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IN-SITU MEASUREMENTS OF THE DENSITY OF AMORPHOUS
GERMANIUM PREPARED IN ULTRA HIGH VACUUM

by

Petr Višcor

(Submitted to Journal of Non-Crystalline Solids 1987)

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ABSTRACT.

Non-destructive, in-situ technique⁽¹⁾ has been used to determine the density of amorphous germanium films, prepared in ultra high vacuum by slow sublimation onto heated single crystal sapphire substrates. Contrary to the previous results, a density deficit with the respect to the crystal has been observed. It is pointed out that the density deficit with respect to the crystalline phase (where such exists) is a general feature common to all bulk glasses and that it should be considered also as a characteristic structural feature of a stable state amorphous germanium. The apparent thickness dependence of the measured density is discussed in terms of a single relaxation mechanism.

IN-SITU MEASUREMENTS OF THE DENSITY OF AMORPHOUS

GERMANIUM PREPARED IN ULTRA HIGH VACUUM

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Non-destructive, in-situ technique⁽¹⁾ has been used to determine the density of amorphous germanium films, prepared in ultra high vacuum by slow sublimation onto heated single crystal sapphire substrates. Contrary to the previous results, a density deficit with the respect to the crystal has been observed. It is pointed out that the density deficit with respect to the crystalline phase (where such exists) is a general feature common to all bulk glasses and that it should be considered also as a characteristic structural feature of a stable state of amorphous germanium. The apparent thickness dependence of the measured density is discussed in terms of a single relaxation mechanism.

I. INTRODUCTION

An understanding of the electronic properties of amorphous semiconductors and insulators⁽²⁾ requires a detailed knowledge of the structure of the investigated material, usually obtained from the diffraction experiments, using

either electrons, X-rays or neutrons. An important parameter in the analysis of the diffraction data and in the subsequent structural models is the density. While it has been known and accepted that in the proper bulk glasses (materials prepared in bulk form by quenching the melt such as chalcogenide glasses, silica etc.) a stable, glassy state is appreciably less dense than the crystalline counterpart, a lot of controversy surrounded the group IV elemental amorphous semiconductors. It has been thought that contrary to the case of glassy materials, no density deficit exists in amorphous germanium (a-Ge) and silicon, when prepared on to heated substrates⁽³⁾ or when annealed to temperatures just below the crystallization⁽⁴⁾. The experimental evidence so far has been rather contradictory, some results indicating no appreciable density deficit relative to the crystalline phase^(3,4,5) while other observations⁽⁶⁾ showed that amorphous semiconductor films seem to be quite less dense than their crystalline counterparts.

In the previous attempts to clarify this important point concerning the atomic structure of amorphous germanium a concept of voids (empty spaces in the amorphous matrix) was usually used in order to explain a finite density deficit in some as-deposited films and their subsequent densification upon annealing (annealing temperatures still below the crystallization temperature) up to the crystalline density. This model, if correct, would represent a situation quite different to that found in bulk glassy materials, where a stable,

disordered phase is usually characterised by a density deficit of up to 20-25% relative to the crystalline phase without invoking the existence of voids.

In order to try to understand the above mentioned contradicting results, they must be put in relation to other measured physical properties and the external factors that control them. It has been pointed out⁽⁷⁾ that the most important parameters controlling the physical properties of amorphous germanium (but also of other thin amorphous films) are substrate temperature, evaporation rate and the cleanliness of the vacuum environment during the deposition. A classification scheme has been suggested to distinguish between films prepared on heated substrates ($T_s \geq 400$ K) using low evaporation rates (class A films) and those prepared on low temperature substrates ($T_s < 300$ K) using higher evaporation rates ($> 5 \text{ \AA sec}^{-1}$) (class B films).

While a degree of inhomogeneity has been detected in films of class B either through the direct high resolution electron microscopy⁽⁸⁾ and/or small angle scattering⁽⁹⁾ (a characteristic "columnar" structure on 10-40 Å length scale), no observable inhomogeneities (of size $> 5-8 \text{ \AA}$) are observed in class A films^(7,8). Since it is these films that represent a stable, disordered state of amorphous germanium (a-Ge), which does not seem to show any changes in its physical properties (electrical and optical)⁽⁷⁾ on annealing it has been a purpose of the present investigation to measure the density of class A films.

To avoid various systematic error sources in the usual density determination, an in-situ technique⁽¹⁾ has been employed to enable the measurement of the density of investigated films to be performed without ever exposing the films to ambient pressure and/or physically removing the films from the ultra high vacuum system. The results of this investigation are reported here.

II. SAMPLE PREPARATION AND EXPERIMENTAL TECHNIQUE

The experiments were performed in the ultra high vacuum (UHV) system with the base pressure of 1.10^{-10} torr after several days bake-out. High purity germanium (10^{13} electrically active impurities per cm^3) was thoroughly cleaned (1 part of HF, three parts of HNO_3), carefully rinsed in the doubly distilled water and placed in the tungsten evaporation coil (the coil was degassed under UHV conditions in the previous pump down), shown in Figure 1. Amorphous germanium films were deposited onto single crystal sapphire substrates, (substrate temperature during the evaporation was 400 K) which were secured onto a sample block, attached to a cold finger of the rotatable liquid helium cryostat. The evaporation rates of approximately 0.1 \AA sec^{-1} were used and the pressure in the UHV chamber was never allowed to rise above 5.10^{-10} torr during the preparation of the films. Two quartz crystal oscillators allowed continuous monitoring of the evaporation rates.

Once the film of required thickness was deposited, the total change in the quartz oscillator resonant frequency was recorded and the cryostat was rotated so that simultaneous measurements of the optical reflectance R and transmittance T could be made. These measurements are normally used in order to determine the optical constants of investigated films as a function of incoming photon energy E , with film thickness either as a free parameter or a fixed parameter to be determined interferometrically ex-situ. For lower values of optical absorption however, the interference effects demonstrate themselves clearly also in $R(E)$, $T(E)$ data as alternating maxima and minima for particular values of E_{int} . Using these values ($R_{Max}(E_{int})$, $T_{min}(E_{int})$) together with the appropriate expressions for R and T in terms of the optical constants of the investigated film and of the substrate (refractive index $n(E)$) and the extinction coefficient $k(E)$) it is then possible to determine the values of $n(E_{int})$, $k(E_{int})$ and the thickness t of a given film selfconsistently in-situ. A non-destructive, in-situ technique⁽¹⁾ used in this work to determine the density of investigated a-Ge films utilises both the total change in the resonant frequency Δf of the quartz crystal oscillator and selfconsistently determined thickness t of the deposited film (R, T measurements). While the quantity Δf is proportional to the mass of the deposited film, the thickness t of the film, obtained through the analysis of the optical measurements is a measure of its volume. The density of the film is then proportional to the ratio of the

two quantities⁽¹⁾

$$\rho = K \cdot \frac{\Delta f}{t} \quad (1)$$

where ρ is the density of the measured film, K is the constant of proportionality (to be determined by calibration), Δf is the total change in the resonant frequency of the quartz crystal oscillator and t is the thickness of the film. As can be seen from the equation⁽¹⁾, once the proportionality constant K is determined independently, a density of any unknown film can be measured non-destructively, directly and in-situ by noting the change Δf in the resonance frequency of the quartz crystal oscillator (\sim mass of the film) after the deposition and the thickness t (\sim volume of the film) through the analysis of the R, T-measurements. For calibration purposes however, both sides of equation⁽¹⁾ have to be determined independently and for this purpose the films have to be removed from the vacuum system. It was for this reason that gold films of various thicknesses were used for calibration (gold is a noble metal, which neither oxidises, nor absorbs water vapour when exposed to ambient pressure). They were deposited in the UHV - system, (in precisely the same way as a-Ge films) onto sapphire substrates, which were weighted prior to the insertion into the UHV system. After deposition, the substrates with gold films were removed from the UHV chamber and the density of the films was measured directly. The mass was measured by weighing the substrates after the deposition and subtracting the weight of uncoated substrates (Cahn 200 model electromicrobalance with resolution of

0.1 μg was used). The volume of the deposited films was determined by measuring the area (using the optical travelling microscope) and the thickness t (interferometrically). Noting also the total change Δf of the quartz crystal oscillation frequency for each gold film, it was then possible to determine the constant K in equation (1).

Results of the calibration measurements are shown in Figure 2, where measured density ρ is plotted against measured ratio $\frac{\Delta f}{t}$ for a number of gold films. The slope of the line determines the constant of proportionality K in equation (1). It was found to be equal to a value of

$$0.435 \pm 0.010$$

It should be pointed out that the precision of the density measurements is given almost entirely by the precision of weighing the gold calibration films. This is reflected in the quoted error of ± 0.01 in the constant K . The errors in Δf and t can be considered to be of the second order relative to the errors in weighing.

III. RESULTS

From five a-Ge films of different thicknesses that have been investigated, four belonged to class A and one to class B films. After each film was deposited values Δf and t in equation (1) were determined through the noted oscillation frequency change of the quartz crystal oscillator and through the analysis of in-situ R,T measurements respectively. Using the constant K (determined through ca-

libration using gold films) the density of a given a-Ge film was then calculated using equation (1). The results are displayed in Figure 3, where the density of the four class A films are plotted as a function of their thickness. For comparison a crystal density is also displayed. Also indicated is the density of a-Ge film of class B (room temperature substrate, higher evaporation rate of 10 \AAsec^{-1} and unbaked UHV system with the background pressure in the region of $1-5 \cdot 10^{-6}$ torr).

The results of the present investigation displayed in Figure 3 can be summarised in the following two points:

- 1) All measured a-Ge films are appreciably less dense than the crystal.
- 2) The measured density in class A films depends apparently on their thickness.

These two points will be now discussed in more detail in the following paragraph, where various structural models (in particular the "void" hypothesis) will be contrasted with the available experimental evidence.

IV. DISCUSSION

For thicknesses between 500 Å and 8500 Å, the measured density deficit (relative to the crystalline density) in investigated a-Ge films varied between 24% and 2%. Leaving for a moment a question of the apparent thickness dependence of the density deficit the important new result

contradicting the previous investigations^(3,5,10) is that an amorphous state of germanium is characterised by a definite density deficit relative to the crystalline phase. The usual explanation of this kind of density deficit in a-Ge has been up till now the presence in the films of voids and/or void networks ("columnar" networks on the length scale of some 10-40 Å). Here it has to be pointed out however that the most of present results have been obtained on films of class A, where voids and/or void networks of sizes just mentioned are expected not to be present⁽¹⁰⁾.

To check the "void" hypothesis, the thin films of the present work were also investigated by "in-situ"⁽⁷⁾ high resolution electron microscopy. Figure 4 shows a typical bright field high resolution electron optical image of the studied films of class A. As can be seen from Figure 4, the films are homogeneous down to the "resolution limit" (smallest void or other density deficient spatial region in the amorphous matrix still giving enough contrast above the noise level⁽¹¹⁾) of some 5-8 Å. Since, as can be seen from the results in Fig.3, it was the thinnest films that showed the largest density deficit, it can be concluded on the basis of the results represented in Fig.4 that the "void" hypothesis seems to be invalid for a-Ge films of class A. For the "void" hypothesis to be valid in the present case, one would have to postulate a large density (some 20% of the total volume) of microscopically small ($\lesssim 5$ Å) voids to be distributed in the amorphous germanium atomic network.

A term "void" or a defect of this microscope size (a missing atom and/or missing bond), occurring with such a large density looses clearly its meaning and the microscopic "empty" spaces would have to be considered as an integral part of the atomic network itself. A more plausible explanation therefore seems to be that the atomic structural network of amorphous germanium is simply more opened (less dense packing of atoms than that of germanium crystal). This is in accordance with similar observations and conclusions coming from the investigations of bulk, glassy semiconductors and insulators such as chalcogenide glasses, silica and others.

Dismissing the "void" hypothesis for class A amorphous germanium films does not mean that it may not be valid for class B a-Ge films. A part of the measured (see Fig.3) large density deficit (some 24%) relative to crystalline density) may be due to the columnar network seen in this type of films using electron microscopy⁽⁸⁾. However, due to lack of direct correlations between electron microscopy studies⁽⁸⁾ and those of small angle scattering⁽⁴⁾ a definite conclusion about the possible effect of the inhomogeneous nature of class B a-Ge films on the overall density can not be made at present and a more systematic and detailed investigation combining both techniques is clearly needed. It should be mentioned at this point also that a simple calculation shows that in order to account for a large density deficit in class B a-Ge films (~ 24%) through the columnar network visualised as a three-dimensional network of tu-

bular columns (assuming no atoms in them) is not sufficient. In order to obtain an agreement between the inhomogeneous model (void hypothesis) and the overall density measurements, the columnar network structures seen in the electron microscope would have to be considered as two-dimensional projection (in the kinematic approximation to the contrast formation from a disordered film) of "grain - boundary"-like low density regions connecting the high density amorphous "grain"- like spatial regions (of some 40 Å in diameter).

The present results indicate therefore that while it is possible that the inhomogeneous nature of class B a-Ge films may play some role in determining both the overall density and the density change (densification) through the structural relaxation on annealing (annealing out of the "void" network), it is the underlying amorphous matrix itself that is characterised by an open atomic structural network with overall density below that of a crystal. One more point seems worthwhile mentioning in this context. It is a qualitatively different correlation that exists between the annealing behaviour (as seen through the d.c. electrical conductivity and the optical absorption measurements) in class A and B films. The d.c. electrical conductivity $\sigma_{d.c.}$ decreases by orders of magnitude ("parallel shifts" of the $\log \sigma_{d.c.}$ versus temperature $T^{-1/4}$) and the optical absorption edge shifts to higher energies ("sharpening" of the optical absorption edge). The important point to note is that both these shifts tend to bring the $\sigma_{d.c.}$ and the optical absorption coefficient α to values cha-

racteristic of class A films. Further annealing at higher temperatures however does not lead to any further changes in these two quantities indicating the stabilisation of the system (at least as seen through the d.c. electrical and optical measurements). It seems therefore logical to conclude that class A films seem to represent a "final, glassy" state of a-Ge into which various class B films tend to anneal.

V. THE THICKNESS DEPENDENCE OF THE DENSITY

A new and unexpected result apparent in Figure 3 is that there seems to be an apparent correlation between the measured density and the thickness of the investigated films of class A. The one film of class B investigated in the present work clearly does not follow this correlation. Also outside lies 1100 Å thick film of class A. The explanation of this apparent correlation based on the effect of the substrate on the overall density of the investigated films is quite unlikely in view of the fact that most properties of solids and amorphous materials in particular seem to be determined by short range effective one-electron interactions. Effects of the substrate which would be felt up to the thicknesses of a micron must be therefore dismissed. A more plausible explanation must be the one based on some structural relaxation model. Bearing in mind that all class A films investigated in the present work were deposited at 400 K and they were held at 400 K only during the deposition for a known length of time, it is then possible to replot the Figure 3

into a plot of the measured density as a function of annealing (deposition) time. This has been done in Figure 5 and it can be seen straight away that all four films of class A investigated in the present work are now lying on a simple curve.

Using a single relaxation time model for the density relaxation of a given film in a form

$$\rho = \rho_0 - \beta_s e^{-t/\tau} \quad (2)$$

(ρ_0 is the final, relaxed density; β_s is short time density deficit and τ is the relaxation time),

and taking into account the fact, that the various parts of the film stay at deposition temperature for different lengths of time during the film preparation, the expression for the density of the film after the deposition time t_{dep} reads

$$\rho = \rho_0 - \beta_s \left(\frac{1 - e^{-t_{\text{dep}}/\tau}}{t_{\text{dep}}/\tau} \right) \quad (3)$$

where the relaxation time τ is defined as

$$\tau = \frac{1}{\omega_0} e^{\Delta E/kT} \quad (4)$$

Here ω_0 is the optical phonon frequency and ΔE is the activation energy of the process responsible for the relaxation of the film density

Although the density measurements were only of a qualitative nature (a relatively small number of thicknesses measured) a rather unambiguous fit to the data in terms of a simply activated relaxation process was possible, leading

to values of $\rho_0 = 5,30 \text{ g}\cdot\text{cm}^{-3}$, $\beta_s = 1,32 \text{ g}\cdot\text{cm}^{-3}$ and $\Delta E = 1,43 \text{ eV}$ in equation two. Fitting the equation three to the data in Figure 4 gave similar values for the fitting parameters. In particular, the activation energy ΔE was found to be almost unchanged ($\Delta E \sim 1,41 \text{ eV}$). Both fits are shown in Fig.4 together with the predicted relaxation curve for samples prepared and/or annealed at room temperature.

As can be seen from Fig.4, the mass density relaxation process seen in films of class A when prepared at 400 K is completely frozen out at room temperature (relaxation time of the order of 500 years). It is now clear why the film of class B studied in the present work yielded a large density deficit which did not relax during the entire length of the experiment (some three weeks).

The simple relaxation process with a simply activated single relaxation time which seems to explain all the data presented in Fig.4 supports the notion that the densification process seen in a-Ge above room temperature involves relatively high, single activation energy, comparable in magnitude to the Ge-Ge sp_3 bond strength ($\sim 1,74 \text{ eV}$).

Due to the qualitative nature of the results it is not possible at present to make definite statement about the final, fully relaxed density of a-Ge films. Indication is however that the final density deficit lies between one and three percent relative to crystal. It has to be stressed that at no point during the investigation of class A films has any degree of crystallinity been de-

tected either through d.c. electrical transport, Raman measurements or direct electron diffraction and/or electron microscopy. The data on both class A (high deposition temperature) and class B (room temperature deposition) films indicate also that unannealed (short time) density of a-Ge is some 24-28% smaller than the Ge-crystalline density. If the proposed simple model for the density relaxation in a-Ge (equations (2), (3) and (4)) is valid generally (the density of a given film given by its temperature/time history) regardless the way of preparation and the vacuum conditions (giving the levels of impurity concentrations), it should be then possible to plot all the observed a-Ge densities published in the literature onto the appropriate temperature/time relaxation curves in Fig.5. All the available experimental data⁽⁶⁾ lie indeed in the density interval between 3.9 and 5.2 g.cm⁻³ predicted by the present model. For the deposition temperatures at and around 300 K (time scales unimportant) the agreement between the observed values and those predicted by the model is very good (see Fig.5) despite a certain degree of scatter^(3,4) (the density deficit measured was found to be only 10-15% as compared to predicted 25%; here the possibility of an increased substrate temperature and therefore an increased degree of density relaxation during the deposition has to be further clarified).

The other two measurements^(5,10) employ a more indirect method to measure the density (X-ray linear absorption coefficient measurements) and will not be discussed here although

even here the measured densities (the density deficit of some 2-5%) fall within the bounds given by the present model. It should be also mentioned that the deposition temperature in ref.5 was 77 K and therefore a different starting density and the subsequent density relaxational behaviour might be expected (point to be discussed later). It is believed that due to atomic immobility at 77 K, the formed initial atomic structure could be viewed as a more tightly packed network of molecular-like structural units⁽¹²⁾ (reflecting the tendency to form a molecular-like solid at these temperatures). However it is clear that much more detailed quantitative data is needed here. The experiments should involve simultaneous measurements of the density (through in-situ structural investigations) and other physical properties (d.c. and the a.c. electrical transport in particular).

Having established the density dependence of the investigated films on the deposition and/or annealing time and temperature, a correlation should be now made with other measured physical properties and in particular with the d.c.-electrical transport and optical measurements.

Here, contrary to expectations no changes in the d.c.-electrical conductivity were observed in any of four measured films of class A (absolute value of the conductivity $\sigma_{D.C.}$ and its temperature dependence was found to be the same - within the experimental error - in all four films of class A shown in Figure 5) although the density in these films varied between 23 and 2 percent. This is a very im-

portant result indicating that the electrical transport measurements seem to be unaffected by the density changes in class A films. The measured class B film on the other hand showed the conductivity some three orders of magnitude larger than in the films of class A in accordance with the general behaviour of class B films⁽¹³⁾. As mentioned previously the conductivity in class B films decreases on annealing towards the values characteristic of class A films ("parallel shifts" of the log of the d.c. electrical conductivity $\sigma_{d.c.}$ versus temperature $T^{-1/4}$). It seems therefore likely that another relaxation process (more directly related to the $\sigma_{d.c.}$) is responsible for this behaviour. In this context it is worthwhile mentioning that the studies of the annealing behaviour of the $\sigma_{d.c.}$ in a-Ge films deposited on substrates held at temperatures between 77 and 300 K during the deposition⁽¹⁴⁾ show that a simple relaxation model involving a single relaxation time τ (a single activation energy ΔE in eq. (4)) is not sufficient to explain the experimental observations. A relaxation model was proposed which is based on a Boltzmann-like exponential distribution of the activation energies. This model is then capable of explaining the relaxational behaviour of the d.c. electrical conductivity $\sigma_{d.c.}$ involving a wide range of substrate temperatures, annealing temperatures and annealing times.

Turning now to optical properties these were also investigated by in-situ optical reflectance $R(E)$ and transmittance $T(E)$ at normal incidence as a function of photon ener-

gy E. Here it was found out that the onset of the optical absorption (optical absorption edge) did not move appreciably for films of class A indicating that no significant changes in the electronic band structure occurred. The low energy ($E \lesssim 0.5$ eV) refractive index n on the other hand did show a definite decrease ($n(E \lesssim 0.5$ eV) ≈ 3.6) for thin films (~ 500 Å) relative to thicker films and relative to the crystalline value ($n = 4.0$). This result can be interpreted as due to mere mass density deficit without having to involve any appreciable changes in the optical matrix elements. In the investigated film of class B on the other hand, the low energy refractive index was found to lie above the crystalline value indicating that despite an appreciable density deficit the weight of the optical absorption shifted to lower energies and therefore a change in the underlying electronic band structure relative to films of class A seems to be involved (optical sum rule). On annealing the refractive index tends to decrease to lower values and it is believed that it is this change that is related to the d.c.-electrical conductivity relaxational behaviour generally observed in class B film (discussed above).

It has to be stressed however that before any definite conclusions can be drawn, more quantitative investigations have to be carried out. Such a study using the UHV-system is rather difficult to undertake mainly because of the complexity of each single experiment when performed under UHV-conditions and in-situ. It seems however that some of the qualitative trends observed in the present investigation should not or might not depend on the UHV-conditions and in-situ

nature of various experiments making the ex-situ quantitative studies possible and feasible.

VI. CONCLUSIONS

Using a non-destructive, in-situ technique, the density of stable, amorphous germanium films, prepared by slow evaporation onto heated sapphire substrates in ultra high vacuum, was measured. Contrary to the previous results, the density found was lower than that of the crystal. The density deficit dependence on the thermal history of investigated films was explained by a simple relaxation process involving a single activation energy of 1.42 eV. This dependence was found to be unrelated to the d.c. electrical properties in class A films, but could explain some changes in the low energy refractive index in these films.

VII. ACKNOWLEDGEMENTS

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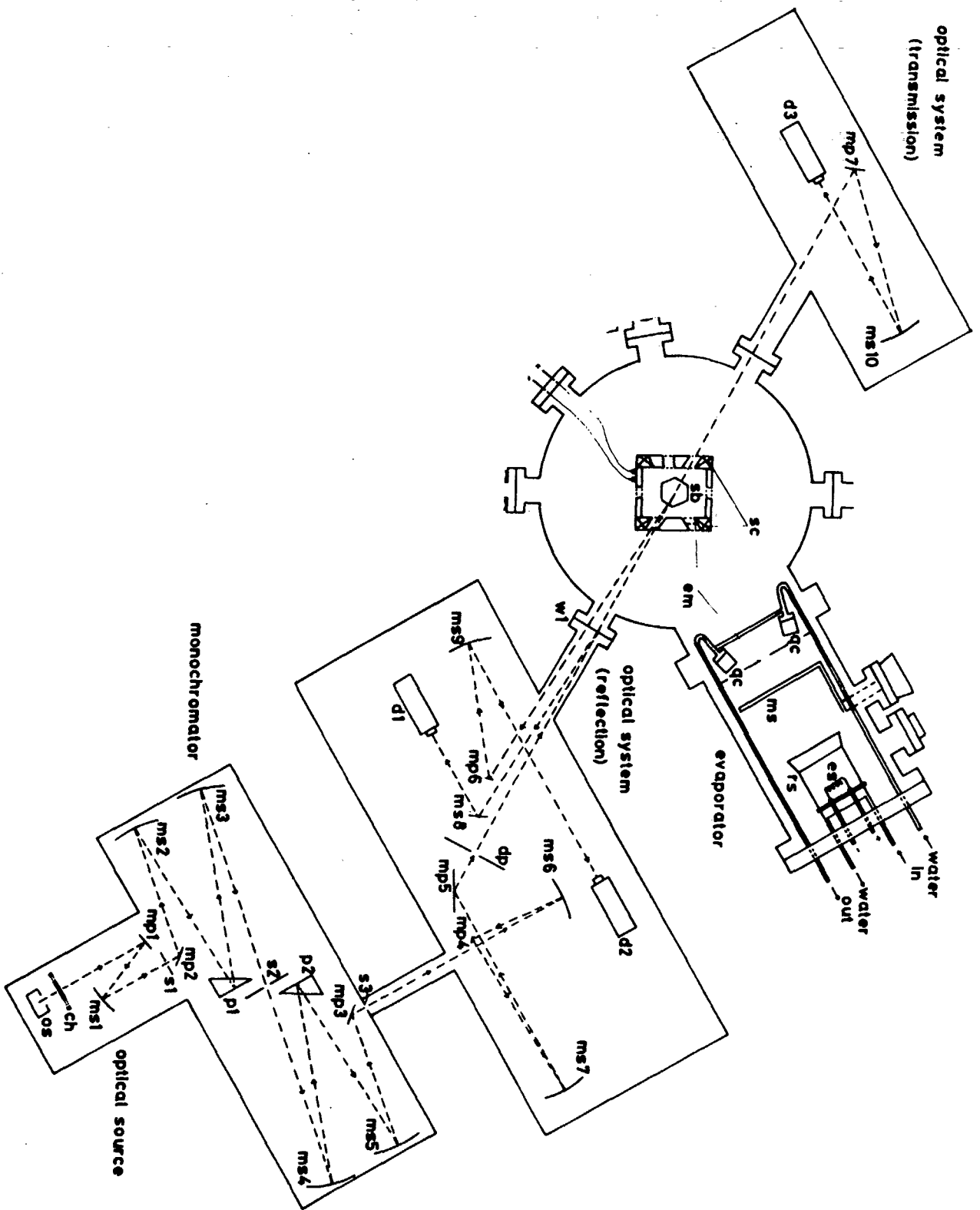
The author would like to thank also N.B. Olsen for bringing to his attention both the unpublished data on the d.c. electrical conductivity annealing behaviour in amorphous germanium films (class B) and the existence of non-exponential relaxation model to explain these.

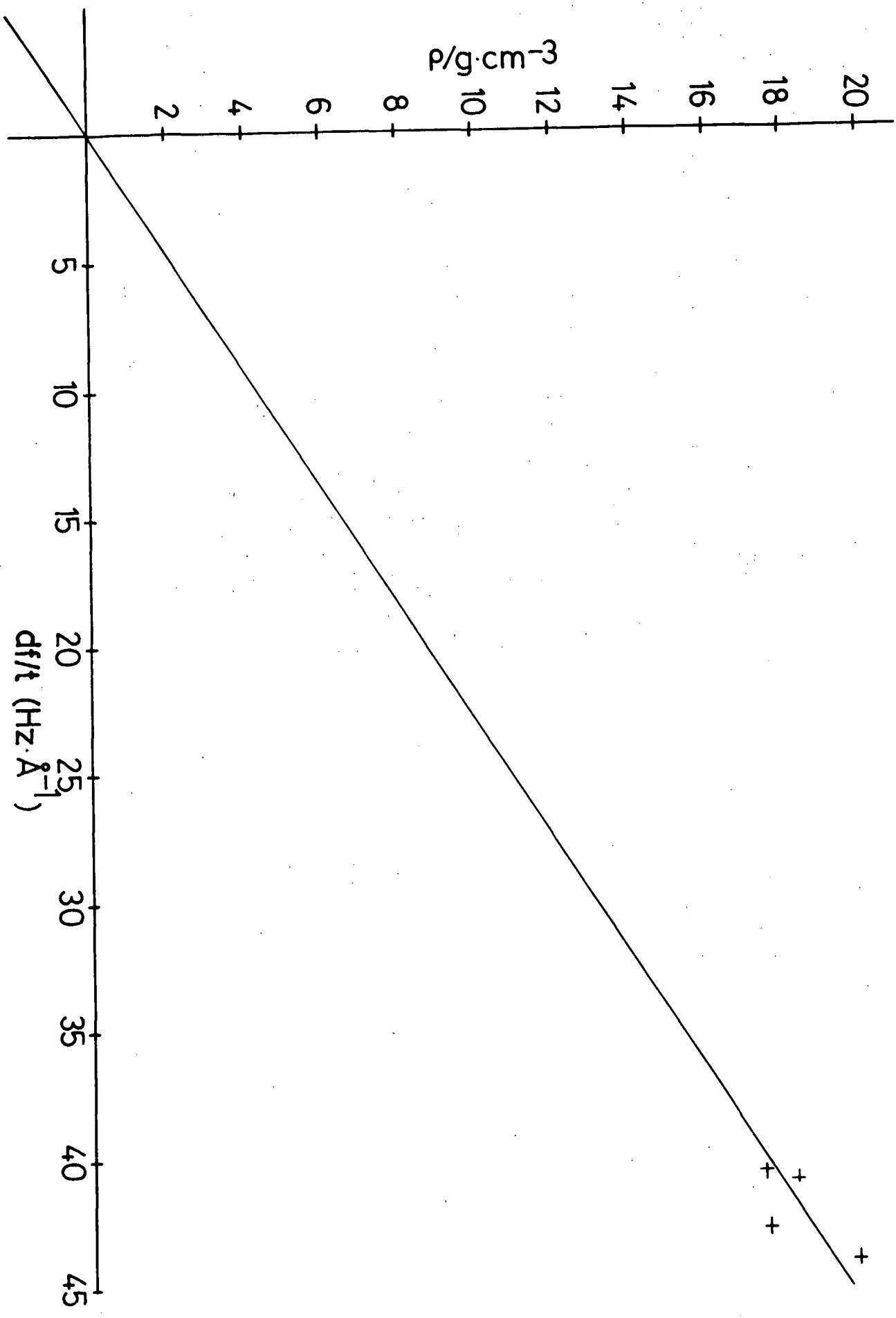
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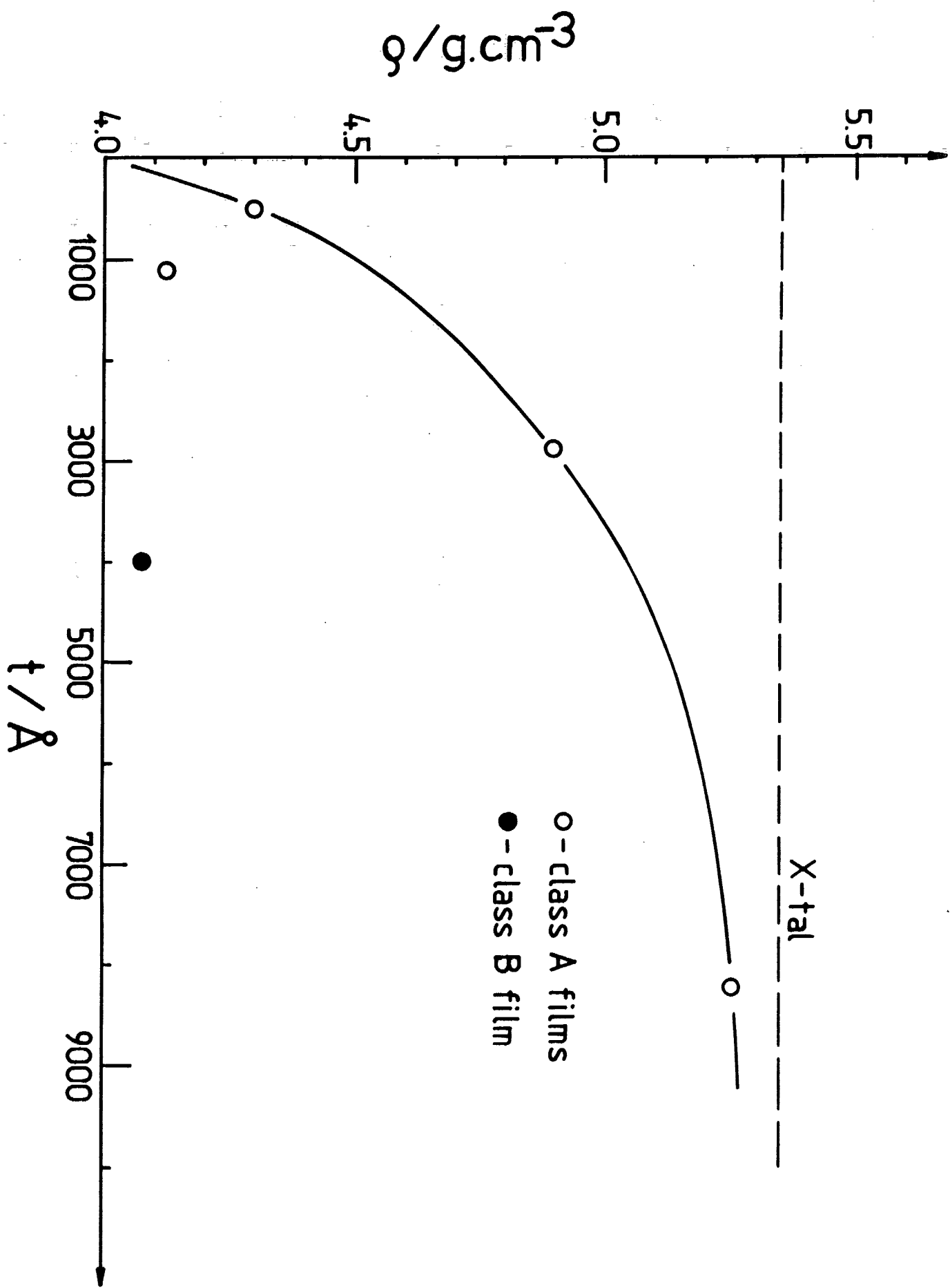
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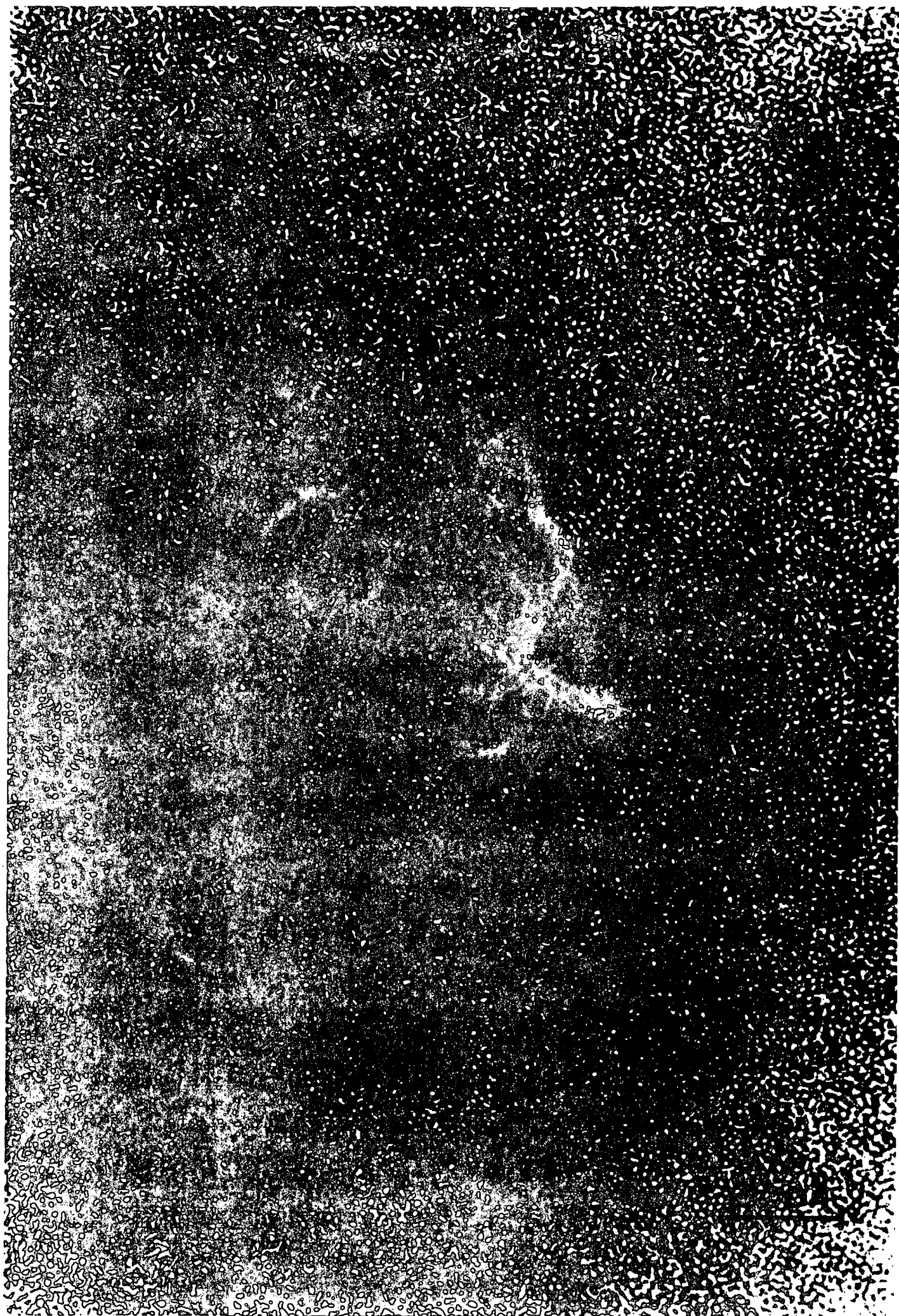
FIGURE CAPTIONS

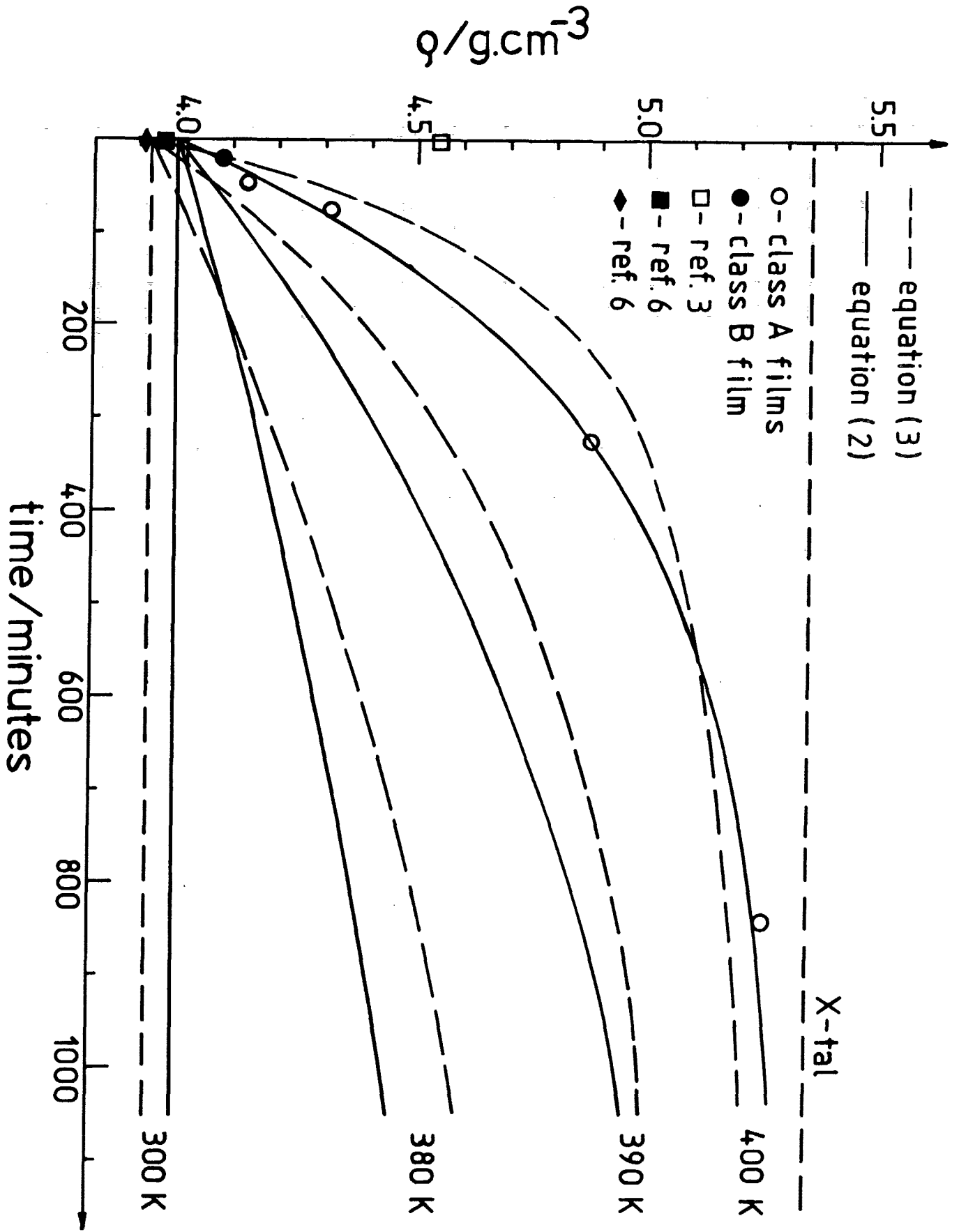
- Figure 1 Schematic diagram of the sample block, the evaporator and the optical system. es-evaporation source, rs-radiation shield, ms-mechanical shutter, qc-quartz crystal oscillator, em-evaporation mask, sb-sample block.
- Figure 2 Calibration plot of measured density ρ versus measured df/t (frequency change divided by the thickness) for a set of gold films.
- Figure 3 The measured density of a-Ge films as a function of their thickness for four films of class A and one film of class B. Also indicated in the figure is the crystalline germanium density.
- Figure 4 High resolution bright field electron micrograph of thin a-Ge film of class A investigated in this work (Magnification $\sim 1.6 \cdot 10^6$).
- Figure 5 The measured density of a-Ge films as a function of the total evaporation and/or annealing time. Also shown are the predicted density relaxation curves according to the simple model discussed in the text.











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