Influence of ultrasonic treatment on the change of monocrystalline silicon defective region

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Abstract. The article presents the results of an experimental study of ultrasonic action on monocrystalline silicon samples. The influence of the processing modes on the surface strength of the material under study was found.

1. Introduction

It is known that ultrasonic treatment of silicon crystals leads to a change in the dislocation movement speed at a constant mechanical load [1]. In this case, the nature of the dislocation velocity change is determined by the sign of the external stresses acting on the sample. Compressive forces lead to a decrease, and tensile forces lead to an increase in the dislocation velocity. In addition, the dislocation movement activation energy decreases and the electroplastic effect is enhanced.

It should be emphasized that by now the optical phenomena associated with dislocations movement in the field of intense ultrasound have been well studied [2]. The formation of a surface hardened layer in dislocation materials, including semiconductors, during ultrasonic treatment of the sample surface is also a known effect. The standard explanation for this hardening is an increase in the concentration of dislocations in the surface layer [3].

An interesting aspect of dislocations mobility studying is to reveal the relationship between electronic excitation of dislocations and ultrasonic excitation.

There are practically no data in the literature on the effect of ultrasound on the dislocations mobility in silicon single crystals in the internal stresses field. Therefore, this work is devoted to the study of the ultrasonic treatment effect of silicon single crystals on the defect region dynamics in the field of internal stresses.

2. Experimental technique

In this work, p-Si samples (ρ = 1 Ohm·cm, surface orientation (100)) with formed stress concentrators – scratches (load during scribing 0.5–2.0 N), being in distilled water, were subjected to ultrasonic processing with a signal from the ultrasonic generator at a frequency of 63.4 kHz. The processing time varied from 30 min to 5 hours, after which the samples were annealed at a temperature of T = 650 °C for t = 180 min. At a given annealing time, the introduced internal stresses completely relax and dislocation transport stops. To experimentally study the distribution of dislocations in crystals, we used the method of dislocations selective etching.

3. Results and discussion

3.1 Influence of internal stresses on dislocation movement in silicon

The indentation of the crystal surface leads to the appearance of stresses. Their relaxation upon high-temperature annealing can lead to the generation and displacement of linear defects in the absence of external mechanical disturbances. Indeed, the dislocations movement lowers the internal stresses formed during the indentation of the surface. Therefore, one of the primary tasks was to study the linear defects transport in silicon crystals in the field of internal stresses in the absence of ultrasonic treatment.

For this, the prepared samples with stress concentrators were subjected to high-temperature isothermal annealing (650 °C) for 10 - 240 minutes. The research results showed (figure 1) an increase in the depth of propagation of linear defects from the starting position during annealing. Their movement rate, determined by the profile of elastic stresses, gradually decreased and after ~ 80 minutes of high-temperature isothermal holding practically stopped. In this case, the maximum dislocation path from the stress concentrator was limited to 25 - 30 µm.

Another feature of this studies series is a clearly recorded delay in transport processes at the initial stages of isothermal annealing. Thus, within the first 10 min, it was not possible to detect any dislocations displacement; however, in the next 5 min, the leading dislocations path reached already 10 µm. The dislocation transport start delay on Si and Ge (within 10 - 1200 s) under external loading of the crystal was observed earlier by other authors [4-7] and was associated with the conditions for removing linear defects to the starting position, temperature pretreatment regimes, and experimental conditions.

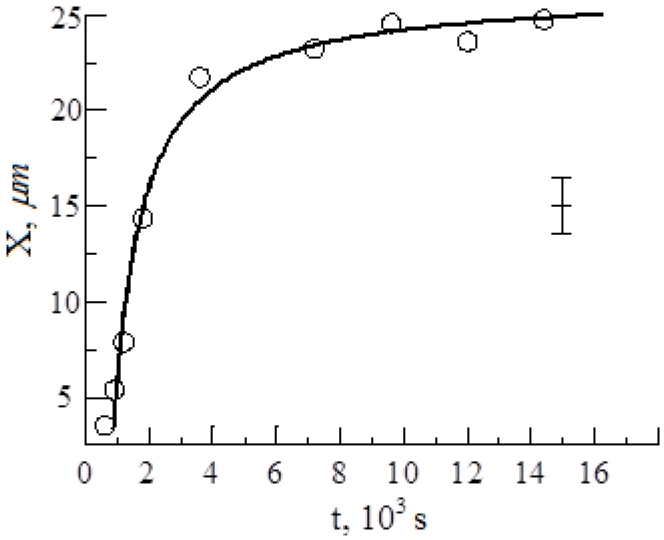

Figure 1. Dependence of the distance traveled by the head dislocations from the scratch (P = 1.75 N) on the high-temperature (T = 650 °C) treatment time.

The results obtained made it possible to describe the temporal changes in the linear defects transport in the field of internal stresses in the form of an empirical dependence:

$$X = \alpha X_{\infty}\left(1 - \frac{t^0}{t}\right) \tag{1}$$

Here X_{∞} – the distance to which dislocations move from the crack at $t \to \infty$; t^0 – time delay of defects movement start; *a* – step function meeting the requirements:

$$\alpha = \frac{f + |f|}{2f} = \begin{cases} 1,\ t > t^0 \\ 0,\ t^0 \geq t > 0 \end{cases},\ \text{where}\ f = \frac{t - t^0}{t}.$$

Approximation of the experimental data by equation (1) (solid line in Figure 1) made it possible to calculate the values of the dislocations maximum path X_∞ = 24.4 μm at $t \to \infty$ and the delay time of the onset of their displacement t^0 = 656.9 s. Consequently, at a larger X_∞ distance, the relaxation of residual elastic stresses cannot be realized by squeezing out dislocations from a scratch.

Thus, to determine the ultrasonic treatment effect on the movement of linear defects, the prepared samples were annealed at a fixed temperature T = 650 °C for t = 180 min. At a given annealing time, the introduced internal stresses completely relax and dislocation transport stops. In this case, the maximum distance of dislocations from stress concentrators is determined only by the microhardness of the surface layers.

3.2 Influence of ultrasonic treatment on the transport of linear defects in silicon

The research results showed a decrease in dislocation paths from stress concentrators with processing time (figure 2). In addition, a decrease in the number of etch pits in the selected slip lines is observed (figure 3).

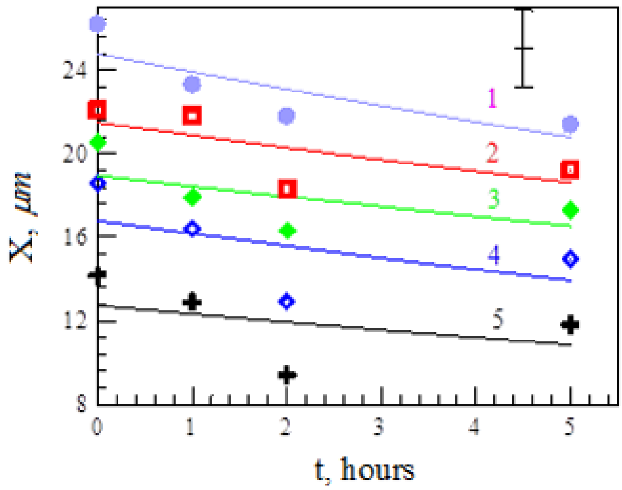

Figure 2. Dependence of the dislocations maximum path in slip lines on the ultrasonic treatment time at various scribing loads: 1 - 2 N; 2 - 1.75 N; 3 - 1.5 N; 4 - 1 H; 5 - 0.5 N. Annealing at T = 650 ° C, t = 3 h.

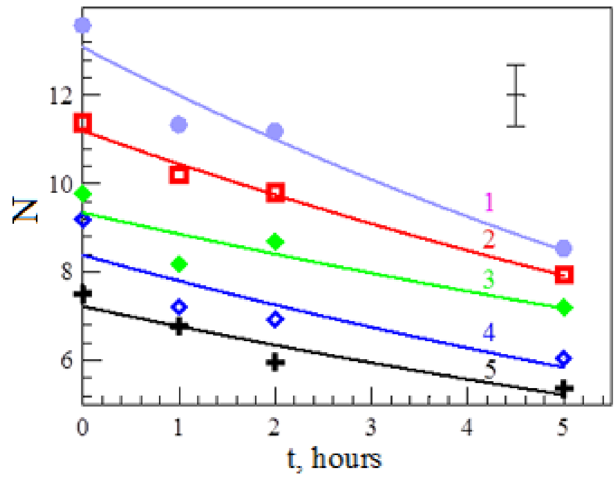

Figure 3. Dependence of the number of locations in slip lines on the ultrasonic treatment time at various scribing loads: 1 - 2 N; 2 - 1.75 N; 3 - 1.5 N; 4 - 1 H; 5 - 0.5 N. Annealing at T = 650 ° C, t = 3 h.

In this case, a regular increase in the controlled parameters with the load on the indenter is clearly recorded when the crystal surface is scribed. The observed decrease in the length of dislocation paths after ultrasonic treatment indicates a decrease in the mobility of dislocations, i.e. the phenomenon of surface hardening is observed.

It should be noted that the action of ultrasonic vibrations by itself did not lead to displacement of dislocations. Clear changes in the dynamics of dislocations are recorded only in the case when isothermal annealing of crystals was carried out after UST. The obtained experimental results can be explained by the influence of a number of factors.

It is known that stimulated diffusion of vacancies and impurities occurs under the action of an ultrasonic wave [8-10]. Taking into account the fact that in our experiments the dislocation half-loop consists of short segments with their ends reaching the surface, as well as the fact that the surface and dislocations play the role of an almost infinite sink of point defects, it can be assumed that the observed effects are associated with an increase in the concentration of point defects under the influence of ultrasonic treatment.

The physical processes of ultrasonic treatment can be associated with the occurrence of acousto-stimulated diffusion of impurities in the samples and an increase in the concentration of impurities in the near-surface layer of silicon. This leads to the predominant nucleation of double kinks on impurities and to an increase in the activation energy of dislocation motion. In addition, the presence of point defects and impurities on the surface and in the bulk, as is known, affects the electronic processes in crystals. That reasons of dislocations motion velocities and the surface microhardness change of the crystal being treated [11].

To check the change in the microhardness of the surface of a single crystal under various modes of ultrasonic processing, studies were carried out to measure the microhardness of crystals. The research results recorded a regular increase in the surface microhardness with the time of ultrasonic treatment (figure 4).

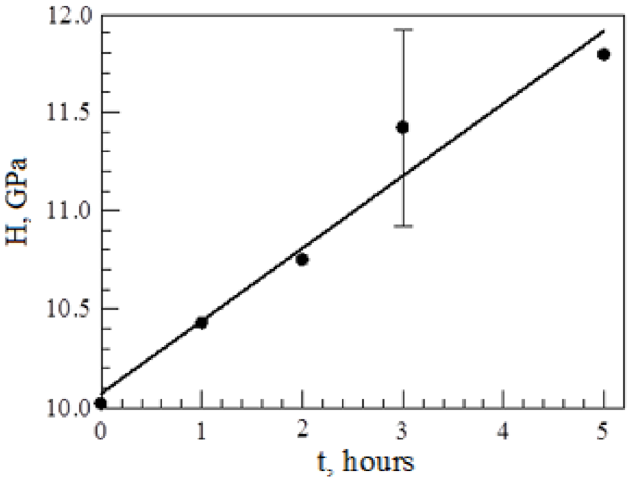

Figure 4. Dependence of the microhardness of crystal surface on the ultrasonic treatment time. Load 100 g at 10 s.

Thus, the ultrasonic treatment of silicon samples containing short near-surface dislocations leads both to a change in the maximum displacement of dislocations with the processing time and to an increase in the microhardness of the crystal surface. It caused by the strengthening of the silicon crystals under study after UST.

4. Conclusion

To check the change in the surface hardness of single crystals under various modes of ultrasonic treatment, a series of experiments was carried out to measure the microhardness of the test samples. The research results recorded a regular increase in the surface microhardness with the time of ultrasonic treatment, which is consistent with the assumptions made on the strengthening of the silicon crystals.

References

[1] Olikh O Y 2018 *Superlattices Microstruct.* **117** 173–188.
[2] Shi C, Shen K, Mao D, Zhou Y and Li F 2018 *Mater. Sci. Technol.* **34**(12) 1511–18.

[3] Mizutani K, Wakatsuki N and Ebihara T 2016 *Jpn J Appl Phys (2008)* **55**(7) 07KA02.
[4] Fan H, Wang Q, El-Awady J A, Raabe D and Zaiser M 2021 *Nat. Commun.* **12**(1) 1845.
[5] Rost H-J, Buchovska I, Dadzis K, Renner M and Menzel R 2020 *J. Cryst. Growth* **552** 125842.
[6] Patel J R 1956 *Phys. Rev. Lett.* **101** 1436–37.
[7] Scandian C, Azzouzi H, Maloufi N Michot G and George A 1999 *Phys. Status Solidi (A) Appl. Mater. Sci.* **171** 67–82.
[8] Ostrovskii I V, Korotchekov O A, Goto T and Grimmeiss H G 1999 *Phys. Reports* **311**(1) 1–46.
[9] Pfahl V, Ma C, Arnold W and Samwer K 2018 *J. Appl. Phys.* **123**(3) 035301.
[10] Olikh O and Voytenko K 2016 *Ultrasonics* **66** 1–3.
[11] Peleshchak R M, Kuzyk O V and Dan’kiv O O 2016 *Ukr. J. Phys.* **61**(8) 741–6.