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THE MAGNETIC BEHAVIOUR OF BARIUM FERRITE PREPARED BY GLASS CRYSTALLIZATION METHOD

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Abstract. — Barium ferrite has been prepared by the glass crystallization method at annealing temperatures \( T_a \) up to 1000 °C. The investigation is concentrated on the annealing temperature region in which the magnetic M-phase dominates. In addition to the data obtained from static hysteresis loops we determined the anisotropy field from microwave measurements. One result is that three annealing temperature regions must be distinguished. In the first region \( (T_a < 600 \, ^\circ C) \) thermal agitation influences the magnetic properties. Above this temperature the crystals achieve very good magnetic properties (e.g. \( H_c = 488 \, kA/m \) at \( T_a = 800 \, ^\circ C) \). The effective anisotropy field \( H_A \) here mainly is a function of the crystal shape. With further increasing temperatures the crystals change into the multidomain state.

Introduction

The glass crystallization method for preparing chemical pure barium ferrites has been the basis of numerous publications (e.g. [1-4]) starting with Shirk and Buessem [1]. This method is attractive, because the crystal size can be very easily controlled by the annealing process.

We investigated the annealing temperature region, in which the crystallized M-phase, analysed by X-ray diffraction, was dominant. Especially stress was put on the determination of the magnetic (shape an crystal) anisotropy of the ferrites from microwave measurements. We received further magnetic data from static hysteresis loops, measured with a Foner-magnetometer \( (H_{max} = 1700 \, kA/m) \).

Preparation

We prepared the glass following the method Shirk and Buessem described in their paper. The starting constituents (s. Tab. 1) were melted 45 min at 1350 °C (air atmosphere) in a Pt crucible. Subsequently, the melt was going to be quenched rapidly between two stainless steel cylinders which rotated in opposite directions at an angular velocity of 1 rps. The surface distance between the rollers was approximately 50 \( \mu m \).

The results were vitreous glass ribbons 5 g in weight with a thickness of 100-150 \( \mu m \). Fragments of the ribbon were now annealed for 5 h. The barium ferrite crystallized during the annealing process was finally leached by a weak acetic acid from the glass matrix.

Results

The typical specific saturation magnetization \( \sigma_s \) measured on barium ferrite at room temperature is 680 A cm\(^2\)/g [5]. This value is reached for all compositions at an annealing temperature \( T_a = 800 \, ^\circ C \) (Fig. 1). The abrupt decrease of \( \sigma_s \) of mixture 4 is caused by an additionally formed paramagnetic phase. The coercive force \( H_{c4} \) (Fig. 2) follows a similar course, but it is not influenced by the paramagnetic crystals.

It is believed, that the abrupt decrease of \( H_{c4} \) at \( T_a > 800 \, ^\circ C \) is caused by a closely defined \( D_c \) (critical diameter for the transition of single- to multidomain magnetization behaviour) of the chemically pure and edge-free formed crystals. Opposed to that the low surplus barium of GKM1 broadens the distribution of the crystal size. Here single- and multidomain crystals exist simultaneously in the sample.

Table I. — Molar composition of the starting constituents. Additionally, the temperature region \( T_M \) in which the M-phase is dominant is given.

<table>
<thead>
<tr>
<th>composition</th>
<th>BaO</th>
<th>Ba(_2)O(_3)</th>
<th>Fe(_2)O(_3)</th>
<th>( T_M / ^\circ C )</th>
</tr>
</thead>
<tbody>
<tr>
<td>GKM1</td>
<td>0.339</td>
<td>0.294</td>
<td>0.367</td>
<td>700 - 900</td>
</tr>
<tr>
<td>GKM2</td>
<td>0.374</td>
<td>0.279</td>
<td>0.347</td>
<td>650 - 900</td>
</tr>
<tr>
<td>GKM3</td>
<td>0.380</td>
<td>0.272</td>
<td>0.338</td>
<td>600 - 900</td>
</tr>
<tr>
<td>GKM4</td>
<td>0.405</td>
<td>0.265</td>
<td>0.330</td>
<td>500 - 800</td>
</tr>
</tbody>
</table>

Fig. 1. — Specific saturation magnetization \( \sigma_s \) as a function of the annealing temperature \( T_a \). For clearness only the results of three compositions are plotted.
The effective anisotropy field strength $H'_A$ (Fig. 3) is determined from microwave absorption measurements (FMR) described in [6]. $H_A$ of uniaxially anisotropic ferrites is influenced by the crystal and shape anisotropy. The minimum value of $H_A$ of barium ferrite at room temperature is 973 kA/m [5]. Regarding first the samples annealed above 600 °C a correlation between the shape achieved of REM graphs and $H'_A$ is obvious. Figure 4 shows a typical REM-graph of a GKM-sample annealed at 700 °C. The ratio of the thickness to the diameter of the crystals is $\approx 1:10$. The corresponding difference of the demagnetization factors is determined to $-0.8$ [7] or $H'_A = 1049$ kA/m, in good agreement with $H'_A = 1058$ kA/m achieved by FMR for this sample. The ferrites grow at higher annealing temperatures to spherically shaped crystals ($T_a \geq 850$ °C), so that in this case $H'_A = H_A$ holds.

The crystals grown at annealing temperatures smaller than 600 °C, especially those of sample GKM4 ($T_a = 500$ °C), are very small. We explain the low anisotropy ($H'_A \leq 973$ kA/m) of these barium ferrites by thermal agitation [8-10].