Materials Science

13C, 27A/2008G575

Surface characterization of β-$FeSi_2$ single crystals by XPS and XAS

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Introduction

Semi-conducting silicides are extensively investigated for using as silicon-based electronic devices. Among silicides, β-$FeSi_2$ having a band gap of 0.85 eV is a candidate as a promising semiconductor. Recently, large single crystalline β-$FeSi_2$ has synthesized by solution growth using Ga solvent [1]. Since then, it is expected to use crystalline β-$FeSi_2$ as a substrate for homoepitaxial growth of β-$FeSi_2$ films. In order to fabricate homoepitaxial films with excellent quality, well-controlled surface of the substrate is necessary.

In the present study, a combination of XPS and XAS is applied to clarify surface chemical states of a β-$FeSi_2$ single crystal and an annealed β-$FeSi_2$ single crystal.

Experimental

The β-$FeSi_2$ single crystals with several millimeter in width were synthesized with the temperature-gradient solution method using Ga-solvent. The (111) face of the single crystal was prepared through mechanical polishing followed by chemical etching with HF solution. The single crystal was annealed at 900°C.

The XPS and XAS measurements were performed at beam lines 13C and 27A [2]. The Si 2p XPS and Fe L-edge XAS spectra were measured at the beam line 13C. In the XPS measurement, excitation X-ray energies were set at 254, 389, 505, 650, 775 and 970 eV. The Fe L-edge XAS spectra were obtained by dividing the signals from the samples recorded on a channeltron in the total-electron-yield mode. The Si K-edge XAS spectra were obtained at the beam line 27A.

Results and Discussion

Figure 1 shows the Si 2p XPS spectra of the β-$FeSi_2$ single crystal. The spectra were measured with the excitation X-ray energy ranging from 254 to 970 eV to perform depth profiling. Mainly three peaks are observed in all spectra. The peak at about 103 eV assigned to SiO_2 increases with decreasing excitation energy. The two peaks at lower binding energies correspond to the Si $2p_{3/2}$ and $2p_{1/2}$ peaks of $FeSi_2$. A simulation of the surface oxide layer suggests that the thickness of the SiO_2 layer is estimated to be 0.77 nm.

The analytical depth in the XPS measurement is below a few nm. In order to obtain information on chemical states at deeper region, XAS measurements were performed. Figure 2 shows the Si K-edge XPS spectra of the β-$FeSi_2$ single crystal before and after annealing at 900°C. Four peaks at about 1840.5 and 1842, 1845 and 1848 eV are observed in the spectra of the samples before and after annealing. The peak at 1840.5 eV can be assigned to the Si-Si bond in $FeSi_2$ structure. The relative intensity ratio of this peak decreases by annealing. In contrast, the relative intensity of the peak at 1842 eV increases by annealing. This peak is attributed to the Si-Fe bond in $FeSi_2$ structure. Therefore, the increase in the relative intensity suggests the formation of Si-Fe structure by annealing. These results indicate that the combination of XPS and XAS is a powerful tool to elucidate the surface chemical states of single crystalline β-$FeSi_2$.

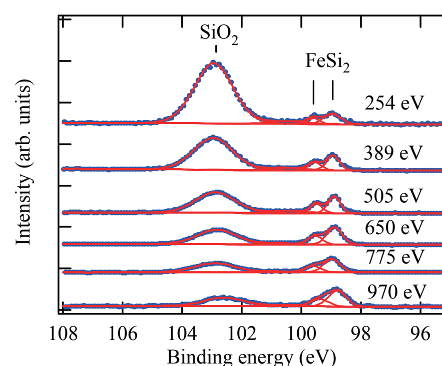

Fig.1. The Si 2p XPS spectra of the β-$FeSi_2$ single crystal measured with the excitation energies from 254 to 970 eV.

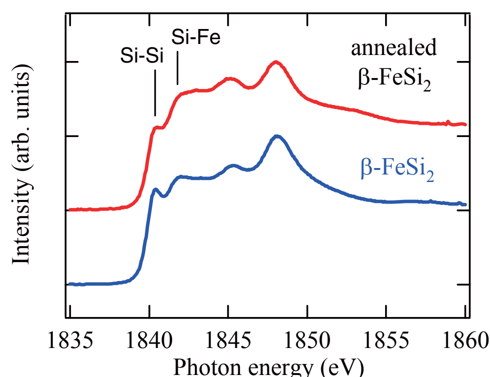

Fig.2. The Si K-edge XAS spectra of the β-$FeSi_2$ single crystals before and after annealing at 900°C.

References

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