

# Controlling the Low-Temperature Ionic Purification of a Silicon Surface by Electron Spectroscopy

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## Research Article

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# Abstract

A comparative study is carried out for etching a surface with negative and positive ions, and the results show completely opposite physical processes occur on a silicon surface. Irradiation with positive ions exhibits oxide removal, while irradiation with negative ions shows an intense oxidation of the sample surface. Technology for low-temperature ion cleaning and the electron-spectrometric control of silicon wafers has been developed. This technology has been shown that only irradiation with caesium ions followed by annealing at 650 °C leads to an atomically clean silicon surface for several minutes, which is dependent on the pressure of residual gases under vacuum. After cleaning in a vacuum of 10<sup>-9</sup> Pa, the silicon surface begins to oxidize again within 10 min.

## 1 Introduction

One of the most popular materials in modern micro- and nanoelectronics is monocrystalline silicon and silicon dioxide [1, 2]. Recently, thanks to the creation of quantum-dimensional structures on silicon [3–5], the difficulty of using the above materials in photoelectronics, due to their low probability of radiative recombination, has been overcome [6–7]; thus, a new impetus has been obtained for its widespread use in the aforementioned fields of science and technology [8]. In the production of silicon integrated circuits, in addition to the quality of the surface structure, the choice of the crystallographic orientation of the substrate crystals is of great importance [9–11]; for example, the (111) planes are oxidized faster than the (100) planes due to the high surface packing density of atoms capable of entering the oxidation reaction [12]. The degree of purification has a direct impact on the quality of products; therefore, an increasing number of microelectronic companies are moving in this direction [13–16]. Much attention has been given to cleaning the surface of semiconductor materials at all development stages of microelectronics [17]. The problem of surface cleanliness has become particularly important with the emergence of planar technology [18], in which the properties of the surface have a significant effect on the parameters of devices. Knowledge of the adsorption and desorption mechanisms of impurities on the surface of semiconductor materials is extremely important for a targeted, efficient purification process [19]. Additionally, orientation effects during the etching of a surface of crystals are considered to be important for obtaining white light from GaN-based silicon LEDs [20–22].

There are various methods for obtaining materials with atomically clean surfaces [23–25]. Each of them has certain advantages and disadvantages. Therefore, there is no universal cleaning procedure. Each material usually requires an individual technique or even a combination of several techniques. Currently, the most promising method for obtaining atomically clean surfaces is low-energy ionic and plasma-chemical etching [26].

A very large amount of research in the field of surface cleaning of silicon crystals has been carried out by many groups of scientists around the world over the past 20 years. Various conditions of the surface cleaning process have been studied, and various methods and approaches have been used to solve identified problems. Improvements in the characteristics of silicon crystals that are required for certain

practical applications have been convincingly shown. Additionally, it is surprising there are practically no studies in the literature comparing surface cleaning with the use of ion bombardment and electron spectroscopic methods. Currently, there is no universal equipment or technological process for cleaning the surfaces of semiconductor wafers that fully satisfies all manufacturers of semiconductor products.

Thus, in the production centres of the world, when obtaining metal-dielectric-semiconductor structures, before the deposition of films, the diffusion of atoms and other technological procedures, it is necessary to clean the surface of the silicon substrate by removing the oxide film through the use of ion bombardment and subsequent annealing. In this case, it is necessary for particles to collide with the silicon surface many times, for instance during oxidation, the physical and chemical adsorption of gases, ion bombardment, etching, and subsequent annealing. Research has repeatedly determined that in the process of oxidation and cleaning a surface, silicon has a large influence on the formed surface defects, and their participation in a particular physical process affects the surface state of the substrate [27–28]. In this regard, a comparative study of the total current (TC) spectroscopy of silicon and silicon oxide is of certain interest.

Various control methods are used to study the surface of silicon wafers. To accurately determine the state and properties of the substrate surface, it is optimal to use a combination of several methods for monitoring the surface of semiconductor wafers.

This work is devoted to the study of using TC spectroscopy for obtaining an atomically clean silicon surface in vacuum by irradiation with negative and positive ions, which is then followed by annealing.

## 2 Experimental Methods

A detailed description of the methods of TC spectrometry [28–32] and secondary ion mass spectrometry (SIMS) is provided in [32–33]. The TC spectra of silicon crystals were obtained by irradiation with electrons at an energy of 0–100 eV, a current of  $\sim 10^{-8}$  A, a current of  $\text{Cs}^+$  and  $\text{Cl}^-$  of  $\sim 10^{-6}$  A, and an energy of 3 keV. The surface cleanliness was monitored using SIMS. Plates of p-type silicon single crystals (SiHB-7.5) with a (111) face orientation were used as samples. The working vacuum during the research was  $10^{-9}$  Pa.

## 3 Results And Discussion

Before starting work on TC spectroscopy, the mass spectra of secondary ions were taken in parallel from the surface of dirty, unannealed silicon by SIMS. The results of studying the band structures of silicon and silicon oxide by TC spectroscopy show that the distributions of the density of states of these semiconductors are similar; in the valence band (0–14 eV), there are three maxima of the density of occupied states. The conduction band is characterized by two extrema in the energy range between the Fermi and vacuum levels. As a function of the density of states, the spectrum of the TC of silicon and silicon oxide should not contain a highly fine structure. However, the surface of these substances is

characterized by a high degree of covalent bonds (51%), and the energy structure of the near-surface region can differ significantly from that of the bulk region, thereby undergoing a strong geometric rearrangement [34].

Figure 1 shows the TC spectra during the adsorption of gases on the silicon surface. The observation of gas adsorption from the TC spectra is determined by the nature of the structural rearrangement based on the density of states in the near-surface region of silicon. In the case of physical adsorption, when the interaction energy of atoms is low, it can be expected that the electronic structure of the surface does not change significantly. The signal intensity of the substrate is attenuated due to additional scattering in the adsorption layer. The adsorption of gaseous molecules such as CO, CO<sub>2</sub> and H<sub>2</sub>O does not introduce features into the TC spectrum of silicon; they only weaken the spectral intensity [33].

In the case of chemisorption, when the energy between the atoms of the substrate and gases is significant and electronic exchange occurs between the substrate and adsorbed particle, one can expect the appearance of additional features in the spectra due to the excitation of hybridized electronic states [34].

One of the features of the TC electron spectroscopy method is the possibility of parallel monitoring of the surface potential by the position of the primary maximum in the spectrum [27, 29]. Carrying out such measurements is very important in the study of adsorption processes, especially since in a comparative analysis, the shift in the primary maximum and/or the entire spectrum, makes it possible to distinguish the two components of change in the work function—one is due to a change in affinity, and the other is due to a change in the bending of zones [30]. Thus, additional control over the condition of the adsorption coating is carried out.

The change in the TC spectrum and the work function of the Si(111) sample during the adsorption of gases at room temperature are observed. The 1-curve in Fig. 1 corresponds to the TC spectrum of a clean Si(111) surface with a 7x7 structure, which is shown for comparison. Curve 2 corresponds to holding the sample for 10 min in vacuum at room temperature, accompanied by a decrease in the work function by approximately 0.5 eV and a decrease in the intensity of the maxima in the TC spectrum of pure silicon. In this case, only a shift in the primary maximum is observed, and the entire spectrum does not change its position relative to the Fermi level (which indicates the invariability of band bending). Consequently, at the first stage of adsorption, a change in the work function occurs due to a decrease in the electronic affinity. At this stage of adsorption (physical adsorption), heating the sample (up to 500°C) leads to the cleaning of the silicon surface from the adsorbed layer, and the surface potential and the form of the TC spectrum return to their original values.

Further holding the sample for 20 min at room temperature (curve 3), a further decrease in the work function is observed (by ~ 0.7 eV), and the entire spectrum shifts together with the primary maximum, which indicates a decrease in the surface bending of the bands. This, in turn, indicates a decrease in the negative charge of the surface states. The obtained result is agrees with the concept of the oxygen

chemisorption mechanism, according to which oxygen occupies the free surface valences of silicon and decreases the density of surface electronic states.

The intensities of the peaks in these curves are very weak; furthermore, these peaks are not sharp. This result occurs due to the scattering of electrons in the polycrystalline lattice of the adsorbed layer. The half-width of the primary peak is approximately 1.2 eV; thus, broadening of the peak has occurred. This result indicates the degree of surface inhomogeneity of the sample under study. The primary peak at electron energies of 3.5 eV shows the origin of the kinetic energy of electrons and corresponds to the position of the vacuum level on the energy axis.

After annealing at a temperature of 200°C, no noticeable change in the spectrum is observed, and only the intensity of the curve characteristic of the spectrum that represents the total SiO<sub>x</sub> current increases. After thermal annealing for 10 min at a temperature of 600°C, the primary peak begins to double. If a bifurcation of the primary peak occurs on the surface of the island film, the energy position of one corresponds to the work function of the substrate, and the other to the work function of the island surface. The bifurcation of the primary peak indicates the formation of carbon and oxide silicon compounds.

As seen in the spectrum of the total current, a maximum of δ appears at an energy of 13 eV, compared with the NEXAFS and TDOS data [29], and this maximum corresponds to the excitation of the 2p state of the carbon atom to unfilled states above the vacuum level. According to the shape of the primary peak, we can say that after annealing at a temperature of 700°C, the surface is completely covered with a continuous oxide layer. The manifestation of the α, β, and γ maxima indicates surface electronic states that are characteristic of oxidized silicon.

It can be noted that at an annealing temperature of 500°C, an increase in the peak of carbon ions on the surface can be observed. When comparing the TC spectra, it is found that at silicon annealing temperatures above 500°C, interstitial carbon leaves the bulk for the silicon surface. Further increasing the annealing temperature to 1000°C does not lead to cleaning carbon and its compounds off the surface.

The diffraction structure present in the TC spectra contains certain information about the properties of the geometric structure of the surface. When studying single-crystal samples, the energy dependence of the reflected beam intensity reveals additional features in the range of energies of primary electrons, which correspond to the conditions for the appearance of diffraction beams. These features are observed at energies somewhat lower than the threshold for the appearance of a diffraction beam, namely, in the form of alternating extrema. According to existing concepts [27], the fine structure is due to multiple scattering of a diffraction beam on a surface potential barrier, which, at certain ratios between the parameters of the barrier and the electron momentum, leads to a resonant change in the intensity of the reflected and transmitted beams. This phenomenon is known as surface resonance, or the resonance of surface states [29], since in the dynamic theory of diffraction, the features of surface scattering are

associated with quasi-stationary surface states located above the vacuum level. Samples with a broken surface do not exhibit a diffraction pattern (for example, ion bombarded, gas adsorbed, or polycrystalline samples); therefore, there is no diffraction component in the spectrum. This observation also applies to measurements of the temperature dependence of TC spectra. It is known that the intensity of diffraction beams decreases with an increasing sample temperature. Since the wavelike feature is associated with multiple scattering of the diffraction beam on the surface potential barrier, a decrease in its intensity with an increasing temperature should lead to a decrease in the intensity of the diffraction feature in the TC spectrum. Using one of the considered criteria, it is possible to confidently distinguish features of a diffraction nature from the spectrum of the total current. The promising nature of the diffraction effects in the TC spectra makes it possible to obtain not only qualitative but also quantitative information on the surface structure and dynamics of surface particles.

Figure 2 shows the TC spectrum of silicon during etching with positive caesium ions and subsequent annealing. Diffraction effects in the TC spectra are shown in parallel.

Curve 1 in Fig. 2 corresponds to the TC spectrum of pure silicon with a 7x7 structure. Curve 2 corresponds to the TC spectrum of the silicon surface after irradiation with positive caesium ions at an energy of 2.5 keV and with a dose of  $10^{13}$  ions/cm<sup>2</sup>. As seen from the spectrum, the primary peak shifts towards low energies, which indicates a decrease in the work function of the surface, while the intensity of the diffraction component of the spectrum also decreases (the right side of the spectrum is 30–40 eV). After increasing the dose to  $10^{14}$  ion/cm<sup>2</sup> (curve 3), peaks appear on the low-energy side of the spectrum at energies of 1.0 ( $H_{Cs}$ ) and 5.0 eV. Notably, the peaks at 7.0 and 10.0 eV disappear, indicating the interband states of silicon. After increasing the dose to  $10^{15}$  ion/cm<sup>2</sup> (curve 4), the intensity of the peak ( $H_{Cs}$ ) at 1.0 eV increases, and the maxima and minima characteristic of the TC spectrum of silicon, which indicates surface degradation, disappear. The peak at an energy of 1.0 eV may correspond to interstitial caesium defects on the surface, since the energy level of the peak lies within the band gap of silicon [29, 34].

A further increase in the dose only leads to an increase in the peak at 1.0 eV; the rest of the fine structure of the spectrum does not change. Annealing at temperatures of 100 and 200°C (curves 5 and 6) leads to an increase in the intensity of the peak at 1.0 eV, which confirms the disrupted nature of the crystal structure of the surface. At such temperatures, there is diffusion of the formed defects from the bulk to the surface. At a temperature of 200°C, the lowest work function of the surface is observed, which indicates the formation of a continuous caesium film on the silicon surface. From the TC spectrum of curve 8, it can be concluded that annealing at a temperature of 400°C leads to a decrease in the concentration of caesium atoms on the surface, which is shown with a decrease in the intensity of embedded defects on the surface (a decrease in the peak intensity at 1.0 eV ( $H_{Cs}$ ))). However, in this case, a doubling of the primary peak is observed, indicating the appearance of islands on the silicon surface. Additionally, the broad peak at 5.0 eV also broadens and doubles.

In the total current spectrum, maxima A and B appear at energies of 6.0 and 7.0 eV, respectively, showing the hybridization of surface silicon atoms with caesium atoms. The diffraction pattern of the TC

spectrum is also restored, which shows the restoration and reorientation of the surface structure. Upon annealing at 650°C, the structure of pure silicon with a 7x7 structure is restored (curve 11).

For comparison, the TC spectrum of silicon was also recorded during etching with negative chlorine ions and subsequent annealing (Fig. 3). Curve 1 in Fig. 3 corresponds to the TC spectrum of pure silicon with a 7x7 structure. Curve 2 corresponds to the TC spectrum of the silicon surface after irradiation with negative chlorine ions at an energy of 2.5 keV and with a dose of  $10^{13}$  ions/cm<sup>2</sup>. The spectrum of chlorine irradiation with a dose of  $10^{13}$  ions/cm<sup>2</sup> leads to irreversible degradation of the silicon surface (the diffraction component of the TC spectrum of 30–40 eV disappears). In this case, an intense peak appears at an energy of 0.8 eV. There is also a doubling of the primary peak; however, it does not shift towards low energies but towards high energies, which shows an increase in the work function of the silicon surface.

Broad maxima of α and β are also observed. The intense peak at 0.8 eV corresponds to embedded chlorine defects on the silicon ( $H_{Cl}$ ) surface. After increasing the dose to  $10^{14}$  ion/cm<sup>2</sup>, curve 3 leads to an increase in the intensity of the 0.8 eV peak, and the α and β peaks in the TC spectrum are also resolved. At irradiation doses of  $10^{15}$  ions/cm<sup>2</sup>, the intensities of the  $H_{Cl}$ , α and β peaks also increase (curve 4).

A further increase in the dose does not lead to noticeable changes in the structure of the TC spectrum. Upon annealing at 100°C, the intensity of the  $H_{Cl}$  peak immediately doubles (curve 5). This result indicates the diffusion of implanted chlorine atoms on the sample surface. Furthermore, a decrease in the intensity of the α and β peaks is also observed, and their shift towards low energies indicates bending of the surface states. A peak also forms at an energy of 11.0 eV –γ. Increasing the annealing temperature by 100–400°C leads to an increase in the intensity of the α, β and γ peaks. The embedded defects are completely annealed, showing a peak at an energy of 0.8 eV (curves 5, 6, 7, 8). A close look at Fig. 3 shows its similarity to the TC spectrum in Fig. 1. However, in Fig. 3 there is no carbon peak at 17.0 eV (δ), and the physics of this phenomenon will be described in detail in future works.

Under irradiation with negative chlorine ions, oxidation of the silicon surface is observed, and in the case of caesium ions, cleaning of the silicon surface is observed. Annealing in both cases enhances the observed effects. Studies carried out under irradiation with different ions show that the structure of the surface state depends not only on the dose, energy, and temperature but also on the mass and value of charge of the primary ion.

Thus, for cleaning the silicon surface, it is necessary to use alkaline ions with a large mass to promote faster etching of the surface oxide.

## 4 Conclusion

The comparative studies carried out on surface irradiation with negative and positive ions showed the completely opposite sides of the physical process taking place on the silicon surface. Under irradiation with positive ions, oxide removal was observed, and upon irradiation with negative ions, intense oxidation of the silicon surface was observed. This phenomenon can be associated with the occurrence of chemical reactions between the primary halogen and alkali ions with atoms of the silicon surface.

A technology for low-temperature ion cleaning and electron-spectrometric control of silicon wafers has been developed. However, it has been shown that only irradiation with caesium ions followed by annealing (at a temperature of 650 °C) of the sample leads to an atomically clean silicon surface for several minutes, which is dependent on the pressure of residual gases under vacuum. Irradiation with caesium ions prevents the formation of oxides on silicon by etching the surface. After cleaning in a vacuum of 10<sup>-9</sup> Torr, the silicon surface begins to oxidize again within a few minutes (10 min).

## Declarations

### \* Ethics approval and consent to participate

Not applicable

### \* Consent for publication

Not applicable

### \* Availability of data and material (data transparency)

Authors are assured that all data and materials support their published statements and comply with industry standards.

### \* Conflicts of interest/Competing interests

The authors report no conflicts of interest include appropriate disclosures. The authors alone are responsible for the content and writing of this article.

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### \* Disclosure of potential conflicts of interest

Not applicable

**\* Research involving Human Participants and/or Animals**

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**\* Informed consent**

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**Authors' contributions:**

All authors contributed to the research conception and design manuscript. Material preparation, data collection and analysis were performed by Utkir Bahodirovich Sharopov, Muzaffar Kadambaevich Kurbanov, Erkin Turobovich Juraev and Dilmurod Shamurodovich Saidov.

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Obtained results of silicon electronic levels and surface states were discussed and agreed with professor Kulwinder Kaur. All authors read and approved the final manuscript.

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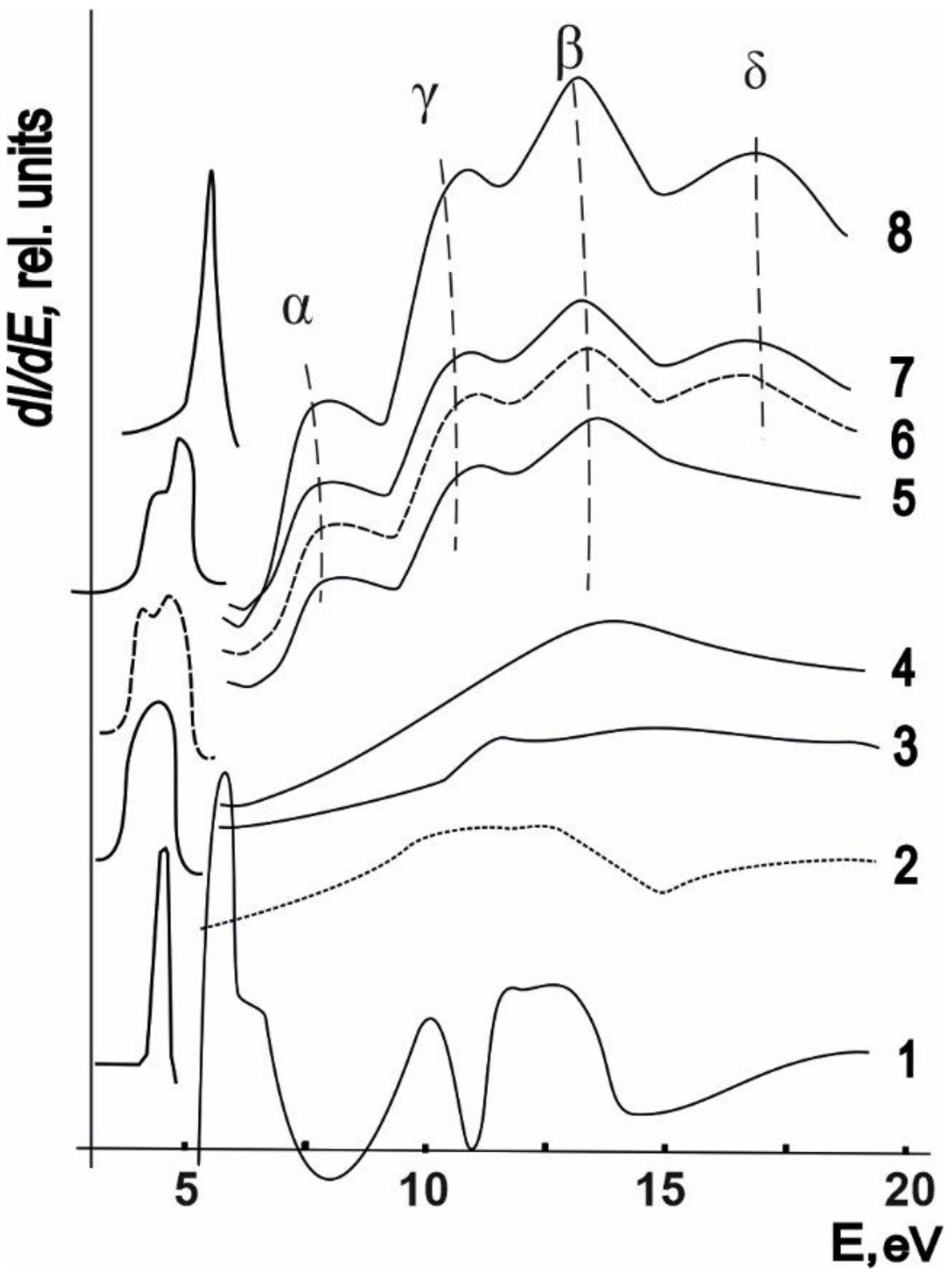
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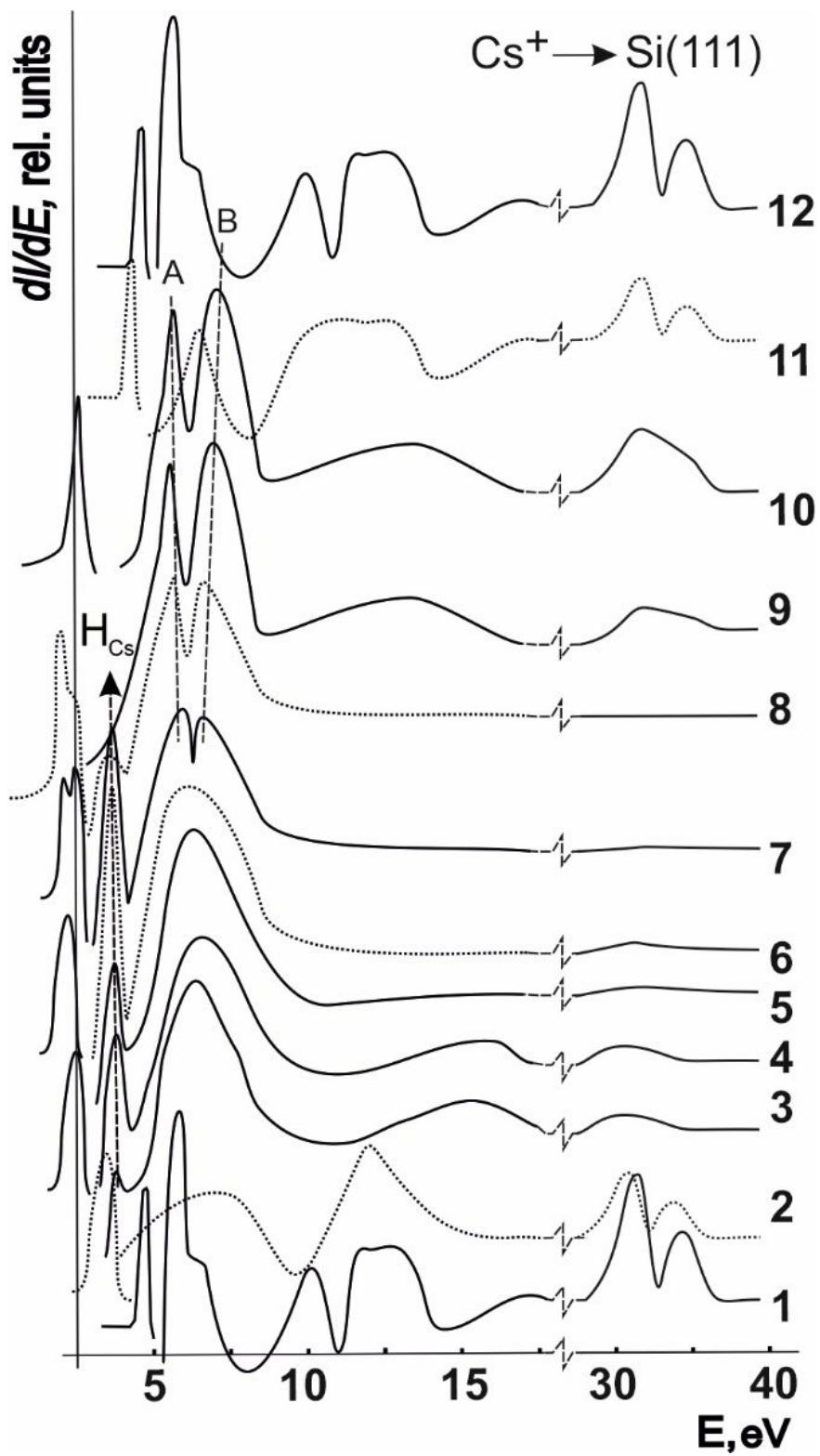
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## Figures



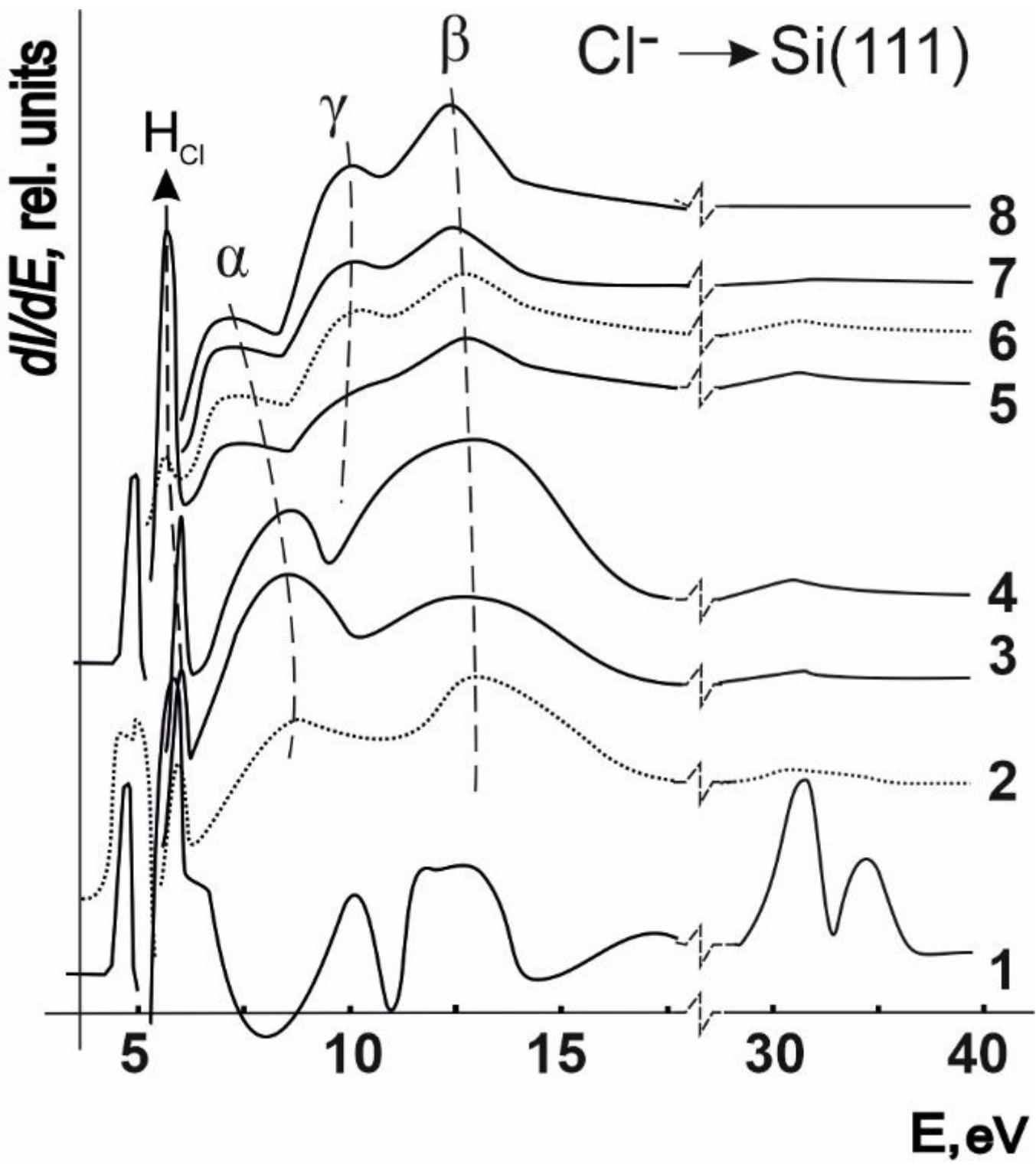
**Figure 1**

Change in the TC spectrum of Si(111) during the adsorption of gases and subsequent annealing 1-Pure silicon with a 7x7 structure. 2-Exposure for 10 min in a vacuum of 10<sup>-9</sup>Pa. 3-Exposure for 20 min in a vacuum of 10<sup>-9</sup>Pa. 4-Exposure for 2 hours in a vacuum of 10<sup>-9</sup>Pa. 5-Annealing at a temperature of 200 °C. 6-Annealing at a temperature of 600 °C. 7-Annealing at a temperature of 700 °C. 8-Annealing at a temperature of 800 °C.



**Figure 2**

TC spectrum of silicon during ion bombardment ( $\text{Cs}^+$ ) and after annealing 1-Pure silicon with a structure of 7x7, 2-Irradiation with caesium ions at an energy of 2.5 keV and with a dose of  $10^{13}$  ions/cm<sup>2</sup>. 3-Irradiation with caesium ions at a dose of  $10^{14}$  ions/cm<sup>2</sup>. 4-Irradiation with caesium ions at a dose of  $10^{15}$  ions/cm<sup>2</sup>. Annealing at the following temperatures: 5-100 °C, 6-200 °C, 7-300 °C, 8-400 °C, 9-400 °C, 10-400 °C, 11-650 °C, and 12-700 °C.



**Figure 3**

TC spectrum of silicon during ion bombardment ( $\text{Cl}^-$ ) and after annealing 1-Pure silicon with a 7x7 structure. 2-Irradiation with chlorine ions at an energy of 2.5 keV and with a dose of  $10^{13}$  ions/cm<sup>2</sup>. 3-Irradiation with chlorine ions at a dose of  $10^{14}$  ions/cm<sup>2</sup>. 4-Irradiation with chlorine ions at a dose of  $10^{15}$  ions/cm<sup>2</sup>. Annealing at the following temperatures: 5-100 °C, 6-200 °C, 7-300 °C, and 8-400 °C.