

FRUSTRATED TOTAL INTERNAL REFLECTION SPECTRA OF DIAZOQUINONE–NOVOLAC PHOTORESIST FILMS

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Radiation-induced effects in thin films of diazoquinone–novolac photoresists on silicon irradiated with high-energy electrons (~5 MeV) were investigated by frustrated total internal reflection (FTIR) IR Fourier spectroscopy. It was found that irradiation with electrons at a dose of more than $3 \cdot 10^{15} \text{ cm}^{-2}$ leads to decrease of the integral absorption at wave numbers in the region of $3700\text{--}400 \text{ cm}^{-1}$. The intensity of bands due to the --O--H-- and particularly aliphatic --C--H-- bonds is increased most strongly during electron irradiation. At a dose of $\Phi = 1 \cdot 10^{17} \text{ cm}^{-2}$ the intensity of the bands due to the methylene ($\text{--CH}_2\text{--}$) and methyl (--CH_3) groups is comparable with the noise level. The intensity of the band at $\sim 1600 \text{ cm}^{-1}$, due to the vibrations of the aromatic ring, does not change at radiation doses in the range of $3 \cdot 10^{14}\text{--}1 \cdot 10^{17} \text{ cm}^{-2}$. The obtained experimental data indicate strong cross-linking of the polymeric components during irradiation with electrons. It was found that the radiation-induced changes in the FTIR spectra depend on the type of photoresist (FP9120 and S1813 G2 SP15). The experimental relationships of change in the optical characteristics of thin films of photoresists due to electrons are explained in terms of the radiation chemistry of diazoquinone–novolac resins.

Keywords: frustrated total internal reflection spectrum, diazoquinone–novolac photoresist, irradiation, electron, silicon.

Introduction. Polymeric composites are widely used as resists in the formation of electronic instruments in submicron and nanolithography [1–3]. Diazoquinone–novolac resists (DQN) have a dominant position among photoresists [4–6]. They consist of a mixture of phenolformaldehyde resins and light-sensitive naphthoquinone diazide usually in a ratio of 5:1 [6]. Mixtures of the acetates of alcohols and glycols are usually employed as solvents. The DNQ photoresists include the FP9120 and S1813 G2 SP15 positive photoresists, which are analogs optimized for the g line of the mercury lamp ($\lambda = 435.8 \text{ nm}$).

By IR Fourier spectroscopy with frustrated total internal reflection (FTIR) it is possible to obtain qualitative and quantitative information on the composition and structure of organic compounds and their mixtures in the solid state of aggregation [7]. We note that there have not been any previous investigations of electron-irradiated films of DQN photoresists on monocrystalline silicon.

The aim of the present work was to investigate the effect of electron irradiation on the FTIR spectra of the various types of positive diazoquinone–novolac resists and of FP9120 and S1813 G2 SP15 in particular.

Experimental. Films of the positive diazoquinone–novolac photoresists FP9120 and S1813 G2 SP15 1.8- μm -thick were deposited on the surface of silicon by centrifuging [8]. Plates of KEF-4,5 monocrystalline silicon 100 mm in diameter with (100) orientation were used as substrates. Before the film of photoresist was formed the silicon plates were submitted to a standard cycle of cleaning in organic and inorganic solvents. The thickness of the films of photoresist was monitored by a MII-4 microinterferometer at five fixed points located on two mutually perpendicular diameters on each plate. The films were irradiated by electrons with energy of 5 MeV on a U-003 linear electron accelerator at doses of $3 \cdot 10^{14}\text{--}1 \cdot 10^{17} \text{ cm}^{-2}$. The density of the electron beam $1 \cdot 10^{12} \text{ cm}^{-2} \cdot \text{s}^{-1}$ was monitored by means of a Faraday cylinder. The temperature of the samples in the process was not higher than 310 K.

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The FTIR spectra of the structures of the silicon photoresist in the region of 400–4000 cm^{-1} were recorded at room temperature on an ALPHA IR Fourier spectrophotometer (Bruker Optik GmbH). The resolution was 2 cm^{-1} , and the number of scans was 24. Background correction was applied before each experiment [9]. The photoresist films were not kept for more than two months before the FTIR spectra were recorded.

Results and Discussion. The FTIR spectra of the initial (unirradiated) FP9120 and S1813 G2 SP15 films are similar (Fig. 1) although they do have a number of differences. Thus, there is no band with a maximum at $\sim 2960 \text{ cm}^{-1}$ (Fig. 2a, curve 1) due to the asymmetric stretching vibrations of the methyl $-\text{CH}_3$ group in the region of the stretching vibrations of the $-\text{C}-\text{H}$ bonds (the region of 2700–3100 cm^{-1}) [10, 11]. For the same photoresist the bands due to the skeletal vibrations of the aromatic ring (a band with a maximum at $\sim 1600 \text{ cm}^{-1}$ [12]) and the stretching vibrations of the $\text{ArC}-\text{C}$ bond (a band with a maximum at $\sim 1500 \text{ cm}^{-1}$ [10]) have lower intensity (Fig. 2b). The reason for these special features of the FTIR spectra of the S1813 G2 SP15 photoresist is the fact that xylene isomers (dimethylbenzene) are not used as solvents in the preparation of the photoresist. In addition, the absence of the band due to the asymmetric stretching vibrations of the $-\text{CH}_3$ methyl group in the spectrum of S1813 G2 SP15 indicates the absence of cresol (methylphenol) residues in this type of photoresist. Another feature of the S1813 G2 SP15 photoresist is the absence of residual formaldehyde. This is demonstrated by the extremely low (at the noise level) intensity of the band with a maximum at $\sim 1650 \text{ cm}^{-1}$ (Fig. 2b, curve 1) due to the $\text{C}=\text{O}$ vibrations in formaldehyde [12].

Differences in the two types of photoresists are also seen in the region of 1150–1240 cm^{-1} , where the planar deformation vibrations of the $\text{C}-\text{O}$ bonds of phenols are observed (Fig. 2b). The structure of the absorption band of these vibrations in the S1813 G2 SP15 photoresist differs substantially from the structure of the analogous band in FP9120. Thus, a band with one maximum at $\sim 1170 \text{ cm}^{-1}$ is observed for S1813 G2 SP15, and a band with three maxima of approximately equal intensity at 1150, 1170, and 1198 cm^{-1} is observed for FP9120. In addition, a band with a maximum at $\sim 1375 \text{ cm}^{-1}$ (curve 1), corresponding to the deformation planar vibrations of $\text{O}-\text{H}$ bonds in the phenyl group, was observed in the absorption spectra of the S1813 G2 SP15 photoresist [13]. At the same time this band is absent in the spectra of the FP9120 samples, but a strong band with a maximum at $\sim 1360 \text{ cm}^{-1}$, assigned to the $\text{C}-\text{O}-\text{C}$ vibrations in furan, appears [12]. A band at 1100 cm^{-1} , the intensity of which is substantially higher for the photoresist FP9120 than for S1813 G2 SP15, also belongs to the $\text{C}-\text{O}-\text{C}$ deformation vibrations (Fig. 2b). In addition, the spectrum of the photoresist S1813 G2 SP15 also contains a band at 1040 cm^{-1} usually associated with vibrations of the $\text{C}-\text{O}$ bond in the $\text{CH}_2-\text{C}-\text{OH}$ fragment [12]. The experimental data show that the intensity of the bands from the phenol group is higher for the S1813 G2 SP15 photoresist.

The observed features of the S1813 G2 SP15 photoresist are most likely caused by the following reasons. The formation of intra- and intermolecular hydrogen bonds through the phenolic hydroxyl group is observed in phenolformaldehyde resins [3]. The presence of such bonds leads to a shift of the frequency of the vibrations of the $\text{C}-\text{O}$ in the phenol group and to the appearance of several bands in the region of 1150–1230 cm^{-1} . Cross-linking of the resin molecules through the oxygen atom of the phenolic group with the formation of $\text{C}-\text{O}-\text{C}$ fragments can be observed under certain conditions during the synthesis of the polymeric composite of the photoresist. They occur in the FP9120 photoresist and are poorly defined in S1813 G2 SP15.

Irradiation with electrons leads to decrease of the integral (background) absorption in both photoresists (Fig. 1). We note that the radiation-induced changes in the FTIR spectra are more pronounced in FP9120 than in S1813 G2 SP15. The decrease of the background absorption is stronger in FP9120 (Fig. 1). The changes in the intensity of the FTIR bands in FP9120 were observed even at dose $\Phi \sim 3 \cdot 10^{15} \text{ cm}^{-2}$ (Fig 3), whereas they are only observed at $\Phi \geq 3 \cdot 10^{16} \text{ cm}^{-2}$ in S1813 G2 SP15.

The intensity of the bands from vibrations of $\text{O}-\text{H}$ and particularly $\text{C}-\text{H}$ bonds decrease most strongly during electron irradiation (Fig. 2a). With a dose of $\Phi = 1 \cdot 10^{17} \text{ cm}^{-2}$ the bands due to methylene ($-\text{CH}_2-$) and methyl ($-\text{CH}_3$) groups (2750–3100 cm^{-1}) in Fig. 2a are not visible in the spectra of FP9120, while in S1813 G2 SP15 their intensity is at the noise level (curve 3). The intensity of the band in the region of 3100–3700 cm^{-1} , due to the stretching vibrations of the $\text{O}-\text{H}$ bonds, decreases substantially (by 3–5 times) during irradiation (curves 3 and 4). It should be noted that during irradiation there is a strong decrease in the intensity of the band at 1375 cm^{-1} caused by in-plane deformation vibrations of the $\text{O}-\text{H}$ bonds in the $\text{C}-\text{O}-\text{H}$ phenol fragment and mostly observed for the S1813 G2 SP15 photoresist (Fig. 4). This band disappears almost completely in the FTIR spectrum of S1813 G2 SP15 at $\Phi \geq 3 \cdot 10^{16} \text{ cm}^{-2}$. This also affects the bands in the region of 630–900 cm^{-1} corresponding to out-of-plane vibrations of the $\text{C}-\text{H}$ bonds in aromatic compounds [13]. They are not observed in the FTIR spectra at $\Phi > 1 \cdot 10^{16} \text{ cm}^{-2}$ (Fig. 4).

Absorption bands not associated with hydrogen are more resistant to electron irradiation. This applies particularly to the band at $\sim 1600 \text{ cm}^{-1}$, corresponding to skeletal vibrations of the aromatic ring [10]. Its intensity decreases weakly even at

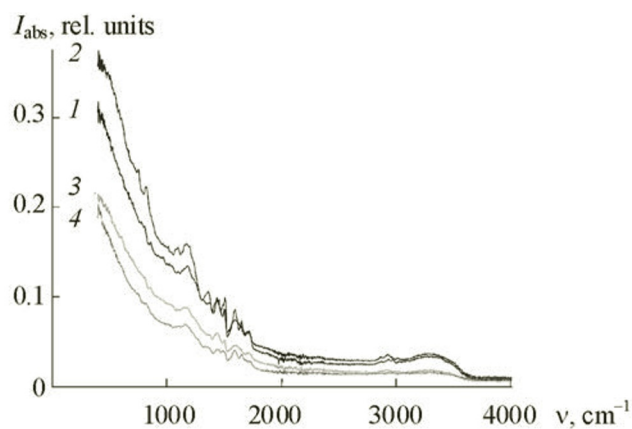


Fig. 1. The FTIR spectra of films of the initial (1, 2) S1813 G2 SP15 (1, 3) and FP9120 (2, 4) diazoquinone–novolac photoresists and of those irradiated with electrons (3, 4) at a dose of $1 \cdot 10^{17} \text{ cm}^{-1}$.

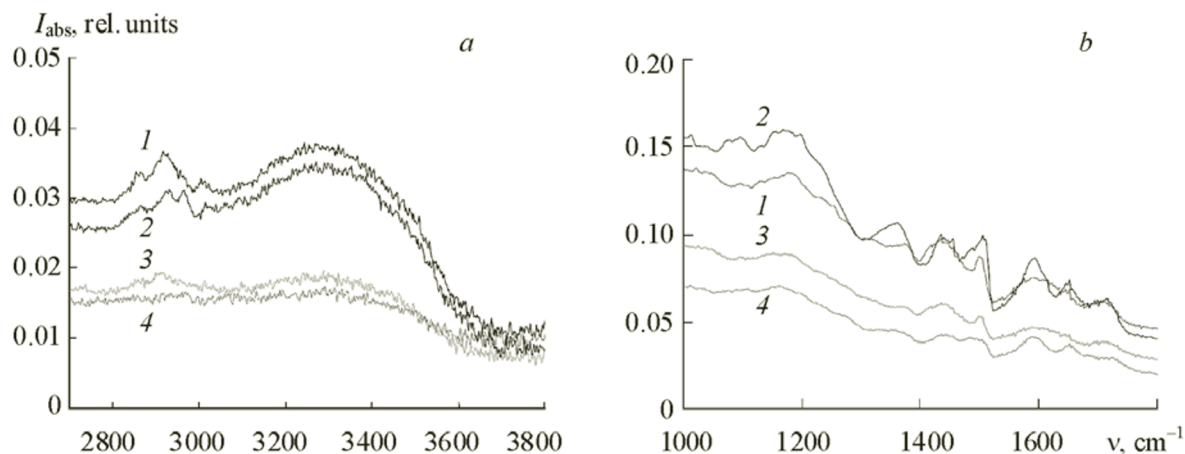


Fig. 2. The FTIR spectra of the initial films (1, 2) of S1813 G2 SP15 (1, 3) and FP9120 (2, 4) diazoquinone–novolac photoresists and of those irradiated with electrons (3, 4) at a dose of $5 \cdot 10^{16} \text{ cm}^{-1}$ in the region of the stretching vibrations of the C–H and O–H bonds (a) and C=O and C–O bonds (b).

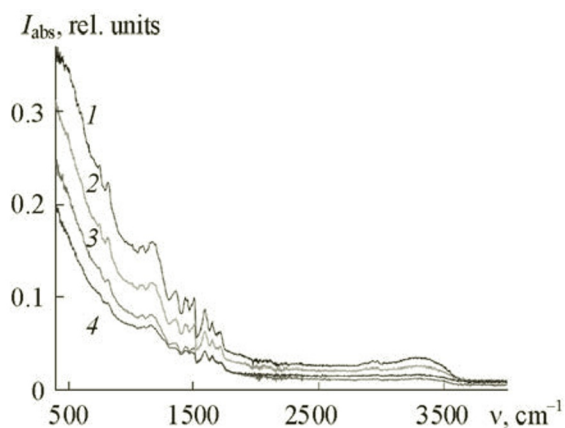


Fig. 3. The FTIR spectra of films of FP9120 photoresist irradiated with electrons at doses of 0 (1), $3 \cdot 10^{15}$ (2), $3 \cdot 10^{16}$ (3), and 10^{17} cm^{-1} (4).

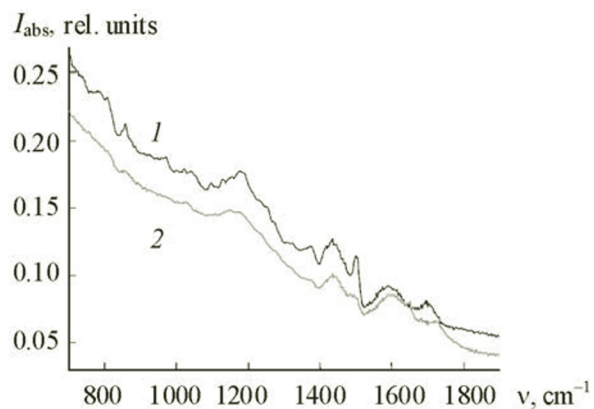


Fig. 4. The FTIR spectra of the initial film of S1813 G2 SP15 photoresist (1) and of the film irradiated with electrons at a dose of $1 \cdot 10^{17} \text{ cm}^{-1}$ (2).

a dose of $1 \cdot 10^{17} \text{ cm}^{-2}$ (Fig. 4). The intensity of the band with a maximum at $\sim 1430 \text{ cm}^{-1}$, due to vibrations of a benzene ring connected to a CH_2 bridge, decreases quite strongly [11, 13]. The reason for this is the superimposition of a weak band for the deformation vibrations of the C–H bonds in formaldehyde [12].

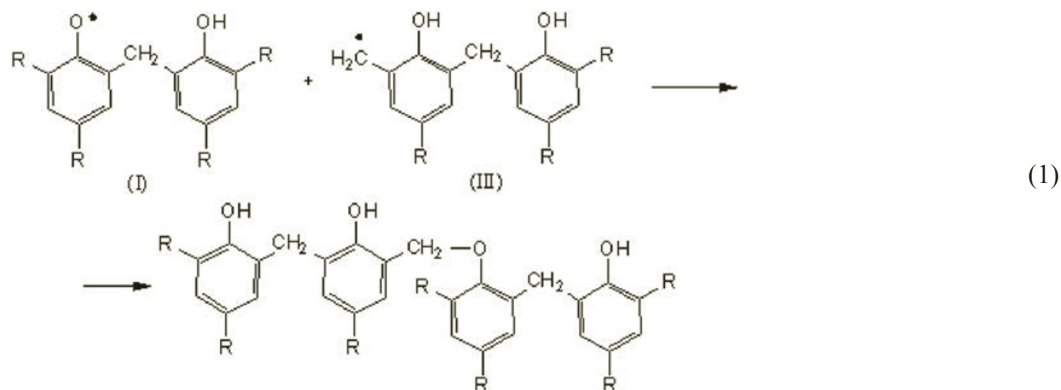
In the region of the stretching vibrations of the multiple bonds the intensity of the band at $\sim 1700 \text{ cm}^{-1}$ decreases during irradiation with simultaneous broadening and a shift of $\sim 30 \text{ cm}^{-1}$ into the high-energy region of the spectrum. As known [14, 15], the band at $\sim 1700 \text{ cm}^{-1}$ corresponds to the stretching vibrations of the C=O bonds in aromatic compounds. With change of the nearest environment the position of the maximum of this band can be shifted both into the low-energy and into the high-energy regions. Thus, during conjugation with C=C bonds the maximum of the band for these vibrations is shifted into the low-energy region [10]. According to [16], structures for which the maximum of the absorption band must be in the region of $1645\text{--}1690 \text{ cm}^{-1}$ [10], depending on the number of conjugated bonds, are formed during implantation of the FP9120 photoresist. In addition, in aromatic esters the maximum of the band for the stretching vibrations of the C=O bonds is shifted into the high-energy region at $1715\text{--}1736 \text{ cm}^{-1}$ [10]. The formation of aromatic esters during irradiation with electrons is probably the main reason for shift of the maximum of this band. Change of the nearest environment of the C=O bonds arising as a result of irradiation leads to broadening of the band at $\sim 1700 \text{ cm}^{-1}$ as a result of the multifaceted action of the above-mentioned factors. The band with a maximum at $\sim 1650 \text{ cm}^{-1}$, observed for the FP9120 photoresist and due to the stretching vibrations of the C=O bonds in formaldehyde $\text{CH}_2=\text{O}$, also remains stable during irradiation with electrons (Fig. 2b).

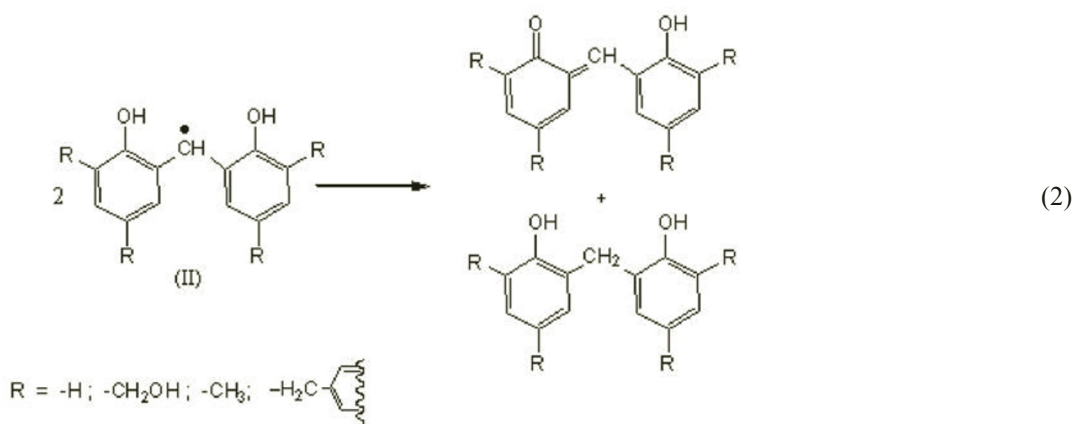
The broad structured absorption band with several maxima in the region of $1100\text{--}1230 \text{ cm}^{-1}$ after irradiation of the photoresist becomes smooth, maxima do not appear, and its intensity decreases (Fig. 4). This band is due, in particular, to the stretching vibrations of the C–O bonds in the phenoxy group (C–OH) [9]. Such behavior of the FTIR bands in the region of $1050\text{--}1230 \text{ cm}^{-1}$ may indicate the formation of ester linkages with OH groups of phenolic type. Such cross linking was observed earlier during implantation of Sb^+ in films of photoresist FP9120, leading to increase of their microhardness [17]. Earlier [18] hardening of FP9120 films was observed during high-energy irradiation with γ quanta. A similar effect can also occur in the oxidation of phenols to quinone methines, as a result of which the C–OH group is converted into C=O.

The intensity of the band with a maximum at $\sim 1500 \text{ cm}^{-1}$, due to the stretching vibrations of aromatic C–C bonds [10], decreases appreciably during irradiation with electrons at doses of $>1 \cdot 10^{16} \text{ cm}^{-2}$, and the decrease is greater in S1813 G2 SP15 (Figs. 2b and 4). This may be due to splitting of substituents from the aromatic ring or to their significant modification during irradiation of the polymer with electrons.

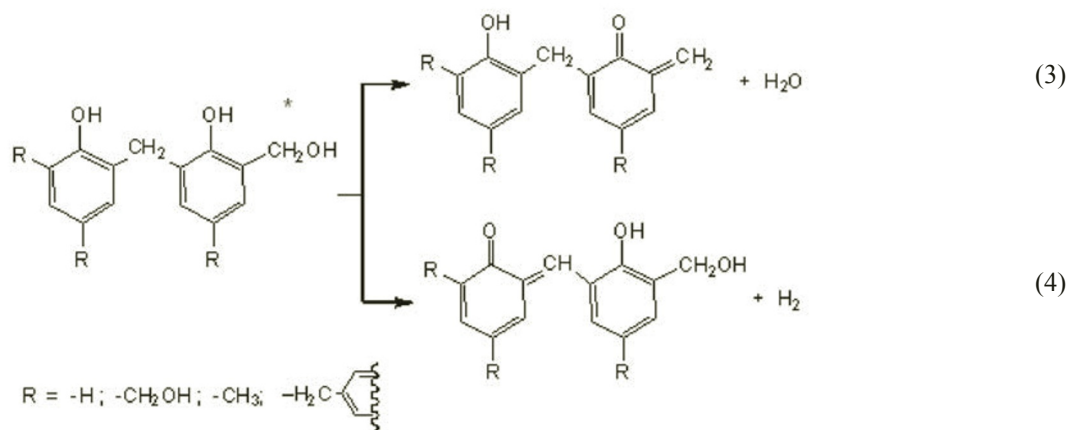
The changes in films of photoresists discovered by FTIR IR spectrometry are caused by the following set of radiation–chemical processes. Absorption of the energy of electron irradiation occurs uniformly throughout the photoresist film unlike ion implantation, where thermalization of ions is observed exclusively in a surface layer 60–150 nm deep [8, 9, 11, 16]. Ionization and electron excitation of the macromolecules occur as a result of interaction of the high-energy electrons with the phenolformaldehyde resin. Subsequent decay of the excited particles and ion–molecular processes will lead to the formation of predominantly oxygen-centered radicals of the phenoxy type (I) and also carbon-centered radicals of the methylene (II) and benzyl (III) types [9]. The latter radicals are only formed when there are arylmethyl fragments in the polymer as, for example, in the FP9120 photoresist.

Because of delocalization in the aromatic system the radicals (I–III) exhibit thermodynamic stability and are mostly consumed in recombination reactions that lead to the formation of new C–C or C–O bonds (in reaction 1, for example) between the polymer chains. With somewhat lower probability the radicals (I–III) will enter into disproportionation reaction (2), as a result of which the quinonemethine fragments are formed:

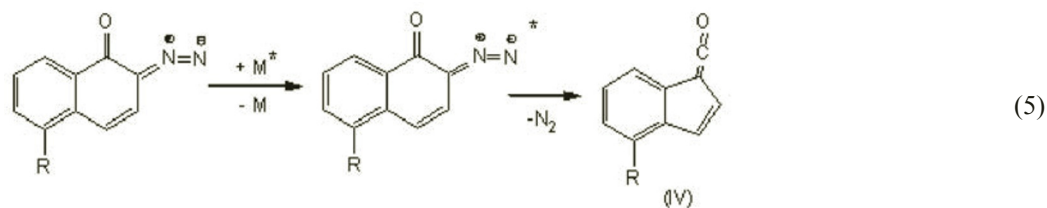




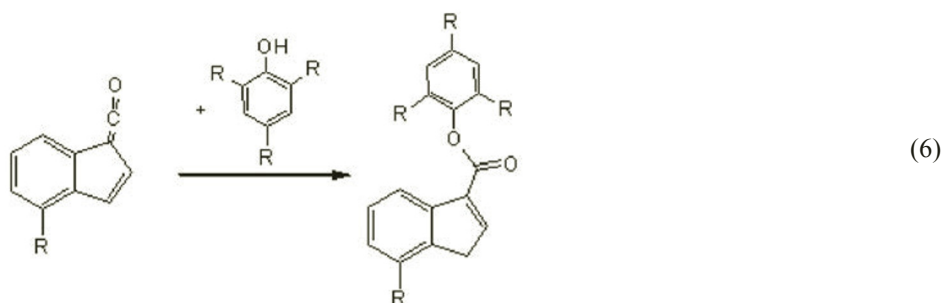
Special features of the radiation chemistry of aromatic compounds are high radiation-chemical yields of particles in an electron-excited state and, as a consequence, their important role in the formation of the final molecular products of radiolysis. The accumulation of quinonemethine structures during irradiation of phenolformaldehyde resins can then also occur in molecular processes involving the electron-excited states of macromolecules and, in particular, as a result of dehydration (3) and dehydrogenation (4) reactions:



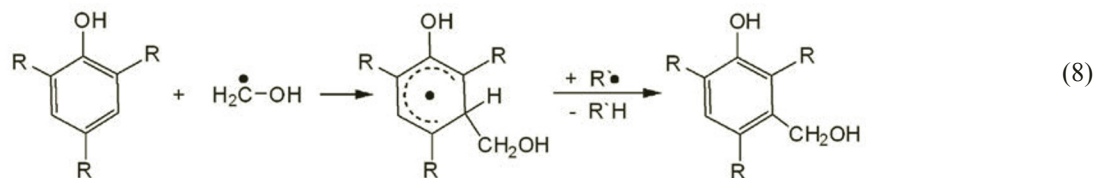
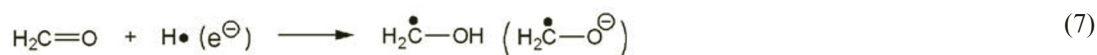
Thus, the decrease in the intensity of the IR absorption bands in the region of $\sim 2800\text{--}3000\text{ cm}^{-1}$, corresponding to the vibrations of C-H bonds in methylene ($-\text{CH}_2-$) and methyl ($-\text{CH}_3$) groups, during irradiation of DQN photoresists arises as a result of the realization of biradical recombination (1) and disproportionation (2) reactions and also of the molecular processes of decay of the particles (3) and (4). Realization of reactions (2)–(4) also leads to the formation of quinonemethine structures, which shows up as decrease of the signal from phenolic $-\text{OH}$ groups in the region of $\sim 3100\text{--}3600$ and also as increase of the intensity of absorption for wave numbers characteristic of $>\text{C}=\text{O}$ groups conjugated with multiple carbon-carbon bonds. It is also necessary to note that the decrease in the amount of phenolic OH groups in the DQN photoresist during electron irradiation can also arise as a result of reactions involving the photosensitive component of the composite. Transfer of electron excitation from the macromolecules of phenolformaldehyde resin to the *ortho*-naphthoquinone diazide leads to its deazotization [9] by reaction (5) with the formation of the highly reactive ketene (IV):



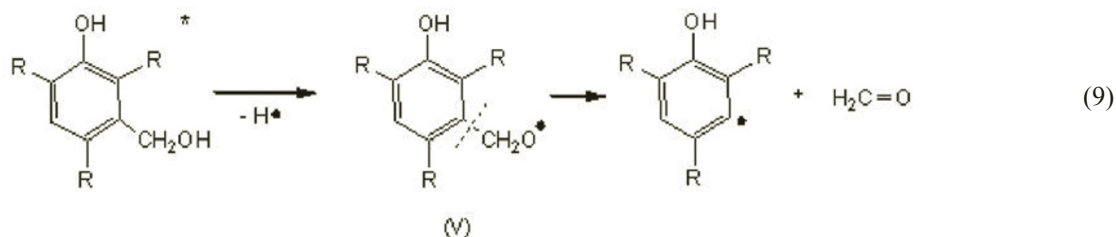
This compound is capable not only of adding water present in the DQN photoresist with the formation of indene-3-carboxylic acid but also of converting the phenols into esters according to reaction (6):



We will now consider the band found for the photoresist FP9120 with a maximum at $\sim 1650\text{ cm}^{-1}$ due to the stretching vibrations of the C=O bonds in formaldehyde $\text{CH}_2=\text{O}$. Together with phenol the $\text{CH}_2=\text{O}$ is used in the synthesis of the phenolformaldehyde resin and could remain entirely in the polymer as a chemical impurity. However, formaldehyde readily accepts hydrogen atoms and electrons in reaction (7) and like other low-molecular aldehydes can add carbon-centered radicals [19]. The $\text{CH}_2=\text{O}$ must therefore quickly disappear from the phenolformaldehyde resin under the conditions of irradiation with electrons, being converted predominantly into new hydroxymethyl groups according, for example, to reaction (8).



The fact that the concentration of formaldehyde changes little from the absorbed dose indicates the realization of processes that lead to its formation in the course of irradiation of the FP9120 photoresist film with electrons. The most likely mechanism of elimination of formaldehyde may be β -fragmentation of the oxygen-centered radical (V) in reaction (9):



Simultaneous realization of reactions leading to consumption (7, 8) and formation (9) of $\text{CH}_2=\text{O}$ leads to the appearance of constancy in its concentration in the FP9120 photoresist film under the conditions of irradiation.

Thus, the lower radiation stability of the FP9120 photoresist compared with S1813 G2 SP15 is due to the presence of methyl groups in the composition of the polymer base of FP9120 and also to the larger content of hydroxymethyl fragments, and this must be taken into account during ion implantation of monocrystalline silicon using photoresist masks.

Conclusions. Analysis of the frustrated total internal reflection spectra indicates that DQN photoresists FP9120 and S1813 G2 SP15 are almost identical in composition. The lower content of cresol fragments and residual formaldehyde must be included among the special features of the S1813 G2 SP15 photoresist. A decrease of integral absorption in the region of $3700\text{--}400\text{ cm}^{-1}$ is observed when the diazoquinone–novolac photoresists S1813 G2 SP15 and FP9120 are irradiated with electrons at doses of $>3 \cdot 10^{15}\text{ cm}^{-2}$. The radiation-induced changes in the FTIR spectra are more pronounced in the FP9120 photoresist compared with S1813 G2 SP15. The intensity of the bands due to vibrations of the hydroxyl ($-\text{O}-\text{H}$), methyl ($-\text{CH}_3$), and methylene ($-\text{CH}_2-$) groups are reduced most strongly during irradiation. The intensity of the band at $\sim 1600\text{ cm}^{-1}$, due to the stretching vibrations of the aromatic ring, remains practically unchanged right up to the highest electron dose $\Phi = 1 \cdot 10^{17}\text{ cm}^{-1}$. The formation of cross links between the macromolecules of the phenolformaldehyde resin with the formation of new C–O–C fragments was observed in the FP9120 photoresist. It was shown that the concentration of formaldehyde in the

FP9120 photoresist as a result of superimposition of the formation and consumption processes does not change with increase of the absorbed dose. An explanation for the obtained experimental relationships with regard to the characteristics of the radiation chemistry of diazoquinone–novolac resins is presented.

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