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TIME EFFECTS IN $^{57}$Fe NMR IN YFeO$_3$

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1. Introduction. — Yttrium orthoferrite YFeO$_3$ is a canted antiferromagnet with the magnetization vector directed along the « c » axis [1]. NMR in YFeO$_3$ was previously observed by a spin echo [2] and by the steady-state methods [3-5]. In the spectrum corresponding to the domain walls the resonance line is split and has two components [4, 6]. The splitting of the resonance line is connected with the dependence of the local NMR frequency on the angle $\theta$ inside the domain wall. The $\theta$ is an angle between the easy axis and magnetization in the domain wall. For the uniaxial ferromagnets the following dependence is presumed [7, 8]:

$$ \nu(\theta) = \nu_0 (1 - \alpha \sin^2 \theta). \quad (1) $$

Where the $\nu_0$ is the NMR frequency inside the domain. The theories presented in ref. [7, 8] predict the existence of two resonance lines at frequencies $\nu_{\text{max}} = \nu_0$ and $\nu_{\text{min}} = \nu_0 (1 - \alpha)$. The enhancement factor $\eta$ for the $\nu_{\text{min}}$ line is about one order of magnitude larger than $\eta$ for $\nu_{\text{max}}$ [8]. The enhancement factor depends on the position inside the domain wall in a following way: $\eta \sim \sin \theta$ [6-9]. However, the orthoferrites show another dependence of the local NMR frequency on the position inside the domain wall [5, 6]. In the approximation of two magnetic sublattices this dependence is as follows:

$$ \nu(\theta) = \nu_0 (1 - \alpha \sin^2 \theta + \xi \beta \sin 2 \theta) \quad (2) $$

where $\xi = 1$ for the one sublattice and $\xi = -1$ for the other one. In these conditions, the spectrum corresponding to the domain walls consists also of the two resonance lines at frequencies $\nu_{\text{max}}$ and $\nu_{\text{min}}$. These lines are associated with the two groups of nuclei, magnetization of which makes with the « c » axis an angle $\theta_{\text{max}}$ and $\theta_{\text{min}}$ respectively.

Nuclei, responsible for $\nu_{\text{min}}$ line are situated closer to the center of a domain wall than the nuclei giving $\nu_{\text{max}}$ line. With increasing temperature the $\alpha/\beta$ ratio increases, $\theta_{\text{max}}$ is going closer to $0^\circ$ or $180^\circ$ and $\theta_{\text{min}}$ to $90^\circ$ (Ref. [5] and Table I).

<table>
<thead>
<tr>
<th>$T(K)$</th>
<th>$\eta(f_{\text{min}}) \times 10^{-6}$</th>
<th>$\eta(f_{\text{max}}) \times 10^{-6}$</th>
<th>$\eta(f_{\text{min}})/\eta(f_{\text{max}})$</th>
<th>$\sin \theta(f_{\text{min}})/\sin \theta(f_{\text{max}})$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.8</td>
<td>4.4</td>
<td>--</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>4.2</td>
<td>0.9</td>
<td>0.6</td>
<td>1.5</td>
<td>1.3</td>
</tr>
<tr>
<td>60</td>
<td>1.7</td>
<td>1.1</td>
<td>1.5</td>
<td>--</td>
</tr>
<tr>
<td>77</td>
<td>2.9</td>
<td>2</td>
<td>1.4</td>
<td>2.4 [5]</td>
</tr>
<tr>
<td>300</td>
<td>6.3</td>
<td>1.7</td>
<td>3.7</td>
<td>4.5 [5]</td>
</tr>
</tbody>
</table>

2. Apparatus. — The oscillator used in the experiments was a modified nuvistor version of the circuit designed by Pound and Knight [10]. Gain control provides the steady value of the driving field $H_t$. The oscillator operates at low rf levels and is sensitive only to power absorbed. When the superregenerative mode of operation is required, the quenching pulses must be supplied to the oscillator.

The double modulation of the frequency was applied...
The low-frequency modulation $f_m$ (32 Hz or 320 Hz) enables the tracing of the first derivative of NMR signal. The high-frequency modulation $f_a$ (1 + 300 kHz) enables the measurement of the enhancement factor $\eta$ by a rotary saturation effect [11].

The coil with a sample is placed inside a cryostat. In the case of a spin echo spectrometer, a cryostat with a sample is placed inside a coaxial cavity tuned at the NMR frequency [12]. In order to obtain the precise tuning and smaller cavity dimensions, a variable capacity was applied. The generator of radiopulses as well as the low-noise preamplifier are coupled with the cavity by means of a loop. In order to measure precisely the spin echo amplitude, the pulse gated integrator was applied.

3. Experiment. — YFeO$_3$ samples, used in the experiments were single crystal plates 0.3 mm thick having a mass of 0.1 g. The abundance of $^{57}$Fe was natural. Some chromium impurities were found in these samples at examining them in the EDAX 707 A type arrangement. «c» axis in these plates was perpendicular to the sample surface. In the steady state technique, the NMR signal at 77 K was so large, that one single plate was enough to carry out the experiment. For spin echo investigations 20 such plates were used. In order to observe NMR in the domain walls, YFeO$_3$ crystals were located in that way, that «c» axis was parallel to the rf magnetic field $H_1$.

In these conditions, $H_1$ field causes the oscillations of the domain walls [4]. The investigations were carried out by a steady-state technique in the temperature range of 1.8 — 450 K and by spin echo method at 77 K.

In the cw experiments the width of the NMR signals was proportional to the driving field $H_1$. This fact is the evidence for the high saturation of NMR signal. Dispersive part of NMR signal is only observed. The measurements of enhancement factor $\eta$ for both lines were carried out by the rotary saturation method [11, 13]. RS effect is based on the additional resonance, that appears in the rotating frame. This resonance occurs at $H_1/2.\eta$ field under the influence of frequency modulation of rf signal [11]. RS effect causes the decrease of nuclear magnetization vector precessing around the effective field. As a result the observed dispersive signal decreases. The smallest signal is obtained when the oscillator is modulated at frequency:

$$f_a = (\gamma/2\pi)(H_1/2.\eta) \quad \text{(Fig. 1).}$$

Below 4.2 K the rapid increase of $\eta$ factor appears (Table I), simultaneously the second line disappears. The lines obtained by a superregenerative technique were much narrower allowing the precise frequency measurements. The NMR in the YFeO$_3$ was investigated until now at the temperature range of 77-400 K [4, 5]. In this paper similar investigations were performed, but at the temperatures 4.2, 60 and 450 K. The measured frequencies which correspond to these temperatures were as follows:

- 4.2 K: $\nu_{\text{min}} = 75.75$ MHz, $\nu_{\text{max}} = 76.27$ MHz
- 60 K: $\nu_{\text{min}} = 75.57$ MHz, $\nu_{\text{max}} = 76.13$ MHz
- 450 K: $\nu_{\text{min}} = 55.23$ MHz

![Fig. 1. — Rotary saturation effect in YFeO$_3$. Driving field $H_1 = 0.6$ mOe.](image1)

![Fig. 2. — The variations of NMR line shape. 1) stable state; 2) unstable state: a) cw mode; b) sr mode.](image2)
The observed signals undergo a violent change after the application of a small magnetic DC field along the $a c$ axis (Fig. 2). The new state is not stable, and after some period of time the situation is the same as before the field application. Now, if the field is switched off after the signal amplitudes are established, once more the unstable state is achieved. By a spin echo method in 77 K a weak, broad line was observed. The measurements of spin echo amplitude were carried out as a function of the driving field $H_1$ for both states (Fig. 3a). Taking into account the differences in the relaxation times $T_2$ it appears, that the spin echo amplitude does not change practically (Fig. 3b). These effects do not depend on the magnitude of the applied field. Apart from NMR investigations, the influence of external field on the imaginary part of susceptibility $\chi''$ was examined. The measurements were performed by observing the changes of rf signal amplitude on the output of the oscillator. In this experiment the rf signal amplitude was not stabilized. In the unstable state the susceptibility $\chi''$ increases. All the effects described above, disappear with the decrease of temperature. At 60 K these effects were not observable. The recovery time decreases with the increase of temperature. At 77 K the NMR signal amplitude as well as the $\chi''$ is stabilized after the time of about 0.5 h.

4. Discussion. — Regarding the fact, that time effects do not depend on the magnitude of the applied DC field, one can assume, that they are caused only by the changes of domain wall positions. Immediately after the change of domain wall position the change of its dynamics properties occurs. Since the intensity of the first line increases more than 4 times and its width about two times, one can associate this with the fact, that $\eta$ factor is twice as large as before. For the second line, $\eta$ increases only 1.4 times (Fig. 2).

On the other hand in spin echo experiments in the unstable state the optimum value of driving field $H_1$ is 3 dB lower (1.4 times). The spin echo signal can be only connected with the second line. A similar situation occurs for CrBr$_3$ [8]. A. Hiari et al. [9] obtained, that the oscillating nuclear magnetization is enhanced by the factor $A, \delta/V$ less than the driving field. ($A$ being the wall surface, $\delta$ wall thickness, $V$ sample volume). No increase of the spin echo signal indicates, that in the unstable state the volume of domain walls responsible for NMR absorption decreases.

The reasons for such a behaviour of domain walls have not been clarified so far. It seems, that this behaviour described above of domain walls can be caused by phenomenon similar to the static conversion [14].

The enhancement factors (Table I) change slowly in the range of 4.2-300 K. This is connected with the change of anisotropy field. The reasons for the rapid increase of NMR signal below 4.2 K are not clear. The similar effects occur in orthoferrites in the neighbourhood of phase transitions [15]. It is probable, that chromium impurities contained in our sample can cause an effect of phase transition at very low temperature.

References