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Magnetometry **measurements**

Magnetic measurements are indispensable in characterizing the magnetic properties of materials used for various modern applications, and lead to advances in both physics and materials science

This paper aims to briefly describe some of the techniques (inductive and force-based) currently used in the characterization of important magnetic materials such as rare-earth permanent magnet, nanostructured magnetic and spin crossover materials. Advantages and disadvantages of each magnetometry technique will be discussed and typical magnetic measurement results for various magnetic materials will be presented.

Magnetic materials

Rare-earth permanent magnet materials are used in many modern devices such as electrical motors, hybrid vehicles, portable communications devices, wind turbines and other electronics. Magnets affect the size, efficiency, stability and cost of these devices and systems. The development of rare-earth permanent magnets in the 1970s profoundly influenced the application of permanent magnetic materials owing to their large energy product (BH_{max}) and increased volume efficiency.

The rare-earth permanent magnets that have been most extensively studied and developed are SmCo and NdFeB. China's rare-earth export restrictions have led to a resurgence of interest in identifying and exploiting sources of rare earths outside of China. Additionally, there is renewed interest in developing strong permanent magnet materials that do not rely as heavily on rare-earth constituents, such as nanostructured magnets that would use smaller amounts of rare-earth metals than standard magnets. Permanent magnets are generally fabricated from magnetically hard materials, since these materials possess a high saturation magnetization (M_{sat}), high remanent magnetization (M_r) after removal of a magnetizing field, and because they have high coercivities (H_c), they are therefore resistant to being easily demagnetized.[1]

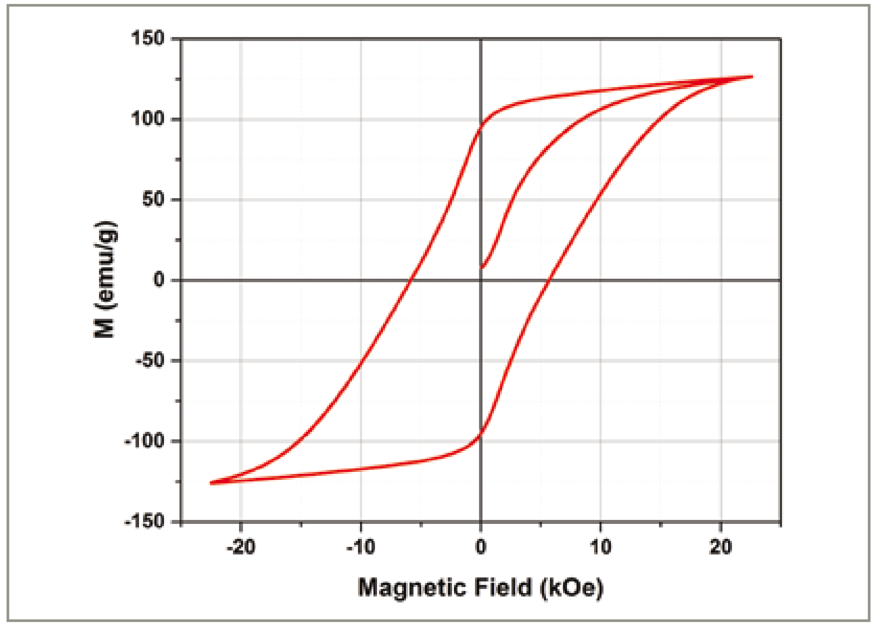


Figure 1: **The initial magnetization and major hysteresis M(H) loop for a 79mg NdFeB sample. The data is presented in terms of mass magnetization (emu/g) versus applied magnetic field (kOe)**

Magnetic nanowires, nanodots and nanoparticles are a very important class of nanostructured magnetic materials. At least one of the dimensions of these structures is in the nm range. Thus new phenomena arise in these materials due to size confinement and they are ideal candidates for important technological applications in spintronics, high-density recording media, microwave electronics, permanent magnets and for medical diagnostics and targeted drug delivery applications. In addition to these technological applications, these materials represent an experimental playground for fundamental studies of magnetic interactions and magnetization mechanisms at the nanoscale. When investigating the magnetic interactions in these materials, one of the most interesting configurations is a periodic array of magnetic nanowires, because both the size of the wires and their arrangement with respect to one another can be controlled. Inter-wire coupling is one of the most important effects in nanowire arrays because it significantly affects magnetization switching, microwave and magneto-transport properties. Experimentally, a method to investigate the strength of these interactions and a method to measure the effect of the interactions is needed. This is accomplished by using a magnetometry technique that measures and analyzes first-order-reversal-curves (FORCs).[2]

> INTER-WIRE COUPLING IS ONE OF THE MOST IMPORTANT EFFECTS IN NANOWIRE ARRAYS BECAUSE IT SIGNIFICANTLY AFFECTS MAGNETIZATION SWITCHING, MICROWAVE AND MAGNETO-TRANSPORT PROPERTIES. EXPERIMENTALLY, A METHOD TO INVESTIGATE THE STRENGTH OF THESE INTERACTIONS AND A METHOD TO MEASURE THE EFFECT OF THE INTERACTIONS IS NEEDED

Spin crossover (SCO) or spin transition materials hold a strong position in the broader field of molecular magnetism. SCO materials are among the few systems showing bistability at molecular scale, with molecular compounds that are switchable between two states in thermodynamic competition: low spin state (LS) and high spin state (HS).[3] The optical and magnetic properties as well as the molecular volumes are different for the two states, and this determines the existence of thermochromes, photochromes, piezochrome and magnetochrome properties (color change following changes of temperature, light irradiation, pressure and magnetic field).[3-6] In compounds with strong elastic interaction, the switch is often accompanied by hysteresis, a property that is essential in information storage.

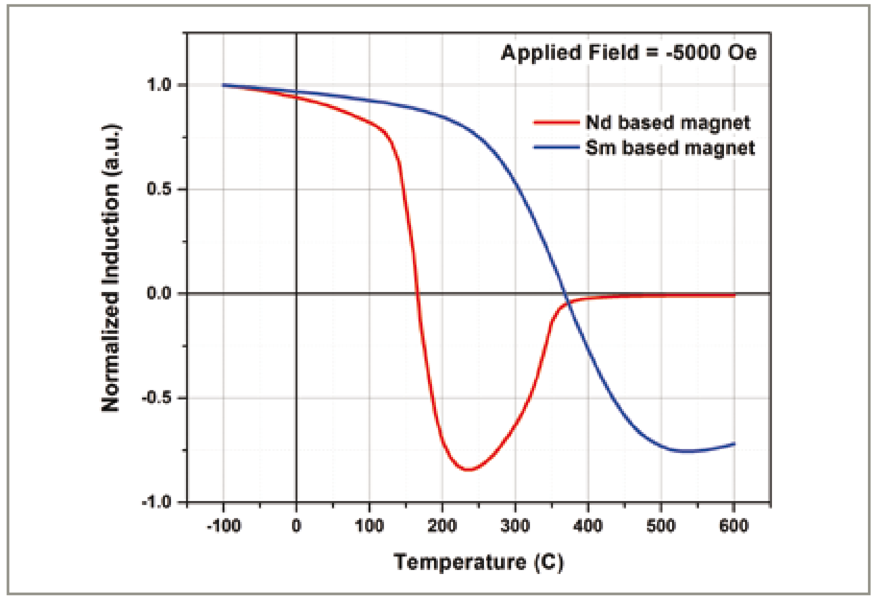
Magnetic measurement techniques

Magnetometry techniques can be broadly classified into two distinct categories: inductive and force based. Common inductive methods include vibrating sample magnetometry (VSM), extraction magnetometry, AC susceptometry and superconducting quantum interference device (SQUID) magnetometry. In the field, the two most commonly used techniques are VSM and SQUID magnetometry.

Figure 2: **The variable temperature measurements for Nd- and Sm-based magnet materials**

In the VSM method, originally developed by Foner,[7] a magnetic material is vibrated within a uniform magnetic field H, inducing an electric current in suitably placed sensing coils. The resulting voltage induced in the sensing coils is proportional to the magnetic moment of the sample. The magnetic field may be generated by an electromagnet or a superconducting magnet. Variable temperatures from cryogenic to high temperatures (<4K to 1,273K) may be achieved using cryostats and furnace assemblies.

Commercial VSM systems are available that provide measurements to field strengths of ~3 T (30,000 Oe) using conventional electromagnets,[8-10] as well as systems employing superconducting magnets to produce fields to 16 T.[11,12] When used with electromagnets, one can make very small step changes in the field (~1mOe) and the



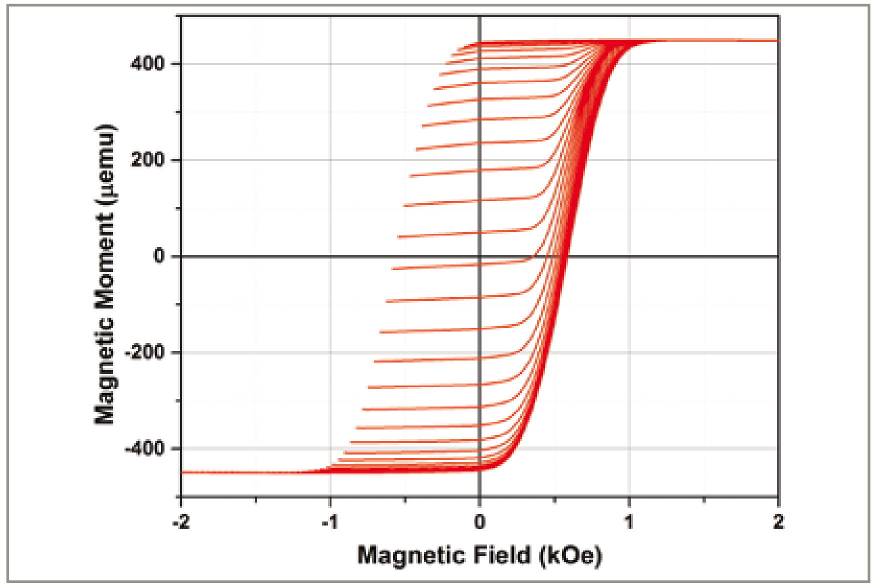


Figure 3: **Charting the first-order-reversal-curves (FORCs) for an array of magnetic nanowires**

measurement is very fast. A typical hysteresis loop measurement can take as little as a few seconds to a few minutes. When used with superconducting magnets, higher field strengths are possible; however, this limits the field setting resolution and the measurement speed is inherently slower due to the speed at which the magnetic field can be varied using superconducting magnets. The ultimate noise floor of commercially available VSMs is 10^{-7} emu. This is sufficient sensitivity for many magnetic materials. However, for materials that are magnetically diluted or that possess very small sample volumes, as is the case with nanoscale magnets, this sensitivity may be insufficient to properly characterize such materials.

Quantum mechanical effects in conjunction with superconducting detection coil circuitry are used in SQUID-based magnetometers to measure the magnetic properties of materials. Theoretically, SQUIDs are capable of achieving sensitivities of 10^{-12} emu, but practically they are limited to 10^{-8} emu, because SQUIDs also pick up environmental noise. As in a VSM, SQUIDs may be used to perform measurements from low to high temperatures (<2K to 1,000K), and to field strengths of 7 T employing superconducting magnets.[11,12] Superconducting magnets are employed in SQUIDs, so, like the superconducting magnet-based VSM systems, the measurement is inherently slow due to the speed at which the magnetic field can be varied. A typical hysteresis M(H) loop measurement can take one hour or more.

Traditional force methods, such as Gouy and Faraday balances, involve determination of the apparent change in weight for a material when it is placed in an inhomogeneous magnetic field. The sample experiences a force f along the axis of the field gradient (dH/dz) that is given by f = m(dH/dz) where m is the magnetic moment. The equipment required for such force methods is either an electro- or superconducting magnet and a balance for force measurements.

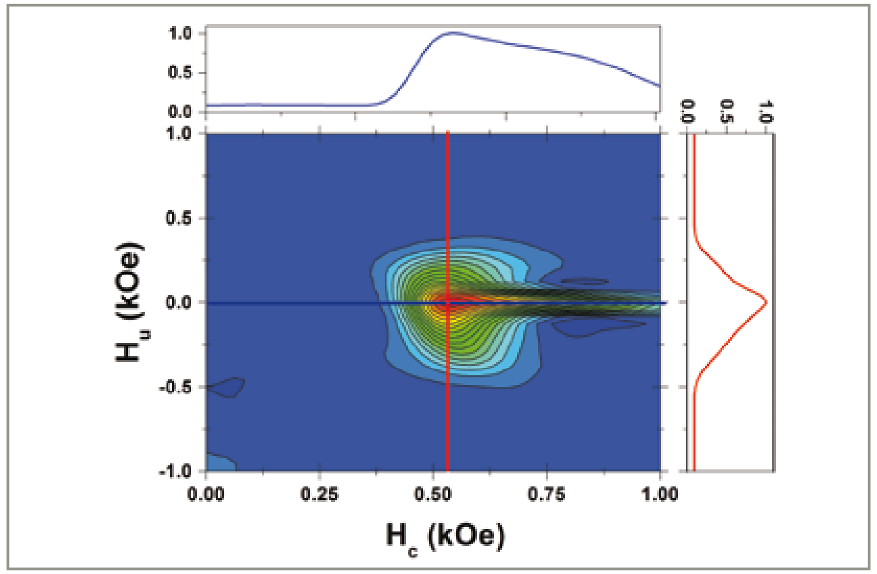


Figure 4: **The distribution of local interaction H_u and coercive H_c fields as determined from further accurate analysis of FORCs**

A commercial variant of these methods, the alternating gradient magnetometer or AGM, in essence extends on the Faraday method.[13-16] AGMs are capable of achieving sensitivities in the 10^{-8} to 10^{-9} emu range, and like the VSM, the AGM is a very fast measurement; a typical hysteresis loop takes seconds to minutes. Commercial AGM systems[9] can be used for ambient and low-temperature measurements (~10K to 300K) to the moderate 2 T to 3 T fields achievable with electromagnets.

Typical measurement results

In this part of the paper, the VSM, AGM and SQUID measurement results recorded on the previously mentioned magnetic materials are presented and discussed.

Figure 1 shows the initial magnetization curve, and the major M(H) loop for a 79mg NdFeB powder sample recorded using a VSM.[8] The most common parameters extracted from the hysteresis loop that are used to characterize the magnetic properties of permanent magnet materials include: the saturation magnetization M_{sat}, the remanence M_r, the coercivity H_c, and the energy product BH_{max}, with the maximum energy product of a magnetic material. BH_{max} is obtained from the second quadrant or demagnetization curve of the material (with regard to the chart, the upper left quadrant in Figure 1), where the curve is recorded starting at remanence M_r and ending at the coercivity H_c.

Figure 2 outlines the variable temperature VSM measurements for NdFeB and SmCo-based magnet materials recorded from -100°C to 600°C. These results show that the Nd- and Sm-based magnets have similar properties below and at room temperature. However, once the temperature rises, the Sm-based magnet retains its magnetic properties better than the Nd-based magnet and is therefore better suited to high-temperature applications. These measurements were recorded using a VSM together with a single-stage variable temperature option, providing a temperature range extending from <100K to 1,000K.[8]

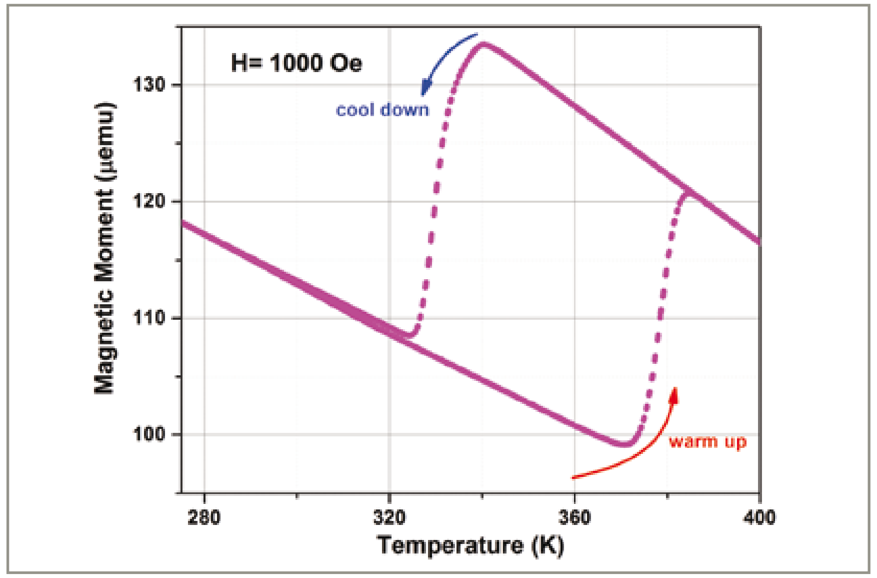


Figure 5: **The total thermal hysteresis of Fe2+-triazole-based spin crossover coordination compound as revealed by SQUID magnetometry**

Figure 3 shows a series of first-order-reversal-curves (FORCs) measured using an AGM[9] for a periodic array of Ni nanowires with a mean diameter of 70nm and an inter-pore distance of 250nm[17]. The nanowire samples were fabricated by electrodeposition using anodic aluminum oxide membrane as a template. As an example of AGM measurement speed, the FORCs consist of 4,640 points and the data was recorded in only 20 minutes.

Analysis of these FORCs yields the local interaction H_u and coercive H_c field distributions, as shown in Figure 4. This measurement protocol and analysis provides additional information regarding irreversible magnetic interactions or processes in this array of nanoscale wires, which cannot be obtained from the standard hysteresis loop measurement.

Figure 5 displays the temperature-dependent magnetic moment data of an Fe2+-triazole-based spin crossover coordination compound in nanoparticulate form,[17,18] measured using a SQUID magnetometer.[11] The particular SCO compound presents 50K thermal hysteresis with values of the transition temperatures of 380K when the measurement is performed in warming mode, and 330K when the complex is cooled down, respectively.

As a consequence, the SQUID magnetometer is well suited to measuring low magnetic moment samples in a broad temperature range spanning from below to above room temperature. However, the measurement time can be relatively long when many datapoints are required.

Conclusion

Magnetic measurements are indispensable in characterizing the magnetic properties of materials used for various modern applications. This paper has briefly described some of the current magnetic materials of technological interest, and the more common inductive and force-based magnetometry techniques currently used to characterize their magnetic properties. The advantages and disadvantages of each magnetometric technique have been discussed.

The technique that is best suited to any given application depends critically on the materials that are being measured, the parameter space (field, temperature, orientation of the field with respect to the sample) over which measurements are to be performed, as well as practical considerations (sensitivity, measurement speed). Advances in physics and materials science will continue to benefit from the application of magnetization measurements in the future. ■

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