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Amorphous Fe Clusters Imbedded in fcc La for Melt-Quenched La-Fe Alloys

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Abstract. Fluorescence XAFS measurements of the Fe K-edge are done for melt-quenched La-rich La-Fe alloys. XAFS results of the as-quenched samples of $\text{La}_{92}\text{Fe}_8$ and $\text{La}_{88}\text{Fe}_{12}$ show characteristic features of disordered structure. The results for $\text{La}_{88}\text{Fe}_{12}$ after annealed at 400°C for 30 min. show that Fe are precipitated to α -Fe in β -La. A strange phenomenon of finding the amorphous clusters in the crystalline phase can be attributed to the immiscible nature and large atomic size difference between Fe and La.

1. INTRODUCTION

La and Fe are insoluble within each other and they do not form intermetallic compounds. The studies of structure and magnetic properties so far by Ino et al, see for example ref. [1] and [2], proved that β -La is formed by rapidly quenching of La-Fe alloys (1~12%Fe). No crystalline Fe is found by X-ray diffraction even at 12%Fe. Lattice parameters of β -La are constant despite of the increase in Fe concentration up to 12%Fe suggesting that Fe atoms do not dissolved in β -La. Super paramagnetism of these La-Fe alloys at room temperature suggests the formation of Fe clusters in β -La. Disordered structure of Fe clusters is inspected from the distributed hyperfine field observed by Mössbauer measurements [3]. The main purpose of this work is to elucidate the local structure of Fe clusters by means of XAFS measurements.

2. EXPERIMENTS

Arc melted $\text{La}_{92}\text{Fe}_8$ and $\text{La}_{88}\text{Fe}_{12}$ were melt-quenched by a single-roller melt-spinning technique. Sample thickness is about 30~50 μm and width about 1mm. Because of the very large absorption coefficient of La at the Fe K-edge, the transmission method is not adequate for XAFS measurements and so the fluorescence method is adopted. XAFS measurements of the Fe K-edge for the as-quenched $\text{La}_{92}\text{Fe}_8$ and $\text{La}_{88}\text{Fe}_{12}$, and the annealed $\text{La}_{88}\text{Fe}_{12}$ (400°C for 30 min) were done at BL7C in Photon Factory of KEK using a Si(111) monochromator and a total reflection mirror. A Mn filter (3 μm) was used as a Z-1 filter and an ion chamber as a detector combined with the Lytle collimating slit.

3. XAFS ANALYSIS

Fluorescence method is usually applied to a thin film or a dilute concentration. La-Fe alloy of 12% Fe seems to be out of dilute concentration range. But the ratio of the absorption jump $\Delta\mu(E_0)$ at the Fe K-edge to total absorption $\mu_{\text{total}}(E_0)$ is only 4% due to the large absorption coefficient of La (3067 cm^{-1}). Rough estimation of the part of the Fe EXAFS oscillation $\Delta\mu_{\text{Fe}}(E)$ to the total absorption $\mu_{\text{total}}(E)$ is less than 0.14%. Therefore a dilute approximation is applicable for the $\text{La}_{88}\text{Fe}_{12}$ as well as $\text{La}_{92}\text{Fe}_8$ alloys.

After an usual background subtraction and normalization procedure, $\chi(k)$ was deduced. The resultant $\chi(k)$ and their Fourier transforms, $F(r)$, are shown in Figs. 1 and 2 together with that for pure α -Fe as a reference. The k -range of the Fourier transform is from 2.5 to 13.5 $\text{\AA}^{-1}$. Curve fitting was done for the present XAFS results using EXAFS2[4]. Cumulant expansion up to the fourth order was adopted as a fitting equation for the as-quenched samples because of the anharmonicity in the radial displacement for the disordered structure. As appropriate model compounds are difficult to be found for the present disordered structure, we use theoretical parameters calculated by ab initio method, FEFF[5]. Two model structures for the as quenched samples are examined; Fe in fcc La and Fe in a hypothetical fcc Fe; fcc is adopted as an alternative structure for dense random packing of hard spheres. The former can not fit the observed results while the latter fits well. Bcc Fe is adopted as a model compound for the annealed sample. The detailed curve fitting procedures were cited elsewhere[6]. The results of the curve fitting are listed in the Table 1.

Fig.1 $k^3\chi(k)$ vs k at the Fe K-edge for the as-quenched and annealed $\text{La}_{22}\text{Fe}_8$ and $\text{La}_{33}\text{Fe}_{12}$, and $\alpha\text{-Fe}$.

Fig.2 Fourier transforms of $k^3\chi(k)$ of the Fe K-edge for the as-quenched and annealed $\text{La}_{22}\text{Fe}_8$ and $\text{La}_{33}\text{Fe}_{12}$, and $\alpha\text{-Fe}$.

Table 1 Curve fitting results of the EXAFS spectra for $\text{La}_{22}\text{Fe}_8$ and $\text{La}_{33}\text{Fe}_{12}$. r :atomic distance, N :coordination number, C_{ij} :Cumulants

sample name	$r_1(\text{\AA})$	$r_2(\text{\AA})$	N_1	N_2	$C_{21}(\sigma)(\text{\AA}^2)$	$C_{22}(\sigma)(\text{\AA}^2)$	$C_3 \times 10^3(\text{\AA}^3)$	$C_4 \times 10^4(\text{\AA}^4)$
$\text{La}_{22}\text{Fe}_8(\text{as-q})$	2.492 ± 0.02		4.07 ± 2		0.081 ± 0.05		0.282 ± 0.005	-0.078 ± 0.005
$\text{La}_{33}\text{Fe}_{12}(\text{as-q})$	2.527 ± 0.02		4.10 ± 2		0.095 ± 0.05		0.980 ± 0.005	-0.271 ± 0.005
$\text{La}_{33}\text{Fe}_{12}(\text{anneal})$	2.469 ± 0.02	2.810 ± 0.02	7.36 ± 1	6.71 ± 1	0.062 ± 0.05	0.088 ± 0.05		

4. DISCUSSION

The characteristics of the present XAFS results for the as-quenched one are that the local structures of Fe atoms in the $\text{La}_{22}\text{Fe}_8$ and $\text{La}_{33}\text{Fe}_{12}$ are very similar to each other and show a high degree of disorder; a small amplitude and a rapid decay of the $\chi(k)$ curve and low and broad peaks with no explicit long-distance structures in the $F(r)$ curve. On the other hand annealed $\text{La}_{33}\text{Fe}_{12}$ exhibits a quite similar to those of pure $\alpha\text{-Fe}$.

The disordered structure of the as-quenched $\text{La}_{22}\text{Fe}_8$ and $\text{La}_{33}\text{Fe}_{12}$ seen in the present XAFS results is agreed with the distributed hyperfine field[3] and local structures of Fe clusters are inferred to be amorphous. It is not easy to determine precise size and concentration of the cluster. However it may be said that the nearest neighbor distance determined by curve fitting is close to iron(bcc and fcc) and no definite structure of $\beta\text{-La}$ are detected within about $6\text{\AA}$ as seen in Fig.2. Therefore the size of the cluster is at least above $12\text{\AA}$ in diameter. Although the reasons of the formation of such Fe clusters in crystalline $\beta\text{-La}$ can not be clearly understood now, the following can be inferred: in a rapid cooling process the immiscible nature of Fe in La inclines to exclude Fe from precipitated $\beta\text{-La}$ and the remained liquid is enriched with Fe. This Fe rich melt can be solidified to a phase of an amorphous state or one with a high disordered structure because of the large atomic size difference, $r(\text{Fe})/r(\text{La})=0.69$.

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