

Anomalous small angle X-ray scattering of melt-spun and aged Fe-Nb-B alloys using BL15XU

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An amorphous Fe-Nb-B alloy is a promising Fe-based fundamental system for practical uses. Because of their high magnetic permeability under high-frequency magnetic field, they are, for example, used as an induction coil of portable telephone. It is known that their soft magnetic properties are largely improved by precipitating nano-scale iron particles in the amorphous matrix by appropriate heat-treatment. The mechanism of the nano-scale precipitation behavior, however, has not been well understood yet. In this work, small angle X-ray scattering (SAXS) has been measured for characterizing the nano-scale precipitates formed in melt-spun Fe₈₅Nb₇B₉ alloy.

Amorphous Fe₈₅Nb₇B₉ ribbons were produced by melt-spun, and a part of the ribbons were subsequently aged at 843 K to precipitate nano-iron particles. This condition was determined based on the result of calorimetric study.

SAXS measurements were performed using BL15XU: WEBRAM (Wide Energy range Beam-line for Research in Advanced Materials). Synchrotron radiations of 20 and 100 eV below Nb K edge was selected by a double Si crystal monochromator. A highly precise goniometer for powder diffraction with vacuum paths was used for the SAXS measurements.

Figures 1 (a) and (b) show SAXS intensity profiles for melt-spun and aged Fe₈₅Nb₇B₉ alloys. Scattering intensity of the aged alloy shows higher intensity in the range between 0.1 and 10 nm than that of the as-spun sample. This indicates that nano-scale

precipitates are formed by aging. In addition, intensity difference between SAXS profiles just below Nb K absorption edge suggests some rearrangements of niobium atoms during the precipitation. Further investigation is currently done.

Consequently, the present results show that the anomalous SAXS measurement using BL15XU is a promising measurement tool to reveal a role of a certain constituent element for nano-precipitation behavior in advanced amorphous materials.

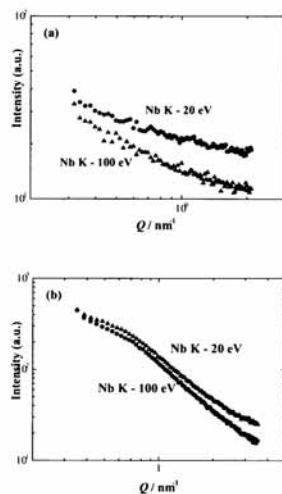


Fig.1 Small angle X-ray scattering intensity profiles for (a) a melt-spun Fe₈₅Nb₇B₉ alloy and (b) a aged Fe₈₅Nb₇B₉ alloys.

Electronic structures of hematite-ilmenite solid solution films by selective x-ray emission or absorption spectroscopy

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Though both α -Fe₂O₃ and FeTiO₃ are antiferromagnetic insulators, the solid solutions between them are known to be unique ferrimagnetic semiconductors. Recent theoretical calculations suggested the system possessed spin-polarized conduction fairly above room temperature. The electronic structures of α -Fe₂O₃-FeTiO₃ are generally considered as the mixed valence states between Fe²⁺ and Fe³⁺ and the nominal valence states of Ti are fixed to 4+. However our previous high-resolution X-ray fluorescence spectroscopy study of various titanium oxides indicated that the Ti K α peak profiles of FeTiO₃ were slightly different from those of other Ti⁴⁺ compounds and were better to assign to have some Ti³⁺ characters. In the present study, in order to clarify the real electronic structures of Ti ions in FeTiO₃, we surveyed the precise peak profiles of Ti K β lines for FeTiO₃ as a function of the incident X-ray energy.

The intense Ti K β _{1,3} fluorescence lines for Ti oxides are usually accompanied by weak lines appearing at around 4945 and 4960 eV. These weak lines are nominally assigned to K β _{2,5} and are explained as the transition from 3d-derived valence band hybridized orbitals to an 1s core-hole. Therefore, peak profiles of K β _{2,5} lines should be very sensitive to the change of valence states of Ti ions. If an additional electron hole was created in the 3s or 3p orbital besides the 1s core-hole, the population of 3d electrons could be decreased due to the 3s(3p)-3d3d super Coster-Kronig transition. And the decreased 3d electrons can influence the K β _{2,5} peak profiles.

Based on the above assumptions, we measured high-resolution Ti K β fluorescence spectra of FeTiO₃ bulk crystals using a Johann type monochromator at

BL15XU in Spring-8. The incident X-ray energies for two-electrons excitation considering super Coster-Kronig transitions for Ti and Fe were selected as follows: Ti K+M_{4,5} (4991 eV), Ti K+M_{2,3} (5020 eV), Ti K+M₁ (5130 eV), Fe K+M_{4,5} (7161 eV), Fe K+M_{2,3} (7203 eV), and Fe K+M₁ (7313 eV).

Figure 1 shows the Ti K β fluorescence spectra for FeTiO₃ bulk crystals measured at the different incident X-ray energies. The Ti K β _{2,5} profiles at 7161 eV seems to be a little different from other profiles at 7203 eV and 7313 eV. It is considerable that the difference may be ascribed to some ionization processes. Unfortunately, due to some beamline trouble, data accumulation time could not serve so sufficient. The statistical error for the spectra was nearly consistent with the probability of the 3d-valence-hole creation through the super Coster-Kronig transition ascribed to low photon flux around the Ti K edge. We can not conclude whether the K β _{2,5} peak profiles were really influenced by the change of the 3d electron population in Fe or not. We need the further measurement and analysis in order to obtain the well-defined spectra.

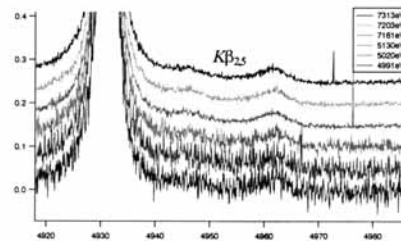


Figure 1 High-resolution Ti K β spectra of FeTiO₃ as a function of the incident X-ray energy.