ION RESONANCE AND SCATTERING TECHNIQUES FOR MEASURING SURFACE AND SUBSURFACE TOPOGRAPHY*

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ABSTRACT

Non- focused ion beams may be employed to investigate the surface profile and the shape of microscopic objects or periodic surfaces by using the known stopping powers of ions in solids. The energy spectra of the scattered or reaction ions are recorded as a function of the angles between the beam, the object and the detector, and of the energy of incident ions. The shape parameters may then be determined using computer codes. Presented also are the typical experimental results.

Keywords: Nuclear resonance reaction Ion scattering techniques Surface profile Element distribution

I . INTRODUCTION

Small objects and rough surfaces are generally observed using optical (400 nm) or electron (1 nm) microscopes[1]. Using ion induced reactions, however, it is possible to observe small objects from the energy spectrum of the emergent ions, varying the energy of the incident ion, and the angles between the beam, the object and the detector. Using a computer technique, the shape and structure of the object may be constructed, such as in tomography.

Many workers have shown that Rutherford backscattering can be used to obtain accurate depth profiling[2]. However, implanted light ions are difficult to see, although Thomas et al.[3] have employed a time- of- flight gating technique. The others have previously described how Rutherford backscattering[4,5], taken for different incident and scattered particle energies and angles, may be employed to obtain the profile for periodic surfaces and many average properties of rough surfaces.

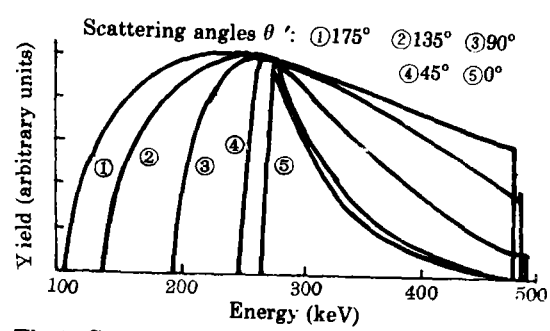

Fig.1 Spectra calculated by computer for 500 keV protons scattered by 0.2 micron diameter silver spheres

We extended a two dimensional computer program from circular fibers to small spheres. The RBS spectrum from 0.2 micron silver spheres, as a function of θ ′,

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the angle between the detector and the incident beam, obtained from our computing method is shown in Fig.1.A inherent problem with RBS surface profiling is that the depth where scattering takes place is not specifically known.

Nuclear reactions may be used to avoid this problem and obtain more exact information in specific circumstances. Computer programs for depth profiling using such techniques have been described by Simpson and Earwaker[6] for flat surface.

Ⅱ. NUCLEAR REACTIONS AND ENERGY STRAGGLING

The most useful reactions for this purpose are sharp resonant reactions in light nuclei leading to the emission of an energetic particle of unique energy. The resonance is preferably isolated in order not to be confused with those nearby as the energy increases. The ion beam handbook[2] contains a listing for many such reactions. More recent information is available in the compilations of Adjzenberg– Selove[7] and Endt[8], which are intended for nuclear rather than solid state physicists.

The maximum size of objects is determined by the range of the incident particles employed. The reaction fragment observed should be of higher energy than the incident particle in order not to be confused with RBS particles, which may be stopped by a foil. In principle, one could employ higher incident energies, but, in practice, for RBS, it is better to stay in the region below the Coulomb barrier, to avoid the complications of resonance scattering. For resonance reactions, the region a little above the Coulomb barrier is best, since at higher energies the density of resonances gets too great. For a similar reason, "magic number" nuclei are best, having widely spaced resonances.

Apart from the width of the resonance, the system resolution is a combination of the detector resolution and the ion straggling within the material. Employed for the most part of this work are silicon surface barrier detectors which, generally, have a full width at half maximum (FWHM) resolution of 12 keV for ^{237}Am alpha particles of 5.486 MeV.

A good approximation for linear straggling is given by Bohr[9]

$$\Omega_B^2 = 4\pi Z_1^2 Z_2 e^4 N \Delta R \qquad (1)$$

Ω_B is the standard deviation of the energy loss, Z_1 is the charge of the incident particle, Z_2 that of the scattering atom, N is the atomic density, and ΔR is the thickness of the material through which the particle passes. This formula has been improved by several people[10] and us[11]; the straggling is less than the Bohr value by 65% for Be and 8% for Au. For convenience we shall assume the simple Bohr value which may be corrected from the reference.

Since the detector resolution is approximately 12 keV, it is of interest to see what thickness of material will provide this value of straggling. This thickness is approximately 0.4 mg/cm^2 for protons and 0.1 mg/cm^2 for alpha particles in many

elemental targets. The thickness for 1 MeV protons to loss 12 keV is much less than this, varying from 0.05 mg/cm^2 for beryllium to 0.2 mg/cm^2 for thorium (for alpha particles, it is 0.007 to 0.025 mg/cm^2, respectively). The thickness for which the proton loss is equal to the standard deviation for straggling varies from 10^{-4} mg/cm^2 for carbon to 10^{-3} mg/cm^2 for the heavy elements, corresponding to 4.4×10^{-8} cm to 8.3×10^{-8} cm, with an energy loss of 23 to 62 eV. Such energies would require a special magnet to resolve, and would be of little use in practice, although this does show the possible capacity to reach the angstrom region of depth resolution. Since the square of the straggling is proportional to the thickness, the fractional resolution improves with size. So, for example, for alpha particles in carbon of density 2.2, we have $\Omega_B^2 = 4.2\times10^{-4}\ \Delta R$, ($6.56\times10^{-3}\ \Delta R$ for protons) where both Ω_B and ΔR are in microns. This gives us a Ω_B of 0.02μ m when ΔR is 1 μ m and 0.2μ m when ΔR is 100μ m. Hence, this technique is clearly valuable in the micron and submicron range.

III. RESONANT REACTIONS PRODUCING CHARGED PARTICLES

We take a beam of energy E_i incident on a rough surface at an angleθ and detected angle φ_o, with respect to surface. The resonance energy is E_R. The useful reaction fragment emits with energy E_F, and leaves the surface with energy E_o, as shown in Fig.2. The target is assumed to have a homogeneous distribution of the resonant material. Stopping powers of a fixed value S_i for the incident particles and S_o for the reaction product employed are assumed. The depth where the resonance occurs then traces out a path parallel to the surface, RR', at a distance l_i that equals to $(E_i - E_R)/S_i$, as shown in the figure. the distance to the surface, l_o is then $(E_F - E_o)/S_o$. If the surface is flat, the particles leaving the surface will be monoenergetic, the energies being related by

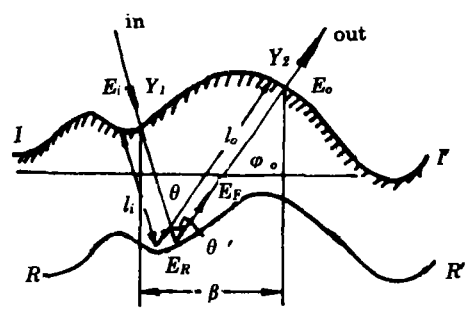

Fig.2 Diagram of the ion beam incident on a rough surface, showing the reaction region

$$(E_F - E_o)\sin\varphi_o/S_o = (E_i - E_R)\sin\theta/S_i \qquad (2)$$

with straggling superimposed. If the surface is rough, this line will spread out to a broad peak, whose width and distribution will depend on the nature of the roughness. It can easily be shown that the yield

$$Y(E_o)\,dE_o \propto dE_o/[S_o(f'(x+\beta) - f'(x))] \qquad (3)$$

where $f'(x)$ is the surface gradient where the particles are entering, $f'(x+\beta)$ is the gradient where they are leaving at an approximately fixed distance β. This gives us information about the surface corresponding to $f(x+\beta)$ and $f(x)$, specifically, the

difference in gradient.

One of the important parameters of a rough surface is the maximum height R_c of peak to valley. This can immediately be obtained from the maximum width of the energy spectrum, taken for all values of β , since $\Delta_{max}(S_0(f(\beta) - f(x))) = \Delta_{max}(E_0)$, where Δ_{max} is the largest excursion. Note this must be done for several values of β , because if the surface has the period β , it is possible to have large variations in $f(x)$ and none in E_0.

So far, we have been discussing scattering at large angles to the surface. However, the most useful spectra are those obtained when we observe particles emitted at small angles to the surface, so that they pass through more than one surface oscillation.

IV. COMPUTER SIMULATION

In the case of periodic surface, such as the one produced by machining, the nature of the periodicity is complex, and a computer simulation provides the only viable solution. We have developed such a program. Particles are assumed incident on the surface equidistantly, and a simulated particle then takes small equal steps in the incident direction within material. At each step, this particle is assumed to produce the reactant product with a probability corresponding to the reaction cross section, a target volume corresponding to the step, and atomic density of the reactant species. The reaction fragment particle is then followed to the surface in the direction of emission, using the energy of the particle emitted from the nucleus as starting energy, and the stopping power of Ziegler et al[12]. The energy and yield are filed and the process repeated, taking one more step along the incident direction. The output is energy sorted to obtain a pulse height spectrum, one more step is taken along the surface, and the whole process repeated. In this way, a complete pulse height spectrum for reaction particles from the periodic surface is obtained for whatever angle of incidence and emission. By trial and error, a surface which will produce this spectrum is found.

To confirm the theory, we obtained the energy spectrum of the alpha particles emitted from an optical grating of 13400 lines per inch, on which was evaporated a very thin layer (< 0.1 micron) of LiF, and it was bombarded by 400 keV protons. Although the ^{7}Li(p,α) reaction is non- resonant at these energies, the depth in which the reaction occurred was so thin that it could be treated as a resonant reaction, since the alpha particles emitted were very energetic ($\sim$ 9 MeV). A comparison of the experimental spectrum with a calculation on the basis of a sinusoidal surface is shown in Fig.3. It will be seen that there is reasonable agreement with a sinusoidal surface of wavelength 1.9μ m and an amplitude of oscillation 0.3 microns, however, a background extends to low energies. This is not slit scattering, since a flat LiF coated surface does not show it. It probably arises from extra deep grooves in the grating (a

depth of 0.6 microns would easily given it). A few of them could be seen under an electron microscope, either deep scratches or surface contamination. It amounts to approximately 15% of the main peak. Difficulties arose, however, because the LiF layer tended to crystallize, even though it was just a thin layer.

The resonant technique is much suitable to obtain the profile and internal structure of solid objects. However, the low yield makes it difficult to get good statistics. To take a specific example, submitting a small teflon fiber of 20μ m in diameter to a 2mm diameter proton beam and using the $^{19}F(p,\alpha\ \gamma\ )$ reaction occurring at 1348 keV proton energy with a detector of solid angle 10^{-3}Sr, the count rate for a 100 nA ion beams will be only about 4/min. Hence, a high beam intensity with consequent radiation damage is required for better statistics. We have found that long running times during which the damage anneals out are preferable. Also, the problems of ambiguity arise in examining a solid object. The ambiguity can generally be resolved by rotating the object through small angles, and taking new spectra. The maximum thickness of a fiber can be found by rotating it through small angles and observing the largest energy loss. It is then possible to construct a suitable shape which will unambiguously agree with all the spectra. This is best done by trial and error as discussed previously.

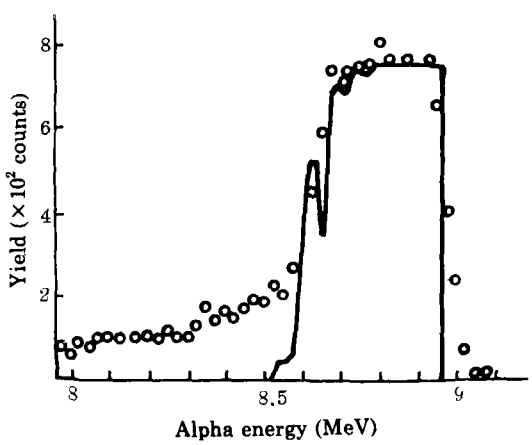

Fig.3 **Comparison of simulation and experiment for 400 keV protons normally incident on a grating of 13400 lines per inch, coated with LiF (<0.1μ m) the detector at 5° from the surface plane**

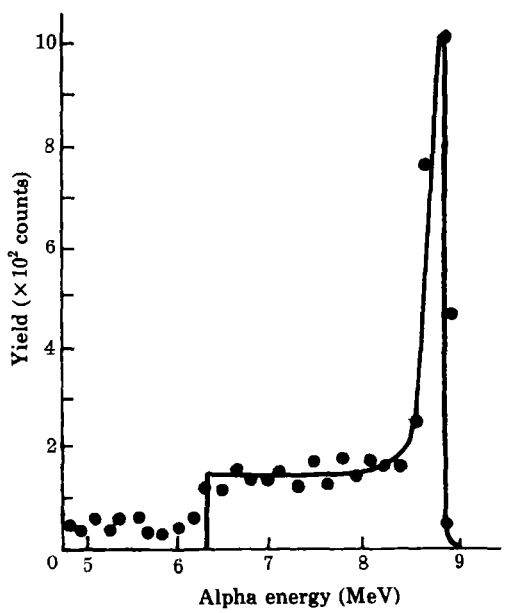

Fig.4 **Theoretical and experimental spectra from the strand of a spider web, 28μ m in diameter, coated with LiF (<0.1μ m)**

In order to investigate this technique using a low energy accelerator, we have examined the profile of a strand from a spider web. The strand was approximately 28μm in diameter. We evaporated a very thin layer($\sim 0.1\mu$ m) of LiF on the fiber, and irradiated with protons. The low energy (340 keV) $^{19}F(p,\alpha\ \gamma\ )$ resonance does not yield a sufficiently monochromatic spectrum of alpha particles, however, the $^{7}Li(p,2\alpha\ )$ reaction for 400 keV protons, behaves much like a resonance approximately 100 keV wide. The theoretical and experimental alpha particle spectra for two cases, under the assumption that the fiber was circular, are compared in Fig.4.

V . CONCLUSION

The surface profile, depth distribution, and shape of objects can be obtained by uniform irradiation of the target with charged particles, either scattering or resonant nuclear reaction method. This technique has advantages in the non- destructive investigation of rough surfaces, with asperities of shorter wavelength than that of light, and for the internal investigation of opaque materials. The stopping power of the incident particles, and of a fragment emitted from the reaction are used to locate the element in the target, and to define the surface. Sharp resonant reactions yielding a unique energetic reaction fragment are most definitive. The principal disadvantage is the low reaction cross section compared with RBS.

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