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AC HYSTERESIS LOOP TRACER FOR SOFT MAGNETIC THIN FILMS

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A high sensitive inductive AC hysteresis loop tracer for the measurements of thin films with the thickness down to few nm is described. The equipment is build entirely with commercially available instruments. The sensitivity is substantially increased by improving the signal-to-noise ratio by means of data acquisition and processing using a digital storage oscilloscope.

Keywords: hysteresis measurement, AC loop tracer, thin magnetic films

1 INTRODUCTION

There is an increasing interest in soft magnetic thin films both from the point of view of fundamental research as well as practical applications. Because of very small volume of samples special experimental techniques are required for their characterization. The basic magnetic characteristic of a ferromagnetic material is the hysteresis loop. With decreasing film thickness the sensitivities of conventional inductive techniques for the hysteresis loop measurement, however, become insufficient and the more sensitive methods have to be used. The commercial vibrating sample, magnetic force or SQUID magnetometers, which provide the suitable sensitivity, are usually combined with electromagnets or superconducting coils, the remanent or residual magnetic fields of which are inappropriate for the low-field measurements of soft magnetic materials. Very sensitive and widely used for thin films investigation is the magnetooptic Kerr effect (MOKE) technique. It, however, takes hysteresis loops from a thin surface layer (typically few tens nm in metals) but the interior of thicker films is not available by this method. The classical “induction coil technique” is therefore still important for thin films investigations. The main problem of the DC loop tracers – the zero offset – can hardly be overcome in high sensitive measurements. Solely AC loop tracers are therefore used for thin film measurements.

Various high sensitive AC loop tracers were described in the literature (see e.g. [1]-[6]). The main problems which appear at the construction of such instruments are: (*a*) field-flux compensation, (*b*) noise reduction and (*c*) signal distortion by the electronic circuits [7]. The requirements for improving (*b*) and (*c*) are, however, usually contradictory and some compromise must be found. In ref. [1-5] different equipments based on home-made electronic integrators (lamp, transistor or IC) are described. Asti et al. [6] developed a sensitive loop tracer for a wide audio-frequency range, where the need of high quality integrator was precluded by the use of numerical integration. The transmission functions of the primary (*H*-channel) and the secondary (*M*-channel) circuits were carefully calibrated and the numerical corrections of the loop shapes were done.

In this paper we describe a simple high sensitive hysteresis loop tracer which is based, with the exception of the magnetizing and pick-up coils, entirely on commercial electronic instruments available in our laboratory. Though the sensitivity is a little lower than the sensitivity of the best equipments, mentioned above, it is quite appropriate for the measurements of thin films with the thickness of few nm.

2 PHYSICAL DESIGN

The principal scheme of the equipment is shown in fig. 1. The primary circuit consists of the signal generator (Stanford Research, Model DS340 Synthesized Function Generator) the power amplifier (Kepco BOP 50-8M) and the magnetizing solenoid.

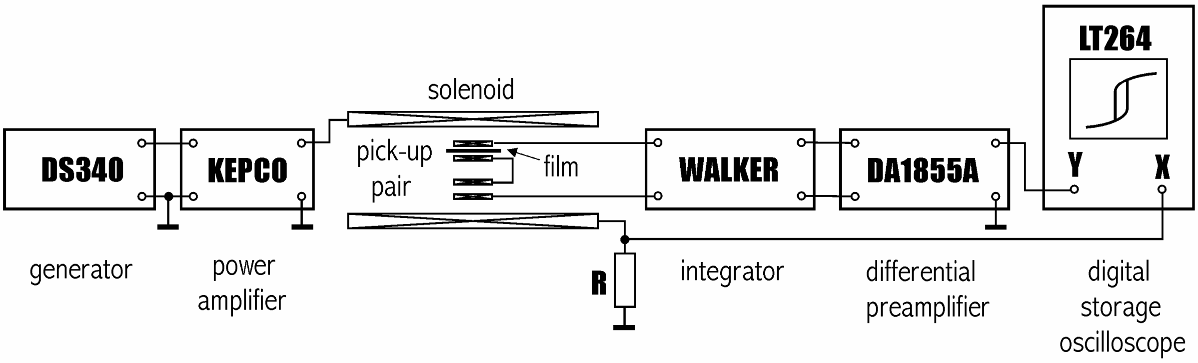

Fig. 1. Principal scheme of the equipment.

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The solenoid is wound by an enamelled and textile insulated 1 mm copper wire on a plastic tube with the outer diameter of 31 mm. The solenoid is 81 mm long and consists of 4 layers (each with 68 turns) and a pair of additional 2 layers (each with 16 turns) at both ends of the solenoid, which are used to improve the homogeneity of magnetic field near the centre. The magnitude of field is within 2 % on the length of 5 cm along the solenoid axis. The solenoid is designed to provide maximum possible magnetic field in the required volume with the inductance less than 1.5 mH, which had been proved to be acceptable for the proper functioning of KEPCO power amplifier. It is supplied by a sinusoidal current with the maximum amplitude of 5.8 $A_{p\text{-}p}$, which can provide the maximum magnetic field of about 10 kA/m for few minutes. The voltage proportional to magnetic field is taken from a low-impedance high power resistor R and is supplied to the *H*-channel of digital oscilloscope.

The secondary circuit consists of the compensated pick-up coil pair (connected in series opposition), the electronic integrator (Walker Scientific, Fluxmeter MF-3D) and the amplifier (LeCroy, DA 1855A Differential Amplifier). The integrated and amplified pick-up voltage is finally supplied to the M-channel of the oscilloscope. The pick-up pair consists of two nearly identical coils wound by 0.04 mm enamelled copper wire on a rectangular plastic coil frame with the inner and outer dimensions of 16 × 1.25 mm^2 and 19.3 × 2.2 mm^2, respectively. The length of the windings is 8 mm. Initially both coils were wound with 1510 turns and then 9 turns were removed from one coil to get the exact compensation.

The voltages proportional to magnetic field *H* and magnetic moment *M* of the sample, which is inserted in one of the pick-up coils, are supplied to two channels of the digital storage oscilloscope (LeCroy, LT264 350 MHz DSO). They can be displayed either in the standard *H(t)*, *M(t)* mode or as a hysteresis loop in the XY mode. To improve the signal-to-noise ratio in the measurements of samples with very small magnetic moment the “summed average” Math tool of the oscilloscope is utilised.

3 MEASUREMENT PROCEDURE

One of the pick-up coils is used to sense the variation of magnetic flux due to the sample. The second coil of the pair serves for the compensation of air flux. If a differential measurement is required, for example to eliminate the paramagnetic moment of the substrate, a dummy sample can be inserted in the second coil. The inner dimensions of pick-up coils allow to measure samples in the shape of ribbons, wires or thin films. The maximum dimensions of thin film substrate are 15×15×1 mm. For the investigation of in-plane magnetic anisotropy a rotatable sample holder can be used, which allows the measurement of samples with maximum dimensions of 10×10×0.5 mm.

The shape and frequency of *H(t)* signal can be changed in a wide range. The maximum measuring frequency, of about 1 kHz, is limited by the transmission function of the fluxmeter MF-3D, which works to about 20 kHz. To minimize the hysteresis loop distortions, which can originate from amplitude errors and phase shifts [7], all the passband filters in the primary and the secondary circuits are disabled, with the exception of 1MHz (18 dB/octave) bandwidth limit filter of the amplifier DA 1855A, which is used to reduce the high frequency noise in the *M*-channel. The main source of noise is the 50 Hz hum of the fluxmeter. It reaches about 3 $mV_{p\text{-}p}$ at the fluxmeter output and completely overlays weak pick-up signals.

To eliminate both the random and periodic noise the capabilities of data acquisition and processing in the digital storage oscilloscope are utilised. The 50 Hz hum can be effectively suppressed in the following way. The measuring frequency is chosen slightly above a multiple of hum frequency (for example, 101 Hz). The time base of the oscilloscope, which is triggered by a signal synchronous with the magnetizing current, is set to display approximately 1 cycle. Then the hum signal moves over the screen with the horizontal speed about 1% of scale per cycle. If the signal is averaged over multiples of 100 cycles the hum is effectively eliminated. This procedure also suppresses the random noise. Oscilloscope LeCroy LT264 allows the signal to be averaged over 4000 cycles, which can theoretically improve the signal-to-noise ratio about 63 times. The averaged signals stored in the scope memory are saved to a diskette and transferred to PC for further evaluation.

4 EXAMPLES OF MEASUREMENTS

4.1 Mumetal films

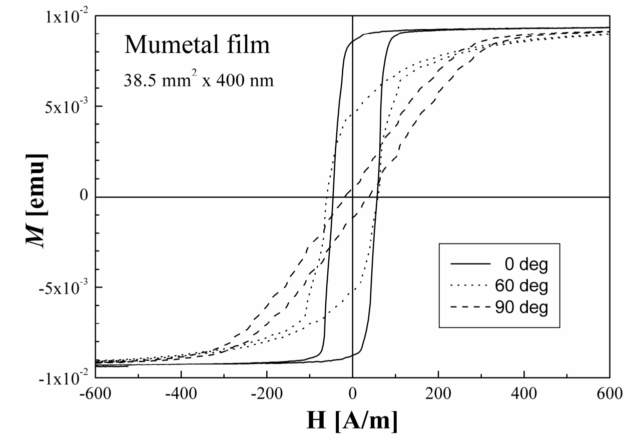

Fig.2. Hysteresis loops of 400 nm Mumetal film. Magnetic field is applied at different angles with respect to the easy axis (freq. = 101 Hz).

The equipment was tested with thin Mumetal films (NiFeCuMoMn alloy) prepared in our laboratory by the plasma jet technique [8]. Circular films with diameter of 7 mm were deposited in pure argon atmosphere on different water cooled substrates with dimensions of 10 × 10 mm^2. The hysteresis loops measured at 101 Hz on a 400 nm film on Si substrate are shown in Fig. 2. The in-plane magnetic field is applied at different angles with respect to the direction of magnetic field present during the film deposition. Such field is used for the plasma confinement near the mouth of plasma nozzle. The hysteresis loops clearly indicate the existence of uniaxial anisotropy with the easy axis along the direction of magnetic field in the vacuum chamber.

4.2 Nanocomposite Fe-Co-Al-N films

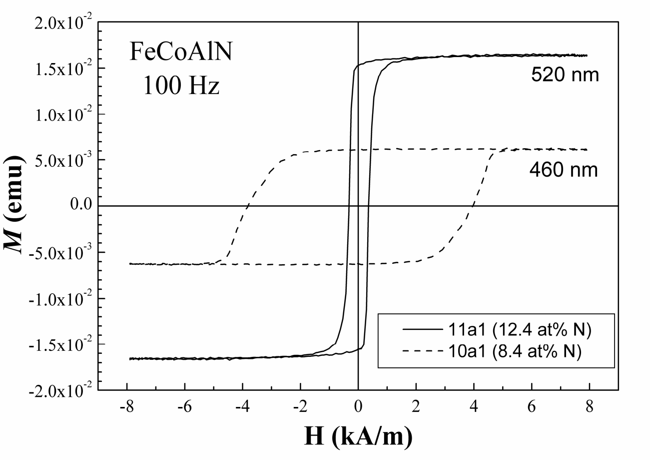

Fig. 3. Hysteresis loops of two nanocomposite FeCoAlN films.

Fig. 3 shows hysteresis loops of two nanocomposite FeCoAlN films prepared by the reactive plasma jet deposition. They consist of nanocrystalline grains of FeCo embedded in an insulating AlN matrix. They were deposited from a combined FeCo/Al nozzle in an $Ar+N_2$ gas mixture. Slightly different partial pressures of nitrogen at the deposition resulted in quite large difference of chemical composition of the two samples, namely in the nitrogen content in the films. Their magnetic properties also differ significantly. The film with lower N content shows much lower magnetization and much higher coercive field. It is probably caused by some metallic Al, which is not bounded to N and is dissolved in the ferromagnetic FeCo phase.

5 SENSITIVITY ESTIMATION

The sensitivity of equipment was tested on a very thin mumetal film prepared by plasma jet deposition. Hysteresis loop measured on the film with the thickness of about 20 nm is shown in fig. 4. The measurement was done following the procedure described in Section 3 with 500 samples/cycle and 4000 cycles averaged. As can be seen, nearly noise-free hysteresis loop was obtained. The signal-to-noise ratio (*S/N*) better than 30 was estimated from this measurement. If we accept the definition of sensitivity as the magnetic moment for which *S/N* = 1 [2], then the sensitivity of our loop tracer is better than 10^{-5} emu, which is equivalent to an iron film with the thickness of 6 nm and the area of 1 mm^2. Though this sensitivity is about 2 orders lower than that reported by Haughdal and Miller [4], it can be already compared with the sensitivities of some VSM and SQUID magnetometers.

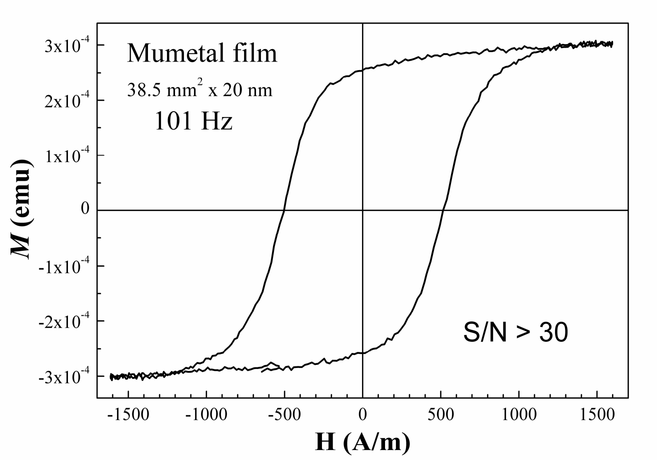

Fig.4. Hysteresis loop of a very thin Mumetal film (thickness about 20 nm) measured at frequency of 101 Hz with 500 samples/cycle, averaged over 4000 cycles.

6 CONCLUSION

We have shown that the signal processing abilities of modern digital oscilloscopes can substantially increase the sensitivity of classical induction AC hysteresis loops tracers. Using this method quite sensitive equipment can be built with commercial electronic instruments.

Acknowledgments

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