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Microstructure observation using MeV-electron-irradiation-induced amorphization

Takeshi Nagase^{a,b,*}, Akihiro Nino^c, Yukichi Umakoshi^d

^a Research Center for Ultra-High Voltage Electron Microscopy, Osaka University, 7-1, Mihogaoka, Ibaraki, Osaka 567-0047, Japan

^b Division of Materials and Manufacturing Science, Graduate School of Engineering, Osaka University, 2-1, Yamada-Oka, Suita, Osaka 565-0871, Japan

^c Department of Materials Science and Engineering, Faculty of Engineering and Resource Science, Akita University, 1-1, Tegatagakuen-machi, Akita-City 010-8502, Japan

^d National Institute for Materials Science, 1-2-1 Sengen, Tsukuba, Ibaraki 305-00471, Japan

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1. Introduction

Amorphous alloys and metallic glasses are produced by thermal processes such as liquid-to-glass (L-G) transition, which can be conducted by liquid quenching (LQ) [1,2]. These alloys and glasses can also be produced by solid-state amorphization (SSA) processes such as crystal-to-glass (C-G) transition, which can be conducted by mechanical processes at temperatures below the glass transition temperature (T_g) . Among the numerous mechanical processes for understanding the SSA mechanism, such as ion and neutron irradiation and severe plastic deformation, the observation of electron-irradiation-induced amorphization [3–5] by high-voltage electron microscopy (HVEM) is an important technique. HVEM is used for the simultaneous achievement of the stimulation of SSA and the in situ observation of the SSA process. Systematic theoretical and experimental studies have been conducted on electron-irradiation-induced SSA to understand the SSA mechanism [6,7]; it was clarified that the tendency of metallic materials to undergo SSA under electron irradiation is related to the position of these compounds in the temperature-composition (T-C) phase diagram [6]. The closer the intermetallic compound is

* Corresponding author at: Research Center for Ultra-High Voltage Electron Microscopy, Osaka University, 7-1, Mihogaoka, Ibaraki, Osaka 567-0047, Japan. Tel.: +81 668797941; fax: +81 668797942.

E-mail address: t-nagase@uhvem.osaka-u.ac.jp (T. Nagase).

ABSTRACT

MeV electron irradiation can stimulate solid-state amorphization in some intermetallic compounds. The irradiation induced amorphization phenomenon facilitates a clearer observation of the composite microstructure of the compounds. MeV electron irradiation is applied to a composite structure containing intermetallic compound "A," which undergoes solid-state amorphization and crystalline phase "B," which does not undergo amorphization. The composite structure transforms into a mixture of amorphous and crystalline phases by the irradiation. The distribution of A and B in the structure can hence be easily determined. High-voltage electron microscopy (HVEM) offers a unique microstructure observation technique that uses the difference between the sensitivities of compounds to undergo solid-state amorphization when MeV electron irradiation is applied to them.

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positioned to the liquidus valley in the T–C diagram, the stronger is its tendency to undergo SSA. Okamoto et al. have proposed the generalized Lindemann melting (GLM) criterion and have theoretically suggested that amorphization is a kinetically constrained melting process [7].

Electron-irradiation-induced SSA by HVEM is useful for the above-described research on the SSA mechanism and also for the evaluation of crystalline material microstructure [8]. In this paper, we report on the concept and demonstration of microstructure observation by using MeV-electron-irradiation-induced amorphization.

2. Concept of microstructure observation by using MeV-electron-irradiation-induced amorphization

A clear understanding of the difference between the amorphization tendencies of different crystalline phases provides a unique opportunity for identifying the mixture of the crystalline phases. Fig. 1 shows a schematic illustration of the microstructure evaluation by MeV-electron-irradiation-induced amorphization. Fig. 1(a) shows a composite structure containing intermetallic compound "A," which undergoes solid-state amorphization, and a crystalline phase "B", which does not undergo amorphization; phase B includes solid solutions and/or intermetallic compounds with a high phase stability against amorphization. In general, the identification of A and B in a conventional polycrystalline com-

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Fig. 1. Schematic illustration of microstructure obtained by MeV-electronirradiation-induced amorphization. The composite structure contains intermetallic compound "A," which undergoes solid-state amorphization and crystalline phase "B" which does not undergo amorphization. (a) TEM microstructure before MeV electron irradiation, (b) TEM microstructure after MeV electron irradiation, and (c) composite microstructure of "A" and "B." Each crystalline phase can be identified by the occurrence of MeV-electron-irradiation-induced amorphization.

posite is difficult if there is no significant difference between A and B with regard to size and morphology. After the MeV electron irradiation is applied to the structure, only A converts to an amorphous phase, whereas B maintains the crystalline structure. The microstructure shown in Fig. 1(b) indicates an amorphous matrix in which crystalline phases B are embedded. Fig. 1(c) shows the composite microstructure of A and B; the crystalline phases can be identified by comparing the microstructures before and after irradiation (Figs. 1(a) and (b), respectively). HVEM is a unique microstructure observation technique that uses the difference between the sensitivities of compounds to undergo solid-state amorphization when MeV electron irradiation is applied to them.

3. Experimental procedure

In the present study, the microstructure of a rapidly solidified Fe_{89.5}Nd_{10.5} alloy was evaluated through MeV-electron-irradiation-induced amorphization. A master ingot of Fe_{89.5}Nd_{10.5} alloy was prepared from pure Fe and Nd (more than 99% purity) in a highly purified Ar atmosphere using a conventional arc-melt technique. A 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 Ar atmosphere. The microstructure before irradiation was determined by X-ray diffraction (XRD) pattern analysis and transmission electron microscopy (TEM). Thin foils for TEM observation and electron irradiation by HVEM were prepared from the ribbon using an ion-milling technique. The foils were electron irradiated in Osaka University using the ultra-high voltage electron microscope (UHVEM; H-3000) operated at an acceleration voltage of 2.0 MV. This acceleration voltage is believed to be higher than the threshold acceleration voltage required for initiating an electron knock-on effect in Fe and Nd. Electron irradiation was carried out at 104 K. The temperature was maintained within ± 5 K of the desired value during electron irradiation. The applied dose rate was selected as 7.0×10^{24} m⁻² s⁻¹. Changes in the bright-field (BF) images and

Table 1

Intermetallic compounds reported to have undergone solid-state amorphization when MeV electron irradiation is applied to them. Most of the data were obtained by a research group at Osaka University using an ultra-high voltage electron microscope (HU-2000 and H-3000); electron irradiation was performed at an acceleration voltage of 2 MV. The temperature during the evaluations was kept at 298 K and below; the dose rate evaluated using a Faraday cup was of the order of $1 \times 10^{24} \, m^{-2} s^{-1}$ [6,10,11].

Yes No Group "A" Group "B" AlşCo AlşCu AlşCr AlşCo2 AlşCr AlşCo2 AlşCr AlşCr3 AlşFe Al2Cu AlşMn AlsFe2 Alıdy AlşFe2 Alıdy AlşMn AlşZr2 AlMn (Y2) AlzT2 AlMn (Y2) AlzT2 AlsNi AlzZr3 AlşNi2 AlzZr4 AlsNi3 BCo2 AlNi3 BCo2 Alx AlzZr3 AlşZr Cy Alz Bre3 Alg BY Alz AlzZr3 Alş Co2 Alx AlzZr3 Alş Cy Cu3 Cu3		
Croup "A" Croup "B" Alg Co2 Al2 Au Aly Cr Al3 Co2 Aly Cr Al3 Co2 Aly Cr Al3 Co2 Aly Cr Al3 Co2 Aly Cr Al3 Cr Aly Cr Al3 Cr Aly Cr Al3 Cr Aly Cr Al3 Cr Aly Cu Al3 Cu (r)2) Aly Mn Al5 Fe2 Al10 V Al2 Fe Alas V7 AlFe Alas V7 AlFe Als Zr Al, MIN Alz Zr Al, MIN BCo2 AlNi BCo3 Al, Ti Brea Al, V ByNia (o) Alg V ByNia (o) Alg V ByNia (co) Alg V ByNia (co) Alg V Cu3 Ti2 Bo2 Ni4 (m)	Yes	No "D"
AlgCo2Al2AuAlpCrAl5CrAlpCrAlpCr4Al4CrAlgCr5AlpFeAl2CuAlpMnAl5Fe2AlaMnAl5Fe2AlaNAl2FeAlaSV7AlFeAl23V4Al3MnAl2TrAl11Mn4Al3Zr2Alm(Y2)AlZrAlaNn(Y2)Al2TrAl12MoAl2TrAlaNn(Y2)Al2TsAlaNn(Y2)Al2TsAlaNn(Y2)Al2TsAlaNiAl2TsAlaNiAl2TsAlaNiAl2TsAlaNiBCo2AlNiBCo3Al3TiBFe3Al3VBNi2Al2T2Co3TiAl3ZrCr2TrAl2T3Cu11BNiCu12TpBNi4 (m)Cu12TpBNi2Cu11BNi4Cu12TpCu11BNi2Cu21Cu11BNi4Cu12TpCu31Fe2TaNbNi3Cu2rCu31Fe2TaNbNi3Cu2rCu31Fe2TaNbNi3Fe2TaNbNi3Fe2TaNbNi3Fe2TaNbNi3Fe2TaNbNi3Fe2TaNbNi3Fe2TaNbNi3Fe2TaNbNi3Fe2TaNbNi3Fe2TaNbNi3Fe2TaNbNi3Fe3Ald2B3Fe3Ald2B3Fe3Ald2B3Fe3Ald2B3Fe3Nd2B3Fe3Ald2B3Fe3Nd2B3Fe3Ald2B3 <th>Group "A"</th> <th>Group "B"</th>	Group "A"	Group "B"
Al ₇ CrAl ₉ Co2Al ₅ CrAl ₉ CrAl ₄ CrAl ₉ Cr ₅ Al ₃ FeAl ₂ CuAl ₈ MnAlCu(n ₂)Al ₄ MnAl ₅ Fe2Al ₁₀ VAl ₂ FeAl ₄ Sv7AlFeAl ₂ ZrAlmnAl ₂ ZrAlmnAl ₂ ZrAlmnAl ₂ ZrAlmn (γ2)AZrAl ₁₂ MoAl ₂ Zr2AlMn (γ2)AZrAl ₂ Mo3Al ₂ Zr3Al ₂ NiAl ₂ Zr3Al ₂ NiBCo3Al ₃ TiBFe3Al ₃ VB ₂ Ni4 (0)Al ₂ ZrBNi2Al ₂ ZrCr ₂ TrAlZrCr2TiAlZrCr2TiAlZrCr2TiAlZrCr2TiBNiCuti12BCoCuti13BFe2Cuti14BNi3CuZr2Cr2TiFe2TiFeTiFe2TiFeTiFe2TiFeTiFe2TiFeTiFe2TiFeTiFe2TiFeTiFeZr3Ni2TiMn2TiFeTiFeZr3Ni2TiNiZrNi3Ni2rNi2rNiZrNi2rNiZrNi2rNiZrNi2rNiZrNi2rNi2rNi2rNi2rPi42PFe3Ndc B3Fe4Nd1.1B4MoNiNiNi2rNi2rNi2rNi2rNi2rNi2rNi2rNi2rNi2rNi2r<	Al ₉ Co ₂	Al ₂ Au
AlsCrAlsCraAlsCrAlsCraAlsFeAl2CuAlsMnAl2Fe2AlaMnAlsFe2AlaVAl2FeAlaVAl2FeAlaVAl2FeAlaVAl2TeAlaVAl3MnAl2TrAl1Mn(Y2)AlZrAl1Mn(Y2)AlZrAl1Mn(Y2)AlZrAl3DrAl2TrAl3NiCo2Al3VBoosAl3VBNiCoCu3Ti2B2Ni4(m)Cu12rCr2TiCu12rCr2TiCu2rCr2TiFe1NAFeTiFeZr3Ni31Cu2rCr2TiFe2TiFeTiFeZr3Ni31Mu2TiFeNNi2rNi2rNi2rNi2rNi2rNi2rNi2rNi2rNi2rNi2rNi2rFe31Al2PNi2rFe31Al2PNi2r <td>Al₇Cr</td> <td>Al₅Co₂</td>	Al ₇ Cr	Al ₅ Co ₂
AlqCrAlgCr5AlgFeAl2CUAlgMnAlCU(m2)AlqMnAlsFe2AlqNnAl2FeAlasV7AlFeAlasV7AlFeAlasV4Al3MnAl2ZrAlm(m4AlzTAl1mm4AlzT2AlMn(y2)AlzT3Al3NiAlzT4Al3NiAlzZr3AlaNiAlzT4Al3NiAlzT4Al3NiAlzT4Al3NiAlzT5AlgMo3AlzT4Al3NiBC02AlNi3BC03Al3TiBFe3Al2VBNi4 (0)AlgY5BNi2Al12WCo2TiAl3ZrCr2TAl2T3Cu3Ti2BC0Cu4Ti3BFe2CuTiBNiCuT2Co7TiCu3Ti2BSNiCuT7CN1Cu72Cr2TiFe1Fe2Cu71Fe1Fe273Ni3TiMn2TiFe1Fe274NiNi3Ni2TNi3Ni3ZrNi3TiMn2TiFe4Nd1.1B4MONiNiZrNiZrNiZrNiZrNiZrNiZrNiZrNiZrNiZrFe3Nd2B3Fe4Nd2BFe81T9B0ZrAlNiKit	Al ₅ Cr	Al ₉ Cr ₄
Al ₃ FeAl ₂ CuAl ₈ MnAl ₂ CuAl ₄ MnAl ₅ Fe2Al ₁₀ VAl ₂ FeAl ₄ SV7AlFeAl ₃ V4Al ₃ MnAl ₃ Zr2Alm(\gamma_2)AlZrAl ₁₁ Mn ₄ Al ₃ Zr2Alm(\gamma_2)AlZrAl ₂ Zr3Al ₄ Zr3Al ₃ NiAl ₂ Zr3Al ₃ NiAl ₂ Zr4Al ₃ NiAl ₂ Zr3Al ₃ NiAl ₂ Zr3Al ₃ NiAl ₂ Zr4Al ₃ NiBCo2AlNiBCo3Al ₃ TiBFe3Al ₃ VBNi2Al ₂ ZrCo2TiAl ₂ ZrCo2TiAl ₂ ZrCr2TrAlZr3Cr2TrAl ₂ Zr3Cu3Ti ₂ BCoCu4Ti ₃ BFe2Cu4Ti3BFi3Cu4Ti3BNi4Cu7TiCo1TiCu2r2Cr2TiFe17NH2Cu7TiFe17NH2Cu7TiFe273Ni ₃ TiMoNiNiNb7Ni6Ni ₃ TiNi ₂ ZrNi ₃ TiNi ₂ ZrNi ₃ TiNi ₂ ZrNi ₂ ZrNiZrNi ₂ ZrNiZrNi ₂ ZrNiZrNi ₂ ZrNiZrNi ₂ ZrNiZrNi ₂ ZrNiZrNi ₂ ZrNiZrFe3Nd2B3Fe3Nd2B3Fe3Nd2B3Fe3Nd2B3Fe3Nd2B3Fe3Nd2B3Fe3Nd2B3Fe3Nd2B3Fe3Nd2B3Fe3Nd2B3Fe3Nd2B3Fe3Nd2B3Fe3Nd2B3 <td< td=""><td>Al₄Cr</td><td>Al₈Cr₅</td></td<>	Al ₄ Cr	Al ₈ Cr ₅
Al ₆ Mn AlCu(η_2) Al ₄ Mn Al ₅ Fe ₂ Al ₁₀ V Al ₅ Fe Al ₄₅ V ₇ AlFe Al ₂₃ V ₄ Al ₃ Mn Al ₂ Zr Al ₁₁ Mn ₄ Al ₂ Zr AlMn (γ_2) AlZr Al ₁₁ Mn ₄ Al ₂ Zr ₂ AlMn (γ_2) AlZr Al ₁₁ Mn ₄ Al ₂ Zr ₂ AlMn (γ_2) AlZr Al ₁₂ Mo Al ₂ Zr ₄ Al ₃ Ni Blo2 Al ₃ Ni BCo ₂ Al ₃ Ni Bre ₃ Al ₃ V ByNi ₄ (o) Al ₈ V ₅ BNi ₂ Al ₁₂ W Co ₂ Ti Al ₃ Zr Cr ₂ Zr Al ₂ Zr Cu ₄ Ti ₃ BFe ₂ CuTi BNi CuTi BNi CuTr CoTi CuZr CoTi CuZr Cu ₄ Ti	Al ₃ Fe	Al ₂ Cu
AlqMnAl ₅ Fe2Al ₁₀ VAl ₂ FeAl ₄₃ V7AlFeAl ₂₃ V4Al ₃ MnAl ₂ ZrAlm(\gamma_2)AlzrAl ₁₁ Mn ₄ Al ₂ Zr2AlMn (γ_2)AlZrAl ₁₂ MoAl ₄ Zr5Al ₈ Mo ₃ Al ₂ Zr4Al ₃ NiAl ₂ Zr3Al ₃ Ni2AlZr2AlNiBCo2AlNi3BCo3Al ₃ TiBFe3Al ₃ VByNi4 (o)Al ₈ V5BNi2Al ₁₂ WCo2TiAl ₂ TCr ₂ ZrAlZrCu3Ti2BCoCu4Ti3BFe2CuTiBNi3CuTi2B ₃ Ni4 (m)CuTi2BNi3Cu2rCoTiCu2r2Cr ₂ TiFe1Fe7Fe2Ti3Ni ₃ TiFe2Ti4Fe1Fe2Ti5Fe4Nd ₁₁ B4MoNiNi ₂ TiNi ₂ ZrNi ₂ TiNi ₂ ZrNi ₂ TiNi ₂ ZrNi ₂ TiFe2 ₃ Nd ₂ B ₃ Fe ₄ Nd ₂ BFe ₁₁ Nd ₂ BFe ₁₁ Nd ₂ BFe ₂₃ Nd ₂ B ₃ Fe ₁₄ Nd ₂ BFe ₁₁ Nd ₂ BFe ₁₁ Nd ₂ BFe ₂₃ Nd ₂ B ₃ Fe ₁₄ Nd ₂ BFe ₁₁ Nd ₂ B<	Al ₆ Mn	$AlCu(\eta_2)$
AlıoVAl₂FeAlısV7AlFeAlızV7AlFaAlızY4Al₃MnAl₂ZrAlınInn4Al₂Zr2Alım (γ2)AlZrAlın (γ2)AlZr3AlagMo3Al₄Zr5AlagMo3Al₄Zr5AlagMo3Al₂Zr3AlajNiAlzT2AlNiBCo2AlNi3BCo3Al₃TiBFe3Al₂VBNi4 (o)Al₂ZrCr2ZrAlZr3Cr2ZrAlZr3Cu3Ti2BCoCu4Ti3BFe2CuTiBNiCuTi2B3Ni4 (m)Cu10Zr7BNi3CuZr2Cr2TiCuZr2Cr2TiFe11Fe71Fe22NbNi3Fe273Ni3TiMoNiHi3Ni2Ti3Fe4Nd1.1B4MoNiNi3TiNi71Ni3ZrNi32Fe4Nd1.1B4MoNiNi3TiNi72Ni3TiNi72Ni3TiNi72Fe4Nd1.1B4MoNiNi2Ni2rNi2Ni2rZraNiNiNi2r<	Al ₄ Mn	Al ₅ Fe ₂
AlasAlFeAlasV4Ala MnAl2ZrAlm(1)m4,Al3Zr2Alm(1)Y2)AlZrAlarbaAl3Zr3AlamaAl2TsAlamaAlazr4AlaniAl2TaAlaNiAl2TaAlaNiAl2TaAlaNiAl2TaAlaNiAl2TaAlaNiAl2TaAlaNiAl2TaAlaNiBlacAlaNiBlacAlaNiBlacAlaNiBlacAlaVBreaAlaVBreaAlaVBriaAlaZrCr2TrAlaZraCu3Ti2BCoCu4Ti3BFe2CuTi1BNiCuTi2BaNi4 (m)CuTaBNi3CuZrCoTiCu2rCr2TiCu2rCr2TiFe2TaNbNi3CuZrCoTiCu2rCr2TiFe2TaNbNi3Fe2TaNbNi3PatiFe1iFe2TaNbNi3NigZrNi3TiNigZrNi3TiNi2rNi2rNi2rNi2rNi2rNi2rNi2rNi2rNi2rFe3Nd2B3Fe3Nd2B3Fe3Nd2B3Fe3Nd2B3Fe3Nd2B3Fe3Nd2B3Fe3Nd2B3Fe3Nd2B3Fe3Nd2B3Fe3Nd2B3Fe3Nd2B3Fe3Nd2B3Fe3Nd2B3Fe3Nd2B3Fe3Nd2B3Fe3Nd2B3Fe3Nd2B3Fe3Nd2B3Fe3Nd2B3	Al ₁₀ V	Al ₂ Fe
$Al_2 3 V_4$ $Al_3 Mn$ $Al_2 Zr$ $Al_1 1 Mn_4$ $Al_2 Zr_2$ $Al Mn (\gamma_2)$ $Al Zr$ $Al_1 2 Mo$ $Al_4 Zr_5$ $Al_8 Mo_3$ $Al_3 Zr_4$ $Al_3 Ni$ $Al_2 Zr_3$ $Al_3 Ni_2$ $Al Zr_2$ $Al Ni$ $B Co_2$ $Al Ni_3$ $B Co_3$ $Al_3 Ti$ $B Fe_3$ $Al_2 V$ $B_3 Ni_4 (o)$ $Al_8 V_5$ $B Ni_2$ $Al_1 2 W$ $Co_2 Ti$ $Al_3 Zr$ $Cr_2 Zr$ $Al_2 Ta$ $Cu_2 Ti$ $Al_2 Rr_3$ $Cu_3 Ti_2$ BCo $Cu_4 Ti_3$ BFe_2 $CuTi$ BNi $CuTi_2$ $B_3 Ni_4 (m)$ CuT_7 BNi_3 $CuZr_2$ $Cr_2 Ti$ CuT_7 BNi_3 $CuZr_4$ $Cr_2 Ti$ $Fe_2 Ti$ $FeTi$ $FeZr_3$ $Ni_3 Ti$ $Mn_2 Ti$ $Fe_4 Nd_{1,1} B_4$ $MoNi$ Nr_7 $Ni_2 Zr$ $Ni_2 Zr_2$	Al ₄₅ V ₇	AlFe
Al ₂ Zr Al ₁₁ Mn ₄ Al ₂ Zr ₂ AlMn (γ_2) AlZr Alm (γ_2) AlZr Alm (γ_2) AlZr Algono Al ₄ Zr ₅ AlgMo ₃ Al ₂ Zr ₃ Al ₃ Ni Al ₂ Zr ₃ Al ₃ Ni AlZr ₂ AlNi BCo ₂ AlNi ₃ BCo ₃ Al ₃ Ti BFe ₃ Al ₃ V BNi ₄ (o) AlgZr Go ₂ Al ₂ W Co ₂ Ti Al ₃ Zr Cr ₂ Zr Al ₂ Ta Cu ₃ Ti ₂ BCo Cu ₄ Ti ₃ BFe ₂ CuTi BNi CuTi BNi CuTr CoTi CuZr CoTi CuZr CoTi CuZr CoTi CuZr Cu ₄ Ti Fe ₂ Ti FeTi FeZr ₂ NbNi ₃ FeZr ₂ NbNi ₃ Ni ₃ Zr Ni ₃ Zr Ni ₃ Zr Ni ₃ Zr Ni ₃ Zr Ni ₃ Zr Ni ₂ Zr Fe ₂ Nd	Al ₂₃ V ₄	Al ₃ Mn
Al ₃ Zr ₂ AlMn (γ_2) AlZr Al ₁ 2Mo Al ₄ Zr ₅ Al ₈ Mo ₃ Al ₃ Zr ₄ Al ₃ Ni Al ₂ Zr ₃ Al ₃ Ni ₂ AlZr ₂ AlNi BCo ₂ AlNi BCo ₂ AlNi BCo ₃ Al ₃ Ti BFe ₃ Al ₃ V B ₃ Ni ₄ (o) Al ₈ V ₅ BNi ₂ Al ₁₂ W Co ₂ Ti Al ₂ Zr ₃ Cu ₃ Ti ₂ BCo Cu ₃ Ti ₂ BCo Cu ₄ Ti ₃ BFe ₂ CuTi BNi CuTi ₂ B ₃ Ni ₄ (m) Cu ₄ Ti ₃ BFe ₂ CuTi BNi CuTi ₂ Co Cu ₄ Ti ₃ Ge Cu ₂ Tr ₂ Cr ₂ Ti Fe ₁ Nd ₂ Cu ₄ Ti Fe ₂ Ti FeTi FeZ ₂ NbNi ₃ FeZ ₇ NbNi ₃ FeZ ₁ NbNi ₃ Fe ₂ Nd ₂ S Ni ₂ Tr Ni ₂ Ti Ni ₂ Zr Ni ₂ Ti <t< td=""><td>Al₂Zr</td><td>$Al_{11}Mn_4$</td></t<>	Al ₂ Zr	$Al_{11}Mn_4$
AlZr Al_2N_5 Al_2Zr_5 Al_8Mo_3 Al_2Zr_3 Al_3Ni Al_2Zr_3 Al_3Ni_2 $AlZr_2$ $AlNi$ BCo_2 $AlNi_3$ BCo_3 Al_3Ti BFe_3 Al_3V $B_3Ni_4(o)$ Al_8V_5 BNi_2 Al_12W Co_2Ti Al_3Zr Cr_2Zr $AlZr_3$ Cu_3Ti_2 BCo Cu_4Ti_3 BFe_2 $CuTi$ BNi CuT_2Cr $CoTi$ Cu_3Tr_2 $B_3Ni_4(m)$ $Cutr_2Cr_2$ Cr_2Ti $CuTar$ $CoTi$ $CuZr_2$ Cr_2Ti Fe_1Nd_2 Cu_4Ti $FeZr_3$ Ni_3Ti $MoNi$ $Nbri_3$ $FeZr_5$ Ni_3Ti $NiZr_2$ $NiZr_1$ $NiZr_1$ $NiZr_1$ $NiZr_1$ $NiZr_1$ $NiZr_1$ $NiZr_1$ $NiZr_1$ $Fe_1Ad_1.84$ $MoRi$ Fe_1RAg_2B	Al ₃ Zr ₂	AlMn (γ_2)
Al_4Zr_5 Al_8Mo_3 Al_3Zr_4 Al_5Ni Al_2T_3 Al_3Ni_2 $AlZr_2$ $AlNi$ BCo_2 $AlNi_3$ BCo_3 Al_3Ti BFe_3 Al_3V BNi_2 Al_2V Co_7Ti Al_2Zr_3 Co_7Ti Al_2Zr_3 Cu_3Ti_2 BCo Cu_4Ti_3 BFe_2 $CuTi$ BNi $CuTi_2$ $B_3Ni_4(m)$ $Cuti_7$ BNi_3 CuT_7 BNi_3 $CuZr_7$ $CoTi$ $CuZr_7$ Cu_4Ti Fe_7Ti $FeTi$ $FeZr_3$ Ni_3Ti $MoNi$ Ni_3 $NiTi$ Ni_3Zr Ni_3Zr Ni_3Ti $NiTi$ $NiTi_2$ $NiZr_2$ $PdZr_2$	AlZr	Al ₁₂ Mo
Al $_3Zr_4$ Al $_3Ni_2$ Al $_2Zr_3$ Al $_3Ni_2$ Al Zr_2 Al Ni BC $_0$ Al Ni_3 BC $_0$ Al Ni_3 BC $_3$ Al $_3Ti$ B Fe_3 Al $_3V$ B $_3Ni_4(o)$ Al $_8V_5$ B Ni_2 Al $_12W$ C $_2Ti$ Al $_2T$ C $_72Tr$ Al $_2T$ C $_13Ti_2$ BC $_0$ C $_14Ti_3$ BF e_2 CuTi BNi CuTi $_2$ B $_3Ni_4(m)$ Cu $_10Zr_7$ BNi $_3$ Cu Zr_2 C $_7Zri$ F e_17Nd_2 C $_2Ti$ F e_2Ti F eTi F eZr_3 Ni $_3Ti$ M $_2Ti$ F $e_4Nd_{1.1}B_4$ MoNi Ni Ni $_2r_2$ V Ni $_3r_7$ N	Al ₄ Zr ₅	Al ₈ Mo ₃
$Al_2 Zr_3$ $Al_3 Ni_2$ $AlZr_2$ $AlNi$ BCo_2 $AlNi_3$ BCo_3 $Al_3 Ti$ Bco_3 $Al_3 Ti$ Bre_3 $Al_3 V$ BNi_4 (o) $Al_8 V_5$ BNi_2 $Al_1 2 W$ $Co_2 Ti$ $Al_3 Zr$ $Cr_2 Zr$ $Al_2 Taabaaaaaaaaaaaaaaaaaaaaaaaaaaaaaaaaaa$	Al ₃ Zr ₄	Al ₃ Ni
AlZr2 AlNi BCo2 AlNi3 BCo3 Al3Ti BFe3 Al3V B3Ni4 (o) Al8V5 BNi2 Al12W Co2Ti Al3Zr Cr2Zr Al2T3 Cu3Ti2 BCo Cu4Ti3 BFe2 CuTi BNi2 Cu17Ti2 BNi3 CuT2r CoTi Cu2r1 CoTi Cu72r CoTi Cu72r CoTi Cu72r CoTi Cu72r CoTi Cu2r2 Cr2Ti Fe17Nd2 Cu4Ti Fe2r2 NbNi3 Fe2r3 Ni2Ti MoNi Fe4Nd1.1 B4 MoNi Ni2r2 NiZr NiZr NiZr NiZr NiZr Pi3Zr5 Fe3Zr5B10 Zr4Ni	Al ₂ Zr ₃	Al ₃ Ni ₂
BCo_2 $AlNi_3$ BCo_3 Al_3Ti Bre_3 Al_3V $B_3Ni_4(o)$ Al_8V_5 BNi_2 Al_12W Co_2Ti Al_2Tr Cr_2Zr $AlZr_3$ Cu_3Ti_2 BCo Cu_4Ti_3 BFe_2 $CuTi$ BNi $CuTi_2$ $B_3Ni_4(m)$ $CuTi_2$ $B_3Ni_4(m)$ $CuTi_2$ BNi_3 CuT_7 BNi_3 $CuZr_1$ $CoTi$ $CuZr_2$ Cr_2Ti Fe_7Nd_2 Cu_4Ti_3 $FeZr_3$ Ni_3Ti $FeZr_3$ Ni_3Ti Mn_2Ti $Fe_4Nd_{1.1}B_4$ $MoNi$ Ni_3Ti $NiZr_2$ $Varti_1 = Varti_1 = Varti_2 = Var$	AlZr ₂	AlNi
BCo3 Al3Ti BFe3 Al3V B3Ni4 (o) Al8V5 BNi2 Al12W Co2Ti Al3Zr Cr2Zr Al2T3 Cu3Ti2 BCo Cu4Ti3 BFe2 CuTi BNi CuTi2 B3Ni4 (m) Cu2r CoTi Cu2r CoTi CuZr Cotati Fe271 FeTi FeZr3 NbNi3 FeZr4 NbNi3 FeZr3 Ni3Ti Nb7Ni6 Ni3Ti Ni3Zr Ni3Zr Ni3Zr Ni2Zr PdZr2 PdZr2 PdZr3 Fe3L79B10 ZrAINi Ki	BCo ₂	AlNi ₃
BFe3 Al_3V $B_3Ni_4(o)$ Al_8V_5 BNi_2 Al_12W Co_2Ti Al_3Zr Cr_2Zr Al_3Zr Cu_3Ti_2 BCo Cu_4Ti_3 BFe_2 $CuTi$ BNi $CuTi_2$ $B_3Ni_4(m)$ $CuTo_2r_2$ Cr_2Ti $CuZr_2$ $CoTi$ $CuZr_2$ Cr_2Ti Fe_2Ti $FeTi$ $FeZr_2$ $NbNi_3$ $FeZr_3$ Ni_3Ti Mn_2Ti $Fe_4Nd_{1.1}B_4$ $NoNi$ Ni_3Ti $NiZr_2$ Pi_3Zr_5 Pi_3Zr_5 Fe_4Nd_2B $Fe_{3}Zr_9B_{10}$ Zr_4Ni $ZrAINi$ $Vi_2Pi_3Pi_5$	BCo ₃	Al ₃ Ti
$B_3Ni_4(0)$ Al_8V_5 BNi_2 Al_12W Co_2Ti Al_3Zr Cr_2Zr $AlZr_3$ Cu_3Ti_2 BCo Cu_4Ti_3 BFe_2 $CuTi$ BNi $CuTi_2$ $B_3Ni_4(m)$ Cu_10Zr_7 BNi_3 $CuZr_2$ Cr_2Ti CuT_2 Cr_2Ti Fe_17Nd_2 Cu_4Ti Fe_2Ti $FeTi$ $FeZr_3$ Ni_3Ti Mn_2Ti $Fe_4Nd_{1.1}B_4$ MoNi Nitri $NiTri_2$ $NiZr_1$ $NiZr_1$ $Fe_4Nd_{1.1}B_4$ $NiZr_2$ Fe_4Nd_2B $Fe_{3}Zr_5$ $Fe_{4}Nd_2B$ $Fe_{3}Zr_9B_{10}$ Zr_4Ni	BFe ₃	Al ₃ V
BNi2 $Al_{12}W$ Co_2Ti Al_3Zr Co_2Ti Al_3Zr Cr_2Zr $AlZr_3$ Cu_3Ti_2 BCo Cu_4Ti_3 BFe_2 $CuTi$ BNi $CuTi_2$ $B_3Ni_4(m)$ $CuTo_2Tr_2$ BNi_3 $CuZr_2$ $CoTi$ $CuZr_2$ Cr_2Ti Fe_1Nd_2 Cu_4Ti Fe_2Ti $FeTi$ $FeZr_2$ $NbNi_3$ $FeZr_3$ Ni_3Ti $MoNi$ Ni_2 Nb_7Ni_6 $NiTi$ $NiTi_2$ $NiZr_2$ $PdZr_2$ Pt_3Zr_5 $Fe_{31}Alg_8$ $Fe_{4Nd_2B_1$ $Fe_{41}Nd_2B_1$ $Fe_{41}Nd_2B_1$ $Fe_{41}Nd_2B_1$ $Fe_{41}Nd_2B_1$ $Fe_{41}Nd_2B_1$ $Fe_{41}Nd_2B_1$ $Fe_{31}Zr_9B_{10}$ $ZrAlNi$	$B_3Ni_4(o)$	Al ₈ V ₅
Co ₂ Ti Al ₃ Zr Cr ₂ Zr AlZr ₃ Cu ₅ Ti ₂ BCo Cu ₄ Ti ₃ BFe ₂ CuTi BNi CuTi ₂ B ₃ Ni ₄ (m) CuT ₁₀ B ₃ Ni ₄ (m) Cu ₁₀ Zr ₇ BNi ₃ CuZr CoTi CuZr ₂ Cr ₂ Ti Fe ₁₇ Nd ₂ Cu ₄ Ti Fe ₂₇ Nd ₂ Cu ₄ Ti FeZr ₂ NbNi ₃ FeZr ₃ Ni ₃ Ti Mn ₂ Ti Fe ₄ Nd _{1,1} B ₄ MoNi NiTi NiTi NiZr NiZr NiZr NiZr Fe ₃ Zr ₅ Fe ₄ Ad ₂ B Fe ₈ Zr ₃ B ₁₀ Fe ₈ Zr ₃ B ₁₀ ZrAINi	BNi ₂	Al ₁₂ W
$\begin{array}{llllllllllllllllllllllllllllllllllll$	Co ₂ Ti	Al ₃ Zr
$\begin{array}{llllllllllllllllllllllllllllllllllll$	Cr ₂ Zr	AlZr ₃
$\begin{array}{llllllllllllllllllllllllllllllllllll$	Cu ₃ Ti ₂	BCo
CuTi BNi CuTi2 $B_3Ni_4(m)$ Cu10Zr7 BNi3 CuZr CoTi CuZr2 Cr2Ti Fe17Nd2 Cu4Ti Fe2Ti FeTi FeZr3 NbNi3 FeZr3 Ni3Ti Mn2Ti Fe4Nd1.1B4 MoNi NiTi NiTi2 Ni3Zr NiZr NiZr NiZr5 Fe32Nd2B3 Fe4Nd2B3 Fe4Nd2B3 Fe4Nd2B3 Fe4Nd2B4	Cu ₄ Ti ₃	BFe ₂
$\begin{array}{llllllllllllllllllllllllllllllllllll$	CuTi	BNi
$\begin{array}{llllllllllllllllllllllllllllllllllll$	CuTi ₂	$B_3Ni_4(m)$
$\begin{array}{llllllllllllllllllllllllllllllllllll$	Cu ₁₀ Zr ₇	BNi ₃
$\begin{array}{llllllllllllllllllllllllllllllllllll$	CuZr	СоТі
$\begin{array}{ll} Fe_{17}Nd_2 & Cu_4Ti \\ Fe_2Ti & FeTi \\ FeZT_2 & NbNi_3 \\ FeZr_3 & Ni_3Ti \\ Mn_2Ti & Fe_4Nd_{1.1}B_4 \\ MoNi & & & \\ Nb_7Ni_6 & & & \\ NiTi & & & \\ NiTi & & & \\ NiTi & & & \\ NiTi_2 & & & & \\ NiZr & & & & \\ NiZr & & & & \\ NiZr_2 & & & & \\ PdZr_2 & & & & \\ PdZr_2 & & & & \\ Fe_{31}Arg_B & & & & \\ Fe_{31}Arg_B & & & \\ Fe_{31}Zr_9B_{10} & & & \\ ZrAlNi & & & \\ \end{array}$	CuZr ₂	Cr ₂ Ti
$\begin{array}{llllllllllllllllllllllllllllllllllll$	Fe ₁₇ Nd ₂	Cu ₄ Ti
$\begin{array}{llllllllllllllllllllllllllllllllllll$	Fe ₂ Ti	FeTi
$\begin{array}{llllllllllllllllllllllllllllllllllll$	FeZr ₂	NbNi ₃
$\begin{array}{llllllllllllllllllllllllllllllllllll$	FeZr ₃	Ni ₃ Ti
$\begin{array}{l} MoNi \\ Nb_7Ni_6 \\ NiTi \\ NiTi \\ NiTi_2 \\ Ni_3Zr \\ NiZr \\ NiZr \\ PdZr_2 \\ PdZr_2 \\ PdZr_2 \\ PdZr_5 \\ Fe_{32}Nd_2B_3 \\ Fe_{14}Nd_2B \\ Fe_{81}Zr_{0}B_{10} \\ ZrAlNi \end{array}$	Mn ₂ Ti	$Fe_4Nd_{1,1}B_4$
$\begin{array}{l} Nb_7 Ni_6 \\ NiTi \\ NiTi_2 \\ Ni_3 Zr \\ NiZr \\ NiZr \\ PdZr_2 \\ PdZr_2 \\ Pd_3 Zr_5 \\ Fe_{23} Nd_2 B_3 \\ Fe_{14} Nd_2 B \\ Fe_{81} Zr_0 B_{10} \\ ZrAlNi \end{array}$	MoNi	
NiTi NiTi ₂ Ni ₃ Zr NiZr NiZr PdZr ₂ PdZr ₂ Pt ₃ Zr ₅ Fe ₂₃ Nd ₂ B ₃ Fe ₁₄ Nd ₂ B Fe ₃₁ Zr ₉ B ₁₀ ZrAlNi	Nb ₇ Ni ₆	
$\begin{array}{l} NiTi_2 \\ Ni_3Zr \\ NiZr \\ NiZr_ \\ PdZr_2 \\ PdZr_2 \\ Pt_3Zr_5 \\ Fe_{23}Nd_2B_3 \\ Fe_{14}Nd_2B \\ Fe_{81}Zr_9B_{10} \\ ZrAlNi \end{array}$	NiTi	
Ni ₃ Zr NiZr NiZr ₂ PdZr ₂ Pt ₃ Zr ₅ Fe ₂₃ Nd ₂ B ₃ Fe ₁₄ Nd ₂ B Fe ₈₁ Zr ₅ B ₁₀ ZrAlNi	NiTi ₂	
NiZr NiZr ₂ $PdZr_2$ Pt_3Zr_5 Fe_3Zr_5 $Fe_{14}Nd_2B$ $Fe_{81}Zr_5B_{10}$ ZrAlNi	Ni ₃ Zr	
NiZr ₂ PdZr ₂ Pt ₃ Zr ₅ Fe ₂₃ Nd ₂ B ₃ Fe ₁₄ Nd ₂ B Fe ₈₁ Zr ₉ B ₁₀ ZrAlNi	NiZr	
PdZr ₂ Pt ₃ Zr ₅ Fe ₂₃ Nd ₂ B ₃ Fe ₁₄ Nd ₂ B Fe ₈₁ Zr ₉ B ₁₀ ZrAlNi	NiZr ₂	
Pt ₃ Zr ₅ Fe ₂₃ Nd ₂ B ₃ Fe ₁₄ Nd ₂ B Fe ₈₁ Zr ₉ B ₁₀ ZrAlNi	PdZr ₂	
$\begin{array}{l} Fe_{23}Nd_2B_3\\ Fe_{14}Nd_2B\\ Fe_{81}Zr_9B_{10}\\ ZrAlNi \end{array}$	Pt ₃ Zr ₅	
$\begin{array}{l} Fe_{14}Nd_2B \\ Fe_{81}Zr_9B_{10} \\ ZrAlNi \end{array}$	Fe ₂₃ Nd ₂ B ₃	
Fe ₈₁ Zr ₉ B ₁₀ ZrAlNi	Fe ₁₄ Nd ₂ B	
ZrAINi	$Fe_{81}Zr_9B_{10}$	
	ZrAlNi	

selected area diffraction (SAD) patterns during electron irradiation were observed in situ using a UHVEM at 2.0 MV. The effect of additional electron irradiation during in situ TEM observations was negligible because of the low dose rate.

4. Results and discussion

Fig. 2 shows the typical example of the microstructure observation using MeV electron irradiation induced amorphization in a rapidly solidified melt-spun $Fe_{89.5}Nd_{10.5}$ alloy [8,9]. Fig. 2(a) shows the XRD pattern of the $Fe_{89.5}Nd_{10.5}$ alloy; the constituent phases can be identified as a mixture of α -Fe and $Fe_{17}Nd_2$ intermetallic compound. Table 1 lists the intermetallic compounds that have been reported to undergo SSA when MeV electron irradiation is applied to them [6,10,11]. Most of the data were obtained by a



Fig. 2. The observed microstructure of a rapidly solidified melt-spun Fe_{89.5}Nd_{10.5} alloy after it undergoes MeV-electron-irradiation-induced amorphization; this alloy is prepared by a single roller melt-spinning method. (a) XRD pattern of Fe_{89.5}Nd_{10.5} alloy. The constituent phases are identified as a mixture of α -Fe and Fe₁₇Nd₂ intermetallic compound. (b) Bright-field (BF) image of specimen before irradiation. (c) BF image after electron irradiation for 600 s and a total dose of 4.2×10^{26} m⁻². (d) Corresponding selected area diffraction (SAD) pattern before the irradiation. (e) SAD pattern after the irradiation. Electron irradiation was performed using an ultra-high voltage electron microscope (UHVEM; H-3000) at 104 K. The acceleration voltage is 2.0 MV, and the dose rate is 7.0×10^{24} m⁻² s⁻¹. Only Fe₁₇Nd₂undergoes solid-state amorphization, while α -Fe is stable against irradiation. The distribution of α -Fe phase and Fe₁₇Nd₂ can be clearly determined by the irradiation [10,11].

research group at Osaka University using a UHVEM (HU-2000 and H-3000); electron irradiation was applied at an acceleration voltage of 2.0 MV, and the temperature was maintained at 298 K or less. The dose rate, evaluated using a Faraday cup, was of the order of $1 \times 10^{24} \text{ m}^{-2} \text{ s}^{-1}$. Table 1 indicates that Fe₁₇Nd₂ belongs to group "A" which undergoes solid-state amorphization induced by MeV electron irradiation. On the other hand, the α -Fe phase belongs to group "B" as the solid-state amorphization of α -Fe solid solution was not reported. Fig. 2(b) shows the BF image of a composite structure of α -Fe and Fe₁₇Nd₂ intermetallic compound in meltspun Fe_{89.5}Nd_{10.5} alloy before it is irradiated. The black arrow in Fig. 2(b) and (c) is a marker. A polycrystalline structure with a crystal grain size of the order of 100 nm can be observed in the figure. The identification of each crystalline grain is impossible only in Fig. 2(b). The SAD pattern in Fig. 2(d) shows the Debye rings corresponding to α -Fe and Fe₁₇Nd₂. The BF image of the composite structure before irradiation (Fig. 2(b)) is in agreement with the schematic illustration shown in Fig. 1(a). The BF image and SAD pattern are drastically changed after the MeV electron irradiation. The BF image irradiated for 600 s (Fig. 2(c)) shows crystalline grains and an amorphous matrix. This BF image is in agreement with the schematic illustration shown in Fig. 1(b). The SAD pattern (Fig. 2(e)) shows Debye rings corresponding to α -Fe and newly appeared halo rings, while it does not show Debye rings corresponding to Fe₁₇Nd₂. In Fig. 2(c), the crystalline grains that remain after the irradiation are identified to be those of α -Fe solid solution, while the crystalline grains that convert to an amorphous phase are identified to be those of Fe₁₇Nd₂ intermetallic compound. The distribution of the α -Fe phase and Fe₁₇Nd₂ can hence be clearly evaluated after the irradiation has been performed [10,11].

The tendency of metallic materials to undergo SSA when electron irradiation is applied to them is related to the position of these materials in the temperature-composition (T-C) phase diagram [6]. The intermetallic compounds that lie close to the liquidus valley in the T-C diagram show a strong tendency to undergo SSA, while those intermetallic compounds that are far from the liquidus valley do not. The effect of the position of an intermetallic compound in the phase diagram on its glass-forming ability (GFA) during electron-irradiation-induced SSA is greater than the effects of the structure [6,12,13], transition temperature [6], and solubility [6,14,15] of the compound. In other words, intermetallic compounds whose positions coincide with a deep eutectic exhibit high GFA through irradiation-induced SSA. Fig. 3(a) shows the relationship between the occurrence of MeV electron-irradiationinduced amorphization in an intermetallic compound and the LQ induced amorphous phase formation in an alloy whose composition is the same as that of an irradiated intermetallic compound [16]; the information regarding the amorphous phase formation is obtained from previously reported experimental data. It can be observed that the intermetallic compounds whose composition is in the LQ induced amorphous phase formation range exhibit a strong tendency to undergo SSA. Fig. 3(b) shows a Fe-Nd binary phase diagram with the composition range for the LQ induced amorphous phase formation. The only Fe-Nd binary intermetallic compound that can be observed in the thermal equilibrium phase diagram is the Fe₁₇Nd₂. An amorphous phase formation was reported in a wide composition range in the binary Fe-Nd alloy system [16]. Fe₁₇Nd₂ lies in the composition range for LQ induced amorphous phase formation, indicating that Fe₁₇Nd₂ lies in the metastable deep eutectic in Fe and Nd solid solutions. The occurrence of irradiation induced SSA in Fe₁₇Nd₂ intermetallic compound can be explained by their position in the phase diagram.

As shown in Table 1, various intermetallic compounds such as metal–metal type binary Al_9Co_2 , metal–metalloid type binary Co_2B , and ternary $Fe_{14}Nd_2B$ were observed to undergo SSA. Thus





Fig. 3. Relationship between the occurrence of MeV electron-irradiation-induced amorphization in an intermetallic compound and the liquid quenching (LQ) induced

amorphous phase formation in an alloys whose composition is the same as that of an irradiated intermetallic compound (a), and the Fe–Nd binary phase diagram with the composition range for the LQ induced amorphous phase formation (b).

far, 50 of 84 intermetallic compounds have been found to undergo SSA [6,10,11]; this phenomenon is commonly observed in metallic materials. This indicates that crystalline composite materials containing the intermetallic compounds belonging to the group "A" in Table 1 can be analyzed using the newly proposed technique. On the basis of the experimental database shown in Table 1, MeV electron irradiation technique by HVEM offers a unique opportunity to evaluate the microstructure of crystalline composites.

5. Conclusions

In the present study, we proposed the HVEM technique for the microstructure evaluation of polycrystalline materials and particularly for the identification of each crystalline grain. HVEM is a unique microstructure observation technique that uses the difference between the sensitivities of compounds to undergo solid-state amorphization when MeV electron irradiation is applied to them.

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