

## EPR Spectroscopy of Diazoquinone–Novolac Resist Films Implanted with P<sup>+</sup> and B<sup>+</sup> Ions

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**Abstract**—The nature of stable radicals in FP9120 positive photoresist films implanted with boron and phosphorus ions and deposited on the surface of single-crystal silicon wafers has been determined using the EPR technique. At an implantation fluence of  $6 \times 10^{15} \text{ cm}^{-2}$ , a narrow singlet isotropic line with a  $g$ -factor of 2.0064 is observed in the EPR spectrum. As the fluence increased to  $1.2 \times 10^{16} \text{ cm}^{-2}$ , the  $g$ -factor decreased to values close to the  $g$ -factor of the free electron. The concentration of paramagnetic centers was higher during implantation of phosphorus ions than in the samples implanted with boron ions. This difference is due to a smaller contribution of nuclear stopping during B<sup>+</sup> implantation, which does not exceed 10–15% of electronic stopping. The formation of long-lived paramagnetic centers recorded by EPR a week after implantation of positive phenol–formaldehyde photoresist is due to the presence of a powerful system of conjugated >C=O and –C=C– multiple bonds in the structure of the radicals.

**Keywords:** photoresist, implantation, phosphorus, boron, EPR, semiquinonoid radical anions

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### INTRODUCTION

One of the most promising methods for controlling the electrophysical, strength, and biological properties of the surface layers of polymers is ion implantation [1, 2]. Thus, ion bombardment leads to a monotonic increase in the electrical conductivity of the implanted layer, the value of which can vary by 10–18 orders of magnitude [3]. The advantage of magnetic resonance methods (in particular, EPR) is high sensitivity, which is especially important in the study of thin films. Electron paramagnetic resonance spectroscopy has been widely used in physics, chemistry, biology, and medicine since the middle of the last century. It is the main, and often the only possible, method for studying free radical processes [4].

The aim of this study was to investigate radiation-induced processes in films of the diazoquinone–novolac photoresist FP9120 during implantation of boron and phosphorus ions and irradiation with  $\gamma$ -rays.

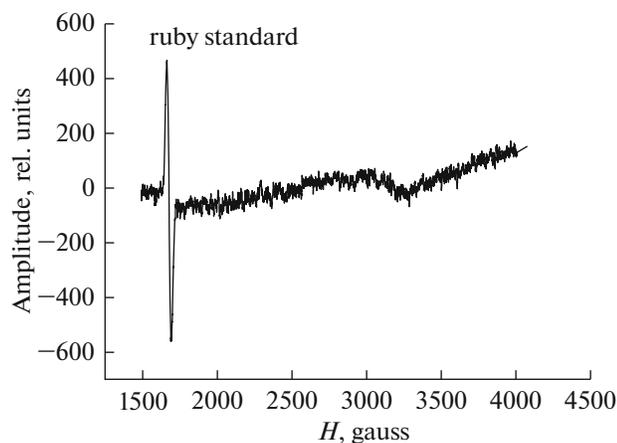
### EXPERIMENTAL

Films of positive photoresist FP9120, which is a composite of photosensitive *o*-naphthoquinone diazide and phenol–formaldehyde resin, with a thickness of 1.0, 1.8, and 2.5  $\mu\text{m}$  were deposited on a Si surface by spin coating [5]. The thickness  $h$  of the photoresist film was determined by the spinning speed to be 1.0,

1.8, or 2.5  $\mu\text{m}$  at a speed of  $\nu = 8300, 2900, \text{ or } 1200 \text{ rpm}$ , respectively. As substrates, wafers (100 mm in diameter) of KDB-10 single crystal silicon with the (111) orientation were used. Before forming the photoresist film, the silicon wafers were subjected to a standard surface cleaning cycle in organic and inorganic solvents. After applying the photoresist to the working side of the wafer, drying at a temperature of 88°C was carried out for 50–55 min. The thickness of the photoresist films was monitored using a MII-4 microinterferometer.

The EPR spectra of the photoresist films were recorded at room temperature on an X-band RadioPan SE/X-2543 spectrometer with an H<sub>102</sub> cavity. The maximum microwave power in the cavity was 200 mW. The magnetic field modulation frequency was 100 kHz with an amplitude of 0.1 mT. To control the quality factor of the measuring cavity, adjust the magnetic field modulation phase, and calibrate the magnetic component of microwave radiation, a ruby crystal mounted on the cavity wall was used. The sensitivity of the spectrometer was  $3 \times 10^{12} \text{ spin/mT}$ .

Implantation with phosphorus P<sup>+</sup> (energy 100 keV) and boron B<sup>+</sup> (energy 60 keV) ions in the fluence range of  $6 \times 10^{14}$ – $1.2 \times 10^{16} \text{ cm}^{-2}$  at an ion current density of  $j = 4 \mu\text{A/cm}^2$  was carried out in a residual vacuum of not higher than  $10^{-5} \text{ mmHg}$  on the Vesuvius-6 ion beam accelerator. Irradiation of polymer films with  $\gamma$ -rays at a dose of up to 270 kGy was carried out on an



**Fig. 1.** EPR spectrum of a photoresist film of a 1.0  $\mu\text{m}$  thickness irradiated with  $\gamma$ -rays with a dose of 270 kGy.

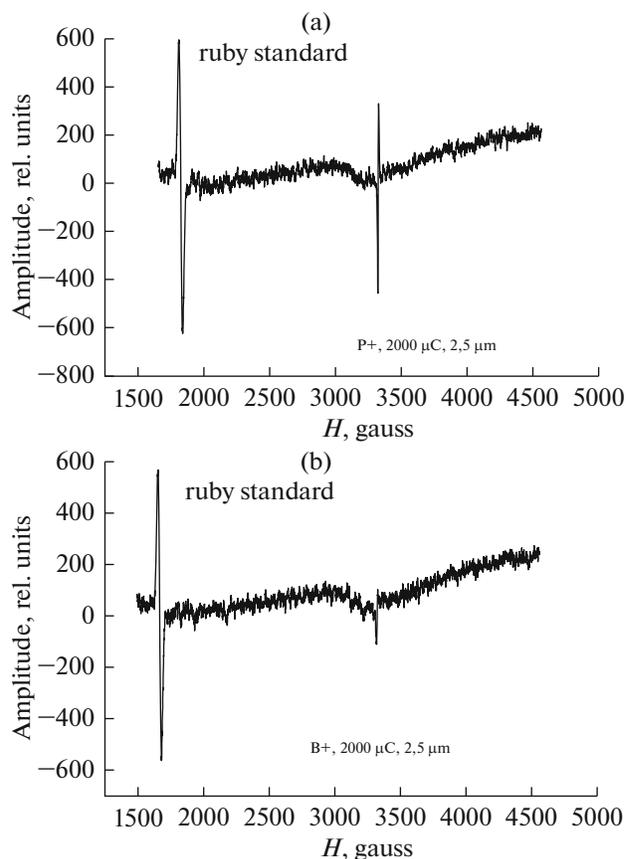
MRKh- $\gamma$ -25M facility with a  $^{60}\text{Co}$  source. The absorbed dose rate was  $0.25 \pm 0.007$  Gy/s.

## RESULTS AND DISCUSSION

There were no EPR signals in the unirradiated photoresist film. Irradiation with  $^{60}\text{Co}$   $\gamma$ -rays at doses up to 270 kGy and ion implantation at fluences up to  $5 \times 10^{15} \text{ cm}^{-2}$  also did not lead to the formation of stable paramagnetic centers. The spectrum measured 4 h after cessation of irradiation (Fig. 1) was identical to the spectrum of the unirradiated photoresist.

At an implantation fluence of  $6 \times 10^{15} \text{ cm}^{-2}$ , a narrow singlet line of a  $\sim 4$  G width appears in the EPR spectrum of the  $\text{P}^+$ -implanted films. This line was not observed in the samples implanted with boron, most likely due to its low intensity. The line shape is characteristic of samples containing paramagnetic centers with an anisotropic  $g$ -factor [6]. Its anisotropic components are  $g_{\parallel} = 2.0064$  and  $g_{\perp} \sim 2.0015$ . Kuzina et al. [7] observed EPR lines with similar anisotropic components in irradiated lignin and associated them with the formation of the semiquinone radical ion. It was noted that partial localization of the unshared electron on the oxygen atom due to strong spin-orbit coupling leads to anisotropy of the  $g$ -factor.

After increasing the ion implantation fluence to  $1.2 \times 10^{16} \text{ cm}^{-2}$ , the EPR line shifted upfield was observed in all the samples studied (Fig. 2). The value of its  $g$ -factor decreased to 2.00127–2.00254 (Table 1) to be very close to the  $g$ -factor of the free electron ( $g_s = 2.0023$ ). The line becomes asymmetric (Fig. 3), and the asymmetry parameter varies widely:  $a/b = (1.2\text{--}1.75)$ , where  $a$  and  $b$  are the amplitudes of the low- and high-field parts of the first derivative of the EPR absorption line, respectively. This indicates strong spin-orbit coupling, which leads to a decrease in the  $g$ -factor in accordance with the expression [6]:



**Fig. 2.** EPR spectrum of a photoresist film a 2.5  $\mu\text{m}$  thickness bombarded with (a) phosphorus and (b) boron ions at an implantation fluence of  $1.2 \times 10^{16} \text{ cm}^{-2}$ .

$$g = g_s(1 - a\lambda/\Delta E), \quad (1)$$

where  $\lambda$  is the spin-orbit coupling constant,  $\Delta E$  is the splitting between the ground and the closest energy orbital states participating in the orbital motion; and  $a$  is the factor depending on the nature of the paramagnetic center and its orientation with respect to the external magnetic field.

Thus, with an increase in the implantation dose, the spin-orbit coupling increases, most likely, due to an increase in the degree of delocalization of the unpaired electron.

The concentration of paramagnetic centers was higher in the case of implantation of phosphorus ions than in samples implanted with  $\text{B}^+$  ions (Table 1). Moreover, the EPR line width in  $\text{P}^+$ -implanted films was almost two times smaller. The narrowing of the EPR line indicates an increase in the exchange interaction, which is due to the overlap of the delocalization regions of paramagnetic centers [8]. Note that the concentration of paramagnetic centers was higher in the thinner films (1.0  $\mu\text{m}$ ) in both boron and phosphorus implantation cases (Table 1). This is due to the fact that the absorbed dose per unit volume of the

**Table 1.** Parameters of the EPR line in photoresist films implanted at an ion fluence of  $1.2 \times 10^{16} \text{ cm}^{-2}$ 

Ion	Film thickness, $\mu\text{m}$	$g$	$\Delta H$ , G	Amplitude, rel. units	Concentration of radicals, rel. units
P <sup>+</sup>	1.0	2.0018	5.30	1029.7	28924
	2.5	2.0020	5.47	782.3	23407
B <sup>+</sup>	1.0	2.0021	8.86	296.6	23283
	2.5	2.0014	9.05	192.9	15799

polymer is less for the thick films (2.5  $\mu\text{m}$ ) and, consequently, the degree of their radiation-induced conversion is lower.

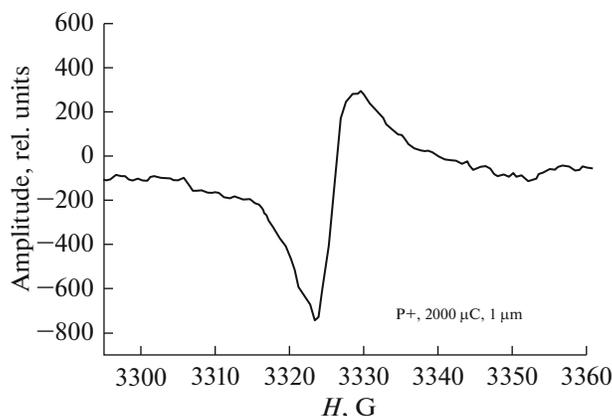
The presence of a single line in the EPR spectrum with a  $g$ -factor close to that of the free electron is characteristic of various carbon-containing materials: coal; graphite; and pyrolyzed, irradiated, or intrinsically conducting polymers [8]. The specific structure of this paramagnetic center has not been finally established. Slightly differing in width and magnitude of the  $g$ -factor, singlets can belong to various radicals, for example, having conjugated carbon–carbon bonds or with partial localization of the spin density on the oxygen atom (phenoxyl radicals, semiquinones, etc.) [7]. For example, the EPR line with a  $g$ -factor of  $2.0025 \pm 0.0005$ , which was previously observed in various polymers (polyethylene, PET, polyimide) implanted with nitrogen and argon ions, is reportedly [8] due to unpaired electrons delocalized over the polyconjugated  $\pi$ -system. A line with a  $g$ -factor of 2.0020 was also observed in poly(methyl methacrylate)–fullerene  $\text{C}_{60}$  composites and was associated with fullerene–oxygen complexes [9].

Note that the monosubstituted phenol radicals are characterized by a  $g$ -factor of 2.0048–2.0037, depend-

ing on the presence and composition of the side groups [4]. Thus, the paramagnetic centers observed in this work most likely include oxygen atoms in their composition, but do not seem to be phenolic radicals.

The change in the cavity loss (cavity  $Q$ -factor) by introducing the test samples into the cavity resonator was monitored by measuring the change in the amplitude of a standard ruby sample glued to the cavity wall. The tuned circuit quality factor (amplitude of the reference signal) decreased by 7–14% with an increase in the film thickness in both the initial and implanted samples (Table 2). After implantation, the amplitude of the reference signal increased, with the increment being greater in the case of the introduction of phosphorus ions (Table 2). This phenomenon is unexpected, since the implantation increases the electrical (ohmic) conductivity of polymer films [8], which should lead to a decrease in the circuit quality factor and, accordingly, to a decrease in the amplitude of the reference signal.

An increase in the quality factor of the cavity with implanted samples compared to the initial films may be due to the presence of a small amount of water in the photoresist film (up to 1% of the photoresist weight). Water can also enter the unirradiated polymer



**Fig. 3.** Line shape of the EPR signal from the 1.0- $\mu\text{m}$  photoresist film bombarded with phosphorus ions at a fluence of  $1.2 \times 10^{16} \text{ cm}^{-2}$ .

**Table 2.** Amplitude of the EPR signal (in rel. units) of a standard (ruby) in the presence of a photoresist film on silicon is the resonator cavity

Film thickness, $\mu\text{m}$	Unirradiated	$\text{P}^+$	$\text{B}^+$
		$1.2 \times 10^{16} \text{ cm}^{-2}$	$1.2 \times 10^{16} \text{ cm}^{-2}$
1.0	1032	1304	1286
2.5	918	1213	1130

from the atmosphere. It is known [8] that water absorbs microwave radiation well. Therefore, when a photoresist film with residual water is placed in the cavity, its quality factor deteriorates and, accordingly, the amplitude of the EPR signal of the standard decreases. Moreover, the thicker the photoresist film, the greater the amount of water in it and the worse the cavity quality factor. Under implantation conditions (vacuum of at least  $10^{-5}$  mmHg), water is removed (evaporation + dehydration reactions) from the photoresist, which should lead to an increase in the cavity quality factor and, accordingly, to an increase in the EPR signal of the reference standard, which was the case observed in the experiment.

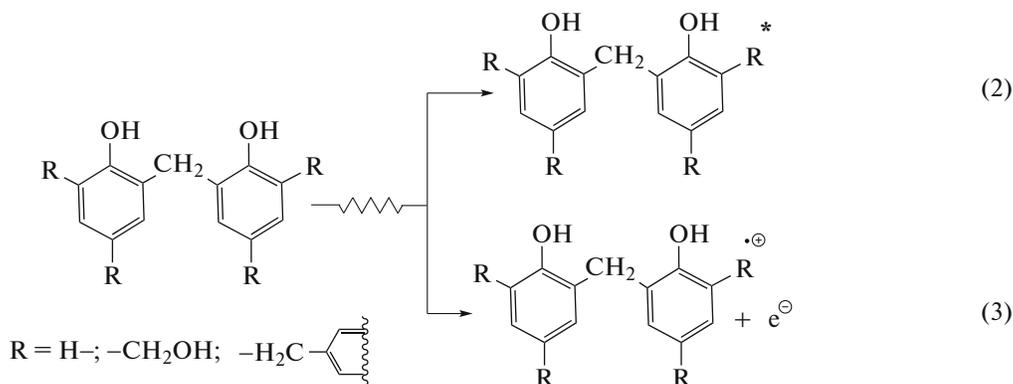
To explain the obtained experimental data, it is necessary to take into account that phenol-formaldehyde resins, which are the main component of the photoresist, are network polymers with a rather variable composition. Their average molecular weight, degree of crosslinking, and other physicochemical properties strongly depend on the phenol to formaldehyde ratio during condensation, as well as on the resin curing conditions (temperature, time, mechanical impact) and the subsequent process operations [10].

Ion bombardment of a photoresist induces a complex set of physical and chemical processes that have a noticeable effect on the structure and composition of polymers. For light ions, in particular  $\text{B}^+$ , the main mechanism of stopping in a polymer is the interaction with electrons of the target material—electronic stopping [8]. In addition to ions, excited polymer macromolecules are also formed during this process, both by direct impact of an ionizing particle and in secondary processes of neutralization of oppositely charged particles in the track.

Nuclear stopping makes a significant contribution at low energies—at the end of the ion range—and leads to the degradation of macromolecules, the formation of disturbed regions, and subsequently (at high doses) to the formation of an amorphized carbon layer. With an increase in the mass of a high-energy particle, the fraction of energy loss due to nuclear stopping will increase. For the  $\text{P}^+$  ion with an energy of 60 keV, it is 40% [8].

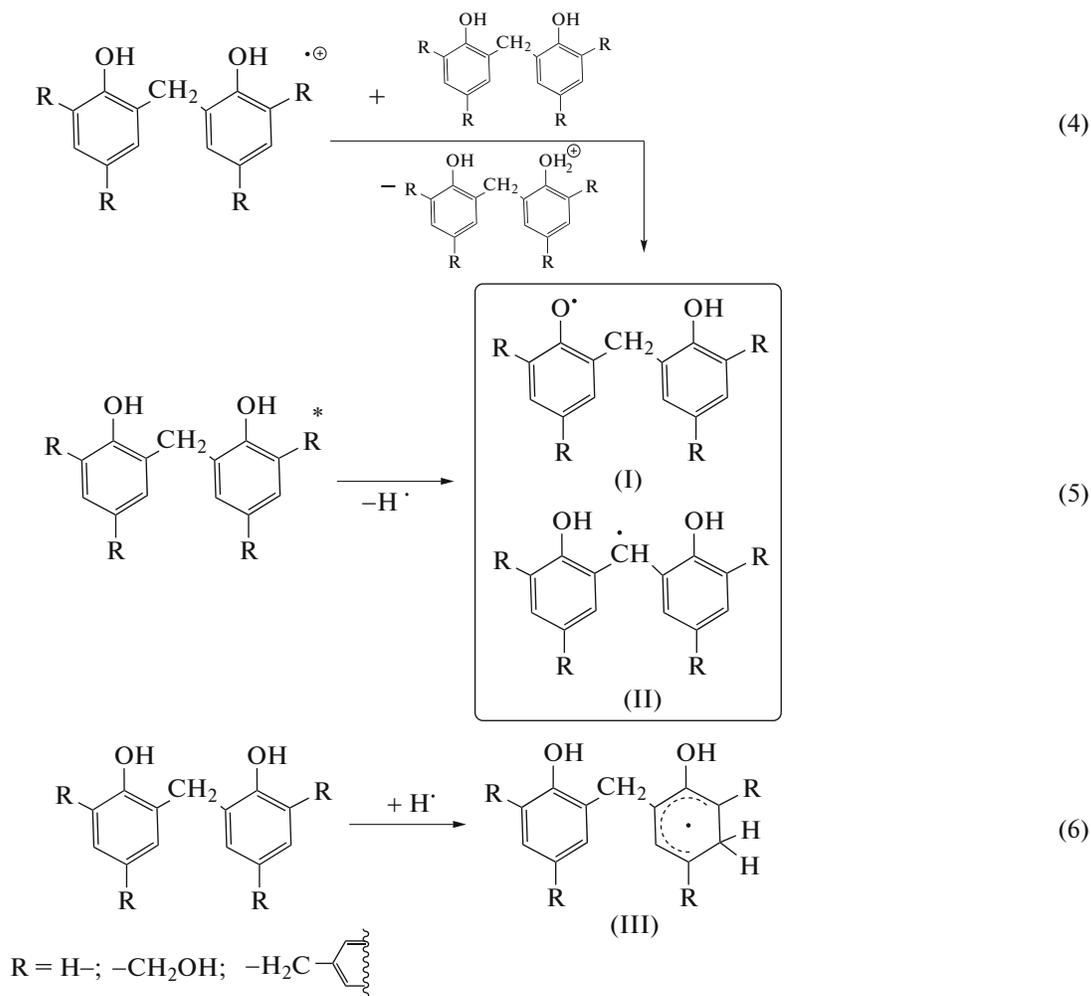
The contribution of the primary ionization of molecules during ion implantation of polymers dominates; it accounts for more than half of the energy lost upon electronic stopping (according to some reports, up to 80%) [11]. Knocked-out electrons are quite mobile, they can move over distances on the order of 100 nm in the polymer, causing radiation-induced processes and forming latent tracks [8, 11].

Unlike corpuscular radiation,  $\gamma$ -rays of isotopic sources transfer energy only to electrons and do not interact with atomic nuclei. In a simplified form, the effect of high-energy ions and  $\gamma$ -radiation on phenol-formaldehyde resin can be reduced to excitation (reaction (2)) and ionization (reaction (3)) of polymer macromolecules.



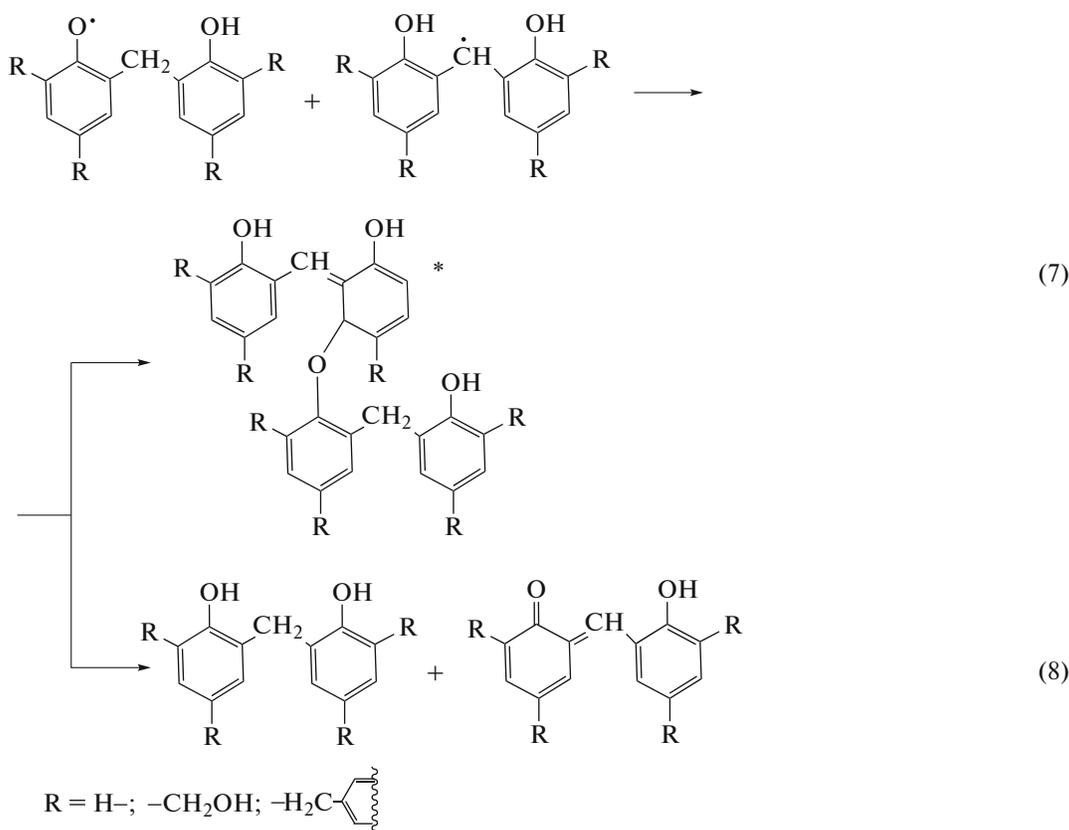
After thermalization, the electron formed in reaction (3) will mainly enter into neutralization reactions with positively charged ions. The neutralization is facilitated by a low dielectric constant of the medium and, in the case of ion implantation, also by a high concentration of ions along the particle track. Electrons that have escaped neutralization can attach to phenolic or carbonyl-containing units of the polymer [12, 13]; the amount of the carbonyl groups will increase significantly with an increase in the absorbed dose.

The high yields of excited species and their relative stability due to the distribution of excess energy over the conjugated  $\pi$ -bonds of the aromatic ring are the reasons for the high radiation stability of phenol-formaldehyde resins in comparison with carbochain polymers [1] or aliphatic alcohols [14]. Free radicals during the irradiation of phenol-formaldehyde resins will be formed as a result of ion-molecule reactions, for example (4); during the decay of excited molecules via reaction (5); and various radical-molecule reactions (mainly, the addition of radicals), for example (6).



The highest thermodynamic stability will be shown by radicals of the phenoxyl (I) and methylene (II) types. The main mechanism of their decay will be combination to form covalent crosslinks between polymer chains, for example, via reaction (7). This process is manifested in

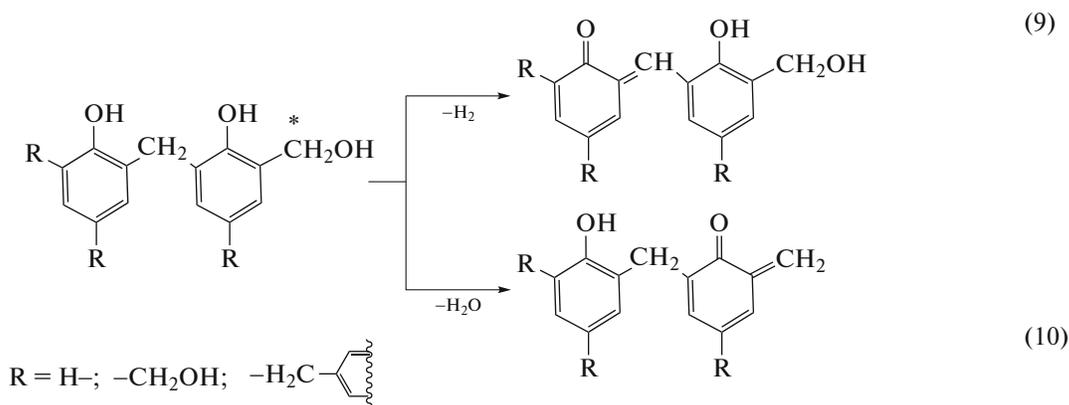
hardening of the photoresist film during irradiation, as we observed in [15]. With a much lesser likelihood, phenoxyl (I) and methylene (II) type radicals disproportionate, for example, via reaction (8), resulting in the formation of quinonemethine moieties in the polymer.



We did not detect long-lived paramagnetic centers in the EPR spectra of the photoresist irradiated with low-intensity <sup>60</sup>Co  $\gamma$ -radiation at a dose of 270 kGy. Therefore, the phenoxy (I), methylene (II), and pentadienyl (III) type radicals formed under these conditions, as well as the electron adducts with *o*-naphthoquinone diazide and other carbonyl-containing impurities in the photoresist, decay in biradical reactions during irradiation. Particles identified by EPR in the photoresist implanted with P<sup>+</sup> and B<sup>+</sup> ions have a different nature.

As the absorbed dose increases during both  $\gamma$ -irradiation and ion implantation, the proportion of qui-

nonoid structures in the phenol-formaldehyde resin will increase as a result of dehydrogenation (9) and dehydration (10) of excited polymer macromolecules. The release of hydrogen, water, and other low-molecular-weight compounds occurs under conditions of deep vacuum during implantation [8]. In the case of transfer of excitation energy from phenol-formaldehyde resin macromolecules to *o*-naphthoquinone diazide, film adhesion to the substrate will increase [16] and polymer hardening will occur [15]. Carbonyl-containing moieties in the irradiated photoresist are also formed as a result of the disproportionation of radicals in reactions similar to reaction (8).

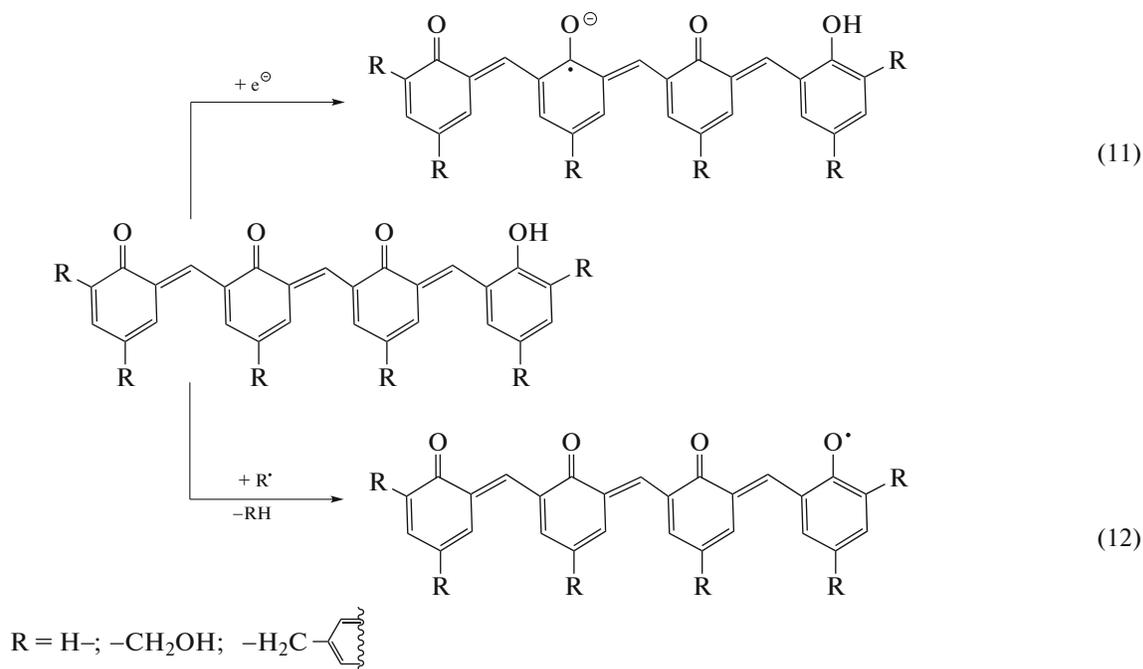


The absorbed dose during  $P^+$  ion implantation at  $6 \times 10^{15} \text{ cm}^{-2}$  in the photoresist amounts to  $\sim 1 \times 10^{12} \text{ Gy}$  in terms of the entire volume of the polymer film or  $\sim 2 \times 10^{13} \text{ Gy}$  in the ion thermalization region. This value is seven to eight orders of magnitude greater than that in the case of  $\gamma$ -irradiation and, as such, will directly affect the degree of radiation-induced conversion of the polymer. The proportion of quinonoid units formed in reactions (8)–(10), as well as the number of conjugated quinonemethine groups in them, will be much larger during ion implantation than during  $\gamma$ -irradiation.

The long-lived paramagnetic centers identified in this study in photoresist samples implanted with  $P^+$  and  $B^+$  ions at fluences of  $6 \times 10^{15}$ – $1.2 \times 10^{16} \text{ cm}^{-2}$  can be formed in two fundamentally different processes. Nevertheless, the possibility of stabilization of the formed radical by conjugation with the “extended” quinonoid fragments is an important factor in both cases. Like most other substances having conjugated  $>C=O$  and  $-C=C-$  bonds [13, 17, 18], quinonemethine groups in the irradiated photoresist will scavenge electrons, for example, via reaction (11) to give a stable radical anion. The possibility of the formation of long-lived radicals due to the oxidation of the phenolic group, for example, by reaction (12),

looks somewhat less obvious. Phenoxy radical (I) may act as an oxidant of  $R \cdot$ . In this case, radical–molecule reaction (12) is thermodynamically favorable because of significantly greater delocalization of the radical formed in the reaction in comparison with the initial radical.

The radicals formed in reactions (11) and (12) have a powerful system of conjugated double bonds and, accordingly, high delocalization of spin density. Therefore, it is possible to detect them several weeks after irradiation of the photoresist. The observed decrease in the  $g$ -factor and the increase in signal intensity in the EPR spectra with increasing ion implantation dose are associated with an increase in the number of conjugated  $>C=O$  and  $-C=C-$  bonds that are part of the stable radical, increasing its delocalization and thermodynamic stability. With a high degree of radiation-induced conversion of the phenol–formaldehyde resin, the number of conjugated multiple bonds can be so large that the unpaired electron in stable radicals will have properties close to those of the free electron. Because of this, in the photoresist bombarded with  $P^+$  and  $B^+$  ions at a fluence of  $1.2 \times 10^{16} \text{ cm}^{-2}$ , the  $g$ -factor of paramagnetic particles decreased to 2.00127–2.00254 close to the corresponding value for the free electron ( $g_s = 2.0023$ ).



Thus, the formation of long-lived radicals detectable by EPR a few weeks after implantation of the positive phenol–formaldehyde photoresist is due to the formation in a macromolecule of a powerful system of conjugated multiple bonds  $>C=O$  and  $-C=C-$ , which stabilizes paramagnetic centers.

The lower concentration of paramagnetic centers in the photoresist samples implanted with boron is due to a smaller contribution of nuclear stopping—not more than 10–15% of electronic stopping—during  $B^+$  implantation. This contribution is significantly lower than in the case of phosphorus implantation, for

which the losses due to nuclear (about 40%) and electronic (~60%) stopping are comparable. In addition, linear energy transfer—the loss of radiation energy per unit path length in the sample—will be significantly less for boron ions. Therefore, the energy of ionizing radiation during B<sup>+</sup> implantation will be distributed over a larger volume of the polymer; as a result, the degree of radiolytic transformations of phenol–formaldehyde resin will be much lower.

Note that the radiation-induced processes described above can occur outside the maximum range of ions. The presence of long molecular chains in the structure of phenol–formaldehyde resin promotes the transfer of energy from the ion range over relatively large distances (more than 1 μm) [1], especially if the excess energy is insufficient for the decay of molecules into radicals. Radiation-induced processes outside the ion implantation region (the so-called “long-range effects”) were observed previously not only for phenol–formaldehyde resin, but also for other polymers (polyethylene, PET, polyimide, polyetheretherketone) during implantation of various ions (Sb<sup>+</sup>, B<sup>+</sup>, Ni<sup>+</sup>, Ag<sup>+</sup>, Fe<sup>+</sup>) [1, 15, 19–21].

## CONCLUSIONS

The EPR spectrum of FP9120 positive photoresist films, implanted with boron and phosphorus ions at a fluence of  $6 \times 10^{15} \text{ cm}^{-2}$  and deposited on the surface of KDB-10 single-crystal silicon wafers with (111) orientation by spin coating, exhibits a narrow singlet isotropic line with a *g*-factor of 2.0064. As the implantation fluence increased to  $1.2 \times 10^{16} \text{ cm}^{-2}$ , its *g*-factor decreased to values close to the *g*-factor of the free electron ( $g_s = 2.0023$ ). The concentration of paramagnetic centers was higher during implantation of phosphorus ions than in the samples implanted with B<sup>+</sup> ions. The implantation of photoresist films also led to an increase in the Q-factor of the cavity (increase in the reference signal) as compared with unirradiated films, which is associated with the removal of residual solvent (water) from the photoresist films during irradiation in vacuum. The formation of long-lived paramagnetic centers detectable by the EPR method a few weeks after the implantation of the positive phenol–formaldehyde photoresist was supposed to be due to the presence of a powerful system of conjugated >C=O and –C=C– multiple bonds in the structure of the radicals.

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