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MS-12-P-2274 Microstructural Characterization by TEM/STEM and XAFS for $Fe_{85.2}Si_1B_9P_4Cu_{0.8}$ nanocrystalline soft magnetic alloy

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The investigation of soft magnetic alloys becomes activated increasingly because of their wide applications, such as transformer, sensors and electric devices. Aiming at the development of the soft magnetic material to achieve lower coecivity (*Hc*) and higher saturation magnetic flux density (*Bs*), new $Fe_{85\text{-}86}Si_{1\text{-}2}B_8P_4Cu_1$ nanocrystalline soft magnetic alloy (NANOMET) has been developed by Makino *et al*.[1] which exhibits low *Hc* of about 5.8 A/m and high *Bs* of about 1.8 T. According to their studies[1], the magnetic properties are highly related to their heterogeneous structure of the as-quenched state and the nanocrystallization induced by the addition of Cu, P and Si. Therefore, it is quite important to understand the nanocrystallization kinetics of NANOMET. In this study, we focus on the structural change during nanocrystallization by means of transmission electron microscope (TEM) and X-ray absorption fine structure (XAFS). Ribbon samples of $Fe_{85.2}Si_1B_9P_4Cu_{0.8}$ alloy were prepared by single roller melt-spinning in air atmosphere. As-quenched ribbons were annealed at several temperatures in an infrared furnace. TEM specimens were prepared by Ar ion milling method. TEM characterizations were performed using an atomic resolution analytical electron microscope (JEM-ARM 200F). XAFS measurements of the Fe and Cu K-edge were performed using synchrotron radiation at BL14B2 in SPring-8. Fig. 1 show STEM-HAADF image and their SAED patterns (inset) of $Fe_{85.2}Si_1B_9P_4Cu_{0.8}$ alloy annealed at (a) 638K for 0s and (b) 693K for 600s, respectively. Due to the higher Fe (85~86 at.%) content, a primary crystalline phase is α-Fe and a few α-Fe nanocrystallites arise in a very limited local area at 638K for 0s even below the onset of the 1st crystallization. The homogeneous structure consisting of fine α-Fe grains with the size of about 12 nm was formed at 693K for 600s. Fig. 2 shows (a) high resolution STEM-HAADF image and (b) STEM-XEDS mapping of an α-Fe nanocrystal precipitated in an $Fe_{85.2}Si_1B_9P_4Cu_{0.8}$ alloy annealed at 638K for 0s. Fe K-, P L- and Cu K-line are superimposed in Fig. 2 (b), some of fine Cu-rich clusters (1.5 nm or less) are observed inside and/or neighboring the precipitated α-Fe and P is rejected and enriched around the precipitated α-Fe, suggesting that P plays the similar roles to Nb in FINEMET[2]. From our results, heterogeneous precipitation of α-Fe crystallites occurs nearby Cu rich clusters and close relationships between the local structural change around Cu and progress of α-Fe precipitation was confirmed by TEM and XAFS.

[1] A. Makino, IEEE Trans. Magn., vol. 48, pp. 1331-1335, Apr. 2012.

[2] K. Hono, D. H. Ping, M. Ohnuma, and H. Onodera, Acta mater., vol. 47, pp. 997-1006, Mar. 1999.

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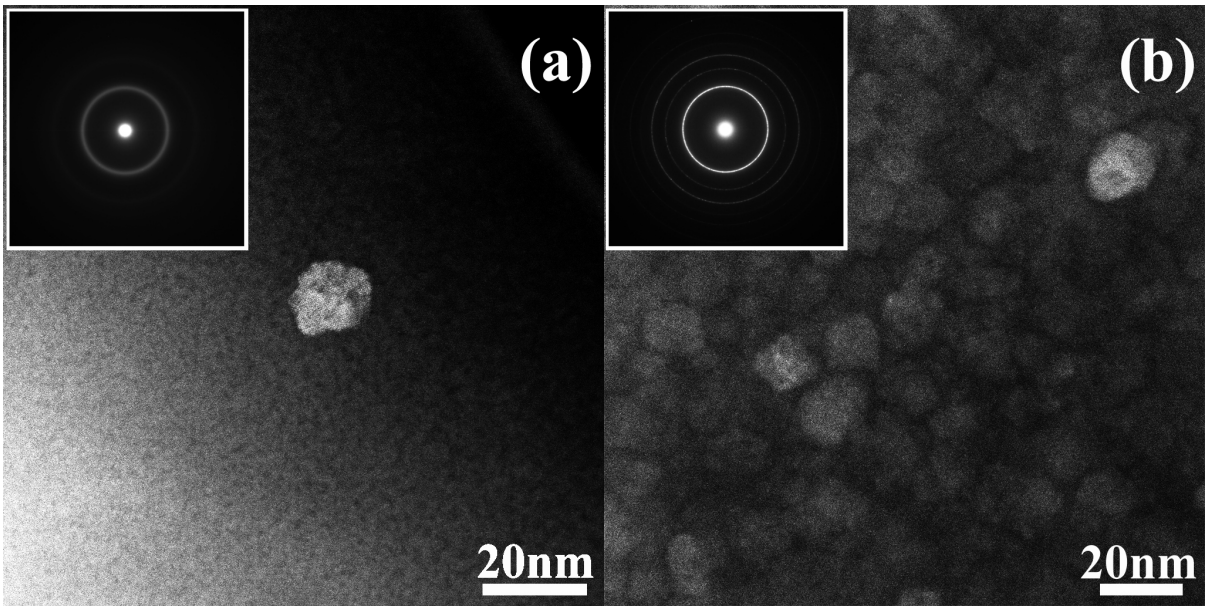

Fig. 1: Fig. 1 STEM-HAADF image and SAED patterns (inset) of $Fe_{85.2}Si_1B_9P_4Cu_{0.8}$ alloy annealed (a)at 638K for 0s and (b) at 693K for 600s.Brighter contrast grains are α-Fe nanocrystallites and dark contrast is residual amorphous matrix.

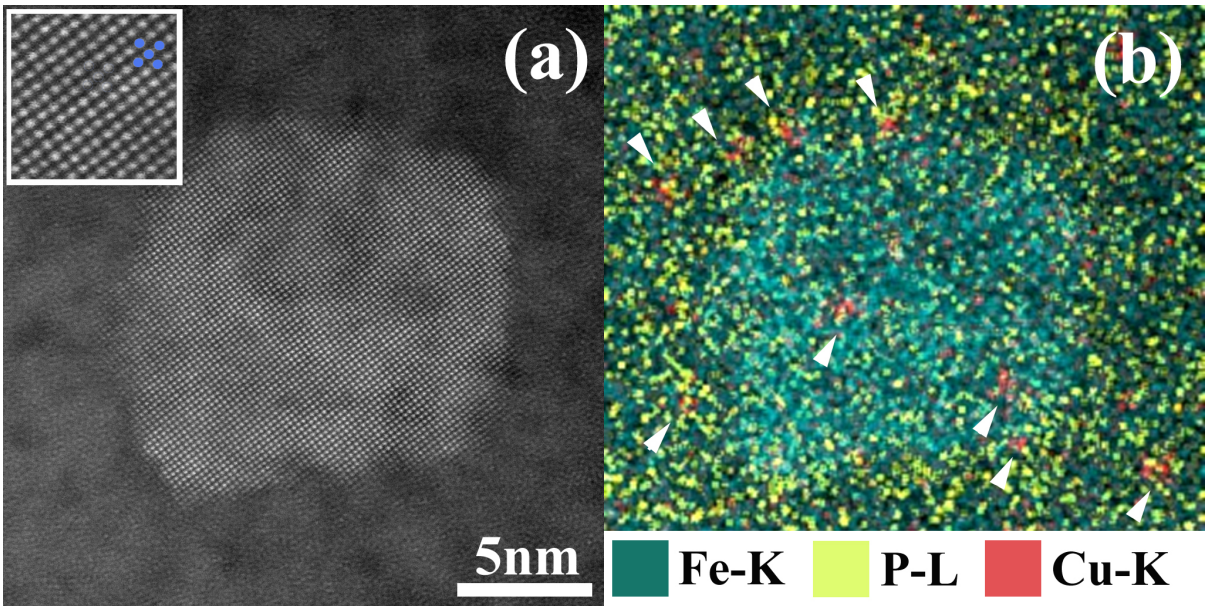

Fig. 2: Fig. 2 High resolution STEM-HAADF image (a) and STEM-XEDS mapping of precipitated α-Fe nanocrystal in $Fe_{85.2}Si_1B_9P_4Cu_{0.8}$ alloy annealed at 638K for 0s. Blue dots in Fig 2(a) indicate the bcc-unit cell of α-Fe (upper left) and white arrow head indicate Cu rich clusters in Fig. 2 (b).