# PAC investigation of qudropole interaction in nano-soft magnetic material $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9^*$

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**Abstract** The quadrupole interaction in nano-soft magnetic material Fe<sub>73.5</sub>Cu<sub>1</sub>Nb<sub>3</sub>Si<sub>13.5</sub>B<sub>9</sub> has been studied by perturbed angular correlation using <sup>62</sup>Zn probe nuclei from the ISOL radioactive nuclear beam facility at CIAE HI-13 tandem accelerator. Two quadrupole interaction frequencies  $\omega_{01} = 440$  Mrad/s with a distribution width  $\sigma = 0$  and  $\omega_{02} = 90$  Mrad/s with a width  $\sigma = 0.48$  are obtained. The fractions of  $\omega_{01}$  and  $\omega_{02}$  are 38% and 62%, respectively. The measured quadruple interaction parameters indicate that 62% of the implanted <sup>62</sup>Zn are located in the grain boundary and 32% in the grain.

Key words PAC, Kev-ISOL, Fe<sub>73.5</sub>Cu<sub>1</sub>Nb<sub>3</sub>Si<sub>13.5</sub>B<sub>9</sub>

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### 1 Introduction

Nanocrystalline materials have a wide spectrum of applications due to their superior properties, which differ much from those of poly- and singlecrystals. For an example, the Fe-based nano-soft magnetic material Fe<sub>73.5</sub>Cu<sub>1</sub>Nb<sub>3</sub>Si<sub>13.5</sub>B<sub>9</sub> exhibits excellent soft magnetic properties due to its ultrafine grain structure<sup>[1]</sup>. The high saturation flux density makes this alloy suitable for being used in many magnetic devices.

In order to gain better understanding of nanostructure on atomic scale, microscopic experimental techniques are needed<sup>[2]</sup>. In this paper the perturbed angular correlation (PAC) with <sup>62</sup>Zn as probe nuclei was used to microscopically investigate the nano-soft magnetic material  $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$  for the first time. PAC has been used widely to obtain microscopic information on the structural and dynamical properties of materials<sup>[3]</sup>. The possibilities for such investigations have been further expanded with the advent of most versatile radioactive nuclear beam (RNB) facilities, one of which is the on-line isotope separator (ISOL). The present experiment was performed with the <sup>62</sup>Zn nucleus as PAC probe provided by the ISOL radioactive nuclear beam (RNB) facility at China Institute of Atomic Energy (CIAE)

#### 2 Experiment Details

The nano-soft magnetic material  $Fe_{73.5}Cu_1Nb_3$ Si<sub>13.5</sub>B<sub>9</sub> was prepared from the magnetic alloy Fe<sub>73.5</sub>Cu<sub>1</sub>Nb<sub>3</sub>Si<sub>13.5</sub>B<sub>9</sub>. The alloy was melted and quench-condensed to room temperature to form Fe<sub>73.5</sub>Cu<sub>1</sub>Nb<sub>3</sub>Si<sub>13.5</sub>B<sub>9</sub> amorphous film. The amorphous film was then sintered at 823 K to become nano-soft magnetic material Fe<sub>73.5</sub>Cu<sub>1</sub>Nb<sub>3</sub>Si<sub>13.5</sub>B<sub>9</sub> with a grain size of 10 nm.

An isotope separation on-line (ISOL) has been constructed at CIAE, which is based on the HI-13

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tandem accelerator<sup>[4]</sup>. The ISOL consists of an electron beam plasma type target/ion source, an analyzing magnet with a mass resolution of better than 180, a beam diagnostic unit of tape transportation and an Einzel lens system. A PAC measurement terminal is located at the down-stream end of the ISOL. The ISOL and PAC set-up are shown in Fig. 1. <sup>62</sup>Zn probe nuclei are produced through the nuclear reaction  ${}^{63}Cu(p, 2n){}^{62}Zn$  by bombarding the Cu target located in the ion source by 23 MeV protons from the HI-13 tandem accelerator at CIAE. The energy of the produced <sup>62</sup>Zn radioactive nuclear beam is accelerated to 25 keV with an intensity of  $4 \times 10^7$  ions/s. The radioactive nuclei  $^{62}$ Zn can be used as both the positron source and the PAC probe nucleus. The decay scheme of  ${}^{62}$ Zn is shown in Fig. 2.



Fig. 1. Schematic drawing of the ISOL facility.

 $^{62}$ Zn decays into the excited states of 596.65 keV and 507.5 keV in its daughter nucleus  $^{62}$ Cu as shown in Fig.2. From these excited states the 596.65 keV plus 507.5 keV and 40.9 keV cascade  $\gamma - \gamma$  rays are emitted via a spin I=2 intermediate state with a halflife of 4.8 ns. The perturbed angular correlation was performed on this  $\gamma - \gamma$  ray cascade. The emitted  $\gamma_1$ (596.65 keV and 507.5 keV) is detected by a "start" detector, which defines the time t=0 when the nucleus decays to the intermediate excited state and most importantly selects a set of nuclei having a higher probability of emitting  $\gamma_1$  and a alignment direction of the angular momentum I. The intermediate excited state is de-excited to the ground state by the emission of  $\gamma_2$  (40.9 keV) detected by a "stop" detector. The detection of  $\gamma_2$  determines the spin precession.



Fig. 2. Decay scheme of <sup>62</sup>Zn.

Delayed coincidence time spectrum of  $\gamma_1 - \gamma_2$  is given by

$$I(\theta, t) = I_0 e^{-t/\tau} W(\theta, t) + B .$$
(1)

 $W(\theta, t)$  is the perturbed angular correlation function

$$W(\theta, t) = 1 + \sum_{k} A_k G_k(t) P_k(\cos\theta) , \qquad (2)$$

where  $A_k$  is the anisotropy coefficient,  $P_k(\cos\theta)$  is the Legendre polynomial and  $G_k(t)$  is the perturbation factor determining the time variation of the perturbed angular correlation function. In case of pure electrical quadrupole interaction the perturbation factor is given by

$$G_2(t)_{\rm ele} = \sum_n S_{2n} [f_0 + \sum_i f_i e^{-n\sigma(i)t} \cos(n\omega_{0i}t)] . \quad (3)$$

In (3) the summation runs over all perturbations,  $f_i$  the fraction associated with the  $i^{\text{th}}$  perturbation,  $f_0$  is the unperturbed fraction of the probe nuclei,  $\omega_{0i}$  the  $i^{\text{th}}$  quadrupole interaction frequency and  $\sigma_i$ the width of the  $i^{\text{th}}$  interaction frequency distribution. For spin I=2, n=0, 1, 3, 4. The quadrupole interaction frequency  $\omega_0$  determines the EFG by the quadrupole coupling constants  $\nu_{Qi} = eQV_{zzi}/h$ . The EFG is highly related to the microscope structure of material. Therefore, the PAC is an advanced technique for microscope investigation of materials structure. A detailed description of PAC can be found elsewhere<sup>[5-7]</sup>.

PAC measurements are typically carried out by using a standard set-up composed of four BaF2 scintillation detectors arranged in a planar  $90^{\circ}$ —180° geometry, yielding simultaneously 4 delayed coincidence time spectra. The time resolution of the set-up used in the experiment is 0.35 ns. In the data analysis the spin rotation function (or counting rate ratio) is usually formed from the background subtracted delayed coincidence time spectra  $I(\theta, t)$ :

$$R(t) = \frac{I(\theta_1, t) - I(\theta_2, t)}{I(\theta_1, t) + I(\theta_2, t)} , \qquad (4)$$

where  $I(\theta, t)$  are the delayed coincidence time spectra recorded at angle  $\theta(=\pm 90^{\circ} \text{ and } \pm 180^{\circ})$ . The measured spin rotation function is fitted with the analytical expressions:

$$R(t) \approx A_2 G_2(t) , \qquad (5)$$

where  $G_2(t)$  is the corresponding perturbation factor given by Eqs. (3) and (4). The fitting yields hyperfine interaction parameters  $A_2$ ,  $f_i$ ,  $\omega_{0i}$  and  $\sigma_i$ , which result in useful information of material properties.

# 3 Result and discussion

The measured and fitted spin rotation functions are shown in Fig. 3. Two quadrupole interaction frequencies  $\omega_{01}$  and  $\omega_{02}$  were gained from the fitting, indicating there are two implantation sites of  $^{62}$ Zn. The measured frequency  $\omega_{01} = 440$  Mrad/s with a distribution width  $\sigma = 0$  is almost equal to the frequency determined in the polycrystalline Fe<sub>73.5</sub>Cu<sub>1</sub>Nb<sub>3</sub>Si<sub>13.5</sub>B<sub>9</sub>. The second frequency  $\omega_{02}$  is shifted greatly to a lower value of 90 Mrad/s and broadened by a width of  $\sigma = 0.48$ . The fractions of  $\omega_{01}$ and  $\omega_{02}$  are  $f_1 = 38\%$  and  $f_2 = 62\%$ . The structure of Fe<sub>73.5</sub>Cu<sub>1</sub>Nb<sub>3</sub>Si<sub>13.5</sub>B<sub>9</sub> grain comprises the nanocrystal core and the amorphous grain boundary<sup>[7]</sup>. There-

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fore,  $f_1$  and  $f_2$  are ascribed to the fractions of lattice sites occupied by <sup>62</sup>Zn in the grain core and in the amorphous outer region of grain boundary, respectively.



Fig. 3. The measured and fitted spin rotation functions.

## 4 Summary

The quadrupole interaction has been measured in nano-soft magnetic material Fe<sub>73.5</sub>Cu<sub>1</sub>Nb<sub>3</sub>Si<sub>13.5</sub>B<sub>9</sub> by perturbed angular correlation using <sup>62</sup>Zn probe nuclei from the HI-13 tandem accelerator based ISOL at CIAE. Two quadrupole interaction frequencies  $\omega_{01} = 440$  Mrad/s with a distribution width  $\sigma = 0$ and  $\omega_{02} = 90$  Mrad/s with a width  $\sigma = 0.48$  were obtained. The fractions are 38% and 62% for  $\omega_{01}$  and  $\omega_{02}$ , respectively. The experimental results clearly indicate that 62% of the implanted <sup>62</sup>Zn nuclei occupy the grain boundary and the rest the grain core.

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