

THE INFLUENCE OF THE RATE OF DEFORMATION ON THE ELECTRICAL PROPERTIES OF ICE MONOCRYSTALS

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ABSTRACT. The influence of plastic deformation on the electrical properties of ice monocrystals has been measured in the frequency range 10^{-2} to 10^5 Hz as a function of strain-rate and strain. The crystals have been tested mainly under uniaxial compression, their orientation being for glide in the basal glide system $(0001)[11\bar{2}0]$. The resolved shear strain-rate ranged from 2×10^{-5} to $1 \times 10^{-3} \text{ s}^{-1}$, the resolved shear strain from 3 to 60%. Transient as well as bridge methods have been used for the electrical measurements before, during and after the deformation. Plastic deformation reduces the electrical conductivity in the space-charge dispersion range. The decrease depends strongly on the strain-rate; the size of the strain has practically no influence. After the end of the deformation recovery processes have been observed, which restored the original conductivity if the total strain was small. Rapid and large deformations however leave permanent changes. The changes of the d.c. conductivity and of the dispersion strength in the space-charge region are discussed and attributed to the increase of the dislocation density and the change of character of the dislocation network.

RÉSUMÉ. Influence de la vitesse de déformation sur les propriétés électriques de monocristaux de glace. L'influence de la déformation plastique sur les propriétés électriques de monocristaux de glace a été étudiée en fonction de la vitesse de déformation et du taux de déformation dans le domaine de fréquence 10^{-2} – 10^5 Hz. Les cristaux ont été soumis essentiellement, à une compression uniaxiale, leur orientation correspondant au glissement dans le système $(0001)[11\bar{2}0]$. La vitesse de déformation effective en cisaillement se situait entre 2×10^{-5} et $1 \times 10^{-3} \text{ s}^{-1}$, le taux de déformation effective en cisaillement entre 3 et 60%. Nous avons effectué les mesures électriques avant, pendant et après la déformation aussi bien avec une méthode transitoire qu'avec des ponts. La déformation plastique diminue la conductivité électrique dans le domaine de dispersion lié aux charges d'espace. Cette diminution dépend fortement de la vitesse de déformation; le taux de déformation n'a pratiquement aucune influence. Après la fin de la déformation, des phénomènes de restauration ont été observés, la conductivité initiale étant retrouvée si la déformation totale avait été faible. Par contre, des déformations importantes et rapides sont suivies de modifications permanentes. Les variations de conductivité en courant continu et de l'intensité de relaxation dans le domaine lié aux charges d'espace, sont analysées et nous les avons attribuées à l'augmentation de la densité de dislocations et à la modification du réseau de dislocations.

ZUSAMMENFASSUNG. Der Einfluss der Verformungsgeschwindigkeit auf die elektrischen Eigenschaften von Eis-Einkristallen. Der Einfluss der plastischen Verformung auf die elektrischen Eigenschaften von Eis-Einkristallen wurde im Frequenzbereich 10^{-2} bis 10^5 Hz in Abhängigkeit der Verformungsgeschwindigkeit und des Verformungsbetrages gemessen. Die Kristalle wurden hauptsächlich unter einachsigen Druck verformt und waren für Abgleitung im basalen Gleitsystem $(0001)[11\bar{2}0]$ orientiert. Die Abgleitgeschwindigkeit erstreckte sich von 2×10^{-5} bis $1 \times 10^{-3} \text{ s}^{-1}$, die Abgleitung von 3 bis 60%. Für die elektrischen Messungen vor, während und nach Verformung wurden Transienten- und Brückenverfahren verwendet. Plastische Verformung verringert die elektrische Leitfähigkeit im Raumladungsdispersionsbereich. Die Abnahme hängt stark von der Verformungsgeschwindigkeit ab, die Grösse der Verformung hat nahezu keinen Einfluss. Nach Beendigung der Verformung wurden Erholungsvorgänge beobachtet, welche die ursprüngliche Leitfähigkeit wiederherstellen, sofern die gesamte Verformung klein war. Schnelle und grosse Verformungen jedoch hinterlassen bleibende Änderungen. Die Änderungen der Gleichstromleitfähigkeit und des Dispersionsbetrages im Raumladungsbereich werden diskutiert und der Erhöhung der Versetzungsdichte und der veränderten Eigenart des Versetzungsnetzwerkes zugeschrieben.

1. INTRODUCTION

The influence of plastic deformation on the electrical polarization and transport properties in ice, i.e. dielectric permittivity and electrical conductivity, has been further investigated since the symposium in Ottawa (Noll, 1973). Glen (1968) first pointed to the importance of the reorientation of H_2O molecules on the plastic deformation, a mechanism originally only thought responsible for mechanical and electrical relaxation phenomena. Since then the interaction of various effects, initially treated separately, has attracted increasing attention.

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Experiments, showing the connection of internal friction with plastic deformation have been carried out by Perez and co-workers (Perez and others, 1976; Vassoille and others, 1978). The various aspects of the interaction between the reorientation of H_2O molecules and dislocation movement is discussed by Whitworth (1978) in his review paper.

The measurements reported here, investigating the interaction between electrical properties and plastic deformation, have concentrated on the low-frequency range, the so-called space-charge dispersion, since the influence on the Debye dispersion seems to be very small and is difficult to measure.

2. EXPERIMENTAL

2.1. Crystal growth and sample preparation

Large pure ice monocystals were grown from deionized and distilled water (electrical conductivity at $25^\circ C \leq 10^{-5} \Omega^{-1} m^{-1}$, rising during growth due to dissolved air) by a modified Bridgman method (crystals with code K) or by a Stöber type method (crystals with code TK) (Noll, 1973). The etch-pit density determined in the usual way (Kuroiwa and Hamilton, 1963) was 5×10^3 to $1.5 \times 10^4 cm^{-2}$ for K-crystals, depending on the crystallographic plane. The density for the TK-crystals was higher on all planes by a factor 4 to 6, the values ranging from 2.5×10^4 to $6 \times 10^4 cm^{-2}$. The modified Stöber method, originally supposed to produce crystals with lower dislocation density by avoiding stresses from the vessel, failed in this respect. The density of Tyndall figures was 0.1 to 0.3 cm^{-3} for the K-crystals, $\leq 1 cm^{-3}$ for the TK-crystals.

The specimens for the uniaxial compression tests were prepared by Ruepp's method 3 (Ruepp, 1973, p. 179). For the shear tests the crystals were simply frozen onto the electrode plates.

2.2. Deformation tests

The specimens were deformed either by shear using an arrangement of the Bausch-type (Bausch, 1935; Steinemann, 1958) or by uniaxial compression, their orientation always being for glide in the basal glide system $(0001)[11\bar{2}0]$. With the design for the shear tests described and illustrated by Noll (1973), a relatively uniform gliding could be achieved, but it was

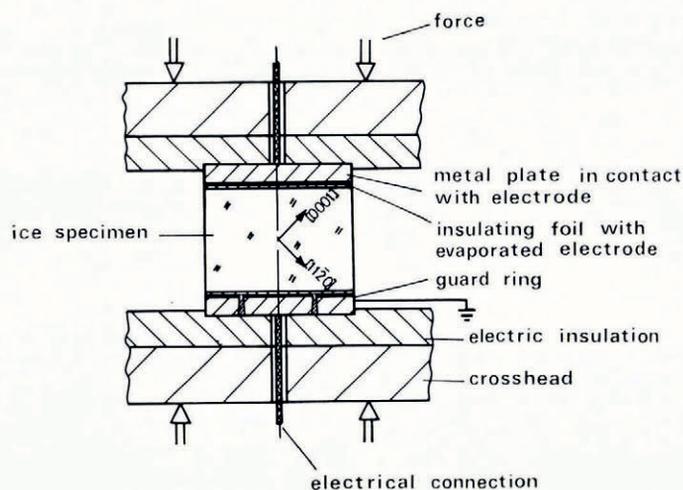


Fig. 1. Apparatus for uniaxial compression test.

difficult to maintain a perfect ice/electrode interface during deformation. This allowed only slow deformation rates at temperatures near to the melting point.

The uniaxial compression avoids such difficulties and has therefore often been used for plastic mechanical investigation (Griggs and Coles, 1954) and in combination with electrical measurements (Brill and Camp, 1957, 1961, p. 67–69; Higashi, 1969; Ackley and Itagaki, unpublished). Figure 1 shows the apparatus used for our uniaxial compression tests. Thin plastic foils (polyethylene or polyfluor(chlor)ethylene 3.5 to 8 μm thick) with evaporated silver electrodes on the outside surface, frozen on the end faces of the cylindrical ice specimen (40 mm \varnothing \times 40 mm), provided blocking electrodes and avoided air gaps between ice and electrodes (Ruepp, 1973). The plastic foils also made the glide planes rotate more easily on the electrode plates, because of the reduced adhesion.

All tests were carried out under constant strain-rate, the resolved shear strain-rate $\dot{\gamma}_s$ in the basal glide system ranging from 2×10^{-5} to $1 \times 10^{-3} \text{ s}^{-1}$, the resolved shear strain γ_s from 3 to 60%.

2.3. Electrical equipment and techniques

All specimens in the experiments had guard-ring electrodes forming a three-terminal capacitor. The impedance of the specimen was measured by a.c. methods described previously (Ruepp, 1973; Noll, 1973, unpublished). The known capacitance of the plastic foils in series with the ice was eliminated by calculation and then the frequency spectrum for the conductivity $\sigma(f)$ and dielectric permittivity $\epsilon(f)$ of the ice was derived. In addition to the a.c. methods a transient method was used to give a quick result over a wide frequency range; Figure 2 shows its circuit diagram.

A voltage step-function U is applied to the ice capacitor and the corresponding current transient $I(t)$ is measured by an electrometer (Keithley 610C) as a function of time t . The transient signal is recorded after passing a logarithmic converter (Hewlett Packard 7562A) either on a storage oscilloscope (Tektronix 564) or on a signal analyser (Hewlett Packard 5480A). This improved the signal-to-noise ratio by averaging several successive signals. The signals can finally be plotted on an XY -recorder. The guarded electrode is controlled at earth potential by the electrometer amplifier; therefore a good guard-ring measurement is

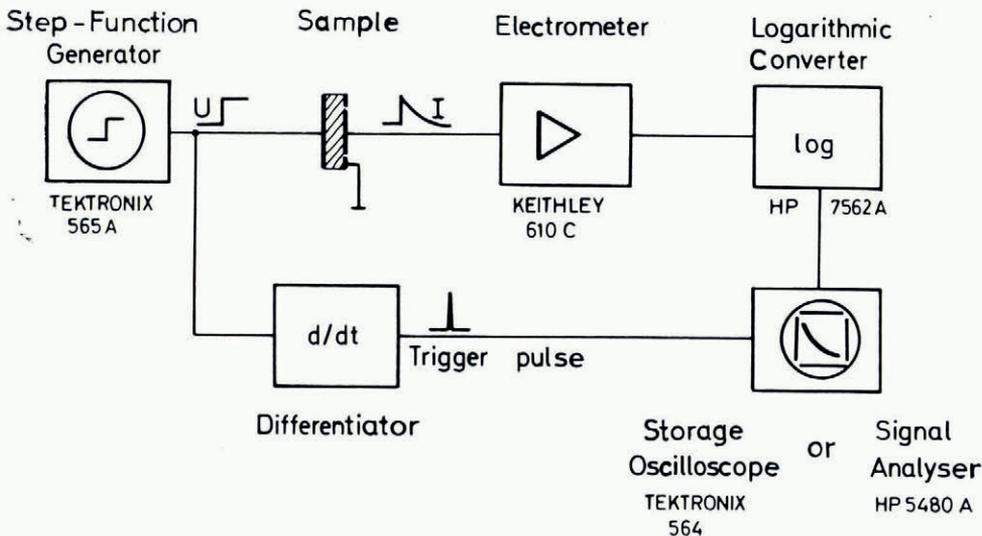


Fig. 2. Circuit diagram for transient measurements.

guaranteed. The measurement has been limited by the rise time of the amplifier, depending on the current range. After Fourier transformation and elimination of the foil capacitance the frequency spectrum $\sigma(f)$ and $\epsilon(f)$ could again be obtained. A variant of the transient method was used by Schenk (unpublished).

Three techniques were used to measure the trend of the dielectric properties during and immediately after the end of deformation: (1) σ and ϵ were recorded at a fixed frequency as a function of strain and time respectively (Noll, 1973). (2) With the transient method the frequency range < 100 Hz could be observed by a succession of measurements. (3) The whole spectrum was taken by quick measurements at various frequencies.

3. MEASUREMENTS AND RESULTS

3.1. Survey

Table I gives a survey of the samples, the deformation parameters resolved shear strain-rate $\dot{\gamma}_s$ and resolved shear strain γ_s , the age of the crystals, the time of annealing after sample preparation, and the symbols used in the figures. The temperature for the tests was $T = 270.2$ K, except for test D8(1).

TABLE I. SURVEY OF SAMPLES AND TEST CONDITIONS

Symbol	Test	Crystal	Temperature T K	Resolved shear strain-rate \dot{a} s^{-1}	Resolved shear strain a $\times 10^{-2}$	Age at time of preparation d	Annealing after preparation d
+	D 1	TK 14	270.2	3.0×10^{-5}	14.0	297	4.0
▲	D 3	TK 28	270.2	3.2×10^{-5}	27.4	310	20.0
▼	D 2	TK 31	270.2	5.2×10^{-5}	24.0	12	2.0
◆	D 6	TK 28	270.2	9.0×10^{-4}	62.0	646	34.0
○	D 7(1)	TK 28	270.2	1.3×10^{-3}	3.2	742	16.5
◐	D 7(2)	TK 28	270.2	1.3×10^{-3}	10.0	742	17.3
◑	D 7(3)	TK 28	270.2	1.3×10^{-3}	20.0	742	19.5
□	D 8(1)	K 37	263.8	2.6×10^{-5}	6.0	3 430	9.0
Y	S 17	TK 2	270.2	1.9×10^{-5}	16.0	124	11.0
人	S 18	TK 2	270.2	1.5×10^{-5}	21.0	162	6.0
×	S 19	TK 11	270.2	3.5×10^{-5}	22.0	6	11.0

3.2. Influence of deformation: qualitative description

First the influence of the deformation on the dielectric spectrum will be demonstrated, using the uniaxial compression test D3 as example. Figure 3 shows the resolved shear stress plotted against resolved shear strain $\tau_s = \tau_s(\gamma_s)$ for a deformation with constant resolved strain-rate $\dot{\gamma}_s$. The changes in the electric properties reach a nearly steady value when the stress τ_s is well over its maximum and approaches its steady value. The period during which the dielectric measurements shown in Figure 4 were taken is marked.

Figure 4 represents the dielectric spectrum in a double logarithmic plot; dielectric permittivity $\epsilon = \epsilon'$ and the conductivity $\sigma = \omega \epsilon_0 \epsilon''$ both as functions of frequency f (ϵ'' is the dielectric loss, ω the angular frequency $\omega = 2\pi f$, and ϵ_0 the permittivity of vacuum). The solid line shows the spectrum before deformation, after the sample has been annealed for 20 d at -3°C . The Debye dispersion D at frequencies above $f = 10^2$ Hz is well separated from the low-frequency space-charge dispersions R1 and R3 arising from blocking electrodes ($f < 10^2$ Hz). They all show the form of simple relaxation processes, each one characterized by one single relaxation time τ and one dispersion strength $\Delta\epsilon$ or, in terms of conductivity σ

$$\Delta\sigma = \epsilon_0 \Delta\epsilon / \tau. \quad (1)$$

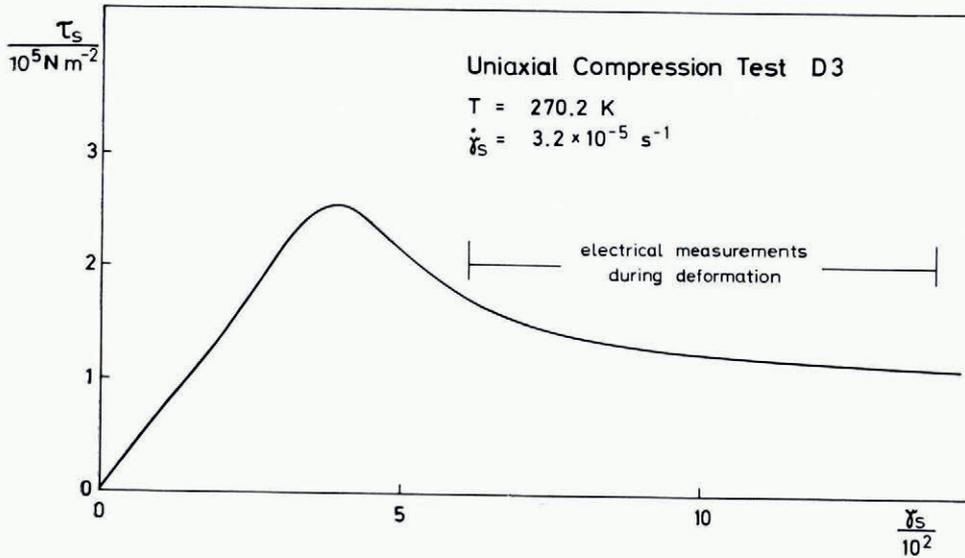


Fig. 3. Resolved shear stress τ_s against resolved shear strain γ_s , resolved shear strain-rate $\dot{\gamma}_s = \text{const}$.

(This holds even for the space-charge dispersions, because very weak electric fields have been applied and the dielectric response contains only the fundamental component. For these processes the time constant τ should be understood as a mean or basic relaxation time.)

The space-charge dispersion has a high-frequency limiting conductivity $\sigma_s = \sum \Delta\sigma_i$, representing the contributions of all slow processes to the conductivity and which is identical with the d.c. conductivity σ_0 .

The influence of the deformation is obvious in the low-frequency part of the spectrum: there the curves $\epsilon(f)$ and $\sigma(f)$ are shifted to lower values. The frequency position of the process R1 is hardly affected, the time constant τ_1 stays nearly constant. The dispersion strengths $\Delta\epsilon$ and $\Delta\sigma$ are decreased, resulting in a lower value of σ_0 . The changes remain at least partially after the deformation has ended and the shear stress relaxed to zero (this will be described later in Section 3.4).

The spectrum keeps its features during and after deformation and can still be characterized by the superimposed dispersions D, R1, and R3. The influence of the deformation will therefore be described in the following by the changes of τ , $\Delta\epsilon$, $\Delta\sigma$, and σ_0 , the emphasis being on σ_0 and process R1.

3.3. Influence of resolved strain-rate and resolved strain

3.3.1. d.c. conductivity

In Figure 5 the change $\delta\sigma_0$ of the d.c. conductivity (as indicated in Fig. 4) is plotted as a function of resolved shear strain-rate $\dot{\gamma}_s$ and resolved shear strain γ_s , respectively. It depends almost exclusively on the rate of deformation and not on the size of the strain. For very slow resolved strain-rate $\dot{\gamma}_s < 2 \times 10^{-5} \text{ s}^{-1}$ the d.c. conductivity decreased only slightly ($\delta\sigma_0 \approx -10^{-9} \Omega^{-1} \text{ m}^{-1}$) but it drops strongly when the deformation becomes faster: $\delta\sigma_0 \approx -10^{-7} \Omega^{-1} \text{ m}^{-1}$ for $\dot{\gamma}_s \approx 3 \times 10^{-5} \text{ s}^{-1}$. For still higher rates, the difference goes on falling, but not as steeply as before: $\delta\sigma_0 = -10^{-6} \Omega^{-1} \text{ m}^{-1}$ for $\dot{\gamma}_s = 10^{-3} \text{ s}^{-1}$.

The amount of strain has almost no influence on the change of σ (Fig. 5(b)). For a given strain-rate, strains between 3 and 60% produce within the limits of experimental error the same changes, which are determined by the parameter $\dot{\gamma}_s$ of the test experiment.

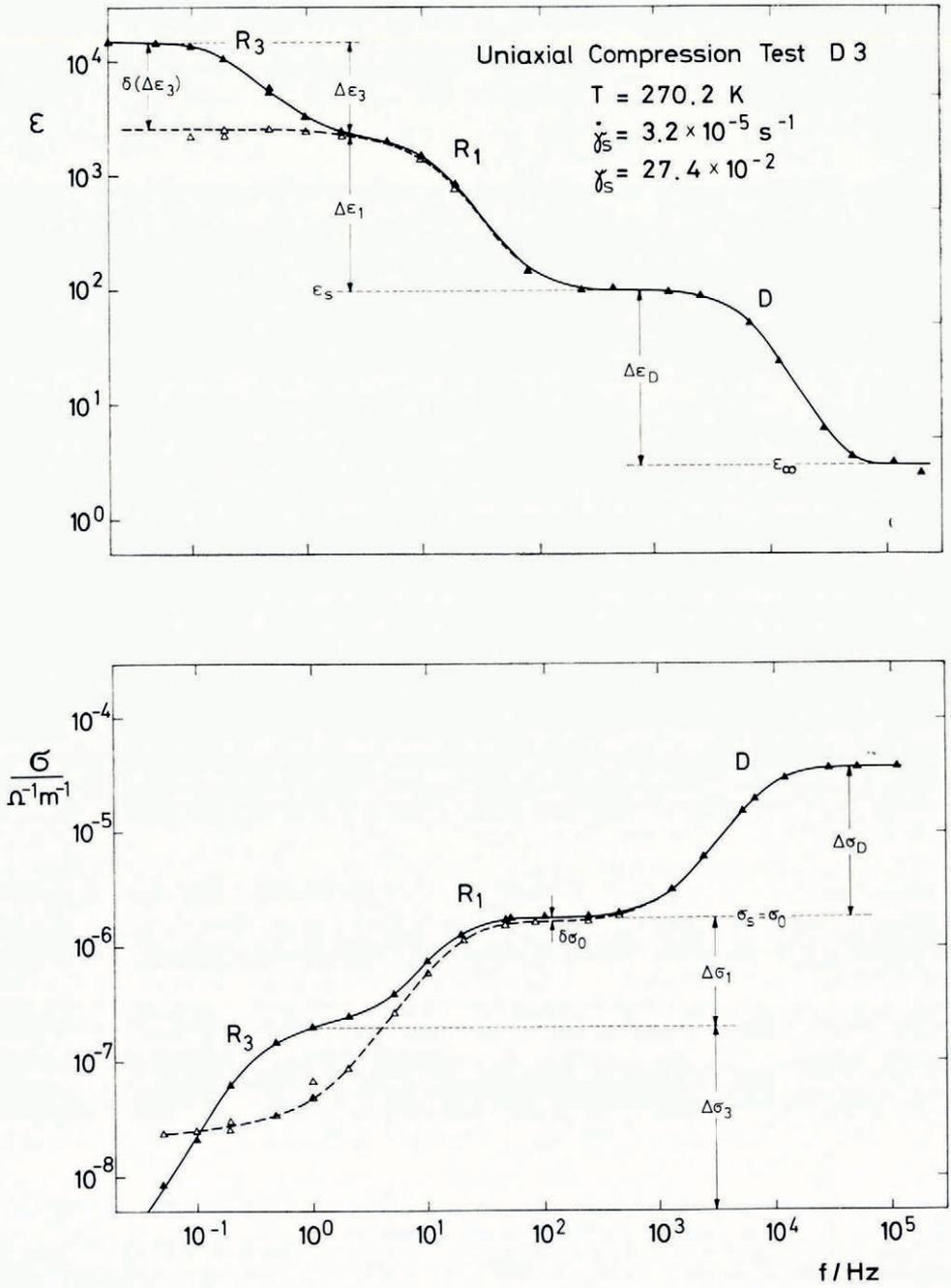


Fig. 4. Dielectric spectrum: frequency dependence of dielectric permittivity ϵ and electrical conductivity σ before (—▲—) and during (---△---) plastic deformation by uniaxial compression.

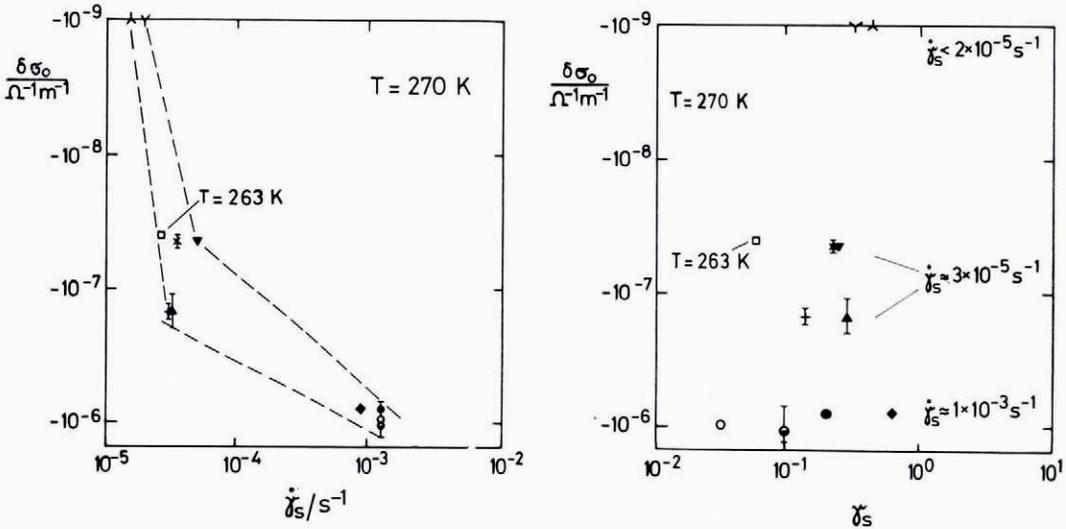


Fig. 5. Change of d.c. conductivity σ_0 : (a) as a function of resolved shear strain-rate $\dot{\gamma}_s$, (b) as a function of resolved shear strain γ_s . Symbols given in Table I.

3.3.2. Space-charge process R1

The frequency position of the process was only minimally affected by the deformation. In the majority of the tests, strains above a value of 4 to 10% resulted in a shift of a few hertz to higher frequencies ($\delta\tau_1 < 0$), but slight displacements to lower frequencies or no change at all also occurred. Since no systematic influence could be found, the time constant is assumed not to alter by deformation.

The changes of the dispersion strength $\Delta\sigma_1$ with resolved strain-rate and strain shown in Figure 6 behave in a similar way to those of the d.c. conductivity σ_0 (Fig. 5): $\Delta\sigma_1$ falls increasingly with higher strain-rates and the alteration is largely independent of the strain. The similar behaviour of $\delta(\Delta\sigma_1)$ and $\delta\sigma_0$ reflects the predominant contribution of process R1 to the d.c. conductivity.

For tests S17(Y) and S18(λ), $\delta(\Delta\sigma_3)$ was positive but very small ($\approx +5 \times 10^{-9}\text{ }\Omega^{-1}\text{ m}^{-1}$) and $\delta(\Delta\sigma_1)$ of similar magnitude and negative ($\approx -5 \times 10^{-9}\text{ }\Omega^{-1}\text{ m}^{-1}$), resulting in $\delta\sigma_0 \approx 0$. These two tests were the only ones with positive changes. This reflects the uncertainty for very slow deformations.

To derive $\Delta\epsilon_1$ from the measured $\Delta\sigma_1$ according to Equation (1), the actual τ_1 -values and their changes $\delta\tau_1$ have been used. This results in a greater scatter of $\delta(\Delta\epsilon_1)$ and even produces one small positive value (\times). For all other tests $\Delta\epsilon_1$ decreases with deformation (Fig. 7).

3.3.3. Space-charge process R3

This very slow process could not often be measured down to sufficiently low frequencies. Therefore the results obtained are only approximate. The relaxation frequency stays unaltered or again changes unsystematically to higher or lower frequencies. The dispersion strengths $\Delta\sigma_3$ and $\Delta\epsilon_3$ also change in either positive or negative direction for small strain-rate $\dot{\gamma}_s$. Both quantities fall if $\dot{\gamma}_s > 4 \times 10^{-1}\text{ s}^{-1}$; this value seems to be a threshold for systematic changes. In contrast to process R1, the strain itself influences the behaviour in this case.

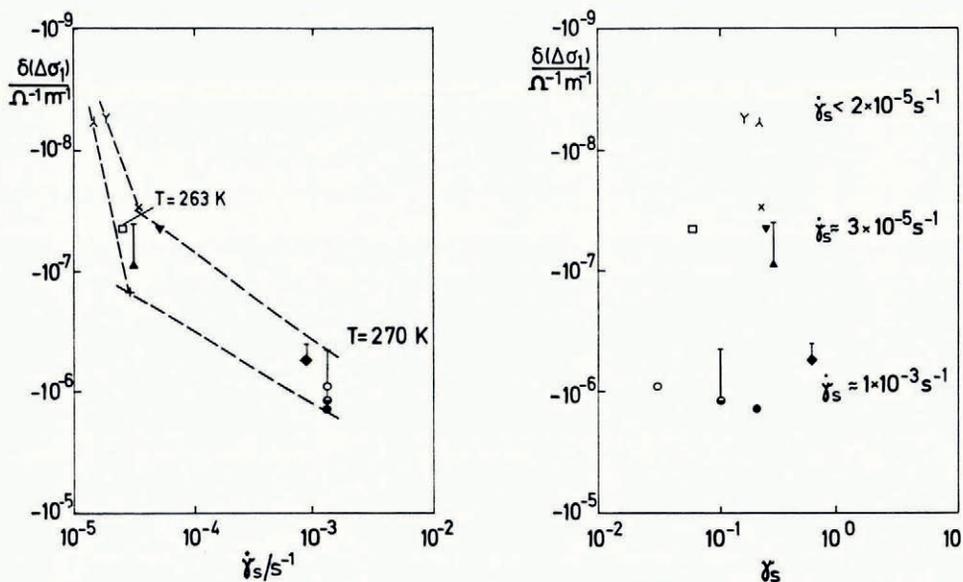


Fig. 6. Space-charge process R1: change of dispersion strength $\Delta\sigma_1$: (a) as a function of resolved shear strain-rate $\dot{\gamma}_s$, (b) as a function of resolved shear strain γ_s . Symbols given in Table I.

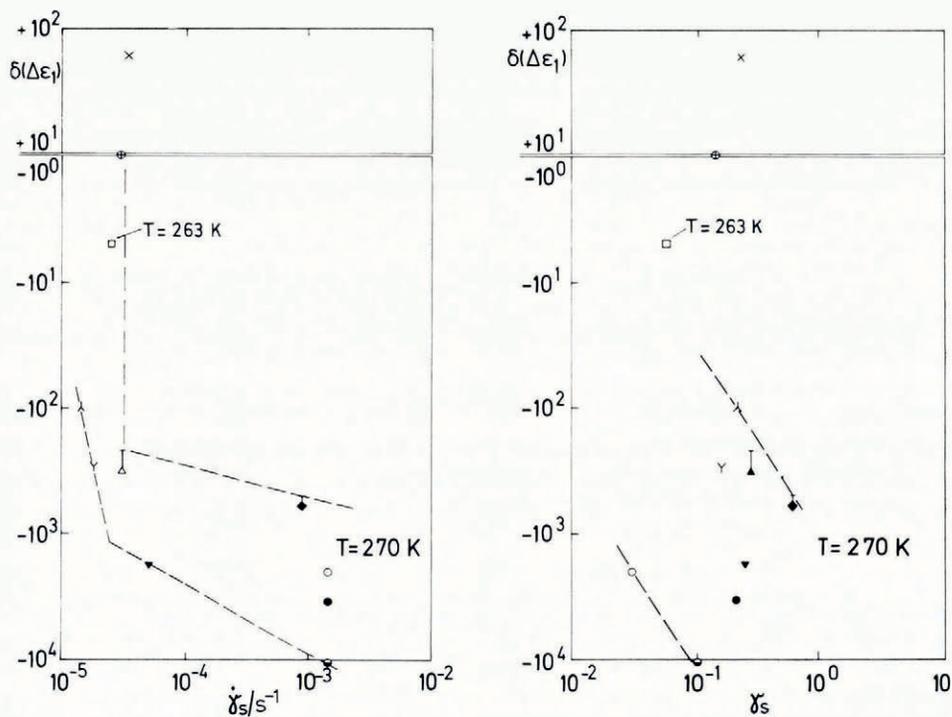


Fig. 7. Space-charge process R1: change of dispersion strength $\Delta\epsilon_1$: (a) as a function of resolved shear strain-rate $\dot{\gamma}_s$, (b) as a function of resolved shear strain γ_s . Symbols given in Table I.

3.4. Behaviour with time: recovery after deformation

The changes depend on the position of the frequency in the spectrum (cf. Fig. 4). In the space-charge region ϵ and σ alter steadily and monotonically with increasing strain. The alterations are proportional to the resolved strain as long as the resolved shear stress rises. The dielectric properties still change after deformation has ended. Figure 8 shows how the space-charge processes depend on time. After a small deformation (test D7(1), $\gamma_s = 3.2\%$) the perturbations of the parameters τ , $\Delta\sigma$, $\Delta\epsilon$ diminish rather quickly; some go beyond their original value. After a stronger deformation (D7(2), $\gamma_s = 10\%$) the parameters of process R1 change a little, the parameters of process R3 attain their final values after some hours. The recovery processes are essentially faster than the ageing processes observed before and to some extent after deformation.

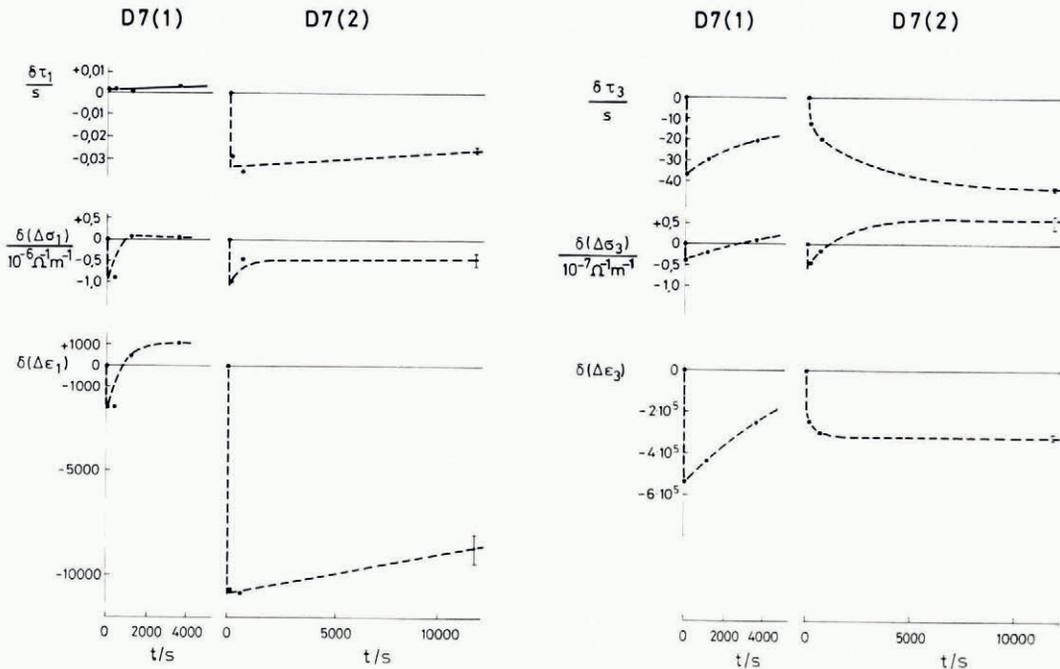


Fig. 8. Time dependence of the changes in the space-charge parameters after the end of deformation. Test D7(1) $\gamma_s = 3.2\%$, test D7(2) $\gamma_s = 10\%$, $\dot{\gamma}_s = 1.3 \times 10^{-3} \text{ s}^{-1}$ for both tests.

4. DISCUSSION

4.1. General aspects

The conspicuous result of plastic deformation on the electrical properties is the reduction of the conductivity in the whole space-charge range, i.e. below 100 Hz. The d.c. conductivity σ_0 and the strengths of both dispersions are diminished. The position of the dispersion in the spectrum, however, does not change significantly.

The electrical conductivity depends basically on the concentration c_i of the charge carriers i involved, their mobility μ_i and their charge q_i . A change of the charge q_i can hardly be imagined. The decrease of σ must therefore originate in smaller c_i or μ_i . But should this happen during deformation, if the charge carriers have contributed to the conductivity and have already been mobile before? As the concentrations c_i includes only active charge

carriers, its value can decrease by trapping mobile charge carriers in lattice imperfections created by the deformation. Charge carriers whose concentration is in thermal equilibrium, i.e. intrinsic ions or defects, would be produced by the lattice anew. A decrease would only be possible if their enthalpy of formation increases. Otherwise it is more likely that the conductivity is due to extrinsic charge carriers. The second possibility is a diminished mobility: either additional obstacles hinder the transport of charges, or previously favourable travel paths are destroyed by the deformation.

4.2. *Dependence on the resolved strain-rate*

The distinct dependence of the deformation effects on the resolved strain-rate (Fig. 5(a) and 6(a)) has to be seen in connection with the behaviour immediately after deformation. We may assume that generating and degenerating processes are already active during gliding (dynamic recovery). At low strain-rates the perturbations produced anneal while the deformation is still going on and major deviations from the original values are prevented. This explains the small effects found for slow deformations. During fast deformations the effects produced cannot be removed sufficiently. If the fast deformation is short (small resolved strain) the crystal recovers after the deformation has ended: the changes are reversible. For long lasting and rapid deformations (large resolved strains), however, large and permanent alterations of the spectrum are found.

During plastic deformation of ice monocrystals the resolved shear strain τ_s is also determined by generating and degenerating processes. The resolved shear strain γ_s , given by the test conditions, is opposed by the movement of dislocations (density ρ , Burgers vector b , velocity v) with a term depending on time t (see Weertman, 1973):

$$\tau_s \propto \gamma_s - \int_0^{\gamma_s} \rho b v dt, \quad (2)$$

or

$$\dot{\tau}_s \propto \dot{\gamma}_s - \rho b v. \quad (3)$$

The dislocation density ρ and the dislocation velocity v are functions of γ_s and τ_s respectively. The second term on the right-hand side of Equation (2) and Equation (3) can be neglected initially, when $\tau_s \propto \gamma_s$ (see Fig. 3). The dislocation density is then very small. With continuing deformation more and more dislocations are produced. When their contribution predominates, the resolved shear stress passes its maximum and decays with increasing strain to a steady-state value. From $\dot{\tau}_s = 0$ it follows that

$$\dot{\gamma}_s = \rho b v. \quad (4)$$

In the steady state the dislocation term $\rho b v$ is directly determined by the resolved strain-rate $\dot{\gamma}_s$; the size of the strain has no influence. The results described in Section 3.3 indicate that this term also determines the decrease of the electrical conductivity. The changes still remaining (at least partially) after the deformation stops, give a hint that the dislocation density plays the determining role, rather than the dislocation velocity. For slow deformations the product $\rho b v$ is small anyway (Equation (4)). During a fast but small strain the velocity v rises, but the density ρ hardly changes. In these cases the product still remains small and its influence decays quickly after the end of the deformation.

The strong influence of the resolved strain-rate and the independence of the strain can be understood. However, a model for the interaction between dislocation and charge carriers is still missing. Possibly it is not the dislocations themselves that are active, but point defects produced with them which anneal later.

4.3. Information from X-ray topography

Perez and co-workers have investigated the dislocation distribution in connection with their measurements of the internal friction (Perez and others, 1976) and ultrasonic propagation (Tatibouët and others, 1975). They used monocrystals, grown by a Bridgman method similar to ours and with a dislocation density of the same size as the etch pit density of our K-crystals, $\approx 10^4 \text{ cm}^{-2}$ (Tatibouët and others, 1975, p. 169; private communication from J. Klinger in 1975). The X-ray topographs of freshly grown crystals show a usual dislocation distribution. During ageing, a network structure develops due to aggregation of dislocations, which has still not reached its final state, even after one year at $T \approx 250 \text{ K}$. By plastic deformation and subsequent annealing it becomes more pronounced, leading to a cellular structure and polygonization (Vassoille and others, 1977).

From these observations it may therefore be concluded that the change of the dielectric spectrum by plastic deformation and the recovery after deformation is not only determined by the increased dislocation density, but also by the changing character of the dislocation network. This is additionally supported by our observations of ageing in the space-charge region of the spectrum. The initially complicated dispersion for frequencies below 100 Hz develops into two well-separated dispersions R₁ and R₃ during about 20 d after the preparation of the sample. During this ageing period the dispersion R₁ shifts to higher frequencies and its dispersion strength $\Delta\sigma_1$ and $\Delta\epsilon_1$ decreases, while at the same time the dispersion R₃ builds up and increases its dispersion strength $\Delta\sigma_3$ and $\Delta\epsilon_3$. This results in a gradual decrease of the plateau of the d.c. conductivity σ_0 . It seems that after 500 h the dispersion R₃ has nearly reached its final stage shown in Figure 4, but the dispersion R₁ and σ_0 are still decreasing. The differential behaviour of the space-charge dispersions might be attributed to the development of the cellular structure of the dislocation network, the two dispersions reflecting regions of the crystal with different density and character. Thus the plastic deformation seems to produce similar effects on the dispersion R₁ as does the ageing.

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DISCUSSION

R. TAUBENBERGER: Could you explain what you mean by "d.c. conductivity", since σ_0 is measured at about 10^2 Hz and below that one has a complicated spectral behaviour?

G. NOLL: The d.c. conductivity σ_0 simply denotes the value of the plateau in the spectrum $\sigma(f)$ at frequencies below the Debye dipole-rearrangement dispersion ($f \approx 100$ Hz). I have followed the usual nomenclature, even if I do not feel happy with it. "Static conductivity σ_s " (corresponding to "static permittivity ϵ_s ", which refers to the corresponding plateau in the spectrum $\epsilon(f)$) implies similar difficulties. I do not want to express a special underlying model by using this term.

TAUBENBERGER: Did you check if there is any correlation between the thickness of plastic foils and any of the strain-rate-induced changes of the parameters of R₃? Could it be that you are seeing mainly electrode effects?

NOLL: At the very low frequencies of the space-charge dispersion R₃, the foil contributes essentially to the impedance of the ice/foil combination. I have not sufficient experimental data in this frequency range to check the influence of the foil thickness and its change with deformation. In the frequency range of the dispersion R₁ and the conductivity plateau σ_0 , the foil plays only a minor part. The effects found there are greater than the possible changes of the foil capacitance. I would further expect such a change to be proportional to the strain, rather than to the strain-rate as observed.

TAUBENBERGER: Did you make comparisons between the development of your spectra on plastic deformation and the ones on ageing, both alone and together with hydrostatic pressure, that I published?

NOLL: In this paper I could only indicate the relationship between the influence of plastic deformation and ageing. The observed ageing effects in the space-charge region of my crystals show a similar behaviour to yours (Taubenberger, 1973). With the extended measurement range the dispersions could be measured to very low frequencies. The dispersion R₁ corresponds to the process called by you "low-frequency dispersion", the dispersion R₃ corresponds to your "blocking dispersion", which is just appearing at your lowest frequencies. These dispersions thus exist in crystals grown by different methods and in samples which are prepared in different ways and with different types of electrodes. The ageing behaviour of both dispersions is correlated. Hubmann has told me that he has investigated the ageing of the dispersion R₁ over a much longer time and confirmed the trend which was indicated in my measurements in the first hundreds of hours. The result of this is finally a completely changed spectrum at frequencies below 1 000 Hz, with a much lower conductivity. I also think that the ageing results make doubtful the interpretation of the conductivity σ_0 by intrinsic proton conductivity alone (Taubenberger, 1973).

TAUBENBERGER: It seems to me that the largest effect you have in your spectra is a tremendous shift of the lowest dispersion region R₃ to lower frequencies, whereas process R₁ stays stable. $\delta\sigma_0(D)$ then stems entirely from the decrease of $\Delta\sigma(R_3) \approx \epsilon_0\Delta\epsilon(R_3)/\tau(R_3)$, assuming that all the processes act in parallel. With this view, your changes of the spectra with strain resemble those I observed for R₃ (blocking dispersion) on ageing. The central common feature then was that $\Delta\sigma_1 = \epsilon_0\Delta\epsilon_1/\tau_1$ stayed largely constant. The relationships of your findings with ours on ageing should be further explored.

NOLL: The major contribution to the change of the d.c. conductivity σ_0 comes from the change of $\Delta\sigma_1$, which can be seen from Figures 5 and 6. The change of the dispersion R₃ may be impressive for the process itself, but contributes little to the conductivity σ_0 .

TAUBENBERGER: I think one has to bear in mind whether one wants to analyse the changes in the two relaxation regions as stemming from processes acting in parallel or in series. One has to find out which hypothesis for process superposition is more consistently described by the spectra and its changes, which in turn have to be linked to a hypothesis for the underlying microscopic process itself.

NOLL: I agree with you that an explanation for the complicated spectrum in the space-charge region and its changes with deformation depends on the kind of interaction of the processes. To clarify this, further experimental data are needed, e.g. temperature dependence, and a further study of the ageing effects is essential too.

TAUBENBERGER: Do your values of $\Delta\epsilon(D)$ fit with or without an anisotropy $(\Delta\epsilon_{\parallel} - \Delta\epsilon_{\perp})/\Delta\epsilon$ of the so-called Debye dispersion D?

NOLL: An anisotropy of the permittivity of the Debye dispersion could not be investigated with the crystal orientations used for the deformation tests.

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