ON THE POSSIBLE INTENSIFICATION OF DISINTEGRATION OF A SOLID SOLUTION OF OXYGEN UNDER ULTRASONIC TREATMENT OF SPECIMENS OF SILICON

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By triple-crystal diffractometry, a comparative study of the scattering of X-ray emission by annealed specimens after ultrasonic treatment and by reference Cz-Si crystals after their analogous annealing at temperatures 850—1050 °C is performed. The corresponding sizes and concentrations of the clusters of point defects and dislocation loops created in the processes of disintegration of a solid solution of oxygen and clusterization.

1. Introduction

As is known, the intensive ultrasonic irradiation of a material can promote not only elastic, but also its plastic deformation and even destruction. In this case, the velocity of motion of dislocations and their density can grow essentially. The latter can increase even by 3—4 orders [1]. Already at room temperature, ultrasound can induce a redistribution of point defects in semiconductor materials such as CdS [2], InSb, etc. [3—5], including Cz-Si, where it not only stimulates the movement of some doped atoms, ions, and their associations [6], but also can be a source of electronic excitations and even chemical reactions [3, 5, 6].

In view of the above-mentioned, it would be assumed that the excitation of specimens by ultrasound should be also realized upon running the processes of disintegration of a solid solution of oxygen which occur in Czochralski-grown Si crystals. The last are intensively studied now, in particular by X-ray diffraction analysis. Triple-crystal diffractometry turned out to be an extremely sensitive, convenient, and efficient tool of the study of not only changes of the diffraction characteristics of crystals which accompany the processes of disintegration, but also the sizes and concentrations formed of dislocation loops and the clusters of, most probably, SiO_x molecules.

We have used triple-crystal diffractometry of Czochralski-grown Si monocrystals of the type KEP-2.0 (called below as reference ones) annealed at temperatures 650—1050 °C for the estimation of characteristics of the formed centers which scatter X-ray emission [7]. Having executed similar researches on specimens subjected to the ultrasonic influence, we were able to directly compare the results with those derived on reference crystals. The last was the main purpose of our studies.

First, it was necessary to establish when the specimens should be subjected to the action of ultrasound more efficiently and which intensity of ultrasound should be used. The previous researches have shown that the middle-intensity ultrasound does not cause the formation of micro- and macrodefects in silicon, and the treatment of crystals by it prior to annealing does not affect the diffractograms of annealed specimens. The effect of ultrasonic irradiation on studied crystals during a shooting of diffractograms (the so-called X-ray-acoustic resonance [8]) is well known. But, in that case, the small intensities of ultrasound are used, and, as a result, no considerable structural changes occur in specimens. Proceeding from all these considerations, we made use of the ultrasonic irradiation of specimens after their heat treatment and cooling to room temperature. The expediency of just such a scheme of researches was indicated by the literature sources [6], according to which the ultrasound stimulates a movement of point defects already present in specimens, rather than their creation.

2. Procedure and Results of Studies

Similarly to work [7], the specimens with the working surface (111) were cut from Si of the type KEP-2.0, polished industrially, and annealed at temperatures of 850, 900, 950, and 1000 °C during 5 h. Then a half was removed, and the second half was repeatedly annealed at a temperature of 1050 °C during 24 h. After the heat treatment, all specimens were etched in hydrofluoric acid for the removal of the oxidized layer and then underwent the ultrasonic treatment during 3 h.

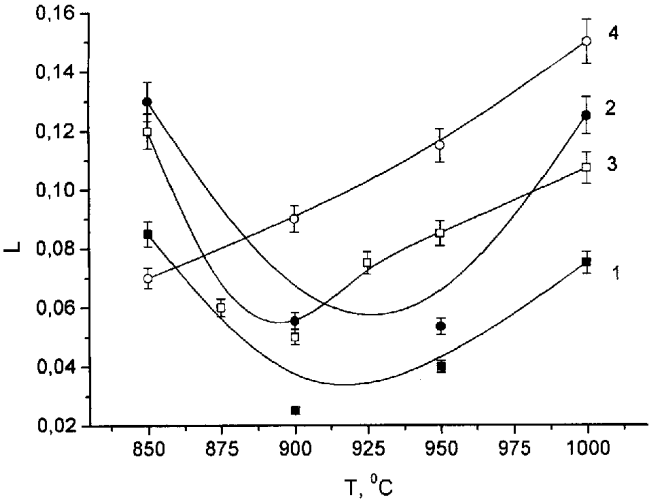

Fig. 1. Debye—Waller static factor L vs the annealing temperature of the samples which underwent the preliminary heat treatment (*1*) and the repeated one at 1050 °C (*2*) and then were subjected to the ultrasonic treatment; *3* and *4* are the similar plots for reference samples

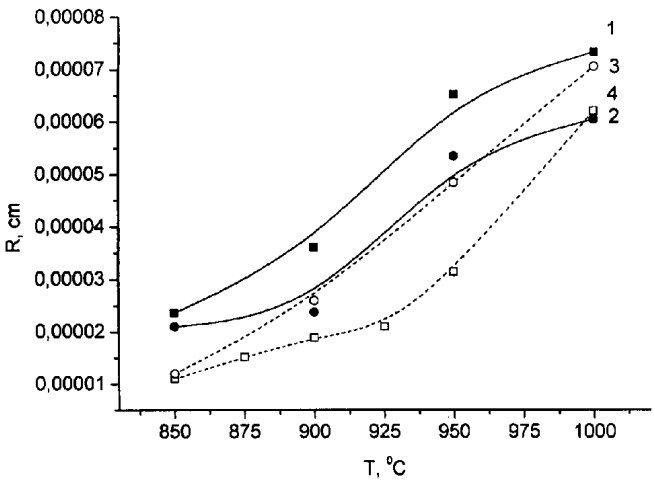

Fig. 2. The same as in Fig. 1 for the effective radii of clusters

On all the specimens, we shot the diffractograms by Bragg on the CuKα_1 emission upon the symmetric reflection from surface (111) at their rotation relative to Bragg's position by an angle α from 10 to 100 "with a step of 5" on a semiautomatic X-ray triple-crystal diffractometer. In all cases, a constant radiation intensity, $I_0 \approx 10^5$pulse/s, was hold.

The diffractograms had a usual three-peak form and therefore are not presented there. The comparative data for the reference specimens cut from the same plate of Si which have passed a similar heat treatment, but were not subjected to the ultrasonic treatment, are taken from [7]. In that work, a technique of calculations of the radii and concentrations of clusters and dislocation loops which are formed in the process of thermal disintegration the solid solution of oxygen in silicon is described in detail. Fig. 1 shows values of the Debye—Waller static factor L derived on the basis of changes of the intensity of the main peaks of diffractograms with increase in α for all specimens under study in comparison with reference crystals.

In Figs. 2 and 3, we give the radii of clusters and dislocation loops derived on the basis of integral intensities of the diffusive peak for the crystals subjected to ultrasonic treatment in comparison with the same data for the reference specimens. We note that the error of calculations of the radii and concentrations is about 10%. In Figs. 4 and 5, we present the concentrations clusters and dislocation loops for the same specimens, as well as those for the reference specimens. These data are got by a technique given in [7, 9] by using the integral intensity of diffusive peaks and the calculated values of the Debye—Waller static factor.

The analysis of the above-presented data indicates that the ultrasonic treatment influences mostly the specimens which underwent the first annealing at middle temperatures (875–975 °C). In this case, the sizes of clusters and dislocation loops noticeably grow with a simultaneous reduction, approximately twice, in the concentration of these defects. The ultrasonic treatment of the crystals repeatedly annealed at a temperature of 1050 °C does not practically change the sizes and concentrations of the centers which scatter X-ray radiation. Changes of the Debye–Waller static factor under various annealing modes are observed to be insignificant. In this case, L even decreases after the ultrasonic treatment. It is clearly seen on the constructed contours of the equiintense scattering of X-rays near site (111) of the reciprocal lattice that the ultrasonic treatment of the crystals causes a certain growth of the diffusive scattering intensity as compared to that for the specimens untreated by ultrasound upon the practically constant intensity of the coherent scattering.

Such a pattern of changes in the characteristics of scattering centers upon the ultrasonic treatment of specimens testifies, probably, to the essential influence of ultrasound on the cluster-dislocation structure of crystals. In particular, these data yield that ultrasound causes the disintegration of a part of precipitates and, possibly, the contraction of some dislocation loops. In this case, it seems that the processes of coalescence are running and promote the reduction of small coagulants

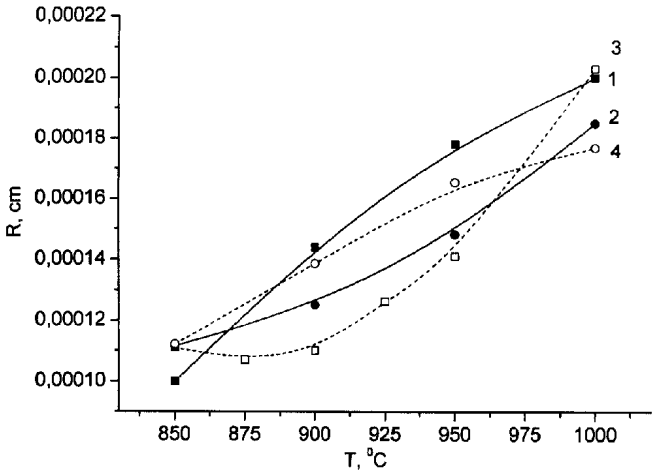

Fig. 3. The same as in Fig. 1 for the effective radii of dislocation loops

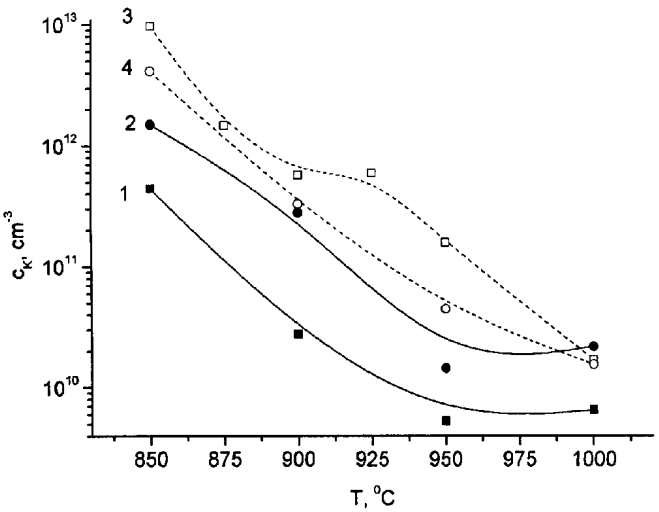

Fig. 4. The same as in Fig. 1 for the concentration of clusters that were formed in the samples processed by ultrasound

and the growth of big ones, which causes the essential growth of magnetic susceptibility. The micron-size coagulants formed upon the repeated high-temperature annealing of crystals take practically no participation in these processes. A similar influence of the ultrasonic treatment should testify to the possibility of the diffusive displacement of interstitial Si atoms and, possibly, oxygen atoms under the influence of ultrasound already at room temperature or a somewhat higher one. We consider such an assumption to be real because the diffusion coefficients of some substances grow [10] sometimes by 5-7 orders if they are measured during the ultrasonic treatment of a material, and this growth increases with decrease in temperature [11].

3. Conclusions

We have observed, for the first time, the influence of the ultrasonic treatment of Czochralski-grown Si crystals which were annealed in the temperature interval where the intense disintegration a solid solution of oxygen in these crystals occurs on the intensity of scattering of X-ray radiation by them and on the physical characteristics of scattering centers.

In particular, it is established that the sizes of coagulants and dislocation loops grow together with a simultaneous reduction of their concentration in the specimens annealed at temperatures 875—975 °C after their ultrasonic treatment. After the repeated annealing of such crystals at a temperature of 1050 °C, the influence of ultrasound on the characteristics of a defective structure becomes slight.

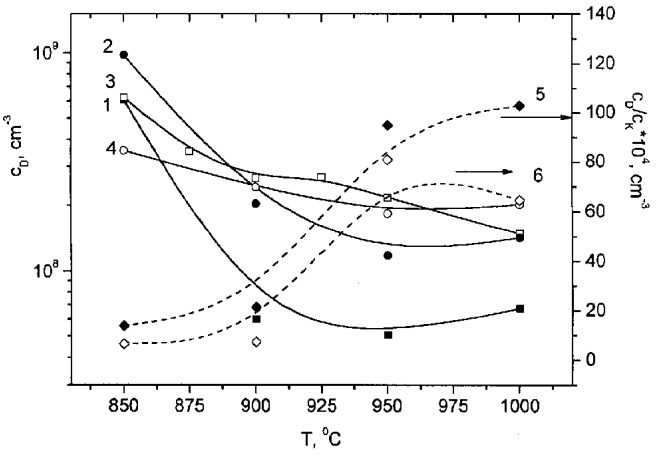

Fig. 5. The same as in Fig. 1 for the concentration of dislocation loops and for the ratio of the concentrations of dislocation loops and clusters in the samples which underwent the preliminary heat treatment (*5*) and the repeated one (*6*)

Some feasible explanations of the observed effects are presented.

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ПРО МОЖЛИВУ ІНТЕНСИФІКАЦІЮ РОЗПАДУ ТВЕРДОГО РОЗЧИНУ КИСНЮ ПРИ УЛЬТРАЗВУКОВІЙ ОБРОБЦІ ЗРАЗКІВ КРЕМНІЮ

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Р е з ю м е

Методами трикристальної дифрактометрії проведено порівняльне вивчення розсіяння рентгенівського випромінювання відпаленими зразками після їх ультразвукової обробки та еталонними кристалами кремнію, вирощеними за методом Чохральського (Cz-Si) після їх аналогічного відпалу при температурах 850—1050 °C. Розраховано відповідні значення розмірів і концентрацій кластерів точкових дефектів та дислокаційних петель, що утворюються в процесах розпаду твердого розчину кисню та кластеризації.