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MAGNETIC PROPERTIES OF BALL MILLED Fe_xCu_{1-x} (x=0.4, 0.5) ALLOY

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The samples $Fe_{0.4}Cu_{0.6}$ and $Fe_{0.5}Cu_{0.5}$ ball milled for 50 h are investigated by X-ray diffraction, Mössbauer spectra, as well as magnetic measurement. The experiments show that the structure of the samples is fcc, with lattice constant 0.361 nm and there are fcc Fe-rich phase and fcc Cu-rich phase in the samples. Most of Fe atoms (91%) are in the fcc Fe-rich phase, which is a ferromagnetic phase. The M-H curve at 1.5 K shows the saturation magnetization of the samples are 80.5 emu/g and 101.6 emu/g for $Fe_{0.4}Cu_{0.6}$ and $Fe_{0.5}Cu_{0.5}$ respectively. The average magnetic moment of Fe atoms is deduced to be 2.40 μ_B . Compared with theoretical predication, the Fe atoms in the fcc phase are in high spin state.

PACC: 7550B; 7680; 6116N

I. INTRODUCTION

There are a lot of investigations on Fe/Cu system prepared by mechanical alloying[1], but many questions still need to be solved. The structure and magnetic properties of these alloys vary with Fe concentration and ball milling time. It was shown by XRD,Mössbauer spectra, EXAFS and magnetic properties measurement that for $x \geq 0.8$ the ball milled sample has bcc structure; for $0.8 > x > 0.6$ bcc and fcc structure coexist and for $x < 0.6$ the sample has fcc structure. For example, for the as-milled $Fe_{0.6}Cu_{0.4}$[2], as ball milling time increases, the proportion of bcc-Fe decreases and disappears at last and the fcc Fe-rich phase emerges instead of bcc Fe phase. When annealed at 500 ℃, fcc Fe-rich phase reverses to bcc Fe phase. Thus in the process of ball milling and subsequent annealing, a phase transition process bcc Fe→fcc Fe-rich→bcc Fe takes place. Similar structure change occurs in $Fe_{0.5}Cu_{0.5}$[3]. Accompanying the changes in structure, the curves of M-T are changed. Ambrose *et al.*[4] provided M-T curve with an applied field of 0.5 T for $Fe_{0.3}Cu_{0.7}$, $Fe_{0.4}Cu_{0.6}$ and $Fe_{0.5}Cu_{0.5}$ milled for 72 h. Jiang *et al.*[1] provided M-T curve with 1 T field for milled $Fe_{0.5}Cu_{0.5}$. Both of these reports show that for the as-milled sample the magnetization decreases with increasing temperature up to a certain temperature and then begins to increase to a maximum, after which decreases to zero. Hernando and Crespo *et al.*[5] reported that the magnetization of as-milled sample $Fe_{0.5}Cu_{0.5}$ below 300 K is greater than that of sample annealed through 900 K.

The magnetic structure of fcc Fe has been investigated by Yu-mei Zhou[6] *et al.* in a range of lattice constants from a=0.350 nm to 0.375 nm using the first-principles theory method. Besides the previously reported antiferromagnetic phase, one high-spin and two low-spin ferromagnetization phases, a ferrimagnetic phase are predicted to coexist in the range of a=0.350 nm to 0.375 nm. The detailed results are shown is in table 1.

Table 1. Atomic magnetic moments (in μ_B) and valence electron number (integrated over contact muffin-tin spheres) for fcc Fe with α=3.61Å in different magnetic states. (Data reproduced from reference [6])

	Para	Ferro			AFM		Ferri	
		LS1	LS2	HS	atom1	atom2	atom1	atom2
electron								
total	7.244	7.245	7.244	7.244	7.243	7.241	7.230	7.254
Spin-up	3.622	4.263	3.786	4.874	4.362	2.880	4.571	2.985
Spin-down	3.622	2.982	3.458	2.370	2.881	4.361	2.659	4.269
Moments	0	1.281	0.328	2.504	1.481	−1.481	1.912	−1.285

In this paper samples of $Fe_{0.4}Cu_{0.6}$ and $Fe_{0.5}Cu_{0.5}$ ball milled for 50 hours are investigated with X-ray diffraction (XRD), Mössbauer spectra, as well as magnetic measurement. The investigation indicates the Fe atoms in the fcc phase are in high spin state.

Ⅱ. SAMPLE PREPARATION

The purity of Fe and Cu powders is better than 99.8%. The Fe and Cu powders were mixed in 4:6, 5:5 atomic proportions respectively and the mixture was placed into a hardened steel vial with steel balls. The samples were milled separately. The mass ratio of the balls to the mixture was 20:1. The vial was sealed under an argon atmosphere to prevent oxidation of the sample. All samples were milled for 50 h and a part of the sample was annealed at 650 ℃ in vacuum of 1×10^{-3} Pa for one hour.

Ⅲ. EXPERIMENT AND ANALYSIS

The samples were measured with a Rigaku-Max-PA rotating target X-ray diffractometer (Cu Kα; λ=0.154 nm). XRD patterns of as-milled $Fe_{0.4}Cu_{0.6}$ (50 h) and $Fe_{0.5}Cu_{0.5}$(50 h) are identical and presented in Figure 1(a). The diffraction peak positions 2θ=43.2° and 50.1° correspond to the series of peaks of fcc Cu with lattice constant 0.361 nm. The main peak of bcc Fe at 2θ=44.3° is not observed. Obviously, the bcc Fe structure disappears and the samples has fcc structure after ball-milling.

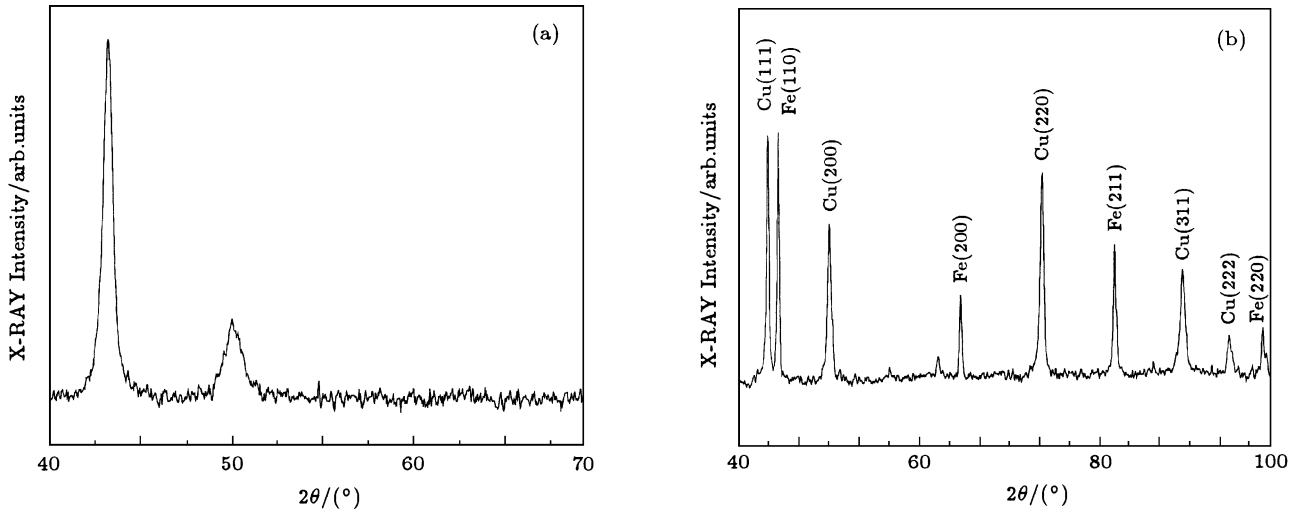


Fig.1. (a) XRD pattern of as-milled $Fe_{0.4}Cu_{0.6}$ (50 h) and $Fe_{0.5}Cu_{0.5}$ (50 h) (b) XRD pattern of ball-milled $Fe_{0.4}Cu_{0.6}$ (50 h) and $Fe_{0.5}Cu_{0.5}$ (50 h) annealed at 650 ℃ in vacuum 1×10^{-3} Pa for one hour.

XRD pattern of ball-milled sample annealed at 650 ℃ in vacuum of 1×10^{-3} Pa for one hour is represented in Figure 1 (b). It has not only a series of diffraction peaks of fcc Cu, but also diffraction peaks of bcc Fe. It's evident that the fcc Fe-rich phase in as-milled sample reverses to bcc Fe phase duing the annealing process. The phase transition (bcc Fe→fcc Fe-rich→bcc Fe) in the ball-milling and subsequent annealing process agrees with our previous result[2,3].

Mössbauer spectra were recorded at room temperature using the MS-500 Mössbauer spectrometer and ^{57}Co/Pd radiation source. Figure 2 shows the experimental, fitted Mossbauer spectra and subspectra of as-milled samples, (a) $Fe_{0.4}Cu_{0.6}$ and (b) $Fe_{0.5}Cu_{0.5}$. Table 2 shows the fitted data of Mossbauer spectrum for $Fe_{0.4}Cu_{0.4}$ and $Fe_{0.5}Cu_{0.5}$. As the samples are pulverous, the area ratio of the first, the second and the third peaks in a sextet is set to be 3:2:1 when fitting Mössbauer spectrum data. The non-recoil fractions of various phases in a sample are assumed to be identical. From fitted Mössbauer spectra it could be seen that $Fe_{0.4}Cu_{0.6}$ and $Fe_{0.5}Cu_{0.5}$ both have broadened sextet and doublet peaks. The hyperfine field of $Fe_{0.4}Cu_{0.6}$ is 18.1–18.4 T and the relative proportion of area of sextet is 91%, and the hyperfine field of $Fe_{0.5}Cu_{0.5}$ is 21.0–24.5 T with a relative area proportion 92%. This result agrees with our previous result that the main part of samples is fcc Fe-rich phase[2,3].

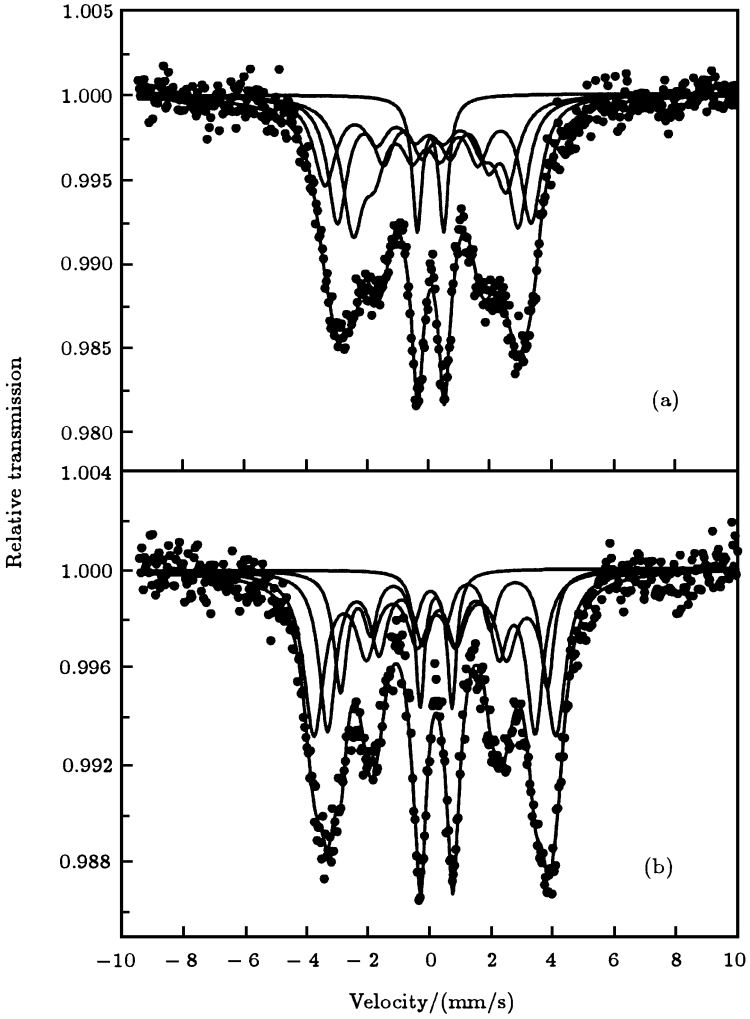


Fig.2. Experimental, fitted Mössbauer spectra and subspectra of samples measured at room temperature, (a) as-milled $Fe_{0.4}Cu_{0.6}$ (50 h) (b) as-milled $Fe_{0.5}Cu_{0.5}$(50 h).

Table 2. Fitted data of Mössbauer spectrum for $Fe_{0.4}Cu_{0.4}$ and $Fe_{0.5}Cu_{0.5}$

		Doublet	Sextet I	Sextet II	Sextet III
$Fe_{0.4}Cu_{0.6}$	IS(mm/s)	0.087	0.214	0.142	−0.168
	QS(mm/s)	0.864	0.487	−0.270	−0.423
	HW(mm/s)	0.194	0.423	0.365	0.452
	H_{hf}(kOe)		180.78	183.32	183.74
	A(%)	9.10	35.62	27.43	27.85
$Fe_{0.5}Cu_{0.5}$	IS(mm/s)	0.227	0.211	0.266	0.202
	QS(mm/s)	1.037	−0.061	0.437	−0.258
	HW(mm/s)	0.177	0.400	0.261	0.325
	H_{hf}(kOe)		244.83	209.28	210.30
	A(%)	8.06	40.32	19.36	32.26

The curve of M-H measurement at T=1.5 K for samples $Fe_{0.4}Cu_{0.6}$ and $Fe_{0.5}Cu_{0.5}$ is represented in Figure 3(a,b). It could be seen that the magnetization increases rapidly with the applied field when H<0.1 T. When H>1 T, the magnetization reaches its saturation magnetization. For $Fe_{0.5}Cu_{0.5}$ (50 h), M_S=101.6 emu/g, for $Fe_{0.4}Cu_{0.6}$(50 h), M_s=80.5 emu/g. Because the proportion of Fe atom in fcc Fe-rich is 91%, the average Fe atomic magnetic moment in the fcc Fe-rich phase

should be 2.40 μ_B.

Comparing with theoretical predication[6], the fcc Fe-rich phase only stands the chance to be the ferromagnetic phase in a high spin state. This atom magnetic moment 2.40 μ_B is smaller than the theoretically predicted 2.50 μ_B, which is reasonable because the latter is in perfect crystal lattice, and its Fe atom number in unit volume is more than that in fcc Fe-rich phase of the experimental sample.

The result of M-T measurement performed in applied field H=0.1 T and H=1.2 T respectively for $Fe_{0.4}Cu_{0.6}$ (50 h) is represented in Figure 4(a, b). By comparing Figure 4(a) with Fig 4(b), it could be seen that the sample $Fe_{0.4}Cu_{0.6}$ displays various magnetic properties in different applied fields. For low field H=0.1 T,at T=300 K, the magnetization before annealing is 28.0 emu/g and 22.0 emu/g after annealing. That is to say, the magnetization of fcc Fe-rich phase is greater than that of bcc Fe phase, i.e. $M(300)_{fcc} > M(300)_{bcc}$. For a high field, the magnetization of as-milled sample is 59.7 emu/g, while that of annealed sample is 77.2 emu/g. The obvious fact is $M(300)_{fcc} < M(300)_{bcc}$, which is opposite to the case of low applied field. The M-H curve of Figure 3 shows that the magnetization of $Fe_{0.4}Cu_{0.6}$ sample does not reach its saturation state in low applied field, while the sample is saturated in high applied field 1.2 T.

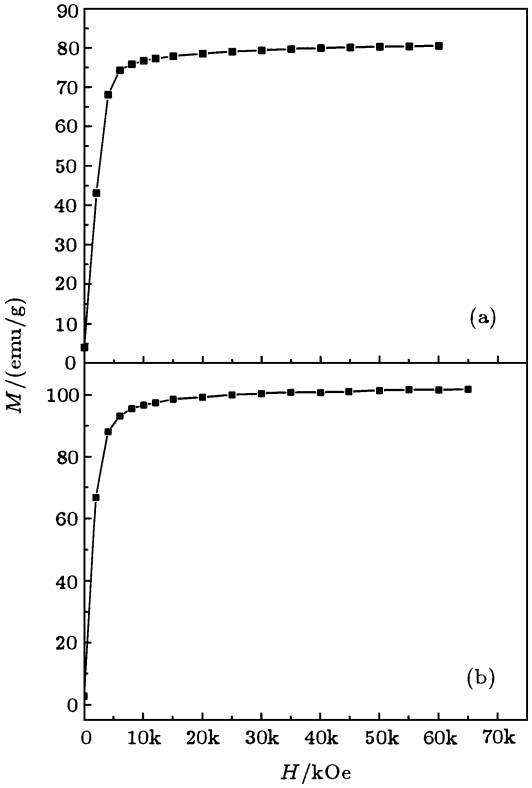


Fig.3. M-H measurement at T=1.5 K for $Fe_{0.4}Cu_{0.6}$ and $Fe_{0.5}Cu_{0.5}$; (a) $Fe_{0.4}Cu_{0.6}$(50 h), (b) $Fe_{0.5}Cu_{0.5}$(50 h).

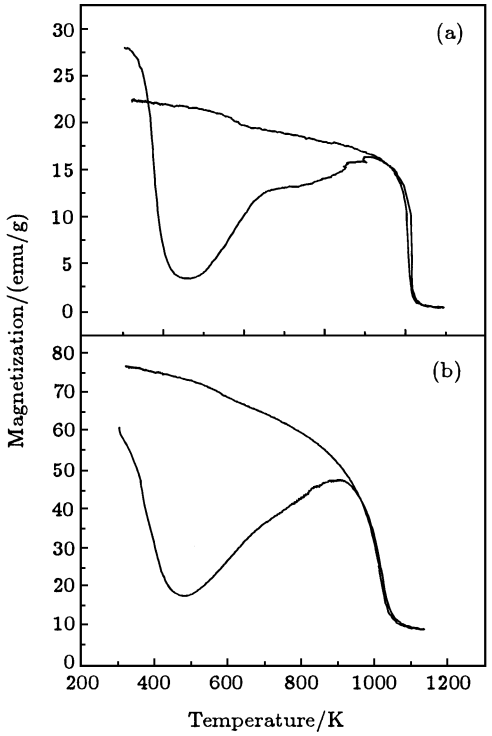


Fig.4. M-T measurement in applied field (a) H=0.1 T, (b) H=1.2 T respectively for $Fe_{0.4}Cu_{0.6}$ (50 h).

IV. CONCLUSION

The samples $Fe_{0.4}Cu_{0.6}$ and $Fe_{0.5}Cu_{0.5}$ ball milled for 50 hours are investigated with X-ray diffraction (XRD), Mössbauer spectra, as well as magnetic measurement. The X-ray diffraction pattern shows the structure of samples is fcc structure with lattice constant 0.361 nm. The Mössbauer spectra and magnetic properties measurement show that there are two phases, fcc Fe-rich phase and fcc Cu-rich phase, in the samples. Most of Fe atoms (91%) is in fcc Fe-rich phase, which is a ferromagnetic phase. From M-H curve, at low temperature 1.5 K, the saturation magnetization of the samples is 80.5 emu/g and 101.6 emu/g for $Fe_{0.4}Cu_{0.6}$ and $Fe_{0.5}Cu_{0.5}$ respectively. Therefor the magnetic moment of Fe atom is deduced to be 2.40 μ_B. Comparing with theoretical predication[6], the fcc Fe-rich phase is ferromagnetic phase and the Fe atoms are in high spin state.

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