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A laboratory magnetometer for express measurements of magnetic hysteresis loops

Subject and Purpose. *The development of technologies for synthesis of nanoscale magnetic materials requires new techniques for measuring magnetic properties of nanoscale magnetic materials in such a way as to provide express post-synthesis measurements of magnetic properties and exclude, in doing so, any mechanical displacements of measured specimens. Despite the fact that numerous techniques exist for studying magnetic properties of materials, the development of such magnetic nanomaterials as magnetic nanoparticles faces the need in novel measuring approaches based on standard procedures. Novel express techniques are called to gain information about how magnetic properties of magnetic materials vary over time and respond to such factors as temperature, storage conditions, stabilizing agents, exposure to an external magnetic field.*

Method and Methodology. *In this work, magnetic hysteresis loops are registered using a newly developed technique based on the method of small disturbances (by an external magnetic field) and combining standard constructions of hysterometers and vibrating-sample magnetometers.*

Results. *Magnetic hysteresis loops of a bulky ferrite (brand 1SCh4) sample and a 40 μm thick YIG film have been registered using the presented technique and compared with the results obtained by the well-known technique for measuring magnetic hysteresis loops. They are in good agreement with a margin error as low as 10%, which can be further improved by means of more precise equipment. With the presented technique, the magnetization and the coercive force of* $Fe_{0.5}Co_{0.5}Fe_2O_4$ *nanoparticles not examined yet have been determined.*

Conclusion. *The developed technique makes it possible to study magnetic materials of various compositions including nanoscale magnets. Fig. 4. Ref.: 13 items.*

Key words: *magnetometer, magnetic hysteresis loop, magnetization.*

In the 20th century, active research into the properties of magnetic materials grew together with research means. Until now, techniques based on the magnetization and coercive force extraction from magnetic hysteresis loops have evolved rather quickly. Since a tendency exists of the transition from bulky samples to nanoscale ones, it is necessary to know how the properties of magnetic materials change with decreasing size.

At the moment, there is a great variety of techniques for studying magnetic properties of materials [1–4]. Many of them are highly accurate and really functional. Yet there are such drawbacks as intricate and costly equipment, time-consuming preparations for launching the setup and lengthy measurements. Thus, SQUID-magnetometers [5, 6] offer high sensitivity but have quite a complex construction, the measurements call for cryogenic temperatures and long preparations of the samples. So, SQUID-magnetometers cannot perform express measurements. The vibrating-sample magnetometer from [7] is such that the sample in a constant magnetic field cannot be off mechanical displacements. In addition, the vibrating magnetometer requires a sufficiently uniform magnetic field. So, the known techniques are needed to be modified so that the sample be kept from mechanical impacts. The technique proposed here does not involve mechanical displacements, which greatly simplifies both measuring process and setup construction. The above-mentioned points make up the purpose of this work.

To perform express registrations of magnetic hysteresis loops of materials, a technique is needed that makes possible measuring various materials of low (~1 mg) concentration with acceptable accuracy. The novelty of the present work lies in the development of an express technique for measuring magnetic hysteresis loops using a hysteresimeter [1] combined with a vibration magnetometer [2]. The setup implementing this technique has a relatively simple structural scheme, performs express measurements and analyzes magnetic hysteresis loops of materials with the H field accuracy of at least 1 Oe.

1. Theoretical information. The proposed technique essentially consists in registering the signal $I = dB/dH$, where B is the magnetic flux density and H is the total magnetic field strength. A similar principle is observed, for example, in Electron Spin Resonance (ESR) spectroscopy method [8] which normally uses the well-known small-perturbation method [9]. An alternating magnetic field $h = h_0 \cos\omega t$ of a small magnitude (where h_0 is the amplitude of the alternating magnetic field h, $h_0 \sim 10$ Oe and ω is the modulation frequency, $\omega \sim 1$ kHz) acts as a small disturbance. Simultaneously with the alternating magnetic field h a permanent magnetic field H_0 is applied with a slow-rate sweep over the range –10…+10 kOe.

The strength of the total magnetic field applied to the sample is determined as

$$H(t) = H_0 + h(t). \qquad (1)$$

Hence, the magnetic flux density B takes the form [10]

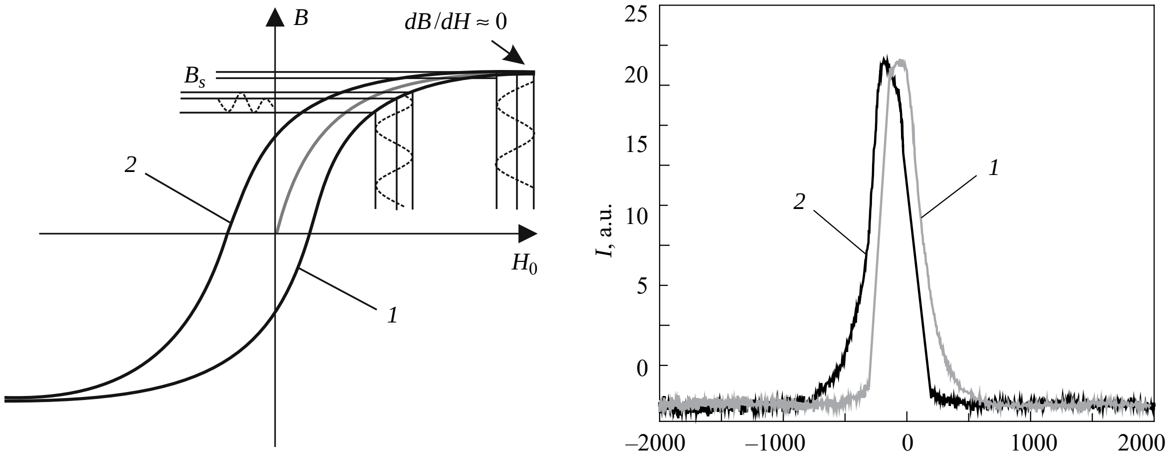
$$B(t) = B_0 + b(t), \qquad (2)$$

where $b(t)$ is the alternating part of the magnetic flux density.

To find the shape of the magnetic hysteresis loop, which is a $B(H)$ function while sweeping H_0 from left to right and then from right to left (Fig. 1, *a*), it is necessary to take integrals along corresponding curves shown in Fig. 1, *b*.

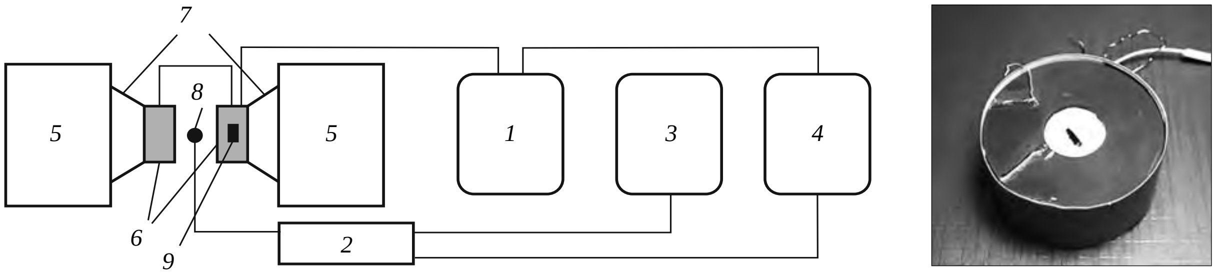
This reasoning is used in the experiments described below.

A distinctive feature of the technique we propose is that signal I as a response from the sample is registered with a magnetic field sensor. After the amplification, the signal is divided into two parts, namely, the time dependent variable h and the constant component H_0. It is important to note that we register signal I as a derivative dB/dH (see above) and do not take into account the constant component H_0. Hence, we obtain magnetic hysteresis loops with B axis, B is proportional to magnetization M. That we omit H_0 from the dependence $B = H_0 + 4\pi M$ [10] offers a clearer view of how the magnetizations of the samples differ. There is no need to omit H_0 for classic-type magnetic hysteresis loops in the form of dependence $B(H)$.



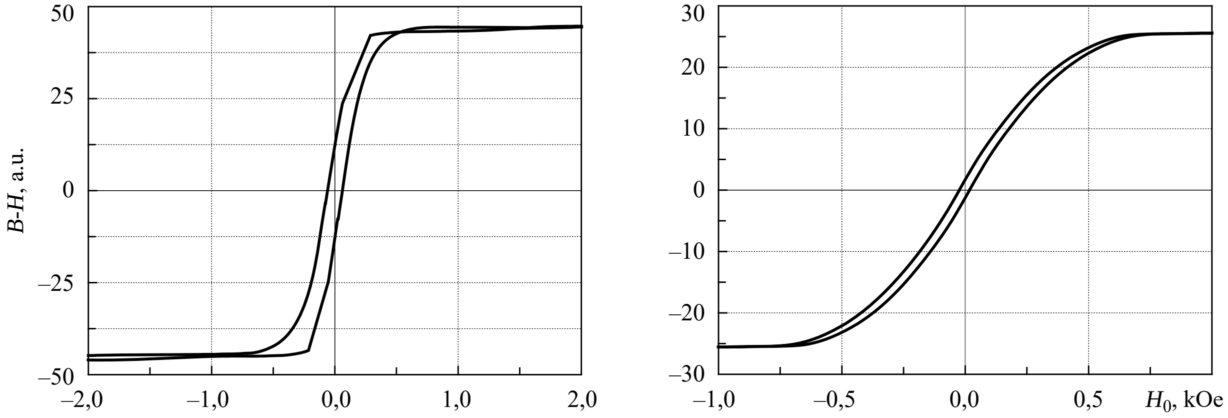
a *b*

Fig. 1. Magnetic hysteresis loop modulated with magnetic field h (*a*). Signal I received from a bulky sample of ferrite (brand 1SCh4, $M_s = 4\,800$ Gs [11]) using the developed technique (*b*). Curve *1* (*a*, *b*) corresponds to the H_0 field sweep from left to right, curve *2* (*a*, *b*) corresponds to the H_0 field sweep from right to left



a *b*

Fig. 2. Schematic block diagram of the measuring setup based on the developed technique: *1* – low-frequency generator with amplifier, *2* – signal splitter, *3* – analog-to-digital converter, *4* – synchronous amplifier, *5* – electromagnet, *6* – modulation coils, *7* – magnetic field concentrators, *8* – magnetic field sensor with amplifier, *9* – sample (*a*). Photograph of the sample placed inside one of the modulation coils (*b*)



a *b*

Fig. 3. Magnetic hysteresis loops obtained by the developed technique for bulky ferrite (brand 1SCh4) (*a*) and for a 40 μm thick YIG film (*b*)

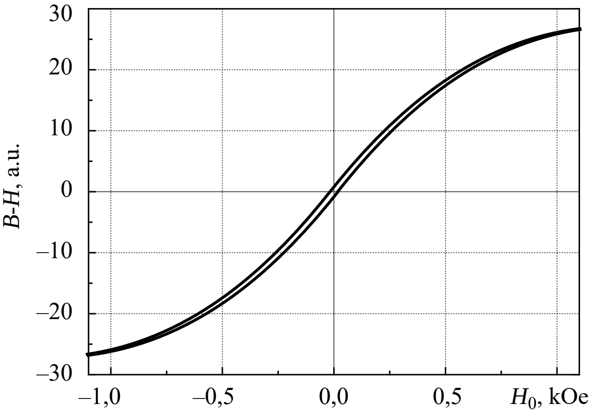


Fig. 4. Magnetic hysteresis loop of $Fe_{0.5}Co_{0.5}Fe_2O_4$ nanoparticles prepared at the synthesis temperature $T = 500$ °C [13]

According to Fig. 2, the field h is induced by two coreless modulation coils 6, and the field H_0 is induced by electromagnet 5. Magnetic field sensor 8 is placed between the coils. The opposite connection of the modulation coils provides compensation of the magnetic field created by the coils in the absence of the sample.

During the experiment, the sample is inside one of the modulation coils (Fig. 2). Moreover, the sample can be placed even in the immediate proximity to one of the coils, which enables express measurements. As a result, an imbalance of the coils appears, producing an alternating signal detected by the magnetic field sensor.

It is important to understand that for samples whose magnetization reaches the saturation point, the condition $dB/dH \to 0$ is satisfied, that is $B \equiv B_S = \text{const}$ at $H \geq H_S$ (Fig. 1), where B_S is the saturation magnetic flux density.

2. Experiment. Test studies of magnetic hysteresis loops were carried out for two magnetically soft samples with known hysteresis loop shapes, a known coercive force and a known saturation magnetization. They are a bulky ferrite (brand 1SCh4) sample of the size 4×4×0.4 mm and a 40 μm thick YIG film. The magnetic hysteresis loops shown in Fig. 3 were evaluated by integrating the dependence $I = I(H)$ (see Fig. 1, *b*).

The control measurements of the magnetic hysteresis loops of the samples were carried out using a magnetometer at the National Technical University "Kharkiv Polytechnic Institute." The analysis of the control measurements showed a good agreement with a margin error down to 10%. For the technique approbation, $Fe_{0.5}Co_{0.5}Fe_2O_4$ nanoparticles [12, 13] were experimentally studied. The magnetic hysteresis loop of this material is plotted in Fig. 4.

We have determined the saturation magnetization $M_s \sim B_s$ using the obtained magnetic hysteresis loop (Fig. 4). The measurement results are presented in [12]. Certainly, the developed technique does not claim to have a high measurement accuracy and is outperformed by expensive commercial developments. But the achieved accuracy of measurements can be significantly increased by technical improvements of particular blocks and components. The main advantage is the availability of express measurements of magnetic hysteresis loops of various substances, including nanoscale materials.

Conclusions. A novel technique for obtaining magnetic hysteresis loops has been developed providing express measurements with no mechanical shifts of the sample. It has been shown that the developed technique is appropriate for both bulky and nanoscale magnetic materials. A simplicity of the design and a block-type structure make it possible to assemble a desired accuracy magnetometer depending on customer requirements.

The measurement results obtained by the novel technique and reported in the paper have been compared with the results provided by the widely used vibrating-sample magnetometer. A good agreement with a margin error down to 10% is observed and can be further improved by the employment of more precise equipment.

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ЛАБОРАТОРНИЙ МАГНІТОМЕТР ДЛЯ ЕКСПРЕС-ВИМІРЮВАНЬ ПЕТЕЛЬ МАГНІТНОГО ГІСТЕРЕЗИСУ

Предмет і мета роботи. Розвиток технологій синтезу нанорозмірних магнітних матеріалів вимагає нових методів експрес-вимірювання їх магнітних властивостей одразу після синтезу та без їх механічного зрушення. Незважаючи на те, що нині існує багато методів вивчення магнітних властивостей матеріалів, розробка нових методик, заснованих на відомих підходах, вивчення властивостей таких магнітних наноматеріалів, як магнітні нанопорошки, все ще залишається важливим завданням. Такі експрес-методики дозволять встановити зміну магнітних властивостей магнітних матеріалів з часом і під впливом різних факторів – температури, умов зберігання, впливу зовнішнього магнітного поля та застосування стабілізуючих речовин.

Методи і методологія роботи. У роботі були зареєстровані магнітні петлі гістерезису з використанням розробленої авторами методики, заснованої на методі малих збурень (зовнішнім магнітним полем). Ця методика поєднує традиційну методику побудови петлескопа і вібраційного магнетометра.

Результати роботи. За допомогою розробленої методики отримано петлі магнітного гістерезису об'ємного фериту (марки 1SCH4) і плівки YIG товщиною 40 мкм. Порівняння результатів використання пропонованої авторами методики та широко відомої методики вимірювання магнітних петель гістерезису показало добре їх узгодження з граничною похибкою на рівні 10 %, яку можна зменшити за рахунок застосування більш точного обладнання. За допомогою розробленої методики визначено намагніченість і величину коерцитивної сили раніше не досліджуваного нанопорошку $Fe_{0,5}Co_{0,5}Fe_2O_4$.

Висновок. Розроблена методика дозволяє досліджувати магнітні матеріали різного складу, а також нанорозмірні магнетики.

Ключові слова: *магнітометр, магнітна петля гістерезису, намагнічування.*