

Influence of surface topography on depth profiles obtained by Rutherford backscattering spectrometry

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A method for determining correct depth profiles from samples with rough surfaces is presented. The method combines Rutherford backscattering spectrometry with atomic force microscopy. The topographical information obtained by atomic force microscopy is used to calculate the effect of the surface roughness on the backscattering spectrum. As an example, annealed Au/ZnSe heterostructures are studied. Gold grains were observed on the surfaces of the annealed samples. The annealing also caused diffusion of gold into the ZnSe. Backscattering spectra of the samples were measured with a 2 MeV $^4\text{He}^+$ ion beam. A scanning nuclear microprobe was used to verify the results by measuring backscattering from grains and from areas of the samples where no grains had been formed during annealing. © 2000 American Institute of Physics.

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I. INTRODUCTION

The purpose of this work is to present a method for extracting depth profiles obtained by Rutherford backscattering spectrometry (RBS) from samples with rough surfaces. RBS is one of the most commonly used tools for depth profiling in various fields of physics, therefore the development of such a method is essential. It is known that surface roughness can make the interpretation of RBS spectra difficult.¹ For example in the study of diffusion from thin films, the annealing often causes grain formation on the sample surface. These changes in the surface topography can cause diffusion-like broadening of the RBS spectra, which has been shown in a study by Campisano *et al.*,² thus making the interpretation of the true diffusion result ambiguous. Recently Baglin *et al.*³ studied the effect of grain formation on RBS on polycrystalline metal films. Metzner *et al.*^{4,5} have proposed a model which can determine the surface properties from measured RBS spectra. Such a model cannot, however, be used in cases where both surface roughness and changes in composition, such as diffusion profiles, are involved and where the signal caused by the surface properties overlaps the signal from the depth distribution inside the sample.

The method we propose for determining correct concentration profiles from samples with rough surfaces combines RBS with atomic force microscopy (AFM). With the topographical information obtained from the AFM data, we can calculate the effect of the surface properties on the RBS

spectra and thus extract the correct concentration profiles.

As an example case we study annealed Au/ZnSe heterostructures. The annealing of these samples caused both diffusion of Au into the bulk ZnSe and formation of gold grains on the sample surfaces. To verify the correctness of the method we use a scanning nuclear microprobe to measure grain free RBS spectra from the samples. The diffusion coefficients obtained from these measurements should match the ones obtained from the combined RBS and AFM study.

Gold is widely used as a contact material on compound semiconductors, thus the electrical properties of the Au/ZnSe system have been extensively studied in the literature, see for example Refs. 6–10. Some of these studies also report on the surface properties. A previous study of ours showed that gold forms grains on the surface of Au/ZnSe samples after annealing¹¹ and is therefore well suited as an example for the method presented.

II. EXPERIMENT

The unintentionally doped *n*-type ZnSe samples used in this study were grown by molecular beam epitaxy on GaAs substrates. The thicknesses of the ZnSe layers were 1.5–1.9 μm . Immediately after growth the samples were transferred to an e-beam vacuum evaporator where a 60–100-nm-thick gold layer was deposited onto the ZnSe.

The annealing was carried out in a quartz-tube furnace in argon atmosphere at a pressure of approximately 660 Torr. During annealing the samples were encapsulated between silicon wafers. The annealing caused diffusion of gold into the ZnSe.¹¹

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The surface properties of the samples were studied with Nanoscope III AFM and scanning electron microscope (SEM). The topographical information of the surface obtained from the AFM images was used to calculate the impact of grains on RBS spectra for the different samples. For the calculation of the RBS spectra we used the computer program Gisa 3.99.¹² Gisa is designed for the analysis of RBS spectra. It calculates theoretical spectra which can be compared to measured ones. Experimental parameters, such as detector resolution, are included in the theoretical calculations. RBS spectra containing concentration independent diffusion profiles can also be calculated with Gisa.

RBS spectra of the samples were measured with a 2 MeV $^4\text{He}^+$ ion beam obtained from a 2.5 MV Van de Graaff accelerator at the University of Helsinki. The scattering angle was 170° and the angle of incidence was 5° in all measurements. The area of the beam spot in these measurements was approximately 0.2 mm^2 . These measurements will hereafter be referred to as macro-RBS.

The scanning nuclear microprobe facility¹³ of the Institute of Nuclear Research of the Hungarian Academy of Sciences in Debrecen was applied for the micro-RBS analysis. The experimental conditions during the measurements were the following: 2.0 MeV $^4\text{He}^+$ beam, $2 \times 2 \mu\text{m}^2$ spot size, 165° scattering angle. All the measurements were performed at normal incidence. By comparing the spectra from the same regions we found that during the measurements the beam heating was negligible. Point scans of $2 \times 2 \mu\text{m}^2$ regions were measured over an area of $20 \times 20 \mu\text{m}^2$. By summing the point scans we got a spectrum that was similar to spectra from macro-RBS measurements. Additional spectra of the samples were measured from grains and from areas where no grains had been formed during annealing.

The AFM and SEM studies showed grain formation on the samples, as can be seen from Figs. 1 and 2. By monitoring the characteristic x-ray emission in the SEM measurements while scanning the samples and by detecting backscattered electrons we could conclude that the grains consisted mainly of gold, as can be seen from the cross-section in Fig. 2. These results were confirmed by the micro-RBS study. The grain width varied from 1 to $3 \mu\text{m}$ on all samples. The grains covered up to 40% of the sample surfaces. An example of an AFM surface profile and a height histogram can be seen in Fig. 1. In the AFM image three spatial coordinates are defined, x and y are the horizontal coordinates and z is the vertical coordinate describing the height of the surface. The height histogram in Fig. 1(a) is the distribution of the z -coordinate points from the AFM image, normalized to the area covered by the image. The grain formation was not uniform over the whole sample. In some areas of the samples no grains had been formed during annealing and the sample structure in these areas was still layered.

In the macro-RBS spectra the yield from the gold grains was clearly overlapping the yield from the diffused gold to a depth of approximately 100 nm below the Au/ZnSe interface. In Fig. 3(a) comparison between spectra obtained by macro-RBS and by micro-RBS from an area where no grain formation has occurred is shown. The diffusion profile is significantly altered to a depth of approximately 100 nm be-

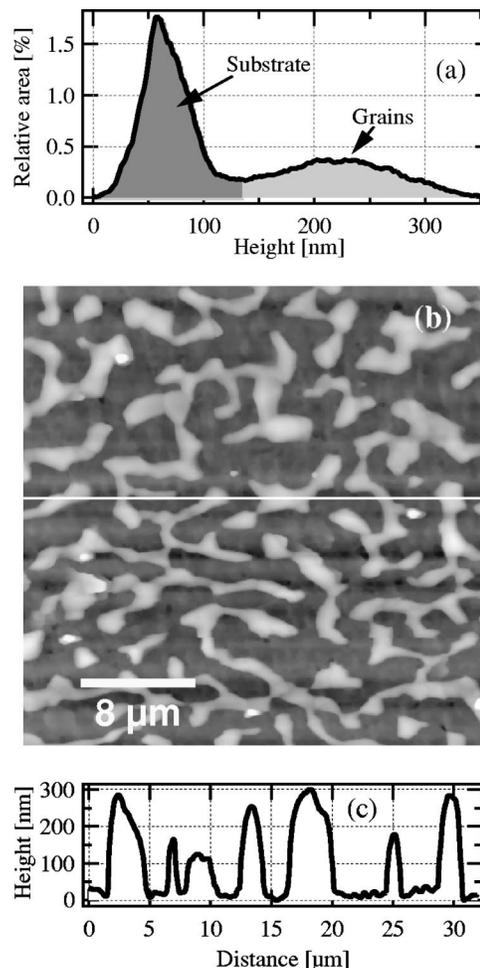


FIG. 1. (a) Height histogram, i.e., the distribution of z -coordinate points, as obtained from the whole area of the AFM image beneath. (b) Typical AFM image of an annealed sample (650°C , 30 min). (c) The cross-section indicated by the line in the AFM image.

yond the Au/ZnSe interface in the macro-RBS spectrum. Micro-RBS spectra obtained from a grain verified that the grains consisted mainly of gold, a small amount of zinc was also detected (less than 5%).

III. DATA ANALYSIS

The topographical information of the sample surfaces obtained from the AFM measurements taken from several spots of the sample, was used to calculate the resulting theoretical RBS spectrum. From the AFM results we calculated the area a certain grain thickness covered, see the height histogram in Fig. 1(a). The step interval for the grain thickness in these calculations was 10 nm. RBS spectra of gold layers on top of ZnSe were then calculated with Gisa 3.99, with the same thickness as in the AFM calculations. The diffusion profile was also included in the calculated spectra. These spectra were then weighted with the relative area of the specific grain thickness and summed. A similar method has been proposed by Baglin *et al.*³ in which the relative area covered by the grains was deduced using a trial-and-error method instead of using experimental AFM data. A comparison between a macro-RBS spectrum and a calculated spec-

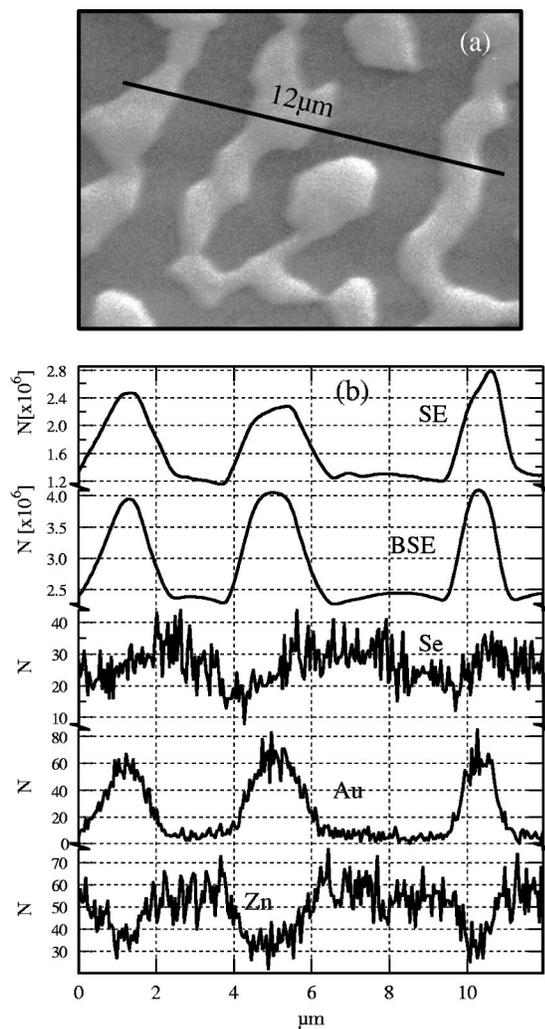


FIG. 2. (a) Typical SEM micrograph of an annealed sample (650 °C, 30 min). (b) The cross-section of secondary electrons (SE), backscattered electrons (BSE), and characteristic x-ray emission (Se-L α , Au-M α , Zn-K α) indicated by the line in the SEM micrograph.

trum can be seen in Fig. 4, the figure also shows the gold grain and gold diffusion contribution to the total spectra. The rest of the gold yield is from areas where no grains have been formed during annealing, i.e., the original gold layer is intact. These results show that the gold grains on the samples do not alter the diffusion profiles at greater depths than 100 nm beyond the Au/ZnSe interface. By comparing the RBS yield from the diffused gold in Fig. 4 with the micro-RBS yield from a grain free area of the sample where the sample structure is still layered (Fig. 3) we see that the yield height is the same in the channel interval 450–480. This verifies the correctness of the diffusion profile determined by combining RBS and AFM.

Besides from overlapping the diffusion profiles the grains will also cause a small depth shift in the diffusion profiles. In the sample considered in Figs. 1–3 the diffusion length is approximately 300 nm and the calculated depth shift is approximately 40 nm. This affects slightly the calculation of the diffusion coefficient for this sample. However, in the diffusion study¹¹ the diffusion lengths are of the order of 500–800 nm and a depth shift of 40 nm is thus negligible.

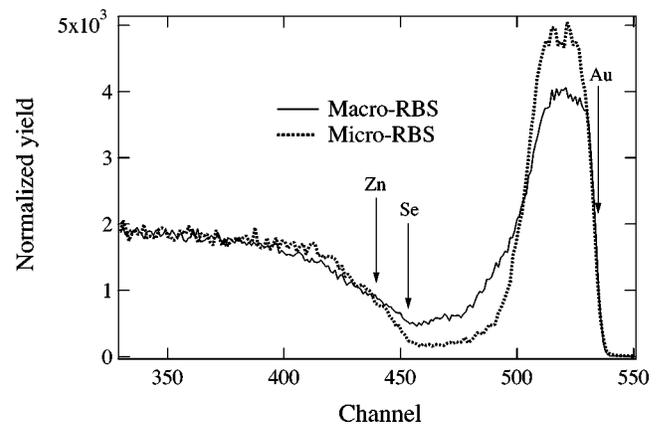


FIG. 3. A comparison between a macro-RBS spectrum and a micro-RBS spectrum from a grain free area of the sample where the sample structure is layered. The sample was annealed for 30 min. in 650 °C. Both spectra were measured with a 2 MeV $^4\text{He}^+$ ion beam and the energy/channel ratio was 3.42 keV/channel. The arrows in the figure indicate the position of the gold surface yield and the zinc and selenium yields at the Au/ZnSe interface in the micro-RBS spectrum.

IV. CONCLUSION

We have presented a general method to obtain correct depth profiles from samples with rough surfaces by combining atomic force microscopy and Rutherford backscattering spectrometry. In the example case of the Au/ZnSe heterostructure we observed gold grains on the sample surfaces. The backscattering signal from these grains overlapped the signal from diffused gold to a depth of approximately 100 nm beyond the Au/ZnSe interface. To verify the accuracy of the depth profiles obtained by macro-RBS, we measured micro-RBS spectra from grain free areas of the samples and from grains with a scanning nuclear microprobe. The diffusion coefficients in Ref. 11 agree very well with diffusion coefficients obtained from the micro-RBS spectra.

The method can in principle be applied to any material system, however, there are practical limitations. If the grains consist of many elements it will be difficult to calculate the theoretical RBS spectrum, since there is one additional quantity in the analysis, i.e., the distribution of elements within

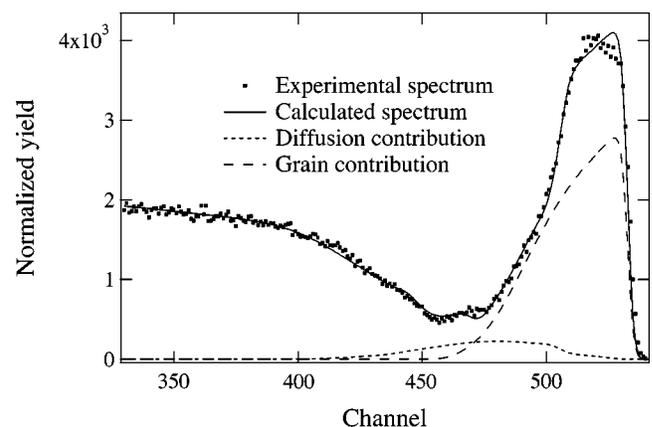


FIG. 4. A comparison between a macro-RBS spectrum, measured with 2 MeV $^4\text{He}^+$ ion beam, and a calculated RBS spectrum. The grain and diffusion contributions to the total yield are also shown.

the grains. Further, if the sample consists of many layers and there is interdiffusion between the layers it will become difficult to resolve the diffusion processes from the grain influence in the RBS spectrum. In addition to these limitations all the limitations of RBS apply as well, i.e., mass and depth resolution etc.

It is noteworthy to emphasize that overlapping of the backscattering signal from the grains and from the diffusing atoms from thin films occur only when the energy separation in the RBS spectra between the diffusing atoms and the grain atoms is small, or the diffusion profile is long. In a previous report¹⁴ we studied platinum diffusion in ZnSe and in this case zinc formed grains on the sample surfaces. However, this did not affect the diffusion profiles since the mass difference between zinc and platinum is over 100 amu. To separate the diffusion profile from the substrate, higher ion beam energies or heavier ions can be used, as was done in Ref. 11.

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