. As described in chapter 4.1, only the crystalline phase which can maintain its original structure under electron irradiation precipitates from an amorphous phase through electron irradiation induced crystallization. A crystalline phase cannot retain its original structure when the creation of lattice defects occurs more frequently than recovery of atoms to their original positions by thermal diffusion. Since the degree of thermal recovery decreases with decreasing irradiation temperature, the stability of a thermally equilibrium phase also becomes weaker with decrease in irradiation temperature. The phase stability of crystalline phases against electron irradiation decreases with reduction in irradiation temperature. The origin of the difference in phase selection between 104 and 298 K is due to the temperature dependence of stability of the crystalline phase against electron irradiation. Therefore, the constituent crystalline phases in nanocrystalline structure can be controlled by the irradiation temperature in Nd₂Fe₁₄B compound. Electron irradiation induced phase transformation is an effective method to control nanocrystalline structure for the further development of nanocrystalline Fe-Nd-B hard magnetic material.

5. Conclusions

Nanocrystalline phase formation in rapidly solidified Nd₂Fe₁₄B compound during electron irradiation was investigated, the results were summarized and the following conclusions were reached.

- (1) Nd₂Fe₁₄B compound obtained by rapid solidification is not stable under electron irradiation. Solid-state amorphization of this phase occurred under 2 MV electron irradiation at 104 K and 298 K.
- (2) Electron irradiation induced crystallization of the amorphous phase occurred after electron irradiation induced amorphization of Nd₂Fe₁₄B compound. This is the first finding of crystal-to-amorphous-to-crystal (C-A-C) transition in Fe-based metallic materials.
- (3) Nanocrystalline structure was obtained by electron irradiation on Nd₂Fe₁₄B alloy. The constituent crystalline phase can be controlled by the irradiation temperature; α -Fe nanocrystalline solid solution dispersed amorphous phase was obtained by C-A-C transition at 104 K, while a nano-duplex structure composed of α -Fe, compounds and residual amorphous phase was formed at 298 K through C-A-C transition.

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