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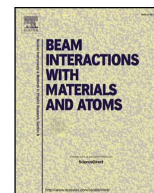

Improving depth resolutions in positron beam spectroscopy by concurrent ion-beam sputtering

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ABSTRACT

The depth resolution of mono-energetic positron annihilation spectroscopy using a positron beam is shown to improve by concurrently removing the sample surface layer during positron beam spectroscopy. During ion-beam sputtering with argon ions, Doppler-broadening spectroscopy is performed with energies ranging from 3 keV to 5 keV allowing for high-resolution defect studies just below the sputtered surface. With this technique, significantly improved depth resolutions could be obtained even at larger depths when compared to standard positron beam experiments which suffer from extended positron implantation profiles at higher positron energies. Our results show that it is possible to investigate layered structures with a thickness of about 4 microns with significantly improved depth resolution. We demonstrated that a purposely generated ion-beam induced defect profile in a silicon sample could be resolved employing the new technique. A depth resolution of less than 100 nm could be reached.

1. Introduction

Positron annihilation spectroscopy (PAS) is a well-established defect characterization method of solids. Positron annihilation lifetime spectroscopy (PALS) or Doppler-broadening spectroscopy (DBS) allows characterization of vacancy-type defects in crystalline material (metallic alloys and semiconductors) [1]. The investigation of near-surface defect structure is done by mono-energetic positron beams. Depth-dependent measurement of defects can be done by variable-energy positron annihilation spectroscopy (VEPAS). The energy-dependent implantation profile of positrons can be described by a “Makhovian profile” (Eq. 1) [2].

$$P(z,E) = \frac{mz^{m-1}}{z_0^m}\exp\left[-\left(\frac{z}{z_0}\right)^m\right] \quad \text{with } z_0 = \frac{AE^r}{\rho\Gamma\left(1+\frac{1}{m}\right)} \tag{1}$$

Γ is the gamma function and ρ is the material mass density. The following values are used for the material-dependent empirical parameters A, m and r: $A = 4{,}0\ \mu g \cdot cm^{-2} \cdot keV^{-r}$, $m = 2$, $r = 1{,}6$ [3]. By increasing the positron energy, the implantation profile broadens, which worsens the depth resolution of this method. Dependent on the material, this resolution is very limited for depths beyond about $d \approx 1\ \mu m$. In order to improve the depth resolution, we now removed the sample surface atoms by sputtering with argon ions during the measurement with constant energy positrons. Former investigations of Börner et al. have shown that by stepwise removing the sample surface atoms by argon ions, the defect structure of the sample region under investigation is not affected if the positron energy is chosen high enough so that the back-diffusion to the damaged surface can be excluded [4]. As a consequence, the positron energy to be selected depends on the diffusion length of the sample to be investigated. Here, we study the application of ion-beam sputtering on depth resolution. PAS for depth-dependent defect analysis and the technique of surface layer removal by ion irradiation are combined in this paper. Through this combination a measurement by means of PAS is carried out during a continuous surface layer removal. A significant reduction in the necessary measurement time is to be expected in addition to an improvement in the depth resolution. The simultaneous execution of PAS and sputtering allows automization of both techniques and it could lead to routinous procedures with the caveat of being destructive technique. The organization of this paper is as follows. In Section 2 an explanation of the experimental details is given. Results are presented and discussed in Section 3, while Section 4 concludes the paper.

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2. Experimental

For the investigation of defect profiles in different materials with Doppler-broadening spectroscopy a mono-energetic positron beam system was used. For this purpose, the positrons, which were generated from a ^{22}Na isotope with an activity of $A \approx 10$ mCi, are moderated by a tungsten foil and accelerated to a transport energy of 28 eV while being guided by a magnetic field to the sample. They have to pass a bended tube which functioned as an energy filter. Behind this filter, the moderated positrons become accelerated to the energy which is used for the measurements and guided to the sample which is kept at ground potential. To avoid collisions between positrons and rest gas molecules a pressure of $p \leqslant 5\cdot10^{-5}$ mbar is necessary. With this setup, it is possible to perform depth-dependent measurements by varying the positron energy. For the conventional $S(E)$ measurement at our beam system, positron energies between $E_{e^+} = 0.03$ keV to $E_{e^+} = 25$ keV are used.

For the simultaneous removal of the sample surface layer ion beam sputtering was used. Therefore, a Kaufman-type ion source [5] was installed on the positron beam system. To reach the operating pressure of the ion source ($p \approx 1\cdot10^{-3}$ mbar) and the sample chamber ($p \leqslant 5\cdot10^{-5}$ mbar) a differential pumping system is required. This system consists of an additional turbo- and pre-pump and two apertures, which allows a pressure difference of two orders of magnitude. For a simultaneous use of both techniques, it is important that both beams (the positron- and the ion beam) hit the sample at the same spot.

Using 5 N argon, Ar^+ ions were generated by the ion source and were accelerated to the sample. When the ions impinge onto the sample surface with the ion energy equal to the acceleration voltage, atoms being removed by sputtering. The sputter rate $\dot{z}$ measures the change of the sample thickness due to sputter removal and depends on material-specific parameters like the molar mass M, the density ρ and the sputtering yield Y. It can be calculated with Eq. (2) [6].

$$\dot{z} = \frac{z}{t} = \frac{M}{\rho N_A e}\cdot Y\cdot j \tag{2}$$

N_A is Avogadro's constant, e is the elementary charge and j is the ion current density. The sputtering yield Y depends on the ion energy, the target, the ion mass and the ion incident angle and was calculated by the program SRIM [7]. Another result of this simulation is the depth distribution of the defects generated by Ar^+ ions. As an example, Fig. 1 shows the depth-dependent defect profile generated by Ar^+ ions with an energy of 1 keV in silicon.

As you can see, with these sputter parameters defects are generated till a depth of about 4 nm beneath the surface. With our setup we generate a maximum ion beam current of $I_B = 10$ mA and a maximum

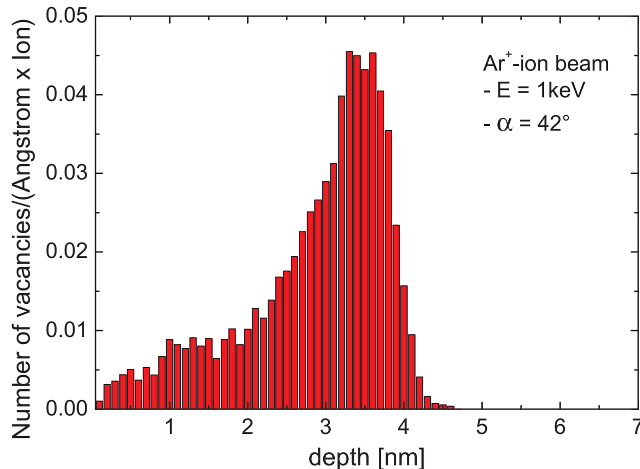

Fig. 1. SRIM simulation of a depth-dependent defect profile generated by Ar^+ ions with an energy of 1 keV in silicon [8].

Table 1
Sputter parameters and the calculated sputter rates.

Material	$M\left[\frac{g}{mol}\right]$	$\rho\left[\frac{kg}{m^3}\right]$	$Y\left[\frac{Atoms}{Ion}\right]$	U_{Ar^+} [V]	$j_{Ar^+}\left[\frac{\mu A}{cm^2}\right]$	$\dot{z}\left[\frac{nm}{h}\right]$
Au	197.0	19320	2.1	460	≈1.5	≈12.0
Cr	52.0	7200	1.6	460	≈1.5	≈7.0
ZnO	81.4	5610	4.0	460	≈2.0	≈40
CdS	144.5	4820	2.6	460	≈2.0	≈55.0
CIGSe	321.2	5700	2.2	460	≈2.0	≈90.0
Mo	95.9	10280	1.0	460	≈2.0	≈10.0
Si	28.1	2336	1.3	920	≈3.5	≈20.0

ion energy of $E_B = 1200$ eV. Depending on the investigated samples, an acceleration voltage of $V_B = 460$ V or $V_B = 920$ V was used. Thereby, a sputter rate between $\dot{z} \approx 7\frac{nm}{h}$ and $\dot{z} \approx 90\frac{nm}{h}$ could be selected. The sputter parameters for some investigated materials are listed in Table 1.

With this experimental technique, three samples were investigated. The first sample is a Au-Cr-layered system consisting of two layers of gold and chromium, which were alternately deposited on a substrate of crystalline silicon with a respective layer thickness of 100 nm. After that, the sputter experiment was performed on a $Cu(In,Ga)Se_2$ (CIGSe) sample. The last investigated sample series was n-type silicon with defined defects and a variation of the implanted ion dose.

3. Results and discussion

Fig. 2 shows the S parameter as a function of the measurement time for the simultaneous sputter experiment (red) on a Au-Cr-layer system. The structure of this sample can also be seen on top of this figure. For the comparison between a conventional DBS measurement and the sputter removal experiment, the $S(E)$ examination (black) was converted to an S parameter as a function of the mean implantation depth by using Eq. (1). In addition, knowing the used sputter rate, the measurement depth can be calculated as a function of the measurement time. Now, the mean penetration depth of the mono-energetic positrons is assumed to be the measurement depth. Thus, the S parameter measured by the energy-dependent DBS can be plotted as a function of the measurement time which is also shown in the same figure. Furthermore, the $S(E)$ measurement was used to determine the theoretical S parameter for each element. For this purpose, Eq. (1) was extended to a multi-layer system in analogy to [3]. The exact formalism can be found

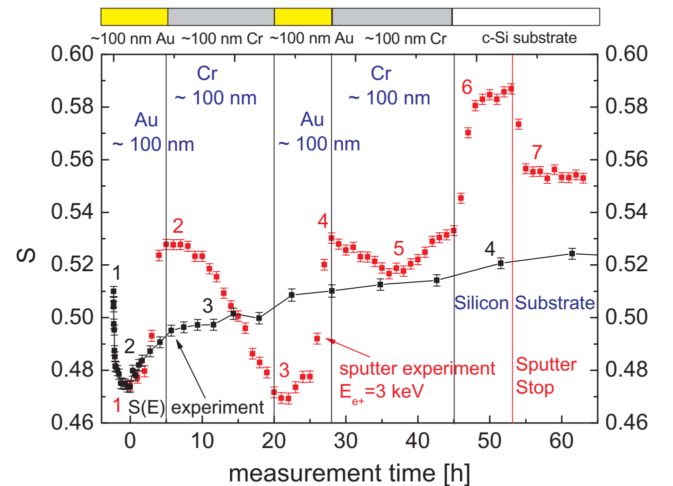

Fig. 2. Comparison between an $S(E)$ measurement from $E_{e^+} = 0.03$ keV to $E_{e^+} = 24$ keV (black) and a measurement of the S parameter during the simultaneous sputter removal (red) on an Au-Cr-layer system. For the removal of the sample surface atoms, Ar^+-ions were accelerated to the sample with a voltage of $V_B = 400$ V. Simultaneously, a measurement of the S parameter was carried out with a constant positron energy of $E_{e^+} = 3$ keV [8].

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