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XAFS Study on Local Structure Change of $Cu_{33}Zr_{67}$ Alloys during Cyclic Transformation from Crystal to Amorphous Phase by Ball-Milling

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Introduction

Mechanical alloying (MA) of elemental powders mixture and mechanical milling (MM) of intermetallic compound by using ball-milling has been successfully employed in recent years as a method for synthesizing amorphous and nano-crystalline alloys. A cyclic phase transformation of alloy by ball-milling was found in $Cu_{33}Zr_{67}$ alloy. The purpose of this work is to study the micro-structural changes during this cyclic phase transformation by ball-milling.

Sample preparation

Pure elemental powders (99.9%) of Cu (20 μm) and Zr (50 μm) were balanced to give the nominal composition of $Cu_{33}Zr_{67}$ and were mixed in a glove box under purified argon atmosphere. The mixed powders of the starting reactant materials were then sealed into a tempered chrome steel vial (250 ml in volume) together with 50 tempered chrome steel balls (10 mm diameter). The ball-to-powder weight ratio was maintained as 14:1 The MA process was performed in a high –energy planetary ball mill (Fritsch P5) at rotation speed of 3.3 s^{-1}. The structural changes of the powders with milling time were followed by XRD, employing Cu K_α radiation. The sample for EXAFS measurements were prepared by peletizing powdered samples together with BN fine powder. EXAFS measurements were carried out at beam line 12C of Photon Factory in KEK. Energy scanning was carried out around the Cu K-edge and Zr K-edge using a Si (111) monochromater in transmission mode at 20K. The radial distribution function (RDF) around the respective atoms were derived by the Fourier transform of EXAFS $k^3\chi$ (k).

Results

XRD measurements show the cyclic phase transformation, crystalline phase-to-amorphous phase occurs. At 24 hours of milling, the sample phase become amorphous, then at 48 hours become to crystalline phase, and then at 100 hours the phase of the sample become to amorphous again.

Table 1 The cyclic phase transformation of $Cu_{33}Zr_{67}$ alloy

Milling time (h)	Phase of $Cu_{33}Zr_{67}$ alloy
0	Crystalline phase
24	Amorphous phase
48	Crystalline phase
100	Amorphous phase

EXAFS measurements

Unfortunately, Ni contamination was found in pre-edge part of Cu K-edge EXAFS, Hf L_{III} and Ta L_{III} absorptions were also detected in post Cu K-edge part. The amount of these contaminations of Ni, Hf and Ta in $Cu_{33}Zr_{67}$ alloy is not small. So that the effect of these contaminations on the Fourier transform of EXAFS around Cu atom is considerable large. This means that the analysis in details has no meaning.

Why contaminations occur?

The contamination of Ni, Hf and Ta in $Cu_{33}Zr_{67}$ alloy seems to be from vial and milling-bal. These vial and balls were used before the sample making for another sample, which contain Ni, Hf and Ta. The cleanings of the vial and balls were not enough, and not complete. So the Ni, Hf and Ta transferred to $Cu_{33}Zr_{67}$ alloy from the surface of vial and balls.

References

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