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Measurement of local stresses in the surface layers of multilayer structures by the null method using phase matching

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A new null method for measuring the values of residual mechanical stresses in surface layers has been developed, based on the measurement of the second harmonic generation signal caused by the anisotropy of polarization of the reflected IR laser radiation from the surface of the rotated sample under study with the subsequent passage of radiation through a nonlinear crystal oriented to the phase matching for the anisotropic polarization. Quantitative data of the amplitude of the second harmonic signal from the magnitude of the stresses were obtained. The residual stresses in the HgCdTe surface layer of the HgCdTe/CdTe/ZnTe/GaAs multilayer structure grown by the MBE method have been measured, which is equal to 0.0045 N. The observed fine structure of the second harmonic signal allows us to conclude about the complex structure of residual stresses.

Keywords: stresses, second harmonic, polarization, semiconductor structure.

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Important parameters in heteroepitaxial structures are the arising stresses and their relaxation after growth, which determine the crystal state of such structures, affecting the electronic parameters of the material and, consequently, the parameters of IR-detectors.

Various optical methods [1], X-ray diffraction [2,3] allow us to determine the residual deformations in the volume of the investigated materials. The second harmonic generation (SHG) method makes it possible to obtain express information on the state of the crystal lattice of the near-surface layers in a complex multilayer structure (013) HgCdTe/CdTe/ZnTe/GaAs, grown by molecular-beam epitaxy [4,5] on the basis of comparison of experimental data and model azimuthal dependences of the SHG signal in an ideal crystal, which require numerical calculations [6]. Our investigations [4–7] showed that there are residual deformations in the substrate and layers.

The previously developed method based on the registration of signal characteristics of the second harmonic of the probing laser IR-radiation excited in a nonlinear crystal using phase matching makes it possible to control weak deformations in the volume of both amorphous and crystalline media [8]. This null-method allowed us to obtain information on the qualitative distribution of volume strain in GaAs and Si wafers with high sensitivity, localization and high speed. However, this method demonstrated only high sensitivity of strain detection when using phase matching, since the question of the choice of the orientation of the laser radiation polarization relative to the crystallophysical axes of the investigated samples and, consequently, the question of the magnitude and orientation of stresses remained outside the scope.

In the present work we report experimental results of weak strain measurements in the near-surface region of amorphous Cr and HgCdTe layers using a new null-method. We used the high sensitivity due to phase matching inherent in the [8] method, and while rotating the sample in a plane perpendicular to the wave vector of the incident laser radiation, we passed the reflected laser radiation through a nonlinear crystal oriented to synchronism for the polarization perpendicular to the laser radiation. Thus, from the characteristics of the SHG signal of the non-linear crystal (amplitude and orientation angle), we were able to obtain a number of quantitative parameters in the investigated samples depending on the applied stresses.

In the null method we have developed, linearly polarized IR-radiation from a picosecond pulsed-periodic YAG:Nd-laser with a wavelength of 1.064 μm is incident on the rotating sample under investigation along the normal. The infrared laser radiation reflected from the rotating sample is passed through a nonlinear crystal made of $LiIO_3$, oriented on an $oo-e$ type synchronism for an excitation polarization perpendicular to the original polarization of the laser radiation. An KS-17 KS filter is placed in front of the sample to suppress the weak reflected second harmonic signal generated by the incident laser beam in the sample. Residual deformations cause the linear polarization of the incident wave to change to a weakly elliptical polarization of the reflected wave. The ellipticity of the wave after interaction with the crystal is determined by the refraction

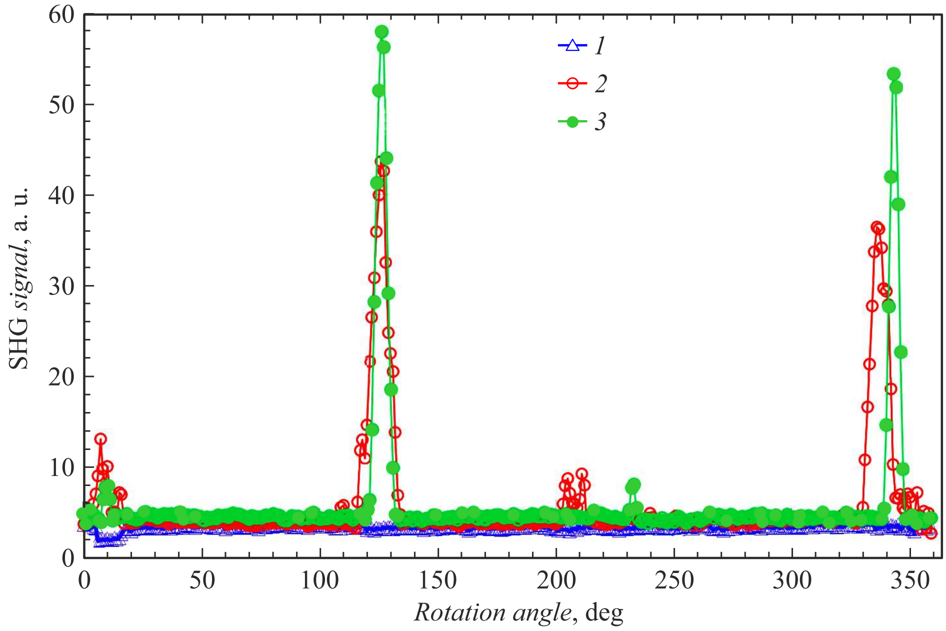

Figure 1. Angular sweep of the experimental values of the PMT signal when -laser IR-radiation reflected from the surface of an amorphous chromium film on glass passes through a nonlinear crystal. *1* — no load, *2* — edge load 10^{-2} N, *3* — edge load $\sim 1.5 \cdot 10^{-2}$ N. The solid lines show the trend of the experimental values.

index difference and depends on the magnitude of the stresses at a given point, which leads to the generation of the second harmonic at the output of the nonlinear crystal, the signal of which is recorded by a photomultiplier tube (model FEU 130) in a measurement path similar to that described in [4].

The rotation of the sample, fixed normal to the end of the shaft with respect to the polarization of the laser beam, was carried out by a stepper motor. The position error of the laser spot relative to the center of the stepper motor rotation axis was ~ 1 mm.

The SHG signal amplitude was measured on a sample of amorphous chromium film deposited in vacuum on a glass plate. The size of the square sample was 18×18 mm. The thicknesses of the glass plate and chromium film were 2 mm and 3.2 μm, respectively. The center of the specimen rested on a point support, the edge load was applied at one vertex of the square with static confrontation of the opposite vertex. Fig. 1 shows the FEU-130 sweep signals from an amorphous chromium film on glass.

For the sample in the free state (without edge load), only the noise of the measuring path of the bench is observed in the FEU-130 signal, which corresponds to the zero SHG signal (curve *1* in Fig. 1). When an edge load $\sim 1 \cdot 10^{-2}$ N (curve *2* in Fig. 1) and $1.5 \cdot 10^{-2}$ N (curve *3* in Fig. 1) is applied perpendicular to the specimen surface, narrow peaks of the FEU-130 signal are observed in the FEU-130 sweep, the magnitude of which is related to the anisotropy and stress differences in opposite directions. It should be noted that the shear force, which provides the anisotropy of the resulting stresses in the chromium film, is directed towards the point of the applied edge load. Indeed, it is known that some isotropic materials become optically anisotropic when stressed under loading, as crystals with temporal birefringence. The observed SHG peaks measured using the null method are related to the occurrence of elliptical polarization in the reflected IR-radiation upon loading, which is determined by the optical anisotropy of the chromium film due to the occurrence of stresses. The SHG signal curves are reproduced by repeating the sample rotation. The dependence of the peak amplitude on the magnitude of the applied load is almost linear:

$$Y = 3.5 \cdot 10^5 X + 428.5, \tag{1}$$

where Y — SHG signal magnitude, X — stress [N]. When the stress was removed, the measured FEU-130 signal corresponded to the noise values of the measuring path, coinciding in magnitude with the initial measurements, which indicates the absence of residual deformation.

The SHG signal from the $Hg_{1-x}Cd_xTe$ heteroepitaxial structure near-surface layer using the developed null method was investigated on the 1MCT180130 sample [7] . The composition of the investigated near-surface outer graded wide band gap layer $x \sim 0.47$, where x — molar fraction of CdTe.

Figure 2 shows a sweep of the FEU-130 signal (SHG signal) from the surface of sample 1MCT180130 in the presence (triangles) and in the absence (circles) of a polaroid in front of the FEU-130.

The insert shows the fine structure of the FEU-130 waveform near the rotation angle 90°. Two narrow maxima of the FEU-130 signal are observed. The first

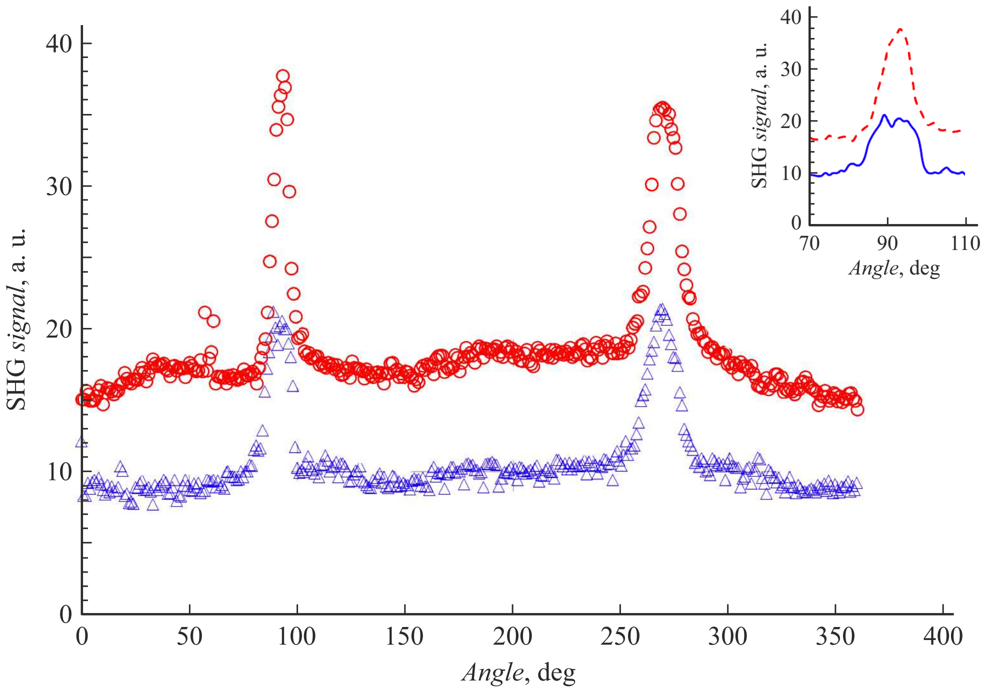

Figure 2. Sweep of the experimental values of the FEU-130 signal for sample 1MCT180130. Triangles — in the presence of a polaroid in front of the FEU-130, circles — in the absence of a polaroid in front of the FEU-130.

maxima (left) are observed in the interval 90–95°. The distance between the first and second maxima is $\sim 180°$ (stepper motor rotation angle). The maxima have an asymmetric shape, which can be seen more clearly in the inset to Fig. 2. The non-symmetrical waveform is due to the complex distribution of stresses in the measured point. The uniform signal between maxima is taken as the zero reference level, the value of which is higher in measurements without polaroid than in measurements with polaroid (as well as in maxima). The average stress value corresponds to an applied load of 0.0045 N according to formula (1). It should be noted that the amplitude of the SHG signal in our method is determined by weak induced birefringence, i.e., it depends on the difference of refraction indices $n_o - n_e$ for ordinary (n_o) and extraordinary (n_e) beams. When there is no deformation in the amorphous matter or cubic crystal $n_o = n_e$, and the SHG signal is zero. An estimated calculation of residual stresses using the procedure presented in [9] allowed us to determine their magnitude in -21 ± 2 MPa. Thus, this value can be compared to the SHG signal amplitude as a value for the calibration curve to be developed. The residual stresses obtained earlier by X-ray diffractometry in similar structures were ~ 2 MPa [10], which is an order of magnitude less than the calculated value. This is due to the thickness of the measured region, which for X-ray diffraction is a few micrometers and captures mainly the region of the HgCdTe layer of constant composition of the order of 0.2–0.3 molar fractions. In our case, the thickness of the measured region is a hundredth of a micrometer and captures the near-surface layer of HgCdTe in which the composition varies with thickness (for the surface sample under study, the composition corresponds to 0.47 molar fractions). The observed features of the maxima and their different shapes correspond to the orientation of the sample in the investigated region, at which a perpendicular polarization component to the polarization of the incident laser radiation appears in the reflected radiation. The appearance of the SHG signal corresponds to the direction of polarization of the incident laser radiation along the base slice of the specimen, in our experiments in the plane (110), which indicates the orientation of stresses in this direction. The obtained experimental dependences indicate that practically the entire SHG signal in the maxima of the FEU-130 sweep corresponds to the weak anisotropy of the crystal structure of the investigated near-surface layer of the sample with thickness ~ 200 nm associated with its weak deformation. The reason for the narrow directivity of the SHG signal is due to the high angular sensitivity of the phase matching employed in the null method used.

The observed differences in the amplitude and shape of the SHG signals when the sample is rotated by 180° are due, apparently, to the formation of different elliptically polarized reflected IR-radiation when the laser beam probes close but different local regions of the 1MCT180130 sample surface due to de-centering of the probing laser beam.

The observed fine structure of the SHG signal is related to the complex structure of deformations in the near-surface layer of the sample due to the presence of disoriented micro-parts [5]. Thus, the obtained data indicate the effect of residual stresses on the polarization characteristics of the reflected radiation, which leads to the appearance of second harmonic signals obtained using a new, more advanced null method.

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Conflict of interest

The authors declare that they have no conflict of interest.

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