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NEUTRON SCATTERING METHODS FOR MATERIALS SCIENCE

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ABSTRACT

This article describes some of the neutron scattering techniques which can be used to probe structures of materials on length scales which range from less than 1 Å to 1 μm. The intent is to remove the veils of secrecy and mystery which have apparently prevented the wide application of these techniques to problems in materials science.

Introduction

As a probe of materials, neutron scattering suffers from a number of important limitations: it is expensive, non-portable, and signal-limited. Quite clearly, the method should not be used unless it provides information which cannot be obtained by other means. Fortunately the technique offers some unique advantages. It is non-destructive, able to give structural information about bulk samples which cannot be penetrated by x-rays or electrons, and well-matched to the length scales which are important in the study of materials.

Although all neutron scattering experiments are conceptually similar, the inherently low flux available from neutron sources means that neutron spectrometers have to be carefully optimised for particular purposes. The same instrument cannot be used to measure residual strain, polymer conformations and surface roughness, for example. For this reason, a veritable zoo of distinctly different neutron spectrometers has been developed since the technique was first used forty years ago. Rather than try to describe each species in the zoo, I shall restrict this article to a consideration of those spectrometers which are most useful for materials science, defined in its narrowest sense. For this reason, I shall not describe spectrometers which measure neutron inelastic scattering, even though this is an area in which neutrons have made seminal contributions to solid-state physics. Readers interested in a perspective which is broader than that provided here should consult earlier review papers [1,2] and books [3,4].

The Principles of Neutron Scattering

Conceptually, neutron elastic scattering is a simple technique: a well-collimated beam of neutrons of a known wavelength, λ , is allowed to impinge on a specimen and the scattered neutrons are detected as a function of the angle, 2θ , through which their trajectory has been deviated. With this arrangement, structure on a length scale of about $\lambda/\sin\theta$ is probed. This

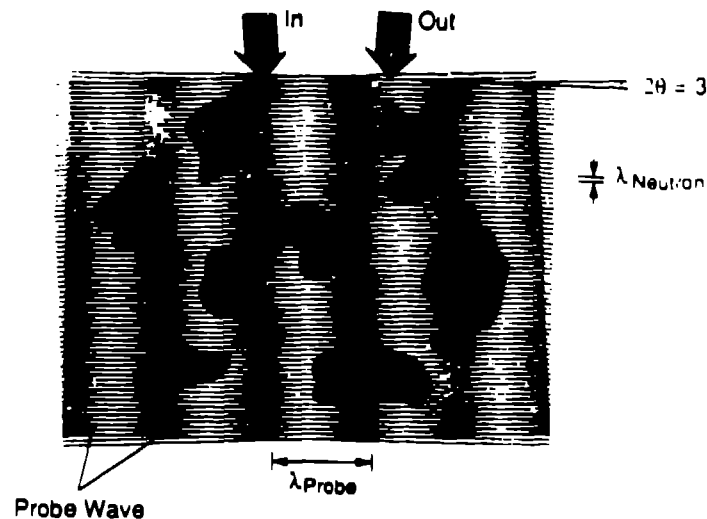


Figure 1. The probe-wave view of neutron scattering. The ingoing and outgoing neutron waves, each of wavelength λ_{neutron} , set up a probe wave of wavelength λ_{probe} . The probe wavelength decreases with increasing scattering angle, 2θ , and decreasing λ_{neutron} . The neutron wavelength and the scattering angle are chosen so that λ_{probe} is roughly equal to the length scale to be probed.

and about 20 Å, while θ is between 0.5° and 90° . Structures of materials may thus be examined routinely by neutron scattering on length scales which vary between 0.1 Å and 1000 Å. This range extends all the way from interatomic to microstructural distances.

The neutrons used in scattering experiments are obtained either from nuclear reactors or from so-called spallation sources where neutrons are produced by bombarding a heavy metal target with energetic protons [3]. The techniques used for neutron scattering experiments at these two sources differ because the neutron spectra differ radically (Figure 2). At a reactor, neutrons are produced continuously with a Maxwellian wavelength spectrum. At a spallation source, on the other hand, neutrons are produced in short ($< 100 \mu\text{sec}$) bursts, twenty to fifty times per second, with a spectrum which extends to shorter wavelengths than the reactor Maxwellian. At a reactor it is necessary to select a small band of wavelengths from the neutron spectrum in order to define the wavelength of the neutrons which impinge upon the sample. Such monochromatisation may be avoided at spallation sources by measuring the time at which each scattered neutron is detected. Since the time at which every neutron pulse starts from its source is known, the velocity of a detected neutron may be calculated from a knowledge of its time-of-flight and the distance between source and detector. The neutron velocity is directly related to its wavelength by the relation $\lambda = h/mv$, where h is Planck's constant, v the neutron velocity and m its mass.

Neutrons are used more efficiently at spallation sources than at reactors. However, the two types of source are either equivalent or complementary from the experimenter's point of view. Usually, similar data may be obtained at either source, although there are a few cases in which one or the other is superior.

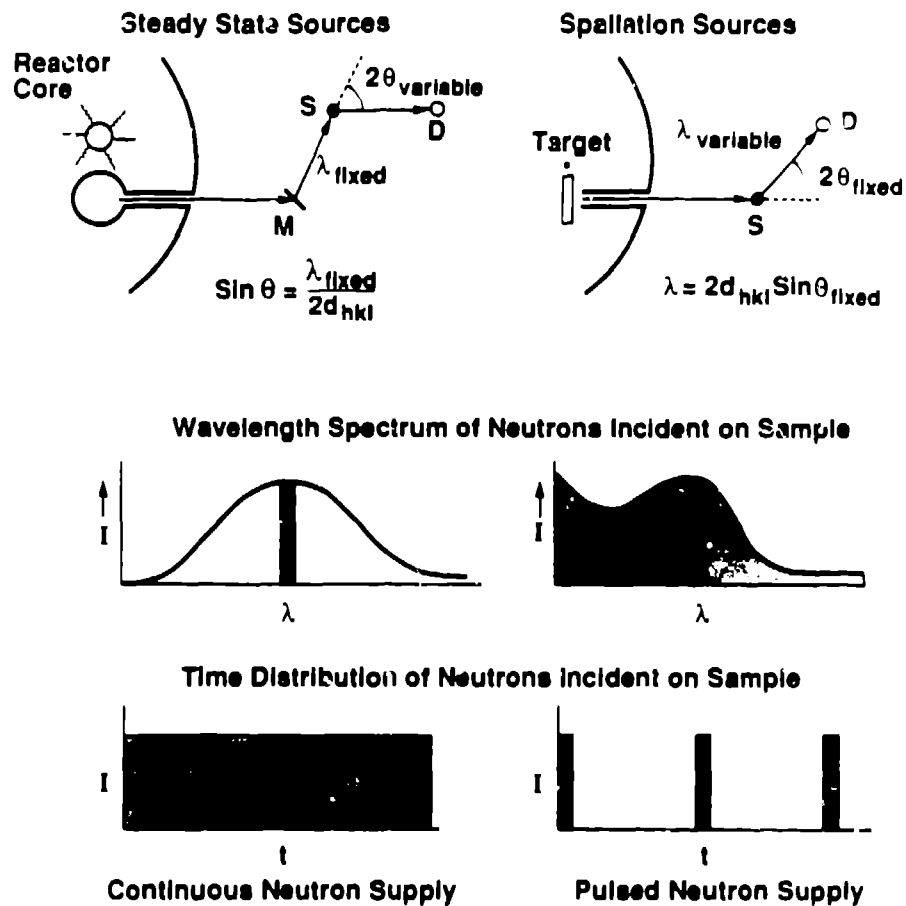


Figure 2. Reactor and spallation neutron sources are used differently for neutron scattering. The neutron spectrum from a *reactor* is continuous so beams must be monochromated (M) before impinging on the scattering sample (S) in a diffraction experiment. The scattering angle, 2θ , is scanned to obtain a diffraction pattern. The pulse structure of neutron beams from *spallation sources* allows the time-of-flight of detected neutrons (D) to be measured and used to determine neutron wavelength, the scanned variable in this case.

Small Angle Scattering

Small angle neutron scattering (or SANS) is by far the most popular neutron scattering technique in the materials community. With this method, structures on length scales between 10 \AA and 1000 \AA in materials as diverse as polymers, ceramics, metals and composites have been studied [4,6] with a distance resolution of about 10%. For such experiments, a position-sensitive neutron detector covering an area of about 1 m^2 is placed directly behind the sample at a distance between 4 m and 40 m (Figure 3). The spatial resolution of the detector is usually between 3 mm and 10 mm.

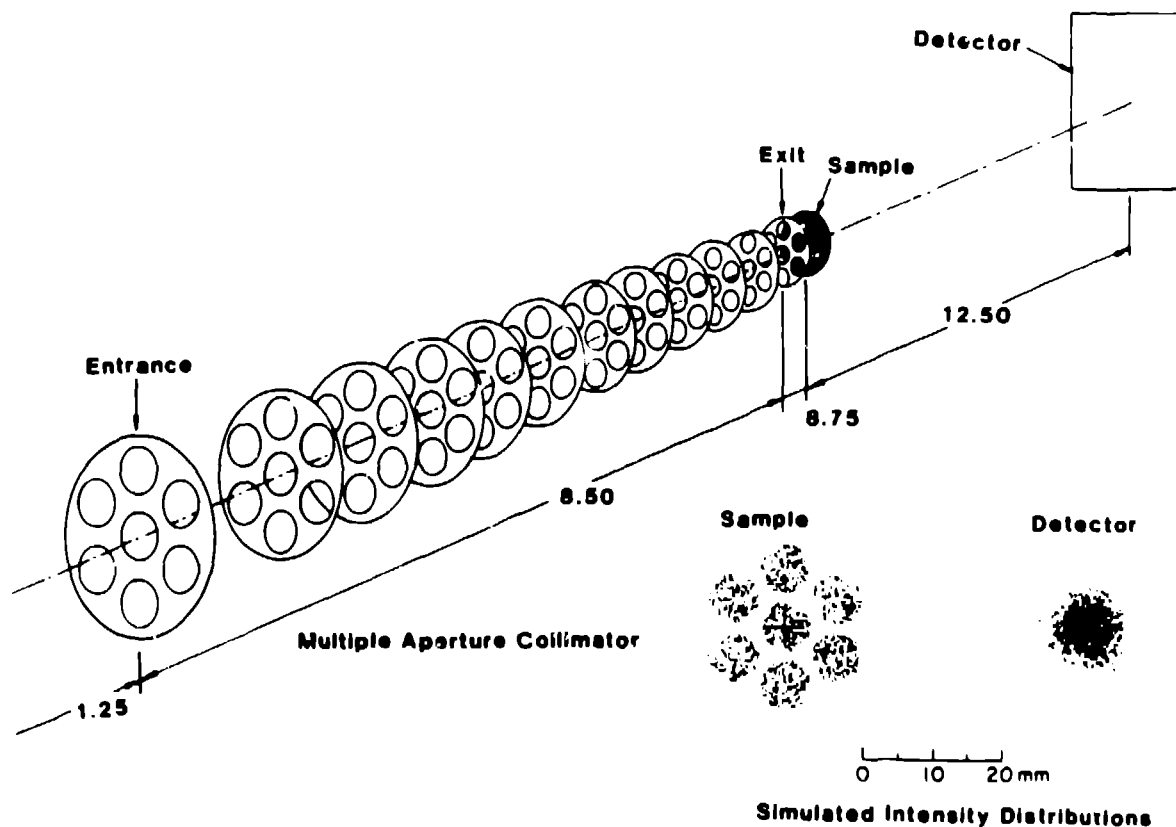


Figure 3. A schematic diagram of a SANS spectrometer. The multiple aperture "pepper-pot" collimator produces seven spots over a large area of the sample but gives only a single spot at the detector (bottom right of figure). The numbers on the sketch of the collimator are distances from the neutron source in metres.

The first SANS spectrometers were built 20 years ago in Germany and France. They proved so popular, however, that they were cloned at most neutron scattering centers, including those in the United States. In contrast to reactors, where experiments at several sample-detector separations are required to access a wide range of length scales, spallation sources provide SANS spectrometers which can probe structures between 10 \AA and 1000 \AA simultaneously. On the other hand, the better reactor spectrometers are able to measure structures larger than 1000 \AA : at the Institute Laue-Langevin in Grenoble, France, length scales of $10,000 \text{ \AA}$ or 1 \mu m can be accessed, for example. Thus the choice of instrument depends on the problem. For studies of composite materials, for example, where one is interested in both the surface structure (length scale $\sim 10 \text{ \AA}$) and the conformation (length scale $\sim 1000 \text{ \AA}$) of a filler [7], a SANS instrument at a spallation source is probably most suitable. For studies of large objects, such as heavy polymers, reactor instruments may be preferable.

Most samples of interest to materials scientists scatter neutrons strongly at small angles. For typical experiments with such materials only about 100 mm^3 of sample is

needed, generally in the form of a sheet approximately 1 mm thick. Liquid samples, such as colloids, microemulsions or polymers in solution can be contained in standard quartz cells which scatter weakly and add very little background. Measurements of strong scatterers take an hour or less at most national user facilities. To simplify studies of multiple samples, many SANS instruments are equipped with automatic sample changers which can be loaded with a dozen or more samples and whose operation is controlled by the same computer that handles data acquisition. Sample changers generally operate at room temperature and pressure, but facilities are often available which allow variable temperature and the application of modest magnetic fields for single samples. Pressure cells suitable for SANS and capable of a few tenths of a GPa and shear cells which achieve 15000 sec^{-1} are available at some facilities but are not yet common.

Although not necessary for many materials science applications, spectrometers which can accommodate weak scatterers have been constructed at the National Institute for Standards and Technology (NIST) at Gaithersburg and, more recently, at the Manuel Lujan Jr. Neutron Scattering Center (LANSCE) at Los Alamos. These instruments use converging collimators composed of successively smaller "pepper-pot lids" (Figure 3). Each aligned series of holes in the collimator plates transmits neutrons which illuminate a small area (usually about 100 mm^2) of the sample. The overall effect is to place several SANS instruments with pin-hole collimators beside one another, with each "instrument" looking at a different small area of a large (1000 mm^2) sample. With this arrangement, weak scatterers can usually be measured.

The computers which control the acquisition and reduction of SANS data are becoming increasingly sophisticated. Most simple analyses, such as radial averaging of the data to obtain the structure factor, $S(Q)$, as a function of wavevector transfer, Q , can be performed during the experiment, almost as soon as the data have been collected. Guinier plots, absolute normalisation, measurement of fractal dimension and even fitting to simple models such as scattering from monodisperse spherical particles can usually be accomplished during a typical one- or two-day experiment. More sophisticated analyses, such as the use of Maximum Entropy methods to determine particle-size distributions [8], are available at some Centers.

Powder Diffraction

Neutron powder diffraction provides a method for determining the atomic structures of polycrystalline materials. The essential difference between powder diffractometers and SANS spectrometers is that the neutron detectors on the powder machines are placed closer to the sample and cover a larger range of scattering angles. The range of scattering angles allows typical interatomic distances between 0.3 \AA and 10 \AA to be probed. Although powder diffractometers at reactors and spallation sources differ in appearance, there is very little difference in the potential performance of spectrometers at the two sources. Powder patterns

from either type of instrument may be analysed by the Rietveld technique [9], in which all of the Bragg reflections in the pattern are fit simultaneously to determine the atomic coordinates of the sample.

A very successful recent addition to techniques for the determination of atomic structures has been the use of simultaneous refinement of x-ray and neutron diffraction data [10]. Since the scattering powers of atoms for x-rays and neutrons do not vary in the same manner across the periodic table, two independent diffraction patterns are obtained when measurements are made with both types of radiation. Simultaneous refinement tends to avoid the false minima sometimes encountered when neutron or x-ray data alone are fitted and the results are usually more accurate than can be obtained with either technique on its own. Sophisticated software is required to accomplish refinement of multiple data sets, but is now available [11]. The joint-refinement technique may be extended by making measurements close to x-ray adsorption edges at synchrotron radiation facilities. Since x-ray scattering cross sections change considerably close to such edges, additional linearly independent data sets can be obtained for Rietveld refinement. Furthermore, site-specific valence of the atoms in a material can be deduced because the x-ray scattering depends on valence close to atomic adsorption edges. Measurements of this sort have been made successfully with 1-2-3 superconductors both above and below their transition temperatures [10]. Neutron diffraction measurements are an essential component for joint refinements because they define the thermal vibration parameters, which are strongly coupled to site occupancies in the refinement.

For most neutron powder diffraction experiments, about 5 cm³ of sample is adequate, although specialised spectrometers are available, albeit with lower resolution, for the investigation of milligram specimens [12]. Most neutron scattering facilities provide variable temperature capabilities on their powder diffractometers. High pressure (up to 0.3 GPa), uniaxial stress and magnetic fields are also frequently available.

Residual Strain

Powder diffractometers can be used to measure residual strain in engineering components made from metals, ceramics and composites [13]. Homogeneous strain gives rise to a shift in diffraction peaks corresponding to changes in the crystal lattice parameters. Fine slits are placed before and after the sample to define the volume within which neutron diffraction occurs and the lattice parameter is measured. This gauge volume is usually a few mm on a side but may be as small as 1 mm³. By moving the sample on an x-y translation stage between measurements, the gauge volume may be moved through the specimen and the residual strain mapped out. Measurement of residual strain places stringent requirements on the ability of a powder diffractometer to determine accurate values for the interatomic spacing corresponding to a particular Bragg peak. Most diffractometers can measure interatomic spacings with an accuracy of 0.01% ($\Delta d/d$), but a value two or three times smaller

than this is probably required for measurements of small strains, for example in ceramics. Such high resolution is achieved only by a few instruments.

Even though they are not generally able to accommodate specimens as large as their reactor counterparts, powder diffractometers at spallation sources offer a number of advantages for measurements of residual strain. The natural scanning variable is neutron wavelength rather than scattering angle, so the gauge volume remains constant during each measurement. Furthermore strains can be measured simultaneously in two mutually perpendicular directions when detectors at $+90^\circ$ and -90° scattering angles are used. Finally, diffraction from several lattice planes is recorded during each measurement, providing information on strain anisotropy and texture. For measurements of strain close to surfaces, for example in shot-peened samples or bearings, the ability to measure several sets of parallel lattice planes simultaneously is probably crucial [14] because it allows the zero of the scattering angle to be defined even when the gauge volume is not entirely within the specimen.

Texture

Measurement of texture in bulk samples have been made using powder diffractometers at many different facilities in Europe and the U.S. [15]. Diffraction patterns are recorded for several orientations of the sample and the intensities of a particular Bragg peak are used to produce a pole figure for that peak. The pole figure for a set of lattice planes is a stereographic representation of the orientation density of the normals to these planes. From a measurement of several pole figures, the full orientation distribution function of the specimen may be deduced by using the Maximum Entropy method [8] or one of its derivatives [16]. While powder diffractometers are generally suitable for measurement of texture in large (several tens of cm^3) samples, texture of small samples (typically 10 mm^3) may be obtained using single crystal diffractometers [17].

Strain Radiography

A technique for measuring the spatial distribution of strains perpendicular to a thin, plate-like sample has been proposed recently by Priesmeyer [18]. The method makes use of a white, pulsed beam of neutrons and is therefore ideally suited to spallation sources. The wavelength (or time of flight) spectrum of neutrons transmitted by a polycrystalline specimen has a sawtooth structure in which each sharp break corresponds to a Bragg edge, that is to neutrons Bragg diffracted out of the transmitted beam (Figure 4). These edges are well defined [19] and their positions can be used to calculate the lattice spacing of atomic planes which are perpendicular to the neutron beam. If a position-sensitive neutron detector is used to record the transmitted spectrum, a map of the lattice parameters, and hence of the strain, may be obtained over an area of several hundred square millimeters with a resolution of a

millimeter or so. The measured strain is an average value along the trajectory of the neutron beam so the most useful results are obtained when the strain does not vary significantly in this direction.

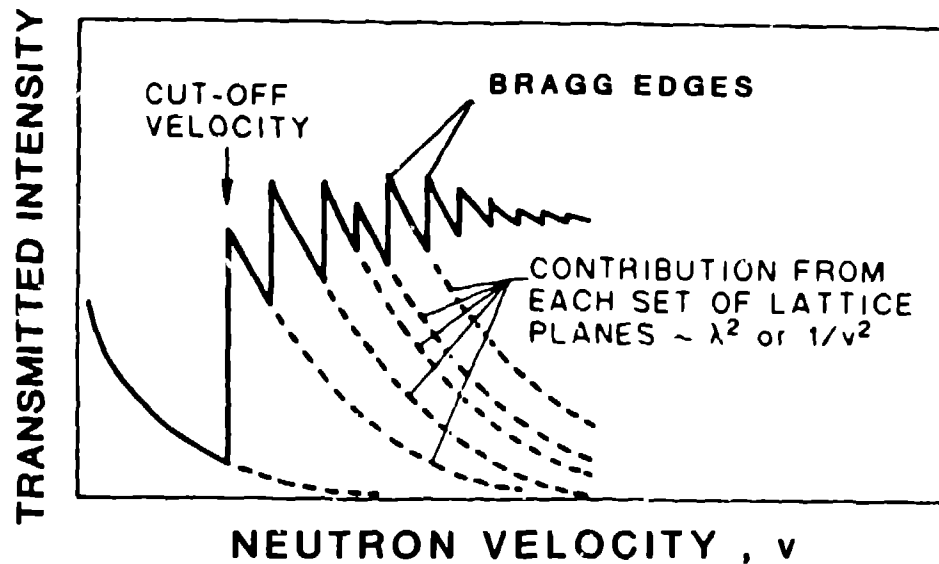


Figure 4. Neutron intensity transmitted through a polycrystalline material.

Very recently, Priesmeyer et al. [20] have shown that the transmission spectrum described above may be measured with acceptable statistical accuracy during a single pulse of neutrons from a high intensity pulsed spallation source. This development opens up the possibility of making time-dependent measurements of the distribution of lattice parameters within a sample because a snapshot of the strain can be taken every tenth second or so. Ultimately, it may be possible to probe high strain-rate processes with this method.

Investigation of Surfaces and Interfaces

One of the advantages of neutrons in the study of materials is that they penetrate bulk samples easily because they are only weakly scattered by matter. For this reason, neutron scattering is a signal-limited technique which one would not expect to be applicable to studies of interfaces. After all, there are many fewer atoms close to the interface than in the bulk of a sample and if the bulk scatters weakly, interfacial scattering ought to be negligible. This is true in most cases. However, at sufficiently small angles of incidence, neutrons are strongly reflected from almost all materials provided the interface from which they are scattered is flat and smooth. This fact allows neutrons to be used to probe the structures of materials within a 1 μm or so of their interfaces with a spatial resolution of a few Angstroms.

To do this, the specular reflectivity, $R(Q_z)$, of the surface is measured as a function of the neutron wavevector transfer, Q_z , perpendicular to the surface ($Q_z = 4\pi\sin\theta/\lambda$ where θ is angle between the neutron beam and the interface). In the simplest approximation, $R(Q_z)$ is related directly to the density gradient of a material evaluated perpendicular to its interface [21].

Reflectometers are a relatively new addition to neutron scattering facilities. The first such apparatus was built at the Intense Pulsed Neutron Source at Argonne National Laboratory less than a decade ago to study vertical surfaces [22]. Subsequently, several similar spectrometers have been constructed at other spallation sources and at reactors to examine horizontal surfaces. Such a geometry allows liquid surfaces, Langmuir films and surfactant layers at liquid-air or liquid-liquid interfaces to be examined. Neutron reflectometers are to be preferred to their x-ray counterparts for studies of hydrogenous materials, such as polymer films or surfactants, and for investigations of magnetic materials. Just as in the case of powder diffraction, it is an advantage to have both x-ray and neutron reflection data for the same surface, because this tends to prevent artifacts in the density profile deduced from the measurement.

To obtain accurate data, the incident neutron beam of a reflectometer must be well collimated. At pulsed sources, the angle between the incident beam and the surface to be studied is fixed at a value between 1° and 2° and the scan of Q_z is accomplished by recording the time-of-flight of specularly reflected neutrons. Angular collimation, which is the principal contribution to resolution, may be adjusted and is generally chosen to be less than 10% (FWHM) of the angle of incidence. At a reactor, the incident beam is monochromated as well as collimated and the scan of Q_z involves changing the angle at which neutrons impinge on the surface under investigation. For this reason, the illuminated surface (generally several tens of cm^2) changes during a scan and the data must be corrected appropriately. At a pulsed source, data for all Q_z are accumulated simultaneously, whereas, at a reactor, the Q_z scan is accomplished point by point. On the other hand, it is probably possible to make measurements close to the critical angle more accurately at a reactor source. In either implementation the factor which limits the performance of reflectometers is background. This determines the lowest value of reflectivity which can be measured (Figure 5) and, hence, the quality of the information which can be derived from the data. The best reflectometers at reactors or spallation sources are currently able to measure reliably reflectivities down to about 10^{-6} . Measurement of a complete reflection profile for a 50 cm^2 sample usually takes less than a day.

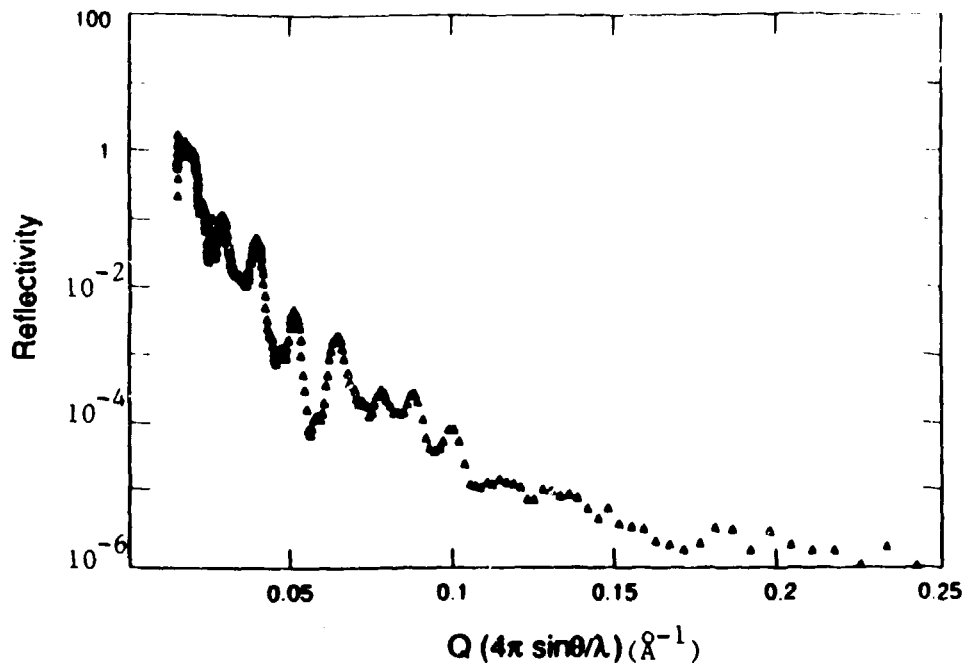


Figure 5. Typical reflectivity profile obtained on an annealed, 1500 Å thick film of block copolymer deposited on a silicon substrate provided by Tom Russell of IBM.

In addition to specular scattering, surfaces which are not perfectly smooth reflect neutrons at angles which differ from the angle of incidence. Such diffuse scattering may be used to measure the height-height correlation function of an interface. Although such measurements are not yet routine, they offer promise for the study of surface roughness on length scales between 1 Å and 50 Å [26].

If the neutron beam of a reflectometer can be spin polarised and if suitable spin flippers are available, contributions to the reflectivity caused by variations of magnetisation close to a surface may be separated from those due to variations in atomic density [22]. Polarised neutron reflectometry has been successfully used to probe magnetic recording materials and to determine the magnetic penetration depth of superconductors [23].

Radiography

Cold neutron radiography has been used widely to examine items as diverse as ancient Egyptian urns, operating helicopter engines, and the wings of fighter aircraft. Neutrons are able to penetrate substantial thicknesses of material and, if the absorption contrast is good, provide accurate images. The examples quoted above all take advantage of the fact that neutrons are strongly scattered by hydrogen and that materials containing this element can be radiographed through metallic or ceramic containers. Since the quality of thermal neutron radiography depends only on the total number of neutrons incident on the

sample, and since monochromatisation is not required, reactor neutron sources are the most appropriate for this technique.

A lesser known type of radiography makes use of the fact that most elements with atomic numbers above 25 have resonances at well defined energies in their neutron scattering cross sections [24]. At these resonances, neutrons are strongly adsorbed, so that when a radiograph is recorded as a function of neutron energy, components of the sample which contain different elements may be imaged. Resonances usually occur at energies of between 1 eV and 100 eV and have widths which are on the order of 100 meV. The high energy of most resonances coupled with the fact that time-of-flight analysis provides a natural method for obtaining energy-dependent neutron radiographs, implies that resonance radiography can only be carried out easily at spallation neutron sources. The method has applications in a variety of areas within the nuclear industry. If required, tomography could probably be developed with a spatial resolution less than 1 mm. Since the resonances in neutron cross sections broaden with temperature, it is possible to use resonance radiography for non-invasive measurements of temperature [25]. It is quite conceivable, for example, that the temperature of turbine blades in an operating jet engine could be measured by this means.

Neutron Adsorption Techniques

Two neutron adsorption techniques have been used at reactors, in particular at the National Institute for Standards and Technology (NIST), for materials testing. Neutron depth profiling allows the concentration profiles of elements such as helium, lithium and boron to be determined in the first few microns below the surface of a sample. The method relies on the detection of prompt energetic charged particles which are produced when neutrons are adsorbed by these atoms [27]. In neutron activation analysis, the energy and intensity of prompt gamma rays which result from neutron-capture are used to determine the abundance of particular elements [28]. Both of these techniques will be enhanced by the cold neutron facilities which are currently being installed at NIST.

Conclusion

Neutron scattering is a technique with a wide variety of applications in materials research. With the growth of national user facilities for neutron scattering in the United States the method has become widely available. The only limit on its application is the imagination of materials scientists.

Acknowledgement

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