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Radiation-Induced Processes in Electron-Irradiated Films of Negative Phenol-Formaldehyde Photoresist on Silicon

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Abstract—It was found that radiation-induced processes in films of negative photoresist *NFR* 016*D* at doses up to 2×10^{15} cm^{-2} occur mainly with the participation of residual solvent molecules or on by-products of the photoresist film synthesis. After irradiation, absorption bands with maxima at 1717 (stretching vibrations of C=O bonds), 1068, and 1009 cm^{-1} (vibrations of C—O—C bond in methyl-3-methoxypropylate solvent) disappear in the attenuated total internal reflection spectra of the photoresist. At irradiation doses above 1×10^{16} cm^{-2}, significant changes in the intensity of the bands associated with the main component of the photoresist (phenol-formaldehyde resin) were observed. A noticeable transformation of the spectrum occurs in the region of 1550–1700 cm^{-1}, in which stretching vibrations of C=O bonds are observed. At irradiation doses above 2×10^{16} cm^{-2}, the intensities of the bands caused by stretching vibrations of CH_2 and CH groups decrease (bands with maxima at 2925 and 3012 cm^{-1}, respectively). The experimental results indicate a change in the composition of substituents at the carbon ring and the formation of conjugated double C=O bonds when the photoresistive film is irradiated with electron doses above 2×10^{16} cm^{-2}.

Keywords: negative phenol-formaldehyde photoresist, silicon, electron irradiation, attenuated total internal reflection

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INTRODUCTION

In modern microelectronics, the *lift-off* lithography method, in which a film of material (usually metal) is applied to an already exposed and etched photoresist layer (PR), is becoming increasingly widespread [1]. The structure on the substrate is formed by removing unexposed PR sections together with the metal film located on them. One of the main factors in the successful application of lift-off lithography is the formation of a negative slope angle of the photoresist mask (the overhanging profile of the sidewalls). Metal is not deposited on such sidewalls of the profile, which facilitates the process of removing the PR layer and ensures the smoothness of the edge of the formed element.

When a negative PR is exposed, polycondensation reactions occur and the polymer molecules become "cross-linked," as a result, the film becomes insoluble in the developer [2]. When it is exposed to electromagnetic radiation, the upper layers of the PR film receive a greater amount of energy than the lower ones. As a result, the lower layers of the film dissolve better in the developer, which contributes to the formation of a negative tilt angle of the PR mask. Novolac resins soluble in a water—alkaline developer are usually used as a film-forming polymer of the PR for reverse lithography. Negative photoresists of the NFR series (JSR, Japan) are such PRs.

The need to increase the resolution of the lithographic process dictates, in accordance with the Rayleigh criterion, the transition from UV radiation to shorter-wave radiation: X-ray or electron [2]. It was shown earlier in [3, 4] that electron irradiation can significantly change the spectral and adhesion characteristics of positive photoresist films on silicon. However, the effect of electron irradiation on the properties of the negative photoresist *NFR* 016*D*4 has not been studied to date. The method of IR Fourier spectroscopy of attenuated total internal reflection (ATR) allows obtaining qualitative and quantitative information on the composition and structure of complex

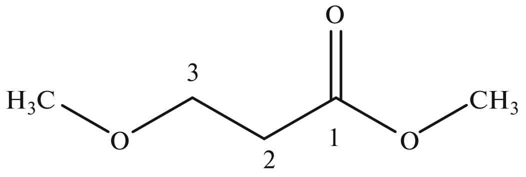

Fig. 1. Structural formula of methyl-3-methoxypropionate.

organic compounds and their mixtures in the solid aggregate state [5].

The aim of the present work is to study radiation-induced processes in *NFR* 016*D*4 photoresist films on silicon under irradiation with high-energy electrons.

EXPERIMENTAL METHODS

Films of negative phenol-formaldehyde photoresist *NFR* 016*D*4 with a thickness of 3.5 and 5.2 μm were applied to the (111) surface of the *p*-type with resistivity of 10 Ohm cm brand single-crystalline silicon by the centrifugation method [6]. Methyl-3-methoxypropionate was used as a solvent (Fig. 1).

Before applying the PR, the silicon wafers were subjected to a standard surface cleaning cycle in organic and inorganic solvents. After the PR film was formed, it was dried at 90°C for 120 s.

Irradiation with 5 MeV-energy electrons with a dose of up to 7×10^{16} cm^{-2} was carried out on a U-003 linear electron accelerator. The electron flux density was 1×10^{12} cm^{-2} s^{-1} and was controlled using a Faraday cup. The temperature of the samples during irradiation did not exceed 310 K.

The ATR spectra of the photoresist/silicon structures in the range of 400–4000 cm^{-1} were recorded at room temperature using an *ALPHA* FTIR spectrophotometer (*Bruker Optik GmbH*). The resolution was 2 cm^{-1}, and the number of scans was 24. Background correction was performed before each measurement [7].

RESULTS AND DISCUSSION

The ATR spectra of the *NFR* 016*D*4 photoresist films on single-crystalline silicon wafers are shown in Fig. 2. The experimentally observed frequencies of the IR absorption bands of PR and their assignment to the corresponding functional groups are listed in Table 1. Their behavior during irradiation is also noted. It should be emphasized that, at initial doses of $\Phi \leq 2 \times 10^{15}$ cm^{-2}, the ATR spectra of the initial and irradiated films with a thickness of 3.5 μm were qualitatively similar (Fig. 2). Only minor changes in the intensity ratios of some closely located bands were observed. Thus, there was an increase in the intensity of the bands at 862, 958, and 1100 cm^{-1} in comparison with the bands at 895, 1009, and 1068 cm^{-1}, respectively.

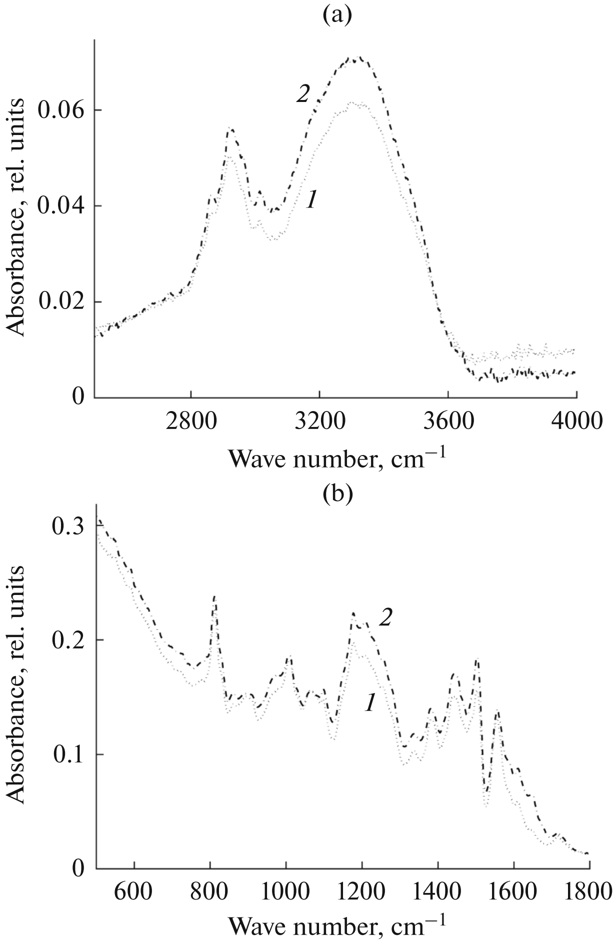

Fig. 2. ATR spectra of the (*1*) initial and (*2*) irradiated with 5 MeV-electrons at the dose of 2×10^{15} cm^{-2} PR films with a thickness of 3.5 μm in the region of (a) OH bonds and (b) stretching vibrations of single (C–C and C–O) and double (C=C and C=O) bonds.

In thick films (5.2 μm), radiation-induced changes in the ATR spectra were significantly more pronounced than in thin films (see Figs. 2b, 3). The most ambiguous transformations of the ATR spectrum upon thick films' irradiation were observed in the range of wavenumbers ν = 1000–1100 cm^{-1}, characteristic of the C–O bonds vibrations. The intensity of all bands in this range decreased upon irradiation. At a dose of $\Phi = 1 \times 10^{15}$ cm^{-2}, a new band appeared in the spectrum at 1043 cm^{-1}, and the intensity of the bands at 1009 and 1068 cm^{-1} sharply decreased. In this case, the maximum of the band at 1068 cm^{-1} shifted to the high-energy region by ~6 cm^{-1} (to 1074 cm^{-1}). A further increase in the dose to 2×10^{15} cm^{-2} led to the restoration of the spectrum structure in this region: the band at 1043 cm^{-1} disappeared from the spectrum, and the maximum of the high-energy band returned to 1068 cm^{-1} (Fig. 3, curves *2* and *3*). Similar behavior was observed for the band with a maximum at 895 cm^{-1}:

Table 1. Experimental values of IR absorption frequencies of functional groups in negative photoresist *NFR* 016*D*4

Energy position	Intensity	Functional group, vibration type	Behavior under irradiation
774 cm^{-1}	Weak	Out-of-plane vibrations of C—H bonds of ortho- and para-substituted rings [8]	
812 cm^{-1}	Strong		Intensity decreasing by three times at $\Phi = 7 \times 10^{16}$ cm^{-2}
~790 cm^{-1}	Weak		Appears at $\Phi = 1 \times 10^{16}$ cm^{-2}
862 cm^{-1}	Weak	No data	Intensity decreasing at $\Phi > 1 \times 10^{16}$ cm^{-2}
895 cm^{-1}	Medium	No data	Disappears at $\Phi = 7 \times 10^{16}$ cm^{-2}
958 cm^{-1}	Weak	C—O—C bonds [9, 10]	No changes recorded
1009 cm^{-1}	Strong	C—C bonds vibrations [3]	Intensity decreasing by two times at $\Phi = 7 \times 10^{16}$ cm^{-2}
1064 cm^{-1}	Medium	C—O—C bonds in methyl-3-methoxypropylate [9, 10]	At $\Phi = 7 \times 10^{16}$ cm^{-2} is nonobservable
1100 cm^{-1}	Weak	Stretching vibrations of Si—O bonds in silicon [7]	Stable
1176 cm^{-1}	Strong	C—O stretching vibrations [7]	Intensity redistribution. 1204 cm^{-1} dominates at $\Phi = 7 \times 10^{16}$ cm^{-2}
1204 cm^{-1}			
1256 cm^{-1}			
1338 cm^{-1}	Weak	No data	Disappears at $\Phi = 7 \times 10^{16}$ cm^{-2}
1381 cm^{-1}	Medium	OH bonds in-plane bending vibrations [9]	Intensity decreasing and shift towards 1375 cm^{-1}
1436 cm^{-1}	Medium	Stretching vibrations of the aromatic ring associated with the CH_2 bridge [9]	Broadening and shift to low-energy region
1503 cm^{-1}	Strong	Stretching vibrations of the aromatic ring [9]	No changes
1556 cm^{-1}	Strong	Pulsation vibrations of the aromatic ring carbon skeleton [10]	Structural change at $\Phi > 5 \times 10^{15}$ cm^{-2}. 1590, 1650 cm^{-1} arise and grow. 1607 cm^{-1} dominates at $\Phi = 7 \times 10^{16}$ cm^{-2}
1607 cm^{-1}	Weak		
1717 cm^{-1}	Weak	C=O stretching vibrations in aliphatic esters [10]	Disappears
~2875 cm^{-1}	Weak	Symmetric stretching vibrations of CH_2 groups [10]	No changes found
2925 cm^{-1}	Medium	Asymmetric stretching vibrations of CH_2 groups [10]	Intensity decreasing
3012 cm^{-1}	Weak	Stretching vibrations of CH groups [10]	Disappears at $\Phi = 7 \times 10^{16}$ cm^{-2}
3290 cm^{-1}	Medium	O—H stretching vibrations, hydrogen bonding [10]	Shift towards low energy region

its intensity sharply decreased, and the maximum shifted to ~875 cm^{-1} at a dose of 1×10^{15} cm^{-2}.

Increasing the dose to 2×10^{15} cm^{-2} led to a further decrease in its intensity and the maximum return at 895 cm^{-1}. The band with a maximum at 1043 cm^{-1} is usually associated with vibrations of the C—O bond in the structural fragment —CH_2—C—OH [10]. The bands at 1009 and 1068 cm^{-1} are due to vibrations of the C—O—C bond in the solvent, methyl-3-methoxypropylate [9, 10]. Note that, in thin films, the intensity

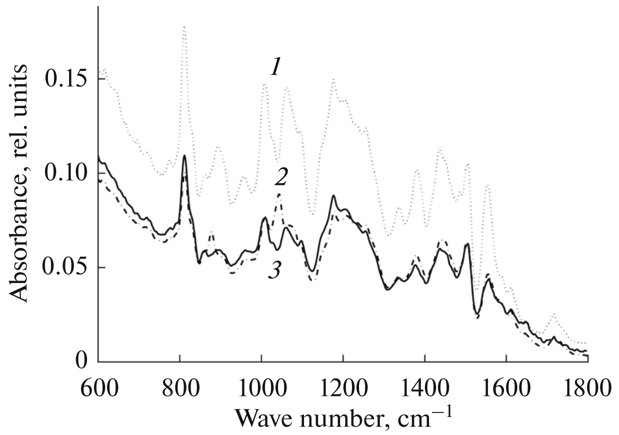

Fig. 3. ATR spectra of the (*1*) initial and irradiated with 5 MeV electrons at doses of (*2*) 1 × 10^{15} cm^{-2} and (*3*) 2 × 10^{15} cm^{-2} PR films with a thickness of 5.2 μm.

of these bands was significantly lower, and it was not possible to track their transformation during irradiation.

The presented data indicate an intensive decomposition of residual solvent molecules in the polymer film volume during electron irradiation. This process becomes noticeable even at initial (5 × 10^{14}–1 × 10^{15} cm^{-2}) doses. Similar transformations of the ATR spectra of thick *NFR* 016*D*4 films were also observed during their drying in a vacuum oven at 70°C for up to 4 h.

The increase in the intensity of the broad band with a maximum at ~3290 cm^{-1}, caused by O–H bonds vibrations (Fig. 2a), during irradiation is most likely associated with the solvent decomposition and the formation of products of its radiolysis. In addition, the band with a maximum at 1717 cm^{-1}, caused by stretching vibrations of C=O bonds in the solvent, practically disappears from the spectrum during drying and irradiation with a dose of 2 × 10^{15} cm^{-2} (Fig. 3). Note that there are no OH groups in the solvent (Fig. 1), but some products of its radiolysis, for example, methanol (CH_3OH), do have such a fragment, which leads to an increase in the intensity of the bands caused by OH bonds. C=O bonds should break when esters are exposed to radiation, forming gaseous products CO_2 and CO.

It should be noted that the residual solvent content in thick films was significantly higher than in thin films. This is explained by the fact that the drying time and temperature of both films were identical, which is why the efficiency of solvent removal was lower in thicker films. The presence of residual solvent in these films is confirmed by the medium-intensity band presence in the ATR spectrum, with a maximum at 1717 cm^{-1} due to stretching vibrations of C=O bonds in the solvent, and its intensity was higher in thick films than in thin films. This band practically disappears from the spectrum upon drying and irradiation with a dose of 2 × 10^{15} cm^{-2} (Fig. 3).

Based on the above experimental data, it can be concluded that radiation-induced processes in *NFR* 016*D*4 films at doses up to 2 × 10^{15} cm^{-2} occur mainly with the participation of residual solvent molecules. Usually, aromatic structures in complex multicomponent mixtures "protect" other compounds under radiolysis conditions from radiation-induced decomposition. In our case, this is not observed. It is possible that a process of the solvent's selective damage occurs under the action of the polymeric matrix, for example, by transferring excitation energy from the aromatic polymer to the solvent. It is possible that hydroxyl-containing compounds, in particular, methyl alcohol and methyl ester of 3-hydroxypropionic acid, are formed as intermediate compounds during solvent radiolysis. Then, under the influence of electron irradiation, the alcohol groups are either converted into the aldehyde ones R–HC=O as a result of the radicals disproportionation, as well as their oxidation by atmospheric oxygen, or they are added to aromatic compounds to form adducts of the R–HC(OH)-Ar type.

It should be noted that the predominant occurrence of radiation-induced processes on the residual solvent was observed previously [11] during a study of the effect of γ-irradiation with a dose of up to 1 MGy on polyimide-based structures.

Increasing the irradiation dose of *NFR* 016*D*4 films from 1 × 10^{15} to 1 × 10^{16} cm^{-2} did not result in noticeable changes in the ATR spectrum. Only a decrease in the intensity of the bands caused by the solvent was noted (in particular, the bands with maxima at 895, 1064, and 1717 cm^{-1} disappeared from the spectrum). Significant changes in intensities of the bands associated with the photoresist main component (phenol-formaldehyde resin) were observed only at doses above 1 × 10^{16} cm^{-2} (Fig. 4). Thus, there was a decrease in the intensities of the bands caused by the stretching vibrations of CH_2 and CH groups (bands with maxima at 2925 and 3012 cm^{-1}, respectively). Also, the intensity of the band with a maximum at 812 cm^{-1} caused by out-of-plane vibrations of the aromatic ring C–H bonds was decreased significantly (by three times, at a dose of $\Phi = 7 \times 10^{16}$ cm^{-2}).

The bands associated with the OH bonds' vibrations (1381 and ~ 3290 cm^{-1}) at doses above 1 × 10^{16} cm^{-2} showed a shift to the low-energy region, which can be explained by strengthening of hydrogen bonds. Note that intensity of the 1381 cm^{-1} narrow band due to in-plane deformation vibrations of OH bonds decreases and becomes barely noticeable at a dose of $\Phi = 7 \times 10^{16}$ cm^{-2}.

The intensity of the 1009 cm^{-1} band, caused by vibrations of the aliphatic C–C bonds, decreased by two times at an irradiation dose of $\Phi = 7 \times 10^{16}$ cm^{-2}. A redistribution of intensities in favor of higher-energy local maxima is observed in the region of the C–O bonds' stretching vibrations (a broad structured band in the region of 1150–1270 cm^{-1} with the main maximum at 1176 cm^{-1}). At $\Phi = 7 \times 10^{16}$ cm^{-2}, the maximum at 1204 cm^{-1} becomes dominant. Vibrations of C–O–C bonds in the solvent and in-plane deformation vibrations of C–O– bonds in phenols are observed in this range of wave numbers [9]. This behavior of the ATR bands in the wavenumber range of 1050–1230 cm^{-1} may indicate the formation of ester "crosslinks" with the phenolic type OH groups. Such crosslinks were observed earlier during the Sb$^+$ implantation into FP9120 photoresist films, which led to an increase in their microhardness [12].

No significant changes in the intensity of the band at 1503 cm^{-1}, corresponding to the aromatic ring stretching vibrations, were observed. However, it should be noted that the band at 1436 cm^{-1}, responsible for the stretching vibrations of the aromatic ring associated with the CH bridge, is broadened and shifted to the low-energy region. This can be explained by the formation of hydrogen bonds or crosslinks along the CH bridge.

The most noticeable transformation of the spectrum upon irradiation with doses exceeding 1×10^{16} cm^{-2} was observed in the range of 1550–1700 cm^{-1} (Fig. 4). Pulsation vibrations of the aromatic ring carbon skeleton (bands with maxima at 1556 and 1607 cm^{-1}) and stretching vibrations of C=O bonds are observed here. The band with a maximum at 1556 cm^{-1} dominates in this range in the initial films. A weak local maximum is observed in the range of 1607 cm^{-1} (Fig. 4, curve *1*). After irradiation with doses up to $\Phi = 2 \times 10^{16}$ cm^{-2}, the absorption intensity on the high-energy shoulder of the indicated band increases in the spectral range of 1580–1700 cm^{-1} (Fig. 4, curve *2*). In this case, the intensity of the maximum at 1556 cm^{-1} remains virtually unchanged, while the intensity of the local maxima at 1607 and 1650 cm^{-1} increases slightly. At a dose of $\Phi = 7 \times 10^{16}$ cm^{-2}, the band structure changes dramatically. The intensity of the maximum at 1556 cm^{-1} decreases, and this maximum turns into an inflection. In this spectral region, the maxima at 1595 and 1607 cm^{-1} become dominant; the maximum at 1650 cm^{-1} are comparable to them in intensity. These experimental results indicate a change in the composition of substituents at the carbon ring and a significant increase in the number of conjugated double C=O bonds when the photoresistive film is irradiated with doses above 2×10^{16} cm^{-2}.

The position of the bands maxima for the C=O bonds stretching vibrations can shift within a fairly wide range when the nearest environment changes. Thus, the participation of the C=O group in the formation of intermolecular hydrogen bonds leads to a decrease in the frequency of its stretching vibrations by 40–60 cm^{-1} [13]. The absorption band maximum of these vibrations shifts to the range of 1600–1650 cm^{-1} wave numbers in the case of conjugation with C=C bonds [9]. Earlier, the formation containing conjugated bonds quinoid structures was found upon the FP9120 novolac photoresist implantation with P$^+$ ions [14]. The maximum of the respective absorption band can be in the range of 1645–1690 cm^{-1} [9] depending on the presence of conjugated bonds.

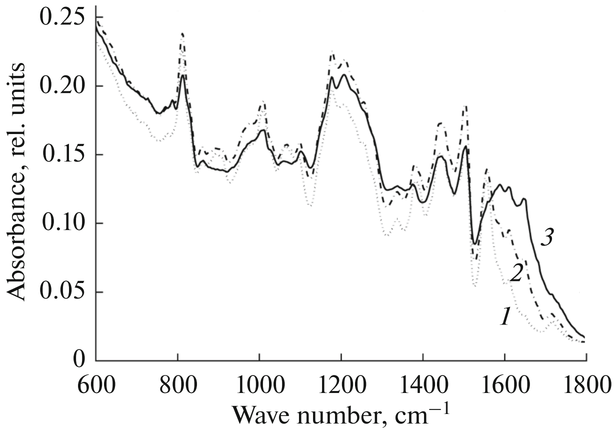

Fig. 4. ATR spectra of the (*1*) initial and irradiated with 5 MeV electrons at doses of (*2*) 1.2×10^{16} cm^{-2} (*2*) and (*3*) 7×10^{16} cm^{-2} PR films with a thickness of 3.5 μm.

CONCLUSIONS

Radiation-induced processes in *NFR* 016*D*4 photoresist films irradiated with fast electrons at doses up to 2×10^{15} cm^{-2} take place mainly with the participation of residual solvent molecules. As a result, after irradiation, the absorption bands with maxima at 1717 (stretching vibrations of C=O bonds), 1068, and 1009 cm^{-1} (vibrations of the C–O–C bond in methyl-3-methoxypropylate) disappear from the photoresist ATR spectra. At the same time, a slight increase in the intensity of a broad band with a maximum at ~3290 cm^{-1} is observed due to the O–H bonds vibrations. A process of selective damage of the solvent under the action of the polymer matrix probably takes place, for example, through the sensitized excitation energy transfer from the aromatic polymer to the solvent.

Significant changes in the intensity of absorption bands associated with the main component of the photoresist (phenol-formaldehyde resin) were observed only at irradiation doses above 2×10^{16} cm^{-2}. The most noticeable transformation of the spectrum was found in the region of 1550–1700 cm^{-1}, corresponding to the stretching vibrations of C=O bonds. The experimental results obtained indicate a change in the composition of substituents at the carbon ring and a significant increase in the number of conjugated double C=O bonds when the photoresist film was irradiated with doses above 2×10^{16} cm^{-2}.

In addition, at irradiation doses above 2×10^{16} cm^{-2}, there was a decrease in the bands' intensities due to the CH_2 and CH groups stretching vibrations (maxima at 2925 and 3012 cm^{-1}, respectively). In the region of the C—O— bonds' vibrations in phenols, (a broad structured band in the range of 1150–1270 cm^{-1} wave numbers with the main maximum at 1176 cm^{-1}), a redistribution of intensities in favor of higher-energy local maxima is observed.

No significant changes in intensity of the 1503 cm^{-1} band corresponding to the aromatic ring stretching vibrations were noted, indicating high radiation resistance of aromatic structures under fast electrons irradiation with an energy of 5 MeV. However, there was a broadening and a shift to the low-energy region of the 1436 cm^{-1} band responsible for the stretching vibrations of the CH bridge-associated aromatic ring.

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CONFLICT OF INTEREST

The authors of this work declare that they have no conflicts of interest.

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