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New pecularities of the conductivity and photoconductivity mechanisms in β-rhombohedral boron

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Abstract

The development of the steady-state dc conductivity of β-rhombohedral boron requires large temperature-dependent time constants (for example $\tau \sim 5$ h at 425 K) and exhibits stochastic precipitous jumps, whose height depends on temperature as well. At low temperatures, these jumps are attributed to trapping and releasing of electrons. At high temperatures, considering the unsteady changes of numerous physical properties between 500 and 600 K, the results are explained by the diffusion of interstitial B atoms causing statistical fluctuation of conductivity paths below the percolation threshold. The time-dependent photoconductivity measured between 88 and 576 K for times up to 45 h is strongly influenced by traps reducing the lifetime of the initially generated electrons considerably. This changes, when the trap occupation increases because of persisting optical excitation. Though under final stationary conditions the concentration of conducting electrons is independent of time, the conductivity persists to change; depending on temperature it increases or decreases. This is probably due to the diffusion of interstitial B atoms as well.

Keywords: Boron; Electrical conductivity; Photoconductivity; Percolation; Traps; Interstitial diffusion

1. Introduction

β-Rhombohedral boron is one of the best-investigated boron-rich solids [1]. Its advanced energy band scheme [2,3] (improved version see Fig. 12) allows consistently describing most of the experimental results hitherto reported. In the gap of about 1.5 eV, the level with localized states at about 0.19 eV above the valence band edge has been attributed to vacancies of the B(13) sites [4,2] (B(13) = six crystallographically equivalent boron sites neighbored to the single B(15) atom in the center of the unit cell [5,6]; measured occupation densities 73.4(15) [7], 74.5(6)% [8]). Moreover, there exists a series of six electron traps in distances of multiples of 0.19 eV off the conduction band, which were attributed to interactions between electrons and specific intra-icosahedral phonons [9,10], and additionally a shallow trap 16(2) meV off the conduction band.

Because of the very high capture cross section of the traps, in thermal equilibrium the contribution of free electrons to the conductivity is generally very small. The charge transport is essentially due to hopping processes in the localized gap states above the valence band and a certain contribution of free holes impeded by the occupied localized states acting as hole traps. Depending on the specific conditions of measurement, hopping or free-hole transport can prevail (see [1]).

Nevertheless, the problems of the correlation between electronic properties and structure, in particular the influence of structural details, which seems to be much more effective than in classical semiconductors with simple periodic structures, have largely remained unsolved. Some principles of the correlation between photoluminescence and photoconductivity of β-rhombohedral boron have been shown in Ref. [2]. The spectral photoconductivity and its dependence of the modulation frequency [3] yielded insight into the kinetics of the charge transport. Below, the course of the onset process of the photoconductivity is investigated in some detail. It is superimposed by a long-term process, which is independent of the optical excitation, and hence attributed to the influence of slow structural modifications.

Between 500 and 600 K, the temperature dependence of the electrical conductivity exhibits a distinct step

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accompanied by a hysteresis of the thermal expansion, a maximum of the internal friction and a minimum of the ESR line width (see Refs. [1,10] and references therein). Accordingly, a correlation between the electronic transport mechanism and structural modifications seems evident.

2. Sample

The sample ($5 \times 7 \times 0.090\,mm^3$) was prepared from single-crystal boron (Wacker, Munich), claimed purity 99.9999%, except of carbon (typically 30–80 ppm). The crystallographic *c*-axis was parallel to the surface, and the current path approximately parallel to the *c*-axis. After cut from the rod, the sample was mechanically polished on both sides with diamond spray (Struers) of gradually decreasing grain size (final 1 μm) and then chemically etched [11] to remove the Beilby layers. For probes, Pt wires of 0.1 mm thickness were adhered by a capacitor discharge forming ohmic contacts of a low-melting Pt–B eutectic. By comparing the resistance of the same sample obtained by four- and two-probe methods, it was proved that the contact resistance is negligibly small compared with that of the sample. Therefore, since only relative measurements were performed, the two-probe method was sufficient.

3. Experimental procedure

The measurements were performed in two groups, from 88 to 425 K and from 445 to 576 K, The experimental setup remained unchanged, in particular within each group, and therefore the results can be immediately compared to one another.

It is well known that the electrical conductivity of boron strongly depends on the antecedent. To realize defined starting conditions, before each measurement the sample was kept at room temperature in complete darkness for at least 12 h. The adjustment of the temperature of measurement required 15–60 min. Irrespectively of the time of cooling, at any temperature the measurement was started 60 min after the final temperature was reached.

The strong influence of the antecedent is demonstrated in Fig. 1, where the procedure described is compared with a well defined different one (Fig. 1).

The sample was in vacuum of about 10^{-4} Pa. For optical excitation a GaAlAs laser diode (Spectra Diode Labs, type SDL-2431-G1; maximum optical power, 500 mW at 816 nm (1.52 eV)) with a narrow, approximately parallel light beam was used. This was screened from the sample by a shade with a monitoring photodetector in its center. When the shade is removed at beginning optical excitation ($t = 0$), the detector signal breaks down, thus giving the starting signal for data recording (see Fig. 7).

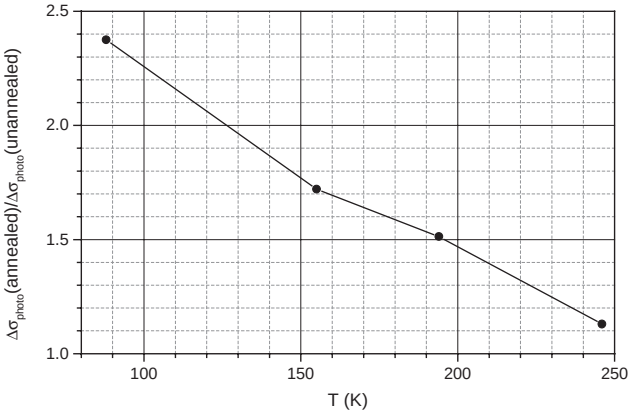

Fig. 1. Relation of the photoconductance of annealed and unannealed β-rhombohedral boron. After having been at room temperature for at least 12 h as usual, the annealed sample was kept at 405 K for 1 h and then cooled down to the temperature of measurement with about 0.2 K/s.

4. Experimental results

4.1. Dark conductivity

As is well known, between 84 and 425 K the temperature dependence of the dark electrical conductance of β-rhombohedral boron exactly follows Mott's law of variable-range hopping (see Ref. [1]).

4.1.1. Time dependence of the dc electrical conductivity

After the drift voltage is switched on, the electrical conductance varies depending on time. As an example, in Fig. 2 the time dependence at 405 K is displayed for 45 h. At that temperature the steady-state value is approached with a time constant of about 5 h. To avoid the extremely long times of measurement, which would have been necessary to precisely determine the temperature-dependent time constants, an approximate method was used: Depending on temperature, the slope $1/\Omega\,s$ of a linear fit to the experimental data within a starting time interval of up to 5 h was determined. The results are displayed in Fig. 3. Such long relaxation times at rather high temperatures make an explanation by pure electronic relaxations improbable.

4.1.2. Stochastic precipitous jumps of the electrical conductivity

Irrespective of noise; the time-dependent conductivity measurements exhibit stochastic precipitous jumps (Fig. 4). Such jumps occur in some electrical conductivity results of boron presented in literature as well. Obviously, till now they were uncritically attributed to noise and therefore not further discussed. By painstaking checks of

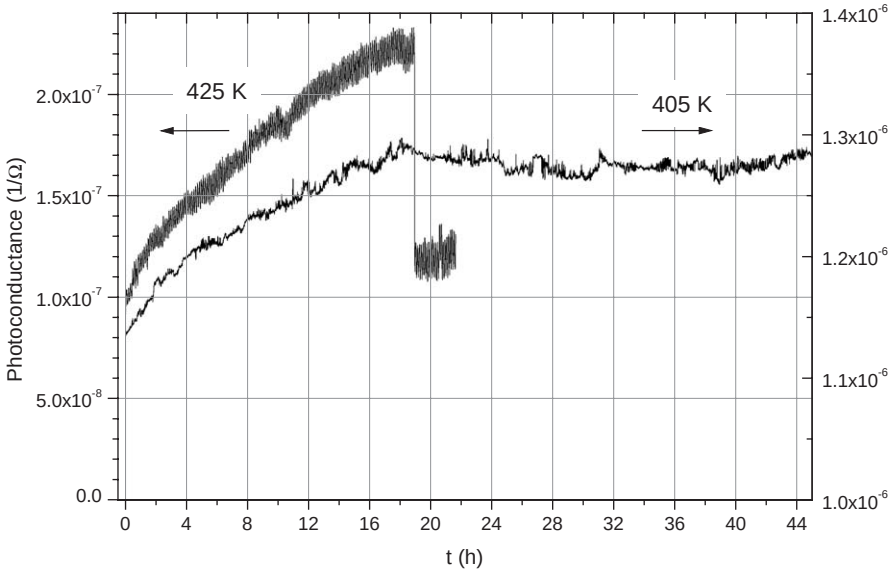

Fig. 2. Dependence of the photoconductance of β-rhombohedral boron for 22 h at 425 K and 45 h at 405 K.

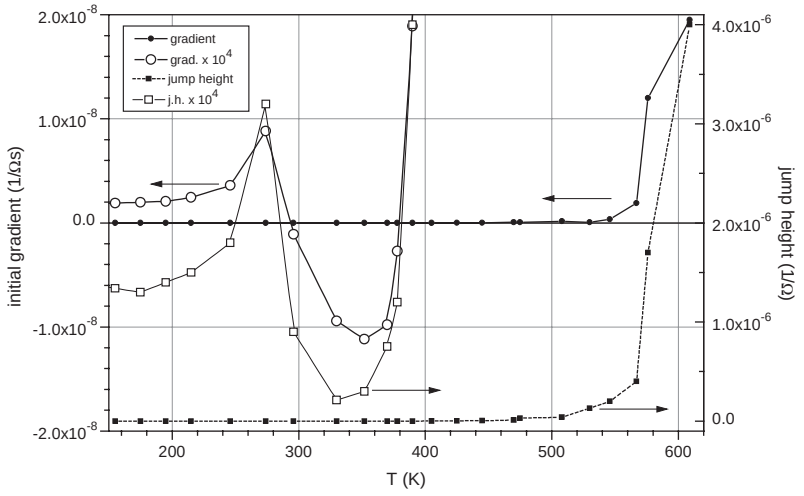

Fig. 3. Initial gradient of the long-term time-dependent electrical conductance (•) and maximum height of stochastic precipitous jumps (■) vs. temperature. For better resolution of the values at low temperatures, the data multiplied with 10^4 are additionally displayed (○,□).

our experimental setup it was proved that these jumps are caused neither by external influences, nor by any compound of the experimental setup but by the boron sample itself. The ohmic contacts are ruled out as well because their resistance is negligible compared with the sample resistance. When the sample is replaced by a comparable ohmic resistor, the stochastic precipitous jumps do not occur. Moreover, the occurrence was verified on other boron samples in different setups. The temperature-dependent maximum values of the height of these stochastic jumps are included in Fig. 4.

The obvious correlation of the time-dependent long-term change of the electrical conductivity and of the stochastic precipitous jumps in Fig. 3 is underlined by the agreement of the thermal activation energies (Fig. 5).

4.2. Photoconductivity

At low temperatures the photoconductivity exceeds the dark conductivity by nine orders of magnitude (Fig. 6). Obviously, there are significantly different mechanisms at lower and higher temperatures. Within the temperature range investigated, the condition of weak optical excitation, characterized by $\Delta I_{\mathrm{photo}} \ll I_{\mathrm{dark}}$, which is the basis of the theory of Simmons and Taylor [12] on the photoconductivity in p-type amorphous semiconductors with narrow trapping levels in the band gap is not fulfilled. Obviously, for the application of this theory on the photoconductivity in β-rhombohedral boron distinctly higher temperatures are required.

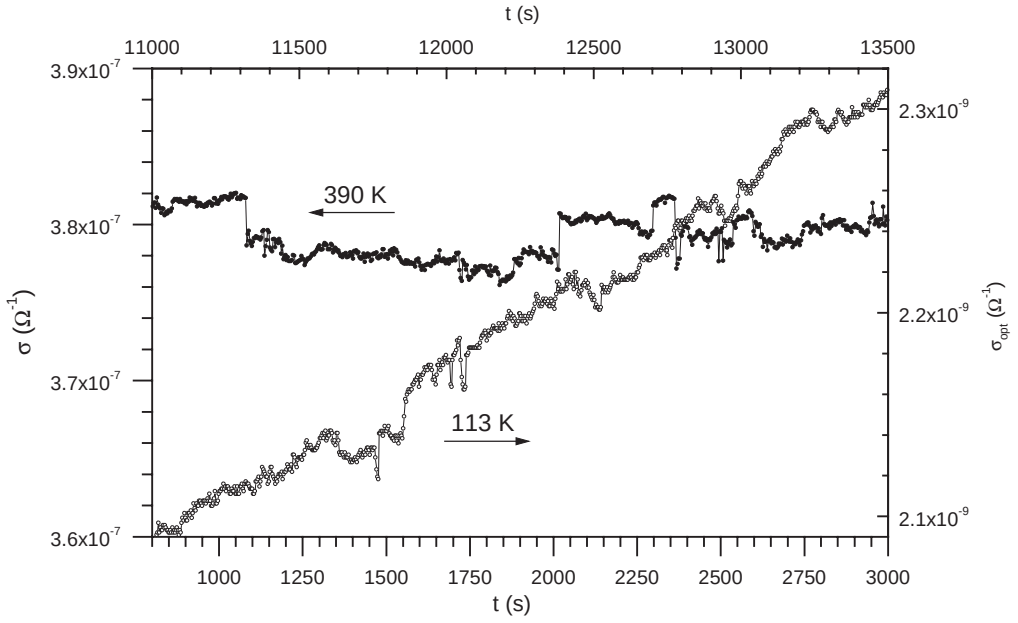

Fig. 4. Sections of the photoconductance at 113 K and the conductance at 425 K vs. time. Examples for the stochastic precipitous jumps in the electrical conductivity at low and high temperatures.

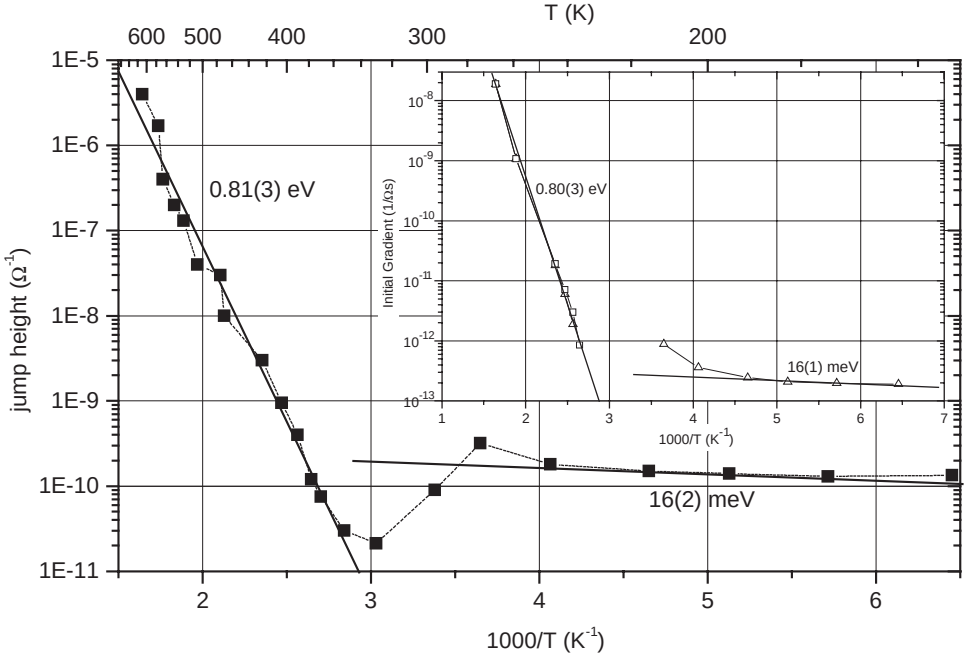

Fig. 5. Maximum jump height of the electrical conductivity vs. reciprocal temperature. Inset: Initial gradient of the long-term variation of the electrical conductivity (•, positive values; ■, values in relation to the minimum at 350 K) vs. reciprocal temperature. Activation energies determined attached to the fitted lines.

The typical time dependence of the photoconductance of β-rhombohedral boron is displayed in Fig. 7. A steep and considerable increase of the conductance after the inset of the optical excitation is followed by a weak steady variation, whose increasing or decreasing tendency depends on temperature. As an example, for 405 K is shown in Fig. 3 that this long-time variation is independent of the illumination because the step-height is exactly the same without and with preceding optical excitation. This was definitely proved by a separate long-term experiment in complete darkness under otherwise same conditions. The weak periodical variations are evoked by the unavoidable temperature variations of about 0.1 K due to the automatic temperature regulation of the experimental setup.

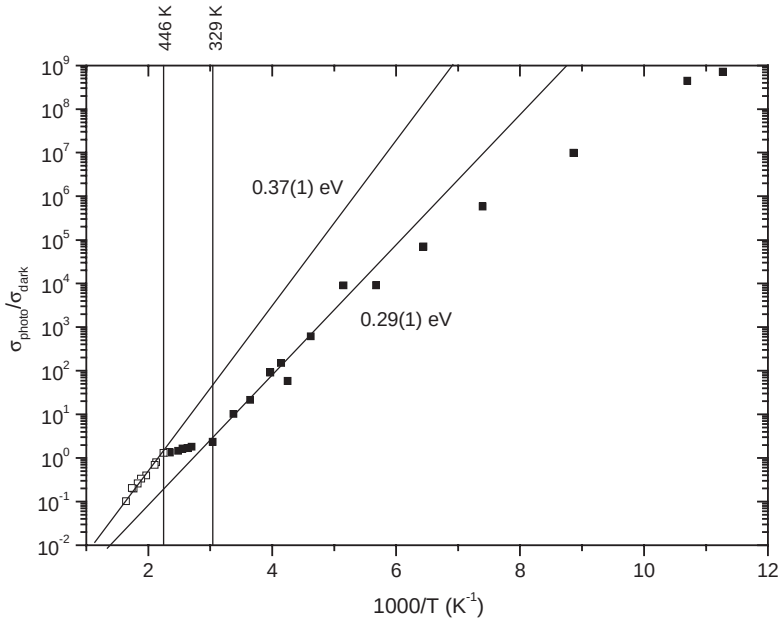

Fig. 6. Relation $\Delta\sigma_{photo}/\sigma_{dark}$ vs. reciprocal temperature. A possible reason for the deviation from the straight line at low temperatures is that the waiting time of 1 h was not sufficient for developing thermal equilibrium. Accordingly, σ_{dark} may be enhanced by frozen-in carriers.

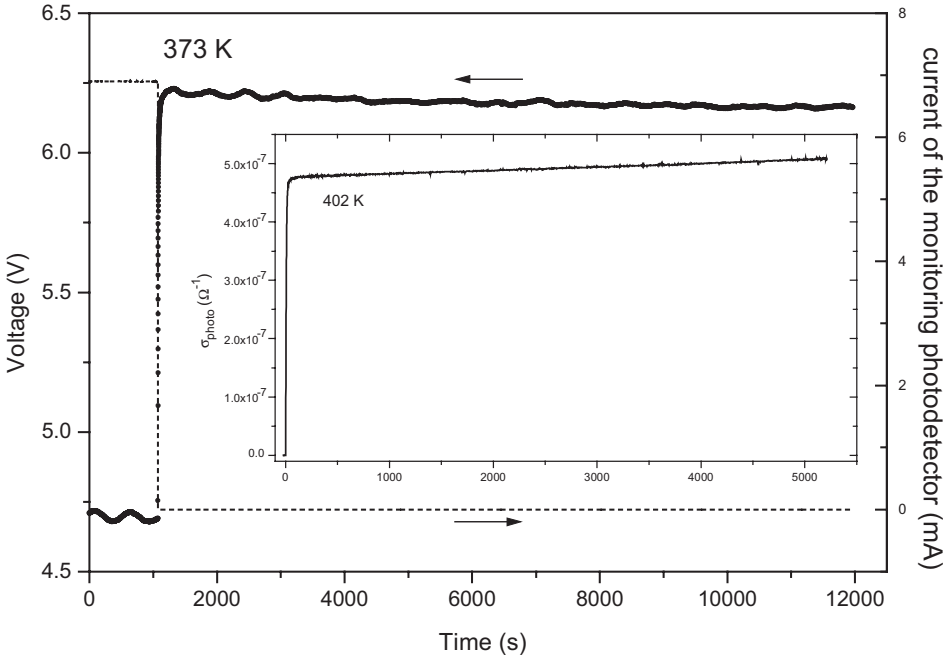

Fig. 7. Typical time-dependent photoconduction of β-rhombohedral boron. $T = 373$ K, long-term variation decreasing. Left ordinate: Voltage at the reference resistor (3888 MΩ); right ordinate: electrical current of the monitor diode determining the starting point of optical excitation and of data recording. The weak periodical current variations are due to the unavoidable temperature variations of about 0.1 K by the automatic regulation and can be removed by calculation using the simultaneously measured sample temperature. Inset: $T = 402$ K. σ_{photo} vs. time. Long-term variation increasing. Temperature-dependent variations removed.

For the whole temperature range investigated, i.e., between 88 and 576 K, the time dependence of the onset of the photoconductance is displayed in Fig. 8.

Before the measured photoconductance data were evaluated, they were largely corrected with respect to long-term changes, temperature variations and stochastic jumps. In Fig. 9 the steady-state values of the photoconductance after sufficiently long optical excitation are plotted vs. reciprocal temperature yielding at least formally the activation energies 17(2) meV at low and

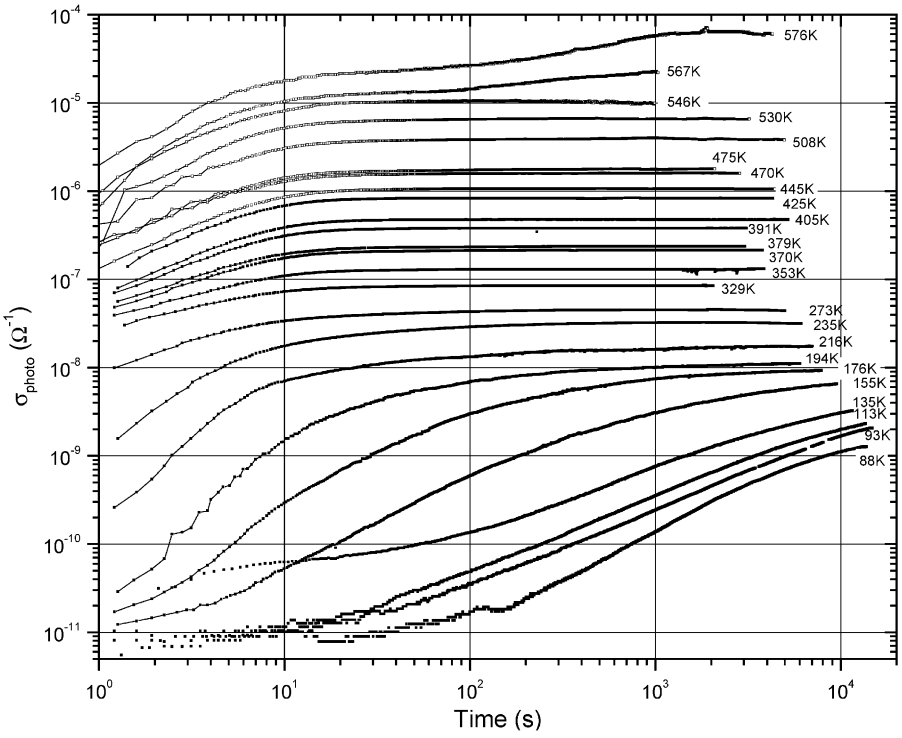

Fig. 8. Time dependence of the onset of the photocondutance of β-rhombohedral boron for various temperatures between 88 and 576 K. The time $t=0$ is the onset of the exciting illumination. The curve at 135 K is falsified at times below about $T\sim 100$ s, possibly because of an accidental weak illumination of the sample.

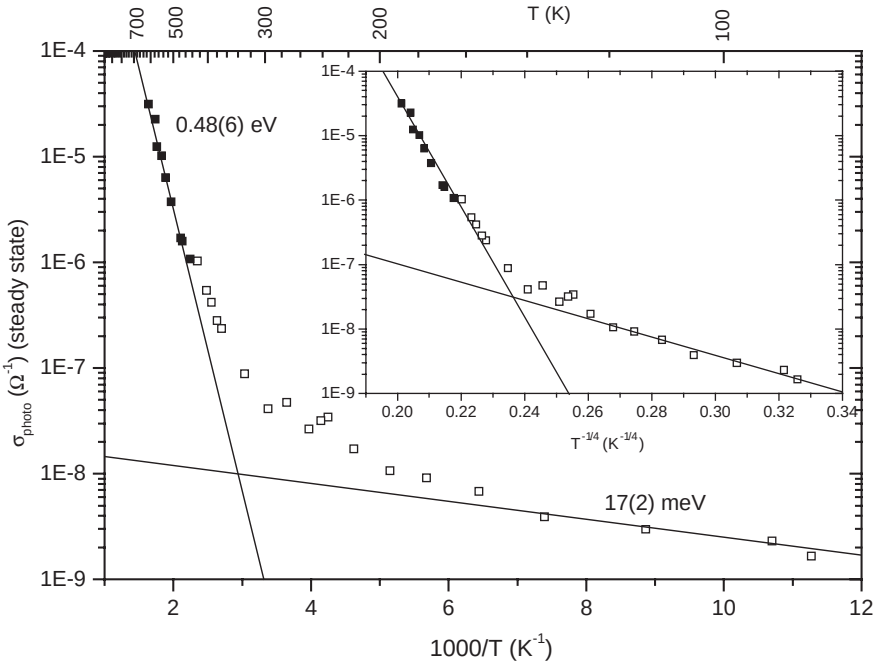

Fig. 9. Steady-state photoconduction plotted vs. reciprocal temperature. Inset: Plot vs. $T^{-1/4}$ (Mott's law of variable-range hopping).

0.48(6) eV at high temperatures. The former value can be easily identified as the activation energy of the electron mobility (0.017(2) eV [3]). However, as shown in the inset to Fig. 9, the fit to Mott's law of variable-range hopping is satisfactory as well, probably even better. Anyway, there seem to be two different characteristic transport mechanisms of the photoconductivity in β-rhombohedral boron at low and at high temperatures, respectively.

It is well known that the free electron transport in β-rhombohedral boron is strongly impaired by the influence of different traps. It is expected that the influence of each trap on the onset of the photoconduction evokes a specific time constant, which is related to the ionization energy of the trap. Accordingly, the onset of the photoconduction in the case of n traps should be describable by the equation

$$I_{\text{photo}} = I_{\text{saturation}}\left(1 - \sum_{i=1}^{n} g_i e^{-t/\tau_i}\right) \quad (1)$$

(n, number of trap; g_i, relative contribution of electrons released from trap i to $I_{\text{saturation}}$; τ_i, time constant of trap i). The extremely long relaxation times did not allow measuring the values $I_{\text{saturation}}$ for temperatures <176 K. They were approximately determined by an exponential fit to the experimentally obtained data. The onset curves (Fig. 8) were decomposed step by step according to Eq. (1). The obtained time constants τ_i are plotted vs. reciprocal temperature in Fig 10. The chosen symbols indicate, that the time constants obtained at different temperatures can easily be attributed to one another allowing determining their thermal activation energies (Table 1).

The good agreement with the ionization energies of the traps obtained from spectral-dependent photoconductivity [3] and other experiments (see [1]) confirms that the onset of the photoconduction is due to electron trapping processes and subsequent filling of the traps. At least below 250 K, this agrees with the spectral photoconductivity showing that the photoconductivity essentially results from the excitation of trapped electron [3].

At temperatures below about 250 K, the described procedure did not allow one to include the considerable delay between the onset of the photoconduction

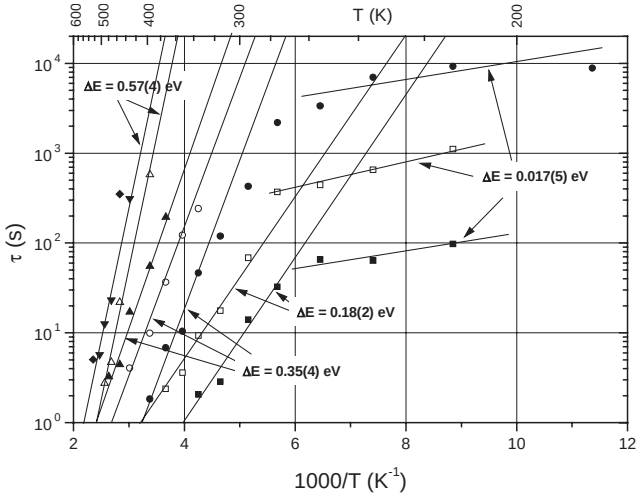

Fig. 10. Time constants of the onset of the photoconductivity obtained from the decomposition of the data measured at well-defined temperatures (see Fig. 4) according to Eq. (1). The symbols indicate the time constants at different temperatures belonging to one another.

Table 1
Thermal activation energies of the time constants derived from the onset of the photoconductivity in β-rhombohedral boron compared with results of spectral photoconductivity

	τ_i activation energy (eV)	Spectral photoconductivity [3] (eV)	Description
1	0.017(5)	0.016(2)	Shallow trap/activation energy of the electron mobility
2	0.18(2)	0.19(2)	Electron trap No. 1
3	0.35(4)	0.38(3)	Electron trap No. 2
4	0.57(4)	0.58(2)	Electron trap No. 3

(see Fig. 8) compared with $t = 0$, the start of the optical excitation. To analyze this range, the difference between the measured data and the best fit obtained after the decomposition according to Eq. (1) was determined (for illustration see inset to Fig. 11). The obtained time constants are displayed in Fig. 11. Their activation energies are 16(4) meV, which can be attributed to the activation energy of shallow traps or electron mobility, and 0.12(2) eV, which is the universal activation energy for the cascade-like recombination of trapped electrons into the valence band range [2,3].

4.3. Discussion

Taking the actual energy band scheme (Fig. 12) and the results of modulated photoconductivity [3] into account and considering the numerous anomalies of physical properties between 500 and 600 K (see Ref. [10]), we interpret these results as follows:

At low temperatures, in both cases the thermal activation energy is that of the shallow traps (16(2) meV). Obviously, the jumps in the conductivity indicate events of capturing and release of conducting electrons, respectively. At room temperature and above, the charge transport in thermal equilibrium can be described by the superposition of hopping processes within the partly occupied localized states at about 0.19 eV above the valence band edge and by free holes, which are generated by the thermal activation of valence electrons into the unoccupied states of this level. The movement of the free holes is strongly impeded by capture processes in the occupied localized states of this level acting as hole traps (see Ref. [1]). After Ref, [2], this level, which is assumed to essentially originate from B(13) vacancies [4], should be separated into two ones, whose energies slightly vary depending on whether they are occupied or not. If the maximum of internal friction between 500 and 600 K is attributed to the diffusion of B(16) to B(20) interstitial B atoms into the vacant B(13) sites or vice versa [3], the step in the temperature dependence of the dc conductivity (see Refs. [1,10]) can be easily explained for both types of possible transport,

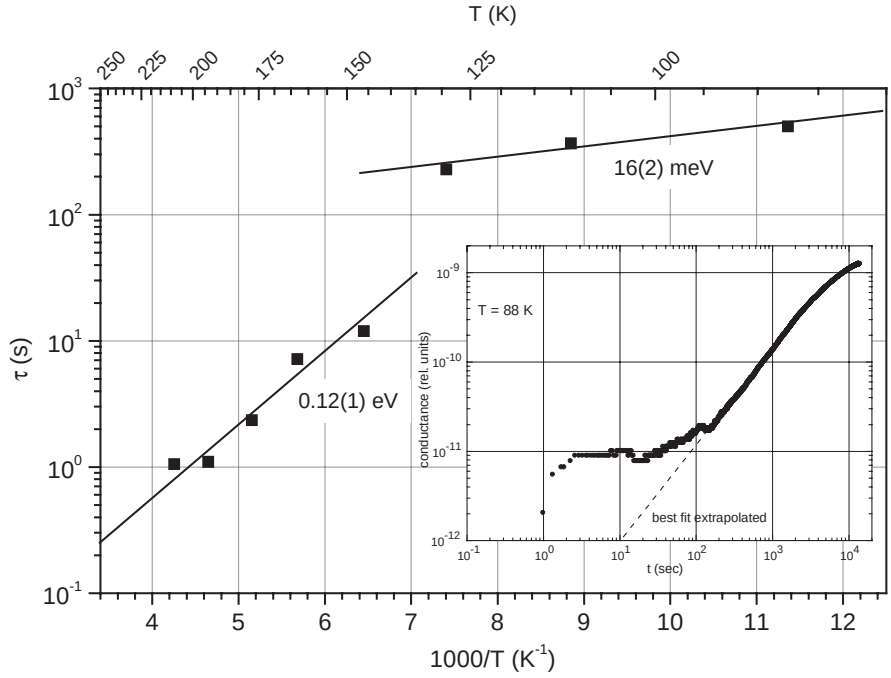

Fig. 11. Time constants of the initial delay of the photoconduction at lower temperatures vs. reciprocal temperature. This initial delay is the difference between the data measured (inset, example for 88 K) and the extrapolation of the best fit to the long-time data.

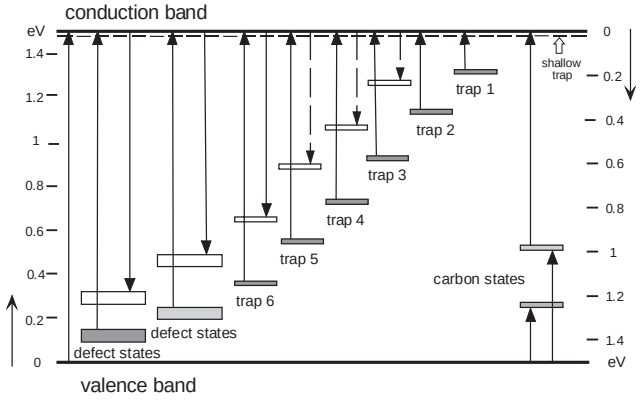

Fig. 12. Energy band scheme of β-rhombohedral boron based on a band gap of approximately 1.5 eV. The energy positions of occupied trapping levels are marked by filled rectangles and those of the corresponding unoccupied states by hollow rectangles. Experimentally determined transitions by optical absorption, photoabsorption or photoconductivity are shown by solid arrows, assumed, but not yet experimentally proved transitions, by broken arrows. The position of the schallow trap, 16(2) meV off the conduction band edge, which became evident in the present paper is indicated by a broken line.

because the density of the hopping sites is modified and that of hole traps in the same way.

Such a diffusion of interstitial B atoms is assumed to require a well-defined activation energy, and therefore it takes place at temperatures below the internal friction maximum as well, with time constants, which increase with decreasing temperatures like indicated by the experimental results. Moreover, the thermal equilibrium of the structure at room temperature is obviously characterized by a $\sim$75% occupancy of the B(13) site [5–8]. This occupancy of the B(13) site, and that of the B(16) to B(20) sites as well determine the entropy under thermal equilibrium at room temperature. Towards higher temperatures the entropy increases, and accordingly the occupation of the B(13) site is expected to decrease. Thus there is a correlation between the occupancy of the B(13) site and the density of electronic gap states, which influences the charge transport.

However, these temperature-dependent specific conditions are averaged ones, and statistical fluctuations around them remain possible. These fluctuations modify the limited conductivity paths assumed for hopping transport by the percolation theory of electrical transport in distorted structures below the percolation threshold (see Refs. [13–15] and references therein). They are statistically changed, for example enlarged or shortened, and we assume this to be the reason for the stochastic precipitous jumps of the electrical conductivity in β-rhombohedral boron. According to Fig. 5, the maximum height $\Delta\sigma$ of these jumps is thermally activated with $\Delta E = 0.81(2)$ eV, which value is compatible with the diffusion of atoms in solids, e.g. $\Delta E = 0.66$ and 1.13 eV for the diffusion of Li and Au respectively in silicon, a covalent bonding material like boron [16], $\Delta E = 1.0(3)$ eV for the diffusion of interstitial atoms in aluminum [17] and energies between about 0.5 and 1 eV in ionic crystals [18]. To check our assumption, the determination of the temperature-dependent occupation densities of the B(13) and B(16) to B(20) sites by X-ray fine-structure investigations is initiated.

The extremely long relaxation times of the steady-state electrical conductivity of β-rhombohedral boron and the stochastic precipitous jumps at higher temperatures can be described consistently with numerous other anomalies of physical properties, when a temperature-dependent diffusion between interstitial B(16)–B(20) and regular B(13) sites is assumed, which modifies the percolation paths of the charge carriers.

The obtained results of time-dependent photoconductivity can be explained within the band scheme of β-rhombohedral boron (Fig. 12). Because of the large capture cross section of the electron traps, the lifetime of the initially generated electrons is extremely small. An especially striking influence comes from the shallow trap 0.16(2) eV off the conduction band. Its function significantly changes at temperatures of about 180–200 K, when the thermal energy reaches its ionization energy. Because of thermal exchange, above these temperatures the electronic states of the trap are no more really separated from the conduction band, and the trap only determines the activation energy of the electron mobility. Taking into account, that at low temperatures the quasi-Fermi level is approximately in the middle between trap and conduction band, it is plausible, that already for $T > 90-100$ K, where $kT > \Delta E_{\mathrm{trap}}/2$, this influence becomes remarkable.

Hence at low temperatures, when this trap has been emptied during the 1 h waiting time before optical excitation, σ_{photo} is determined by the short lifetime of the optically excited electrons in the conduction band, largely independent of T (88–113 K) (see Fig. 8). This changes with proceeding time of optical excitation, when the concentration of unoccupied traps and accordingly the trapping probability decrease in consequence of the persisting optical excitation. Accordingly, in this range the thermal activation energy of σ_{photo} corresponds to the ionization energy of this trap (see Fig. 10). This holds for the delay of the significantly increasing photoconduction as well (Fig. 11).

At low temperatures, after the shallow trap has been filled, and at higher temperatures more or less immediately, σ_{photo} increases, while the deeper traps become occupied, and the trapped electrons are thermally re-excited into the conduction band. Accordingly, the thermal activation energies of the time constants in this range correspond to the trap energies (Fig. 10). The higher the temperature, the deeper are the traps, which are involved in that process. In this range of temperature, the influence of recombination becomes visible. As shown in Ref. [2], the prevailing recombination in β-rhombohedral boron is cascade-like: Electrons captured in trap n are thermally activated into the level of the unoccupied trap $n+1$, whose energy afterwards decreases. The energetical distance between the occupied trap n and the unoccupied trap $n+1$ is 0.12 eV [2,3]. The time constant of the declining delay between the onset of the optical excitation and the increasing contribution of electrons thermally re-excited from traps to σ_{photo} is thermally activated with exactly this energy (Fig. 11). This shows that the electrons are at least preferably captured in the trap, which is closest to the conduction band, and then distributed over the deeper traps by the cascade-like transitions.

Under the final stationary conditions, the concentration of free electrons is independent of time. Nevertheless, the conductivity persists to change: Depending on temperature it increases or decreases. According to Fig. 6, this process is thermally activated with 0.80(3) eV. As discussed in connection with the temperature-dependent stochastic jumps of the conductivity in some detail, this is probably due to the diffusion of B atoms between the interstitial sites B(16) to B(29) and the regular, partly unoccupied sites B(13). The temperature-dependent different direction of this diffusion indicates that the equilibrium occupation of these atomic sites varies with temperature. On the first view, it may be surprising that the interpretation of the photoconduction considers electrons only, while the holes are not taken into account. However, optical transitions between the valence band and the adhered defect states are strictly forbidden because of selection rules (see Refs. [1–3] and references therein). Accordingly, the contribution of holes is largely restricted, in particular at low temperatures. If there is a measurable contribution of holes in the valence band because of thermal exchance with defect states, it may be covered by the excitation of electrons from trap 1 having nearly the same ionization energy. At higher temperatures, the activation energy 0.48(6) eV of the steady-state photoconduction (Fig. 9) can probably be attributed to the ionization of carbon states in exchange with the valence band in accordance with the spectral photoconduction at these temperatures [3].

5. Conclusion

The large temperature-dependent time constants of the development of the steady-state dc conductivity of β-rhombohedral boron at high temperatures are attributed to the diffusion of interstitial B atoms between different sites in the structure explaining the unsteady changes of numerous physical properties as well. This diffusion of atoms evokes a statistical fluctuation of the conductivity paths below the percolation threshold, which is indicated by stochastic precipitous jumps of the electrical conductivity. At low temperatures, such jumps occur as well; there they are attributed to the trapping and releasing of electrons. The time-dependent photoconductivity is strongly influenced by traps reducing the lifetime of the initially generated electrons considerably. This changes, while the trap occupation increases

because of persisting optical excitation. Though under final stationary conditions the concentration of conducting electrons is independent of time, the conductivity persists to change. This is attributed to the diffusion of interstitial B atoms as well.

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