STUDIES OF MAGNETIC SUSCEPTIBILITY IN PLASTICALLY DEFORMED SILICON AND GERMANIUM SINGLE CRYSTALS

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The nature and the type of magnetic ordering in plastically deformed Si and Ge single crystals have been discussed. The nonlinear component of magnetic susceptibility has been found, and its features have been analyzed. An interpretation of the experimental results in the framework of Langevin superparamagnetism has been proposed. The concentration of superparamagnetic clusters in the crystals and their magnetic moments have been estimated. The size distribution functions for magnetic clusters have been plotted on the basis of the superparamagnetic curves obtained for the magnetic susceptibility.

1. Introduction

The EPR spectra associated with dislocations in plastically deformed silicon are known to have a complicated character [1–3]. The analysis of the fine structure of the so-called peripheral lines testified to a strong interaction between nonpaired electrons in the dislocation core, although the issue on the character of such an interaction remained unresolved. Further researches [3, 4] showed that the electronic properties of dislocation cores are mainly determined by the end groups of atoms which have dangling valence bonds, the so-called chains of dislocation dangling bonds (DDB chains). However, the complexity of this picture becomes enhanced due to the simultaneous presence of dislocations of different types and the strong dependence of the real structure of their cores on the conditions of plastic deformation and heat treatment. It is evident that the further progress in the study of dislocation structure features is possible, only provided that various techniques are applied simultaneously and the results obtained are confronted. In this case, informative turned out to be the application of the magnetic susceptibility (MS) method, because the matter is about the cooperative phenomena associated with the magnetic ordering in a system of electron spins on dislocation structures. In this work, our main experimental results obtained when studying the magnetic properties of plastically deformed Si and Ge single crystals are reported, and their theoretical interpretation is proposed.

2. Experimental Part

The magnetic susceptibilities of n-Si ($\rho = 10^4\ \Omega\cdot$cm) and n-Ge ($\rho = 0.3 - 0.5\ \Omega\cdot$cm) single crystals were studied at various degrees of their deformation (ε,%). The concentration of growth dislocations in the crystals did not exceed 10^2 cm^{-2}. Specimens were cut out in the form of rectangular prisms with side orientations (110), $(1\bar{1}1)$, and $(\bar{1}12)$ for Si and (001), (110), and $(1\bar{1}0)$ for Ge. The specimens were $10 \times 4 \times 3$ mm^3 in dimensions. After mechanical-chemical polishing – HF:HNO_3=1:4 for silicon and HNO_3:HF:CH_3COOH=1:1.5:3 for germanium– specimens were deformed by squeezing them along the long edge (along the direction [110] for Si and $[1\bar{1}0]$ for Ge). Specimens were deformed in the temperature interval 670–700 °C. The deformation degree was determined by the relative squeezing of crystals. The reference, nondeformed specimens were subjected to the same thermal and mechanical-chemical treatments. Therefore, all differences between the magnetic characteristics of the reference and deformed specimens, which were established experimentally, can be explained only by the deformation-induced effects.

The MS was measured at two orientations of the external magnetic field **H**: in Si, along the direction $[1\bar{1}1]$ ($\chi_\parallel$) and perpendicularly to it, i.e. along the crystallographic direction $[\bar{1}12]$ ($\chi_\perp$); and in Ge, along the direction [001] ($\chi_\parallel$) and perpendicularly to it, i.e. along the crystallographic direction [110] ($\chi_\perp$). The comparison between the dependences $\chi_\parallel(H)$ and $\chi_\perp(H)$,

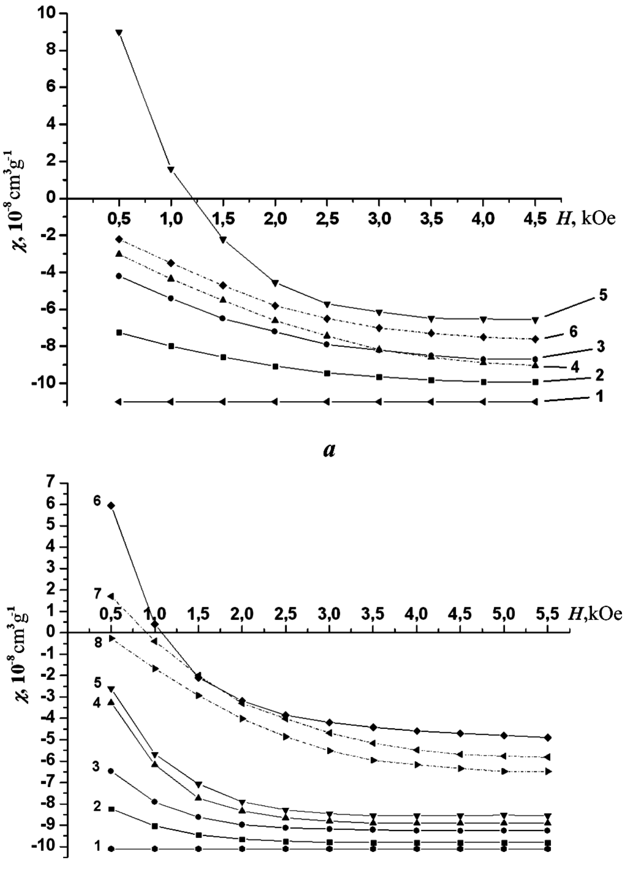

Fig. 1. Dependences $\chi(H)$ for plastically deformed single crystals of silicon (*a*) and germanium (*b*) at 77 K. The deformation degree ε (in per cent): (Si crystals) 0.05, 0.15, 0.3, 0.4, and 1.5 (curves *2* to *6*, respectively), (Ge crystals) 0.3, 2, 4, 10, 14, 22, and 25 (curves *2* to *8*, respectively). Curves *1* correspond to reference (nondeformed) specimens

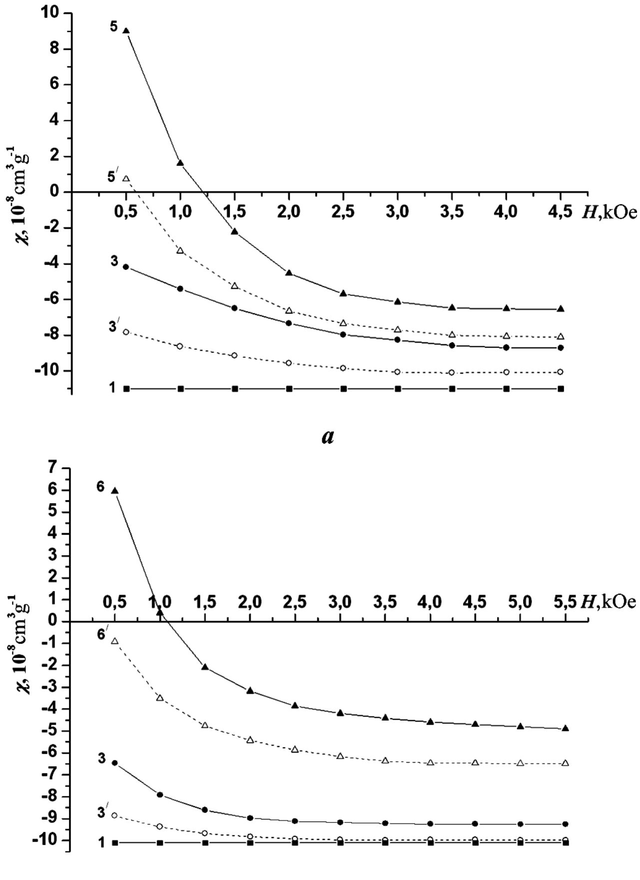

Fig. 2. Dependences $\chi(H)$ for plastically deformed single crystals of silicon (*a*) and germanium (*b*) at 77 (solid symbols) and 300 K (hollow symbols). The deformation degree ε (in per cent): (Si crystals) 0.15 (*3* and *3'*) and 0.4 (curves *5* and *5'*), (Ge crystals) 2 (*3* and *3'*) and 14 (*6* and *6'*). Curves *1* correspond to reference (nondeformed) specimens

which was made for every measured specimen (Si and Ge), testifies that there is an anisotropy with respect to the chosen directions, with $\chi_{\parallel} > \chi_{\perp}$ for every plastically deformed crystal. Since no MS anisotropy was observed in initial, nondeformed Si crystals, we consider that its occurrence is associated with the appearance of dislocations introduced in the course of plastic deformation. Taking the similarity of the dependences $\chi_{\parallel}(H)$ and $\chi_{\perp}(H)$ into account (although the relative difference between the $\chi_{\parallel}$- and $\chi_{\perp}$-values was approximately 25% in a field of 2 kOe and about 5% in a field of 5 kOe), we analyze, in what follows, only the $\chi_{\parallel}$-values. For the simplification of further notations, the indices at the MS and other physical quantities that specify the direction of measurement are omitted.

The dependences $\chi(H)$ were measured on a modernized installation [5], the functioning of which is based on the Faraday method, in the temperature range 77−300 K and at magnetic fields within the interval $0.3 - 5.0$ kOe. The relative measurement error $\varepsilon \leq 1\%$. The reproducibility of the values obtained was good.

3. Results and Their Discussion

In Fig. 1, the dependences of magnetic susceptibility on the magnetic field strength are depicted for plastically deformed single crystals at 77 K: Si ($\varepsilon = 0.05 \div 1.5\%$) and Ge ($\varepsilon = 0.3 \div 25\%$). For the sake of comparison, the dependences $\chi(H)$ obtained for some specimens at temperatures of 77 and 300 K are exhibited in Fig. 2. Since $\chi(H)$ varies more effectively at 77 K, we will analyze them below. The figures demonstrate that the plastic deformation of those crystals, which gives rise to the formation of a significant amount of non-growth dislocations, changes their magnetic properties essentially. On the one hand, a reduction of diamagnetism is observed; on the other hand, there emerges a non-linearity in the dependences of MS on H. The decrease of crystal diamagnetism can be explained 1) by the growth of paramagnetism χ^{par} which is independent of the magnetic field, and 2) by the growth of paramagnetism $\chi^{\text{ord}}(H)$ which depends on H. In our opinion, in the former case, paramagnetism is induced by paramagnetic centers or such ones which are located in less dense groups and do not transit into a magnetically ordered state at $T \geq 290$ °C – these can be EPR-active D-centers (the chains of atoms with dangling bonds) and "isolated" dangling bonds [3]. In the latter case, paramagnetism depends on the magnetic field strength in the interval 0 kOe $< H <$ 5 kOe. The character of the dependence $\chi^{\text{ord}}(H)$ evidences for its superparamagnetic nature. The absence of hysteresis in the studied crystal also confirms this conclusion.

As a physical reason for this paramagnetism to appear, the formation of clusters with magnetically ordered spins (elementary paramagnetic centers) in plastically deformed crystals may be taken. Such clusters can be considered as single-domain magnetic particles. The magnetism of such clusters is similar to the paramagnetism of atoms which possess a magnetic moment and are described by the Langevin function. The main difference consists in that their magnetic moment can be $10^3 - 10^5$ times larger than the magnetic moment of individual atoms.

If the dislocation concentration increases, those effects become more pronounced; it is especially noticeable for plastically deformed germanium crystals (Fig. 1,*b*). However, deviations from this tendency are also observed (Fig. 1,*a*, curves *4* and *6*). In plastically deformed crystal 6, visually observable cracks and cleavages were formed. It is natural to suppose that the anomaly in the MS of silicon specimen 4, for which no cracks or cleavages were detected visually, was caused by the emergence of microcracks in it.

The revealed regularities in the influence of plastic deformation on the magnetic properties are traced especially clearly for Ge specimens. Deviations from the indicated tendency manifested themselves at a much higher deformation degree (Fig. 1,*b*, curves *7* and *8*) than it was in the silicon case. It is evidently connected with a higher plasticity of germanium crystals in comparison with that of silicon ones.

Every experimental dependence $\chi(H)$ presented in Fig. 1 can be considered as a sum of two components – field-independent, χ^{ind}, and field-dependent, $\chi^{\text{ord}}(H)$, ones–the latter is due to the ordering of magnetic centers in the crystal:

$$\chi(H) = \chi^{\text{ord}}(H) + \chi^{\text{ind}}. \tag{1}$$

The component χ^{ind} is composed of the lattice susceptibility χ_L and a paramagnetic component χ^{par} independent of the magnetic field strength:

$$\chi^{\text{ind}} = \chi^{\text{par}} + \chi_L, \tag{2}$$

Hence, with regard for our assumptions and remarks made above, the expression, which could describe the observable dependences of the magnetic susceptibility on the magnetic field strength for plastically deformed Si and Ge crystals, can be written down in the following form:

$$\chi\,(H) = \chi^{\text{ord}}\,(H) + \chi^{\text{par}} + \chi_L =$$

$$= N\mu L'\left(\frac{m_{cl}H}{kT}\right) + \chi^{\text{par}} + \chi_L, \tag{3}$$

where N is the concentration of magnetically ordered clusters, $\mu = N_0\mu_B\varrho\sqrt{s(s+1)}$ is the magnetic moment of such a cluster (as the first approximation, we assume that the magnetic moments of clusters are identical), $L'(x)$ is the derivative of the Langevin function, k the Boltzmann constant, T the temperature, N_0 the number of paramagnetic centers in a magnetic cluster, μ_B the Bohr magneton, ϱ the g-factor (for estimation, we admit $\varrho = 2$), and s the spin of every paramagnetic center the cluster consists of (for estimation, $s = 1/2$). Then, Eq. (3) looks like

$$\chi\,(H) = NN_0\mu_B\varrho\sqrt{s(s+1)}\,\left(\frac{N_0\mu_B\varrho\sqrt{s(s+1)}}{kT}\times\right.$$

$$\times\left(1 - \text{cth}^2\left(\frac{N_0\mu_B\varrho\sqrt{s(s+1)}}{kT}\,H\right)\right) +$$

$$\left. + \frac{kT}{N_0\mu_B\varrho\sqrt{s(s+1)}\cdot H^2}\right) + \chi^{\text{par}} + \chi_L. \tag{4}$$

By approximating the experimental curves (Fig. 1) with the use of function (4), it is possible to evaluate the quantities N_0 and N. Examples of approximation are given in Figs. 3 and 4. The same function approximates well

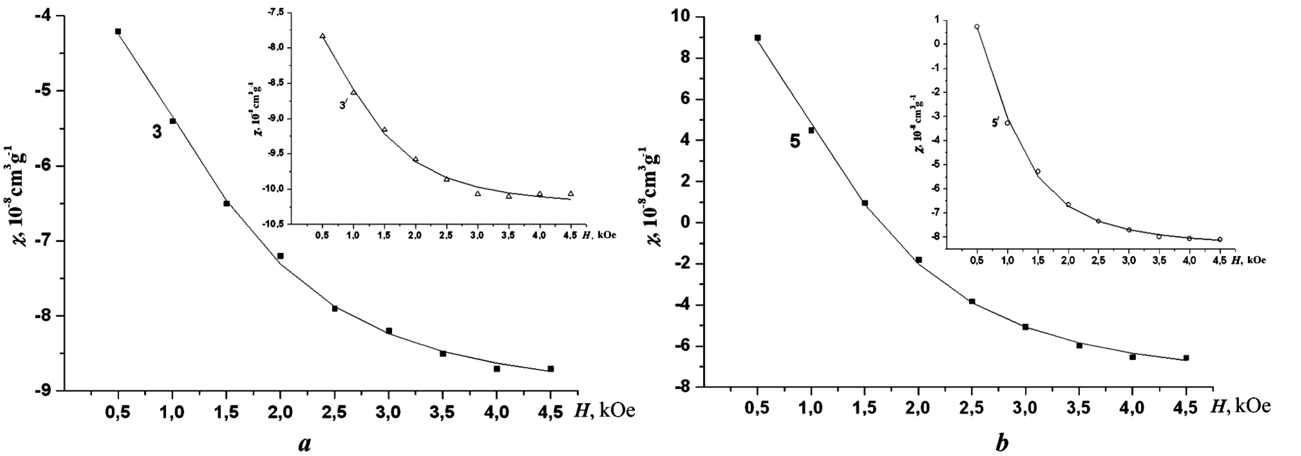

Fig. 3. Approximations by expression (4) of the experimental dependences $\chi(H)$ for plastically deformed Si specimens 3 (*a*) and 5 (*b*) measured at 77 and 300 K (in the insets)

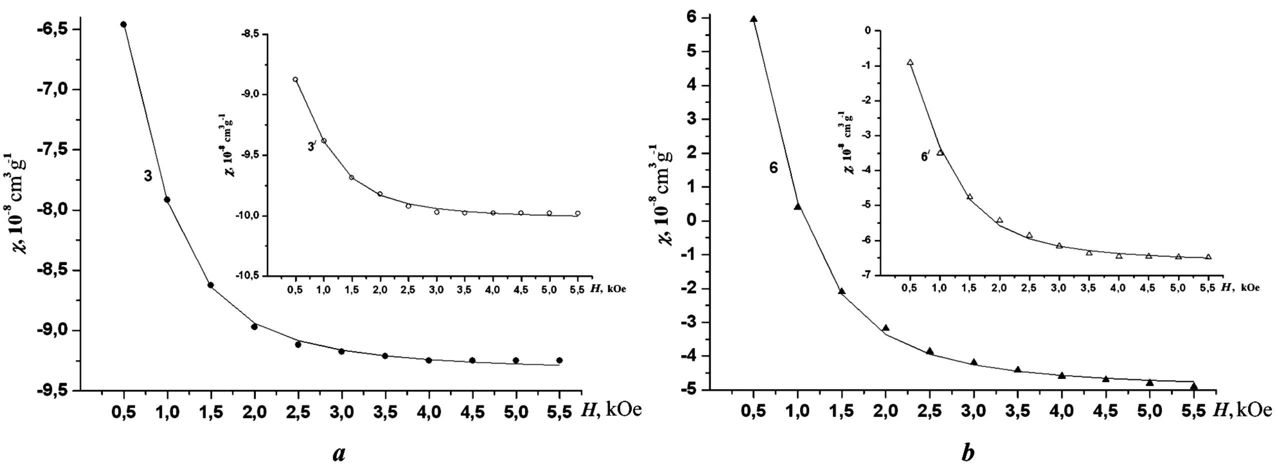

Fig. 4. The same as in Fig. 3 but for Ge specimens 3 (*a*) and 6 (*b*)

the dependences $\chi(H)$ measured at room temperature (see the insets in the figures).

When analyzing the results obtained, a number of interesting regularities can be revealed: 1) as the deformation degree increases, the concentration of paramagnetic centers in clusters remains constant in both the Si and Ge cases, provided that the specimens are free of observable microcracks or cleavages; 2) in the course of plastic deformation, the concentration of emerging magnetic clusters in germanium crystals is lower than that in silicon ones, but their magnetic moments are approximately twice as large. Two more, clearly distinguished regularities are also observed (especially for Ge crystals): the increase of deformation degree is accompanied by the growth of superparamagnetic cluster concentration, and the appearance of cracks and cleavages gives rise to a reduction of cluster magnetic moments (by means of the diminution of the concentration of paramagnetic centers in clusters), may be owing to the pairing between dangling bonds.

The values for N_0 and N obtained by the approximation procedure are quoted in Tables 1 and 2.

T a b l e 1. Silicon

Specimen No.	2	3	4	5	6
Deform. deg. (ε), %	0.05	0.15	0.3	0.4	1.5
$N_0 \times 10^3$	0.92	1	0.74	0.95	0.74
$N \times 10^9$, cm^{-3}	0.39	7.3	15	24	12

T a b l e 2. Germanium

Specimen No.	2	3	4	5	6	7	8
Deform. deg. (ε), %	0.3	2	4	10	14	22	25
$N_0 \times 10^3$	1.77	1.81	1.81	1.74	1.74	0.92	0.81
$N \times 10^9$, cm^{-3}	0.84	1.4	2.9	3.3	5.9	1.2	1.3

The experimental results obtained allowed us to identify the nature of magnetic ordering in plastically deformed Si and Ge crystals. The mentioned experimental fact concerning the influence of cracks and cleavages on the MS of plastically deformed crystals gives us a reason to reject the assumption that the observed ordering was induced by the accumulation of magnetic impurities at dislocations, because, if so, the mechanical damages of the crystals could not affect the MS so substantially.

There is another important proof that the paramagnetic components of MS in plastically deformed silicon crystals are related to dislocations. Namely, making use of the D-center annealing technique described in work [6], we subjected plastically deformed Si crystals to a thermal treatment and measured their MS. It turned out that, after annealing, the susceptibility values for plastically deformed crystals and the reference specimen coincided. In other words, the components $\chi^{\rm par}$ and $\chi^{\rm ord}(H)$ in every plastically deformed crystal subjected to thermal annealing vanished. This is what should be expected, if one takes into account that the thermal treatment of crystals carried out following the technique of work [6] brings about the complete disappearance of the EPR signal from D-centers, as well as from donor and acceptor levels in the energy gap coupled with them. This may occur as a result of the dislocation core reconstruction accompanied by the pairwise closure of dangling bonds into the state with $S = 0$ [6, 7].

The alternative assumption that the complete disappearance of $\chi^{\rm par}$ and $\chi^{\rm ord}(H)$ is caused by the migration of the magnetic impurity away from dislocations should be considered as poorly substantiated, even because the chosen heat treatment regime ($T = 900$ °C, $t = 15$ min) is evidently insufficient for the dislocations to get free of impurity atoms accumulated at them, so that the values of $\chi^{\rm par}$ and $\chi^{\rm ord}(H)$ would be different from zero. We also note that, according to our estimations, the concentrations of magnetic impurities (e.g., Fe^{2+}), which are needed for the MS values obtained experimentally for plastically deformed Si and Ge crystals to be achieved, exceed their solubility limit in those substances.

In the model proposed above, we assumed, as the first approximation, that the magnetic moments of clusters are identical. However, the actual situation is evidently quite different. Therefore, a no less important problem is to study the cluster distribution over their magnetic moments. For this purpose, we introduced the corresponding distribution function $f(\mu)$, so that $f(\mu)d\mu$ is the concentration of clusters with the magnetic moments ranging from μ to $\mu + d\mu$. Then, for the magnetic susceptibility, we obtain

$$\chi^{\rm teor} = \int\limits_0^\infty \mu f(\mu) \left(\frac{\mu}{kT} \left(1 - {\rm cth}^2 \left(\frac{\mu H}{kT} \right) \right) + \frac{kT}{\mu H^2} \right) d\mu. \tag{5}$$

Usually, the function $f(\mu)$ is accepted to be logarithmically normalized [8, 9], i.e.

$$f(\mu) = \frac{n}{\sqrt{2\pi}\sigma} \frac{1}{\mu} \exp\left[-\frac{\ln^2(\mu/\langle\mu\rangle)}{2\sigma^2} \right]. \tag{6}$$

It is driven by three parameters: n, σ, and $\langle\mu\rangle$. Therefore, the construction of the distribution function is reduced to finding those parameters. One of the ways to obtain them is the least square method, which consists in the minimization of the expression

$$\Delta_\chi = \sum_{i=1}^N \left(\chi^{\rm teor}(H_i) - \chi^{\rm exp}(H_i) \right)^2, \tag{7}$$

where $\chi^{\rm theor}(H_i)$ is determined by formula (5), $\chi^{\rm exp}(H_i)$ are experimental values of the magnetic susceptibility, and N is the number of experimental points. The parameters n, σ, and $\langle\mu\rangle$ are found from the necessary condition for the minimum of functional (7) to exist:

$$\begin{cases} \frac{\partial\Delta_\chi}{\partial n} = 0, \\ \frac{\partial\Delta_\chi}{\partial\sigma} = 0, \\ \frac{\partial\Delta_\chi}{\partial\langle\mu\rangle} = 0. \end{cases} \tag{8}$$

Hence, we obtain system (8) of three nonlinear algebraic equations to determine n, σ, and $\langle\mu\rangle$. The parameter n linearly enters the first equation of the system, so it can be expressed in terms of σ and $\langle\mu\rangle$. Therefore, the system of three equations (8) is reduced to a system of two nonlinear equations

$$F_1 = \sum_{i=1}^N \left[n(\sigma,\ \langle\mu\rangle) \int\limits_0^\infty \frac{1}{\sqrt{2\pi}\sigma} W(\sigma, \langle\mu\rangle) \times \right.$$

$$\left. \times \left[\frac{\mu}{kT} \left(1 - {\rm cth}^2 \left(\frac{\mu H_i}{kT} \right) \right) + \frac{kT}{\mu H_i^2} \right] d\mu - \chi^{\rm exp} \right] \times$$

$$\times \int\limits_0^\infty \frac{\left(2\ln^2(\mu/\langle\mu\rangle) - \sigma^2\right)}{\sqrt{2\pi}\sigma^4} W(\sigma, \langle\mu\rangle) \times$$

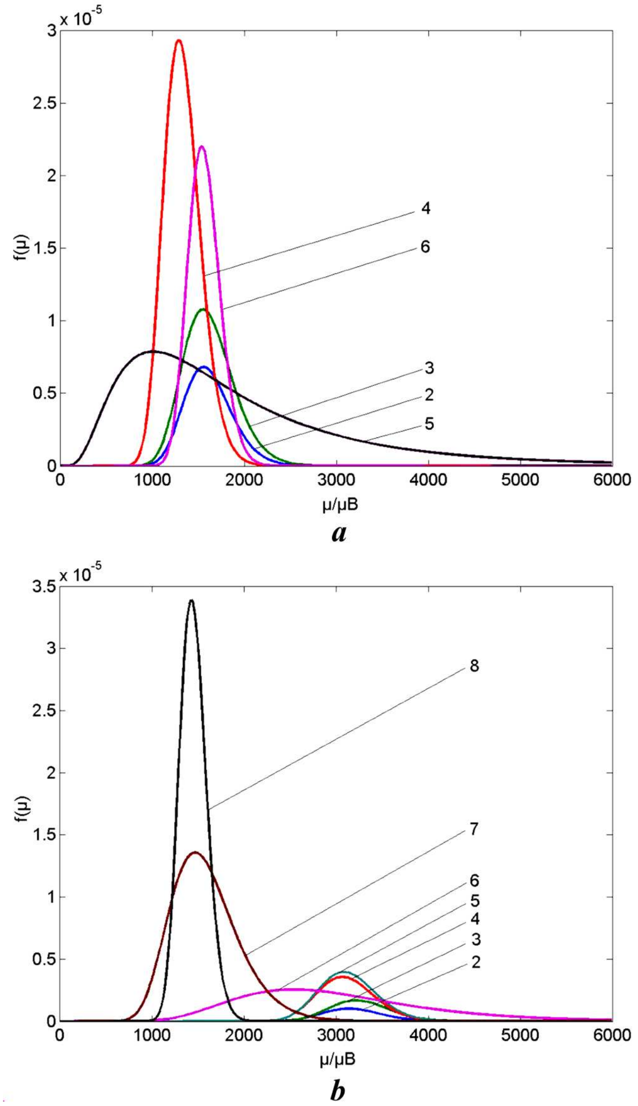

Fig. 5. Distribution functions of clusters over their magnetic moments in silicon (a) and germanium (b)

$$\times \left[\frac{\mu}{kT}\left(1-\mathrm{cth}^2\left(\frac{\mu H_i}{kT}\right)\right)+\frac{kT}{\mu H_i^2}\right] d\mu = 0,$$

$$F_2 = \sum_{i=1}^{N}\left[n\left(\sigma,\ \langle\mu\rangle\right)\int_0^\infty \frac{1}{\sqrt{2\pi}\sigma} W\left(\sigma,\langle\mu\rangle\right)\times\right.$$

$$\left.\times \left[\frac{\mu}{kT}\left(1-\mathrm{cth}^2\left(\frac{\mu H_i}{kT}\right)\right)+\frac{kT}{\mu H_i^2}\right] d\mu - \chi^{\exp}\right] \times$$

$$\times \int_0^\infty \frac{\left(\mu\ln\left(\mu/\langle\mu\rangle\right)-\sigma^2\right)}{\sqrt{2\pi}\sigma^3\left\langle\mu\right\rangle^2} W\left(\sigma,\langle\mu\rangle\right) \times$$

$$\times \left[\frac{\mu}{kT}\left(1-\mathrm{cth}^2\left(\frac{\mu H_i}{kT}\right)\right)+\frac{kT}{\mu H_i^2}\right] d\mu = 0.$$

The system obtained is analytically complicated, and its approximate solution can lead to cumbersome calculation operations. Therefore, we found the minimum of functional (7) by tabulating the latter within the intervals $\sigma = 0.1 \div 3$ and $\mu = 1000 \div 10000$, making allowance for the relation $n(\sigma, \langle\mu\rangle)$. As the initial approximations $\sigma^{(0)}$ and $\langle\mu\rangle^{(0)}$, we took those σ- and $\langle\mu\rangle$-values, at which functional (7) is minimal. The values obtained for n, σ, and $\langle\mu\rangle$ were used to plot the distribution of particles over their dimensions. The corresponding results are depicted in Fig. 5. The magnetic moments of clusters, which were obtained on the basis of their distribution, agree well with the relevant values determined in the framework of model (4) (Tables 3 and 4).

From Fig. 5, one can see that, depending on the deformation degree, the most probable sizes of magnetic particles vary from 7×10^{-6} to 3×10^{-5} cm in the Si case, and from 2×10^{-6} to 3.5×10^{-5} cm in the Ge one.

We now comment, in brief, on the results, which are mainly of practical importance. Along with the MS of plastically deformed crystals, we also studied the susceptibility of silicon doped with the elements – in particular, Gd and Ni – which actively interact with it by forming silicides, and Si crystals with a defective surface structure [10]. The whole body of the results obtained (taking also into consideration those obtained for plastically deformed crystals) led us to a conclusion that non-growth dislocations – irrespective of the mechanism of their formation – stimulate a magnetic ordering which, in turn, is responsible for the emergence of a non-linearity in the field dependences of MS. This hypothesis can be easily verified experimentally. It enables the MS measurement method to be applied to the solution of some practically important problems, in particular, for detecting microcracks formed in the course of plastic deformation of crystals, for revealing the non-growth dislocations in defective surface layers,

T a b l e 3. Silicon

Specimen No	2	3	4	5	6
μ/μ_B by model (4)	1593	1732	1282	1645	1282
μ/μ_B by model (5)	1598	1597	1318	1586	1053

T a b l e 4. Germanium

Specimen No	2	3	4	5	6	7	8
μ/μ_B by model (4)	3065	3135	3137	3014	3004	1593	1403
μ/μ_B by model (5)	3171	3250	3255	3116	2852	1550	1444

as well as precipitated atoms of doping and residual elements.

4. Conclusions

1. The presence of non-growth dislocations, irrespective of the mechanism of their formation, in covalent semiconductors has been found to give rise to the emergence of a non-linearity in the dependences $\chi(H)$. This fact can be effectively used to detect the non-growth dislocations and, on this basis, to monitor the quality of semiconductor materials.
2. From the analysis of the nonlinear dependences $\chi(H)$ of plastically deformed Si and Ge single crystals, the assumption has been made that some part of paramagnetic centers includes nanoclusters, the magnetism of which has the superparamagnetic nature. The proposed model has been used to evaluate the concentration of such clusters and their magnetic moments.
3. On the basis of the superparamagnetic curves for a magnetic susceptibility, the distribution functions of magnetic clusters over their size have been constructed. The corresponding analysis of experimental results has been carried out to determine the most probable sizes of magnetic clusters and their magnetic moments.
4. It has been demonstrated that, in the range of low deformations, the concentration of individual paramagnetic centers (supposedly, D-centers) smoothly increases with the dislocation concentration, and so does their contribution to the MS. At the same time, in the range of strong deformations, the contribution of dislocation dangling bonds to the MS substantially diminishes. This effect is associated with the appearance of cracks and cleavages, which can be detected visually in the specimens, as well as microcracks.

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ДОСЛІДЖЕННЯ МАГНІТНОЇ СПРИЙНЯТЛИВОСТІ ПЛАСТИЧНО ДЕФОРМОВАНИХ МОНОКРИСТАЛІВ КРЕМНІЮ ТА ГЕРМАНІЮ

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Р е з ю м е

Обговорено природу і тип магнітного впорядкування у пластично деформованих монокристалах Si та Ge. Виділено нелінійну складову магнітної сприйнятливості та проаналізовано її особливості. Запропоновано інтерпретацію експериментальних результатів у рамках моделі ланжевенівського суперпарамагнетизму. Оцінено концентрацію суперпарамагнітних кластерів у кристалах та їх магнітні моменти. На основі суперпарамагнітних кривих магнітної сприйнятливості побудовано функції розподілу магнітних кластерів за розмірами.