EBIC and DLTS study of dislocation trails in Au-doped Si

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Properties of extended defects introduced by plastic deformation at 600°C in n-Si co-doped with gold are studied by the EBIC and DLTS methods. It is shown that intrinsic point defects generated by moving dislocations stimulate the gold redistribution. As a result, regions depleted with gold are formed near dense dislocation rows. It is shown that dislocation trails decorated with gold are charged that is a reason for the formation of bright EBIC contrast outside the Schottky barrier.

1. Introduction

It was well established that dislocations gliding in Si at 600-800°C generate some defects in the slip planes, so called the dislocation trails (DTs).[1-12] DTs can be revealed by the selective chemical etching [1] and by the electron- and laser-beam induced current methods (EBIC and LBIC, respectively).[2-6, 8-11] Due to their electrical activity DTs introduce deep levels in the bandgap [3,5,12]

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and affect the electrical conductivity.[1] These defects are rather stable. While their effect on the electrical conductivity anneals at 700-750°C,[1] the recombination activity of DTs can be observed up to annealing temperatures of about 850°C.[13] However, in spite of numerous studies the microscopic nature of these defects and the origin of their electrical activity has not totally understood up to now. Extended defects are known to getter point defects, therefore, it is expected that the study of DT interaction with metal atoms allows to separate the contributions of intrinsic and extrinsic mechanisms to the DT electrical activity. Probably, the investigations of DT interaction with gold seem to be the most promising because Au atoms diffuse via kick-out mechanism, i.e. they quickly diffuse over interstitial sites and to occupy the substitutional positions they should push out Si atoms or occupy vacancy sites.[14] The effects of DTs and dislocations on Au in-diffusion have been studied in a few papers.[6,9,14,15] It was shown that the concentration of substitutional Au essentially increases in plastically deformed Si. This can be explained taking into account that interstitial Au is highly mobile at deformation temperature, however, its solubility limit is rather low. Therefore, the Au concentration can be only increased by the occupation of vacancy substitutional sites or by pushing out Si atoms. The second process needs sinks for the excess self-interstitials, thus, the effect of dislocations on the gold in-diffusion at temperatures higher than 800°C was usually explained by the role of dislocations as sinks for the excess self-interstitials.[14,15] However, in Si deformed at rather low (600-700°C) temperature the gold concentration was essentially higher than that which can be provided by dislocations themselves. This allowed to assume that DTs also affect the gold concentration after in-diffusion.[6,9] The increase in the gold concentration determined by DTs was explained under the assumption that some vacancy complexes are formed in the DTs,[9] although in[6] it was concluded that the measured Au profile does not allow to exclude also the interstitial generation by moving dislocations. These interstitials could precipitate in the slip planes forming sinks for the self-interstitials. Thus, in the in-diffusion experiments the defects introduced by deformation stimulated the transition of Au atoms to the

substitutional position. In Si doped with gold, Au atoms occupying the substitutional sites are immobile at deformation temperature and to reveal any effects of deformation on the concentration of Au atoms they should be transferred to the interstitial positions. Thus, it can be expected that investigations of plastic deformation effect on the gold concentration can provide some new information about the defects responsible for the DT formation.

In the present paper the DT properties were studied in n-Si co-doped with Au to check if the intrinsic point defects generated by gliding dislocations can stimulate the redistribution of Au atoms and to gain further insights into Au interaction with DTs.

2. Experimental Section

Silicon crystal with a diameter of 2" and donor and Au concentrations of 1.6×10^{14} and 5×10^{13}cm^{-3}, respectively, was grown by the Floating Zone method in the Leibniz-Institut für Kristallzüchtung. Samples were cut in the form of rectangular prisms with edge orientations of <110>, <110>, <100> and sizes of 35x4x1.5 mm^3, respectively. Dislocations and DTs were introduced by four-point bending at 600°C under the resolved shear stress of about 30 MPa. Indentations produced with a Vickers indenter on the {100} surface serve as dislocation sources for the EBIC measurements. The distance between indentations was of 0.5 mm. To obtain the higher dislocation density scratches were also used as nucleation sites. Four-point bending introduces dislocations only in the central part of samples and the outer parts without dislocations can be used as reference ones. After deformation dislocations were revealed by selective chemical etching in the Sirtl solution (1 CrO_3 +2 H_20 + 3 HF). In the regions, in which indentations serve as nucleation sites the dislocations are located in rows generated from the indentations with an average distance between them about 5-10 μm while in the regions with scratches as nucleation sites the dislocation are distributed more or less homogeneously. The scheme of two acting slip planes {111} inclined to the surface by 55° and dislocation half-loops are shown in **Figure 1**.The Schottky barriers with a diameter of 2 mm were

prepared by thermal evaporation of gold. The ohmic contacts were fabricated by rubbing Al–Ga paste into the rear side of the sample.

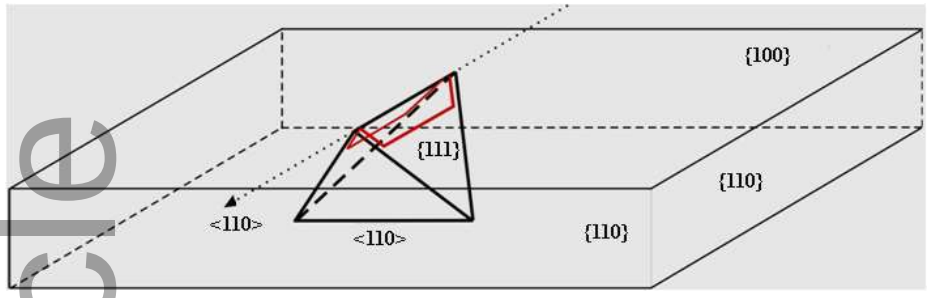

Figure 1. Scheme of acting slip planes {111} and dislocation half-loops. The bending axes is <110> (shown with dotted line) and dislocations segments are parallel to <110> directions.

The EBIC investigations were carried out at room temperature with beam current about 0.1 nA in JSM-840 scanning electron microscope with a Keithley 428 current amplifier. The diffusion length in the reference diodes was estimated by fitting the dependence of collected current in the EBIC mode on beam energy[16] as about 25 μm that is lower than the value, which such method allows to reveal using beam energy up to 40 keV (about 30 μm). It should be noted that the obtained diffusion length value correlates well with that calculated using the measured gold concentration and the capture cross-section for holes of 7.6×10^{-15}cm^{-2}.[17]

The spectra of deep levels were studied by the deep level transient spectroscopy (DLTS) in temperature range from 77 to 300 K. The relaxation signal of the diode capacitance measured at the test signal frequency 1 MHz was processed with a Lock-in amplifier operating as a correlator. The repetition period and filling pulse width in the DLTS measurements were usually of 48 and 1 ms, respectively. The concentration of deep level centers was calculated from the amplitudes of the DLTS peaks using the phosphorous concentration determined from the capacitance–voltage (C–V) characteristics. C-V measurements were carried out at a frequency of 1 MHz. The spectra of deep levels demonstrated below were measured at the reverse bias and pulse amplitude of -9 and 9 V, respectively.

It well known that hydrogen can be introduced into silicon crystal during chemical etching. As a result, in Au-doped Si Au-H complexes are formed.[18,19]. For this reason the Au concentration is estimated as a sum of substitutional gold concentration (energy level E_c- 0.54 eV) and the concentration of Au-H complexes with one hydrogen atom (energy level E_c- 0.20 eV).

3. Results and discussion

The DLTS spectra measured on the reference and plastically deformed diodes are shown in **Figure 2**. The average dislocation density in the diodes with dislocation rows is lower than 3×10^4cm^{-2}, while in the diodes with scratches as dislocation sources it is of about 5×10^5 cm^{-2}. On the spectra measured on the diodes with dislocation rows and on reference one four peaks can be revealed (Curves 1 and 2). The peaks at 109 and 264 K are well known and are associated with Au-H center (the energy level E_c-0.2 eV) and with substitutional Au (the energy level E_c-0.54 eV), respectively.[17-19] In the reference diode, the total gold concentration does not practically change after deformation while in the diode with dislocation rows the total gold concentration decreases by about 15-30%. Such scattering is probably determined by a variation in the number of dislocations producing DTs. The nature of peaks at 158 and 203 K with the energy levels about E_c-0.35 eV and E_c-0.4 eV, respectively, is not clear. The first level is close to that of Au-Fe center,[20] however, it should be unstable at deformation temperature (600°C). The concentration of this center in the reference diode is about 7×10^{11}cm^{-3} and decreases in the diodes with dislocations (Curves 1 and 2). The energy level of the second centers is close to that of substitutional/interstitial Au pair (E_c- 0.44 eV).[17] Its concentration is of about 9×10^{11}cm^{-3} in the reference diode and increases in deformed ones. If these centers indeed are Au_s/Au_i pairs, it could indicate that some gold atoms move to the interstitial positions during plastic deformation. However, not so much is known about this center. C-V measurements show that the carrier concentrations in the diodes with dislocation rows and in the reference ones are practically the same. It can be expected because the gold concentration

decreases about 20% and the initial gold concentration is more than 3 times lower than the donor concentration. Thus, the increase in uncompensated donor concentration due to the decrease in the Au concentration should be smaller than 6%. In the diodes with the high dislocation density the carrier concentration decreases from 1.6×10^{14} to 1.2×10^{14}cm^{-3}. The DLTS spectrum measured on these diodes is mainly determined by the dislocation related peaks (curve 3).[12,21] and it is impossible to estimate the Au concentration from this spectrum due to peaks overlapping.

The average dislocation density of 3×10^{4}cm^{-2} can hardly explain the observed decrease in gold concentration without taking DTs into account since otherwise the dislocations should pick up 10^{8} Au atoms per cm of their length**.** The diffusivity of substitutional Au at 600°C is very small, therefore, a decrease of substitutional gold concentration demonstrates that under deformation gold atoms are moved to the mobile interstitial sites or their diffusion is enhanced by vacancies and then they are gettered by the extended defects or by the surface. The decrease of Au concentration in the diodes with a few dislocation rows allows to estimate the width of regions, in which gold diffusion is stimulated. Under an assumption that the region depleted with Au covers of 15-30% of diode area (as follows from the DLTS data) and if, as in **Figure 3**, three or four dislocation rows per the diode generate the defects stimulated the gold redistribution, the depletion region width is about 60-150 μm per one row**.**

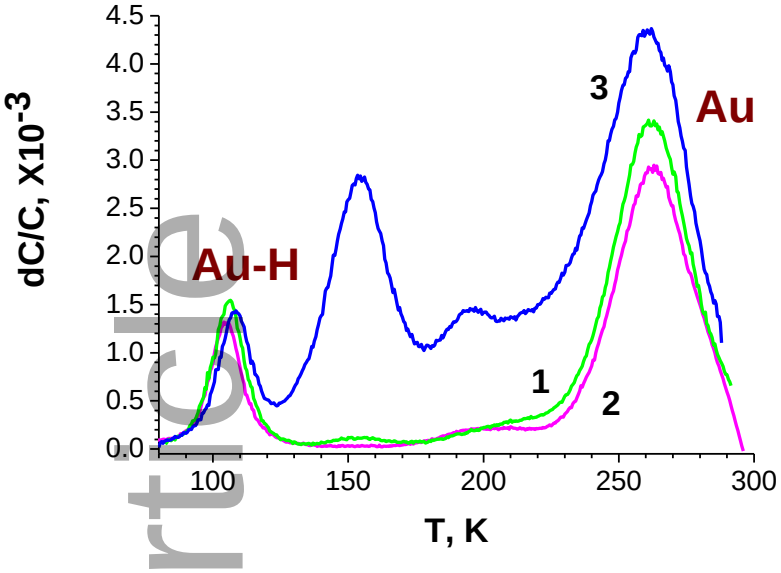

Figure 2. DLTS spectra of reference diode (1), diode with a dislocation density less than 3×10^4 cm^{-2} located in rows (2) and diode with dislocation density about 5×10^5 cm^{-2} (3).

To reveal these depletion regions the EBIC study of diodes with a few dislocation rows was carried out. It was observed that if only a few dislocations are generated from the indentation, the DTs behind them demonstrate the usual dark EBIC contrast (**Figure 4**). However, the dislocation rows with an average distances between dislocations of about or smaller than 5-10 μm produce the bright contrast both under the metal and outside the Schottky barrier (Figure 3). The electrical properties of DTs after gold gettering can differ from those of nominally clean DTs. Unfortunately, in FZ Si it is difficult introduced long DTs with a small dislocation density due to a number of internal defects serving as nucleation sites for the dislocation generation. Therefore, now we cannot measure the DT properties, at least on the samples studied. However, the dark EBIC contrast shown in Figure 4 demonstrates the DT recombination activity that means that DTs introduce energy levels in the gap. Let us discuss the bright contrast outside the Schottky barrier. In **Figure 5** the DT outside the Schottky barrier is shown at a higher magnification. Dislocations in this image can be seen as bright dots probably due to a topography contrast of etched pits. It should be stressed that the bright EBIC

contrast does not change between dislocations, thus, it can be concluded that it is determined by the DTs. The bright lines outside the barrier extend over 1.5 mm (Figure 3), i.e. on distances essentially exceeding the diffusion length value. It seems that the only way to explain this result is to assume that electrons are captured by some traps on DTs and the resulting potential barrier produces a spike of the depletion region that separates excess electrons and holes on the large distance from the depletion region boundary. The bright EBIC contrast of the DTs outside a metal contact similar to that observed in the present work was already revealed in the samples intentionally contaminated with Cu[22] and in samples probably contaminated unintentionally.[4,23] In the samples contaminated with Cu the bright contrast disappeared after annealing at 350°C for 30 min. [22] Silicon can be easily contaminated with Cu, therefore, to check, if the bright contrast observed is indeed associated with Au, the samples were annealed at 350°C. It was revealed that such annealing practically does not change the EBIC images. Moreover, the bright contrast in [22] was observed in p-Si, while in the present work n-Si is studied. The bright EBIC contrast outside the Schottky barriers were not observed on uncontaminated DTs, therefore, it can be assumed that the observed bright contrast outside the metal contact is determined by a capture of electrons on the traps formed due to Au gettering by the defects in the DTs**.**

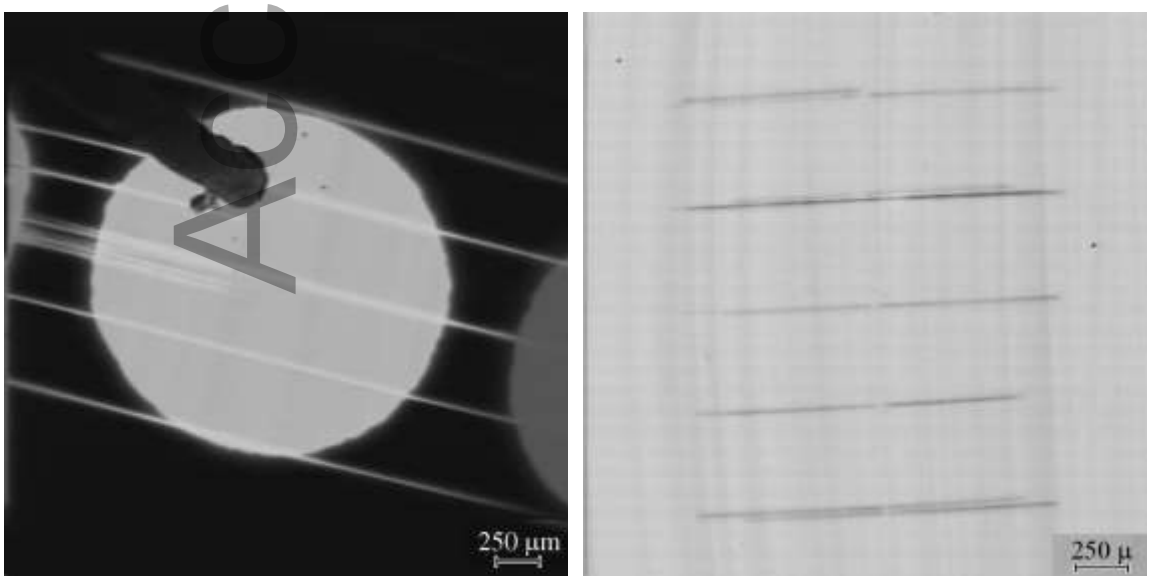

Figure 3. Typical EBIC image at 35 keV of the Schottky diode with dense dislocation rows.

Figure 4. EBIC image at 35 keV of DTs produced by a few dislocations**.**

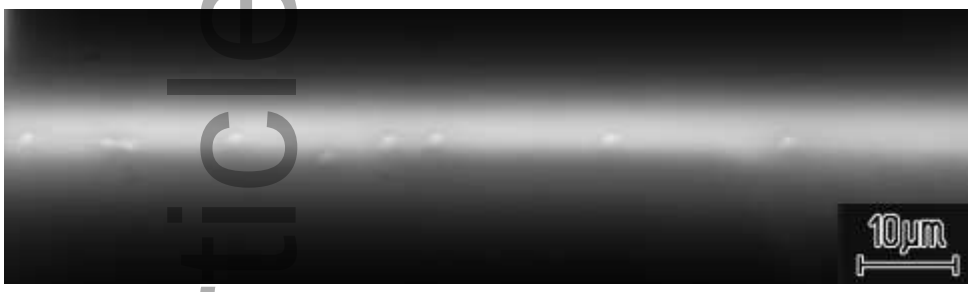

Figure 5. EBIC image of DT outside the Schottky barrier obtained at 35 keV.

The excess carriers are generated by the 35 keV electron at a depth of about 9 μm, i.e. essentially smaller than the diffusion length. Thus, the spikes of the depletion region cannot produce the noticeable bright contrast under the metal and the reason for this contrast formation should differ from that outside the Schottky barrier. The profiles of EBIC contrast under the metal and outside it are shown in **Figure 6**. The asymmetry of the contrast profile seen in the Figure is caused by the inclination of the slipe planes by 55° to the surface. It is seen that width values of contrast profile under the metal and outside it distinctly differ. Besides, the contrast width under the metal is practically independent of beam energy and the bright regions disappear at beam energy lower than 15 keV. Contrary, outside the metal the profile width of the bright contrast coincides well with that of the dark contrast of DTs shown in Figure 4 and decreases with beam energy, i.e. with the width of the generation region. Such behavior well correlates with the assumption that outside the metal the contrast width is mainly determined by the width of the generation region.

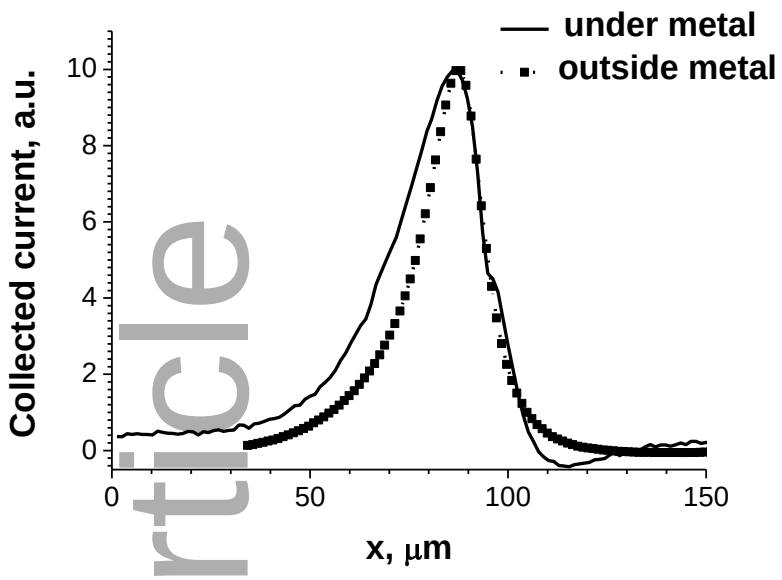

Figure 6. Profiles of bright EBIC contrast under and outside the metal measured at beam energy of 35 keV, *x* is the coordinate in the direction perpendicular to lines of DT intersection with the surface.

Under the metal, the width of the bright contrast can be estimated as 30-40 μm that correlates rather well with the estimated from the DLTS data width of regions near DTs depleted with Au, especially if one takes into account that in the samples studied the diffusion length is close to values, at which the collection efficiency weakly depends on the diffusion length, therefore, the sensitivity of EBIC to the diffusion length changes are rather low. Thus, it is reasonable to assume that the bright contrast under the metal contact is determined by the Au gettering and, as a result, by an increase in the diffusion length near dislocation rows. Under such assumption, the width of regions adjusting to DTs depleted with Au can be estimated as exceeding 20 μm. To be gettered by DTs, Au atoms should be pushed out to the interstitial positions due to an interaction with self-interstitials or diffuse via vacancies if a flow of vacancies is formed during the dislocation motion. The second mechanism of Au redistribution in Si was already observed after rf plasma etching.[24] In any case, the intrinsic point defects stimulating the Au diffusion should be able to diffuse over distances of

the order of 20-70 μm. From this, $(Dt)^{0.5}$ can be estimated as a half of this distance, i.e. about 10-35 μm, where D is the diffusivity of corresponding defects and t is the diffusion time, and Dt is of 1-12×10^{-6}cm^2. The deformation time is about 6 hours that allows to estimate D as 5×10^{-11}-5×10^{-10}cm^2/s. According to reference,[25] diffusivities of vacancies and interstitials in Si at 600°C can be estimated as 1.3×10^{-9} and 9×10^{-8} cm^2/s, respectively, i.e. higher than values obtained from the DLTS and EBIC data. Thus, it cannot be concluded which type of intrinsic point defects generated during dislocation glide stimulates the Au diffusion. However, as shown in,[26] the experimental diffusivity values for vacancies at elevated temperatures are usually essentially lower than the value predicted by the expressions presented in,[25] therefore, it is reasonable to assume that the gold diffusion is stimulated by self-interstitials. If the DLTS peak at 203 K is indeed associated with the Au_s/Au_i pairs this confirms that the self-interstitials are generated in Si by gliding dislocations. Then these self-interstitials push the Au atoms to the interstitial positions. Of course, such assumption does not allow to exclude vacancy type defects, which can stimulate the capture of gold atoms on DTs.

3. Conclusion

Thus, the effect of plastic deformation on the properties of gold-doped n-Si has been studied. It is shown that moving dislocations generate intrinsic point defects, which lead to the formation of DTs and enhance the gold transport. As a result, the regions adjusting dislocation rows are depleted with gold. In these regions the diffusion length increases that leads to the formation of the bright EBIC contrast under the Schottky barrier. The interaction of gold with DTs generates electron traps, which capture electrons forming electric field. This field suppresses electron-hole recombination and leads to the formation of the bright EBIC contrast outside the Schottky barrier.

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Conflict of Interest

The authors declare no conflicts of interest.

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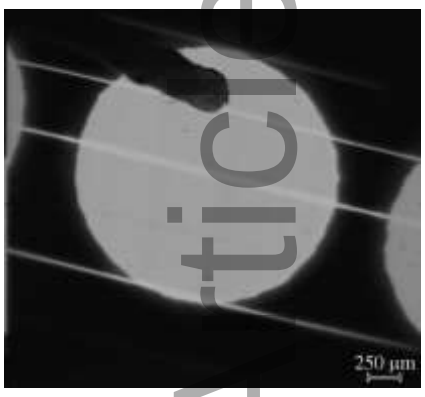

O. V. Feklisova, V. I. Orlov, E. B. Yakimov

EBIC and DLTS study of dislocation trails in Au-doped Si

Plastic deformation of gold co-doped n-Si leads to a formation of regions depleted with Au near dense dislocation rows. Au atoms gettered by dislocation trails form charged two-dimensional defects. These two mechanisms determine a formation of the bright EBIC contrast under the Schottky barrier and outside it.