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VERSATILE MEASURING DEVICE FOR THE STUDY OF MAGNETIC PROPERTIES OF SOLIDS

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ABSTRACT. As a zero apparatus, our measurement device is based on the Faraday method. We have made two compensation systems; mechanical and electromagnetic. It is adapted to the translational movements for insertion of the sample between the poles of the electromagnet that creates a field with an appropriate gradient of operation. To the balance can attach either a non-inductive furnace that provides sample heating up to 1900 K, or a cryostat that provides a controlled cooling temperature and a return temperature between ambient and to liquid nitrogen temperature. Susceptibility and magnetization are assessed in a relative mode; calibration using standard samples of high trust. Magnetic properties studies, versus temperature and intensity the magnetic field, on hundreds materials of thousand different metallic, vitreous and crystalline and oxidic superconductor samples, have been confirmed with similar research, demonstrating the versatility and utility of our measuring system for many years.

Keywords: *Susceptibility, magnetization, Faraday balance, magnetic field*

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1. INTRODUCTION

Experimental methods for studying the static magnetic properties of materials are based either on the Faraday interaction of a non-homogeneous magnetic field on the sample or on the induction of an electrical current in a circuit by changing the magnetic flux to remove the sample from a homogeneous field [1.2].

The analytical method and device, which ensures high sensitivity, is based on the action of a non-homogeneous magnetic field on the material samples. When the dH / dz gradient is known, it is easy to assess the magnetic moment of a sample of observed force. Resistance is usually measured by offsetting an analytical balance. That's why this type of equipment is called Faraday Balance. The Faraday magnetic interaction is evaluated from the difference between the apparent and real weight of the sample recorded with the on-off and off-on electromagnet.

More known analytical method and device that provide a high sensitivity is based on the action of a vertical non-homogeneous magnetic field upon material samples. As soon as the gradient dH/dz is known, it is easy to evaluate the magnetic moment of a sample from the observed force. The force is usually measured with an analytical balance. That's why this kind of equipment is called Faraday Balance. The magnetic part of the vertical force is evaluated from the difference in apparent sample mass with, respectively, the magnet switched on and off.

Development and use of these scales of type Foerrer-Weiss by adapting to the arm of an analytical balance scales fixed on an electromagnet has sample port vertical rods whereby the sample is placed in non-uniform field. The measurements weighing operation and the electro-mechanical design of the balance give rise to various effects which influence the data obtained. Among this influence are the repeatability, nonlinearity, sensitivity tolerance and the temperature coefficient of the sensitivity. Eccentric load can be another effect.

The magnetic balances with horizontal gradient field [3, 4, 5] have many advantages on sensitivity, adaptability and control based on measurements of magnetic field and temperature parameters test sample, thereby eliminating the short comings but also introducing other vertical ones.

To the both types of balance the interaction of scratchy field is applied to the sample in the direction of the magnetic field gradient. This has the value:

$$F_x = m\chi H \frac{dH}{dx} = m\sigma \frac{dH}{dx} \quad (1)$$

where σ, χ, and *m* are magnetization, susceptibility and mass of the sample; *dH/dx* is the gradient of magnetic field.

Magnetic properties study involves to obtaining more accurate values of the studied susceptibilities according to the required temperature and magnetic field for correlating and describing the sample-specific behaviour through different theoretical models. These studies constitute one of the fundamental problems of knowledge at the macroscopic and microscopic physical properties of materials.

2. INSTALLATION AND EXPERIMENTAL TECHNIQUE

The device operated by us has a number of facilities and features for study materials paramagnetic, fero, diamagnetic, Shun, antiferromagnetic in a broad spectrum of susceptibility and conditions which provide ground sample up to temperatures of 1900K and controlled cooling of the sample until the liquid nitrogen temperature.

In order to prevent chemical reactions in the sample space at high temperatures, this space is filled with inert gas. The disadvantage of the use of an inert atmosphere is that the whole system has to be placed in a vacuum vessel, in open connection with the sample space in the furnace.

In Figure 1 it can see the constructive and operation scheme of device and of the measuring systems. Swinging system with one degree of freedom D_1D_2 made of thin glass tube suspended by flexible and inextensible silk thread of AD_1A' and BD_2B' [6]. To left end of horizontal rod

is attached a quartz port sample cup – *E*. At right end longitudinal with the rod is glued a bar magnet *M* having mass below one gram, with a length of more than 8 times as large as in diameter.

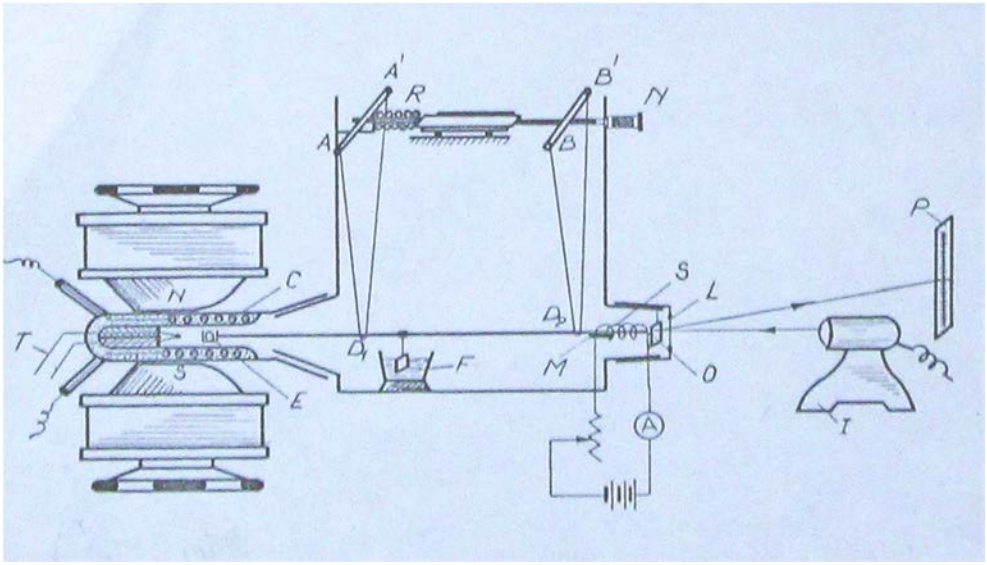

Figure 1. Principle sketch of the constructive with measuring systems of magnetic balance for study of static magnetic properties of materials.

The sample is subjected to the force F_x along the glass rod, carried on by non-homogenous field generated between of *N* - *S* poles of an Weiss type electromagnet. To enable axial centring in furnace and cryostat *C*, the suspension points of oscillation system are adjustable vertically. They can be moved by their horizontal support on a distance measurable using a micrometer *N* and a resort *R*. On the opposite rod of sample-cup is suspended a mirror, which reflect a beam of light passing through the lens it twice focusing a reticular image on the *P*.

The oscillation system behaves normally like a nearly critically damped oscillator. The damping is a viscous damping *F* in transformer oil.

After a suitable d. c. is coupling through the excitation electromagnet coils, force *Fx* of the magnetic field over sample is compensated by means of micrometer by moving the suspension system swinging up the spot returns

to zero. Thus the suspension treats form an angle θ with the vertical. I mean we have:

$$m\chi H\frac{dH}{dx} = (m_p + m)g\tan\theta \quad (2)$$

Because the mass of the system swinging, m_p, on the order of a few grams is much greater than the sample mass *m* smaller of the meters submitted to measurement the latter can be neglected. After clearing the travel *d* measured with the micrometer being very small comparatively with the physical pendulum length *l.* Vertical angular tilting of the silk yarn will be also very small; i.e.: $\theta \cong tan\theta = l/d$. With good above the clearing balance of forces is writing:

$$m\chi H\frac{dH}{dx} = mg\tan\theta = mg\frac{l}{d} \quad (3)$$

Working with the same current through the electromagnet excitation for a standard sample with known susceptibility χ_0 corresponding sizes will be m_0 and d_0 and relationship to relative measurements will be:

$$\chi = \frac{m_0}{m}\frac{d}{d_0}\chi_0 \quad (4)$$

Although the using of mechanical method is relatively simple and has a very high sensitivity, this does not ensure sufficient stability balance. Sample rising in the camp, it has other drawbacks. Such compensation forces range is too small and the precision decreases as a result of instability of zero point, the rising there retains its place in the field.

Shortfalls mentioned and versatility growth a lot and balance was done by adapting an electromagnetic compensation system [5] different from the one used by Sucksmith [6].

Our compensation system consists of a fixed coil and a horizontally mobile bar-magnet glued axially in front of coil. Compensation current *I* through the coil, is adjustable and measurable, being supplied by a galvanic or stabilized source. The fixed coil is centreline along the rod in front of which a barium ferrite magnet *N - S* moves (Figure 2).

Theoretically, we can consider [6] as a magnet-coil is assessed through an axial force of interaction between the magnetic field and the elementary element of the current *ds* spire:

$$df_c = H_c I ds \sin\alpha.$$

For an entire field this force/spire becomes

$$f_c = \int_0^{2\pi r} H_c I ds \sin\alpha = 2\pi r H_c I sin\alpha \qquad [5]$$

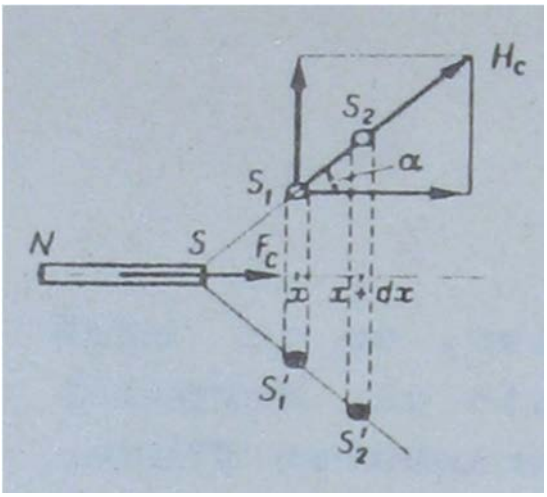

Figure 2. Axial force of interaction between a cylindrical magnet and the current through a coil

where *H*, N and α are constants for a date. Why magnetic field flow crosses two different layers in turn at a distance dx infinitesimal considered one another is:

$$\pi r^2 H = \pi(r + dx\tan\alpha)(H + \frac{\partial H}{\partial x}dx).$$

Neglecting the terms which contain small-order infinity, for small angles and the coil has a comparable section of cylindrical magnet we have:

$$\tan\alpha \cong \sin\alpha = \frac{r}{2H}\frac{\partial H}{\partial x}.$$

Value inserted in the expression [1] give the formula of interaction between spire from layer *j* and the field magnet bar:

$$f_{ij} = \pi r_{ij}^2 I \frac{\partial H}{\partial x_i}$$

Claims for coil spirals all obtain expression of the force

$$F = \sum_{i,j} \pi r_{ij}^2 I \frac{\partial H}{\partial x_i}$$

for a position because all date coefficients of I is constant we can write:

$$F = I \sum_{i,j} \pi r_{ij}^2 \frac{\partial H}{\partial x_i} = const I \qquad [6]$$

I.e. the force of interaction between the voice coil and magnet bar with the cylindrical section is proportional to the current through the coil.

For the same current through the electromagnet excitation coils in case of standard samples with known susceptibility χ_0 and a test of the unknown susceptibility χ mass m_0 respectively m, obtaining compensation currents I_0 and I the relationship for relative measurements will be:

$$\chi = \frac{m_0}{m} \frac{I}{I_0} \chi_0 \qquad (7)$$

Practically, based on formula (7), the susceptibility of the materials under study and thus of the magnetic behaviour depending on the temperature, the magnetizing field, the structure, the composition can be analyzed. For this is necessary to weight the etalon and the bulk samples and to determining their intensity of the compensating currents. We use salt Mohr etalon sample with susceptibility at room temperature $\chi_0 = 32 \cdot 10^{-6}$ emu/g.

3. MEASUREMENT METHODS AND USES

The utility of the two mechanical and electromagnetic compensation systems described in the above paragraph, and their adaptation to calibration procedures and appropriate measurement methods at very diverse thermal and magnetic conditions, highlights the versatility, reproducibility and credibility of data obtained with high- precision.

By feeding the electromagnet coils N-S from a DC source, we can obtain magnetic fields of up to 1 Tesla at a distance of 20 mm between the poles during each measurement, which takes about 10-20 seconds. The compensation coil circuit is powered from an adjustable source with currents measured by analogue or digital multimeters. The two DC, excitation and compensation circuits close or open simultaneously with a manual switch

The quartz sample holder cup can move longitudinally into space of furnace / cryostat, type test-tube, outer diameter 12 mm, inner 8 mm. The temperature is measured with thermocouples. A type Pt / Pt(10%Rh) thermocouple is installed inside the furnace space close to the sample. It is directly connected to the input of the micro voltmeter via dedicated extension wire. This thermocouple is isolated with thin alumina tubes

The heating power of 120W of this furnace set-up showed to meet our objectives well and, moreover, to be very fast. With currents up to 3 A at 20 V a temperature of 900 ^{0}C is attained within 10 minutes. After switching off the power the temperature is below 50 ^{0}C within, again, 10 minutes. The maximum temperature amounts to above 1600 ^{0}C (close to 1900 K). From time to time we do checks of the temperature readings with the Curie points of nickel and iron. In all cases, we found results that differed not more than $\pm 1^0$C. Literature values scatter between 354 and 358 ^{0}C.

Attaching the balance through a two-wire standard ground mouth of small noninductive furnace wound with wire kantal ensure heating samples at temperatures that can achieve the values of 1900K. The same kind it can be attached to a standard ground mouth circulation cryostat, in which sample will be cooled up to temperatures of approximately 93K of evaporation of liquid nitrogen. Knowing the temperature of the sample is assessed through a thermocouple T, Pt – PtRh, in small furnace, respectively Cu - Constantan -in nitrogen cryostat. Thanks of higher facility and accuracy for measurement of electromagnetic compensation scheme, the mechanic system was dropped from. The sensitivity of a balance sheet is based on the value of *HdH/dx* product which can be adjusted by changing the distance between both poles of electromagnet but also through the variation of current

excitation and the belt alignment distance between poles. Sensitivity maximum insured through the optical system of compensation is $\Delta\chi \cong 5 \cdot 10^{-9}$ e.m.u/g. This is sufficient for sample under 100 mg to put in evidence diamagnetic, paramagnetic and one-dimensional antiferromagnetic substances. The interfere field from which assured a given constant force is 2/2 mm and 9 mm in a horizontal plane. This surveying was achieved by casing the magnetic poles cap so that the magnetic field decrease in parabolic axis Ox. These characteristic of non-uniform magnetic field implies relationships:

$$F_x = m\chi H \frac{dH}{dx} \leftrightarrow F_x = \frac{1}{2} m\chi H \frac{d}{dx}(H^2) = m\chi H \frac{dH}{dx} = m\sigma \frac{dH}{dx}$$

In the case of 2 sufficiently small ferromagnetic (~10mg) samples operating under the same conditions of the magnetizing field, the unknown magnetic saturation σ will be evaluated by the relation:

$$\sigma = \frac{F}{F_0} \frac{m_0}{m} \sigma_0 = \frac{I}{I_0} \frac{m_0}{m} \sigma_0 \quad (8)$$

By this relationship, using reliable standard samples, magnetic saturation of ferromagnetic and ferrimagnetic samples can be obtained.

The calibration of the balance was carried out in two steps. In the first step, m_0 and I_0 were measured for a nickel sample which at 20 ^{0}C have the saturation σ_0=54,4emu/g, and in the second step the accuracy of the measured values was checked by studying the thermal saturation of an iron sample in a magnetic field of 14000 Oe. In figure 3, the results of our measurements are given by points and by the continuous curve dependence σ (T) for iron after Bozorth [6].

Paramagnetic susceptibility calibration was done on nickel at temperatures above the Curie point until temperatures above 1400K relative measuring with samples of about 100 mg of Mohr salt having standard susceptibility $\chi_0 = 32 \cdot 10^{-6}$emu/g. In the same figure 3 is presented *1/χ (T)* dependency graph of the magnetic harnessed (inverse of the susceptibility).

For diamagnetic samples, which are rejected by magnetic field, the susceptibility measurements compensation current requiring being repellents proportional with magnetic field. In the case of our device of quartz port

sample cup implicate a correction to each sample measuring. Otherwise the systematic errors in the susceptibility of the sample studied, which generally represents the percentage values more or less significant.

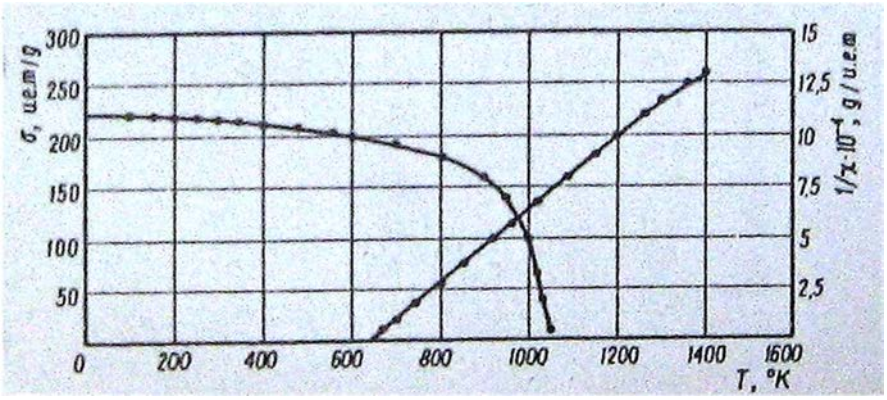

Figure 3. The balance calibration in temperature for Fe magnetization and for Ni susceptibility

The degree of confidence in measurement accuracy check if our method through repeatability compensate current through some optical feedback to spot over the landmark ruler graduated in 0 mm. Predictability of appreciation under a 1mA/mm lead we provide values that do not exceed 2%. Calculating these errors in the case of measurements of the masses and current or with the compensator class and scales can be done with the relationship:

$$\frac{\Delta\chi}{\chi} = \frac{\Delta\sigma}{\sigma} = \Delta m\left(\frac{1}{m} + \frac{1}{m_0}\right) + \Delta I\left(\frac{1}{I} + \frac{1}{m_0}\right) \quad (9)$$

In our case, errors are less than 1%. Of course, the stability of the magnetic field and the measurement of the sample temperature are particularly important to ensure the credibility of the determined values.

The type of magnetic balance also allows the magnetic field to be measured instead of the sample. This is particularly important because the use of the Hall (GaussMeter) probe is hard to locate where the sample is

positioned. Thus, will determine for a paramagnetic sample and the susceptibility χ and mass mp and for a ferromagnetic sample magnetic saturation σ and mass mf with and compensation currents Ip respectively If, the intensity of the magnetic field will be:

$$H = \frac{I_p}{I_f}\frac{m_f}{m_p}\frac{\sigma}{\chi} \tag{10}$$

The results in determining field after relation (10) are in agreement with those obtained by soak up gaussmeter with the probe.

Magnetization measurements depending on the temperature range 90-1300K are facilitated by the fact that we can attach through the same standard ground mouth a small cryostat respectively furnace for study magnetic behaviour with temperature. By maintaining the temperature at a constant value can we assign magnetization isotherms according to camp through excitation current intensity variation through the electromagnet. Determination of temperature of transition (points) Curie can be done also described within the installation. For this the temperature at which the sample of the material by heating ferromagnetic becomes paramagnetic. For the exact determination of the sample are measured in arbitrary unit's magnetization to the excitation currents as small. The values obtained are draw graph thermal variation of Curie temperature magnetization is considered the place where the tangent point of inflection of the curve cut temperature axis. The data found for Ni 631K and Fe 1043K represent reliable values for thermal studies of magnetic properties of concordant with those presented by Bates [9]. So I could continue and expand studies [10] on the Curie points of alloys of nickel with non-magnetic elements initiate by my teacher Victor Marian.

4. CONCLUSIONS

The study of magnetic properties is one of the fundamental problems of the knowledge, at the macroscopic and microscopic level, of the physical properties of the materials. Their research involves knowing as

accurately as possible the values of susceptibility of the studied sample, depending on temperature and field, necessary to correlate and describe the specific behaviour of the sample through different theoretical models. Besides the theoretical impetus, the magnetic behaviour of new and new materials is of great applicative importance in new technologies.

Our installation, which has been verified by hundreds and hundreds of studies on the magnetic behaviour of thousands of conductive, semiconductor, insulating and superconducting, metallic, oxide, crystal and glass samples, has proven its usefulness and credibility. Its versatility in the determination of static magnetic parameters as well as their dependence on variable external factors, allow decisive correlations with the dynamic studies of magnetic structures at microscopic and atomic level.

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