

PECULIARITIES OF INELASTIC CHARACTERISTICS CHANGES OF P-TYPE $\text{Si}_{0.96}\text{Ge}_{0.04}$ ALLOYS, IRRADIATED BY X-RAY PHOTONS

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ABSTRACT. *Present work deals with the investigation of the amplitude dependences of internal friction and dynamic shear modulus of p-type coarse-grained $\text{Si}_{0.96}\text{Ge}_{0.04}$ alloy, grown by Czochralski method, radiated by X-rays with a fluencies of $5 \cdot 10^{15} \text{cm}^{-2}$. It is shown, that radiation exposure by X-rays modifies the subsystem of the structural defects, formed in a crystal growth process. Radiation point defects (vacancy-oxygen atom, vacancy-interstitial Si atom) change content and spatial distribution of the impurity atmosphere around the dislocations. This type of change in the defect subsystem stipulates observed variations of intensity of internal friction, dynamic shear modulus and critical strain amplitude at which microplastic deformation appears in $\text{Si}_{0.96}\text{Ge}_{0.04}$ alloy.*

After annealing at 200-400°C temperatures there is a tendency for dynamic mechanical hardening due to the enlargement of radiation defects in the atmospheres around the dislocations and conditions for their blocking.

Keywords: *Radiation defects, internal friction, shear modulus, microplasticity, impurity atmosphere.*

Effective implementation of purposeful changes in mechanical properties of silicon-germanium bulk crystals is possible by controlling crystallographic and energetic parameters, characteristic to their dislocation structure. They are closely linked to the process of formation of the impurity atoms atmospheres in the vicinity of dislocations. Primary radiation defects, in particular, vacancies make significant contributions to the changes of the impurity atmosphere composition and configuration. Concentration and configuration transformations in complexes formed by the interactions of the vacancies and impurity atoms stipulate changes of dislocation mobility in a wide range. In such conditions possibilities to control mechanical strength and microplasticity of SiGe alloys are detected.

Works [1-3] show, that point defects and dislocation origin defects stipulated by thermal stresses and mechanical deformation cause sharp changes of dynamic mechanical properties of $\text{Si}_{1-x}\text{Ge}_x$ ($x \leq 0.05$) bulk crystals. Influence of dislocation structure and changes of Ge content in SiGe alloys on the characteristics of shear modulus and relaxation and

hysteretic internal friction have been studied [4-6]. Activation characteristics of internal friction maxima are determined in SiGe alloys with different Ge content. Causes of reducing strain amplitude and critical temperature for revealing microplastic deformation in condition of increasing Ge content have been analyzed. Regularities of changes of mechanical characteristics determined by internal friction and traditional mechanical methods have been established [7-9]. In spite of the high sensitivity and possibilities internal friction methods are not properly applied in the studies of structural-sensitive physical-mechanical properties of Si-Ge alloys.

In recent years many interesting research works of radiation defects in Si-Ge alloys have been carried out. [10-12]. In particular, structure and electronic properties of vacancy-oxygen atom complexes have been studied. Thermal stability and regularities of electronic properties of Si-O-Si and vacancy - O-Ge complexes have been analyzed. Data is too scarce in the formation and changes of mechanical properties of Si-Ge alloys. Radiation defects influence on relaxation and hysteretic processes of acoustic oscillation damping in Si-Ge alloys is not practically studied.

The present work deals with the investigation of the dynamic shear modulus and torsional oscillations damping processes in polished substrates of initial and X-ray photons irradiated coarse-grained p-type Si_{0.98}Ge_{0.02} alloy. Experimental samples are obtained by the Czochralski method. In their structure oxygen atoms concentration is 10¹⁸cm⁻³. Ge content in the experimental Si_{0.98}Ge_{0.02} alloy is marginal, above which interaction between Ge atoms occurs and the probability of cellular structure forming increases. 1-2 mm thickness plates are cut on the diamond disc and perpendicular surfaces of crystal growth direction. High quality polished samples are obtained by chemical and mechanical treatment combined methods.

X-ray radiation has been performed from cooper anode tube of diffractometer DRON-3. Exposure dose was 5·10¹⁵ photon/cm². Metallographic studies of initial and irradiated samples have been performed on the optical microscope NMM-8RF/TRF. Chemical etching of the samples has been conducting in the solution: 1HNO₃+2H₂SO₄+ 3HF. Logarithmic decrement of torsional vibrations damping and frequency squared have been determined in vacuum (10⁻⁴Torr.) at fixed temperature in ranges of frequency 0,5-5,0 Hz and strain amplitude 10⁻⁵-5·10⁻³. Strain amplitude during the oscillations has been determined, as torsion angle of the sample fixed on vertical axis of reverse pendulum:

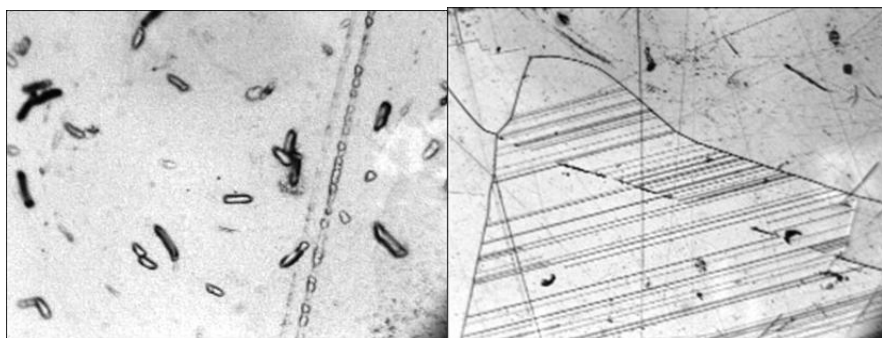
$$\varepsilon = \frac{r \cdot L}{l \cdot R} ,$$

where l is a length of the sample; r - transverse section circumradius of the sample; R - distance between the sample and registration scale; L -deviation of the reflected beam from the mirror, placed on the surface of the sample, on the horizontal registration scale. Error for determining strain amplitude does not exceed 10%. The value of internal friction proportional to the logarithmic decrement of the oscillation damping was determined by the formula [13]:

$$Q^{-1} = \frac{\ln A_m}{\pi \cdot n \cdot A_{m+n}},$$

where n is a number of oscillations, performed in a reducing process of oscillation amplitude from A_m -to A_{m+n} . Error of determining internal friction does not exceed 5%.

Metallographic studies of polished sample of p-type $\text{Si}_{0.98}\text{Ge}_{0.02}$ alloy reveals coarse-grained structure. There are chaotically distributed etching pits with different sizes and shapes. Most of them are "noncrystallographic" defects. They are formed by etching of localized defects in a process of chemical polishing (fig.1,a). Small sized light and dark figures located in deformation lines with different thickness are also revealed. They may be connected to the dislocations formed on the surface of the sample. In inner structure of the coarse grains abundance of stacking faults are revealed. They are decorated with impurities complexes and dispersive inclusions (fig.1,b). The same figure shows disorderly distributed etching pits, that are "noncrystallographic" defects. Irradiation of the experimental sample by $\sim 5 \cdot 10^{15} \text{ cm}^{-2}$ fluence X-ray does not cause substantial changes in microstructure. After the irradiation „non-crystallographic“ and dislocation origin etching pits is still fixed. Fig.1,c shows light etching figures distributed disorderly and in rows. It can be assumed, that they are non-decorated dislocations. Abundance of high density microtwins and stacking faults are revealed in the microstructure of X-ray photons irradiated samples. They contain numerous small bright and dark figures. Dislocation origin defects are spread to the grain boundaries, dispersive inclusions and in the intersection areas (fig.1, d).



a

b

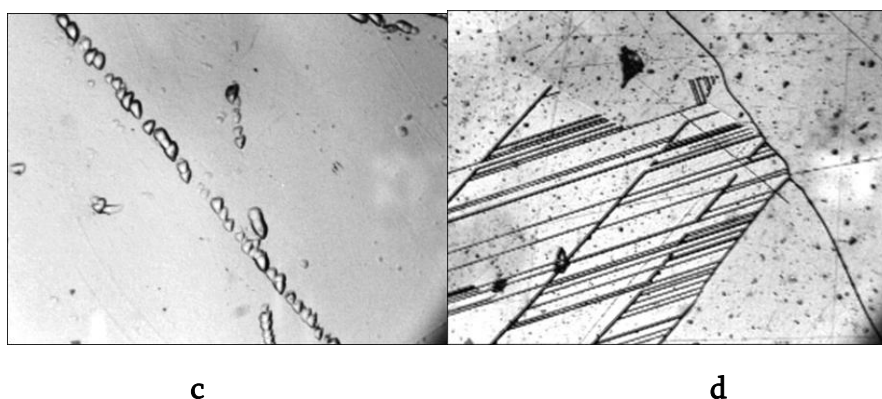


Fig. 1. Microstructure of coarse-grained p- type Si+4at.%Ge alloy

a–initial state, Noncrystallographic defects are shown

n- initial state, inclusions of dispersive phases are revealed.

c–irradiated state, rows of dislocation etching pits are revealed

d- irradiated state, dislocation etching pits and dispersive phases in microtwins and stacking faults are revealed

According to the metallographic studies, the structure of the coarse-grained $\text{Si}_{0.96}\text{Ge}_{0.04}$ alloy contains dislocation origin defects, also complexes of point defects and dispersive inclusions with different sizes in the volume of crystal and near to the linear and flat dislocation defects. It must be noted, that radiation fluence is relatively low and by their influence it is possible to form only point radiation defects and their simple complexes. In realized conditions of radiation it is expected to modify dislocation atmospheres formed in crystallization process of $\text{Si}_{0.96}\text{Ge}_{0.04}$ alloy with radiation point defects. To confirm this opinion internal friction and dynamic shear modulus studies of coarse-grained $\text{Si}_{0.96}\text{Ge}_{0.04}$ alloys in initial and X-rays irradiated states were conducted in a wide range of strain amplitude.

Intensity of internal friction $Q^{-1}(\epsilon)$ at the room temperature is practically unchanged in a wide range of strain amplitude. From the critical deformation of $8 \cdot 10^{-5}$ increase of $Q^{-1}(\epsilon)$ linear character begins. In a wide range of strain amplitude $1 \cdot 10^{-5}$ – $5 \cdot 10^{-3}$ internal friction change is reversible. Annealing in vacuum (10^{-4} Torr.) at 200, 300 and 400°C temperatures for 1hr, practically does not influence on the value of critical deformation. After annealing at 400°C temperature slight decrease of $Q^{-1}(\epsilon)$ line inclination is revealed in high amplitude range ($\epsilon > 10^{-4}$). This circumstance shows that thermal annealing below 400°C temperature does not influence on composition and configuration of point defects formed near dislocations.

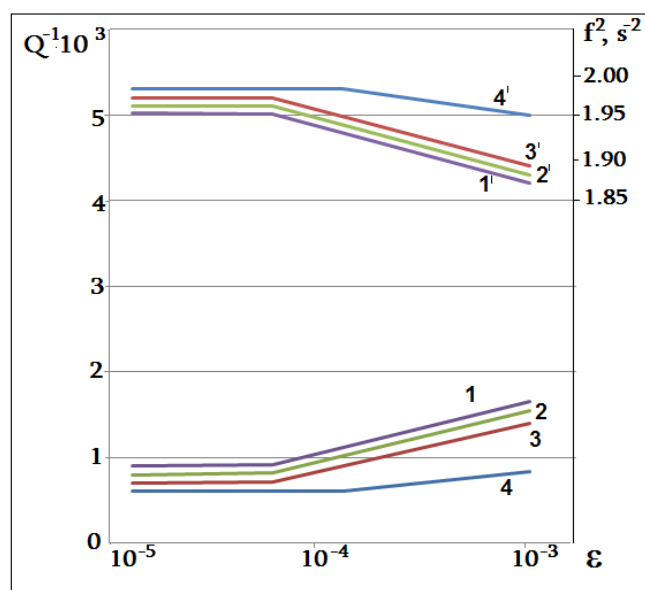


Fig. 2. Influence of thermal annealing on the amplitude dependences of internal friction (1-4) and shear modulus (1'-4') of coarse-grained $\text{Si}_{0.96}\text{Ge}_{0.04}$ alloys
 1,1' –initial state, $f_0^2=1,96\text{s}^{-2}$; 2,2' - annealed state, 200°C 1hr. $f_0^2=1,965\text{s}^{-2}$; 3,3' - annealed state, 300°C , 1hr. $f_0^2=1,97\text{s}^{-2}$; 4,4' - annealed state, 400°C , 1hr. $f_0^2=1,98\text{s}^{-2}$

Torsional oscillations frequency squared $f^2(\epsilon)$ proportional to the shear modulus of initial $\text{Si}_{0.96}\text{Ge}_{0.04}$ alloy is unchanged up to the value of critical strain amplitude ($\sim 8 \cdot 10^{-5}$). Its slight decrease is observed in a high range of strain amplitude ($\epsilon > 8 \cdot 10^{-5}$). Consistent thermal annealing at 200 and 300°C temperatures for 1 hr. does not influence on the character of shear modulus changes. Annealing at 400°C temperature reveals its increase by 10-15%. Linear decrease trends of the shear modulus are observed at high torsional deformations.

The following peculiarities have been revealed on the amplitude dependences of the internal friction and shear modulus in the $\text{Si}_{0.96}\text{Ge}_{0.04}$ alloy irradiated with X-ray photons. Like the non-irradiated sample on the amplitude dependence of internal friction the critical value of strain amplitude appears, in the upper range of which linear increase of intensity of torsional oscillation scattering takes place (fig.2). The critical strain amplitude is increased up to $2 \cdot 10^{-4}$, that reveals strengthening of dislocation bonds stipulated by the radiation and increasing of materials strength. It can be assumed, that radiation point defects are formed in the volume of crystal as well as in atmospheres existing near dislocation nucleus and cause its enrichment.

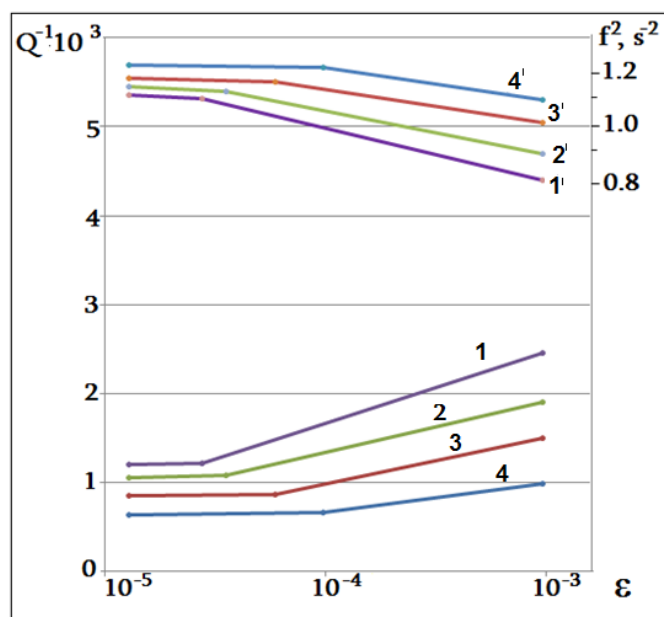


Fig. 3. Amplitude dependences of internal friction (1-4) and shear modulus (1'-4') of coarsegrained $\text{Si}_{0.96}\text{Ge}_{0.04}$ alloy, irradiated by X-ray photons:
 1,1' –initial state 2,2' - annealed state, 200°C 1hr; 3,3' - annealed state, 300°C, 1hrs.;
 4,4' - annealed state, 400°C, 1hr. $f_0=1,1\text{Hz}$.

Annealing at 200°C temperature in vacuum causes a decrease of internal friction intensity in a low amplitude range ($\epsilon < 2 \cdot 10^{-4}$) by 10-15%. There is also a slight increase of critical strain amplitude. Changes in the internal friction amplitude dependence are more clear in the annealed syaye (300°C, 1hr.). Annealing at 400°C temperature causes an increase of critical strain amplitude up to the critical value characteristic of the initial alloy. Amplitude dependence of shear modulus of the irradiated sample is linear in a wide range of deformation. Up to the critical point, it practically does not change, and in the high amplitude range of deformation it linearly decreases. The linear change of the shear modulus is kept in the thermal annealed states at 200, 300 and 400°C temperatures. In this conditions on the amplitude dependence of shear modulus increase of critical strain amplitude is revealed up to the value characteristics for non-irradiated samples. Results obtained by the influence of thermal annealing and irradiation by X-ray photons on the critical values of $\text{Si}_{0.96}\text{Ge}_{0.04}$ alloys are presented in Table 1.

Critical values of strain amplitude in $\text{Si}_{0.96}\text{Ge}_{0.04}$ alloy

Table 1

p-type coarse-grained $\text{Si}_{0.96}\text{Ge}_{0.04}$ alloy	State of the sample			
	Initial state	Annealed, 200°C, 1 hr	Annealed, 300°C, 1 hr	Annealed, 400°C, 1 hr
Non-irradiated	$8 \cdot 10^{-5}$	$6 \cdot 10^{-5}$	$8 \cdot 10^{-5}$	$20 \cdot 10^{-5}$
Irradiated by X-ray photons	$5 \cdot 10^{-5}$	$6 \cdot 10^{-5}$	$8 \cdot 10^{-5}$	$10 \cdot 10^{-5}$

The obtained results confirm that concentration of radiation defects reduces after thermal annealing. Consequently, the interatomic bonds are restored and the strength increased to the initial level. The critical strain amplitude raising might be stipulated by dislocation braking with vacancy-oxygen atom (V-O) complexes, that is formed in a X-ray irradiation process. For a clear separation of the contribution of thermal and radiation origin defects in the changes of structural-sensitive physical-mechanical properties of $\text{Si}_{0.96}\text{Ge}_{0.04}$ alloy it is necessary to investigate monocrystalline samples in initial and irradiated states.

REFERENCES

1. *Berdzenishvili K., Darsavelidze G., Gabrichidze L., Kekua M.* Doping effect on the properties of oxidized monocrystalline silicon. *Inorganic Materials*, 1997, vol. 33, №11, p. 1298-1300. (in Russian)
2. *Kurashvili I., Sanaia E., Darsavelidze G., Bokuchava G., Sichinava A., Tabatadze I.* Physical-mechanical properties of germanium doped monocrystalline silicon. *J. Materials Science and Engineering*. A3 (10)(2013) 698-703.
3. *Kurashvili I., Darsavelidze G., Bokuchava G., Tabatadze I.* Influence of germanium and boron doping on structural and physical-mechanical characteristics of monocrystalline silicon. (2014) 8, ISSN 1314-7269, <http://www.scientific-publications.net.298-302>.
4. *Kurashvili I., Sichinava A., Bokuchava G., Darsavelidze G.* Physical-mechanical characteristics of monocrystalline $\text{Si}_{1-x}\text{Ge}_x$ ($x \leq 0.02$) solid solution. *International Scholarly and Scientific Research and Innovation* 9 (7) 2015.
5. *Kurashvili I., Darsavelidze G.* Mechanical relaxation processes in monocrystalline Si-Ge alloys. *Proceedings of the Conference "Advanced Materials and Technology"* 2015.
6. *Kurashvili I., Darsavelidze G., Bokuchava G.* High-amplitude internal friction in monocrystalline germanium-doped silicon. *J. Phys. Stat. Sol. A* 214 No 7, C14(2017) 1700107 (1-4).
7. *Tetsuo Fukuda and Akira Ohsawa.* Mechanical strength of silicon crystals with oxygen and/or germanium impurities. *Appl. Phys. Lett.* Vol. 60. No 10, 1992, 1184-1186.
8. *Yonenaga Y. and Sumino K.* Mechanical strength of GeSi alloy. *J. App. Phys.* vol. 80, No 6, 1996, 3244-3247.

9. *Wang P., Yu X., Li Z., Yang D.* Improved fracture strength of multicrystalline silicon by germanium doping. *Journal of Crystal Growth* 318 (2011), 230-233.
10. Khirunenko L.I., Pomozov Yu.V, Sosnin M.G., Shinkarenko V.K. Oxygen in silicon doped with isovalent impurities. *Physica B* 273-274 (1999) 317-321
11. Londos C.A., Sgourou E.N., Hall D., Chroneos A. Vacancy-oxygen defects in silicon: the impact of isovalent doping. *J. Mater. Sci: Mater Electron* (2014)25 : 2395-2410
12. Sgourou E.N., Londos C.A., Chroneos A. Vacancy-oxygen defects in p-type Si_{1-x}Ge_x// *J.Appl. Phys.* 133502 (1-6) (2014)
13. Blanter M.S., Golovin I., Neuhauser H., Sining H. Internal friction in metallic materials. A handbook Series: Springer Series in Materials Science 90 (2007) p.539.

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