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SCANNING PERMEABILITY VERSUS TEMPERATURE CURVES THROUGH IMPEDANCE MEASUREMENTS

A. González Arias

DPTO. FÍSICA APLICADA, FAC. DE FÍSICA, UNIVERSIDAD DE LA HABANA, CUBA

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The convenience of dynamic scanning, as compared with the traditional point-by-point methods, to obtain the permeability versus temperature curves is discussed, together with the use of a simple and reliable dynamic method based on impedance measurements. Dynamic scanning may provide additional indirect information, which can usually not be detected by X-ray diffraction, DTA, DSC or conventional thermomagnetometry. Some results obtained on NiZnCo ferrites are shown.

In soft polycrystalline magnetic materials, such as ferrites, an analysis of the relative initial permeability μ_i as a function of temperature may provide information about additional properties besides the Curie or Néel point and the thermal stability coefficient

$$\alpha = (1/\mu_i)\, \mathrm{d}\mu_i/\mathrm{d}T$$

Thus, $\mu_i(T)$ curves also provide indirect information about the homogeneity of the analyzed sample, or about changes in the chemical composition or cationic distribution caused by different thermal treatments while synthesis is being carried out. These changes are usually detected as a shift of the Néel temperature or as a curve misshape in the region nearby [1–4]. The conventional thermomagnetic methods, being essentially based on measurement of the specific magnetic saturation (an intrinsic parameter less sensitive to structural changes than the extrinsic permeability), can not provide this type of information [5, 6].

Traditional static "point-by-point" methods (e.g. with the use of an alternating current bridge or a Q-meter for measuring μ_i at different fixed temperatures) are time-consuming. Besides, they have a strong tendency to smooth out the curves, and small magnetic effects may very easily pass unnotices, especially if they occur in a narrow range of temperature. Further, static methods are inadequate in the regions where permeability changes rapidly, e.g. near the Néel temperature.

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On the other hand, when dynamic methods are used, the literature on the subject usually gives no details about the method itself, and topics such as the thermal homogeneity in the sample, or the accuracy of the reported results, are not discussed. However, it is always advisable to make certain of the direct reading of the permeability on the recorder scale, and not that of some signal merely proportional to it (which may be less troublesome from the technical point of view). In the latter case, the parameter α will remain unchanged, but to obtain the real $\mu_i(T)$ dependence it will be necessary to replot the whole curve, a simple change of origin not being enough.

In soft ferrite materials, $\mu_i(T)$ dynamic curves may be obtained in a simple way with the help of an impedance meter, such as the Tesla BM–507 meter. This instrument, in the frequency range of 50 Hz–500 kHz, provides absolute impedance values $|\hat{Z}|$ in the $\Omega - k\Omega$ ranges automatically, together with the phase angle φ in separate scales, also including analog outputs for recording.

Theory

The impedance of a toroidal core with a rectangular cross-section and complex permeability $\hat{\mu}$ may be written as:

$$\hat{Z} = w\hat{\mu}L_0 = (\mu' - j\mu'')wL_0 \tag{1}$$

where $w = 2\pi f$, f being the frequency and $L_0 = (1/2\pi)\mu_0 N^2 h ln(b/a)$. In this expression, μ_0 is the permeability in vacuo, N the number of coil turns, and h, b and a, the height, and outer and inner diameters of the core, respectively. Multiplying (1) by its conjugate, grouping terms and making use of the fact that, in the frequency range of practical application, $\tan\delta = \mu''/\mu' < 0.1$, the former expression leads, with excellent approximation, to

$$|\hat{Z}| = \mu' w L_0 \tag{2}$$

If the number of turns and the frequency are chosen in such a way that the applied signal is small and $wL_0 = 1$, then $\mu_i = \mu' = |\hat{Z}|$, and the small signal permeability values μ_i may be read directly on the impedance meter scale, and recorded. In the International System of Units, the condition $wL_0 = 1$ may be written as:

$$fN^2 = 10^7/4\pi h ln(b/a)$$

Experimental

Curves of $\mu_i(T)$ have been scanned on many different ferrite materials using this method, with satisfactory results. The mean diameter of the toroidal cores ranged between 10 and 20 mm, and the number of coil turns from 20 to 40. The values of the relative initial permeability of these cores, at 25°, ranged between 40 and 1000. Besides the impedance meter, the equipment includes an electromechanical temperature programmer, with several available heating rates (curves were scanned at 4 deg/min); an xy recorder, with a scale of 1 mV/cm on the temperature axis (this scale provides a resolution of 2.5 deg/mm using a chromel-alumel thermocouple); and a vertical furnace with an inner diameter of 40 mm, aluminium sample holder and metallic walls, devised in a manner to make certain of the temperature homogeneity of the sample (Fig. 1).

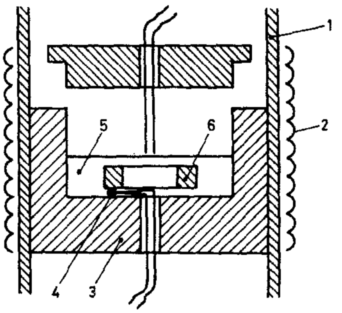

Fig. 1 Scheme of the vertical furnace. 1. Metallic wall, 2. heating wire, 3. aluminium sample holder, 4. thermocouple, 5. alumina powder, 6. toroidal core

With this same aim, the furnace has a double cover, the inner one being metallic. Covers are sectioned in halves, to facilitate the handling of the connecting coils. The heating elements were chosen in such a way that a high enough temperature may be set up with small current values, in order to make induction in the toroidal core as small as possible; the curves obtained are free of noise effects. Measurements may be carried out from below 0° by previously cooling the furnace inner block and the core sample with liquid nitrogen.

As an example of the results that can be obtained with this method, Fig. 2 shows several curves recorded on NiZnCo ferrite samples of general formula $Ni_xZn_yCo_zFe_{2+y}O_4$, with approximate molar compositions 23% NiO, 25% ZnO (samples *A* and *B*), and 32% NiO, 16% ZnO (samples *C* and *D*). The CoO content varies from 1 to 1.4% in samples *A* and *B*, and the Fe_2O_3 content from 50.5 to 51% samples in *C* and *D*, the changes in the remaining components not being significant. The synthesis procedures have been described elsewhere [7].

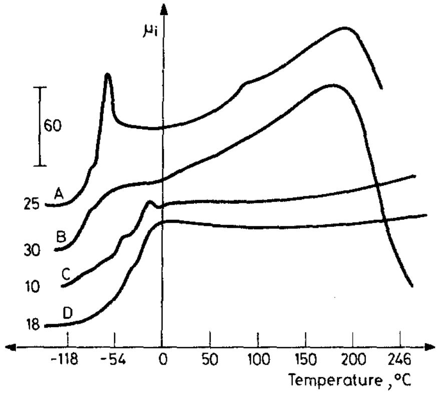

Fig. 2 Permeability curves of NiZnCo ferrites with small changes in iron or cobalt content. The minimum value of permeability for each curve appears at the left

The thermal stabilization that can be seen in curves *B* and *D*, near 0°, is a feature of the Ni—Co crystalline anisotropy compensation in this range of compositions and temperatures [4]. The misshaping of curves *A* and *C* was obtained by adding CoO in excess (0.4%), or diminishing the iron content (0.5%), respectively. The peaks observed in these curves below 0° were not identified, but suggest the presence of at least one additional cobalt-rich magnetic phase, besides the main spinel one. Structural changes caused by such small variations in composition can usually not be detected by traditional methods such as bulk sample X-ray diffraction, DTA or DSC [8, 9].

The accuracy of the method is controlled, in principle, by that of the impedance meter used. In practice, without carrying out the corrections advised by the maker to obtain the best results, permeability values were found to differ, in the worst case, by no more than 10% as compared with the results of more exact methods. However, the main advantage of the method is not based on its accuracy, but on the high resolution that can be attained. For instance, in curve *C*, the small maximum near $-54°$ represents a change of 2 units in μ_i, and covers a range of 10°. Such a small effect would be completely smoothed out when using point-by-point methods, if its occurrence were not known beforehand.

Conclusions

The measurement of $\mu_i(T)$ dynamic curves through impedance, which in principle are not so accurate as those obtained with the traditional high-precision static methods, provides much higher resolution and simplicity, using non-specialized commercial equipment. The dynamic method can detect magnetic effects related to structural changes that would commonly pass unnoticed in static methods, and also in techniques such as DRX, ATD, DSC or conventional thermomagnetometry. Because of the relatively large size of the samples, care should be taken with regard to the heating rates and the thermal homogeneity in the furnace.

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Zusammenfassung — Die Vorteile, die das dynamische Scanning im Vergleich zur traditionellen Punkt-für-Punkt-Methode zur Ermittlung der Permeabilität/Temperatur-Kurve bietet, werden zusammen mit einer auf Impedanzmessungen beruhenden, einfachen und zuverlässigen dynamischen Methode diskutiert. Dynamisches Scanning kann zusätzliche indirekte Informationen liefern, die durch Röntgendiffraktometrie, DTA, DSC order konventionelle Thermomagnetometrie nicht zu erhalten sind. Einige für NiZnCo-Ferrite erhaltene Ergebnisse werden mitgeteilt.

Резюме — Обсуждено удобство динамического сканирования по сравнению с традиционными поточечными методами получения графической зависимости проницаемости от температуры. Основой является простой и надежный метод измерения полного сопротивления. Динамическое сканирование предоставляет такую дополнительную информацию, которая, как правило, не может быть получена рентгено-диффракционным анализом, методами ДТА, ДСК или обычной термомагнетометрии. Представлены некоторые результаты исследования ферритов типа NiZnCo.