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X-ray reflectivity and non-specular scattering investigation of amorphous W/Si multilayers after rapid thermal annealing

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The specular reflectivity and non-specular scattering measurements at grazing incidence were used to characterize the evolution of the amorphous W/Si multilayer structure with the nominal period of 7 nm after the rapid thermal annealing. No mixing or interdiffusion worsening the perfection of the interfaces takes place up to the 773K/5s annealing. The complex interface phenomena start after the 773K/20s annealing connected with the displacement and coarsening of the interfaces. After the 1023K/5s annealing the ML structure collapsed.

1. Introduction

W/Si multilayers (MLs) are interesting as artificial X-UV mirrors as well as starting structures to prepare silicides for the VLSI technology. In our previous papers we analyzed the phases arising successively during their rapid thermal annealing (RTA) [1] as well as the accompanying changes of the electrical properties [2]. In this paper we complete this information by the results from the X-ray specular reflectivity and non-specular scattering measurements to study the thermal evolution of the ML stack on the background of the previous results. The specular and non-specular results are combined for a more complete characterization of the ML.

2. Experimental

The preparation of the samples was described elsewhere [2]. The results discussed below were obtained from the nominally (6nmSi/1nmW)9x sample. The oxidized Si (100) wafers with the SiO₂ 0.5 μ m thick layer were used as the substrates. The grazing incidence measurements were done with the STOE high resolution diffractometer working with the GaAs double crystal monochromator at 400 reflection. The CuK _{α 1} radiation and a scintillation counter were used.

3. Results and discussion

A series of the RTA processed samples was prepared, the annealing temperature and time being $T = 523\text{K}$, 773K, 1023K, 1273K, and $t = 5\text{s}$, 10s, 20s, 40s, respectively. The specular reflectivity curves are shown in Fig.1 together with the simulation results provided by the Fresnel optical computational code [3]. The Bragg maxima due to the ML period given by the

basic bilayer thickness and the Kiessig fringes in between due to the finite size ML structure are clearly visible. The ML period Λ and interfacial roughness σ coming from the simulation decrease up to the 773K/5s annealing. No distinct reflectivity changes on the Bragg maxima occur. After the 773K/20s annealing the 2nd Bragg maximum disappeared which implies the redistribution of the atomic density within the basic bilayer connected with the displacement of the interface to its middle. However, the interfaces are obviously still present and the ML character of the structure is preserved. This vanishing of the 2nd Bragg maximum took place also after the 773K/40s annealing and needs a more detailed elucidation in the future as it cannot be explained only by a simple interdiffusion and mixing at the interfaces. After the 1023K/5s annealing the ML structure collapsed. The rest small maxima come mainly from the interferences of the waves reflected from the air/ML and ML/substrate interfaces. After the 1273K/5s annealing also these features vanished.

The interfacial roughness σ was included in the simulations by the Debye-Waller factor $\exp(-4\pi\sigma\sin\theta/\lambda)^2$, corresponding to the Gaussian distribution, which describes the reduction of the specular reflectivity. However, a simulation of the specular spectrum cannot reliably distinguish between the "rough" and the "diffuse" interface which is principally possible by the non-specular scattering. Some examples of the 2θ scans with the fixed sample are shown in Fig.2. Except for the main specular peak there are also other maxima coming from fulfilling the interference condition, namely the phase difference $(2\pi/\lambda)\{\sqrt{(n^2 - \cos^2\theta_1)} + \sqrt{(n^2 - \cos^2\theta_0)}\}\Lambda = 2\pi m$ for $m=1,2,3$. λ , n , θ_1 and θ_0 are the wavelength, mean refractive index, incident and takeoff angle, respectively. The 2θ positions of the non-specular maxima agree with the positions of the specular Bragg maxima as they come from the same periodicity Λ . However, in the non-specular case this periodicity manifests itself by the constructive interferences of the small-angle scattering from structural inhomogeneities associated with the individual interfaces, i.e. of the scattering from the laterally imperfect rough interfaces [4], and by the interferences of the coherent scattering from the atoms in the sublayers forming ML if they are amorphous [5]. The intensities of the non-specular maxima expressed in the percents of the specular peak intensity in the same scan are also shown in Fig.2. They decrease for the 773K/5s annealing, similarly as σ does, but as the sublayers are still amorphous [1] this fact need not necessarily mean the decrease of the geometrical roughness of the interfaces. It may be connected with the relaxation ordering in the amorphous regions, too. On the other hand, the increase of these relative intensities after the 773K/20s annealing is connected with the increasing interfacial roughness as the crystallization in the sublayers begins [1] and a higher atomic order is clearly established. The observed formation of the crystalline grains may contribute to the increased roughness. The absence of the specular 2nd Bragg maximum after the 773K/20s annealing manifests itself also in the disappearance of the 2nd non-specular maximum. Similar results were obtained for the 2θ scans with other angles of incidence.

In Fig.3 some results of the ω -scans (rocking curves) are shown. Except for the central peak, in this case the specular 2nd Bragg maximum, the width of which depends basically on the mosaicity of the ML stack, there are broad non-specular wings stretching symmetrically in both directions from the central peak. These wings are due to the ML enhancement of both scattering mechanisms mentioned in the previous paragraph and in the reciprocal space may be viewed as the intensity streaks around the specular Bragg points along the in plane component of the wave vector with respect to the surface [4]. In fact, we crossed these streaks corresponding

Fig.1. - The specular reflectivity scans (full lines) with their simulations (dotted lines). See also the text.

Fig.2. - The 2θ scans for the angles of incidence θ_i .

Fig.3. - The ω -scans with the detector fixed at 2θ values.

to different Bragg orders when getting the non-specular maxima in the 2θ scans. We checked that there are no wings when the detector is fixed out of the 2θ position for a specular Bragg maximum. Small dips, symmetrically placed around the central peak in the positions where θ_1 or θ_0 is equal to the 1st Bragg angle, may be seen on the wings. Such features, either dips or small peaks, have already been observed [4,5]. The presence of dips in our case implies that standing waves excited when θ_1 approaches the 1st Bragg angle reduce the non-specular scattering as the consequence of the extinction controlled reduction of the penetration depth. Out of this angle the penetration depth is increased, being limited only by the absorption. This mechanism prevails over the enhancement of the electric field and confirms that a good quality of the interfaces in the ML is preserved after the 773K/5s annealing. The dip arising when θ_0 equals to the 1st Bragg angle may be explained by the reciprocity theorem [5].

4. Conclusions

The specular reflectivity on the ML Bragg maxima shows no distinct changes up to the 773K/5s annealing, i.e. no progressive interdiffusion or mixing at the interfaces takes place. Instead, the interfacial roughness together with the ML period decreases, the character of the interfaces being not unambiguously established. However, a high degree of their perfection is evidenced by the ω -scans. After the 773K/20s annealing the 2nd Bragg maximum vanished but the presence of other Bragg maxima as well as the Kiessig fringes shows that ML character of the structure is preserved. Therefore the interdiffusion and mixing processes are not simple and involve also the interface displacements. At the same time their geometrical roughness increases as judged from the 2θ scans. After the 1023K/5s annealing the ML structure collapsed.

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