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PIEZOSPECTROSCOPIC ANALYSIS OF MECHANICAL STRESSES IN Si_3N_4 AND AlN IRRADIATED WITH HIGH-ENERGY BISMUTH IONS

Depth-resolved Raman piezospectroscopy was used to study residual mechanical stress profiles in polycrystalline silicon and aluminum nitrides irradiated with 710 MeV bismuth ions to fluences of 1×10^{12}, 2×10^{12}, and 1×10^{13} cm^{-2}. It was found that stress fields of opposite signs are formed in the irradiated Si_3N_4 layer, separated by a buffer zone located at a depth coinciding with the thickness of the sample layer, amorphized at high ion fluences due to multiple overlapping of track regions. At great depths, tensile stresses witch magnitude reaches their maximum value in the region of the end of the ion range are detected. In contrast to Si_3N_4, radiation-stimulated changes in mechanical stresses in AlN were within the measurement error throughout the entire thickness of the irradiated layer, except of the near-surface region. The observed effect is associated with the different structural sensitivity of silicon and aluminum nitrides to high-density ionization - the formation of amorphous latent tracks in Si3N4 and their absence in AlN.

Key words: silicon nitride, aluminum nitride, swift heavy ions, Raman spectra, mechanical stress, piezospectroscopy.

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Жоғары энергиялы ауыр иондармен сәулеленген Si_3N_4 және AlN механикалық кернеулерді пьезоспектроскопиялық талдау

Кеңістіктік ажыратылымдылығы бар Рамандық пьезоспектроскопия әдістері 1×10^{12}, 2×10^{12} және 1×10^{13} $см^{-2}$ флюенске дейін энергиясы 710 МэВ жоғары энергиялы висмут иондарымен сәулеленген бұл жұмысьа поликристалды кремний мен алюминий нитридтеріндегі қалдық механикалық кернеулердің профильдерін зерттеу үшін пайдаланылды. Сәулеленген Si_3N_4 қабатында трек аудандарының бірнеше рет қабаттасуы есебінен иондардың жоғары флюенсі кезінде аморфизацияланған үлгі қабатының қалыңдығына сәйкес келетін тереңдікте орналасқан тректік аймақпен бөлінген түрлі белгілердің кернеу өрістері қалыптасатыны зерттеу нәтижесінде анықталды. Үлкен тереңдікте созылу кернеулері тіркелетіні көрсетілді, олардың деңгейі иондар жүгірісінің соңындағы аймақтағы максималды мәнге жететіні анықталды. Si_3N_4 - тен айырмашылығы, AlN - дегі механикалық кернеулердің радиациялық - ынталандырылған өзгеру деңгейі беттік аймақты қоспағанда, сәулеленген қабаттың бүкіл қалыңдығы бойынша өлшеу қатесінің шегінде болды. Байқалатын әсер кремний мен алюминий нитридтерінің жоғары тығыздықтағы ионизация әсеріне әртүрлі құрылымдық сезімталдығымен –

Si_3N_4 - те аморфты латентті тректердің түзілуімен және олардың AlN-де болмауымен байланысты деген қортынды жасалды.

Түйін сөздер: кремний нитриді, алюминий нитриді, жылдам ауыр иондар, комбинациялық шашырау спектрлері, механикалық кернеулер, пьезоспектроскопия.

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Пьезоспектроскопический анализ механических напряжений в Si_3N_4 и AlN, облученных тяжелыми ионами высоких энергий

Методы рамановской пьезоспектроскопии с пространственным разрешением в данной работе были использованы для изучения профилей остаточных механических напряжений в поликристаллических нитридах кремния и алюминия, облученных высокоэнергетическими ионами висмута с энергией 710 МэВ до флюенсов 1×10^{12}, 2×10^{12} и 1×10^{13} см$^{-2}$. По результатам установлено, что в облученном слое Si_3N_4 формируются поля напряжений различного знака, разделенные буферной зоной, находящейся на глубине, совпадающей с толщиной слоя образца, аморфизованного при высоких флюенсах ионов за счет многократного перекрытия трековых областей. На больших глубинах регистрируются растягивающие напряжения, уровень которых достигает максимального значения в области конца пробега ионов. Показано что, в отличие от Si_3N_4, уровень радиационно-стимулированного изменения механических напряжений в AlN был в пределах ошибки измерений по всей толщине облученного слоя, только за исключением приповерхностной области. Наблюдаемый эффект связывается с разной структурной чувствительностью нитридов кремния и алюминия к воздействию ионизации высокой плотности – образованием аморфных латентных треков в Si_3N_4 и их отсутствием в AlN.

Ключевые слова: нитрид кремния, нитрид алюминия, быстрые тяжелые ионы, спектры комбинационного рассеяния, механические напряжения, пьезоспектроскопия.

Introduction

The effect of heavy charged particle irradiation in solids, as a rule, is accompanied by generation of mechanical stresses in the irradiated layer, which, in own turn, affect the defect formation processes. Assessing the stress level and establishing the relationship between the nature of radiation damage and stresses are very important for a correct description of the evolution of a defect structure under various experimental conditions. First of all, such problems were considered for low-energy ion implantation into semiconductor materials. These issues have been studied to the least extent for high-energy $(E > 1\, MeV/amu)$ion irradiation, the possibilities of which for solving practical problems are beginning to be in demand only recently. Such research are, in particular, of considerable interest for predicting the long-term radiation stability of ceramic and oxide materials, showing promise for nuclear applications, against fission fragments impact. The main experimental methods for measuring mechanical stresses are usually the following - curvature measurement method, X-ray, electron and neutron diffraction, electromagnetic and ultrasonic methods [1].

The stress level in a variety of materials can be determined using an approach based on the use of the piezospectroscopic effect. As is known, this effect connects the changes in optical absorption, luminescence, or Raman scattering spectra with the magnitude of mechanical stresses (see, for example, [2-4]). As known, in general, the change in the radiation frequency with stress level can be expressed as [4]:

$$\Delta\nu = \Pi_{ij} \times \sigma_{ij}, \qquad (1)$$

where Π_{ij} are the so-called piezospectroscopic coefficients, and σ_{ij} are the stress tensor components. Piezospectroscopic coefficients are determined using of appropriate calibrations, during which a known stress is applied to the material and the corresponding change in frequency relative to the unstressed state is registered [4,5]. In contrast to the methods listed above, which provide information on the stresses in the entire investigated volume of the sample, piezospectroscopy makes it possible to determine the stress level with a spatial resolution up to several hundred nanometers, that is especially important in the case of irradiation of materials with ions of fission fragment energies for which the projected range does not exceed 10 microns. In [6], this approach was used to measure the stress profiles in Si_3N_4 irradiated with high-energy xenon and bismuth ions. As is known, silicon nitride is the only nitride ceramic in which latent tracks of swift heavy ions have been revealed [7-16]. At the same time, no tracks were detected in aluminum nitride even at very high specific ionization energy losses [17]. This paper presents the results of a comparative analysis of residual mechanical stress profiles in AlN and the previously obtained data on Si_3N_4 [6] – nitride ceramics with different structural responses to the high-energy heavy ion impact.

Materials and Methods

The objects of study of this work were polycrystalline silicon and aluminum nitrides samples manufactured by MTI Corporation. The grain sizes ranged from several hundred nanometers to several microns. The samples were irradiated with 710 MeV ^{209}Bi ions at room temperature to fluences of 1×10^{12}, 2×10^{12}, and 1×10^{13} cm^{-2}. The irradiation experiments were carried out at the U-400 cyclotron of the FLNR JINR (Dubna, Russia). According to calculations, the projected range of bismuth ions in AlN was 29.8 μm.

Irradiated samples were studied by Raman spectroscopy using Solver Specrtum, NT-MDT laser confocal scanning microscope. Raman spectra excited at a wavelength of λ=473 nm were recorded on the edge of polished sample along the ion trajectory with a spatial resolution of ~ 1 μm. The measurement time was optimized for maximum intensity and was 60 seconds.

Results and Discussion

Raman spectra of intact silicon and aluminum nitride specimens excited at a wavelength 473 nm are shown in figure 1.

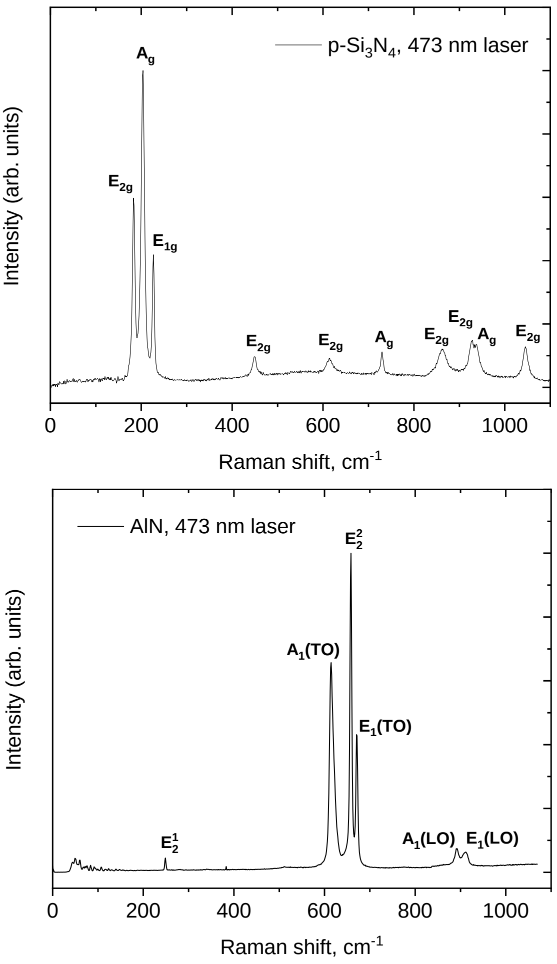


Figure 1 – Raman spectra of initial Si_3N_4 and AlN polycrystals.

Without stopping on the nature of the vibrational modes represented by the corresponding peaks in the Raman spectra studied in detail in [18-23], we note that in piezospectroscopic studies, the most often used is the measurement of the position of the peaks at 862 cm^{-1} (Si_3N_4) [24] and 658 cm^{-1} (AlN) [25].

The piezospectroscopic coefficients for these peaks are 2.22 GPa/cm^{-1} and 2 GPa/cm^{-1}, respectively [24, 4]. The 658 cm^{-1} line in AlN is most significant since because exhibits the greatest stress sensitivity, resulting in the shortest acquisition time for Raman measurements and the highest signal-to-noise ratio. Another advantage of using this band is that the frequency of the E_2 (high) mode, in contrast to the A_1(LO) phonon mode, does not depend on the concentration of free carriers; therefore, only the stress will affect this phonon frequency for measuring the residual stress. All values of the peak frequencies in the Raman spectra in aluminum nitride known from the literature are presented in table 1.

Table 1. Experimental data on Raman spectra in AlN

Position	248.69	612.87	658.26	671.12	892.52	910.81	cm^{-1}
Phonon mode	E_2^1	$A_1(TO)$	E_2^2	$E_1(TO)$	$A_1(LO)$	$E_1(LO)$	

Figure 2 shows the changes in the position of the 658 cm^{-1} (AlN) line, which were measured at a depth of 5 µm. First of all, it should be noted that mechanical polishing of AlN introduces defects, which leads to both broadening and a shift of the main peak relative to the initial position from 658.26 cm^{-1} to 657.78 cm^{-1}. To take this circumstance into account, the frequency value measured at a distance of ~ 100 microns from the surface was taken as the initial position of the peak, which significantly exceeds the depth of the irradiated layer. As can be seen from the part of the spectrum shown in the inset, irradiation with bismuth ions leads to the signal intensity decrease and the peak broadening, which is associated with the general disordering of the crystal lattice. In addition, a shift of the peak towards lower frequencies is also observed. A negative value of $\Delta\nu$ indicates that compressive mechanical stresses are detected at a given depth of the irradiated layer of the aluminum nitride sample.

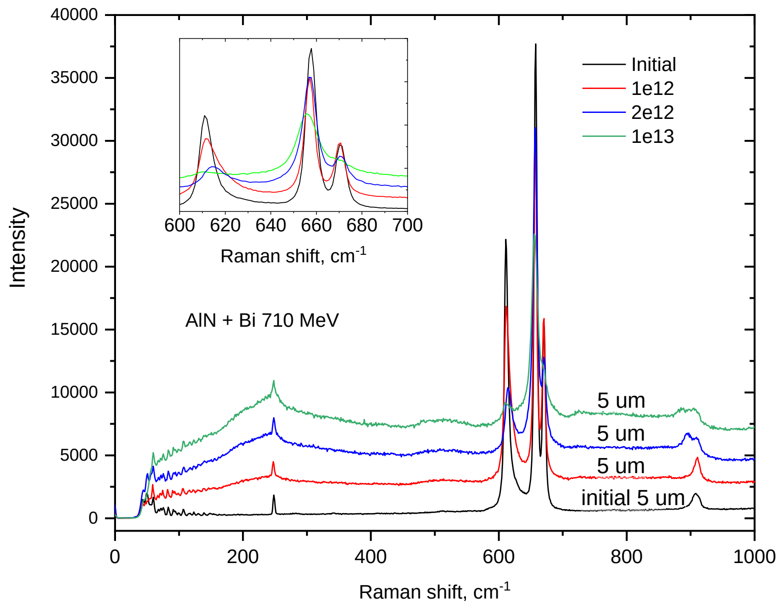
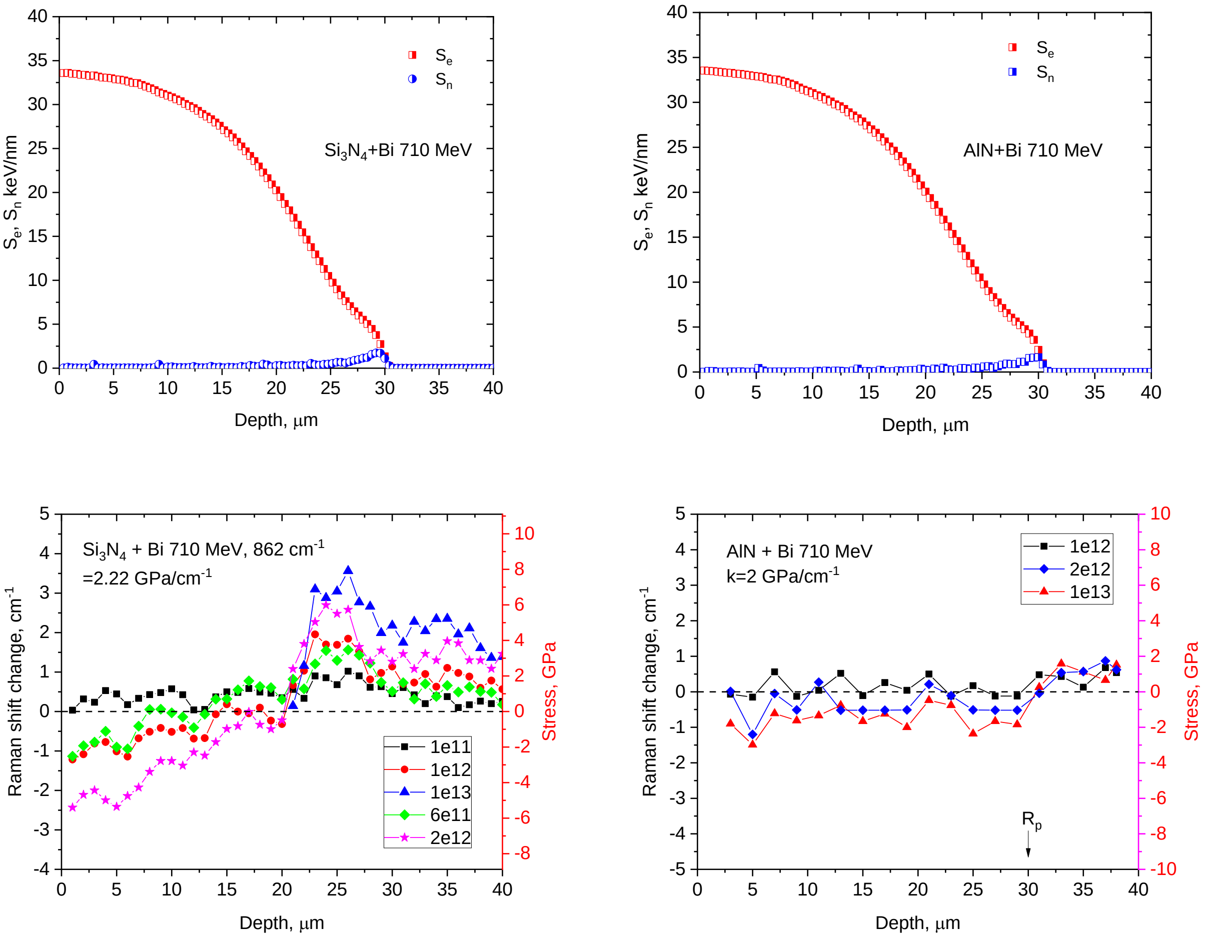


Figure 2 – Raman spectra measured at a depth of 5 µm from the surface of AlN irradiated with bismuth ions to various fluences. The inset shows the spectral region near the 658 cm^{-1} line.

The generation of compressive stresses in ceramics irradiated with high-energy ions was previously revealed not only in Si_3N_4 [6], but also in oxides Al_2O_3 and ZrO_2:Y [26-28]. In all these materials are formed latent tracks as disordered regions around ion trajectories, both amorphized (completely or partially) and retaining the crystalline structure. In all cases, an important feature of the tracks is their reduced density in comparison with the density of the surrounding matrix. Their accumulation with an increase in the ion fluence leads to a decrease in the density and, as a consequence, to an increase in the volume of the sample layer in which the tracks are formed, which is the source of compressive stresses due to the difference between the volumes of the irradiated and unirradiated parts of the material. The appearance of stresses in AlN, in which the tracks are not detected by transmission electron microscopy, indicates that defects formed in elastic collisions also lead to mechanical stresses but much lower magnitude in comparison to silicon nitride.



(a) (b)

Figure 3 – Variation of the spectral position of the 862 cm^{-1} (Si_3N_4) - a) and b) - 658 cm^{-1} (AlN) lines via the Bi ion fluence and the depth of the irradiated layer

Figures 3a and 3b show the change in spectral position of lines used for evaluation of stress level in the silicon (a) and aluminum (b) nitrides irradiated with bismuth ions with respect to the values for the non-irradiated material. The corresponding piezospectroscopic coefficients for frequencies 862 cm^{-1} (Si_3N_4) and 658 cm^{-1} (AlN) according to [24,4] are 2,22 and 2,0 GPa. As can be seen, stresses in AlN are detected only at the highest fluence of bismuth ions - 1×10^{13} cm^{-2}, which clearly indicates the differences in the formation of structural defects in these ceramics. The data shown in fig. 3b, do not also allow drawing a conclusion about the correlation of the stress profile in AlN obtained for a fluence of 1×10^{13} cm^{-2} with the profiles of ionization and nuclear energy losses. Although tracks in aluminum nitride are not observed, it cannot be ruled out that a high level of specific ionization energy losses can affect the evolution of the defect structure, which requires further detailed studies.

Conclusions

Polycrystalline Si_3N_4 and AlN irradiated with high-energy (710 MeV) bismuth ions to fluences of 1×10^{12}, 2×10^{12}, and 1×10^{13} cm^{-1} were studied by Raman piezospectroscopy with spatial resolution about one micron. In the irradiated silicon nitride layer, stress fields of different signs are formed, separated by a buffer zone located at a depth coinciding with the thickness of the sample layer, amorphized at high ion fluences due to multiple overlapping of track regions. Deeper, tensile stresses are registered and reach its maximum in ion end of range. Compared to Si_3N_4, mechanical stresses in AlN are found to be out of experimental inaccuracy only at a bismuth ion fluence of 1×10^{13} cm^{-2}.

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