plexes during annealing in the presence of a strong disorder of the lattice created by ion implantation.

The authors are grateful to A. V. Spitsyn and V. A. Dravin for the implantation of ions in the High Voltage Engineering Europa accelerator, to N. A. Shuvalova for the annealing of the samples, and to E. V. Sobolev and E. A. Konorova for discussing the results.

[1)] Our data on the symmetry of the 575 nm center differ from the results of Davies.[15] The origin of this discrepancy is not clear. A similar discrepancy occurs between the data of the various authors on the symmetry of the 503 nm center (see Sec. 3.3).

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Formation, annealing, and interaction of defects in ion-implante layers of natural diamond

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The cathodoluminescence method was used to determine the characteristic features of the formation and annealing of defects in diamond subjected to ion implantation. It was found that these features could be explained on the basis of the existing ideas on the properties of disordered regions created by ions. A specific influence of nitrogen on the formation of defects in ion-implanted layers was discovered. It was found that the defect environment influenced the annealing of the luminescence centers created by different types of radiation.

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1. INTRODUCTION

Ion implantation doping is used to alter the properties of diamond in a desired manner and, in particular, to obtain layers exhibiting semiconducting properties. This doping method is particularly important because other methods for doping diamond have not yet been developed sufficiently. In principle, to realize the advantages of ion implantation as an effective method of clean precision doping one should know the implantation and annealing conditions which ensure that the properties are governed by the newly introduced impurities and not by the radiation defects created as a result of implantation. One should point out that although ion implantation has been used to prepare semiconducting layers of diamond,[1] the characteristic features of the formation of defects in the cou of ion implantation in diamond have not been investigat much, particularly in respect to the nature of spec fects. The main attention has been concentrated on the electrical activity of the implanted impurity[1,2] and stu have been made of the influence of defects on the mac scopic properties of ion-implanted layers.[3] Relativ little information can be gained by the application of th ESR method to ion-implanted layers of diamond. It has been found that the implantation temperature influences the signal associated with dangling bonds or with the "amorphous phase," and anisotropic ESR centers hav been observed (but only after the implantation at rel high temperatures) some of which are likely to be cancies.[4,5] We investigated defects in ion-implanted la of diamond with the aid of luminescence which is an effective "microscopic" method for determining the nature of local centers in solids.

EXPERIMENTS

Our samples were prepared from natural type IIa crystals ("nitrogen-free" crystals, i.e., those containing lower than 10^{18} cm^{-3} of nitrogen atoms) as well as type Ia crystals (with nitrogen concentrations in the range ≥ 10^{19} cm^{-3}). Implantation of ions of energy 30–350 keV in doses [illegible]-10^{15} cm^{-2}, required to obtain a specified distribution of the impurity with depth, was carried out using a heavy-ion accelerator made by High Voltage Engineering Europa. Some of the measurements were carried out on samples irradiated with electrons of energies 2.2 MeV and 10 keV (subthreshold defect formation[6]). The samples were annealed in a graphite container evacuated to 10^{-5} torr and temperatures of 150–1400°C were applied for 2 h. Cathodoluminescence was excited by a beam of 2–10 keV electrons (the electron current was ≈ 5 μA) and its spectra were investigated in the range 300–900 nm when a sample was kept at a temperature of 80°K.

RESULTS AND DISCUSSION

Figure 1a shows the cathodoluminescence spectra of type IIa and Ia crystals before implantation. These spectra were dominated by a wide structure-free band (called the A band) with a maximum at 400 and 430 nm and a width of 0.4 and 0.7 eV for the type IIa and Ia crystals, respectively. This band was clearly associated with dislocations in IIa crystals and with platelet nitrogen defects in Ia crystals.[7,8] Moreover, there were several weak narrow lines (389, 415, 441, 496, 503, 575 nm). The cathodoluminescence spectra of the original crystals were not affected by annealing right up to 1400°C.

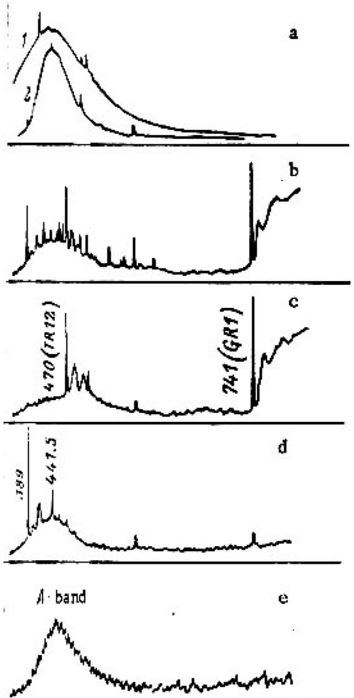


FIG. 1. Cathodoluminescence spectra: a) original type Ia (1) and IIa (2) samples; b-d) samples subjected to bombardment with H$^+$, C$^+$, and N$^+$ ions, respectively, in doses lower than D_{cr}; e) samples bombarded with ions in doses exceeding D_{cr} (see Sec. 3.3.2).

Implantation reduced the intensity of the A band and gave rise to new bands and groups of lines consisting of a narrow zero-phonon line and more or less intense phonon wing. The majority of the narrow cathodoluminescence lines were clearly associated with intracenter electron transitions. The nature of the radiative transitions responsible for the luminescence was not investigated and we used these characteristic lines simply for luminescence spectral analysis of ion-implanted layers, i.e., to determine the concentrations of the various luminescence centers identified in accordance with the wavelength of the zero-phonon line. Since the A band was associated with nonradiation defects, whose number was not affected by implantation (at least up to a certain dose) or annealing, we took the ratio of the intensity of the band or line of interest to us to the intensity of the A band and thus eliminated the changes in the carrier lifetimes; this allowed us to obtain information on changes in the luminescence centers as a result of implantation and annealing (in the discussion below we shall use the term line intensities to denote the intensities relative to that of the A band).

3.1. Defects in Ion-Implanted Diamond Layers (Before Annealing)

The nature of defects formed in ion-implanted layers may generally depend on the original impurity composition of a sample and also on the nature of the implanted ions. In our experiments the cathodoluminescence spectra of the IIa and Ia type samples subjected to ion bombardment were the same before annealing, i.e., the defects created by implantation (and manifested in the cathodoluminescence spectra) were independent of the presence of the only controlled impurity (nitrogen). On the other hand, there was a definite relationship between the cathodoluminescence spectra and the nature of the implanted ions. This was manifested by the difference between the results obtained for the crystals subjected to bombardment with H$^+$ and with heavier ions. After implantation of H$^+$ we found that the spectra had many narrow lines (389, 415, 441, 468, 470, 491, 503, 545, 555, 575, and 741 nm), whereas after implantation of the heavier ions the spectrum was dominated by the 741 and 470 nm lines (Fig. 1b and 1c). The defects associated with hydrogen in diamond had not been investigated thoroughly so that some of the lines observed after the implantation of H$^+$ and absent in the case of implantation of the heavier ions could be due to centers containing hydrogen. How-

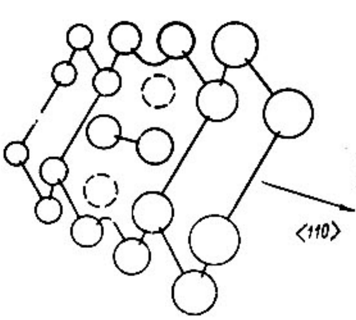


FIG. 2. Model of a TR12 center (470 nm).

ever, the reduction in the intensities of the 389, 415, 503, and 575 nm lines associated with the centers containing nitrogen, which was observed as H^+ was replaced with the heavier ions, clearly indicated a reduction in the importance of the impurity–defect complexes. After ion irradiation a major proportion of the defects were concentrated within disordered regions when the concentration of intrinsic defects was very high and increased on increase in the ion mass. The dominant role of the 741 and 470 nm lines observed after implantation of the heavy ions reflected the increase in the proportion of the intrinsic defects and the saturation of the process of complex formation within disordered regions.

It is known that the 741 nm line is due to isolated vacancies (GR1 centers[10]). The 470 nm line was also due to intrinsic defects as indicated by the nature of the centers (TR12 – Refs. 11-13), but the intensity of the 470 nm luminescence was practically the same for all the investigated crystals of both types after implantation of different ions having comparable masses. Our unpublished results enabled us to put forward some ideas on the nature of these centers. Firstly, judging by the superlinear (quadratic or cubic[11–13]) dependence of the intensity of the 470 nm line on the radiation dose, the center responsible for the line consists of several elementary radiation defects. Next, if we turn to the "compliance" of the center, i.e., to a parameter representing the response of an optical center to uniaxial deformation of a crystal,[1)] we find that in the case of the 470 nm centers the compliance is $s = 0.76\ \mathrm{Pa}^{-1}$, which is intermediate between the values typical of vacancy ($s = 4\text{-}5\ \mathrm{Pa}^{-1}$) and interstitial ($s < 0.1\ \mathrm{Pa}^{-1}$) centers. Hence, we may conclude that this center contains both vacancies and interstitial atoms. Finally, the nature [illegible] splitting of the zero-phonon 470 nm line by uniaxial [illegible] pression (as found in Ref. 12 and in our study) shows [illegible] the center has the monoclinic-I symmetry and the symmetry plane is (110). All these properties are [illegible] by a model shown in Fig. 2 representing a double [illegible] pair in which interstitial atoms occupy hexagonal [illegible] positions. It is interesting to note that this TR12 [illegible] is formed by ion implantation in crystals of both [illegible] diamond, whereas in the case of electron irradiation [illegible] formed only in the nitrogen-free crystals. It follows [illegible] because of the high concentration of intrinsic defects [illegible] disordered regions, the "quenching" effect of nitrogen [illegible] the type Ia crystals (the mechanism of this effect is not yet clear) is greatly weakened.

Quenching of the luminescence associated with [illegible] intrinsic defects was also caused by the implanted nitrogen atoms. The cathodoluminescence spectrum of the samples implanted with nitrogen had specific lines [illegible] and 441 nm) associated with the nitrogen-containing centers[14,16] and the intensity of the luminescence due to [illegible] GR1 and TR12 intrinsic defects was considerably [illegible] than in the case when other ions were implanted (Fig. [illegible] In principle, this effect could be explained in two ways. Firstly, it may be due to a specific (typical only of [illegible] implantation of nitrogen) position of the Fermi level [illegible] which the luminescence associated with the GR1 and [illegible] centers (introduced at the same rate as in the case of [illegible] plantation of other ions) is not observed. Secondly, [illegible] feature of nitrogen may be due to the fact that the implanted nitrogen atoms act as traps or even precipitation centers of intrinsic defects and they thus reduce the concentration of the GR1 and TR12 centers. Both these explanations are not very convincing primarily because [illegible] concentration of the implanted nitrogen atoms is very [illegible] compared with the concentration of radiation defects [illegible] unannealed crystals). Moreover, in the case of the [illegible] mechanism the radiation defects must be mobile at [illegible] temperature, which is certainly not true of vacancies (GR1 centers). Therefore, the mechanism of a [illegible] influence of nitrogen on the formation of defects in [illegible] implanted diamond layers (manifested also in annealing as described below in Sec. 3.2.1) is not yet clear.

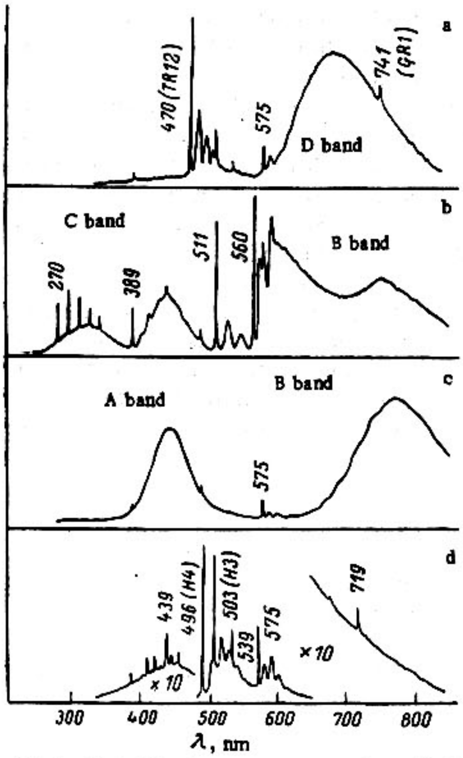


FIG. 3. Cathololuminescence spectra of type IIa (a-c) and Ia (d) crystals after ion bombardment and subsequent annealing at temperatures (°C): a) 600; b) 1000; c, d) 1400.

3.2. Annealing of Defects in Ion-Imp[illegible] Diamond Layers

The annealing of ion-implanted crystals alters [illegible] siderably the cathodoluminescence spectra indicating

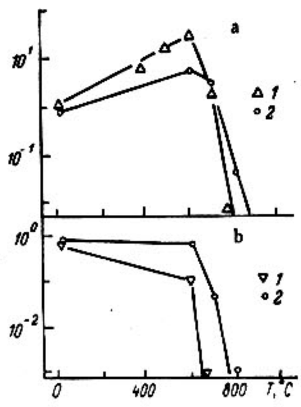
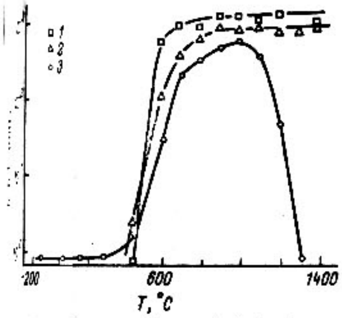


FIG. 4. Dependences of the [illegible] I of cathodoluminescence [illegible] annealing temperature: a) [illegible] (470 nm) centers created by [illegible] ation of Si^+ (1) and B^+ (2) [illegible] GR1 (741 nm) centers created [illegible] plantation of O^+ (1) and [illegible]



Dependences of the cathodoluminescence intensity I on the anneal-mperature in the case of the H3 (503 nm) centers formed by ion im-ation (1), irradiation with 2.2 MeV electrons (2), and irradiation with V electrons (3).

ges in the concentrations of various defects. Since ese changes depended strongly on the presence of the rogen impurity in the original samples, we shall con-der separately the results applicable to the type IIa and iamond samples.

3.2.1. **Nitrogen-free type IIa diamond ystals.** Changes in the cathodoluminescence spectra itrogen-free diamonds as a result of annealing (Fig. 3) e qualitatively the same for any implanted ions with the eption of nitrogen. The GR1 and TR12 centers were an-aled at ≈ 800°C. New centers with zero-phonon lines 1 and 560 nm appeared at the same temperatures. eover, wide bands with maxima at 770 nm (B band) 80 nm (D band) were observed, as well as a C band nm) and a group of narrow lines (270, 386, 302, ... On the basis of the available data it was difficult to nything definite about the nature of the centers re-sible for the lines and bands that appeared after an-ing. It was worth noting an interesting feature of the and, which was its anticorrelation with nitrogen: all crystals containing nitrogen as an impurity (either ent originally or introduced by implantation) were acterized by B-band intensity at least two orders of gnitude less than that exhibited by nitrogen-free crys-under identical implantation conditions. Once again encountered an effect of nitrogen on the formation of ects in diamonds which was not well understand.

3.2.2. **Nitrogen-doped type Ia diamonds.** radiation defects which appeared immediately after ion plantation (Sec. 3.1) were annealed in the type Ia crystals at same temperature of ≈ 800°C as in the case of the nitrogen-free type IIa samples. The special property of the type Ia diamonds appeared at high annealing temperatures and was manifested by the appearance of strong luminescence lines (Fig. 3d) associated with the centers containing nitrogen.[14,16] The strongest were the lines at 503 nm (H3 centers) and 496 nm (H4), which appeared as a result of association of radiation defects (vacancies) with the A and B_1 forms of nitrogen. The ratio of the intensities of these lines varied strongly from sample to sample. Moreover, we observed lines at 439, 539, and 575 nm also associated with the centers containing the nitrogen atoms. In view of the high intensity of the luminescence associated with nitrogen it was not possible to observe the lines at 511 and 600 nm or the bands C and D typical of the nitrogen-free crystals annealed at T ≈ 1000°C.

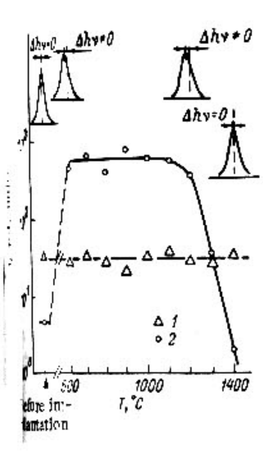


FIG. 6. Dependences of the intensity I of the 389 nm line on the annealing temperature in the case of unimplanted samples (1) and samples implanted with ^{15}N ions (2). The isotopic shift of the cathodoluminescence line is shown schematically for the ion-implanted samples.

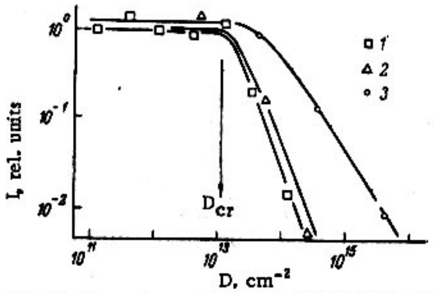


FIG. 7. Dependences of the intensity I of the A band on the implantation dose: 1, 2) implantation of N+ in the type Ia and IIa samples; 3) implantation of B+ in the type IIa samples.

3.3. Interaction of Defects in Ion-Implanted Diamond Layers

Since the implantation of ions created defects in very high (compared with electron irradiation) concentrations, their interaction was very important. The cathodoluminescence method provided a microscopic technique for obtaining information on the presence of specific centers with more or less known microstructure and thus collecting data on the interaction between defects, particularly on its influence on the defect annealing processes.

3.3.1. **Influence of defect environment on the annealing of luminescence centers.** The defect environment of a luminescence center is understood to be the spatial distribution of defects and impurities aro around it. This distribution depends on the previous history of of a sample and on the nature of the radiation which creates defects. We shall see that the specific nature of the defect environment is manifested in the annealing characteristics of the same centers present in unirradiated crystals or created by different types of radiation.

We shall now turn to Fig. 4 showing the annealing-induced changes in the intensities of the 470 and 741 nm lines associated with the TR12 and GR1 centers. Clearly, the temperature at which the concentration of the centers decreased strongly and the general behavior of the curves depended on the nature of the implanted ions. In particular, it should be pointed out that an increase in the concentration of the TR12 centers at T ≈ 600°C increased with the ion mass (in the case of the Si ions this increase reached two orders of magnitude). On the other hand, the concentration of the GR1 centers decreased in the same temperature range (although not as strongly as in the case of the TR12 centers) and once again the effect became magnified on increase in the ion mass. These observations could be explained in a natural manner by the existing knowledge of this structure of the GR1 and TR12 centers and of the characteristics of defect formation as a result of ion implantation. In fact, ion bombardment produces disordered regions consisting of a core where vacancies (and their complexes) predominate and a surrounding "atmosphere" where interstitial atoms are the main effects.[9] Therefore, vacancy and interstitial defects are spatially separated and the separation increases with the mass of the implanted ions. Under these conditions the formation of the complex vacancy–interstitial TR12 defects is relatively unlikely. A considerable increase in the concentration of the TR12 centers on increase in the annealing temperature can be interpreted (on the basis of the proposed model) as evidence that at least one of the species of the particles forming these centers (most probably the interstitial carbon atoms) becomes mobile at temperatures of 400-600°C. This hypothesis is in agreement with our data on the changes in the structure of the luminescence centers containing nitrogen, according to which the activation of the motion of interstitial nitrogen atoms also occurs beginning from ≈ 500° (Ref. 14). Annealing up to T ≈ 600°C reduces the number of vacancies (GR1) by an amount smaller than the increase in the number of TR12 centers. This is clearly due to the first that the latter are formed as a result of association of interstitial atoms not only with isolated vacancies, but particularly with divacancies whose concentration in the disordered regions may be fairly high.

The influence of the defect environment on the annealing of the luminescence centers can be followed also by comparing the annealing curves of the centers containing nitrogen and created by a variety of methods. Figure 5 shows the annealing-induced changes in the intensity of the 503 nm line associated with the H3 centers (postulated to consist of two neighboring substituted at nitrogen atoms and two vacancies[14,17]) in the type Ia crystals. The results represented by the symbols 1 were obtained for the samples subjected to ion bombardment, whereas the results denoted by 2 and 3 were obtained for the samples irradiated with electrons of energies below and above the elastic displacement threshold. Clearly, in all cases the H3 centers appeared at T ≈ 500°C but an increase in the intensity of the 503 nm line was faster in the case of the ion-implanted crystals. Moreover, the H3 centers created by ion bombardment and suprathreshold electron irradiation were not annealed up to 1400°C, whereas the same centers introduced by subthreshold electron irradiation disappeared at ≈ 1000°C. These differences were associated with the specific nature of the defect–impurity and environment of the centers formed by subthreshold irradiation. In this case the formation of defects clearly occurred in some special impurity-rich parts of a crystal.[18] In particular, a transition metal atom and a donor were assumed to be located near a luminescence center formed in this way.

The influence of the defect environment was observed very clearly in a study of the annealing of the 389 nm luminescence centers. The nature of these centers was not quite clear but it was known that they contained nitrogen atoms probably in the interstitial form.[14] The 389 nm centers appeared as a result of implantation of nitrogen ions in diamond; they were sometimes observed in low co trations in unirradiated crystals. We found that the nm centers introduced by ion implantation were anne at T ≈ 1100°C, whereas the same centers in unirradi crystals were retained only up to T ≈ 1400°C. The d ence between the 389 nm centers introduced by vario methods appeared most clearly in the experiments i ing implantation of the ^{15}N isotope (Fig. 6). Before im plantation we observed a weak 389 nm line. After im ation of ^{15}N the line intensity was much higher and it shifted toward shorter wavelengths by 0.2 meV (isoto shift). This shift demonstrated that the implantation ated a large number of the 389 nm centers containing ^{15}N isotope. After annealing at 1400°C the intensity o 389 nm line decreased to a value obtained earlier bef irradiation and the isotopic shift disappeared, i.e., th nm centers formed by the implantation were annealed only those 389 nm centers remained which were pres before implantation. Such a strong difference in the nealing behavior between the 389 nm centers introdu by implantation and those present in the unirradiated tal was clearly due to the fact that in the former cas were within disordered regions where the intrinsic d concentration was high. The influence of disordering the lattice caused by ion bombardment on the modifi of the structure of defects was observed by us also i study of the transformation of the centers containing trogen and present in ion-implanted diamond layers.

3.3.2. Cathodoluminescence after implantation doses. The effects discussed in S 3.3.1 were due to the interaction of defects present in hig centrations within disordered regions. The volume-a defect concentration could still be low. An increase in plantation dose was accompanied by a number of specif fects associated with an increase in the average def concentration. Firstly, beginning from a certain "cr dose D_{cr} (which decreased on increase in the ion mas the intensity of the A band which was reduced by imp ation was not restored by the subsequent annealing Next, at approximately the same dose the luminescen lines of the individual defects (GR1, TR12, 3H) disap peared from the cathodoluminescence spectrum reco immediately after implantation and only the A band r mained (Fig. 1e). These observations indicated cons able changes in the structure of the ion-implanted la clearly resulting in an overlap of the disordered reg and beginning of the amorphization process. These changes in the structure were confirmed by the satu

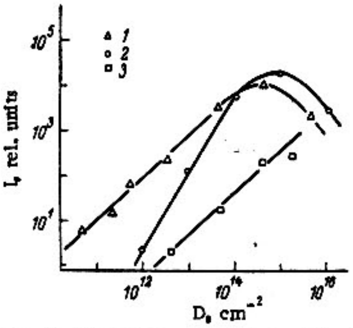


FIG. 8. Dependences of the intensities I of the cathodolumines and bands of the IIa type crystals (after annealing at 1400°C) on th plantation dose: 1) 575 nm line, implantation of N⁺; 2) 736 nm li implantation of Si⁺; 3) B band, implantation of B⁺.

or even nonmonotonic behavior of the dose dependences of the intensities of the luminescence lines associated with the centers which appeared after the implantation of nitrogen and silicon and subsequent annealing (Fig. 8). Weakening of the dose dependences at high doses could be interpreted as a reduction in the effective volume of a "normal" crystal (where these centers could form) resulting from an increase in the proportion of the volume occupied by the regions which became amorphous or at least strongly disordered.

Enhancement of the lattice disorder by large implantation doses was manifested also by considerable (up to ≈30 meV) broadening of the lines of the "soft" centers containing nitrogen. The broadening was much stronger than that discovered for crystals irradiated with 2 MeV electrons[21] (in the latter case the broadening of the lines to 1-2 meV was attributed to local stresses created in the lattice by the A form of nitrogen[22]). Clearly, the strong broadening of the lines due to the centers containing nitrogen was due to the strong disorder of the lattice caused by the ion implantation and retained even after annealing at 1400°C.

The cathodoluminescence spectrum of ion-implanted diamond included a band which was clearly associated with the residual (i.e., that retained after annealing at 1400°C) lattice disorder and with the possible formation of amorphous regions. This was the band B mentioned in Sec. 3.2.1, which was the only band of radiation origin not associated with known impurities and retained after annealing at temperatures up to 1400°C in the case of the nitrogen-free crystals bombarded with any ions apart from nitrogen. The annealing temperature needed for the appearance of the B band increased on increase in the implantation dose. The dose dependence of this band was linear and it showed no saturation up to ≈ 10^{15} cm^{-2} (Fig. 8).

4. CONCLUSIONS

We can summarize the results reported above by pointing out that the application of cathodoluminescence to the study of defects in ion-implanted diamond layers provided new information on the properties of these defects and also on the formation and annealing of the defects. This information confirmed the current views on the properties of disordered regions created by ions and indicated that the interaction between defects present in high concentrations was an important effect.

The authors are grateful to A. V. Spitsyn and V. A. Dravin for the implantation of ions with the aid of the High Voltage Engineering Europa accelerator, to N. A. Shuvalova for annealing the samples, and also to E. A. Konorova and V. V. Krasnopevtsev for their valuable comments and discussion of the results.

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[1] This parameter is introduced by the expression $s = (\Delta h\nu_{max}/h\nu_0\sigma)\cdot 10^{12}$ Pa^{-1}, where $\Delta h\nu_{max}$ is the shift of the most strongly split-off component of the zero-phonon line from its position $h\nu_0$ observed for an undeformed crystal and σ is the elastic stress. For details see Ref. 14.

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