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XANES of Mechanically Alloyed Y-Fe System

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Abstract We have measured X-ray absorption near edge structure (XANES) of mechanically alloyed Y_2Fe and YFe_2 system as a function of milling time. An amorphous phase is obtained by mechanical alloying for 100 hours in the Y_2Fe system and for 500 hours in the YFe_2 system. In both systems the intensity of Fe $1s-4p\pi$ transition grows up significantly with increasing the milling time and approaches to that of a melt spun amorphous $Y_{67}Fe_{33}$ alloy. On the other hand, the shape of $1s-4p\sigma$ transition becomes broad with the milling time.

1. INTRODUCTION

Solid state reaction techniques have attracted attention for synthesizing amorphous materials as well as traditional solidification methods such as thermal evaporation, sputtering and melt quenching. Mechanical alloying is among the techniques, which means milling a mixture of crystalline powders of the pure elements [1]. The amorphization by mechanical alloying is due to a solid state interdiffusion reaction near boundaries between the pure constitutive elements, the kinetics of which is controlled by the excess point and lattice defects created by plastic deformation [2]. When the binary system has a negative heat of mixing between the pure elements, mechanical alloying is a method promising of making homogeneous amorphous alloy powders. This is because the negative heat of mixing promotes the interdiffusion reaction and the homogenization between the elements in the atomic scale.

The Y-Fe system possesses an eutectic composition of Y_2Fe . It is well known that it is easy to obtain amorphous phase at the eutectic composition using the traditional melt spinning method. On the opposite composition in the system there is a YFe_2 compound with a cubic C15 Laves phase structure [3]. To investigate how mechanical alloying changes the structure and the electronic states of Fe in the Y-Fe system, we attempt to measure X-ray diffraction patterns and X-ray absorption near edge structure (XANES) spectra of mechanically alloyed Y_2Fe and YFe_2 as a function of the milling time.

2. EXPERIMENTAL

We made powder specimens with the nominal compositions of Y_2Fe and YFe_2 by mechanical alloying. The starting material was a mixture of pure crystalline Fe and Y powders in the purity of 99.95 %. They were milled in a stainless steel vial with stainless steel balls in vacuum; then the vial was cooled by water. Four kinds of samples were prepared as references: pure crystalline Fe powders, a compound YFe_2 with C15 Laves phase structure, a melt spun amorphous $Y_{67}Fe_{33}$ alloy and a Cu foil 6 μm thick. We made the metallic compound YFe_2 by arc melting in an argon atmosphere. The ingot was ground into a fine powder of less than 20 μm in diameter. The mechanically alloyed powder and these reference powders were stuck uniformly on an adhesive tape. Specimens for the measurement were a few pieces of tapes piled up. The $Y_{67}Fe_{33}$ ribbon 20 μm thick was made by quenching on a rotating copper disc; the amorphous state of the ribbons was confirmed with X-ray diffraction using Cu K α radiation. We also measured X-ray diffraction patterns in a $\theta-2\theta$ scan mode for the mechanically alloyed Y_2Fe and YFe_2 samples to investigate the change in their structure on the milling time.

XANES measurements were carried out at the beam line 10B station of the Photon Factory (the National Laboratory for High Energy Physics, Tsukuba, Japan) [4]. The storage ring of the synchrotron radiation was operated with an electron energy of 2.5 GeV and a current of 300 mA in maximum. X-rays were monochromatized with a Si (311) channel-cut crystal which enabled us to obtain the energy resolution of 0.45 eV near the Fe K edge. The XANES spectrum was measured near the K edge of Fe at 300 K in a transmission mode. Intensities of photons incident upon the specimen and passed through it were detected with two independent ionization chambers: one was of 17 cm in length filled with a nitrogen gas flowing and the other was of 31 cm with a mixed gas of 85% nitrogen and 15% argon. The energy of photons was corrected with the energy value for a prepeak near the Cu K edge (8978.8 eV).

3. RESULTS AND DISCUSSION

The X-ray diffraction intensity of mechanically alloyed Y_2Fe is plotted against the angle 2θ in Figure 1. The main reflections from Y (200) and Fe (110) planes sharply decrease with increasing the milling time and almost disappear after 100 hour milling. The width of the reflection increases slightly with the milling time. The other reflections from Fe and Y vanish faster than the Y (200) and Fe (110) reflections; we cannot detect any other reflections than Y (200) and Fe (110) after 50 hour milling. These mean that mechanical alloying for 100 hours makes the Y_2Fe sample homogeneously amorphous. In the mechanically alloyed YFe_2 system we observe no reflections but a small peak of Fe (110) after 400 hour milling; after 500 hour milling, only a broad lump remains near the angle of the Fe (110) reflection. Therefore we regard the YFe_2 specimen after 500 hour milling as an amorphous alloy. It is noted that we have not observed any reflections suggesting formation of the YFe_2 compound during the milling. The amorphous phase of Y_2Fe can be obtained by mechanical alloying for much shorter time than that of YFe_2 . This is because Y_2Fe is just an eutectic composition in the Y-Fe phase diagram as mentioned above.

Figures 2 and 3 show observed XANES spectra near Fe K edge for mechanically alloyed Y_2Fe and YFe_2 together with the pure crystalline Fe powder, the melt spun amorphous $Y_{67}Fe_{33}$ ribbon and the metallic compound YFe_2 . The spectrum for the crystalline Cu foil is also shown for comparison. The energy indicates the difference from the threshold for Fe and Cu K edges. We pay attention to broad peaks at 1.1 eV and 19.6 eV above the threshold energy for pure crystalline Fe. We consider these peaks as $1s \rightarrow 4p\pi$ ($1s \rightarrow 4p\pi$) and $1s \rightarrow 4p\sigma$ ($1s \rightarrow 4p\sigma$) transitions. The intermetallic compound YFe_2 and the amorphous $Y_{67}Fe_{33}$ alloy have clearer peaks of $1s \rightarrow 4p\pi$ transition and broader peaks of $1s \rightarrow 4p\sigma$ transition than those of pure Fe. For mechanically alloyed Y_2Fe and YFe_2 , the peak of $1s \rightarrow 4p\sigma$ transition becomes broader and the peak of $1s \rightarrow 4p\pi$ transition clearly grows up as the milling time increases. However the energy for these peaks do not shift within the experimental accuracy. When Fe powders are mechanically alloyed in both systems, invasion of Y atoms at an interstitial site between Fe atoms makes BCC lattices of Fe deformed and the crystal field on Fe sites changed; thereby the $4p$ electronic state of Fe as a final state of transition is changed. We emphasize that Y_2Fe after 100 hour milling and YFe_2 after 500 hour milling are almost same as the melt spun amorphous $Y_{67}Fe_{33}$ alloy in the shape of $1s \rightarrow 4p$ transition. This means that the electronic states of Fe in both these mechanically alloyed samples approach to those of the melt spun $Y_{67}Fe_{33}$ alloy. As shown in Figure 3, the FCC Cu metal has a sharp $1s \rightarrow 4p\pi$ transition like the melt spun alloy and these mechanically alloyed samples. Therefore Fe atoms in the mechanically alloyed Y_2Fe after 100 hour milling and YFe_2 after 500 hour milling feel the same crystal field as Cu atoms in the pure Cu metal; this suggests that the mechanically alloyed specimen has a local structure like FCC lattices. It should be added that there is no difference in XANES of Y K edge between the mechanically alloyed specimens at different milling time.

Figure 1: X-ray diffraction patterns for mechanically alloyed Y_2Fe together with crystalline Fe and Y powders. Intensities for crystalline Fe and Y are reduced to 1/3 and 2/3 of observed results.

Figure 2: XANES spectra near the Fe K edge for mechanically alloyed Y_2Fe , crystalline Fe powder and melt spun amorphous $Y_{67}Fe_{33}$ alloy. The energy is measured from the Fe threshold energy.

Figure 3: XANES spectra near the Fe K edge for mechanically alloyed YFe_2 , metallic YFe_2 compound and crystalline Fe powder. XANES of Cu foil is also shown. The energy shows the difference from the threshold energy.

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