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RADIATION-INDUCED PHASE TRANSFORMATIONS, POINT DEFECT AGGREGATION, AND NANOPARTICLE FORMATION IN GAMMA-IRRADIATED NaCl CRYSTALS

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For the first time, the influence of growth Na/Cl non-stoichiometry at the surface of a NaCl single crystal on its phase composition, gamma-irradiation-induced point-defect aggregation in both sublattices, and nanoparticle growth was studied using a combination of experimental methods and modern analytical instruments. It was found that under irradiation with doses <1 MR, the initial impurity nanophases $NaClO_3$ and Na_2O on the surface are ruptured, and instead Na2Cl, NaCl, NaOH, and metallic Na and NaH are formed.

Key words: *NaCl crystal; Gamma-ray irradiation; Color centers; Impurity nanophases; Irradiation-induced phase transformations; Local elemental composition; X-ray diffraction spectra; Optical absorption spectra*
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INTRODUCTION

Recently, sodium chloride (NaCl) crystals have attracted particular interest as a matrix for the disposal of highly active nuclear waste; therefore, several studies of gamma-induced structural defects are again relevant [1-5]. A hundred-year history of research on the effect of ionizing radiation on solids began with the discovery of color centers associated with point defects in NaCl and other ionic crystals when irradiated with UV light and X-ray quanta [2,6-15], and continued with irradiation with nuclear particles and the addition of X-ray and electron microscopic methods for the analysis of structural defects [16-23].

Optical methods formed the basis of the theory of the creation of Frenkel paired anionic defects (charged α-*I* and neutral *F-H*) as a result of the decay of anionic excitons (their creation energy is 8-8.4 eV), cationic excitons (60-62 eV), and the recombination of conduction electrons with self-trapped holes (V_K centers) [12,15,19]. There are three types of Schottky vacancies (the transfer of an atom from the bulk to a site on the surface S): a cation vacancy, an anion vacancy, and a bound pair of vacancies of the opposite sign, as well as pairs of Frenkel defects (vacancies and interstitials) in the bulk.

It was shown that at low temperatures of 5-200 K, color centers are stable, and above the anionic interstitials (*H*- and *I*-centers) as well as anionic and cationic vacancies (υ_a and υ_c) already straightforwardly migrate along the crystal lattice [2,6,9,12-15,19-23]. The concentration of paired defects was estimated using the Smakula-Dexter formula (assuming a uniform volume distribution of defects and an unchanged dielectric constant) from the experimental spectra of optical absorption and refractive index, as well as the calculated oscillator strength (0.8–0.9 for the *F* center in NaCl) [7,12,14,15]. The minimum energy of incident electrons for the collisional displacement of Cl in NaCl is estimated at ~ 290–320 keV [2,8]. The enthalpies of migration h^m were determined for the vacancy Na 0.66 eV and interstitial Na 0.29 eV, for the vacancy Cl 0.72 eV and Cl_2^- only 0.16 eV, but the maximum value 0.9 eV was obtained for the divacancy complex $\{V_{Na}V_{Cl}\}$. Thus, it was proved that molecular Cl migrates more easily than the Na atom. This explains why, during radiolysis, Cl evaporates faster as a gas, while Na remains on the surface and forms colloids [2,5,10-13,15,18,19,21,22]. However, the non-stoichiometry of the surface after evaporation of Cl during radiolysis was not measured directly. The total surface area of the crystal should increase as Schottky defects and their agglomerates (nanopores and nanopillars) form, but this can only be measured with modern atomic force microscopes.

Therefore, the purpose of this work was to experimentally investigate the effect of the initial (growth) Na/Cl non-stoichiometry of the surface composition of a NaCl single crystal on the gamma radiation-induced aggregation of point defects and the growth of nanoparticles (metal or other nanophases) using a set of experimental methods.

OBJECTS AND METHODS OF RESEARCH

We used pure high-quality NaCl crystals for IR windows, grown in an inert atmosphere by the classical Kiropulos method in a graphite crucible, with a standard diameter of ∅35 mm and a thickness of 4 mm; optical polishing was performed at LOMO (Russia). The samples were wrapped in Al foil for isolation from air (possible oxidation of the surface) and irradiated in a dry channel at 320 K ^{60}Co with gamma quanta at a dose rate of 145 R/s (energies 1.17 and 1.32 MeV, when corpuscular properties prevail over wave properties) at 4π geometry (isotropic irradiation) to doses

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from 10^5 R, when Frenkel pairs accumulate to 10^7 R (10 MR), when agglomerates and colloids are effectively formed [2,5,13,15,17-22].

Micrographs of the samples before and after irradiation were taken on a scanning electron microscope EVO MA10 (Zeiss) at different magnifications, and the local elemental composition was determined using a built-in EDS attachment with an electron backscattering detector (Oxford Instr. Resolution 128 eV) in the energy range of X-ray quanta from 0.1 to 30 keV. Scanning probe microscopy was carried out on SPM (Shimadzu) to see nanoscale roughness of the surface layer and its change induced by the gamma-irradiation (radiolysis etc). Optical absorption spectra were recorded on an SF-56 spectrophotometer in the wavelength range of 190–1100 nm and optical density range from -1 to +5 in the mode of strongly absorbing objects (slit spectral width 5-10 nm). The phase composition was determined by X-ray diffraction over a wide angular range from 5 to 140 degrees on an Empyrean diffractometer (PANalytical).

RESULTS AND DISCUSSIONS

NaCl crystals were irradiated with gamma rays at Debye temperature = 322 K (which turns out to be very close to the temperature of the dry channel of the gamma source), while all vibrational modes of the lattice were excited and effective diffusion of Na and Cl along interstices was ensured [18, 20].

Taking the known ratio 1 R/s = $2\cdot10^9$ quant/cm^{2}s for the energy of a gamma quantum of 1 MeV, the dose rate of 145 R/s = $2.9\cdot10^{11}$ quant/cm^{2}s and the exposure dose in the flux range from $2.9\cdot10^{16}$ to $3\cdot10^{17}$ quant/cm^2. Since one 1.17 MeV gamma quantum can create either one electron-positron-pair with an energy of 0.585 MeV each, or up to 10^3 Compton electrons with an energy of ~ 100 eV each, sufficient to displace a light atom (A<40) upon collision (25-50 eV) and generation of a pair of defects [2, 8]. However, the minimum energy of an incident electron for the displacement of Cl in NaCl is estimated in the range of 290–320 keV and the radiation-chemical yield of stable *F*-centers 1.5/100 eV and unstable 1.75 [2,17,18,21]. However, according to another estimate, for A = 30, the electron energy in the beam is 0.6-0.8 MeV [8].

The experiment began by determining the local elemental composition of the defective surface. Figures 1A–1D show microimages and nanoscale surface roughness of a NaCl crystal in the non-irradiated state and after irradiation with a dose of 10 MR, as well as local spectra of characteristic X-ray radiation from the near-surface layer. The growth dislocations and tracks of polishing are clearly visible even on non-irradiated sample with high-quality optical polishing, which indicates the presence of both aggregated and nanoscale structural defects. Bright spots indicate areas where the surface charge is localized.

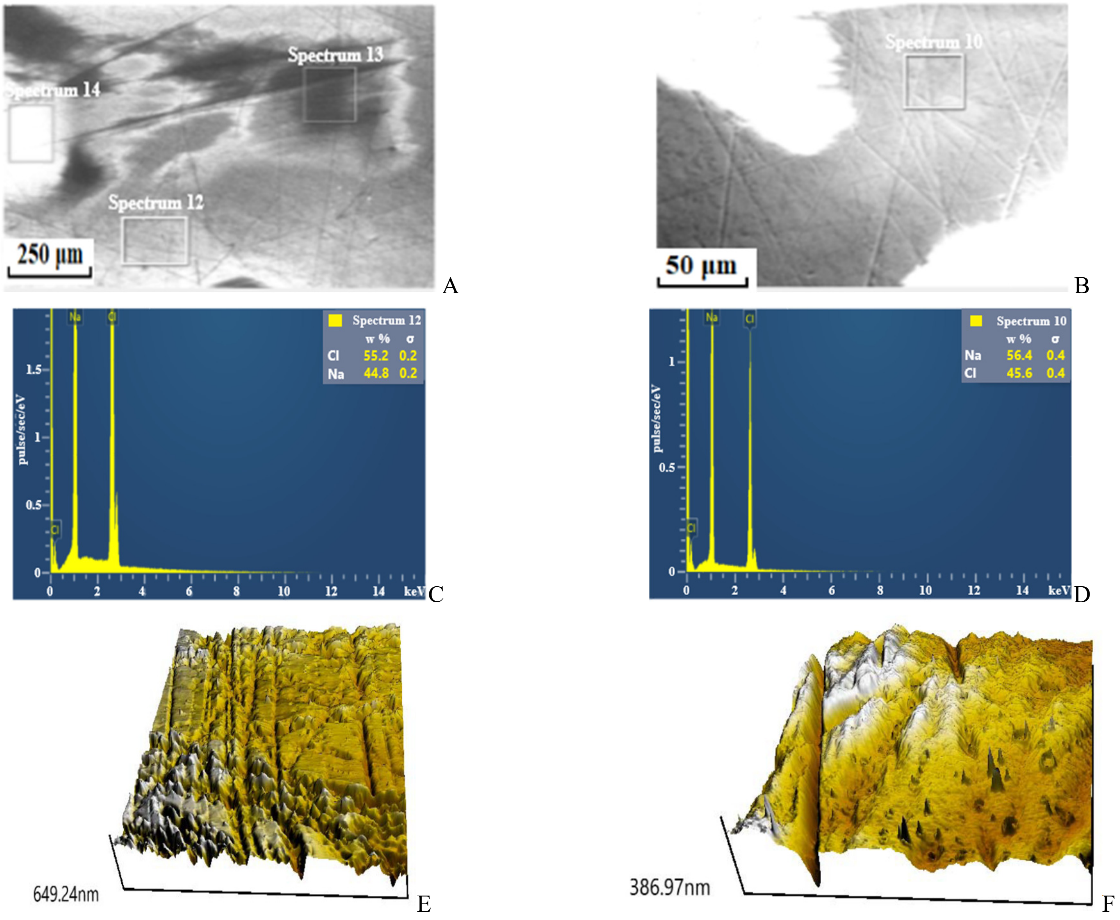

Figure 1. SEM and SPM images of the polished surface of a NaCl crystal: A, E) unirradiated and B, F) after gamma irradiation at 320 K and 140 R/s with a dose of 10^7 R; the squares show the places where the local characteristic spectrum C) and D) were recorded

Figures 1E and 1F show the surface profiles before and after γ-irradiation. It can be seen that the surface roughness (according to the Z scale) after the initial optical polishing reaches about 650 nm due to the presence of frequent nanosized channels and protrusions of the dominant cubic phases $NaClO_3$ and Na_2O. The most anisotropic orthorhombic phase, Cl_2, as well as nanosized cubic HCl and Na, appear to be located within nanogrooves on the channel walls. After irradiation, the depth of the furrows and the height of the peaks decrease by approximately a factor of two due to radiation-enhanced diffusion, and the NaCl matrix becomes exposed. At the same time, a new NaH phase forms in the shape of sharp-edged cones with heights of up to 100 nm, while the furrows deepen as a result of chlorine loss (Table 1).

The chlorine content even on a non-irradiated surface is less than the stoichiometric (60.65%), but still more than sodium. The chlorine content on the irradiated surface is already so much less than stoichiometric that it is already less than sodium. This is direct quantitative evidence of surface radiolysis with the removal of chlorine. It is energetically disadvantageous to create such a quantity of chlorine vacancies; therefore, an effective agglomeration process occurs with the formation of metal colloids and pores [10, 11, 17-22].

Table 1. Local elemental composition (wt %) of the surface layer of the NaCl crystal (the second column is the stoichiometric ratio according to the chemical formula) before and after gamma irradiation

	NaCl	**Unirradiated**			**Dose 10^7 R**		**Error, %**
Element	**formula**	**12**	**13**	**14**	**10**	**11**	
Na	39.35	44.8	44	44.2	54.6	52.6	0.2-0.6
Cl	60.65	55.2	56	55.8	45.4	47.4	0.2-0.6

To confirm this, the X-ray diffraction spectra were measured, and the structure and phase composition were determined, as shown in Fig. 2 and Table 2.

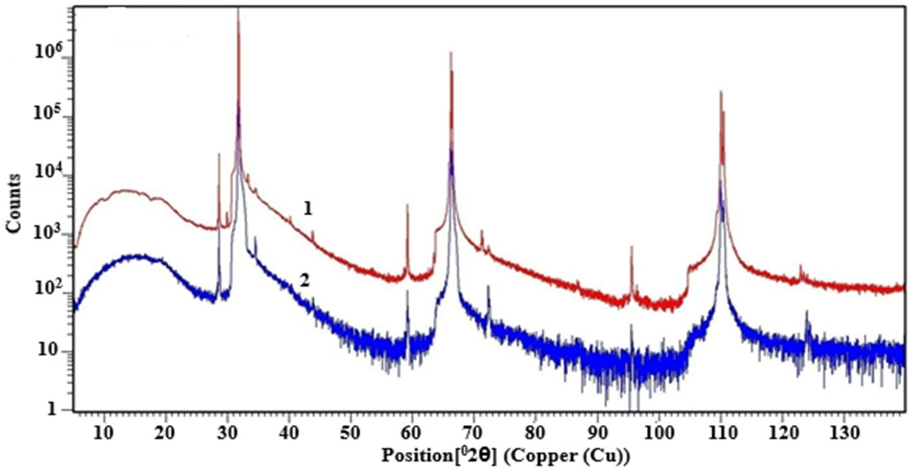

Figure 2. Wide-angle X-ray diffraction spectra of a pure NaCl crystal before (1) and after gamma irradiation at 300 K and 140 R/s with a dose of $7 \cdot 10^5$ R (2)

The spectra show that each high-intensity matrix reflection (h00) is accompanied by incoherent Compton scattering due to the interaction between X-rays and weakly bound valence electrons, which is an important effect in the case of light elements such as Na and Cl. A weak reflection (110) at 29.5^0 was attributed to Na nanoparticles [17,18,20,21]. After gamma irradiation, a systematic shift of reflections towards small angles is observed, a decrease in their amplitude and broadening at the level of half the amplitude are due to phase transitions as a result of additional loss of chlorine from the surface.

Table 2. Structure and phase composition of the surface layer of a NaCl crystal before and after gamma irradiation at 300 K and 140 R/s with a dose of $0.7 \cdot 10^6$ R.

Sample	**Phase %**	**Group**	**Structure**	**Lattice parameters, Å**		
				a	**b**	**c**
Reference NaCl	Na -1%	Im-3m	Cubic	4.221	4.221	4.221
	NaCl-6%	halite HP, syn Fm-3m	Cubic	5.453	5.453	5.453
	Na(ClO_3)-47%	sodium chlorate(V) P213	Cubic	6.340	6.340	6.340
	Cl_2-4%	dichlorine Cmca	Orthorhombic	6.290	4.500	8.210
	HCl-2%	Fm-3m	Cubic	5.482	5.482	5.482
	Na_2O-40%	Fm-3m	Cubic	5.560	5.560	5.560

Sample	Phase %	Group	Structure	Lattice parameters, Å		
				a	b	c
NaCl D=0.7 MR gamma irradiation	NaCl-16%	Fm-3m	Cubic	5.620	5.620	5.620
	NaH-4%	Fm-3m	Cubic	4.880	4.880	4.880
	Na(ClO$_3$)-8%	sodium chlorate(V) P213	Cubic	6.486	6.486	6.486
	NaOH-11%	α-NaOH Bmmb	Orthorhombic	3.399	3.399	11.377
	Na-3%	P63/mmc	Hexagonal	3.767	3.767	6.154
	Na$_2$Cl-58%	Cmmm	Orthorhombic	3.291	10.385	2.984

As you can see from Table 2, the unirradiated polished surface contains nanopores with Cl_2 and HCl and nanoparticles of Na and NaH, as well as impurity nanophases $NaClO_3$ and Na_2O, which are all cubic and practically cover the bulk cubic structure of NaCl. After the accumulation of a gamma dose of 0.7 MR, the impurity oxygen-containing phases decompose with the formation of dense orthorhombic and hexagonal structures and partial reduction of the basic NaCl lattice. So, for the first time, gamma-radiation-induced phase transitions in the near-surface layer were discovered, for which much more energy from absorbed gamma rays is spent than for simple displacements of atoms from site to interstice to saturating concentrations of 10^{17}-10^{19} cm^{-3} [9,13,15,19,22], which must be taken into account in the energy balance.

Having determined experimentally the deficit of chlorine and the phase composition of the near-surface layer, the dose dependences of the absorption spectrum were measured to establish the dose when pair chlorine vacancies are formed, from which, presumably, chlorine molecules evaporate. In fig. 3 shows the absorption spectra of NaCl crystals measured in steps of dose accumulation from $2\cdot10^4$ R to $3\cdot10^7$ R. The results obtained do not contradict the literature data on the position of absorption bands and color instability [7,12,13,15-19,22].

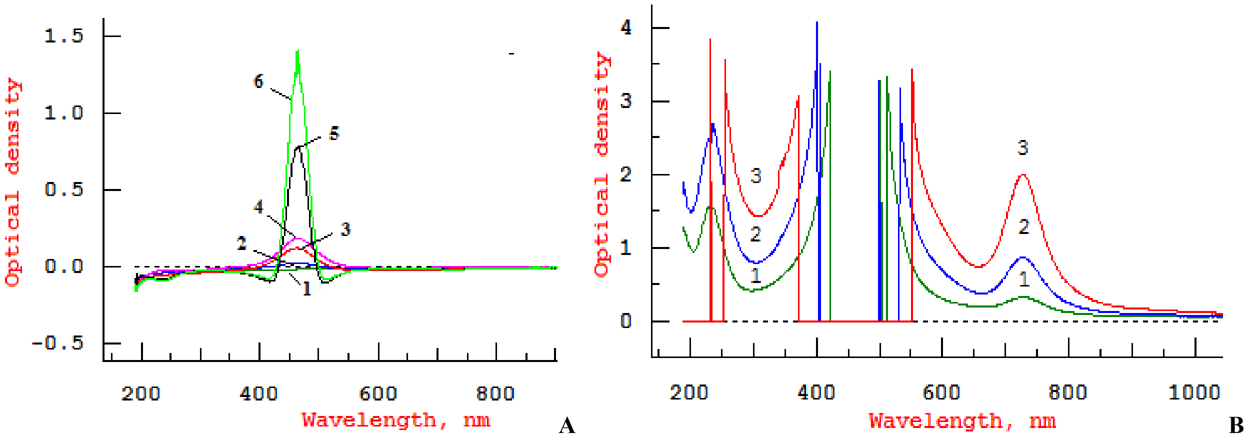

Figure 3. Absorption spectra of NaCl crystals scanned from 190 to 1100 nm on an SF-56 spectrophotometer:
A) 1- non-irradiated, small doses: 2(blue) – 2×10^4 R, 3(red) – 5×10^4 R, 4(pink) – 10^5 R, 5(black) – 2×10^5 R, 6(green) – 3×10^5 R;
B) large doses 1(green) – 7×10^5 R, 2(blue) – 10^7 R, 3(red) – 3×10^7 R

However, measurements in the mode of strongly absorbing materials (Fig. 3B) made it possible to see that the saturation of absorption by *F* centers noted by all authors and the simultaneous appearance of aggregate F_2 and F_3 centers does not exist. Instead, there is a break a continuous extinction function on the wavelength, as if an impurity band were formed in the forbidden band.

The splitting of the *F*-center band appears at a dose of $5\cdot10^5$ R, when paired *F-F* centers with electric dipole interaction are formed. In fig. 3A, for the first time, strong dispersion was found around the intense absorption band of the *F* center, where the optical density even takes negative values. According to G. Mie's theory of light scattering by metal nanoparticles in a transparent dielectric [24]. This means that this band of light is effectively reflected, which characterizes metal clusters at the site of anion vacancies, which fully agrees with the above results of the elemental and phase composition of the surface (the presence of metallic phases of sodium). Figure 3B shows that the higher the radiation dose, the higher the concentration of paired centers (category of molecular centers), the closer the distance between the centers in each pair, the stronger the electron-electron interaction and, accordingly, the greater the splitting between the levels. The sodium plasmon resonance line is observed at 500 nm. When the transition of an electron between an occupied and an unoccupied level is allowed by the rules of optical selection, this leads to a strong absorption of a photon with an energy equal to the difference between the energies of the occupied and unoccupied levels. The maximum of the imaginary term of the dielectric function ϵ_i can be expected at $h\nu_1$ and $h\nu_2$. Since these maxima correspond to strong absorption, they determine the color of the solid. The physical condition for strong absorption is that the states in the valence and conducting bands are dispersed in parallel over large parts of the

Brillouin zone. This condition is obviously satisfied for very flat bands arising from strongly localized states [21,23]. In our case, under ionizing irradiation, strongly localized states appear near the level of the chemical potential μ near the middle of the forbidden band with excited levels very close to the valence and conducting bands. At high doses, these states come so close to each other that the electrical dipole interactions between them become significant. The electric dipole moment arises due to ionic polarization: the negatively charged chlorine sublattice is displaced relative to the positively charged sodium ions.

CONCLUSIONS

The use of a set of experimental methods for studying the crystal structure, the spectra of intense optical absorption by colour centres, and the elemental and phase composition of the near-surface layer on modern analytical instruments enabled the discovery of new effects even in the widely studied ionic NaCl crystal. The X-ray diffraction spectra, presented for the first time on a semilogarithmic photon-counting scale, clearly show strong reflections from the matrix and weak reflections from impurity phases. For the first time, the effect of the initial (growth) nonstoichiometry of the surface of a NaCl single crystal with a Chlorine deficiency on the aggregation of point defects in both the anionic (pores with Cl_2 and HCl) and cationic (Na and NaH nanoparticles) sublattices was found. Impurity nanophases $NaClO_3$ and Na_2O were identified, which passivate the superstoichiometric aggressive Na. For the first time, gamma-radiation-induced damage of the existing surface phases and formation of new nanophases were discovered, as a result of which the surface layer of the basic lattice of NaCl was partially restored, which should be taken into account in the distribution of the absorbed dose in the bulk and in the near-surface layer.

The obtained results and research methods can be applied for the dosimetry of long-term irradiation in depositories of high-level waste in the NaCl matrix and their structural changes.

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РАДІАЦІЙНО-ІНДУКОВАНІ ФАЗОВІ ПЕРЕТВОРЕННЯ, АГРЕГАЦІЯ ТОЧКОВИХ ДЕФЕКТІВ ТА УТВОРЕННЯ НАНОЧАСТИНОК У ГАММА-ОПРОМІНЕНИХ КРИСТАЛАХ NaCl

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Вперше за допомогою комбінації експериментальних методів та сучасних аналітичних приладів досліджено вплив нестехіометрії росту Na/Cl на поверхні монокристала NaCl на його фазовий склад, агрегацію точкових дефектів в обох підґратках, індуковану гамма-опроміненням, та ріст наночастинок. Було виявлено, що при опроміненні дозами <1 MR початкові домішкові нанофази $NaClO_3$ та Na_2O на поверхні розриваються, а замість них утворюються Na2Cl, NaCl, NaOH та металеві Na та NaH.

Ключові слова: *кристал NaCl; гамма-опромінення; центри забарвлення; домішкові нанофази; фазові перетворення, індуковані опроміненням; локальний елементний склад; рентгенівські дифракційні спектри; оптичні абсорбційні спектри*