

Microscopy with Spin-Polarized Electrons

NBS scientists combine a novel detector of spin-polarized electrons with a field-emission SEM to image the magnetic structure of surfaces

A scanning electron microscope (SEM) makes images from the secondary electrons emitted by a specimen as a focused electron beam rasters across its surface. Three years ago, physicists at the National Bureau of Standards discovered that the spin angular momenta of the low-energy secondary electrons emitted by a magnetic material retained the orientation they had prior to being ejected from the solid. Now, by combining a high-resolution SEM with a novel, compact detector of electron spin orientation, NBS scientists have converted this observation into a tool for imaging the magnetic microstructure of solid surfaces with a spatial resolution as good as that of the SEM. Instruments with field-emission electron guns should achieve a 10-nanometer resolution or better.

Magnetic microstructure refers to the local magnetization of a material. Because of electron spin and the orbital motion of the electrons around the nucleus, atoms have a magnetic moment. In most materials, the moments are randomly oriented, so there is no net magnetization, whereas magnetic materials can lower their energy by aligning the atomic moments over relatively large volumes. However, the lowest energy state is not that in which the specimen comprises a single domain of aligned atomic moments. Instead, there are several domains whose orientation is different. It is possible to make a detailed study of these domains and the walls between them with the addition of electron spin orientation analysis to the SEM.

Robert Celotta of NBS mentions two current examples of the potential usefulness of such information. One is understanding the origin of the improved properties of new permanent magnet materials, such as the neodymium-iron-boron compound that has attracted so much interest in the last 2 years (1). In unmagnetized materials, the magnetizations of the several domains cancel out. To make a magnet, one first applies a magnetic field. In the field, the walls separating neighboring domains move so that domains aligned with the field grow at the expense of those that are not. One then removes the applied field, but the domain walls relax only partially to their original positions, thereby leaving the material with a net magnetization.

For permanent magnets, one would like the domain walls to relax very little when the magnetizing field is removed, so that the magnet is as strong as possible, and to resist moving when the material is subsequently exposed to external fields, so that it does not become demagnetized. These properties depend partly on the intrinsic nature of the magnetic material and partly on features of the physical microstructure of the material, such as the presence of small particles of a second compound that may inhibit motion of the domain walls. The new instrument, which takes conventional SEM pictures as well as independent images of the magnetic microstructure, enables a direct, high-resolution comparison of the physical and magnetic structures.

The low-energy secondary electrons emitted from a magnetic material were spin-polarized even though the probing electron beam was not.

Celotta's second example deals with magnetic recording media in which binary bits are represented by domains of "up" and "down" magnetization in a thin film. Here the size of the domains is the most challenging feature at the moment. The distance between the centers of neighboring domains is now as short as 100 nanometers. Metallurgists must devise microstructures that allow domains this small to have the somewhat contradictory properties of retaining their magnetization upon removal of the magnetizing field that writes each binary bit into the recording medium, yet reversing their magnetization when a relatively weak field is applied during subsequent write operations. The high spatial resolution of the SEM equipped with electron spin orientation detectors once again could help with this task.

Researchers at NBS, as well as elsewhere, have been interested in the spin orientation of electrons scattered from solids for many years. Electron beams constitute one of the primary probes for

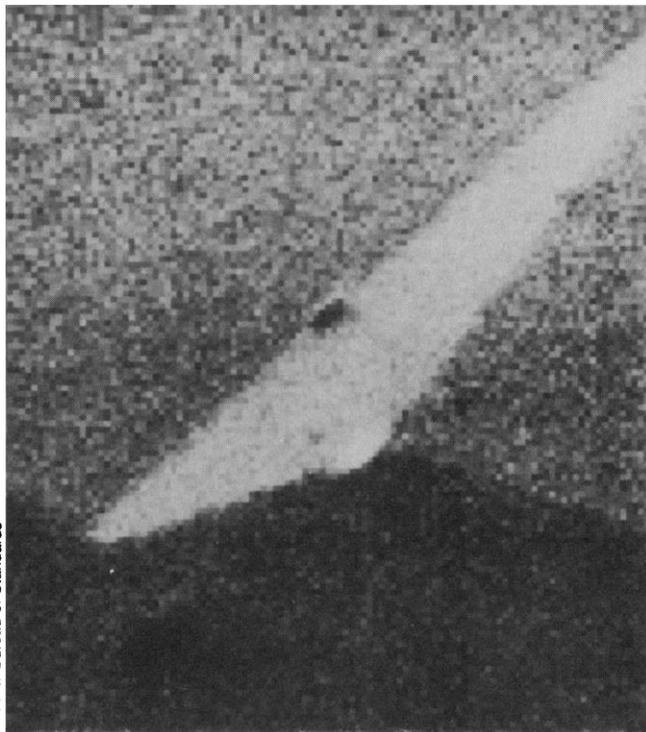
structural and spectroscopic studies of materials. If electron spin is neglected, the intensity and the velocity (speed and direction) of the scattered electrons are the only variables that can be monitored. Measurement of the spin orientation gives an extra dimension to the data and, at least in principle, the possibility of more detailed information.

One obvious way to look at spin orientation effects would be to use a so-called polarized electron beam. Polarized means that a substantial fraction of the electrons in the beam have their spin angular momentum vectors in a fixed orientation with respect to the direction of the beam. If the spins of the electrons in a specimen were oriented in a special way, as in a magnetic material, then the intensity of the scattered electrons in a particular direction might depend on the relative orientation of the spins of the electrons in the beam and in the sample. Some years ago, for example, Daniel Pierce, Celotta, and several NBS co-workers developed a high-intensity source of polarized electrons for this purpose.

The source did not lend itself to incorporation into an SEM so that spatially resolved information could be obtained, however. Part of the problem was that the magnetic lens system that focuses the beam also disrupts its polarization. Another part was that the kiloelectron-volt energy of the SEM beam so overshadows the small energy of the exchange interaction between the spins of the electrons in the beam and in the specimen that any polarization effect is washed out.

In July 1982, another approach to imaging magnetic microstructure became immediately evident when John Unguris, Pierce, Annija Galejs, and Celotta reported that the low-energy secondary electrons emitted from a magnetic material were spin-polarized, even though the probing electron beam was not (2). In this case, the polarization refers to the orientation of the magnetization of the sample; that is, the ejected electrons retained the spin orientation they had in the solid. Since the magnetic moment of an electron is in the direction opposite to the spin angular momentum, the polarization is antiparallel to the magnetization.

Secondary electrons are those



Magnetic domains

The micrograph shows a 10 micrometer by 10 micrometer area of an iron-silicon sample. Four domains are visible, two of which have the same magnetization orientation. The dark spot on the upper edge of the light domain is a surface defect. Another defect barely shows up as a white spot in the bottom right corner of the light domain, where three domains meet. These defects prevent the domain walls from moving.

knocked out of the solid by the energy they receive from collisions with the primary electrons in the beam and from collisions with other electrons excited by the beam. The bonding or valence electrons from the near-surface region of the sample are usually the ones ejected. These electrons emerge with a spectrum of kinetic energies whose peak lies at a few electron volts.

The experiment involved an iron-boron-silicon amorphous metal film. Not every valence electron in the magnetized film has the same spin orientation, but from elementary considerations one can calculate a spin polarization given by the ratio of the difference between and the sum of the numbers of electrons with spins parallel and antiparallel to the magnetization. The measured spin polarization of secondary electrons was, within experimental error, the same as that calculated for the iron-boron-silicon sample for electrons with kinetic energies of a few electron volts but decayed for higher energy electrons.

Other groups have reported similar findings, although it appears that spin-dependent scattering processes can enhance the polarization of the secondary electrons in some cases. It has also been shown by Martin Landolt and Daniele Mauri of the Eidgenössische Technische Hochschule (ETH) in Zürich that Auger electrons from magnetically ordered solids can be spin-polarized, leading to new opportunities for Auger spectroscopy of magnetic materials.

The first researchers to implement the idea of combining spin polarization de-

tectors with an SEM to map out the magnetic domain structure of a magnetic material were Kazuyuki Koike and Kazanobu Hayakawa of Hitachi's Central Research Laboratory in Tokyo (3). These investigators used a conventional SEM without a field-emission source and were limited to a rather low spatial resolution of 10 micrometers in their experiments with cobalt and iron-silicon. (Recently, the resolution has been improved to 1 micrometer.) Nonetheless, they were able to make quite striking images of stripe-shaped domains in both materials. Typical time to image about 1 square millimeter was 10 minutes.

To find the direction of the magnetization vector in a domain, Koike and Hayakawa rotated their sample around an axis normal to its surface. During rotation, the image contrast between the domains oscillated from a maximum positive value to zero and back again with a periodicity of 180 degrees, suggesting that the magnetization vectors in adjoining stripes were antiparallel. The angle of maximum contrast gives the direction of the magnetization.

Koike and Hayakawa used a conventional spin polarization detector, called a Mott detector. Mott detectors have three drawbacks but are widely used because there has been no good alternative. They are inefficient, recording only about 1 electron in every 1000. They also require quite high operating voltages, 100 kilovolts in the present case, with attendant safety problems. And they are bulky and hence do not lend themselves easily to the role of an add-on detector that is

simply bolted on to the vacuum chamber of an SEM.

The new spin polarization detector devised at NBS by Unguris, Gary Hembree, Celotta, and Pierce solves two of these problems. The one it does not help with is efficiency, which remains about the same. However, it does operate at low voltages (150 volts) and it is compact (the size of a fist). The principle of the detector is that the intermediate energy electrons scatter asymmetrically off the surface of a polycrystalline gold film according to their spin orientation.

The idea would be similar to that already discussed in connection with scattering of spin-polarized electrons by a magnetic material except that, as gold is not magnetic, the spins of its electrons do not have any preferred orientation. It turns out, however, there is another way to obtain a spin-dependent scattering. From the point of view of the moving spin-polarized electrons, the static electric charge of the gold nuclei in the film appears as a current, which generates a magnetic field. The interaction between this motional magnetic field and the spin of the electrons causes a polarized electron beam to scatter with different intensities on opposite sides of the gold film. From the measured intensity difference, one can deduce the polarization.

A test system constructed at NBS comprises an SEM with a field-emission source capable of generating an electron beam as small as 10 nanometers in diameter with two orthogonal spin polarization detectors (4). Having orthogonal detectors permits mapping out all three components of the magnetization of a specimen, which need not lie in the plane of the surface. In a first experiment with an iron-silicon sample, the NBS group imaged the same striped domains as seen by Koike and Hayakawa, but with a spatial resolution of 50 nanometers. Reduction of vibrations on the specimen stage should improve the resolution to the 10-nanometers of which the SEM is capable.

All in all, there are other techniques for magnetic imaging, but the combination of the high resolution of the SEM with the ability to distinguish physical or topographic features from magnetic ones promises to make this a powerful method of studying magnetic microstructures.

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