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Structural Transformation of Immiscible Metals Induced by Mechanical Alloying

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Abstract X-ray absorption fine structure(XAFS) is used to investigate the local structural change of $\text{Fe}_{60}\text{Cu}_{40}$ and $\text{Fe}_{80}\text{Cu}_{20}$ with the ball milling time. The XAFS results verify that the solid solution of mechanically alloyed Fe-Cu samples are inhomogeneous. We propose that the bcc to fcc structural transformation of Fe small particles of $\text{Fe}_{60}\text{Cu}_{40}$ (160h) is induced by interfacial Cu atoms dissolving in small Fe grain boundary during the mechanical alloying process.

1. Introduction

Recently, a new interest has arisen by the discovery that mechanical alloying(MA) can make some immiscible metals to form the metastable phase alloys. This phenomenon has been considered as that the solid solubility of immiscible binary metals can be greatly extended by MA. The mechanically alloyed Fe-Cu binary metals is a representative alloy, its solid solubility and structure have been widely investigated by Shingu[1], Yavari[2], Crespo[3] and us[4] with x-ray diffraction(XRD), electron microscopy, and Mossbauer spectroscopy. The XRD patterns show the distinct peaks of fcc copper and bcc iron for initial $\text{Fe}_{60}\text{Cu}_{40}$ mixture. The bcc Fe peaks gradually decrease with the milling time, fully disappear after 160 hours milling and only fcc peaks remain. The bcc Fe peaks subsequently return after annealing at 773K. However, the local structure of Fe and Cu of mechanically alloyed Fe-Cu samples has not been clear yet: whether or not the Cu atoms in the Cu lattice are homogeneously substituted by Fe atoms according to their chemical composition. In more recent studies, we found that $\text{Fe}_{60}\text{Cu}_{40}$ supersaturated solid solution forming from MA is inhomogeneous alloy by Mossbauer spectroscopy[4].

In this short report, XAFS has been performed to measure the local structures of Fe and Cu atomic neighbor environment of $\text{Fe}_{60}\text{Cu}_{40}$ and $\text{Fe}_{80}\text{Cu}_{20}$ samples at various ball milling time. Our aims are to study separately the structural change of Fe and Cu near neighbor coordination with the ball milling time, verify whether the Fe atoms are homogeneously dissolved into fcc metal Cu phase for $\text{Fe}_{60}\text{Cu}_{40}$ (160h) and the Cu atoms are dissolved into bcc Fe phase for $\text{Fe}_{80}\text{Cu}_{20}$ (160h).

2. Experimental

Crystalline iron and copper powders with particle sizes less than 200 mesh and purity higher than 99.9% were mixed to the desired composition. 10g mixtures and 100g tungsten carbide balls were sealed inside a cylindrical stainless steel vial filled with argon gas, and then MA was performed in a planetary ball with a rotation speed of about 210 r/min. Fe-Cu mixtures milled for 10, 40 and 160 hours, were represented as Fe-Cu(10h), Fe-Cu(40h), Fe-Cu(160h), respectively.

The Fe and Cu K-edge x-ray absorption spectra of mechanically alloyed Fe-Cu samples were measured at the XAFS station of 4W1B beamline of Beijing Synchrotron Radiation Facility. The electron beam energy was 2.2GeV and maximum stored current is 50mA. Data were collected with a fixed exit monochromator using two flat Si(111) crystals. The energy resolution was about 2~3 eV by the Cu foil 3d near edge feature. Data were collected in transmission mode using ion chambers with mixed Ar/N_2 fill gas at room temperature. The X-ray harmonics were minimized by detuning the two flat Si(111) crystal monochromator to about 70% of the maximum incident light intensity.

3. Results and discussion

The radial distribution functions(RDF) of Fe and Cu atoms of mechanically alloyed Fe-Cu binary metals are shown in Fig. 1. The RDF of bcc α -Fe has three strong amplitude peaks($R=2.20\text{\AA}$, 3.66\AA , 4.48\AA) in the region of 1~6 \AA . The magnitude intensity of the third peak of bcc α -Fe is very stronger, many times larger than the corresponding third peak of fcc metal Cu. We can then recognize the local neighbor structure of Fe or Cu for mechanically alloyed Fe-Cu samples with this character. It can be observed in Fig.1 that the magnitude intensity of the first peak of $\text{Fe}_{60}\text{Cu}_{40}$ sample gradually decreases with the milling time. After 160 hours ball milling, The magnitude intensity of main peak drops about one third. The third peak ($R=4.48\text{\AA}$) disappears for the $\text{Fe}_{60}\text{Cu}_{40}$ (160h), its feature of RDF is completely different from that of bcc α -Fe, but is similar to that of fcc

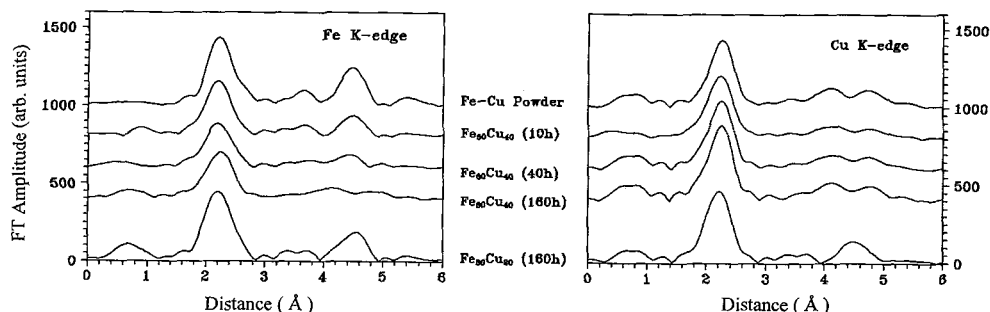


Fig.1 The RDF of Fe and Cu atoms for mechanically alloyed Fe-Cu samples

metal Cu (three stronger amplitude peaks of fcc metal Cu are at 2.26Å, 4.12Å and 4.74Å). This result confirms that the MA can change the neighbor environment of Fe atoms of $\text{Fe}_{60}\text{Cu}_{40}$ from bcc structure to fcc structure. The Fe atoms of $\text{Fe}_{80}\text{Cu}_{20}$ exist still in the form of bcc structure in spite of 160 hour milling. Moreover, the magnitude intensity of main peak is the same as that of its initial $\text{Fe}_{80}\text{Cu}_{20}$ powder mixture. We noted an interesting XAFS results, the RDF of Cu in $\text{Fe}_{80}\text{Cu}_{20}$ (160h) appears the third stronger peak at 4.48Å, which is similar to that of bcc α -Fe. It suggests that the Cu neighbor coordination environment of $\text{Fe}_{80}\text{Cu}_{20}$ is changed from fcc structure to bcc structure after 160 hour milling.

Although the $\text{Fe}_{60}\text{Cu}_{40}$ was milled 160 hours, the magnitude intensity of the main peak of Cu appears no evident change. It means that the Cu local neighbor structure of Fe-Cu (160h) is the same as that of its initial Fe-Cu mixture. MA does not increase the disorder degree of Cu neighbor coordination of $\text{Fe}_{60}\text{Cu}_{40}$ samples, even if the disorder degree of the other element Fe increases with the milling time. Therefore, it is impossible that much amount metal Fe is dissolved into Cu phase. Table 1 lists the fitting results for the $\text{Fe}_{60}\text{Cu}_{40}$ (160h) and $\text{Fe}_{80}\text{Cu}_{20}$ (160h). The structural parameters show that Fe atoms or Cu atoms are coordinated by both of Fe and Cu atoms in the nearest neighbor shell. The Fe atoms are mainly surrounded by Fe atoms and the Cu atoms are still tending to coordinated by Cu atoms for $\text{Fe}_{60}\text{Cu}_{40}$ (160h), the ratio of Fe coordination number to Cu coordination number in Fe first coordination shell greatly deviates from its chemical composition. XAFS results indicate that Fe is rich in some region and Cu is rich in other region for these MA Fe-Cu samples. This conclusion is supported by the results of Mossbauer spectroscopy[4] and high-resolution transmission electron microscopy[5]. XAFS results further verified that Cu atoms of fcc Cu lattice in $\text{Fe}_{60}\text{Cu}_{40}$ (160h) are not homogeneously substituted by Fe atoms after 160 hour milling.

Table 1. The structural parameters of the first shell for Fe-Cu samples fitting with Fe and Cu K-edge XAFS spectra

sample	K-edge	bond type	R(Å)	σ (Å)	N	ΔE_0	K-edge	bond type	R(Å)	σ (Å)	N	ΔE_0
$\text{Fe}_{60}\text{Cu}_{40}$ (160h)	Fe	Fe-Fe	2.56	0.094	8.2	1.0	Cu	Cu-Cu	2.56	0.089	6.6	4.0
		Fe-Cu	2.56	0.089	3.3	-3.7		Cu-Fe	2.56	0.087	5.2	-1.1
$\text{Fe}_{80}\text{Cu}_{20}$ (160h)	Fe	Fe-Fe	2.49	0.079	7.0	2.0	Cu	Cu-Cu	2.50	0.079	5.5	2.0
		Fe-Cu	2.50	0.080	1.0	-2.7		Cu-Fe	2.50	0.080	3.6	2.5

During the milling process to make Fe-Cu powder mixture down into small particles, the interdiffusions of Fe and Cu are simultaneously produced in the region of Fe grain boundary coherent with Cu grain by MA. We observed that the mechanically alloyed Fe-Cu samples are nanometer particles with transmission electron microscope[6]. Moreover, the higher Cu concentration is more effective to make the Fe-Cu powder mixture the small size particles. For $\text{Fe}_{60}\text{Cu}_{40}$ (160h), we consider that only little amount of Cu atoms can diffuse into the core of Fe grain which corresponds to a Fe-rich region, deducing from the structural parameters in table 1. The bcc structural Fe grain is unstable while Cu atoms reach some concentration in the interfacial region and Fe grains are broken down to very small sizes. Interfacial Cu atoms drive phase transition from bcc α -Fe to metastable fcc Fe phase. For $\text{Fe}_{80}\text{Cu}_{20}$ (160h), the Cu atomic concentration is lower, so that Fe particles are not broken down to small size. Fe particles keep the bcc α -Fe structure. On contrary to $\text{Fe}_{60}\text{Cu}_{40}$ (160h), fcc Cu phase is transformed to bcc phase, inducing by bcc α -Fe phase.

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