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INFLUENCE MECHANICAL TREATMENT, ELECTRICAL CURRENT, CHANGING OF DEFECT STRUCTURE ON INELASTIC CHARACTERISTICS OF Si + SiO₂ WAFER-PLATES, GeSi AND SiO₂

The method is created for nondestructive control of structure defects in semiconductors plates, which determines the integral density of structure defects, their distribution and the broken layer from the internal friction difference of free elastic vibrations on neighboring harmonics f_1 and f_2 . The dislocation density and the broken layer are measured from the curve of dependence for plates Si + SiO₂. The results of influencing of direct and variable electrical current at simultaneous influence of ultrasonic deformation on internal friction and the elastic module of crystal GeSi after cutting and polishing were studied. The decreasing of elastic module and the raise of internal friction was obtained under condition when the critical value of the electrical current is exceeded.

Introduction

Internal friction (IF) depends essentially from nature and density of structure defects. IF depends on different relaxation processes, that connected with plate defect structure. The local heterogeneities are the factor, which gains strength the influence of direct current I on state of impurities in GeSi. The measuring of amplitude dependent IF (ADIF) can be used as the highly sensitive control method of nanoplastic deformation GeSi crystals. The measuring of ADIF allows to fix the moment of tearing away of dislocation segments L_C from stops. Therefore the purpose of this work was a study of influence of electrical current I on tearing away from the stops of dislocation segments L_C during simultaneous ultrasound deformation σ_{US} GeSi crystals with orientation [111] after mechanical cutting and polishing.

Experiment

For measuring of the temperature dependences of IF $Q^{-1}(T)$ and elastic module $E(T)$ of mono crystal GeSi of orientation [111] with sizes $2 \times 2 \times 16$ mm³ with the dislocation density $N_d \approx 10^8$ m⁻² after cutting and polishing the methods of complete

piezoelectric oscillator on frequency $f \approx 118$ kHz and bending resonance vibrations on frequency $f \approx 1$ kHz during alternative deformation $\varepsilon \approx 10^{-6}$ in vacuum $P \approx 10^{-3}$ Pa were used [1-3]. The measuring error of IF was $\Delta Q^{-1}/Q^{-1} \approx 10\%$ and the elastic module relative changing was $\Delta E/E_0 \approx 0,1\%$. The temperature dependencies of IF $Q^{-1}(T)$ and elastic module $E(T)$ experimentally simultaneously were measured, that are allowed more exactly to determine the critical deformation amplitude \mathcal{E}_c^* , at which the dislocation segments average length \bar{L}_C tear off from stops — point defects. The high exactness of the elastic module E measuring, especially its relative changing, is conditioned the high exactness of determination resonance frequency specimen oscillation f .

The method of determining the integral density structure defects mono crystal discs Si + SiO₂ — plates, which were element of metal- dielectric-semiconductor (MDS) — structure at manufacturing the big integral schemes (BIS) for industrial express operation technology control of structure perfection of multilayer epitaxial structures and the increase of reliability valid p-n crossing was created.

The method and the machinery were developed for nondestructive for the control of the structure defects semiconductors plates, which permit on IF difference ΔQ^{-1} free elastic vibrations on neighboring harmonics f_1 and f_2 to determine the integral density of structure defects N_d , their distribution and the broken layer h_b . It is necessary take into account attenuation, arising as a result of absorption of elastic mechanical vibrations energy – IF and of fastening of plate at examination the vibrations of disks with recording of their structure imperfections. In developed acoustic machinery for operation control at manufacture of semiconductor devices the research plate - disk with thickness $h \gg 500 \mu\text{m}$ and diameter $D = 100 \div 200 \text{ mm}$ fasten off in centre and electrostatic method in it are excited the elastic umbrella vibrations. The measuring of difference IF $\Delta Q^{-1} = Q^{-1}_1 - Q^{-1}_2$ on different frequencies f_1 and f_2 takes to minimum measuring error. The method and the machinery were developed – machinery, that generate elastic bending vibrations in disk for study of free elastic vibrations in disks Si + SiO₂ – plates, which are fastened in centre. The principle of machinery work is consisting. The variable voltage U gives to the disk and capacitive gage, which is on distance $d = 50 \div 100 \mu\text{m}$. Resonance elastic bending free vibrations, umbrella type, are excited at coincidence of frequency electrical value with own frequency $f \approx 1 \text{ kHz}$. The capacitive gage is for generation and receiving of free vibrations of disk. Own frequency f vibrations of disk and IF are determined with help electronic frequency meter and amplitude discriminator, which makes way for the value in given amplitude interval from A_1 to A_2 .

Results and discussion

The research results of elastic US wave's absorption are on imperfections of crystal structure after **mechanical, temperature and radiation treatments**. There are studied the kinetics of structure defects annealing. This method may be used for the control of structure defects Si, Si + SiO₂, GaAs plates after technological mechanical, temperature and radiation treatments. The measuring results are presented on fig. 1 and fig. 2. The dependence of IF difference ΔQ^{-1} in Si + SiO₂ plate with orientation (111), diameter $D \approx 60 \text{ mm}$ and thickness

$h \approx 500 \mu\text{m}$ on neighboring harmonics f_1 and f_2 from the dislocation density N_d is presented on fig. 1. The integral dislocation density after different treatments changes in limits $N_d = 10^2 \div 10^5 \text{ cm}^{-2}$ on fig. 1.

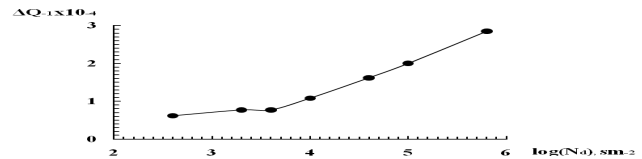


Fig. 1. Dependence of internal friction difference ΔQ^{-1} of Si + SiO₂ plate with orientation (111) from the dislocation density N_d

IF difference $\Delta Q^{-1} = Q^{-1}_1 - Q^{-1}_2$ on different frequencies f_1 and f_2 deposit on vertical axis, and on horizontal – dislocation density N_d . The dislocation density N_d was determined by standard etch-pit method for construction of the calibration graphic. Specimens Si + SiO₂ were washing in alcohol and in distilled water before etching. The using of method layer-specific polishing, which alternate with chemical etching at $T = 290 \text{ K}$ in Sirtl etching agent $\text{Cr}_2\text{O}_3 : \text{H}_2\text{O} : \text{HF} = 1 : 2 : 3$, allow to observed, that US treatment (UST) hardens the layer, which ranges from surface on depths $h \gg 100000 \text{ nm}$. The etch pits for vacancy clusters V-V-V were disappeared on this depth. The etch pits were oval, rounded form, which connected with presence in near-surface crystal layers vacancy clusters, coagulants of diffuse impurities on surface Si specimens after UST. The dependence of difference IF ΔQ^{-1} Si plate with diameter $D \approx 60 \text{ mm}$ and thickness $h \approx 500 \mu\text{m}$ on neighboring harmonics f_1 and f_2 from the broken layer depth h_b , that was created by the polishing of Si plates with help of diamond pastes with different grain was studied. The broken layer depth h_b were measured by roentgen method. The IF difference ΔQ^{-1} Si plate with diameter $D \approx 60 \text{ mm}$ and thickness $h \approx 500 \mu\text{m}$ on neighboring harmonics f_1 and f_2 linear increases with the broken layer depth h_b can see from fig. 2. The broken layer depth is presents on curve fig. 2 and was changing in limits $h_b = 1000 \div 3000 \text{ nm}$.

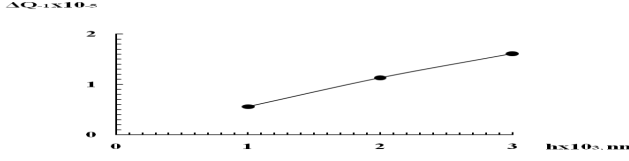


Fig. 2. Dependence of internal friction difference ΔQ^{-1} of Si + SiO₂ plate with orientation (111) from the broken layer h_b

The solution of differential equation of round disks vibrations under action of periodical external force allow to get circular resonance frequency of disc free vibrations [1]:

$$\omega = \frac{\beta}{R^2} \sqrt{\frac{D}{\rho h}}. \quad (1)$$

It is necessary take into account that the attenuation arise as a result of the attaching of plate and absorption of mechanical vibrations energy – IF at examination of resonance frequency ω . IF обусловлено different relaxation processes, that connected with plates defect structure. The account of dispersion of elastic mechanical vibrations energy of Si+SiO₂ plate on the structure defects results in expression for frequency of disk free vibrations [1]:

$$\omega = \sqrt{\frac{D\beta^2}{\rho h R^4} - 2\pi^2 \left(\frac{Q^{-1}}{T} \right)^2}, \quad (2)$$

where cylindrical inflexibility of disc D , determined through the elastic module E , plate thickness h and Poisson coefficient μ :

$$D = \frac{Eh^3}{12(1-\mu)^2}, \quad (3)$$

$$\mu = \frac{\left(\frac{1}{2} V_{\downarrow}^2 - V_{\leftrightarrow}^2 \right)}{\left(V_{\downarrow}^2 - V_{\leftrightarrow}^2 \right)} \quad (4)$$

where V_{\downarrow} — longitudinal velocity US of elastic wave, V_{\leftrightarrow} — quick shear velocity US of elastic wave. β — is dimensionless coefficient, the value of which depends on the number of key circumferences, ρ — the specific density of plate, R — the disk radius, Q^{-1} — IF, T — the disk vibrations period. The measuring of vibrations resonance frequency f allow to determine the elastic module E plate (without attenuation recording) on formula [1]:

$$E = \frac{12\rho\omega^2 R^4 (1-\mu)^2}{\beta^2 h^2}. \quad (5)$$

IF was measured in regime of free attenuate vibrations on formula [4,5]:

$$Q^{-1} = \frac{h \left(\frac{A_1}{A_2} \right)}{\pi N}, \quad (6)$$

where A_1 and A_2 — upper and lower discrimination level accordingly, N — number of free vibrations at amplitude decreasing from A_1 to A_2 .

The parameters of vibrations on neighboring harmonics f_1 and f_2 were measured for exclusion of contribution in IF, which depend on the attaching of plate — disk in centre. The changing from first harmonic f_1 to second f_2 without transposition the plate allow to except the instrumental errors, which connected with it fastening in centre. The measured losses of mechanical vibrations energy of plate consist from two summands: own IF plates Q_p^{-1} and IF instrumental losses Q_o^{-1} , which connected with fastening of plate. The difference of measured loss mechanical vibrations energy on two neighboring harmonics f_1 and f_2 is equal the difference of values IF on this frequencies $\Delta Q^{-1} = Q_1^{-1} - Q_2^{-1}$, and instrumental losses are reduced. There was a small value of IF background in SiO₂ $Q_o^{-1} \approx 2 \cdot 10^{-6}$ to $T < 385$ K. In comparison of results on the same Si + SiO₂ plate on propose method $h_b = 7000 \pm 1000$ nm and with standard roentgen method $h_b = 6000 \pm 5000$ nm, at that productivity of experiment increase on two order.

The influence of the external fields (electrical and magnetic) is possible to explain on IF within the framework of string dislocation mechanism of elastic vibrations attenuation in GeSi, designing the motion of the charged dislocations in these fields. Will consider GeSi crystal with orientation [111], which periodic mechanical tension $\tau = \tau_0 e^{i\omega_0 t}$ is added to, and which is under the combined influence of external electric field \vec{E} and external magnetic field \vec{B} . If dislocation segment $\xi(x, y)$, that are vibrated under the act of tension τ , is charged, additional forces will operate on it $\vec{F}_e = e\rho(\xi)\vec{E}$ and $\vec{F}_m = e\rho(\xi)\left[\frac{\partial \xi}{\partial t}, \vec{B}\right]$, where $\rho(\xi)$ - is the distribution function of electrical charge density on the dislocation segment.

The system of equations, which describes the movement of the charged dislocation under act of the mechanical, electrical \mathbf{E} and magnetic \mathbf{B} fields, within the framework of string dislocation model acquires the following kind:

$$M \frac{\partial^2 \xi}{\partial t^2} = V_d \frac{\partial^2 \xi}{\partial x^2} - Q \frac{\partial \xi}{\partial t} + b\tau - b\tau_a - N_j \frac{\partial U}{\partial \xi} + e\rho(\xi)E + e\rho(\xi)\left[\frac{\partial \xi}{\partial t}, B\right], \quad (7)$$

$$\frac{\partial^2 \tau}{\partial y^2} - \frac{\rho}{G} \frac{\partial^2 \tau}{\partial t^2} = \rho b \frac{\partial^2}{\partial t^2} \left\langle \int_0^\infty \left[\int_0^l \xi(x) dx \right] N(l) dl \right\rangle, \quad (8)$$

where $\left(M \cdot \frac{\partial^2 \xi}{\partial t^2} \right)$ — inertial force, $M \approx \rho b^2$ — effective mass of unit of dislocation length, ρ — crystal density, b — Byurgers vector, t — time, $\left(V_d \cdot \frac{\partial^2 \xi}{\partial x^2} \right)$ — force which is conditioned effective strain of bended dislocation line, $V_d \approx G b^2$ for screw dislocations and $V_d \approx \frac{Gb^2}{(1-\mu)}$ for line dislocations, G — the displacement module, $\left(Q \cdot \frac{\partial \xi}{\partial t} \right)$ — the force of the viscid braking, Q — damping constant or coefficient viscid braking, $b\tau$ — external force, which operates on unit of dislocation length, $b\tau_a$ — force, which operates on unit of dislocation length and tensions is conditioned, which arise up as a result of potential pattern of crystalline grate, $\left(N_j \cdot \frac{\partial U}{\partial \xi} \right)$ — force, which operates on unit of dislocation length from the side of point defects, which co-operate with dislocation and located on dislocation line with the density $N_j(x)$, $U(\xi)$ — potential energy of co-operation, l — length of dislocation segment, $N(l)$ — the distributing function of dislocation segments for lengths, which is conditioned $N_j(x)$.

Practically interesting is a case, when the external electrical and magnetic fields are periodic,

that: $E = E_0 e^{i\omega_E t}$, $B = B_0 e^{i\omega_M t}$. When the variable electrical current density J^\sim is the same ultrasonic (US) frequency $\omega_j = \omega_{US}$. Examinations for influence of J^\sim on IF $Q^{-1}(J^\sim)$ on fig. 3 and elastic module $E(J^\sim)$ on fig. 4.

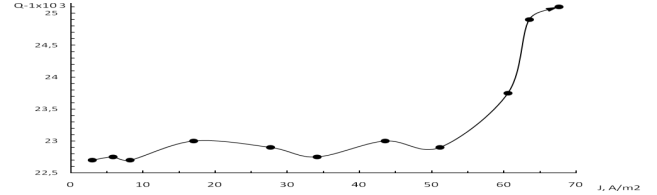


Fig. 3. Current dependence internal friction $Q^{-1}(J)$ in GeSi of orientation [111] with the dislocation density $N_d \approx 10^8 \text{ m}^{-2}$ after cutting and polishing from variable electrical current with frequency equal US frequency $\omega_i = \omega_{US}$ at $T = 296 \text{ K}$ and constant amplitude US deformation $\varepsilon_{US} \approx 2 \cdot 10^{-6}$

The variable electrical current J^\sim , which flowed through the sample, was in the phase with the voltage, which applied on piezoelectric vibrator to make ultrasonic strain σ_{US} .

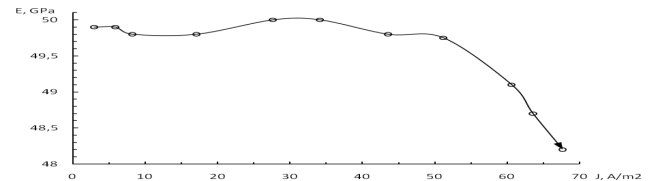


Fig. 4. Current dependence elastic module $E_{111}(J)$ of GeSi after cutting and polishing from variable electrical current with $\omega_i = \omega_{US}$ at $T = 296 \text{ K}$ and constant amplitude US deformation $\varepsilon_{US} \approx 2 \cdot 10^{-6}$

As one can see from fig. 5, for variable electrical current J^\sim when the critical value $J_c^* \approx 60 \cdot 10^3 \text{ A/m}^2$ realized the raise of IF $Q^{-1}(J^\sim)$ with simultaneous the decrease the value of elastic module $E(J^\sim)$ is observed at the subsequent increase of variable current.

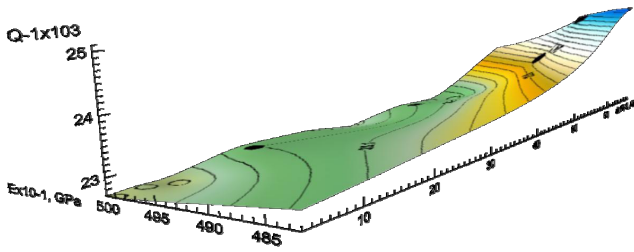


Fig. 5. Current dependence internal friction $Q^{-1}(J)$ and elastic module $E_{111}(J)$ (indicatory surface of inelasticity-elasticity body) GeSi after cutting and polishing from variable electrical current with frequency equal ultrasound frequency $\omega_1 = \omega_{US}$ at $T = 296$ K

For explanation of correlation $Q^{-1}(J)$ and $E(J)$ one can see equations (7) and (8). Compare the equations (7) and (8) with the experimental results on fig. 3, fig. 4 and fig. 5 made conclusion, that the influence performance of electrical current on IF $Q^{-1}(J)$ and elastic module $E(J)$ sharply increasing, when variable electrical current with frequency equal US frequency $\omega_J = \omega_{US}$.

The increasing of IF $Q^{-1}(J)$ is related to that the electrons of certain energy co-operate with the dislocation segments L_C , which vibrate under the action of US deformation ε_{US} , and assist in their tearing away from stops – point defects. The changing of absolute value US deformation amplitude ε_{0US} did not influence on quality motion of IF dependence from the electrical current $Q^{-1}(J)$, although here US deformation amplitude changed to $\varepsilon_{0US} \approx 4 \cdot 10^{-6}$, at the same time at temperatures to $T < 470$ K there was ANDIF. The diminishing of critical value current J_c^* at the increase of temperature testifies that the thermofluctuations vibrations of dislocation segments L_C grow with temperature T growth, which in same allows the lesser stream of electrons to tear away dislocations from stops. The changing of steepness of curves dependences IF from the current $Q^{-1}(J)$ during constant US deformation on fig. 3 can be consider as an indicator of tearing-fixing away of dislocations. An eloped direct and reverse motions of dependence IF from the current $Q^{-1}(J)$ represents the absence of irreversible changes of dislocation structure in the process of measuring IF. 3D atomic-force microscopy (AFM) of microstructure image of GeSi with orientation (111) is represented on fig. 6.

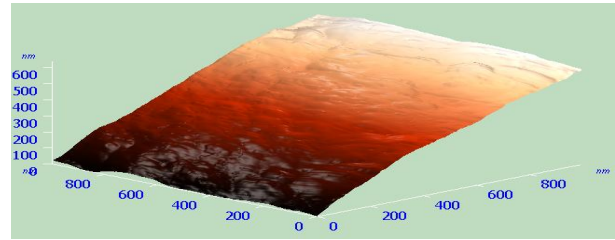
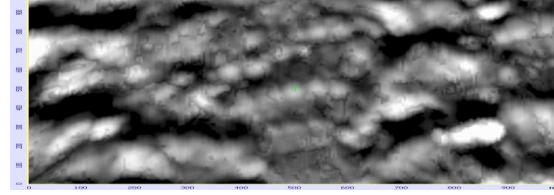


Fig. 6. 3D atomic-force microscopy of microstructure image of GeSi with orientation (111) (1x1 mkm)

The shallow dispersion fleshy plain surface, the intergrowth of little islands are observed, their configuration are rounded. The crystal sites have the fragmentary structure, which formed from weakly disorient one from another islands.

The single-row chain of impurity atoms under super-plane can't provide all relaxation of dislocation elastic field σ_i [6,7]. The impurity atoms Si accumulate in planes, which perpendicular to dislocation line, to full compensation of it's field σ_i [8-10]. The beginning of temperature dependence IF $Q^{-1}(J, \varepsilon, T)$ correspond to peripheral atoms Si going out of stopper atmosphere, that reduce attaching effect of dislocation segments L_C . The resorption of atmospheres around stoppers take place at further increasing of temperature. The increase of length between stoppers – the increasing of the middle effective dislocation segments length L_C begins after completion this process. When the resorption of impurity atmospheres near the stoppers finished, IF $Q^{-1}(J, \varepsilon, T)$ intensive decrease with temperature increasing. The values of critical amplitude deformation ε_c^* are stabilizing at temperature T , which correspond to full absence of fixing centres. It's showed, that inelastic Q^{-1} and elastic E characteristics are essentially depended from morphology of surface layer. 3D atomic-force microscopy (AFM) of microstructure image of Si + SiO₂ with orientation (100) is represented on fig. 7.

AFM are testified the presence of wafer-plate relief. There are many defects at film from abroad the influence of wafer-plate, which formed at increasing of islands. The structure defects don't to skin over in time at first growth stages at little film thickness $h \leq 1000$ nm.

Conclusion

Thus, the studying of structure defects influence on attenuation of elastic vibrations in Si + SiO₂ plates allow to estimate the degree of crystal structure perfection. The maximal error of definition of dislocation density N_d don't exceed 1,5÷2 twice, that in combination with small measured time allow to use the modify method for experiment. The integral dislocation density in limit $N_d = 10^6 \div 10^9$ m⁻² and broken layer depths in limit $h_b = 0 \div 40000$ nm were measured from calibrating curves for Si + SiO₂ plates. The measuring of IF background Q^{-1}_0 after different heat, mechanical, radiation treatments gives information about the changing of the thermoelastic strains fields σ_i in Si+SiO₂, GeSi plates. The growth of IF maximums heights Q_M^{-1} testifies the growth of structural defects concentration, and the broadening of IF maximums ΔQ_M^{-1} represents the relaxation processes of structural defects new type.

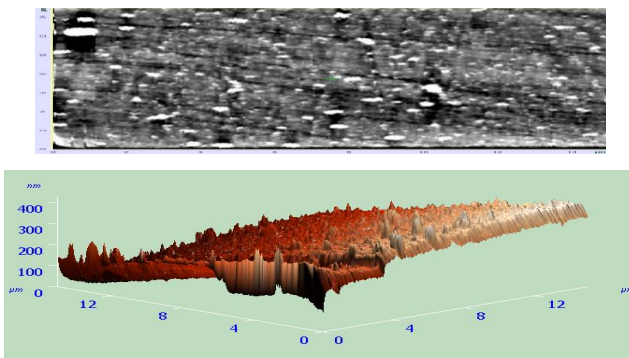


Fig. 7. 3D atomic-force microscopy of microstructure image of Si + SiO₂ type KDB-6.0 with orientation (100) (15x15 mkm)

It is found out in crystal GeSi with orientation [111] the influence of direct J and variable electric current J^\sim on growth of IF $Q^{-1}(J)$ and on the simultaneous diminishing of absolute value of the elastic module $E(J)$, that may be related to that the electrons of certain energy co-operate with

dislocation segments L_C under US deformation ϵ_{US} and is instrumental in their tearing off from points defects. It is obtained, that at presence of variable electrical current with the same frequency and phase, as the strain, that leads to US deformation ϵ_{US} , the critical value of variable current J^\sim^* is nearly by an order of magnitude lesser, than in case of direct current J_σ^* , which is necessary to observe the same IF $Q^{-1}(J)$ raise at increasing of direct current. It is suggested the highly sensitive method of nanoplasticity deformation control of GeSi crystals by measuring of amplitude dependent IF.

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Abstract

The method is created for nondestructive control of structure defects in semiconductor plates, which determines the integral density of structure defects, their distribution and the broken layer from the internal friction difference of free elastic vibrations on neighboring harmonics f_1 and f_2 . The dislocation density and the broken layer are measured from the curve of dependence for wafer-plates Si + SiO₂. The results of influencing of direct and variable electrical current at simultaneous influence of ultrasonic deformation on internal friction and the elastic module of crystal GeSi after cutting and polishing were studied. The decreasing of elastic module and the raise of internal friction was obtained under condition when the critical value of the electrical current is exceeded.

Keywords: semiconductor wafer-plate, structure defects, ultrasound deformation, internal friction, elastic module.

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ВПЛИВ МЕХАНІЧНОЇ ОБРОБКИ, ЕЛЕКТРИЧНОГО ТОКУ, ЗМІНИ ДЕФЕКТНОЇ СТРУКТУРИ НА НЕПРУЖНІ характеристики підкладок Si + SiO₂, GeSi и SiO₂

Резюме

Для неруйнівного контролю структурних дефектів напівпровідникових пластин розроблена методика, що дозволяє по різниці внутрішнього тертя вільних пружних коливань на сусідніх гармоніках f_1 і f_2 визначати інтегральну щільність структурних дефектів, їх розподіл і глибину порушеного шару. Для підкладок Si + SiO₂ зміряна інтегральна щільність дислокацій і глибина порушеного шару по градувальній кривій. Розглянутий вплив постійного і змінного електричного току при одночасній дії ультразвукової деформації на внутрішнє тертя і модуль пружності монокристала GeSi після різання і шліфування. Виявлено зменшення модуля пружності і зростання внутрішнього тертя досягнувши критичної величини електричного току.

Ключові слова: напівпровідникова підкладка, структурні дефекти, ультразвукова деформація, внутрішнє тертя, модуль пружності.

Онанко А. П., Ляшенко О. В., Продайвода Г. Т., Выжва С. А., Онанко Ю.

ВЛИЯНИЕ МЕХАНИЧЕСКОЙ ОБРАБОТКИ, ЭЛЕКТРИЧЕСКОГО ТОКА, ИЗМЕНЕНИЯ ДЕФЕКТНОЙ СТРУКТУРЫ НА НЕУПРУГИЕ характеристики подложек Si + SiO₂, GeSi и SiO₂

Резюме

Для неразрушающего контроля структурных дефектов полупроводниковых пластин разработана методика, позволяющая по разности внутреннего трения свободных упругих колебаний на соседних гармониках f_1 и f_2 определять интегральную плотность структурных дефектов, их распределение и глубину нарушенного слоя. Для подложек Si + SiO₂ измерена интегральная плотность дислокаций и глубина нарушенного слоя по градуировочной кривой. Рассмотрено влияние постоянного и переменного электрического тока при одновременном действии ультразвуковой деформации на внутреннее трение и модуль упругости монокристалла GeSi после резки и шлифовки. Обнаружено уменьшение модуля упругости и рост внутреннего трения при достижении критической величины электрического тока.

Ключевые слова: полупроводниковая подложка, структурные дефекты, ультразвуковая деформация, внутреннее трение, модуль упругости.