

Chromium and Chromium-Boron Pairs in Silicon

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Abstract. Four different measurement techniques: EPR, NAA, DLTS, and luminescence were applied to characterize the properties of chromium in silicon. The solubility of chromium in silicon was determined and its pairing reaction with boron was studied. The energy level at $E_c - 0.23$ eV was attributed to interstitial chromium and a second level at $E_v + 0.27$ eV was correlated to chromium-boron pairs. The luminescence band of the chromium-boron pairs was clearly identified. The properties of chromium are compared with those of other transition metal impurities in silicon crystals.

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Transition metals in silicon are mostly fast diffusers which influence the performance of semiconductor devices if introduced unintentionally into the crystal during device production. The properties of transition metals in silicon have experienced even growing interest in the last years. Chromium in silicon is one of the less intensely studied systems. Since the pioneering work of Ludwig and Woodbury [1] there have been only a few publications [2–4] till very recently [5–11]. The identification of chromium in silicon on interstitial sites achieved by Electron Paramagnetic Resonance (EPR) analysis [1] is generally accepted. However there are conflicting results concerning its electrical properties [3–11]. These discrepancies may be partly caused by the use of insufficiently defined samples, containing uncontrolled further impurities and impurity complexes.

In this paper, results on chromium in silicon are presented which were obtained by means of Deep Level Transient Spectroscopy (DLTS), EPR, Neutron Activation Analysis (NAA), and photoluminescence investigations. By using mostly identical samples for the different techniques we were able to achieve clear conclusions on the chromium species responsible for the observed sample properties.

1. Experimental

The chromium-doped silicon samples used in our experiments were prepared by diffusing chromium into dislocation- and swirl-free *n*- and *p*-type silicon crystals. The shallow doping concentration in the starting material ranged from 10^{13} to 10^{16} cm⁻³ corresponding to an electrical resistivity of 2000–0.5 Ωcm. Most of the samples were prepared from float-zone refined silicon crystals, only a few from Czochralski-grown material. The experimental results, however, were identical in both crystal types.

Before evaporation of a thin chromium film onto the sample surface, the wafers were etched in a mixture of nitric and hydrofluoric acid and stored in methanol in order to prevent the adsorption of oxygen or nitrogen at the silicon surface. Thus the formation of chromium oxide or -nitride preventing the diffusion of chromium into the silicon crystal could be effectively avoided. In addition, different techniques were used during the diffusion process which was performed at temperatures between 960° and 1150 °C: Diffusions were carried out in vertical and horizontal silica furnace tubes either in a nitrogen atmosphere, a reducing nitrogen-hydrogen atmosphere [12] or in evacuated ampoules. In the pure

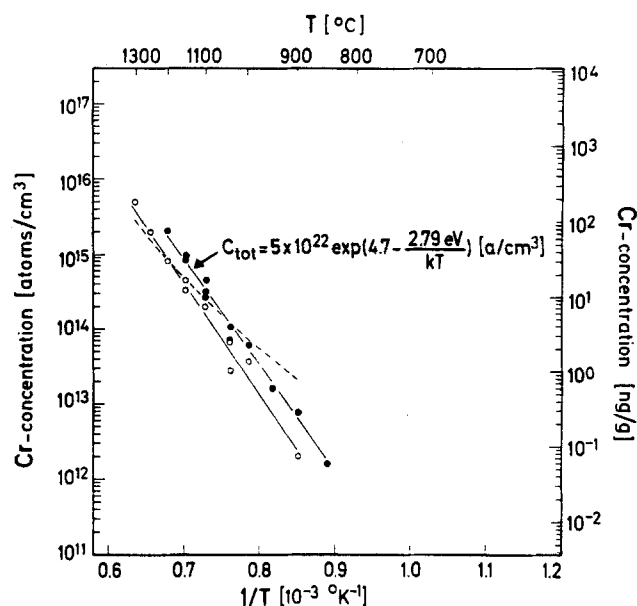


Fig. 1. Solubility of chromium in silicon, as measured by NAA (C_{tot}) (●) and EPR (Cr_i^+) (○) of suitably doped *p*-Si [15]. Further indicated (---) are NAA results of Würker et al. [17]

nitrogen atmosphere a sandwich technique was applied by masking the chromium layer with the aid of a second silicon specimen [8]. After diffusion the samples in the open systems were quenched to room temperature by dropping them into silicone oil. The ampoules used for the diffusion in vacuum were rapidly cooled in water. All specimens were then mechanically and chemically polished to remove the alloy layer.

The EPR measurements were performed with a Bruker BER 420 spectrometer working in the X-band and using a He gas flow cryostat. The EPR spectra of Cr_i^+ and $(\text{CrB})^0$ pairs in *p*-type silicon as described by Ludwig and Woodbury [1] were recorded in slow passage conditions at a temperature of 30 K. EPR of Cr_i^0 was not observed since strong line broadening prevents its detection at temperatures above 4 K [13].

Absolute concentrations of Cr_i^+ were determined by comparison with a NBS calibrated sample [14] and by the use of *p*-type silicon with low boron content in which the Cr_i^+ concentration turned out to be equal to the shallow acceptor concentration. Both methods resulted in the same concentrations with an error margin of 30%.

The total chromium concentration in the samples was obtained using instrumental neutron activation analysis. The experimental details have already been described elsewhere [15]. Using a thermal neutron flux of $10^{14} \text{ n/cm}^2 \text{ s}$ typical irradiation times for the ^{50}Cr measurement were 20 days.

The experimental arrangement to record DLTS signals in a temperature range between 30 and 350 K has been published previously [18]. Schottky barriers were formed by evaporating metal spots 1 mm in diameter on top of the etched silicon surfaces. For *p*-type silicon Mg or Ti was used, for *n*-type silicon Pd contacts were evaporated. Ohmic contacts on the reverse side of the wafers were formed by rubbing emery with liquid Ga which has proved as a fast and successful technique.

The activation energies of the deep levels were determined either by measuring the emission and capture rates of the majority carriers or by an Arrhenius plot of the temperature-corrected emission rates at various fixed measurement temperatures. The latter method exhibits incorrect results if the capture cross-section of the majority carriers depends on the measurement temperature. This should be examined in every experiment if possible.

For luminescence measurements the samples were mounted either in a variable-temperature cryostat or in a liquid helium immersion cryostat which enabled the application of an uniaxial stress to the specimens. For the excitation an argon-ion laser was used operating in the cw-mode. The luminescence was analysed with a Spex monochromator (Model 1702) and a germanium detector (Northcoast type EO 817 L). The signal was amplified in usual lock-in technique.

2. Results

2.1. Interstitial Chromium

Two different chromium-associated EPR signals were found in *p*-type silicon: The spectrum of Cr_i^+ corresponding to an ion with $S=5/2$ (two 4s electrons are promoted to the 3d shell) on the T_d interstitial position and the spectrum of $(\text{CrB})^0$ which is $\langle 111 \rangle$ axially symmetric consistent with the model of an interstitial Cr_i^+ next to a substitutional B_s^- .

Immediately after chromium diffusion no signal of CrB pairs could be detected and the measured chromium concentration was independent of the boron concentration of the starting material (for $[\text{B}] > [\text{Cr}]$). Thus the existence of Cr^{2+} and thus a second donor level [1, 2] could not be confirmed. These observations might be influenced by uncontrolled complexing reactions.

In *n*-type silicon no chromium-related signal was found at measurement temperatures between 10 and 300 K.

Chromium concentrations obtained by NAA were in good agreement with the EPR results on suitably *p*-doped samples ($10^{14} \leq [\text{B}] \leq 10^{16} \text{ cm}^{-3}$) and were found to be independent of type and concentration of the doping with shallow dopants. Figure 1 gives an Arrhenius plot of the maximum chromium concen-

In contrast to iron-doped *p*-type silicon where Fe_i and FeB pairs can be recorded simultaneously, only the increase of the newly formed CrB pairs can be observ-

ed in chromium-doped *p*-type silicon by DLTS. The chromium level cannot be detected in the same specimen since its energy level is situated in the upper half of the band gap of silicon. The activation energy of the CrB pairs was determined to $E_v + 0.27$ eV by measuring the emission- and capture rates of the holes. A slightly differing value $E_v + 0.29$ eV resulting from the determination by means of an Arrhenius plot was published previously [8]. From this difference it may be deduced that the capture cross-section of the holes is temperature dependent. The amount of this rather poor dependency, however, was found to be within our measurement error for the determination of the capture rates in the temperature interval of about 30 K where the DLTS signal was detected. The mean hole capture cross-section resulted to $(1.0 \pm 0.2) \times 10^{-14} \text{ cm}^2$.

In boron-doped silicon we found a new structured luminescence band after chromium diffusion. It consisted of three equispaced lines: CrB^0 , CrB^1 , CrB^2 , and the 0^F lattice phonon replica of the CrB^0 line. The spectrum is similar to luminescence bands detected previously in silicon samples doped with other transition metals [19–21]. The main line CrB^0 at $1.469 \mu\text{m}$ is followed by two phonon replicas CrB^1 and CrB^2 at the long wavelength side. These are due to the excitation of local vibrations of the complex with a typical energy of 13.6 meV. Investigations of 3d transition metal complexes in silicon have shown that the phonon energy seems to be characteristic for the interstitial component of the complex and decreases with increasing atom weight [21]. Typical energies for example are about 10 meV for iron and 7.0 meV for copper [19, 20]. For the lighter element chromium therefore a somewhat higher phonon energy can be expected in agreement with the measured line spacing of 13.6 meV.

The intensity of the luminescence strongly depended on the boron and chromium concentration of the sample. An increase of the main line by a factor of about 50 was observed changing from a sample with $66 \Omega\text{cm}$ to another one with $1 \Omega\text{cm}$ resistivity. Higher boron concentrations of the starting material did not further increase the intensity. On the other hand, if the chromium concentration was varied in samples with a fixed boron content of 10^{17} cm^{-3} a linear correlation between the line intensity and the chromium concentration was found. From these experimental findings we concluded that the luminescence is due to CrB pairs and its intensity is proportional to the number of pairs, which is limited by the component with the lower concentration. We also studied aluminium- and gallium-doped silicon crystals. The respective luminescence lines were not observed after chromium diffusion or only with very low intensities due to a known boron

impurity content of the starting material. In *n*-type silicon we never detected this luminescence band.

Measuring the luminescence intensity as a function of the sample temperature we compared the energies derived from luminescence with the DLTS donor level by making the following assumptions: The energy level measured by DLTS is determined by the deep binding of a hole to the neutral CrB pair. In luminescence we observe the recombination of a bound electron and a bound hole, which is an excitonic decay at an isoelectronic center [22]. According to the model of Thomas and Hopfield [23] the hole is bound very deep and forms a positive core which binds the electron rather weakly. The ionisation energy E_i of this donor electron was determined from an Arrhenius plot to about 20 meV [22]. Therefore we get a hole binding energy $E_h = E_g - E_i - h\nu = 0.3$ eV, with E_g being the gap energy and $h\nu$ the photon energy. The value of E_h is in good agreement with the activation energy for CrB pairs of about 0.28 eV as determined by DLTS.

Application of uniaxial stress to oriented silicon bars ($2 \times 2 \times 7 \text{ mm}^3$) yielded a splitting of the luminescence line which is typical for the lifting of the well-known $\langle 111 \rangle$ orientational degeneracy of the CrB pairs [22].

In analogy to iron-boron pairs [18, 24] the binding of the CrB pairs is rather weak and dissociation of the pairs was observed at elevated temperatures. In thermal equilibrium the dissociation could be described by the law of mass action:



$$C_{\text{CrB}}/C_{\text{Cr}} = C_{\text{B}} A \exp(U/kT) \quad (2)$$

with U : Pair binding energy, C symbolizing the respective concentrations of the participants, k : the Boltzmann constant, T : the sample temperature, and A a proportionality constant.

Annealing experiments were performed in chromium-doped *p*-type silicon with different boron concentrations. Whereas silicon with low boron concentration ($n_{\text{B}} = 2.5 \times 10^{14} \text{ cm}^{-3}$, $n_{\text{Cr}} = 2 \times 10^{13} \text{ cm}^{-3}$) was used in DLTS, investigations by means of EPR and luminescence were carried out on specimens of higher boron concentrations ($n_{\text{B}} = 5 \times 10^{15} \text{ cm}^{-3}$, $n_{\text{Cr}} = 1 \times 10^{15} \text{ cm}^{-3}$, and $n_{\text{B}} = 2 \times 10^{16} \text{ cm}^{-3}$, $n_{\text{Cr}} = 1 \times 10^{15} \text{ cm}^{-3}$, respectively). In all samples the chromium concentration was low compared to the initial boron content. To determine the variation of the pair concentration by means of luminescence we used the linear correlation between the number of CrB pairs and the line intensity. In Fig. 4 the pair-dissociation is demonstrated as function of $1/T$ for various boron concentrations. As is expected by the law of mass action, the higher boron content shifted the dissociation of the CrB pairs to higher temperatures. The solid

lines in Fig. 4 are calculated using the respective boron concentrations and a pair binding energy U of 0.65 eV.

In order to reach thermal equilibrium at slightly elevated temperatures (60 °C) and lowest boron concentrations rather long annealing times (about 20 h) were needed. At higher boron concentrations and the corresponding higher annealing temperatures the required annealing durations reduced drastically because of increased diffusivity of the chromium atoms and the reduced spacing of the substitutional boron atoms in the silicon matrix. For the high boron content of $2 \times 10^{16} \text{ cm}^{-3}$ the reaction velocity was so fast, that in the time between annealing and the experimental determination a substantial re-formation of pairs took place. Therefore we observed for this sample a systematic deviation of the measurement points to higher CrB concentrations (Fig. 4).

3. Discussion

3.1. Diffusion and Solubility

A couple of papers on the properties of chromium in silicon have been published previously starting with the work of Ludwig and Woodbury [1] in 1960. The diffusion coefficient of chromium in silicon has been measured by Bendik et al. [2] and Würker et al. [17] using *pn*-junction methods. Both experiments resulted in

$$D = 0.01 \exp(-1.0 \text{ eV}/kT) \text{ cm}^2/\text{s}$$

giving $D(1100 \text{ °C}) = 2 \times 10^{-6} \text{ cm}^2/\text{s}$. In addition, a similar activation energy of chromium diffusion at low temperatures was derived [2]. These values are close to the activation energy for the diffusion of interstitial iron in silicon (0.86 eV [25], 0.68 eV [26]) and can be considered as typical for interstitial diffusion of transition metals in silicon [26].

Similar to iron, chromium not only diffuses in silicon via an interstitial mechanism but prefers the interstitial site at high temperatures in thermal equilibrium. Thus a high fraction of these interstitial ions is maintained after quenching the samples to room temperature. Substitutional chromium has been obtained by Ludwig and Woodbury [1] only by means of codiffusion with copper. In the present study no comparable experiments were performed.

The enthalpy of formation of interstitial chromium near 2.9 eV, as derived from the solubility results, is within the error limits the same as that determined for Mn, Fe, and Co in silicon. A further evaluation of the solubility parameters can be found in [26]. The entropy of formation is somewhat smaller than that of these other elements. This lower entropy results in

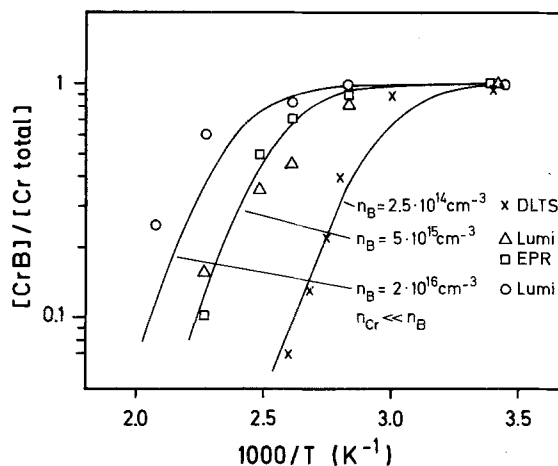


Fig. 4. Relative CrB pair concentration as a function of the annealing temperature in samples of various boron content. Measurement by EPR, DLTS and luminescence (Lumi). The solid lines are calculated equilibrium concentrations for a pair binding energy of 0.65 eV

about a factor of 10 smaller total chromium concentrations. Nevertheless, the interstitial mechanism of diffusion and solution of these 3d transition elements in silicon is obviously the same.

3.2. Electronic Properties

Doping of silicon with transition metal impurities results in a variety of electrically active levels in the band gap. It is a problem to identify the chemical nature of the corresponding defects, because the transition metals built up a lot of complexes with other impurities and they often have very low solubilities. Caused by these difficulties, for chromium a variety of different energy levels was published [3–11], which covers the whole band gap of silicon.

In the present study we compared results for identical samples obtained by DLTS and luminescence investigations, with those obtained by EPR and NAA. The latter methods allow a clear identification of the chromium impurities. The four measurement techniques lead to completely consistent results, if we associate the donor levels $E_c - 0.23 \text{ eV}$ and $E_v + 0.28 \text{ eV}$ to Cr_i or CrB pairs, respectively, and the luminescence band at $1.5 \mu\text{m}$ to CrB pairs. Therefore we are sure that these identifications are correct.

Only a part of these previously published activation energies [4, 5, 8, 10, 11], is in agreement with the Cr_i donor state, which is presented in this study. The respective activation energies varied from 0.20 till 0.25 eV with most favoured results between 0.22 and 0.23 eV. In agreement with Hall-effect measurements of Feichtinger and Czaputa [10] we believe that this will be the best value determined so far. All other energy levels related to chromium in silicon could not be

reproduced. Some of these additional levels may be related to Fe, FeB pairs, and Au impurities introduced unintentionally during the preparation of the specimens. We assume that a couple of authors were not successful in diffusing any chromium into their silicon crystals because they may have used common reacting atmospheres during heat treatment.

Interstitial chromium in silicon exhibits only one single donor state as iron and possibly nickel and copper do whereas the three remaining 3d transition metals Ti, V, and Mn form three charge states [8]. The activation energy of the chromium donor measured from the top of the valence band shows the highest value compared to all other 3d transition metals. In spite of this experimental finding no double donor was detected although a double positively charged chromium ion should exist in CrB pairs in crystals with moderate to high boron content. So far no reasonable assumption was found to explain this observation.

Chromium-acceptor pairs (B, Al, Ga) have been observed and investigated by means of EPR very early [1] but no energy levels for CrB pairs were determined until very recently [8]. In analogy to iron, CrB pairs form donor states. The energy level of the pair is situated between those of chromium and boron forming a deeper level than FeB pairs. The activation energy was determined to about 0.28 eV above the top of the valence band by two independent methods. The hole capture cross-section was found in the order of magnitude of 10^{-14} cm^2 in the temperature region of 160 K.

A new type of luminescence was observed corresponding to these deep centers in silicon [21]. A similar luminescence band was associated to FeB pairs [20]. The main characteristic feature of these bands is a sharp zero phonon line which is followed by several equispaced phonon replica. The energy spacings of the replica are typical for the interstitial component of the complex. For CrB pairs we observed 13.6 meV, for FeB pairs about 10 meV were reported [20]. We could explain the luminescence as an excitonic transition at an isoelectronic center [22]. The hole binding energy corresponds to the DLTS level. It is much larger than that of the weakly bound electron and determines therefore the energy of the luminescence transition in a first approximation.

3.3. Impurity Reactions

After annealing of the specimens we observed a dissociation of the CrB pairs into the components Cr_i and B_s . From the annealing behaviour a pair binding energy of 0.65 eV was deduced. This value is similar to that obtained by splitting FeB pairs (0.5 eV [7]). The values are roughly in accordance with the model of

two Coulombic bound point charges with binding energies of $U = e^2/\epsilon r = 0.55 \text{ eV}$ where ϵ symbolizes the dielectric constant of silicon, r the distance between the nearest T_d interstitial site and the substitutional site (2.4 Å), and e the electron charge. Following the law of mass action the fraction of pairs with respect to the entire impurity content depends on the boron concentration in the specimen at a certain temperature since a high concentration of one component shifts the equilibrium towards pair formation.

In contrast to FeB pairs [18] the CrB pairs cannot be dissociated by an intensive illumination of the specimen. In iron-doped *p*-type silicon the Fermi level is situated usually between the level of the interstitial iron ($E_v + 0.38 \text{ eV}$) and that of boron. According to their donor state the FeB pairs appear in two charge states: $(\text{FeB})^+$ if the Fermi level is beneath the level of FeB ($E_v + 0.1 \text{ eV}$) and $(\text{FeB})^0$ if the Fermi level exceeds the activation energy of FeB. During illumination of the specimen with near band gap light free electrons and holes are created which shift the Fermi level towards the intrinsic level. Since the FeB pairs are proved to be in a thermal equilibrium with the unpaired components [18] the rate of pair formation must be equal to the rate of pair splitting. If, however, the Fermi level exceeds the E_i level, the positively charged iron ion will be discharged after pair splitting. By this way the iron atom will be excluded from pair formation since there is no remaining Coulomb attraction to the boron ion. It has been shown experimentally that neutral iron does not form FeB pairs for example in boron-compensated *n*-type silicon [16].

This mechanism cannot work in chromium-doped silicon. Interstitial chromium is positively charged even at a midgap Fermi level since its energy level is situated in the upper half of the bandgap. So illumination does not discharge the Cr^+ ion. This explains why CrB pairs are not splitted by illumination.

Conclusion

Application of four basically different measurement techniques on chromium-diffused silicon allowed us to relate defect properties to well-defined chromium species. Chromium enters the silicon lattice interstitially and stays in interstitial sites in thermal equilibrium at high temperatures and after rapid quenching. Interstitial Cr as well as CrB pairs give rise to donor levels. Photoluminescence measurements yielded no radiative emission of isolated interstitial chromium in the wavelength region up to 1.8 μm . An emission of 0.85 eV occurring in boron-doped silicon can be related to the electrical level of CrB pairs. The behaviour of EPR spectra, DLTS signal and photoluminescence

emission after annealing the samples confirm this identification.

A summary of relevant data obtained for chromium in silicon is given in Table 1.

Although chromium is a widely spread heavy metal it is scarcely found as an unwanted impurity in semiconductor devices. The main reason may be its high reactivity with oxygen and nitrogen which prevents the diffusion of chromium into the silicon wafer during heat treatments in common atmospheres. Furthermore the solubility and diffusivity of chromium in silicon are less than those of iron, nickel, and copper which are frequently found as unwanted precipitates in devices in contrast to chromium.

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Table 1. Properties of chromium and CrB pairs in silicon

Diffusion coefficient	$D = 0.01 \exp(-1.0 \text{ eV}/kT) \text{ cm}^2/\text{s}$		
Solubility NAA	$\text{Cr}_{\text{tot}} = 5 \times 10^{22} \exp(4.7 - 2.79 \text{ eV}/kT) \text{ cm}^{-3}$		
EPR	$\text{Cr}_i = 5 \times 10^{22} \exp(5.4 - 2.95 \text{ eV}/kT) \text{ cm}^{-3}$		
Activation energy Cr_i	$E_c - 0.23 \text{ eV}$		
Electron carrier cross-section	$\sigma_e = 2 \times 10^{-13} \text{ cm}^2$		
Activation energy CrB	$E_r + 0.28 \text{ eV}$		
Hole carrier cross-section	$\sigma_h = 1 \times 10^{-14} \text{ cm}^2$		
Luminescence lines	CrB ⁰	1.469 μm	0.844 eV
	CrB ¹	1.493 μm	0.830 eV
	CrB ²	1.518 μm	0.816 eV
Pair binding energy	0.65 eV		