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ENHANCED SENSITIVITY AND  
DEPTH RESOLUTION OF OXYGEN DETECTION  
COMBINING RESONANCE SCATTERING AND  
TILTED TARGET METHODS

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ENHANCED SENSITIVITY AND DEPTH RESOLUTION OF OXYGEN  
DETECTION COMBINING RESONANCE SCATTERING AND TILTED  
TARGET METHODS

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## ABSTRACT

Using glancing incidence (a target tilt between  $80^{\circ}$ - $83^{\circ}$ ), the depth resolution of  $^{16}\text{O}(\alpha,\alpha)^{16}\text{O}$  resonance for oxygen detection has been improved by a factor 5-7 depending on the depth. The more buried the oxide, the more straggling contribution is observed. Besides, at present scattering geometry an additionally 3.5 times better detection limit is achieved.

## АННОТАЦИЯ

Разрешение по глубине измерения распределения кислорода в тонких пленках с помощью ядерной реакции  $^{16}\text{O}(\alpha,\alpha)^{16}\text{O}$  улучшается в 5-7 раз, если пучок гелия падает на образец под большим углом,  $80$ - $83^{\circ}$  к нормали поверхности. При увеличении расстояния атомов кислорода от поверхности увеличивается разброс падающего пучка по энергии и ухудшается разрешение. В данном случае и чувствительность повысилась в 3,5 раз.

## KIVONAT

A  $^{16}\text{O}(\alpha,\alpha)^{16}\text{O}$  magreakción alapuló oxigénmeghatározási módszer mélységfelbontása 5-7 szeresére javítható, ha az analizáló nyaláb és a vizsgált minta felületének normális közötti szöget  $80$ - $83^{\circ}$ -ra megnöveljük. Eltemetett oxidrétegek esetén a He ionok energiaszórásából a felbontáshoz adódó járulékot is kimértük a mélység függvényében. Az általunk használt geometriai elrendezésben a detektálás érzékenysége 3.5-szeresére nőtt.

## 1. INTRODUCTION

For quantitative oxygen determination, both on the very surface and in bulk, the 3.045 MeV  $^{16}\text{O}(\alpha,\alpha)^{16}\text{O}$  resonance proved to be an effective method <sup>1)</sup>. The sensitivity for oxygen detection in silicon of conventional RBS is about  $3 \times 10^{15}$  atom/cm<sup>2</sup> <sup>2)</sup>. We have pointed out earlier that this limit could be lowered by a factor 17 using the resonance scattering. These sensitivity values are valid, of course, only for experiments which are performed on single-crystals where resonance scattering can be combined with channeling by which the signal to background ratio increases at the energy of oxygen in the backscattering spectrum. In an earlier paper the sensitivity of this method has also been compared with other surface analyses <sup>3)</sup>. It was shown that native oxide formed on <100> silicon could be easily studied using the resonance while this measurement proved to be impossible with standard RBS<sup>2)</sup>. The improved sensitivity, however, is associated with poor depth resolution because of the half width of resonance (10 keV) that is equivalent to about 50 nm in silicon. On the other hand, the glancing angle geometry is generally used to improve the depth resolution of RBS <sup>4)</sup>. This paper deals with the application of tilted target method to the resonance scattering of helium on oxygen in order to get 6-10 nm depth resolution even for buried oxides, while preserving high sensitivity.

## 2. EXPERIMENTS

For the experiments, a special sandwich sample configuration was prepared. 5  $\Omega\text{cm}$  Si single-crystals were thermally oxidized in dry oxygen atmosphere at 800 °C. The thickness of oxide was

16 nm determined by ellipsometer. Aluminium overlayers of 50 or 100 nm were evaporated onto the oxides. On the Al surface thin native oxide was formed. In some cases 7 nm thick SiO<sub>2</sub> layer on Si was also used.

Backscattering measurements were made in a vacuum of  $5 \times 10^{-5}$  Pa. During experiments the target was tilted from 0° to 89° in several points trying to find the optimum scattering geometry. At buried oxides the quantity of oxygen was determined by increasing the energy of the probing <sup>4</sup>He<sup>+</sup> beam. Another surface barrier detector with collimation was placed at 165° scattering angle in the plane which is perpendicular to the target tilt. At each energy the area of the oxygen peak was calculated and plotting these values as a function of bombarding energy, excitation curves were obtained. The oxygen quantity can be derived either from the FWHM of these curves or from their maximum value <sup>1)</sup>.

### Results and discussion

Fig. 1a shows the excitation curve of 16 nm SiO<sub>2</sub> on Si taking the spectra along <100> direction using the channeling effect to diminish silicon scattering yield at oxygen energy. The FWHM of the curve is 10 keV equal with that of resonance. If one defines the "energy width" of the oxide as

$$\beta = n t \epsilon \quad (1)$$

where  $n$  is the density of oxygen atoms,  $t$  the physical thickness of the oxide and  $\epsilon$  the stopping cross-section of the incoming beam, then at resonance energy  $\beta \approx 3.5$  keV so  $\beta < T$ , the half width of the resonance.

Fig. 1b shows the energy scan of this oxide at tilt angle of  $79.2^\circ$ . The FWHM is 21 keV now. If one uses the approximation

$$\text{FWHM} = (\beta^2 + \Gamma^2)^{1/2} \quad (2)$$

then  $\beta$  is 18.5 keV which is equivalent to an effective thickness of 85 nm corresponding to a 5.3 times magnification. This value coincides with a calculated one at this tilt angle.

Fig. 2 gives examples for the measurement of buried oxides. Here the previous 16 nm thick  $\text{SiO}_2$  with a 50 nm aluminium overlayer was investigated. Fig. 2a shows the excitation curve taken at normal incidence. It can be seen that there is no clear separation of surface native oxide and the buried one because the energy loss of  $^4\text{He}^+$  particle in Al at 3.045 MeV is less than 10 keV. Using  $80.7^\circ$  tilt, however, excellent separation was found (Fig. 2b). The 92 keV energy distance between the oxygen curves corresponds a 6.2 times magnification of the 50 nm layer. The half width of the excitation curve of buried oxide is 27 keV, broader than eq. 2. would predict, because the energy straggling of the analysing beam has a significant contribution, too. For evaluation

$$\text{FWHM} = (\beta^2 + \Gamma^2 + \delta(\Delta E)^2)^{1/2} \quad (3)$$

formula should be applied instead of eq. 2. Here  $\delta(\Delta E)$  is the energy spread due to straggling. Fortunately, the energy straggling data owing to both theoretical and experimental works are tabulated <sup>5)</sup>. The relationship,  $\delta(\Delta E) = 2.355\Omega_{\text{str}}$  where  $\Omega_{\text{str}}$  is the standard deviation of energy broadening calculated from

Bohr's theory <sup>5)</sup> with W.K. Chu's correction <sup>4)</sup>, for aluminium was experimentally verified <sup>6)</sup>.

The energy spread calculated this way together with  $\Gamma$  and magnified  $\beta$  results in 27 keV for FWHM through eq.3. Both surface and buried oxygen peaks were separated in the backscattering spectrum. In Fig. 2 the open circle means the surface oxygen peak and the full circle the buried layer.

In present experiments eq.3. served only for checking the results but this way one can also calculate unknown oxide thicknesses. Theoretically eq.3. is not strictly valid, because this way only the convolution of the Gaussian type contributions can be taken into account. Practically, however, this approximation proved to be satisfactory. Nevertheless, a more elaborate work was made to include the straggling <sup>6)</sup> and it gives practically the same result.

In the last example, where the Al overlayer is 100 nm thick, eq.3. still holds. Fig. 3a shows the case of normal incidence. The peaks in the excitation curve just separated. By tilting the sample with  $81.5^\circ$ , the peak-to-peak energy distance becomes 150 keV equivalent with the magnification of 6.75. The 3 keV increase in FWHM can exactly be described by larger energy spread of longer ion path for the incoming direction.

If one defines the depth resolution as a clear separation of excitation curves for two infinitesimally thin oxides in a given depth, some estimates can be made calculating aluminium as intermediate layer. The thin oxide will always be 10 keV thick and the FWHM is increased only by straggling. Fig. 4 shows the variation of FWHM of oxygen peak in the excitation curve (the

depth resolution) as a function of depth.

Using glancing incidence, the sensitivity of oxygen detection is also improved additionally to the factor of 17 that gives the resonance itself. The variation of the signal to background ratio was investigated as a function of the tilt angle. Fig. 5 shows the ratio of the area of oxygen peak to silicon background in backscattering spectra taking 7 nm  $\text{SiO}_2$  on a Si sample with different tilt angles. All spectra were measured in random direction. The sensitivity increased to a maximum of 3.5 at  $83^\circ$  owing to our scattering geometry. If the detector is placed in the plane which coincides with that of the target tilt, the sensitivity improvement would be proportional to the inverse cosine of the tilt angle.

It is clear, however, that sensitivity increase occurs only for an oxide so thin that increasing effective depth is still below the half-width of resonance, otherwise saturation would be experienced.

### Conclusion

The combination of  $^{16}\text{O}(\alpha,\alpha)^{16}\text{O}$  with glancing incidence improved the depth resolution of oxygen determination by a factor 5-7. The best resolution of 6 nm can be obtained near the surface region, but in 500 nm depth it is still 10 nm instead of 50 nm. Besides, for thin oxides, the sensitivity also increases depending on the applied tilt angle. In the scattering geometry used in the present experiment the increase was an additional factor of 3.5, combined with the factor of 17 caused by the resonance itself.



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FIGURE CAPTIONS

Fig. 1a Excitation curve of 16 nm SiO<sub>2</sub> on silicon, spectra taken along <100> direction using channeling to diminish silicon scattering yield at oxygen energy.

Fig. 1b Excitation curve of 16 nm SiO<sub>2</sub> layer using 79.2° tilt. The depth magnification is 5.3.

Fig. 2a Excitation curves both surface native oxide and 16 nm SiO<sub>2</sub> on Si buried by 50 nm Al in normal incidence.

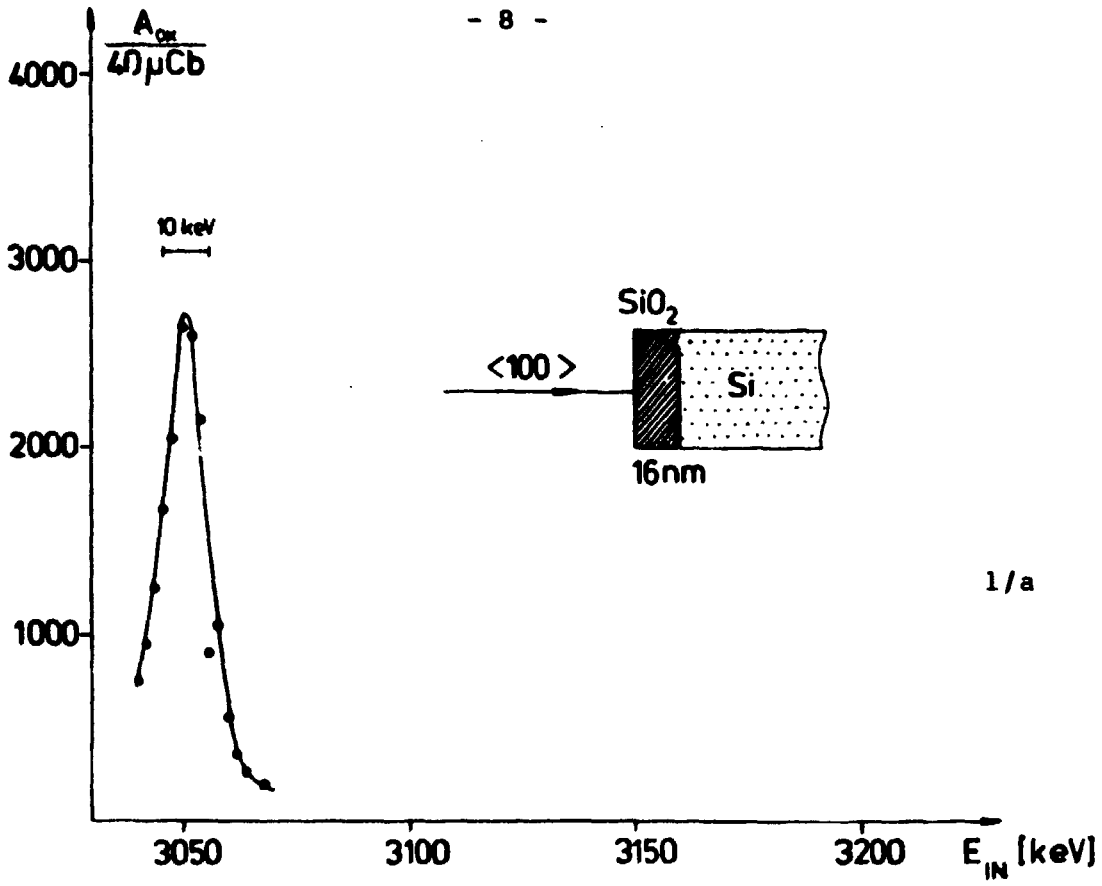
Fig. 2b The investigation of the sandwich of Fig. 2a using 80.7° target tilt. There is excellent separation between excitation curves between surface oxide and buried one.

Fig. 3a Excitation curves for surface native oxide and buried one for normal incidence.

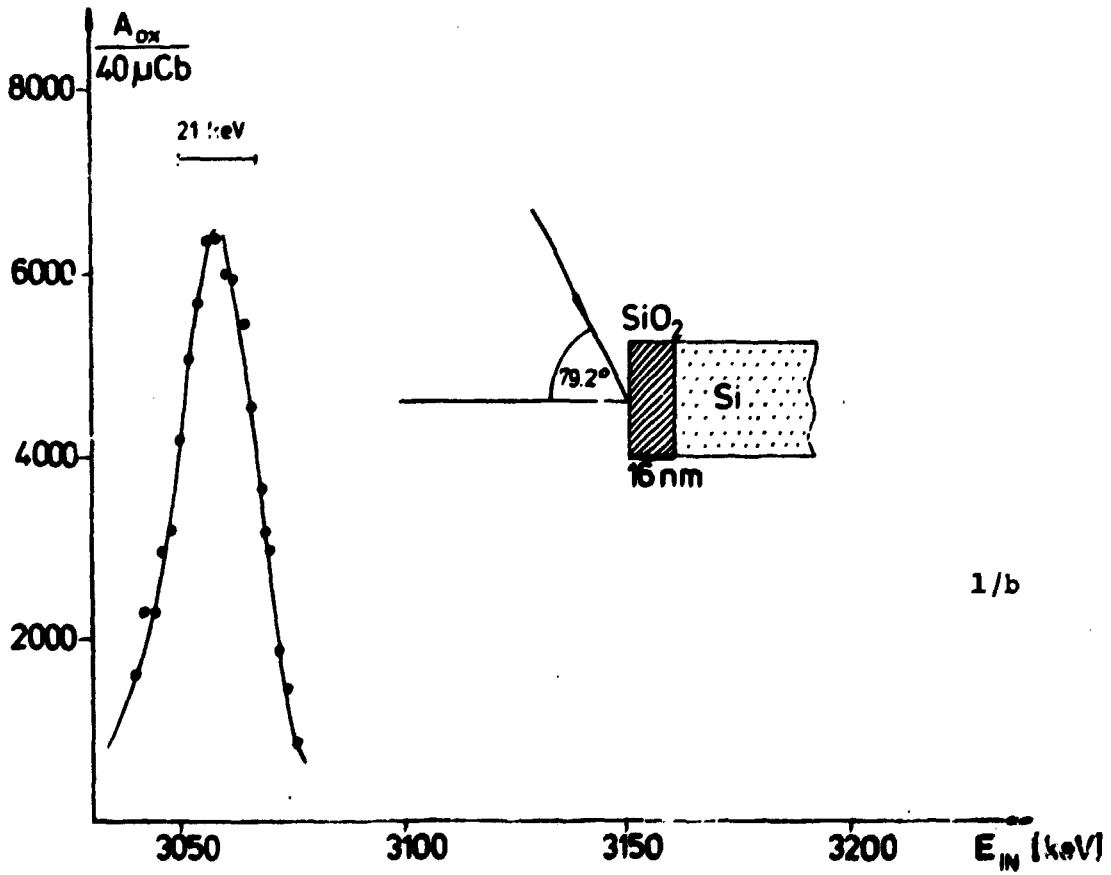
Fig. 3b The energy scan of a sandwich structure using 81.5° tilt which means 6.75 magnification.

Fig. 4 The calculated depth resolution for oxygen resonance as a function of depth for buried oxides.

Fig. 5 The change of sensitivity for 7 nm SiO<sub>2</sub> on Si as a function tilt angle.



1/a



1/b

Fig. 1/a; 1/b

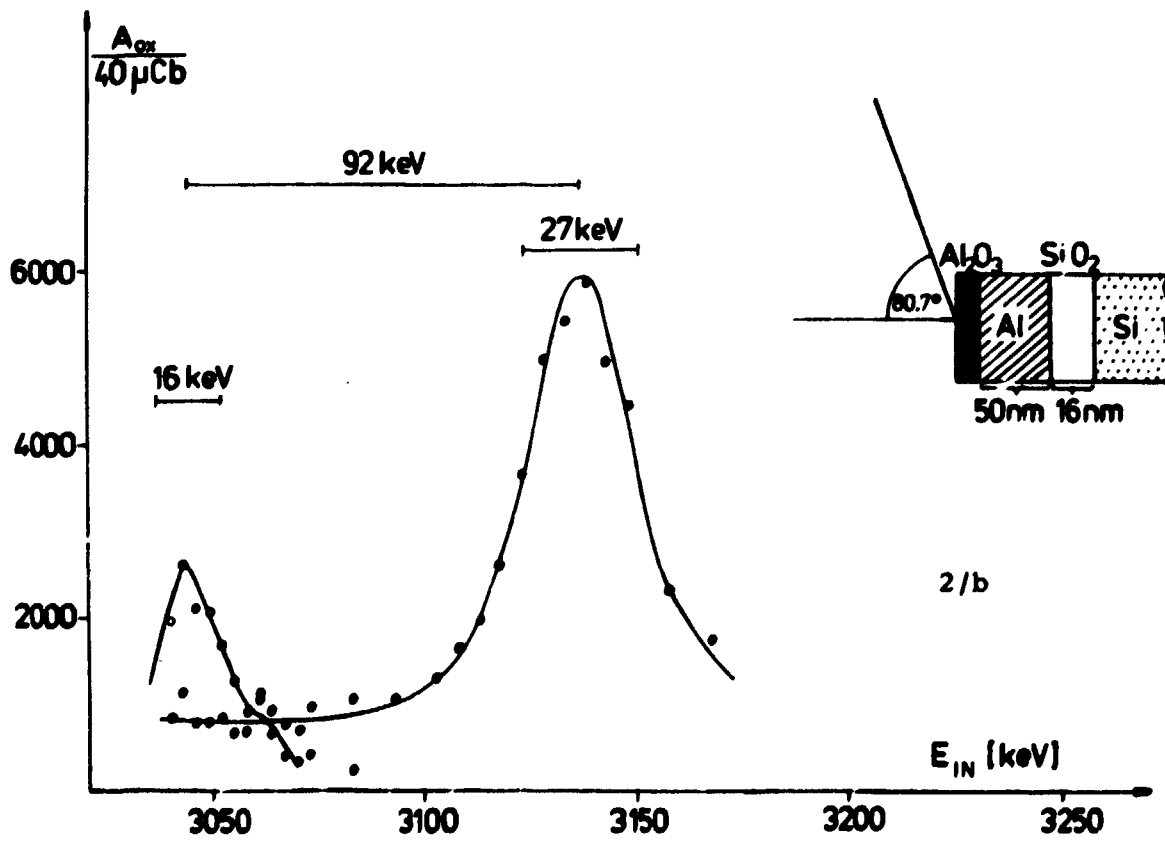
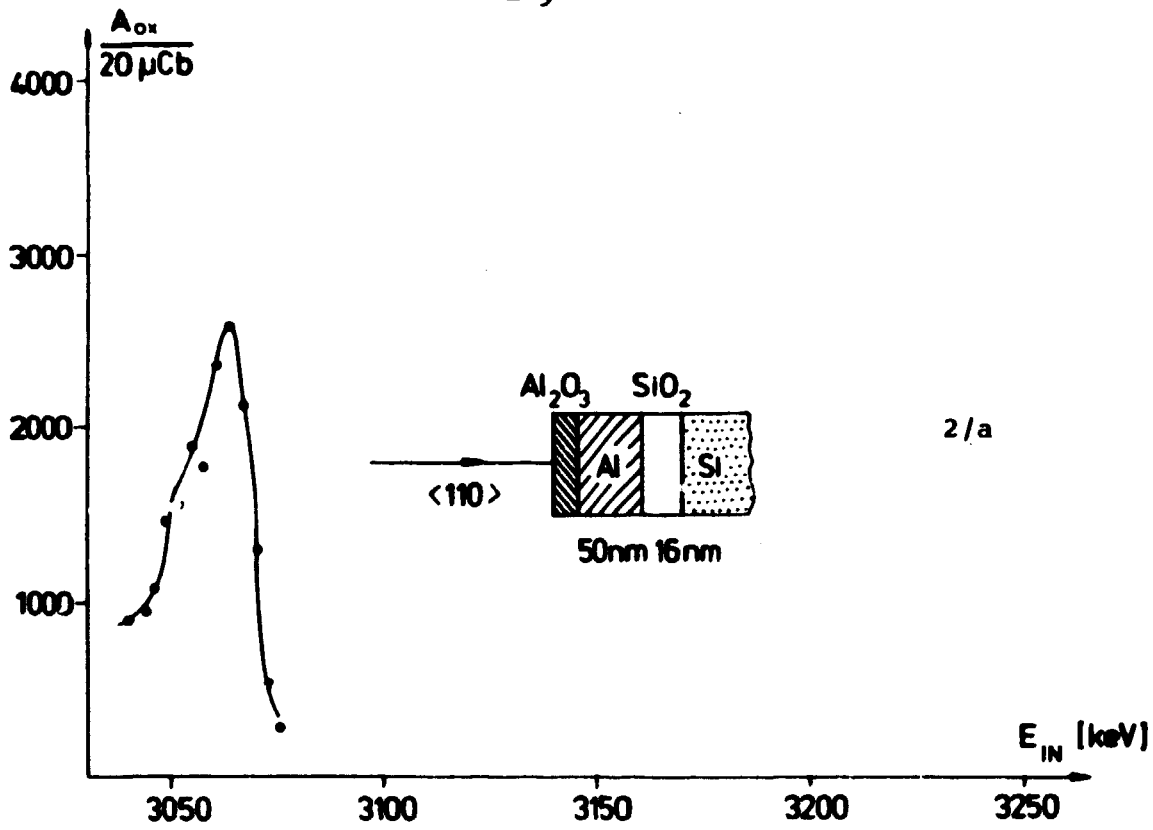


Fig. 2/a; 2/b

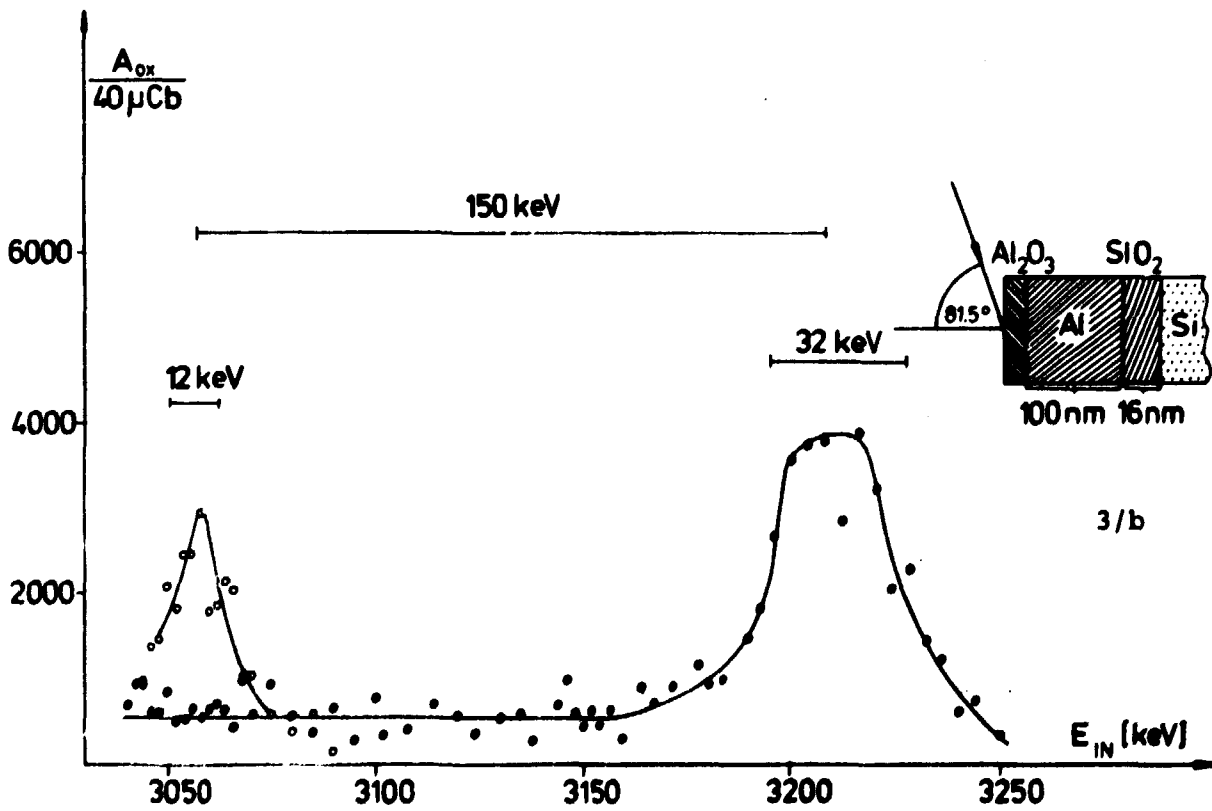
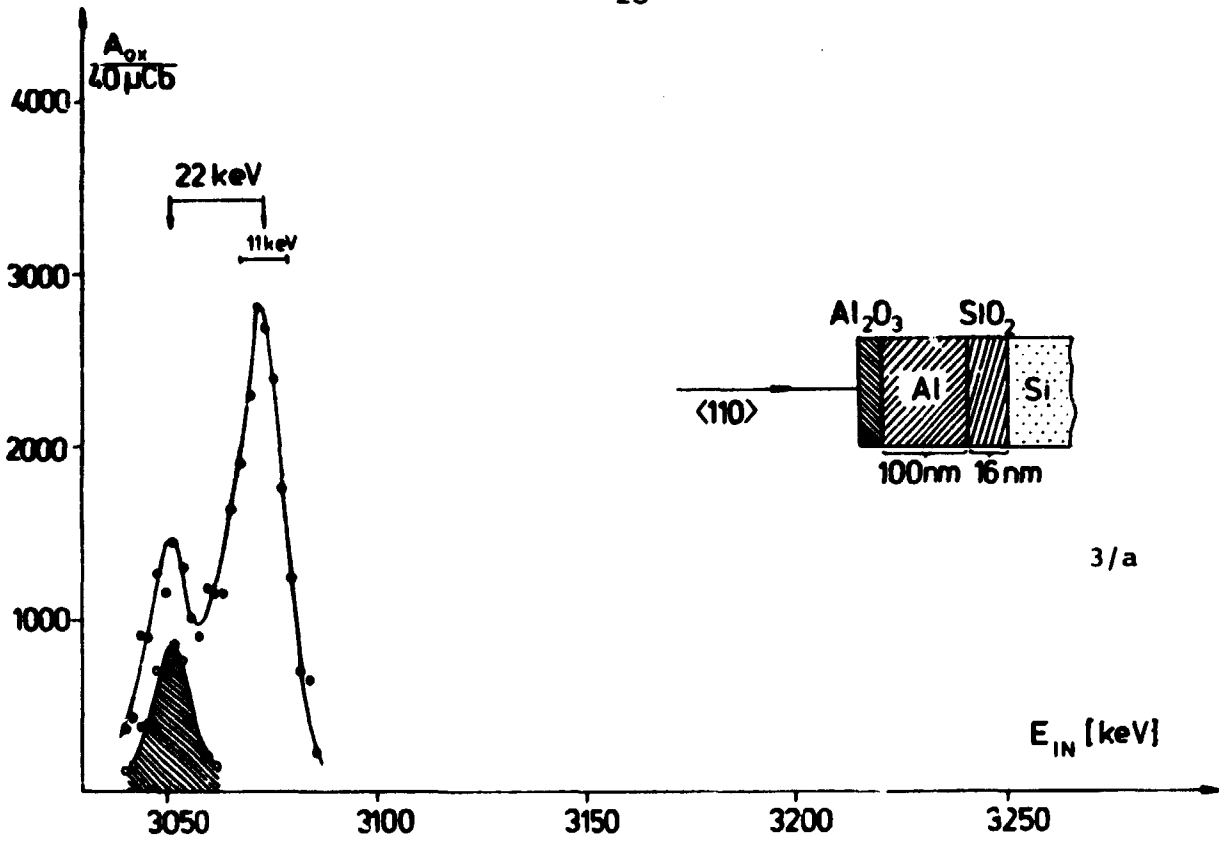
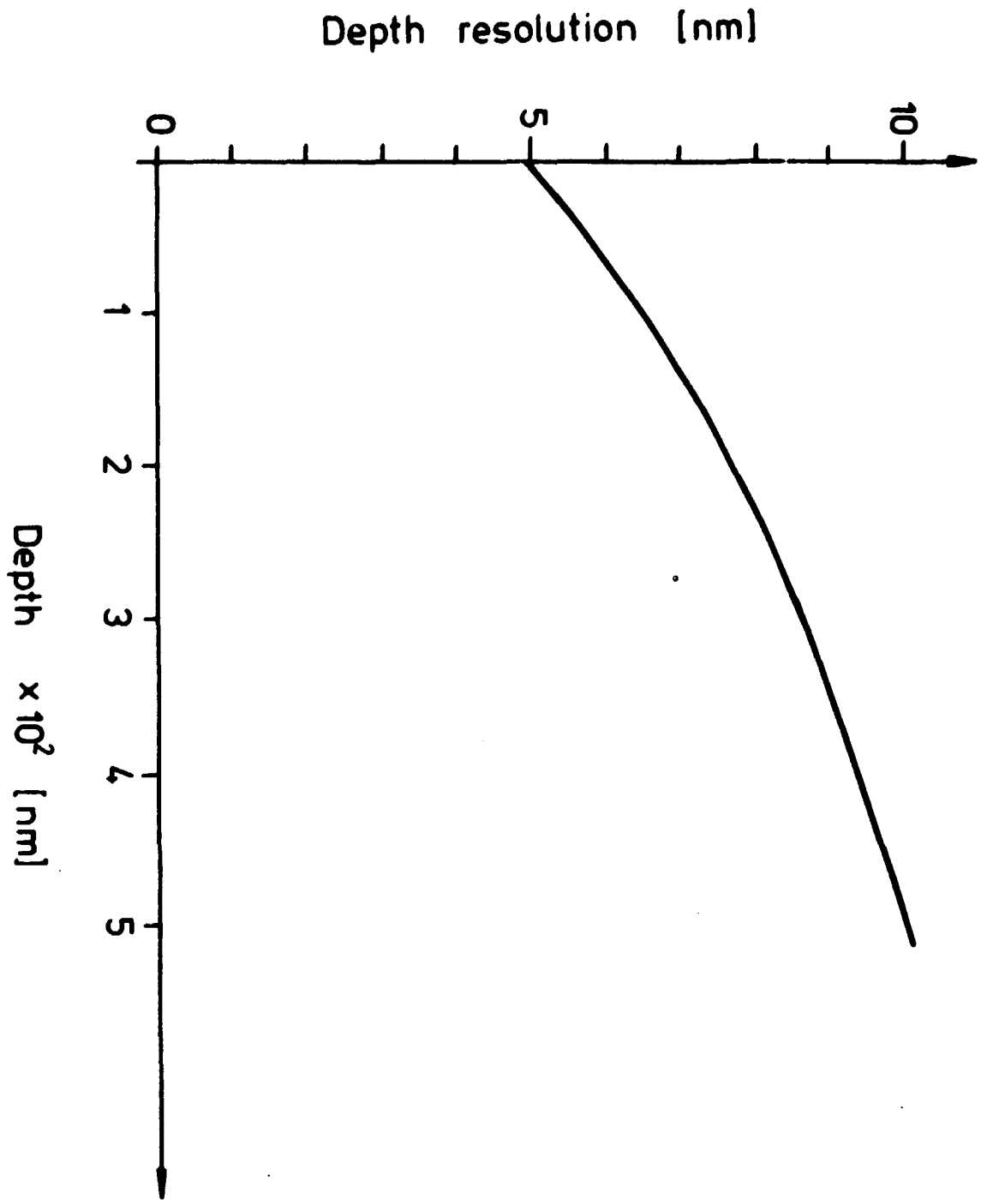


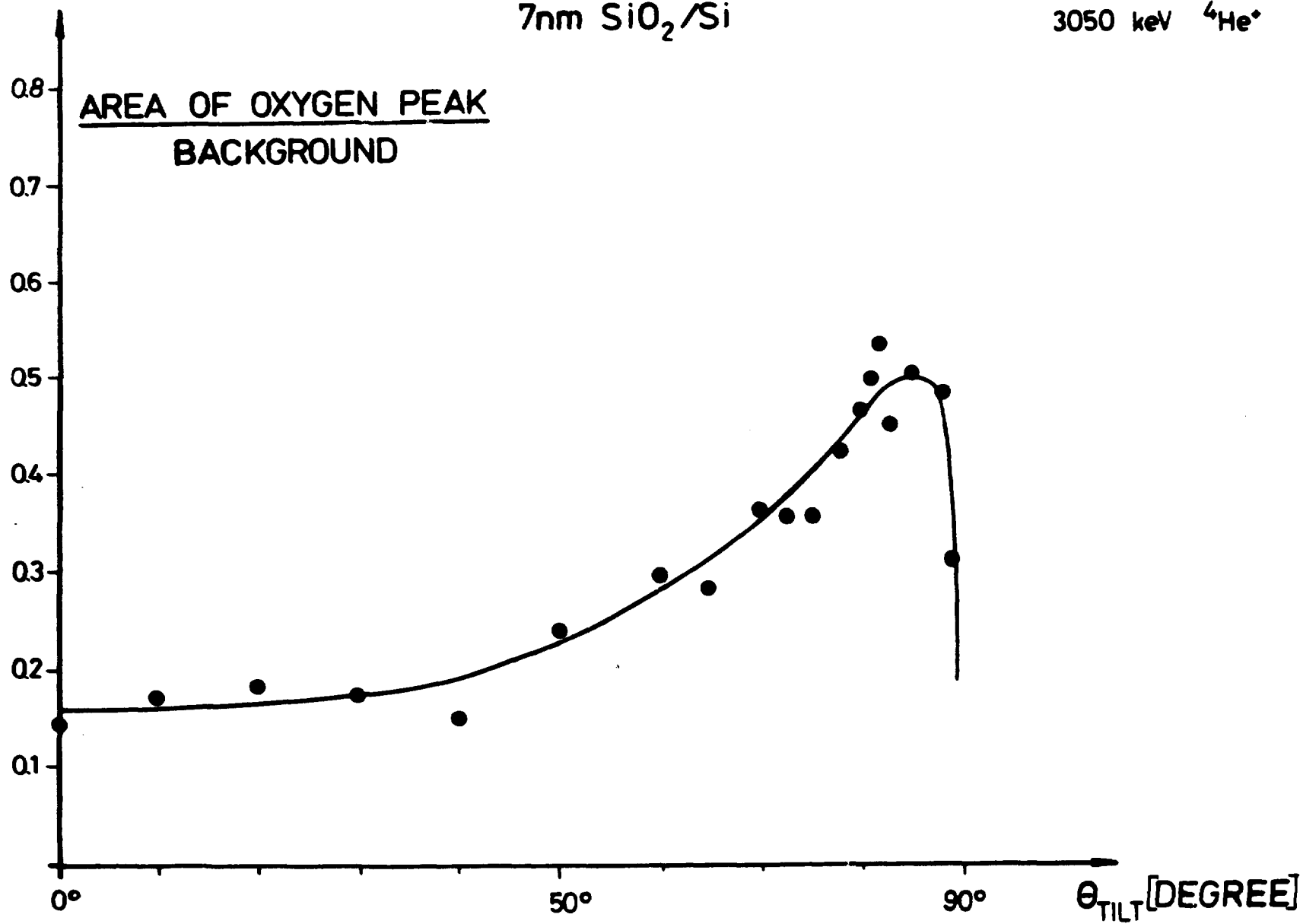
Fig. 3/a; 3/b

Fig. 4.



7nm SiO<sub>2</sub>/Si

3050 keV <sup>4</sup>He<sup>+</sup>



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