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Dependence of electrical conductivity of nanocrystalline silicon on structural properties and the effect of substrate bias

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ABSTRACT

The dark electrical conductivity of undoped and phosphorus-doped nanocrystalline (nc) and amorphous (a-) thin films of silicon has been studied as a function of substrate bias between $V_b \sim 0$ (floating) and -1000 V under conditions in which all other deposition parameters, such as temperature, deposition rate, discharge current and chemical composition of the plasma, were held constant. With increasing bias from $V_b \sim 0$ to -100 V, the room-temperature conductivity σ_{RT} of undoped nc-Si decreases from $\sim 10^{-4}$ to about $10^{-9}\,\Omega^{-1}\,cm^{-1}$. A further increase in the bias results in the appearance of a second conductance path which could be attributed to grain boundaries. At a bias of $V_b < -600$ V, a mixture of nc- and a-Si phases are formed with σ_{RT} showing a rather complex dependence on V_b and approaching that of a-Si for $V_b \sim -1000$ V, where no crystalline component is detectable by X-ray diffraction. Phosphorus-doped nc-Si films prepared under the same conditions and with a gas-doping ratio of $[PH_3]/[H_2] = 10^{-4}$ exhibit an almost constant conductivity over the whole bias range between 0 and -850 V. An interpretation of these results is suggested.

§ 1. INTRODUCTION

The future technological importance of nanocrystalline silicon (nc-Si) for solar cells, sensors and integrated circuits, as well as for basic solid-state research is widely recognized. Since the first report on the preparation of nc-Si and nc-Ge by chemical transport in a hydrogen plasma (Vepřek and Mareček 1968), a number of papers on various properties of these materials has been published (for reviews see, Matsuda (1983), Mishima, Miyazaki, Hirose and Osaka (1982), Vepřek (1982, 1984) and Osaka and Imura (1984)). The present paper deals with the dependence of the dark electrical conductivity of undoped and P-doped nc-Si on the substrate bias applied during deposition in a d.c. discharge. The bias determines the structural properties of the deposited films. An appropriately designed deposition apparatus (Vepřek 1982, 1984), enables one to vary the substrate bias without significantly changing other conditions, such as deposition temperature, deposition rate, discharge current density and concentration of SiH_x species in the gas phase. This is a substantial improvement compared to the majority of work on nc-Si§ which involve r.f. discharges and a mixture

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§ As the crystallite size of this material typically ranges between $\sim 2{\cdot}5$ and 35 nm we prefer the more appropriate term 'nanocrystalline' Si, in agreement with the nomenclature of nc-metals.

of hydrogen and silane (e.g., Matsuda, Kumagi and Tanaka (1983), Komuro, Aoyagi, Segawa, Namba, Masuyama, Matsuda and Tanaka (1984)). In the latter case, one typically obtains a mixture of amorphous and nanocrystalline phases, and the basic plasma parameters determining their relative contributions remain buried in the complexity of the system.

A series of studies performed by several research groups (Vepřek and Mareček 1968, Matsuda 1983, Komuro *et al.* 1984, Spear, Willeke, Le Comber and Fitzgerald 1981, Le Comber, Willeke and Spear 1983, Willeke, Spear, Le Comber 1984, Tsu, Hernandes, Chao, Lee and Tanaka 1982, Augelli, Murri, Alba and Ligonzo 1983, Richter and Ley 1981) has shown that the electronic properties of nc-Si depend mainly on the relative amount of the crystalline and amorphous phase and on the crystallite size. However, little attention has been devoted to the effect of mechanical stress in these films, which can reach high values (30–40 kbar) and thus significantly affect the properties of the intergrain material (Vepřek 1982, 1984, Vepřek, Iqbal, Kühne, Capezzuto, Sarott and Gimzewski 1983, Sarott, Iqbal and Vepřek 1982). It has also been shown that Raman spectra fail to provide unambiguous data on the relative amount of the amorphous component in nc-Si (Vepřek 1984).

The aim of the present paper is to show the effect of these parameters on the dark electrical conductivity of nc-Si and of mixed amorphous and nanocrystalline films and to give an interpretation of these results.

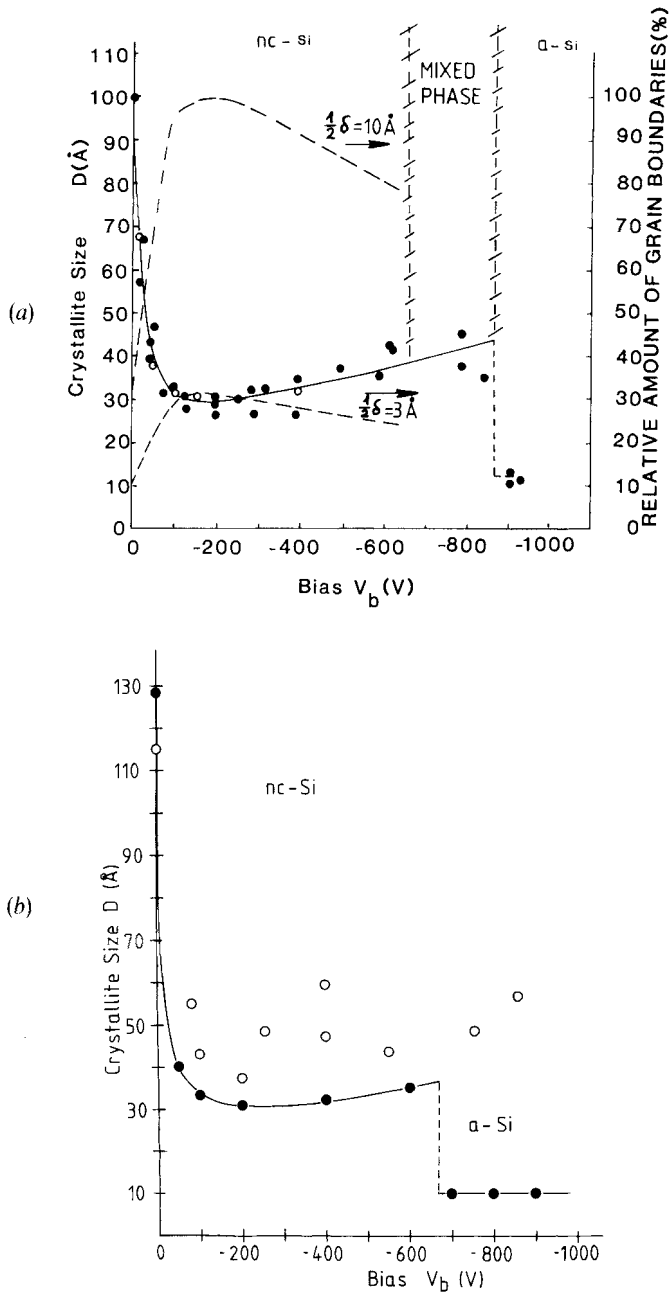
§ 2. Experimental

The samples of nc-Si and a-Si were deposited via chemical transport of silicon in hydrogen discharge (Vepřek and Mareček 1968, Vepřek 1982, 1984, Vepřek *et al.* 1983) using, in the undoped case, a later version of the apparatus described elsewhere (Vepřek 1984). The advantage of this preparation technique is the excellent control and reproducibility of the deposition process which allows one to prepare pure nc-Si films with a mean crystallite size between ~ 30 and 350 Å, as well as a-Si and a mixture of both. The application of d.c. glow discharge offers the possibility of a simple scaling and, therefore, an excellent reproducibility of the properties of samples prepared in different apparatus (see, e.g., fig. 1). Furthermore, the appropriate design of the substrate holder enables us to control and measure the bias between the 'wall' potential (termed 'floating') $V_b = 0$ and -1000 V. Here, the bias is defined as the true difference of the substrate potential and that of the surrounding unperturbed plasma (for details see Vepřek (1984)).

With increasing bias, i.e. with the substrate becoming more negative, the flux towards the surface of the growing film increases, as does the impact energy. The ion flux can be measured exactly but the impact energy $E_{imp} \sim (0{\cdot}2\text{–}0{\cdot}3)V_b$ (for $V_b \sim 0$ to -300 V). The deposition rate increases only by a small amount ($\sim 20\%$) but the hydrogen content (~ 3–4 at.%) remains constant for $V_b = 0$ to -1000 V (Vepřek 1984). These results were also verified for the samples prepared for the present study.

X-ray diffraction data were taken on each of the deposited samples using a Siemens D 500 powder diffractometer with CuK_α radiation and digital data processing. The crystallite size was calculated according to the Scherrer formula from the measured (111), (220) and (311) Bragg reflections. From the apparent crystallite size $D(hkl)$, the lengths of the cube edge $D(100)$ were calculated using the appropriate crystallographic relations. The mean values of $D(100)$ obtained in this way from the (111), (220) and (311) peaks for undoped and P-doped samples are given in figs. 1 (*a*) and 1 (*b*).

Fig. 1



Mean crystallite size (cube edge $D(100)$) calculated from the (111), (220) and (311) Bragg diffraction peaks. (*a*) Undoped samples: ● data obtained using a previous apparatus (Sarott *et al.* 1982); ○ present work; $T_d = 260°C$. The broken lines show the relative amount of the grain boundaries material assuming the mean half-width of the grain boundaries to be ~3 and 10 Å, i.e. about one and three (111)-lattice spacings. (*b*) Phosphorus-doped samples: ● 'large area' samples deposited on Mo substrates for the X-ray diffraction studies; ○ 'small area' thin samples used for the conductivity measurements.

Silica and molybdenum substrates were used for the measurement of the electrical conductivity σ with evaporated gold or aluminium electrodes in gap and sandwich arrangements, respectively. In the former case, a film of nc-Si of several μm thickness of a limited area of $4 \times 7\,mm^2$ had been deposited on silica substrate using a molybdenum mask. Immediately afterwards, the electrodes were evaporated in order to avoid oxidation of the silicon surface (Gimzewski and Vepřek 1983). For the sandwich structure, about 1000 Å of gold was evaporated as the bottom electrode on the molybdenum substrate prior to the deposition of a 10–20 μm thick silicon film. Approximately 100 upper Au electrodes each of an area of $0{\cdot}25\,mm^2$ were evaporated on the surface of the film, carefully avoiding its oxidation.

The sandwich arrangement was used for undoped films deposited on Mo substrates under negative bias. Undoped samples deposited on silica at floating potential, as well as doped samples (all deposited on silica substrates), were measured using a gap arrangement. The conductivity of doped films could not be determined in the sandwich arrangement because of their relatively high value of $\sigma \approx 1\,\Omega^{-1}\,cm^{-1}$ resulting in too small resistances of the probes of $<0{\cdot}1\,\Omega$ for film thicknesses of $\approx 10\,\mu m$.

The deposition of the films under controllable bias implies a certain minimum conductivity of the substrate. On the other hand, the measurement of the film conductivity in the gap arrangement requires a highly resistive substrate. In order to compromise between these two requirements we first deposited an undoped 0·1–0·2 μm thick nc-Si film on a silica substrate at floating potential and, subsequently, the doped one deposited under the desirable bias on top of it. The film area was typically about $2 \times 5\,mm^2$ and the thickness of the doped one, 1–2 μm. From the measured density of the ion current from the plasma towards the substrate, the relatively high conductivity of the undoped 'substrate' nc-Si film ($\approx 10^{-2}\,\Omega^{-1}\,cm^{-1}$) at the deposition temperature of 260°C and the small film area we calculated that the voltage drop across the film during deposition was of the order of 1 V or less. This allowed accurate control of the bias to within a few volts, i.e. much better than in r.f. discharge.

The effect of the underlying undoped film on the conductivity measurement was negligible because of its much reduced thickness and conductivity as compared to those of the doped one (the corresponding error of the measured σ-value was $\leqslant 0{\cdot}1\%$).

Current–voltage characteristics were measured for each sample in order to check for good Ohmic contact and the absence of perturbing interface effects. The voltage used in the measurement of the temperature dependence of the electrical conductivity was typically 10 V and the current density was below $0{\cdot}5\,A\,cm^{-2}$. The measurements were performed under vacuum better than 2×10^{-6} Torr using a computer-controlled system described elsewhere (Vepřek *et al.* 1983) over the temperature range 20 to 230°C. In each case we verified that any effects of gas adsorption which might affect the measured data (see Vepřek *et al.* (1983)) were absent.

§3. Results and discussion

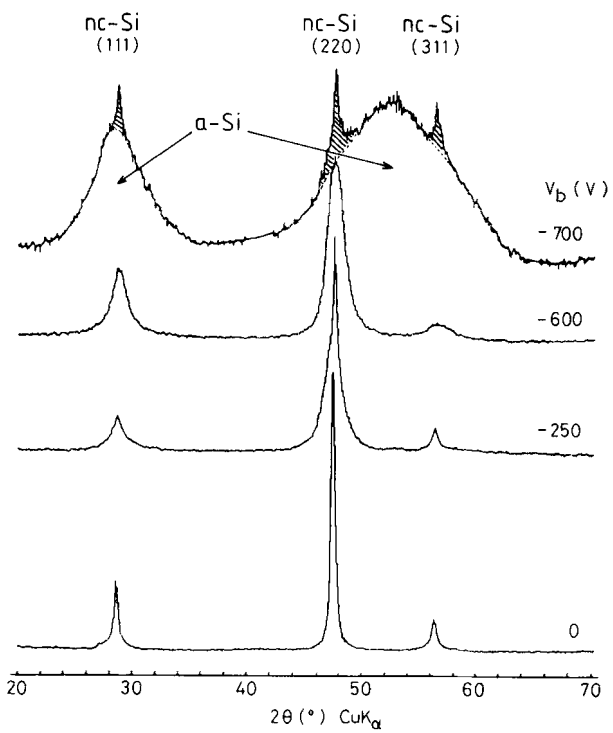
3.1. *X-ray diffraction and transmission electron microscopy data*

Figure 1 (*a*) shows the mean crystallite size of undoped n-Si versus bias at a constant deposition temperature of 260°C. The solid circles represent previous data (Sarott *et al.* 1982) obtained using an older deposition apparatus, which was used in the present work for the preparation of doped films. Open circles are for samples from the new apparatus (Vepřek 1984) used for the deposition of undoped films, illustrating excellent reproducibility. For a discussion of these data we refer to Vepřek (1984) and Sarott *et al.* (1982).

Figure 1 (*b*) shows the mean crystallite size of the phosphorus-doped samples. The solid-circles data were obtained from 5–16 μm thick samples of area $\sim 1{\cdot}5\,cm^2$ deposited on metallic substrates (Al and Mo). Open circles correspond to the samples used for the conductivity measurement. Owing to the small area and thickness of the latter, one obtains very weak Bragg reflections from the nc-Si sample superimposed on a strong signal from the amorphous silica substrate. In spite of the resulting larger error in the measurement, the data agree well with those obtained from the thick 'large area' samples, and both agree surprisingly well with the data for undoped samples (fig. 1 (*a*)). The only difference in the data given in figs. 1 (*a*) and 1 (*b*) is the range of the bias at which the crystalline-to-amorphous transition occurs. This transition has been discussed previously (Vepřek 1984, Vepřek, Iqbal and Sarott 1982). The somewhat lower value of bias at which the amorphization of the doped samples occurs can be understood in terms of a lower yield strength of P-doped Si. However, more quantitative data are necessary in order to clarify this question in detail.

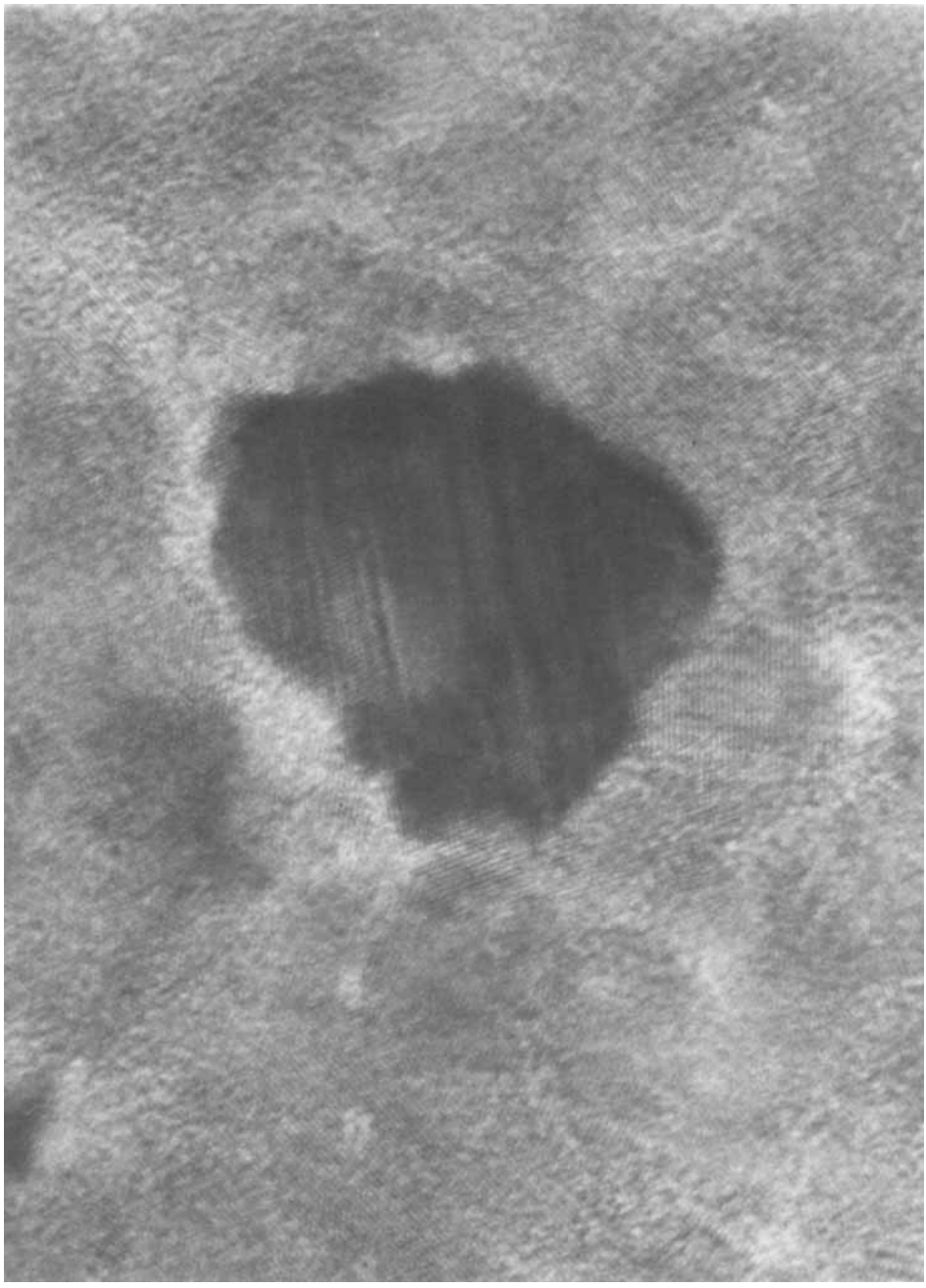
Typical examples of X-ray diffraction patterns of undoped samples are shown in fig. 2 for samples deposited under different substrate bias. Similar data were obtained also with the doped samples. We have shown previously (Vepřek *et al.* 1982) that the nanocrystalline and amorphous phases can be distinguished by the shape of the diffraction pattern inbetween the (220) and (311) Bragg reflections of c-Si. Accordingly, the films deposited at $V_b = 0$ to -600 V comprise a pure nc-Si phase with negligible amorphous component. In contrast, the sample deposited at $V_b = -700$ V consists of a mixed phase in which the amorphous component is dominant.

Fig. 2



X-ray diffraction pattern of nc-Si deposited at 260°C and substrate bias V_b between 0 and -600 V. The upper diagram corresponds to a mixed a-Si and nc-Si phase obtained at $V_b = -700$ V.

Fig. 3



High-resolution transmission electron photomicrograph showing a direct image of the (111) lattice planes in a ~ 200 Å thick nc-Si film deposited at floating potential.

This conclusion is further supported by fig. 3, which shows a typical high-resolution transmission electron micrograph of an area ($\sim 540 \times 430$ Å^2) of ~ 200 Å thin film deposited at $V_b = 0$. The fine lines, which can be seen over the whole area, correspond to direct images of the (111)-lattice planes of crystalline silicon. Close examination of a

series of such photomicrographs taken on several samples confirmed the absence of extended amorphous tissue, but revealed only narrow grain boundaries, which arise when growing crystalline nuclei approach each other.

Of course, we do not exclude the possibility of the growth of a heterogeneous mixture of a-Si and nc-Si under conditions of hard ion bombardment, as shown in fig. 2 for the sample at $V_b = -700$ V, or far away from partial chemical equilibrium (Vepřek 1982, Vepřek *et al.* 1981, 1982, Wagner and Vepřek 1982, 1983), where the nucleation and growth are poorly controllable (e.g., the deposition from a mixture of silane with hydrogen).

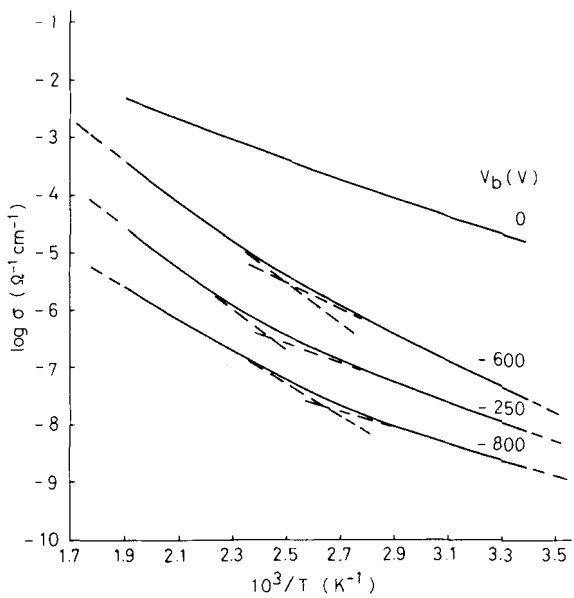
3.2. *Dark conductivity of undoped samples*

The Arrhenius plot of $\log \sigma$ against the reciprocal temperature (fig. 4) is a straight line for samples deposited at a V_b between 0 and about -100 V, but becomes curved for larger bias. As for the latter samples ($V_b < -100$ V), the plot of $\log \sigma$ against $T^{1/4}$ does not yield straight lines, the conduction mechanism by variable-range hopping (Mott and Davis 1979) can be excluded:

$$\sigma = \sigma_0 \exp\left[-\left(\frac{T_0}{T}\right)^{1/4}\right]. \tag{1}$$

As has been shown by Spear, Le Comber and their co-workers (Spear *et al.* 1981, Le Comber *et al.* 1983, Willeke *et al.* 1984, Willeke, Spear, Jones and Le Comber 1982, Spear, Willeke and Le Comber 1983), electronic transport in nc-Si can be described using the grain boundary trapping model that was proposed for polycrystalline

Fig. 4



Arrhenius plot of the logarithm of the dark conductivity against reciprocal temperature for silicon films deposited at 260°C and different bias. For $V_b = 0$ to -600 V the samples are microcrystalline, for $V_b < -700$ V they are largely amorphous.

semiconductors (Orton and Powell 1980). Accordingly, the temperature dependence of σ is of the form

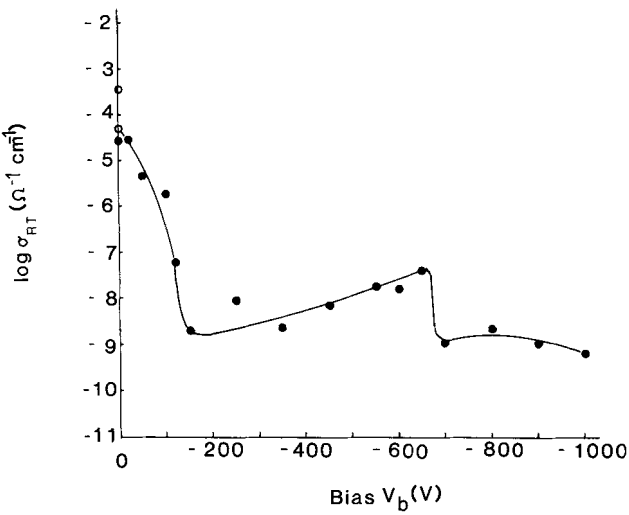
$$\sigma = \sigma_0 \exp(-\varepsilon_a/kT), \tag{2}$$

where the activation energy depends on the dominant charge-transport path. Thus, for the purpose of further discussion, we separate the measured dependence of $\log \sigma$ on $1/T$ into a low and a high-temperature parts which can be approximated by eqn. (2) (see also fig. 4).

Figure 5 shows the dependence of the logarithm of the dark conductivity at room temperature upon the substrate bias. Accounting for the fact that in the gap and sandwich arrangement of the electrodes the conductivity is measured in the direction parallel and perpendicular to the substrate, respectively, the agreement of the data at $V_b = 0$ is fairly good. (Films deposited at $V_b = 0$ V have, to a certain degree, a columnar structure and, therefore, inherently anisotropic properties.) One notices the decrease in σ of about five orders of magnitude as the bias changes from 0 to about -150 V, followed by a relatively small, yet noticeable, increase between $V_b \sim -200$ and -600 V. At even greater bias, when either a mixed amorphous and nanocrystalline, or even only the amorphous phases, are formed, the conductivity decreases again. In the pure nc-Si phase between $V_b = 0$ and -600 V the change in σ resembles that of the crystallite size shown in fig. 1, and its lowest value ($\sim 10^{-9}\,\Omega^{-1}\,\mathrm{cm}^{-1}$) is close to that of a-Si.

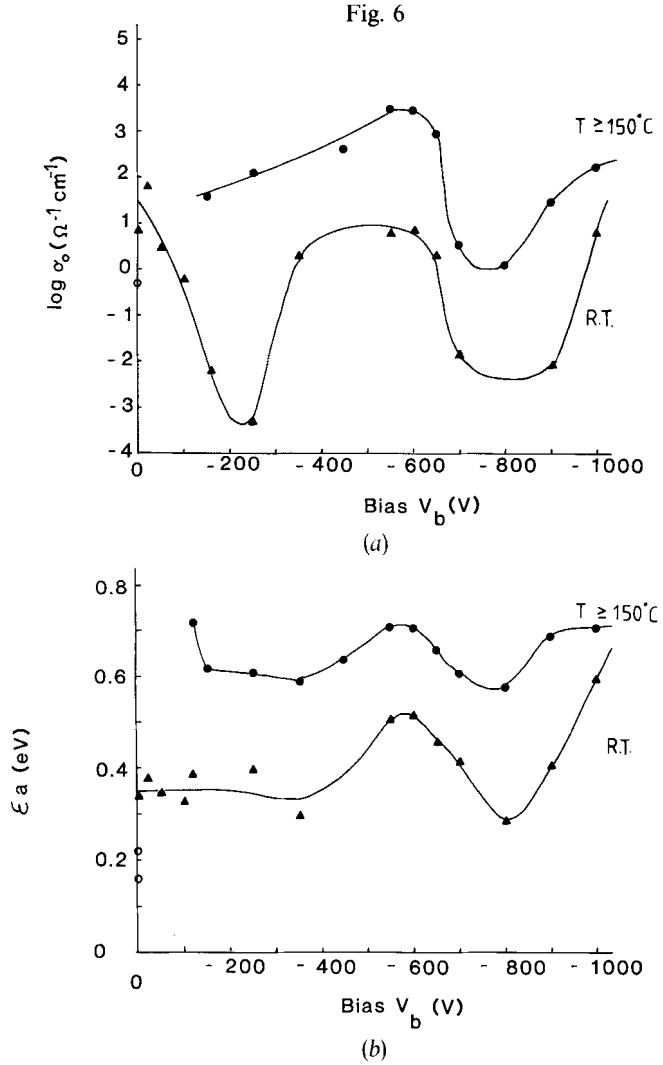
Figures 6(*a*) and (*b*) show the corresponding dependences on the bias of the logarithm of the pre-exponential factor σ_0 and of the activation energy ε_a for the low- and high-temperature parts of the Arrhenius plot, respectively (for examples, see fig. 4). As the plots of $\log \sigma$ against $1/T$ are linear for $V_b = 0$ to -100 V there is only one value of σ_0 and ε_a for such samples indicated in fig. 6. However, for $V_b < -100$ V, there is an additional charge-transport mechanism dominant at $T > 150$°C, which is characterized by greater values of σ_0 and ε_a. It is seen that the decrease in σ_{RT} with V_b changing from 0 to ~ -100 V (fig. 5) is due predominantly to the decrease in the pre-exponential

Fig. 5



Dependence of the logarithm of the dark electrical conductivity of undoped nc-Si at room temperature on the substrate bias during deposition: ○ gap-cell geometry; ● sandwich geometry.

Fig. 6



Dependence of (*a*) the pre-exponential factor, and (*b*) the activation energy ε_a for the room-temperature (▲) and the high-temperature ($\gtrsim 150$°C) (●) range. ○ gap arrangement (undoped nc-Si).

factor σ_0. Such behaviour is expected within the framework of the grain-boundary trapping model when the electron tunnelling through the grain boundaries is the rate-limiting step of the charge transport.

One also notices that the high-temperature conductance path comes into operation when the relative amount of the grain-boundary material (see fig. 1) reaches a certain critical value. Assuming that for this conduction path, charge transport takes place within the boundaries (Orton and Powell 1980) and assuming a percolation threshold between ~ 30 and 50% (Mott and Davis 1979, Essam 1980), we conclude that essentially only a relatively narrow part of the grain boundary of a mean width of 6 to 20 Å contributes to this kind of charge transport. One also notices that the observed apparent activation energy of $\sim 0{\cdot}6$ to $0{\cdot}7$ eV changes relatively little with V_b, and that it is in the range typical of a disordered Si network.

Both the activation energies and the pre-exponential factors display a relatively complex, but fairly reproducible, dependence on the substrate bias. Let us first briefly discuss the pure nc-Si phase that is deposited at $V_b = 0$ to ~ -600 V (see figs. 1–3). In our previous paper (Vepřek 1984) we show that the compressive stress in the film increases with increasing bias owing to ion bombardment and, for a bias of -600 V, it reaches a value of about 40–50 kbar. Thus, as a preliminary interpretation of the results given in figs. 5 and 6, we suggest that the increase in σ_0 over this range of V_b is associated with the improved 'connectivity' of the Si network, i.e. with a decrease in the amount of density-deficient regions. This interpretation is supported by our finding that incorporation of gases into the films is completely prevented if nc-Si is deposited under negative bias (Vepřek *et al.* 1983).

Further support is lent by detailed X-ray diffraction studies that show that the region of grain boundaries, which in films deposited at $V_b = 0$ V show expansion of the lattice spacing, are compressed in films deposited at $V_b \sim -150 - 600$ V (Vepřek *et al.* 1982).

For bias $V_b < -600$ V, where a mixture of a-Si and nc-Si is deposited, both σ_0 and ε_a decrease as is expected. The decrease in the electrical conductivity of silicon films upon transition from the crystalline to the amorphous phase has been reported previously (Johannessen 1974, Müller and Kalbitzer 1980, Kamiya, Kishi, Ushirokawa and Katoda 1981). However, although the data presented in figs. 5 and 6 are fairly reproducible, their interpretation is not simple owing to the complex structural changes associated with the strong decrease of compressive stress in the films which occurs between $V_b \sim -650$ and -800 V (Vepřek 1984).

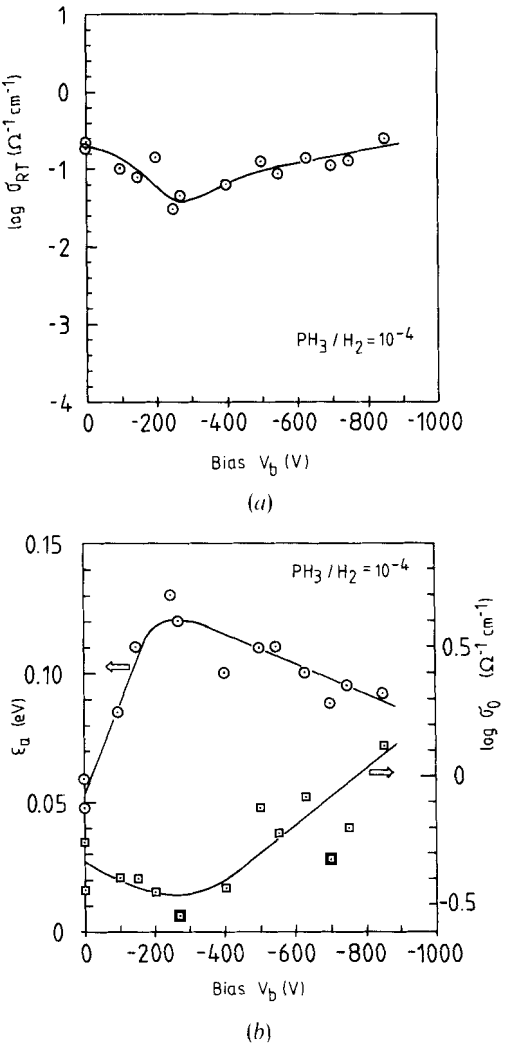
With a further increase of bias towards $V_b \sim -1000$ V the low- and high-temperature values of σ_0 and ε_a converge. The Arrhenius plot of $\log \sigma$ against $1/T$ for samples deposited at $V_b \sim -1000$ V is almost linear and the corresponding values of σ_0 and ε_a are typical of undoped a-Si. One further notices that the decrease in σ_{RT} for such samples, as compared with that of nc-Si deposited at $V_b \sim 0$ V, is due predominantly to the increase of activation energy (see fig. 6). These results are in agreement with the X-ray diffraction (see fig. 2 and Sarott *et al.* (1982), Vepřek *et al.* (1982)) and Raman scattering data (*Ibid.*, Vepřek 1984), which show that undoped samples deposited at $V_b \sim -1000$ V are entirely amorphous.

3.3. *Dark conductivity of phosphorus-doped nc-Si*

Figure 7 (*a*) shows the dependence of the logarithm of the room-temperature dark conductivity of nc-Si doped with phosphorus at a constant concentration of phosphine in the gas phase, $[PH_3]:[H_2] = 10^{-4}$, on the bias applied during deposition. Although the relative change of $\log \sigma$ with bias resembles that of undoped material (fig. 5) its magnitude is smaller by about a factor of 2×10^4.

The temperature dependence obeys strictly the exponential law, eqn. (2), for all doped samples. The corresponding values of σ_0 and ε_a are plotted in fig. 7 (*b*) against the bias voltage. The magnitude of the changes is much less than that for the undoped samples and, in addition, the dependence of ε_a and $\log \sigma$ on V_b is somewhat different (cf. fig. 6). As the dependence of $\log \sigma$ on $1/T$ remained fairly linear for all samples measured, there is only one value of ε_a and σ_0 for each of them. Thus, there is only one dominant conduction mechanism for the whole range of temperature and applied bias. In view of the results of other authors (Spear *et al.* 1981, 1983, Le Comber *et al.* 1983, Willeke *et al.* 1982, 1984, Orton and Powell 1980) we conclude that electron transport is via extended states at E_C.

Fig. 7



Dependence of (*a*) the logarithm of the dark electrical conductivity σ, (*b*) activation energy ε_a and pre-exponential factor σ_0 of phosphorus-doped samples ($[PH_3]/[H_2] = 10^{-4}$, constant) on bias.

The slight decrease in σ_{RT} at a bias of about -200 V, (fig. 7 (*a*)) is due to a decrease in σ_0 and an increase in ε_a. For a larger bias, σ increases (σ_0 increases and ε_a decreases) monotonically and no amorphous-to-crystalline transition is observed for these samples. A similar conclusion may be drawn from the X-ray diffraction data (fig. 1 (*b*)) for 'small-area samples'. It is likely that the crystalline phase is stabilized in these tiny samples deposited on relatively thick silica substrates owing to the high compressive stress. Such an effect has been found experimentally and has been discussed theoretically in our previous papers (Vepřek *et al.* 1982, Vepřek 1984).

The small changes in the dark conductivity of doped samples compared to the undoped ones between $V_b = 0$ and -200 V are explained in terms of preferential doping

of the grain boundaries due to dopant segregation. Impurity segregation is a well-known phenomenon and it has been shown to occur also for boron-doped nc-Si (Hamasaki, Ueda, Osaka and Hirose 1983). In the present case, grain boundary defect passivation due to dopant segregation results in a decrease of the energy barrier for electron tunnelling which is no more the charge transport limiting step.

§4. Conclusion

The effect of substrate bias on the dark electric conductivity σ of nc-Si and a-Si is a complex phenomenon. The observed changes in σ, σ_0 and ε_a of nc-Si are due to a combined effect of the change of crystallite size, relative amount of grain-boundary material and compressive stress in the films. For samples deposited at a bias between -700 and -1000 V the values of σ_{RT}, σ_0 and ε_a approach those of a-Si, in agreement with the increasing amorphous component in the films.

A preliminary and tentative interpretation of these results has been given. For a more detailed understanding of these complex phenomena, additional data, such as thermoelectric power, Hall effect and density-of-states measurements would be necessary. Such measurements cannot be done on samples with a sandwich arrangement of the electrodes. However, such an electrode configuration had to be used in the present work in order to assure accurate control of the substrate bias. Further work is in progress.

A result which deserves particular emphasis is the fact that nc-Si films without an extensive amorphous network can be deposited at a bias $V_b \sim 0$ to -600 V if adequate control of the steady state ('equilibrium') concentration of silane in the gas phase is provided. This result is further supported by Raman-scattering data published previously (see fig. 13 of Vepřek (1984)).

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