Research Square

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Thermally Stimulated Transformation of Oxygen Atoms between Phase States in a Silicon Single Crystal

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Research Article

Keywords: monocrystal, silicon, oxygen, transformation, X-ray diffraction, thermal stimulation

Posted Date: December 11th, 2023

DOI: https://doi.org/10.21203/rs.3.rs-3690667/v1

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Additional Declarations: No competing interests reported.

Version of Record: A version of this preprint was published at Silicon on April 13th, 2024. See the published version at https://doi.org/10.1007/s12633-024-02985-y.

Abstract

The high-temperature X-ray diffraction method showed the presence of two phase states of oxygen in the volume of a single crystal of silicon grown by the Czochralski method: a dissolved (interstitial) state in the composition of “quasimolecules” - SiO_2 and a chemically bound (precipitate) state in the composition of silicon dioxide - SiO_2; the precipitate state of oxygen in the bulk of silicon is in the form of crystalline SiO_2(c), and in the near-surface layer of a single crystal in the amorphous form of SiO_2(a). The thermally stimulated transformation of oxygen atoms between dissolved and precipitate states of oxygen in crystalline form in the bulk of a silicon single crystal has been established.

1. Introduction

High-purity monocrystalline silicon is widely used in various fields of the electronics industry and at the same time is the object of the latest fundamental research and advanced technological developments associated with entering the nanometer range in the production of integrated circuits (ICs) [1, 2]. According to leading organizations producing electronic equipment, silicon single crystals will remain the main material for the production of the elemental base of modern electronics for several more decades [3].

2. Materials and methods

Currently, most silicon single crystals for integrated circuits are produced by the Czochralski method, which is the main industrial method [4, 5]. However, according to the Czochralski method, silicon single crystals are contaminated during the growth process, in addition to metallic ones, by two more background impurities - oxygen and carbon, which determine the impurity phase state of silicon single crystals, due to a number of factors; the magnitude of the concentration and uniformity of distribution throughout the crystal, the formation of its own interstitial atoms, changes in the parameters of the matrix lattice, the formation of precipitates, etc. [5, 6]. Sources of contamination of silicon single crystals with these impurities are the supply of oxygen into the melt at the growth temperature (1688 K) due to the partial dissolution of the quartz crucible and carbon in it - in the form of dust and gaseous oxides, released by parts of the graphite equipment of the melting chamber of the crucible. Therefore, silicon crystals grown using the Czochralski method contain a high concentration of dissolved oxygen $1{\cdot}10^{18}$cm^{-3} ($2{\cdot}10^{-3}$at.%) [7] and carbon – $2{\cdot}10^{16}$ cm^{-3} ($4{\cdot}10^{-5}$ at.%) [8].

Of these, carbon in silicon is a substitutional impurity and does not become electrically active. If the carbon concentration is low, a solid solution of carbon substitution in silicon is formed, and at a high carbon content, the formation of β– SiC with a fluorite structure is possible in the form of precipitates of the impurity phase. The lattice parameter β– SiC (3C-SiC) is equal to $a_{\beta SiC}$ = 4.3596 Å, but the discrepancy between the specific volumes of silicon and its carbide $V_{Si} / V_{\beta\text{-}SiC} \approx 2$, causes the appearance of mechanical stresses in the silicon matrix lattice of sufficient magnitude, affecting the behavior of charge carriers and metal impurities [5, 6, 9, 10].

Oxygen forms a saturated interstitial solid solution in a silicon single crystal, being in the interstitial position (dissolved state) and in this position oxygen is optically active and does not exhibit electrical activity. In the dissolved state, oxygen occupies the position of a bridge between two silicon atoms, forms impurity “quasi-molecules” Si – O – Si, and the axis of the quasi-molecule is oriented along the crystallographic direction < 111>, is somewhat bent, forming an angle of the order of $\phi_{Si\text{-}O\text{-}Si}$ = 150° [5, 6, 10]. However, a high-temperature equilibrium saturated solid solution of oxygen in silicon at low temperatures becomes supersaturated (unstable in concentration), since the solubility of oxygen is proportional to temperature. Therefore, when a single crystal ingot is cooled, a supersaturated solid solution of oxygen in silicon decomposes into dissolved (interstitial) and precipitate (chemically bonded) impurity phases. It is assumed that the precipitate impurity phase is formed in the following way: as a result of the decomposition of a supersaturated solid solution, small oxygen complexes SiO_n (n = 1, 2, 3..,) are first formed with their own silicon atoms, the concentration of which sharply decreases with increasing index n and, that some of them are even electrically active [5, 11]. With further cooling of the ingot, polymerization of the complexes into oxygen precipitates occurs and, ultimately, a stoichiometric impurity crystalline phase is formed from the precipitates in the bulk of the single crystal - SiO_2(c) (silicon dioxide), a crystal lattice, which is conjugated (coherent) with the silicon matrix lattice. According to [5, 6, 12], in Si crystals grown using the Czochralski method, up to 20% of the total oxygen concentration is in the form of precipitate. But, due to the discrepancy between the specific volumes of silicon and its crystalline dioxide V_{SiO2} / $V_{Si} \approx 2$, compressive stresses arise in the silicon matrix lattice (up to $2 \cdot 10^5$ N$\cdot$ cm^{-2}) [13, 14], causing silicon dioxide to become a drain for charge carriers, as well as an internal getter for rapidly diffusing metal impurities that enter the wafer during single crystal growth and during the formation of IC elements.

Consequently, growth single crystals of silicon grown by the Czochralski method are not completely homogeneous in phase terms, but inhomogeneities in the phase state (impurity phases) of silicon crystals can be both SiC and SiO_2(c) particles. Due to the strong chemical bonds of atoms, SiC particles ($E_{Si\text{-}C} \approx 3\ E_{Si\text{-}Si}$), in a fairly wide temperature range up to the melting point of Si, (T_m = 1688 K) do not experience a change in the initial phase state [5, 6, 15]. In particles of crystalline SiO_2(c), on the contrary, in this temperature range, instability is observed in its phase state, despite the rather enhanced chemical bond of atoms ($E_{Si-O} \geq 2E_{Si-Si}$). This phase instability of the SiO_2(c) particle is a prerequisite for the possible transformation of oxygen atoms. Thus, in [16] they proposed the possibility of decomposition of silicon dioxide precipitates and the transfer of oxygen atoms into the dissolved (interstitial) phase state at silicon wafer processing temperatures $T_{ht} \approx T_m^{Si} - 120K$. Indeed, a number of experiments have shown that high-temperature dispersive annealing lasting several hours at a temperature of $\sim$1350° C (1623K) followed by quenching, dissolves existing oxygen precipitates SiO_2(c) in silicon and leads to the distribution of oxygen atoms over interstitial phase positions in the matrix lattice single crystal [5, 17, 18]. However, these works did not consider the possibility of transforming oxygen atoms from a dissolved position back to the precipitate phase state during heat treatment of a silicon wafer, and this question remained open. The results of the above works and IR absorption data by silicon [19] indicate that the

main impurity phase in the bulk of a growing silicon single crystal is stoichiometric crystalline silicon dioxide (SiO_2(c)), and not its carbide (SiC). However, analyzes of some electron diffraction patterns from a silicon single crystal showed that the precipitates of a silicon single crystal are most likely SiC particles [6, 15]. Consequently, despite the fact that there is a lot of experimental data regarding the phase states of oxygen and carbon in silicon, it is not clear what specific impurity phase is formed in a silicon single crystal during growth by the Czochralski method. In this regard, this work is aimed at studying the real impurity phase in the volume of a Czochralski single crystal of silicon, its thermal stability and the possible transformation of any background impurity using high-temperature X-ray diffraction.

3. Results and Discussion

The sample used was single-crystal n-Si type silicon with a resistivity $\rho \approx 10$ Ω•cm, an oxygen concentration $N_O \approx 2 \cdot 10^{17} cm^{-3}$ according to IR absorption data and a dislocation density $N_D \approx 10^2$ cm^{-2} grown by the Czochralski method along the axis growth [111]. The dimensions of the samples were $10 \times 6 \times 1$ mm^3 and the surface of all samples was parallel to the crystallographic plane (111) of the single crystal. X-ray diffraction patterns of the samples were obtained on a DRON-UM1 diffractometer (Russia) using copper X-ray radiation with a wavelength $\lambda_{CuK\alpha}$ = 1,54178 Å with a nickel (Ni) filter. X-ray diffraction patterns of the samples were taken in the angle range from 8 to 73° using the Θ – 2Θ scheme in step scanning mode with a step of 0.01°. To study the thermal stability of the silicon impurity phase and the possible transition of the background impurity from one phase state to another, the X-ray diffraction pattern of the sample was measured in the temperature range 293–1593 K. The required sample temperature – 1593 K, 693 K and 673 K - was set directly on the X-ray beam using a high-temperature attachment of the UVD-2000 type with an accuracy of ± 1°C. After high-temperature heat treatment at 1593 K, the sample was subjected to additional low-temperature annealing at a temperature of 673 K in order to bring it into a thermodynamic equilibrium state. The duration of heat treatment at each temperature was two hours. Additionally, a separate measurement of the X-ray diffraction patterns of the sample subjected to longer annealing (4 hours) at a temperature of 693 K was carried out for a reliable interpretation of the precipitate impurity phase of the Czochralski single crystal of silicon.

In Fig. 1,a shows a complete X-ray diffraction pattern of a plate of the original n-Si silicon single crystal measured at room temperature (T = 293 K) in the angle range 2Θ = 8–101°. In the diffraction pattern at different scattering angles, six selective reflections and one diffuse reflection at small scattering angles are distinguished. Analysis of the X-ray diffraction pattern showed that of the intense narrow structural lines, lines with indices $(222)_{Si}$ at 2Θ = 58,9° (d_{222} = 1,5679 Å) in the middle and $(333)_{Si}$ at 2Θ = 94,93° (d_{333} = 1,0461 Å) at large scattering angles are second and third order, the main diffraction reflection of $(111)_{Si}$ at scattering angles 2Θ = 28.6° (d_{111} = 3.0998 Å) of the silicon matrix lattice. From these three reflections, the lattice parameter of the studied sample was determined using the Nelson–Riley extrapolation function $\{1/2(Cos^2\Theta/Sin\Theta + Cos^2\Theta/\Theta)\}$ [20] and it was a_{Si} = 5.4305 Å, which corresponds to the tabulated silicon data a_{tab} = 5.4307 Å [21]. Somewhat ahead in angles, their β-components $(111)_\beta$ and $(333)_\beta$ are observed at scattering angles 2Θ = 26.5° and 2Θ = 83°, respectively. Due to the low

intensity of the $(222)_{Si}$ reflection, its β- component is not observed in the x-ray diffraction pattern. According to the law of extinction of diffraction reflections, the $(222)_{Si}$ reflection is forbidden for the undistorted lattice of the diamond-like structure of silicon [20]. It appears in the diffraction pattern only in the presence of distortions in the matrix lattice of the growth silicon single crystal, caused by the inhomogeneous distribution of background impurities, preferably oxygen in it, since the oxygen concentration is two orders of magnitude higher than the carbon concentration [7, 8, 22]. This is evidenced by the ratio of the intensity of the forbidden reflection $(222)_{Si}$ to the intensity of the main reflection $(111)_{Si}$ of the silicon matrix lattice, $I(222)_{Si} / I(111)_{Si} = 4.31 \cdot 10^{-3}$, which is significantly greater than 10^{-4}, corresponding to the uniform distribution of oxygen over the interstices of the silicon lattice [23]. These results suggest that the inhomogeneous distribution of oxygen apparently ultimately causes the formation of an impurity phase from precipitation of stoichiometric crystalline silicon dioxide in a highly distorted region of the silicon lattice during cooling of a single crystal ingot. This assumption is confirmed by the presence in the X-ray diffraction pattern of a low-intensity but selective reflection at relatively small scattering angles of silicon that does not belong to the matrix lattice. Analysis showed that this diffraction reflection at scattering angles 2Θ = 36°(d_{110} = 2.4968 Å) with the Muller index $(110)_{SiO2}$ is caused by the crystalline impurity phase SiO_2(c) (Fig. 1,a). The structure of crystalline silicon dioxide SiO_2(c) at room temperature is known; it is trigonal and has lattice parameters – a_{tab} = 4.9043 Å, c_{tab} = 5.3974 Å [21]. Calculations carried out based on the experimental angular position of the $(110)_{SiO2}$ reflection gave the lattice parameter a = 4.9894 Å, which is a slightly higher value compared to the tabulated value. Using the Scherrer formula ($D = K \cdot \lambda / \Delta(2\theta) \cdot \mathrm{Cos}\theta$, where K is the Scherrer coefficient) from the half-width (FWHM = $4{,}07 \cdot 10^{-3}$ rad) of this reflection, the effective size of silicon dioxide crystallites was determined equal to L ≈ 36 nm. Consequently, despite the presence in the diffraction pattern of the initial sample with a noticeable intensity of the forbidden reflection $(222)_{Si}$, indicating the presence of distortion in the silicon matrix lattice and the presence of the $(110)_{SiO2}$ structural line of the impurity phase, the narrow width and high intensity of the main reflection $(111)_{Si}$ and its third order $(333)_{Si}$, as well as the low level and monotonicity of the inelastic background, indicate the high perfection of the crystal structure of the studied growth single crystal of silicon.

The low-intensity wide diffuse maximum at small scattering angles, 2Θ ≈ 17,2° (d_{dif} ≈ 5,21 Å), is most likely caused by the surface amorphous phase of silicon dioxide SiO_2(a), since the plates are in an oxidizing environment (atmosphere) and therefore the amorphous phase is always present in all initial plates of a single crystal [6]. In shape, diffuse scattering corresponds to short-range order in the arrangement of silicon and oxygen atoms in the surface layer of a single crystal, that is, each silicon atom tends to surround itself with nearby oxygen atoms (Fig. 1,b).

However, in the X-ray diffraction pattern of silicon (Fig. 1,a) from the impurity phase there is a single structural reflection $(110)_{SiO2}$ at a scattering angle of 2Θ = 36°. Based on single structural lines, it is not correct to say that it belongs to certain impurity phases, since there are always possible alternatives that require further research. In this case, it is possible that structural lines belonging to different particles with

different chemical components may overlap. For example, in a single crystal of silicon, on a given structural line from a particle of crystalline SiO_2, it is possible, according to the angular position, to overlap some structural line from a SiC particle. The analysis of the possibility of such a situation showed that on the $(110)_{SiO2}$ structure line at 2Θ = 36.0° (d_{110} = 2.4968 Å) from the SiO_2 particle, the $(004)_{SiC}$ structure line at 2Θ = 35.93° (d_{004} = 2.508 Å) is completely superimposed in angular position from a SiC particle of the 4H–SiC polytype (hexagonal structure, lattice parameters; a_{tab} = 3.0730 Å, b_{tab} = 10.0530 Å [21]). This situation requires that in order to unambiguously determine the real impurity phase of a growing silicon single crystal, at least one more selective structural line from one or another supposed impurity phase is necessary.

It was experimentally established that during long-term low-temperature heat treatment of a silicon wafer at about 400–450°C (673–723 K), the processes of generation of interstitial silicon atoms and precipitation of a solid solution of oxygen into the second precipitate phase occur [24, 25]. Since silicon single crystals grown by the Czochralski method are rich in oxygen, it is appropriate to assume that the second precipitate phase is a heterogeneous defect consisting of oxygen atoms and an intrinsic silicon atom, which leads to a decrease in the level of thermal stresses caused by the temperature gradient in the sample. To test this assumption, according to the results of [25], a separate wafer of n–Si silicon growth single crystal was subjected to a longer low-temperature annealing (4 hours) at a temperature of 693 K and its X-ray diffraction pattern is shown in Fig. 1,c. A comparison of the X-ray diffraction patterns of the original sample (Fig. 1,a) and the annealed one (Fig. 1,c) showed that they differ from each other only with the appearance on the diffraction spectrum of the annealed sample of an additional selective reflection with noticeable intensity at small scattering angles 2Θ = 26, 71° (d_{101} = 3.338 Å). Analysis of the nature of this reflection showed that it is also due to particles of crystalline silicon dioxide, but with a different orientation – $(101)_{SiO2}$ relative to the normal to the surface of the plate. Calculations carried out based on the experimental angular position of the $(101)_{SiO2}$ reflection gave the lattice parameter c = 5.2631 Å, which is a slightly underestimated value compared to the tabulated one c_{tab} = 5,3974 Å [21]. In addition, they showed that near the location of the $(101)_{SiO2}$ reflection, with the exception of $(111)_{\beta}$ at 2Θ = 25.90°, there are no structural lines from particles of silicon carbide polytypes 3C-SiC or 4H-SiC. This indicates that low-temperature long-term annealing of the wafer caused an increase in the content of oxygen precipitates in the volume of silicon along the < 101 > crystallographic direction; the additional amount of oxygen and its own silicon atoms was apparently carried out by the process of absorption of oxygen from the external environment through the oxide layer on the surface and due to the partial departure of oxygen atoms from the interstitial position. This assumption is consistent with the data of [6, 26], where a decrease in the content of interstitial oxygen was observed during low-temperature annealing of silicon.

Consequently, as a result of low-temperature annealing at 693 K, on the diffraction spectrum of a silicon wafer with a (111) orientation, there is a clear presence of two selective reflections $(101)_{SiO2}$ and $(110)_{SiO2}$ with significant intensity at scattering angles 2Θ = 26.71° and 2Θ = 36.0°, due to particles of crystalline silicon dioxide of different orientation in the bulk of the single crystal clearly indicates that the real

impurity phase of a growth single crystal of silicon grown by the Czochralski method is indeed crystalline silicon dioxide SiO_2, and not its carbide SiC. Since the simultaneous superposition of two selective reflections from a particle with different chemical components is very unlikely. This conclusion is consistent with the result of work on α-β – transitions in the SiO_2 impurity phase of a Si single crystal [27]. It should be noted that all these processes associated with the behavior of oxygen depending on temperature apparently occur in the near-surface layers of the volume of a silicon single crystal, where the SiO_2 – Si phase boundary is formed.

Thus, in the X-ray diffraction spectrum of a growth silicon single crystal with a (111) cut, in addition to diffraction reflections of the (HHH) type of different orders from the silicon matrix lattice, structural reflections reliably appear with sufficient intensity, corresponding to the precipitate state of oxygen in silicon, in the form of its dioxide - SiO_2: in the bulk in crystalline form - SiO_2(c), and in the near-surface layer of a single crystal in amorphous form - SiO_2(a); and the atomically dispersed phase state of oxygen in silicon is best determined by IR spectroscopy, since in diffraction studies this phase state of oxygen affects only the level of inelastic background, the change of which is only a few percent, which is not enough to identify the phase being determined. This conclusion is consistent with the results of works [17, 24].

In Fig. 2a shows the diffraction spectrum of the original sample at a temperature of 293K measured in the angle range 2Θ = 8–73°, which corresponds to the X-ray diffraction pattern shown in Fig. 1,a, without the high-angle sections. The X-ray diffraction pattern does not show the structural reflection $(101)_{SiO2}$ at 2Θ = 26.71°, since the sample was not subjected to long-term low-temperature annealing. Therefore, the dissolution of the bulk impurity phase SiO_2(c) of a silicon wafer during high-temperature heat treatment was studied by changes in the intensity of the $(110)_{SiO2}$ structural reflection from this phase at scattering angles 2Θ = 36°. After annealing for 2 hours at a high temperature of 1593K, the following anomalies are observed in the diffraction pattern of the sample: complete disappearance of diffuse scattering from amorphous silicon dioxide – SiO_2(*a*) at 2Θ ≈ 17.2°, a noticeable increase (6%) in the average level of the inelastic background and the background acquiring a monotonic character throughout the entire angular range, a noticeable ∼12% increase in the intensity of the main structural line (111) from the silicon matrix lattice, a significant ∼ 80% loss of intensity of its second order $(222)_{Si}$ at 2Θ ≈ 59° and a decrease of more than 90% in the intensity of the selective reflection $(110)_{SiO2}$ at 2Θ ≈ 36°from a crystalline silicon dioxide particle SiO_2(c) (Fig. 2,b). These listed effects indicate the transformation of oxygen atoms from a chemically bound state to a dissolved one, that is, a transition to a phase corresponding to a high temperature of a saturated solid solution of oxygen in silicon with a more evenly distributed matrix lattice interstices. Such a solid solution usually becomes supersaturated upon cooling or at low temperatures. Therefore, when cooled to room temperature, it should decompose into two phases; a phase containing little dissolved oxygen – SiO_2 (oxygen-depleted phase) and a precipitate (crystalline) phase – SiO_2 (oxygen-rich phase).

Thus, the X-ray diffraction pattern of a slowly cooled sample with a stove from a temperature of 1593 K to room temperature 293 K is shown in Fig. 2,c. In general, the x-ray diffraction pattern of the sample cooled to room temperature is similar to the x-ray diffraction pattern of the original sample with minor differences. The main difference is observed in the intensity of diffuse reflection at $2\Theta \approx 17{,}2°$ from a particle of the amorphous SiO_2(a) phase of a silicon crystal. In Fig. 2c only traces of diffuse reflection are observed at $2\Theta \approx 17.2°$. This shows that the formation of amorphous silicon dioxide on the surface layers of a single crystal plate by natural oxidation requires more time than the cooling time of the crystal from high temperatures to room temperature. To speed up the oxidation processes of the sample, it was subjected to additional low-temperature annealing at a temperature of 673 K for two hours. After such annealing, the intensity of the diffraction reflections of the impurity phase corresponded to the intensity of the same structural lines of the impurity phase of the initial plate. The intensity of the main reflection (111) of the silicon matrix lattice was also equal to the intensity of the same structural line of the original sample, as well as the level of the inelastic background. Consequently, at low temperatures, as a result of the decomposition of a saturated solid solution of oxygen at high temperatures, particles of stoichiometric silicon dioxide are again released in the crystalline phase (SiO_2)(c) in the bulk and in the amorous phase (SiO_2)(a) on the surface layers of the silicon single crystal.

Conclusions

Thus, the high-temperature X-ray diffraction method has established that the real impurity phase of a silicon single crystal grown by the Czochralski method is not SiC, but stoichiometric SiO_2; in the bulk in crystalline form - (SiO_2)(c) and in the surface layers in amorphous form - (SiO_2)(a). The presence of two phase states of oxygen in the bulk of silicon is shown: a dissolved (interstitial) state in the composition of “quasimolecules” - SiO_2 and a chemically bound (precipitate) state in the composition of crystalline silicon dioxide - SiO_2(c). The thermally stimulated transformation of oxygen atoms between dissolved and precipitated states in crystalline form (SiO_2)(c) in the bulk of a silicon single crystal has been established.

Declarations

Ethics Approval All author declared that the entire ethical standard required for the preparation and publication are complied.

Consent to Participate All person named as author in this manuscript have participated in the planning, design and performance of the research

Consent for Publication All authors have indorsed the publication of this research.

Availability of data and materials All data generated or analyzed during this study are included in this published article.

Competing Interests The authors declare no competing interests.

Funding The Fundamental Research Programs of the Uzbekistan Academy of Sciences financially supported this work.

Author Contributions M.U. Kalanov: Writing-review & editing, I.I. Sadikov: Writing-original draft, A.V. Khugaev: Validation, Data curation, Sh.R.Malikov: Project administration, A.S. Saidov: Funding acquisition. Sh.N. Usmonov: Supervision. D.V.Saparov: carried out the experiment.

Acknowledgments The authors are sincerely grateful to B.S. Yuldashev for constant attention and support at all stages of this scientific research.

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Figures

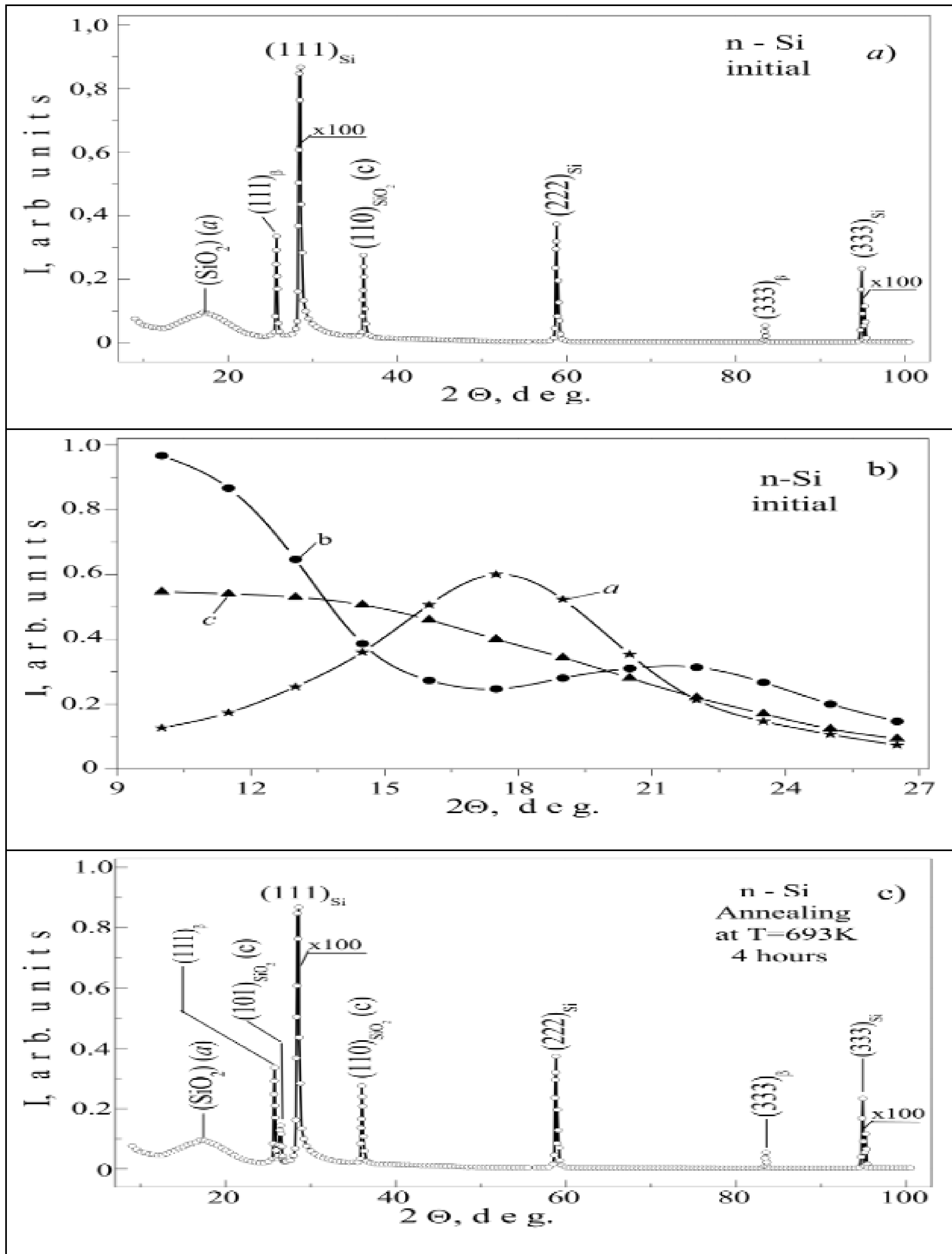

Figure 1

a) complete X-ray diffraction pattern of a growth single crystal n-Si; – b) diffuse scattering of X-rays (l_{CuKa} = 1.54178 Å) from the surface layer of the initial n-Si: «) short-range order; ●) near stratification; ▲) near disorder; – c) X-ray diffraction pattern of annealed n-Si at T = 693 K for 4 hours.

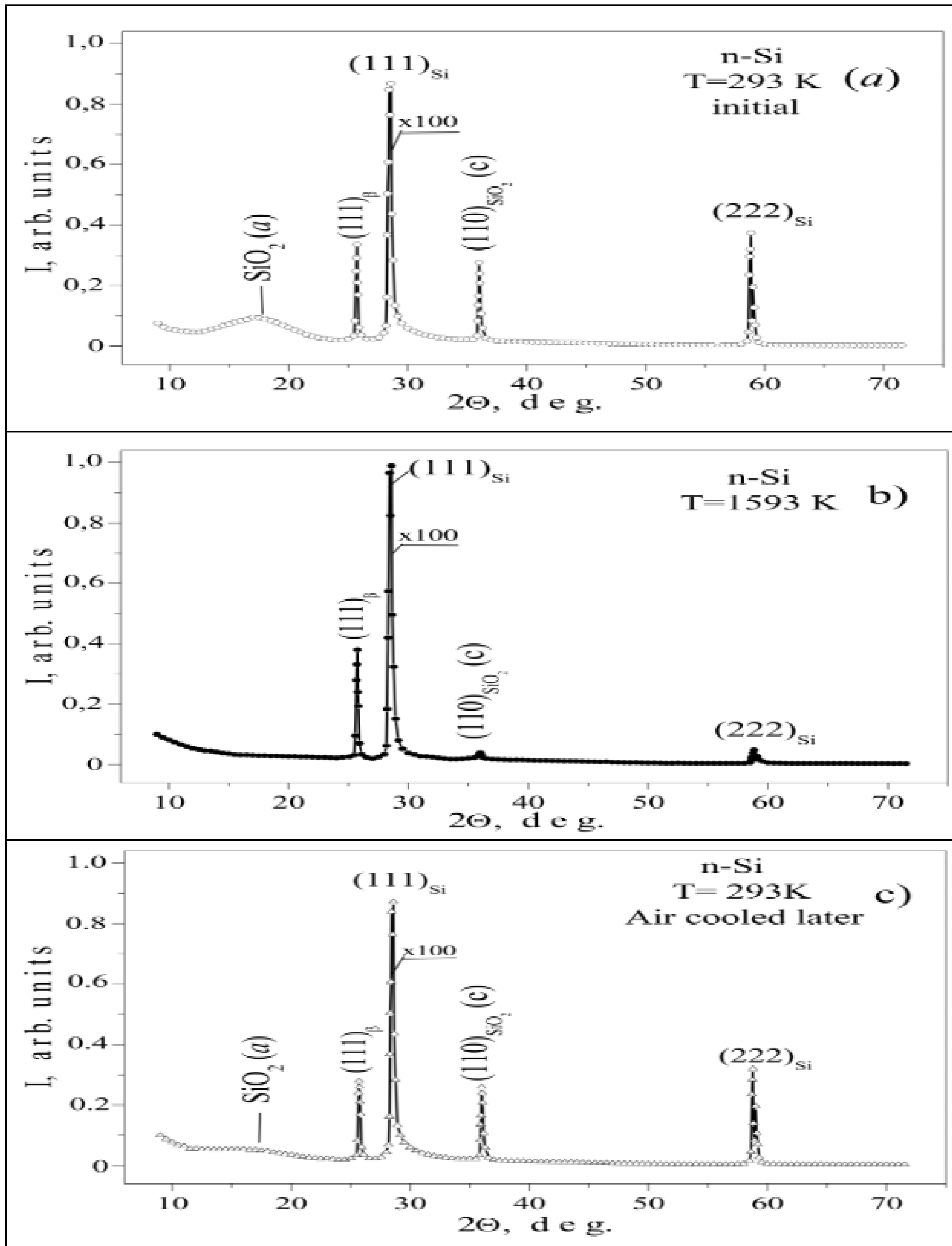

Figure 2

Diffractograms of single crystal silicon n - Si at different temperatures: – a) at T = 293 K; – b) at T = 1593 K; – c) at T = 293 K, after cooling the sample in air.