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NON-DESTRUCTIVE QUALITY CONTROL AND MICROANALYSIS
OF ION-IMPLANTED SOLIDS.

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SUMMARY: Non-destructive nuclear physics testing techniques - the Rutherford backscattering in conjunction with ion channeling method and method of nuclear reactions have been used to control quality and element composition of single-crystal GaAs implanted by Al⁺.

1. INTRODUCTION

Industrial production of components and constructions used in crucial conditions has stimulated developing of the non-destructive radiographic methods, such as x-ray, neutron and electron radiography [1].

The large scale application of the ion beam technology for the semiconductor devices fabrication, modification of the metal surfaces properties requires special testing techniques which should be in agreement with the requirements of the high depth resolution, satisfy the sensitivity and selectivity of the element composition in microanalysis. These requirements are conditioned by a very small thickness of the tested layers (about 10-100 nm) and the high sensitivity of solid's properties in dependence on negligible amounts of impurity.

There are two relatively new nuclear physics testing techniques - the Rutherford backscattering in conjunction with ion channeling method and method of nuclear reaction. Both of them satisfy the above mentioned requirements and are used in practice.

II. NON-DESTRUCTIVE NUCLEAR PHYSICS TESTING TECHNIQUES

II.1. The Rutherford backscattering method

The channeling-Rutherford backscattering method uses basic

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rules of interaction of MeV light ions with atoms in crystalline materials /2,3/.

The basic principles of the channeling of an ion beam and backscattering technique are illustrated in Fig.1. When a beam of energetic ions is incident on a monocrystalline target, the interaction between the ions and target atoms depends strongly on the relative orientation of beam and target. Under certain orientation, the repulsive forces between the ion and atom cores can act to gently steer the ion along an oscillatory trajectory through the lattice. This effect is referred to as "channeling". Both axial and planar channeling are possible. Since axial channeling is of more importance in the application of channeling to the measurement of crystal's structure quality control, the parameters basic to it will be considered briefly.

Figure 1 shows the energy spectrum of backscattered ions (M_1, Z_1) on target (M_2, Z_2), for particles following a random trajectory (curve labelled "random"). When a particle of energy, E_0 , strikes the target surface, it may backscatter with energy, E_1 , through angle, θ , to the detector. Since the collision is elastic, energy and momentum are conserved and $E_1 = k^2 E_0$. k is the kinematic factor:

$$k = (M_1 \cos \theta + \sqrt{M_2^2 - M_1^2 \sin^2 \theta}) / (M_1 + M_2). \quad /1/$$

When a particle penetrates the surface and subsequently backscatters at depth, t , it will lose energy to ionization and excitation of the target atoms before and after scattering. If $S(E)$ is the electronic stopping power, a continuous spectrum of backscattered particles will result for $E < E_1$, where:

$$E(t) = k^2 (E_0 - \int_0^t S(E) dt) - \int_{t/\cos \theta}^0 S(E) dt. \quad /2/$$

The energy scale of Fig. 1 converts to a depth scale such that one energy channel, δE , becomes a depth increment, Δt :

$$\Delta t = \delta E / [S], \quad /3/$$

where $[S]$ - is the backscattering energy loss factor. Surface-energy approximation gives:

$$[S] = k^2 S(E_0) + S(E_1) / \cos \theta. \quad /4/$$

The resolution, ΔE_d , of the detection system is revealed in the finite slope of the surface edge in the spectrum at $E=E_1$.

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For a surface barrier detector $\Delta E_d = 15$ keV (FWHM), this will translate to a depth resolution through Eq. 4/ for 1 MeV He^+ -GaAs and $\theta = 150^\circ$, $\Delta t \sim 200\text{\AA}$.

Shown also in Fig. 1 is the backscatter yield when the beam is incident along a low-index crystallographic axis (curve labeled "aligned"). The strong influence of channeling on the backscatter yield is evident.

When the energetic ions are subsequently channeled along a major crystal axis of the target, they will backscatter normally from the displaced atom that lie in the channel, while backscatter from lattice atoms is suppressed in the manner characteristic of the channeling effect. Therefore, the channeling-backscattering technique is a powerful analytical method of investigating both the damage and damage distribution in ion implanted crystals.

Let us consider a heavy impurity of mass i on a light substrate of mass M_2 . In Fig. 1, A_i is the area of the impurity signal and H_M is the height of the signal due to scattering from the surface of the substrate. The amount of the impurity $(Nt)_i$ (atoms per unit area) is

$$(Nt)_i = A_i / Q\Omega\sigma_i \quad /5/$$

where subscript i stands for impurity. The height of the signal generated at the total number of incident particles and the solid angle of detection (Q and Ω consequently), i.e..

$$Q\Omega = H_M \cdot N_M \cdot [S] / \sigma_M \delta E_1 \quad /6/$$

where N_M is the atomic density of the target atoms and δE_1 is the energetic thickness or the energy width of one channel in the spectrum.

By substituting Eq. 6 into Eq. 5,

$$(Nt)_i = A_i \cdot N_M \cdot \sigma_M \cdot \delta E_1 / H_M \cdot \sigma_i [S] \quad /7/$$

However, the resolution between adjacent masses decreases as the mass of the target atom increases because of the resolution of the detector and the dependence of k on the mass difference squared. On the other hand, the rapid rise in sensitivity for the high mass elements - due to the rise in Rutherford cross section means that extremely low concentrations of heavy elements can be detected.

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II.2. Application of the channeling-RBS technique for determination of the nature of defect types in implanted crystals.

Ion channeling, in conjunction with ion backscattering measurements, has been used extensively to control and study disorder in the near-surface region of single crystals. Recent years this technique has been better and deeper understood and developed. In this way it has been found that channeling measurements as a function of various experimental parameters can greatly help in understanding the nature of the disorder.

Primary, Quéré /4/ has theoretically suggested that channeling-RBS technique may be possible to determine the nature of defect clusters by observing the dependence of dechanneling cross-section σ_d on the probing ion energy E . The calculated σ_d shows widely different dependence on energy such as an $E^{1/2}$ dependence for dislocation loops where distortion effects dominate; an E^0 dependence for cavities or gas bubbles; and $E^{-1/2}$ dependence for interstitial atoms where obstruction effects dominate /5,6/. Then these theoretical predictions of the energy dependences σ_d have been experimentally examined and confirmed /7,8/.

In the procedure for analysis of disorder in channeling measurements the aligned yield normalized by the random yield, χ_D , is used which can be described at depth t in terms of two components:

$$\chi_D = \chi_R + (1 - \chi_R) \cdot n_D / N \quad /8/$$

where the first term, χ_R , is the dechanneled fraction of the aligned beam and the second term represents the direct scattering of the channeled fraction of the aligned beam from displaced atoms of density n_D for crystal atom density N . The dechanneling component is given by

$$\chi_R = \chi_V + (1 - \chi_V) \cdot [1 - \exp(-\sigma_d N_D)] \quad , \quad /9/$$

where χ_V is the aligned yield at depth t for a virgin crystal and N_D is the total number of defects per centimeter² integrated from the surface to the depth t .

For particular example (typical for implanted metals) when contribution to direct scattering relative to the dechanneling is negligible, we would have, $\chi_D \approx \chi_R$. Therefore from Eq. /9/

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we obtain

$$(1 - \chi_D)/(1 - \chi_V) = \exp(-\tilde{\sigma}_d N_D). \quad /10/$$

Thus using Eq. /10/, the total damage per unit length, $\tilde{\sigma}_d N_D$ is determined at each bombarding energy. Since we are looking only for the energy dependence of $\tilde{\sigma}_d$, we need not evaluate N_D which is a constant for the same target. Because of the different energy dependences of the cross-sections the various defect types may be distinguished by energy dependent measurements. More than that, Eq. /10/ gives us the possibility to express the depth dependence of N_D and to build up the quantity profile of radiation damage. However, it is necessary emphasis that a high density of dechanneling types damage is required for detection by dechanneling measurements. For example, a minimum dislocation density of approximately $10^9 - 10^{10}$ lines/cm² is required for detection by single alignment channeling measurements. This density is introduced by only moderately high implantation fluences.

II.3. Control of depth profiles of ion-implanted Aluminum using nuclear resonance reaction $^{27}\text{Al}(p,\gamma)^{28}\text{Si}$.

Nuclear resonance reactions have been used extensively for the determination of trace elemental quantities near the surfaces of solids /9/ and have also been used to determine impurity atom depth profiles without the removal of successive layers /10,11/.

We discuss the measure of space distribution of ion-implanted aluminum by means of sharp resonances. The original analytical method and usual experimental procedures are employed in the present work.

The measurements which provide the data for profile determination are made by bombarding the target with a proton beam of well-defined characteristics, counting a fraction of the gamma rays emitted by the sample as the average proton energy is varied in steps ($\Delta E = 1$ keV), and recording the number of gamma rays counted at each step. For profiling Al implanted into GaAs at different temperatures, the resonance in the $^{27}\text{Al}(p,\gamma)^{28}\text{Si}$ reaction at a proton energy of 991.9 keV with a FWHM of 100 eV is used. The gamma-ray yield curve (if plotted as a function of

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bombarding energy relative to resonance energy) serves as an approximate profile with the abscissa giving the depth in terms of the average energy loss of protons in the host material. A more exact profile may be determined by comparing of γ -radiation of Al atoms in GaAs Y_{Al}^{GaAs} with the yield of γ -rays emitted by the aluminum standart. Then we have $Y_{Al}^{GaAs} \sim N_{Al}^{GaAs}$ and $Y_{Al}^{Al} \sim N_{Al}^{Al}$. The Al content in standart is $N_{Al}^{Al} = \delta E' / \epsilon^{Al}$, where $\delta E'$ - energetic width of the analysed layer, ϵ^{Al} - the stopping cross section per atom. Now it is easy establish

$$N_{Al}^{GaAs} = (Y_{Al}^{GaAs} / Y_{Al}^{Al}) \cdot (\delta E' / \epsilon^{Al}) \quad /11/$$

In Eq. 1 yields and $\delta E'$ - are the experimental data, ϵ^{Al} may be taken from the Tables /12/.

For the relative concentration of Al in GaAs we obtain

$$N_{Al}^{GaAs} / N_{GaAs}^{GaAs} = (Y_{Al}^{GaAs} / Y_{Al}^{Al}) \cdot (\epsilon^{GaAs} / \epsilon^{Al}) \quad /12/$$

The data of Al distribution in implanted at different temperatures GaAs are given in Table 1. The doses are 0.8 and 1.2 10^{17} cm^{-2} .

Table 1.

depth, nm	0	20	40	60	80	100	140	180
relative concentration of Al	20°C : 0.19	0.17	0.16	0.14	0.11	0.06	0.015	0
	100°C : 0.23	0.17	0.15	0.14	0.12	0.07	0.04	0.015
	375°C : 0.5	0.28	0.23	0.23	0.21	0.19	0.09	0.023

III. EXPERIMENTAL AND RESULTS.

Peculiarities of radiation damage, spatial distributions of implanted atoms and structural transformations are problems of academic and applied field of science. Consequently, it is a matter of practical interest to control and study radiation damage in GaAs implanted by aluminum ions.

GaAs single crystals of (100) orientation were implanted by Al^+ ions at energy 50 keV. Implantation doses $8 \cdot 10^{16} \text{ cm}^{-2}$ at 20°C and 100°C and $1.2 \cdot 10^{17} \text{ cm}^{-2}$ at 375°C were obtained. The average beam intensities were 2-3 $\mu\text{A/cm}^2$. Control of damage behaviours of ion bombarded GaAs has been done by the channeling technique using He^+ ions with the energy 0.6, 1.0, 1.4 and 2.0 MeV and for measurement of Al spatial distribution protons with the energy 980-1020 keV were applied. Energies of RBS ions were measured using a surface barrier detector with energy resolution $\Delta E = 15 \text{ keV}$. A NaI gamma-ray detector was chosen for γ -radiation registration.

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In Fig. 2 the backscattering spectra are shown for Al^+ implanted GaAs crystals. Curves 3 and 4 show the RBS spectra from non-aligned and oriented crystals implanted at 20°C respectively. The curves indicate that amorphisation has been achieved in the layer 40-45 nm of thickness. The theoretically estimated projected range of 50 keV Al^+ in GaAs is $0.044 \mu\text{m} / 13\%$. However, the differences in yields of random spectra speaks that Al is distributed 2.5-3 times deeper than $\bar{R}_{p, \text{theor}}$. These data are in a good agreement with data in Table 1. The rising of implantation temperature leads to deeper Al distribution and increasing of it's content on the surface of GaAs crystals. The quality of GaAs crystals implanted at high temperature is considerably improved. This is confirmed by the decreasing of aligned yield and by the lack of even a small disorder peak in the vicinity of the implanted region. The high level of the dechanneling reflects the formation in implanted crystals defect clusters of the different types.

We have analysed the types of defects in the implanted layers (200 nm thickness) using the above discussed in the section II.2 technique. In the Fig. 3 we plot the energy dependence of dechanneling parameter (Eq. 10). These curves show that 100°C Al^+ implantation into GaAs leads to formation of extend defects like dislocations. The 375°C implantation of Al^+ into GaAs results in the synthesis of ternary compound $\text{Al}_x\text{Ga}_{1-x}\text{As}$ in the layer epitaxial oriented to the matrix with relatively high concentration of stacking faults.

IV. CONCLUSIONS.

The present results indicate that modern nuclear physics testing techniques - the channeling RBS method and method of nuclear reactions are very powerful and useful methods of non-destructive quality control and microanalysis of ion implanted materials.

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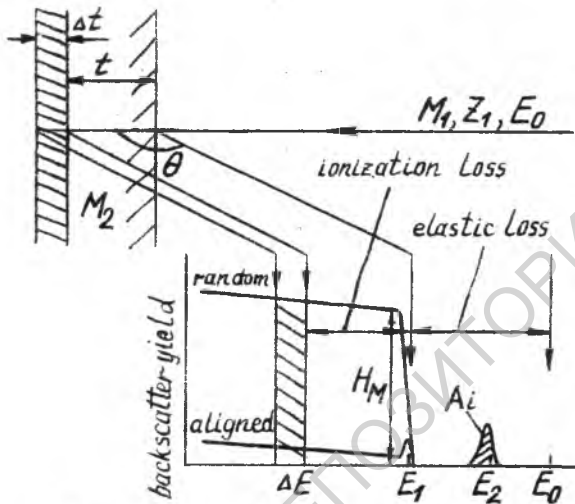


Fig.1. Principles of elastic scattering experiments.

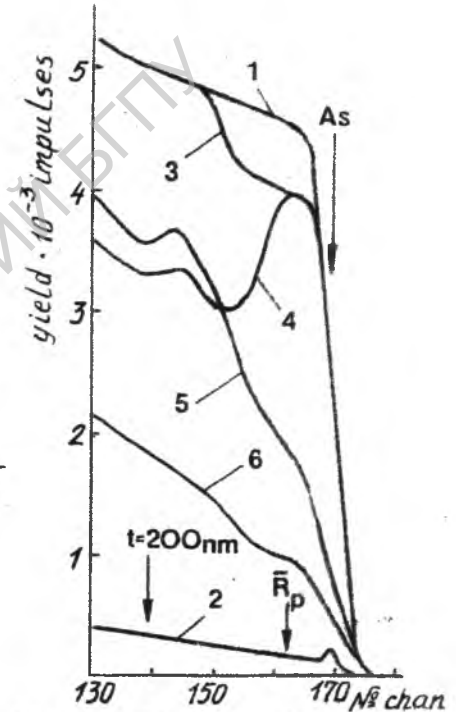


Fig.2. RBS for 1.0 MeV He^+ incident on virgin GaAs - 1, 2; implanted by Al^+ at RT -3, 4; 100°C - 5; 375°C - 6.

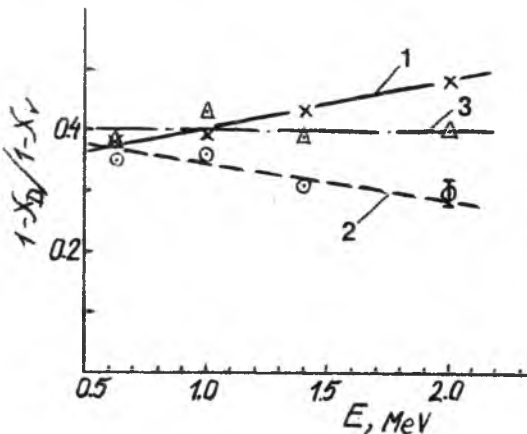


Fig.3. Dechanneling parameter vs energy of He^+ . GaAs implanted by Al^+ at RT -1; 100°C - 2; 375°C - 3.