

Investigation of dislocations in Ge single crystals by scanning electron beam

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The comparative analysis of the dislocation images obtained by optical and scanning electron microscopy in Ge single crystals has been carried out. The results obtained by both methods agree well with each other. When there is no impurity atmosphere, the etching pit or the image spot on the dislocation is about 1 μm in diameter. By creating an electric field at the semiconductor surface, the spatial charge area around the dislocation can be polarized. A contrast appears around the dislocation when the area is scanned by electron beam. This enables the distinguishing of dislocations created by low-temperature deformation among neutral impurity inclusions in a crystal.

Проведен сравнительный анализ изображений дислокаций в монокристаллическом Ge, полученных методами оптической и сканирующей электронной микроскопии. Результаты обоих методов хорошо согласуются между собой. При отсутствии примесной атмосферы ямка травления или пятно изображения на дислокации имеют диаметр ~1 мкм. Путем создания электрического поля на поверхности можно добиться поляризации области пространственного заряда вокруг дислокации, а при сканировании этой области электронным пучком — появления контраста. Последнее дает возможность выделить дислокации, созданные низкотемпературной деформацией, среди нейтральных примесных включений в кристалле.

A characteristic feature of dislocations induced in Ge and Si by straining at low temperatures (<500 K) consists in that tiny etching pits about 1–1.5 μm size [1, 2] are revealed thereon by selective chemical etching in standard etchers [3]. Pits of approximately the same dimensions arise at the etching on the inclusions such as GeO_x in Ge and SiO_x or A-clusters [1] in Si. In view of the fact, there are difficulties in identification of dislocation loops emerging in thin subsurface layers at low-temperature straining. Comparative analysis of the dislocation images obtained by optical microscopy and by scanning electron probe is done in this work.

The first series of experiments was carried out on the growth dislocations having some impurity atmosphere. It was established that the dimension of dislocation image decreases to about 1–1.5 μm if the dislocation leaves the impurity atmosphere while moving. In the second series of experiments, optical and electron probe images of fresh dislocations induced in Ge by low-temperature straining in the absence of impurity atmosphere around the dislocations were compared.

Single crystals of Ge (ГЭ-45г3 brand) doped with Sb and having the density of the growth dislocations about $2.5 \cdot 10^3$ cm^{-3} were used. Samples were cut out as rectangular parallelepipeds of edge sizes of $4 \times 4.5 \times 10$ mm^3 oriented in [112], [111], and [110] directions, respectively. The samples were strained at $T = 300$ K by pressing along the [110] direction. The samples of the first series were tested by uniaxial straining under step-by-step loading regime. The loading step value was $\Delta\sigma = 10$ MPa.

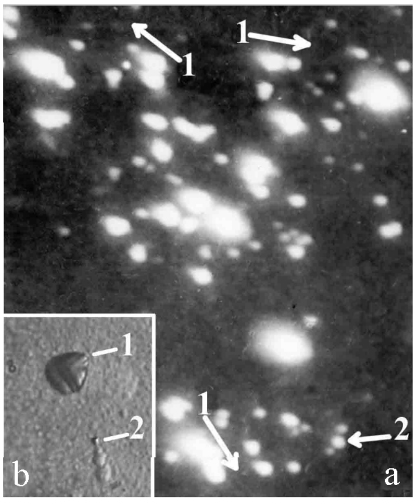

Fig. 1. (a) SEM image of the (111) surface of a strained Ge sample by pressing with load increasing step wisa up to $\sigma_\Sigma = 100$ MPa. $\times1250$. (b) Optical image total pressing stress $\sigma_\Sigma = 30$ MPa. $\times1100$.

The time delay at each loading step was 1.5 h. The number of steps varied from 4 up to 10. So at the total loading $\sigma_\Sigma \leq 60$ MPa, the step-by-step creep in the field of growth dislocation exhaustion mechanism [4] was assured. At the total loading $\sigma_\Sigma > 60$ MPa, microplastic strain arose due to formation of new dislocations. The selective etching on dislocations was carried out in chromic etcher [3].

The dislocation structure revealed on the [111] surface of a strained Ge sample by scanning microscopy in the mode of the secondary electrons extraction is shown in Fig. 1a. It can be seen that while moving (in the direction 1), the growth dislocations leave the impurity cloud. When a dislocation is completely separated from Cottrell atmosphere, it is seen as a small (~1–1.5 μm) light spot. On the same image, the origin of new half-loops on the inclusions (marked as 2) can be seen. A possible mechanism of the half-loop origin has been described in [5]. A metallographic image (Fig. 1b) confirms the above-described effect: at a partial displacement, a large triangular pit (marked as 1) is revealed at the dislocation. When the dislocation is completely separated from the impurity cloud, the triangular pit size decreases to ~1 μm (marked as 2). During low-temperature straining, dislocations are displaced and separated from impurity clouds in the thin subsurface Ge layer (not thicker than 5–6 μm). Therefore, if the layer of the

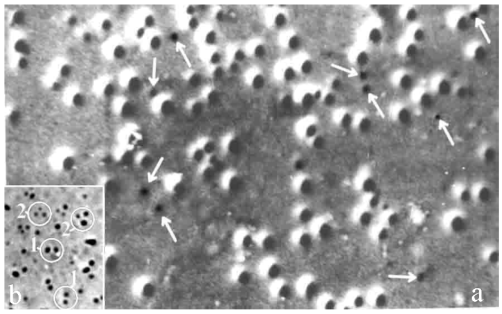

Fig. 2. (a) SEM image of strain-induced dislocations. Energy of hitting electrons is 25 keV, the beam current 0.25 μA, electrostatic bias field strength on the surface, 450 V/cm. $\times1300$. The neutral impurity inclusions are shown by arrows. (b) Optical image of new dislocations induced in Ge crystal by cycle straining under simultaneous ultrasound irradiation at $T = 310$ K. $\times625$.

mentioned thickness is removed by chemical polishing and then the crystal is etched in the selective etcher, a large pit can be etched again at the dislocation.

There is no common opinion in literature on the impurity atmosphere influence on the etching pit size. So, in [6], it is noted that in the case of an "aged" dislocation, the impurity atmosphere bound therewith causes a decrease its kernel energy as well as elastic energy. Therefore, the trend to etching pit formation is decreased. However, when the difference between the atom chemical potential in a crystal and in surrounding etcher is small enough, the site of the aged dislocation may be a preferred source of dissolution steps. As a result, an etching pit is formed [6].

Ge samples of the second series were strained using pressing-unloading cycles at 310 K. The highest stress value was 400 MPa, the cycle duration was 2 h and the total testing time, 24 h. Simultaneously with cyclic straining, the sample was irradiated with 22.5 kHz ultrasound at 5 W power. Under these conditions, the low-temperature microplasticity diffusion mechanism inherent in subsurface crystal layers is realized and density of strain-induced dislocations is raised [7].

An optical image of structural defects on the (111) lateral surface of the strained sample is shown in Fig. 2. Half-loop outlets are seen as pairs of pits (marked as 1) or their triads (marked as 2). One of the pits in a triad corresponds to the inclusion that has caused a dislocation loop. The sizes of loops are within 2 to 4 μm that is near to the thickness (~5 μm) of the anormalous plastic surface layer. The both images in Fig. 2 are taken in an area of dislocation accumulation where the dislocation density is ~10^6 cm^{-2}. At the rest of the surface, it is essentially lower ($5\cdot10^3$ to $5\cdot10^4$ cm^{-2}). The short dislocation loops cannot be revealed and identified reliably by usual X-ray topography methods [8].

There are problems in investigation of dislocations with so low density also when the electron translucent microscopy is used. The dislocation density should be high enough (10^7–10^{12} cm^{-2}) in order that the investigated object will fall into the very restricted foil area.

The gap between opportunities of structural examinations by X-ray diffraction and translucent electron microscopy is filled to a certain extent with the raster-type microscopy [9]. While working in the regime of absorbed electrons, the electron-hole pairs generated by the scanning electron beam change the current in the sample according to the number of excited carriers. This number depends on the state of the crystal surface and the subsurface layer, the local high field areas generating carriers more effectively. The dislocation outlets carrying an electrostatic charge are just such local areas. The lattice atoms located on the edge of the dislocation excess half-plane get a negative charge due to attraction of electrons to the uncompleted bonds. Thus, a linear charge surrounded with a cylindrical positive charge is formed on the uncompleted bonds. Application of an electric field along the surface can change the charge distribution (Fig. 3). The spatial charge can be stretched or squeezed by the field that is revealed as contrast formation around the dislocation. The charged dislocations can be distinguished from the neutral impurity inclusions in a crystal basing on the contrast appearance and changes. The model of an edge dislocation spatial charge tube is shown in Fig. 3. The field applied to the crystal stretches out the positive charge area on the left side and squeezes it on the right side of the dislocation. Therefore, electrostatic intensity decreases on the left side and increases on the right side. The current in the sample is greater and the oscillogram is brighter (Fig. 2a) to the right from the dislocation where carriers generation and recombination rate is higher. Dark spots from neutral inclusions (marked with arrows) also can be seen in Fig. 2. Radius of

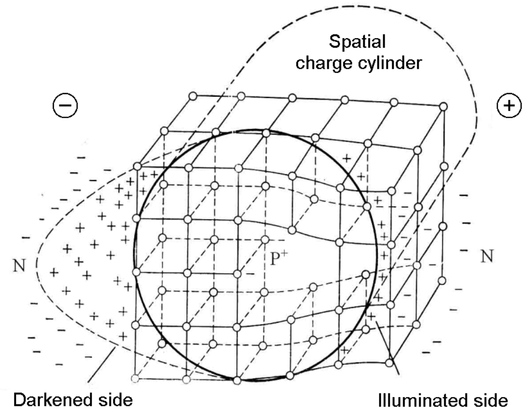

Fig. 3. The model of edge dislocation spatial charge tube. External electric field changes the charge distribution.

the spatial charge tube can be estimated by the formula [10]:

$$R = \left(\frac{f_0}{\pi c(N_d - N_a)}\right)^{1/2},$$

where f_0 is function of filling by electrons of the torn-off bonds on the dislocation line ($f_0 \approx 0.1$ at T = 300 K)); c, gap between unsaturated bonds (for Ge c = 0.4 nm); $N_d - N_a$= 10^{14} cm^{-3}, donors and acceptors concentration difference for the selected semiconductor. Calculation gives that is close R = 0.89 μm to values determined using contrast and etching pits in Fig. 1 and Fig. 2.

Thus, the chemically etched pit size on a dislocation in Ge depends on the presence of impurity atmosphere. When impurity atmosphere is lost, the pit is diminished down to 1–1.5 μm in diameter. By application of an electric field along the semiconductor surface, the spatial charge area around the dislocation can be polarized. A contrast appears around the dislocation when the area is scanned with the electron beam. This enables the distinguishing of dislocations induced by the low-temperature straining among neutral impurity inclusions in a crystal.

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Дослідження дислокацій у монокристалічному Ge сканучим електронним пучком

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Виконано порівняльний аналіз зображень дислокацій у монокристалічному Ge, одержаних методами оптичної та скануючої електронної мікроскопії. Результати обох методів добре узгоджуються між собою. При відсутності домішкової атмосфери ямка травлення або пляма зображення на дислокації мають діаметр ~1 мкм. Шляхом утворення електричного поля на поверхні можна досягти поляризації області просторового заряду навколо дислокації, а при скануванні цієї області електронним пучком — появи контрасту. Останнє дає можливість виділити дислокації при низькотемпературній деформації серед нейтральних домішкових включень у кристалі.