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Significantly extended analytical potential of Rutherford backscattering spectrometry by *in situ* combination with low-energy sputtering

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We have assembled a dual-beam system which allows solid samples to be analyzed by Rutherford backscattering spectrometry (RBS) at deliberately chosen stages of *in situ* sputter etching by low-energy ion bombardment. Using the novel setup we demonstrate that, for a large variety of samples, the analytical potential of conventional RBS can be extended remarkably. Apart from the obvious possibility of increasing the accessible depth of analysis, three specific advantages are illustrated: (i) removal of the interference between the signals due to buried dopants and a (low-mass) substrate, (ii) decoupling of the mass and the energy scale, and (iii) high-resolution depth analysis of deep-lying structures (glancing angle RBS) with the ability to circumvent the problems usually associated with sputter-related mixing effects.

Rutherford backscattering spectrometry (RBS) is known as a very useful technique for quantitative analysis of the composition of solid samples up to a depth of about 1 μm.[1] Near the surface the depth resolution δz may be as good as 2 nm if a solid-state detector is employed in combination with a glancing angle scattering geometry.[2] Even better resolution data can be achieved with systems featuring magnetic[3,4] or electrostatic[5,6] energy analyzers.

Unfortunately, however, δz increases rapidly with increasing depth due to multiple scattering and energy loss straggling of the probing beam.[1,2,5] Another limitation of the RBS technique arises from the fact that the measured backscattering energy E_i is determined by two different contributions to the energy loss, firstly the elastic (kinematic) loss occurring in the very scattering event and secondly, the inelastic (electronic) loss experienced by the probing beam along its path through the sample

$$E_i = K_i E_0 - [\epsilon]_i N z. \tag{1}$$

K_i denotes the kinematic scattering factor,[1] which is determined by the masses of the projectile (M_1) and the scattering center (M_i) as well as by the scattering angle Θ. E_0 is the primary ion energy, z is the depth normal to the surface, N is the number density of target atoms, and $[\epsilon]_i$ is the stopping cross-section factor,

$$[\epsilon]_i = K_i(\epsilon_{\text{in}}/\cos\theta_{\text{in}}) - (\epsilon_{\text{out}}/\cos\theta_{\text{out}}). \tag{2}$$

ϵ signifies the (mean) stopping cross section for the projectile-target combination under study and θ the angle between the direction of beam propagation and the surface normal. The subscripts "in" and "out" denote the ingoing and outgoing paths, respectively.

According to Eq. (1) the depth of location of atoms producing a certain feature in the backscattering spectrum can only be determined from the energy E_i if K_i, i.e., if M_i is known prior to the analysis (measurements at different tilt angles may solve the problem, but only for shallow dopant distributions). Moreover, Eq. (1) indicates that analysis of the sample composition is possible only up to certain depth.

An additional limitation is brought about by spectral interference. In a matrix composed of atoms of mass M_m the sensitivity for impurities of mass $M_i < M_m$ is very poor due to the superposition of the impurity signal and the matrix "background." The same kind of problem occurs for $M_i = M_m + \Delta M_{i,m}$ if the impurities are located at a depth $z > z_{i,m}$, where

$$z_{i,m} = (K_i - K_m)E_0/N[\epsilon]_i. \tag{3}$$

The basic idea of the present work is that the limitations discussed above can be removed, largely or fully, by performing additional RBS measurements after controlled sputter etching of the sample ("sputter/RBS"). This is an obvious approach for extending the depth accessible in RBS analysis. More important, however, are the following advantages.

(i) Sputter etching provides means to shift buried concentration distributions for dopants of (known) mass $M_i = M_m + \Delta M_{i,m}$ out of the region of interference with the matrix signal. With reference to Eq. (3) this can be accomplished simply by removing a layer of thickness Δz so that after sputtering the dopants are located at a depth $z' = z - \Delta z < z_{i,m}$.

(ii) According to Eq. (1) the differential shift ΔE_i resulting from the removal of a layer of thickness Δz is

$$\Delta E_i/\Delta z = -N[\epsilon]_i. \tag{4}$$

If $N\epsilon$, θ_{in}, and θ_{out} are known one may thus use Eqs. (2) and (4) to determine the unknown mass $M_i = f(K_i, \Theta)$ from the measured quantities ΔE_i and Δz. The thickness Δz of the removed layer can be determined either by measuring the depth of the sputtered crater after the analysis or by a calculation based on experimental data for the sputtering yield and a measurement of the applied low-energy ion fluence. If $N\epsilon$ is not known, the identity of the unknown dopant can be determined by repeated steps of sputter etching and RBS analysis up to the point where the high-energy edge of the feature under investigation does not shift any more during the following step of etching. In that case the distribution of dopants extends to the instantaneous surface ($z' = 0$) so that

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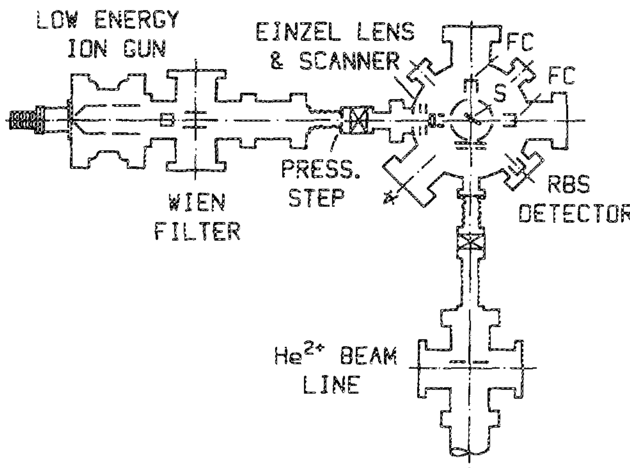

FIG. 1. Cross section of the experimental setup. S = sample, FC = Faraday cup.

mass identification according to Eq. (1) becomes unambiguous,

$$K_i = E_i/E_0. \quad (5)$$

(iii) The sputter/RBS technique can be employed to measure buried dopant distributions or multilayer structures with good or excellent depth resolution. For this purpose the region of interest is shifted close to the instantaneous surface so that the advantages of glancing-angle RBS can be fully utilized. In contrast to depth profiling techniques based upon mass analysis of the sputtered particles or upon surface analysis of the etched sample, sputter/RBS offers the exciting possibility of getting around the problems usually associated with atomic mixing, a process which inevitably takes place during sputtering.[7,8] If the intermixed zone is kept shallow by sputtering with low-energy heavy ions at oblique incidence, there will be an undamaged region underneath the altered layer which is close enough to the instantaneous surface so that in this part of the sample RBS analysis can be performed with high depth resolution.

The experimental setup employed to verify the ideas outlined above is shown in Fig. 1. The sample was mounted on a manipulator which allowed full rotation as well as translation along three axes. The sample surface was located at the crossing point of the two ion beams which propagated at right angle with respect to each other. Backscattering analysis was performed using a collimated beam of 700 keV He^{2+} ($\Theta = 135°$; $\theta_{in} - \theta_{out} = 45°$). The probing beam entered the scattering chamber through a series of apertures, the last of which having a rectangular shape (width in the plane of drawing 0.1 mm, height 0.5 mm). Due to the use of this narrow aperture the width of the helium beam on target did not exceed 1 mm up to angles of incidence as large as 84°. The low-energy ion beam used for sputtering was produced by a telefocus ion gun.[9] Mass analysis of the ion beam was accomplished using a Wien filter. After having passed through a pressure step the mass filtered beam was focused onto the target. Beam rastering was employed in order to produce a uniform bombardment fluence across an area which was sufficiently large compared to the area seen by the probing helium beam. In this study we used a 4 keV Ne^+ beam for sputtering. The angle of incidence θ_{sp} was 60°.

The first example showing the usefulness of the sputter/RBS technique is depicted in Fig. 2. The sample originated from an investigation of As segregation produced during the thermal growth of SiO_2 on an As-doped polycrystalline Si ("poly-Si") layer.[10] This layer had previously been deposited on a SiO_2-covered Si substrate. Subsequent to the removal of a thin (15 nm) surface oxide grown during annealing in O_2, RBS analysis was carried out with a sample orientation such that the accessible depth of analysis was maximized ($\theta_{in} = 28°$). Nevertheless, the large-depth part of the As spectrum was found to interfere with the near-surface part of the Si "background," cf. spectrum (a) in Fig. 2. This interference could be removed easily by sputter etching an appropriate fraction of the As-doped layer, as illustrated by spectra (b) and (c). Segregation of As at the Si/SiO_2 interface can now be identified easily.

The spectra in Fig. 2 may also serve to illustrate the above procedure (ii). In fact, if the identity of the dopants had not been known prior to the RBS analysis, one would have been able to determine their mass from two features, firstly the shift of the low-energy edge of the dopant spectrum and secondly the constancy of the high-energy edge. Note that in this case the shift of the low-energy edge of the polycrystalline Si spectrum provides a simple means for determining the eroded depth Δz.

One might also point out with reference to Fig. 2 that sputtering can be employed to circumvent, for low-concentration impurity distributions in a uniform matrix, any ambiguity in quantitative analysis which might arise from un-

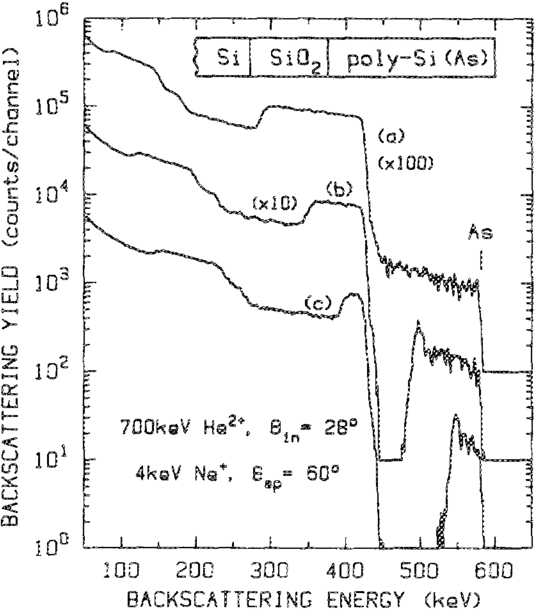

FIG. 2. RBS spectra of a sample composed of a Si substrate covered with an intermediate 300 nm layer of SiO_2 and a 220 nm top layer of As-doped polycrystalline Si (poly-Si), (a) as prepared, (b) and (c) at two different stages of sputter etching. A simple smoothing routine was employed to reduce scatter of the data in the low-yield region. The concentration of Ne implanted during sputtering was too low to be detectable in the presence of the Si or SiO_2 "background" ($K_{Ne}E_0 = 350$ keV).

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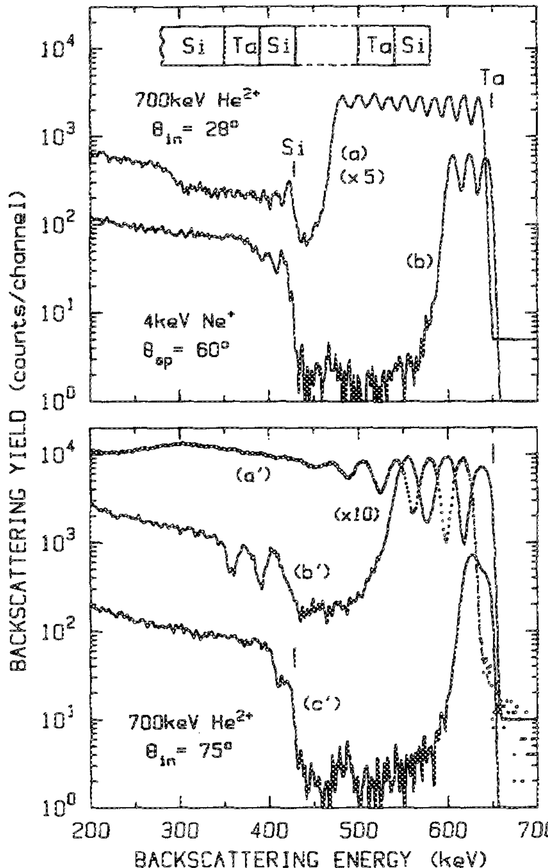

FIG. 3. RBS spectra of a 185 nm multilayer sample of Si/Ta on a silicon substrate. (a) and (a') as-prepared sample (outermost layer Si); (b), (b'), and (c') spectra taken at various stages of sputter etching [the same stage for (b) and (b')]. The thickness of an individual Ta layer amounted to between 6 and 7 nm.

certainties in the energy dependence of the stopping cross section. Using sputter/RBS one can shift the spectrum through a fixed narrow window ΔE within which the stopping cross-section factor is sufficiently constant. If ΔE is chosen to correspond to the (constant) sputtering interval Δz, the concentration distribution can be determined quite accurately in a stepwise fashion.

The second example demonstrating the potential of sputter/RBS for high-resolution depth profiling is shown in Fig. 3. The sample consisted of 10 Si/Ta double layers grown on a silicon substrate by sputter deposition. The total thickness of the multilayer was found to be (185 ± 10)nm; the average composition corresponded to $TaSi_2$. Due to the limited energy resolution of the employed surface barrier detector (7 keV at $-120\,°C$, 0.97 keV/channel) details of the structure could not be identified too well at $\theta_{in} = 28°$, cf. spectra (a) and (b) in Fig. 3. Much better depth resolution was achieved using a glancing incidence scattering geometry, e.g., $\theta_{in} = 75°$, as shown in spectrum (a') of Fig. 3. In that case, however, energy straggling of the probing beam limits the useful part of the backscattering spectrum to only a small fraction of the multilayer. Accordingly, the composition of the complete multilayer could be determined with high resolution only in a stepwise manner.

The sample composition observed after removal of about seven and nine double layers can be derived from spectra (b') and (c'), respectively, of Fig. 3. The shoulders seen at the high-energy edge of the Si and the Ta features in spectrum (c') clearly reveal intermixing of Ta and Si produced as a by-product of sputter etching. However, the thickness of the intermixed region is sufficiently small so that analysis with rather good resolution ($\delta z < 4$ nm) could be achieved over a depth from about 10 to 50 nm. Using this high performance we were able to show, for example, that the thickness of the first two double layers grown on the substrate was about 15% larger than of the remaining layers.

To summarize, we have shown that sputter/RBS is capable of extending the analytical potential of conventional RBS considerably. The amount of intermixing observed in this study can be reduced by using lower energies and more oblique angles of incidence for sputter etching. Limitations of the novel technique may be encountered, as with other sputter-based methods of analysis, if cone formation occurs as a result of heavy-ion bombardment.

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