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STUDY OF POINT DEFECTS AT LOW CONCENTRATIONS BY INTERNAL FRICTION MEASUREMENT OF THEIR INTERACTION WITH DISLOCATIONS.

C. MINIER

Section de Physique du Solide,
Département de Recherche Fondamentale,
Centre d'Etudes Nucléaires de Grenoble
BP 85, Centre de tri 38041 Grenoble Cedex

SUMMARY

Different internal friction theories based on the presence of point defects at dislocations are compared with experimental results in irradiated metals and their condition of validity are discussed. Then the experimental results obtained in irradiated copper on the creation of point defects at dislocations and subsequent annealing behavior are compared with several theoretical models.

Internal friction permits direct study of point defects by analysis of anisotropic defect relaxation peaks (e.g. Snoek peak) or of pairs of defects (e.g. Zener peaks). When the concentrations become quite low (10^{-10}... 10^{-6} at. conc.) there is better sensitivity to indirectly study point defects by their modification of the damping of dislocations. In the first part, we shall discuss different theories which permit an interpretation of the internal friction due to dislocation damping in the presence of point defects. In the second part, we will analyze the mechanism of point defect creation at dislocations and finally the information given by this kind of measurement on the models of point defect annihilation.

I. Theories of dislocation internal friction in the presence of point defects

Much internal friction analysis has been based on the interpretation that, in addition to viscous drag and inertial effects, dislocation line motion - in response to external stress - is impeded by discrete pinning defects as proposed by Koehler 1 and Granato-Lücke 2 . Despite the noted successes in applying this vibrating string theory to explain point defect induced changes in the modulus and damping of materials, open questions remain.

In the strain amplitude independent region, at 0°K and at KHz frequencies, the fundamental relationships are given by

$$Q^{-1} = \alpha_1 \frac{N\omega L^4}{T_e^2} \qquad 1$$

$$\frac{\Delta M}{M} = \alpha_2 \frac{NL^2}{T_e} \qquad 2$$

where N is the dislocation density, ω the angular frequency, L the average dislocation length, T_e the line tension and α_1, α_2 are terms dependent on other parameters including a distribution of dislocation lengths.

Heiple and Birnbaum 3 have shown that the variations in modulus and the internal friction with frequency well verify the Granato-Lücke model for frequencies $\omega > 20$ KHz, but for lower frequency values, the internal friction deviates from prediction and gives too large values of the damping.

The arrival of point defects on dislocations results in a reduction of the average dislocation length L, hence Q^{-1} and ΔM/M, and a proportionality between $(\Delta M/M)^2$ and Q^{-1} is expected. This has been experimentally verified during irradiation in copper in the KHz frequency range, in 1956 by Thompson and Holmes 4 . Since then, other investigators have observed deviations from this relationship which they have attributed in the vibrating string model to two dislocation types 5 . More recently, Simpson, Sosin and coworkers 6 have shown a relationship nearly linear between the modulus defect and the internal friction in irradiated copper. Lücke et al. 7 have given evidence that the measurement strain amplitude influences the results obtained.

As expected from theory, a simultaneous decrease in both the modulus defect and the damping should occur during irradiation as dislocation pinning is accomplished. Almost all investigators report experimental changes following prediction, but several observations have been reported which exhibit marked deviations. Simpson-Sosin et al. 6, 8 have pointed out that during irradiation and measurements at low frequencies (500 Hz) in copper, the damping may increase, reach a maximum and decrease, simultaneously, the modulus defect decreases monotonically. Clearly this peaking effect is inconsistent with the K.G.L. model. Simpson and Sosin have proposed that point defect drag accompanying dislocation motion in response to external stress can account for this effect, and they have presented a model valid in the strain amplitude independent range based on this assumption. Qualitative and reasonably good quantitative agreement was found between experimental results and their model predictions in irradiated copper. Using two dislocation components, account was also possible for the linear dependence between the damping and modulus defect.

In our Grenoble group, Lauzier and coworkers 9 have studied the effect of strain amplitude in low temperature irradiated copper. Figure 1 shows the damping change during electron bombardment plotted as a function of irradiation time for several strain amplitudes. The magnitude of the peak in damping effect is larger at higher strain amplitudes, diminishes and no longer is apparent at the lowest measured strain amplitudes. This result is in contrast with the essentually amplitude-independent observations of Simpson, Sosin et al., and the theoretical results of the dragging model. The temperature dependence of the peaking in our results is also interesting ; in this sample, a peak is no longer observed for irradiation temperatures below 60 K, even at large strain amplitudes. Although the defect dragging model does not explicitly include a treatment of temperature dependence, it would suggest that a peaking effect in the damping be observable at all temperatures. Figure 2 presents a logarithmic plot of the normalized modulus defect, damping during irradiation at 9 K for several strain amplitudes. The slopes of the curves decrease with strain amplitude towards the value 0.5, at low strain amplitude ($\sim 10^{-7}$), expected from the Granato-Lücke model. In summary for the samples we have studied, the peaking effect and deviation to the Granato Lücke theory appear with increasing strain amplitude.

Seeger and Hornung 10 11 have proposed another analysis of the internal friction of dislocations in the presence of point defects. In pinning models, a point defect interferes only when it finds itself on a dislocation, which is what Seeger qualifies as a model of "all or nothing", whereas physically one should think that the internal friction and associated modulus defect vary progressively when point defects approach dislocations. Seeger and Hornung have made a theoretical study of this long-range point defect-dislocation interaction which is formally equivalent to the point defect-Bloch wall interaction in ferromagnetic materials. They show that in the case of anisotropic defects which can reorient, as for example, the dumbbell in f.c.c.materials, two effects can be present :

1. A redistribution of dumbbell axes occurs through dislocation interactions

characterized by a relaxation time $\tau_{1,2}$ as

$$\tau_{1,2} = 1/6(\nu_R + 2\nu_M)$$

where ν_R is the reorientational jump frequency without migration and ν_M is the migrational jump frequency. If a dislocation vibrates with a frequency $f >> \tau_{1,2}^{-1}$, its amplitude is reduced gradually when establishing an inner structure orientation and a mechanical after effect is observed comparable to the reorientational magnetic after-effect, where the role of dislocations is played by Bloch walls.

2. The second phenomenon to occur is defect diffusion in the stress field of dislocations to minimize the system energy. The change in spatial distribution of defects would be characterized by another relaxation time $\tau_3 \neq \tau_{1,2}$. Hornung 12 has shown that for a screw dislocation, <110>, in an isotropic crystal

$$\tau_3 = 1/7\nu_M$$

Using this form, Seeger 11 analyses the experimental results of stage III internal friction in copper and nickel. For the case of nickel, both the reorientation and diffusion are observed separately in different temperature ranges, corresponding to $\tau_3 >> \tau_{1,2}$, thus $\nu_R >> \nu_M$. For copper, $\tau_{1,2}$ and τ_3 should be of the same order, hence, $\nu_R << \nu_M$ and reorientation occurs together with migration.

It is particularly interesting to analyze the results during stage I using this model where at present two conflicting interpretations appear : crowdion, one-dimensional migration versus three dimensional migration. In the case of crowdion, one should view uniquely the phenomenon of migration to dislocations or to Bloch walls. For dumbbell, one should observe a superposition of orientation effects and diffusion in two different ranges of temperature, if the relaxation constants $\tau_{1,2}$ and τ_3 are different.

Figure 3 presents the results of Soulie et al. 13 for the internal friction as measured at 4 K in electron irradiated nickel following bombardment at 10 K with a flux of $\emptyset = 12.10^{18}$ electrons/cm^2, shown as a function of isochronal anneal temperature. The upper curve was measured after demagnetization to remove the Bloch wall contribution and it is observed that a reduction in dislocation motion occurs in the temperature range of 55K to 80K. The lower curve measured before demagnetization shows evidence of the point defect-Bloch wall interaction. In effect, because of the strong magnetostriction of nickel a periodic mechanical stress induces Bloch wall vibration. The magnetic after effect occurs and the Bloch walls are stabilized by reorientation or diffusion of point defects. The internal friction corresponding to wall movement diminishes between 45K to 55K. The phenomenon disappears at higher temperatures by annihilation of interstitials by vacancies during stage I_E.

These two appearing effects are rather displaced in temperature and we are led to attribute the interstitial-dislocation interaction near 65K to the diffusion of interstitials to dislocations with a relaxation time τ_3 which we have verified experimentally is independent of the dislocation density, and the effect of the interaction of interstitials with Bloch walls near 50K by the mechanism of reorientation of interstitials with a relaxation time $\tau_{1,2} << \tau_3$. The experimental results, obtained during an isothermal anneal to 52K correspond perfectly with the results deduced from the theory of Neel 14 for after effect reorientation.

Seeger equally accounts for the peaking effect by the theory of long-range interactions. Dumbbells reorient themselves in the neighborhood of dislocations with a relaxation time $\tau_{1,2}$. If dislocations vibrate under the influence of an external field, with a comparable frequency f, the distribution of dumbbell axes will be out of phase with respect to the stress and one observes a large energy dissipation. It is the "dislocation enhanced Snoek effect" which would be independent of the amplitude as long as the vibrational amplitude of the dislocation remains less than the surrounding "Snoek cloud". For the conditions, $\tau_{1,2} \sim 1/f_1$, a peaking effect may be observed which decreases and disappears rapidly when the temperatures is reduced, contrary to the predictions of the dragging model of Sosin.

Experimentally, for the most part, in the copper samples studied by Lauzier, the peaking effect was absent at irradiation temperatures below 60K. Nevertheless, in samples with a large Bordoni peak present, the peaking effect exists at 30K. In the Seeger hypothesis, this last observation could be attributed to an enhanced Snoek effect of interstitials in Stage I.

To conclude this first section, we see that as point defects arrive at dislocations, several effects are superimposed : a) a pinning effect due to defects situated on dislocation which reduce the free segment lengths, and b) a long range interaction between point defects and dislocations may occur related to reorientation and defect diffusion mechanisms.

When the number of point defects arriving on dislocation segments is small, Seeger indicates that the Granato-Lücke pinning mechanism can be valid if the effects of loop length reduction becomes more important than long range interactions.

Experimentally then, as ascribed by the results just presented the best conditions for observing G. L. type pinning occur when very low concentrations of point defects are used together with very low strain amplitudes to eliminate complicating effects, as for example, the peaking effect. Subsequently, the following sections, centered on point defect creation and annealing studies in copper, will present results under those experimental conditions.

II. Point defect creation mechanisms

When a metal is irradiated at very low temperature, below that of stage I_E, it is found that progressive pinning of dislocation lines occurs. At these temperatures, interstitials created in the bulk crystal are immobile and the pinning points are those which result from dynamic creation of point defects on the dislocations.

One can easily estimate the direct creation of Frenkel pairs in the stacking fault region between two partial dislocation components. Additionally, other creation processes which consider focussed events such as the dynamic crowdion or the focusson have been discussed by Leibfried [15], Bauer and Sosin [16], and Kamada et al. [17].

The first mechanism is known as the dynamic crowdion. During interaction with an electron, an atom is ejected if energy greater than the threshold energy is transmitted and a vacant site remains. Computations show that a collision serie with replacement follows the main event with a tendency to focalize along the close packed directions of the crystal. The energy lost Λ per interatomic distance traversed is several tenths of an ev. Gibson et al. [18] suggest a value of 2/3 ev. in copper. If the focussed chain intersects with a dislocation on the faulted region between partial dislocation components, defocussing occurs, followed by the creation of an interstitial near the dislocation which leaves far behind a vacancy. The threshold energy T_o of this event is taken as the same as that in the crystal.

The second mechanism known as focusson creation was first proposed by Silsbee 19 who considered a transfer of energy along the close-packed crystallographic directions but without interstitial transport. In a perfect crystal, focussons propagate, progressively losing their energy by thermal vibration and do not produce defects along their trajectory. However, if the focusson traverses a disordered region, a Frenkel pair can be created. The threshold energy T_o for this phenomenon can be much less than in the crystal and vacancies as well as interstitials may be created near dislocations.

Bauer and Sosin 16 have estimated the cross section of the focussed collision mechanisms

$$\frac{n_F}{\phi} = \frac{l_o}{a^3} \int_0^{\frac{T_m - T_o}{\Delta}} \omega \, d\lambda \int_{T_o + \Delta\lambda}^{T_m} \frac{d\sigma}{dT} \, dT$$

n_F is the number of pinners on a dislocation segment of length l_o, ϕ is the electron dose, T_m is the maximum energy transmitted to an atom and T_o the displacement threshold energy on dislocation, Δ is the energy loss by an atomic jump in the focussed chain, λ is the length of the chain in atomic units, ω is the width of the stacking fault of the dissociated dislocation and $d\sigma/dT$ is the effective differential Rutherford cross section.

In our Grenoble group, Lauzier 20 has studied these mechanisms in copper. He has measured on the same sample, dislocation pinning during electron bombardment at 20K for different values of the incident electron energy.

Figure 4 shows the variation with T_m of the experimental values n_{ex}/ϕ and of the calculated values by direct creation n_D/ϕ or by focussed collisions n_F/ϕ, using the numerical values $\Delta = 0.6$ eV, $\omega = 25.10^{-8}$ cm, $l_o = 10^{-4}$ cm, $T_o = 18$ eV and 20 eV.

This comparison indicates that the focussed collisions are probably not very important in copper and that most of the defects are created on the dislocations by direct mechanism. Also it appears that few creation events have a threshold energy lower than 16 eV ; additional experiments are needed to very low energies to further clarify this point.

III. Point defect annihilation models

Dislocation pinning experiments have been extensively used to study various models of point defect annihilation since for small concentrations of defects (10^{-9} --- 10^{-10} for example) the probability of forming defect complexes (divacancies and diinterstitials) is low. The observed pinning stages have been attributed to the migration of simple interstitials or vacancies.

The experiments performed by Thompson and Buck 21 in copper have generated considerable discussion. They have been interpreted by different theoretical models and have been submitted to numerous criticisms of their experimental conditions. The pinning rate measured at temperatures above 77K goes through a pronounced minimum near 160K. The ratio (n_{80K}/n_{160K}) is the order of 15. The authors interpret the minimum by a two-interstitial model with conversion. Crowdions migrate during stage I_E (50K) and dumbells during stage III (above 180K in these experiments). The activation energy observed in this temperature range is in effect too small for vacancies. The diffusion length of a crowdion before conversion to a dumbbell diminishes with the temperature which explains the lower pinning rate between 80K and 160K.

A quantitative analysis of these results has been proposed by Simpson and Sosin 22 using a two-interstitial model with thermal conversion, but where the two interstitial types can migrate in three dimensions. Defects present in stage I correspond to a <111> type, and in stage III, to an <100> interstitial type. By introducing an impurity interaction term, the quantitative results of Thompson and Buck are directly fitted by this model.

Concurrently, Frank and Seeger 23 have proposed a quantitative analysis of the results of Thompson and Buck based on the hypothesis of one dimensional migration in stage I. This necessitates the existence of two types of crowdion defects : the on-line crowdion migrating on the direction of its own vacancy and having a very weak chance of pinning a dislocation if it is not converted, and the off-line crowdion, to the contrary, having a possibility to intercept dislocation before converting.

Stage I pinning corresponds, therefore, to the arrival of off-line crowdions. Between 80K and 150K, there will be thermal conversion of the two crowdion types into dumbells, with stage III corresponding to the pinning of dislocations by dumbbells. The threshold energy for creation should be 9 eV for on-line crowdions and on the order of 19 eV for off-line crowdions. This theory is supported by the experimental results of Roth and co-workers 24 who show that the threshold energy is much less for pinning rates measured at 400K than for those measured at 80K. On the other hand, the hypothesis is not confirmed in the results obtained in our group 20 during isochronal annealing of a copper sample for different values of the incident energy. Figure 5 shows the number of pinners n per dislocation length deduced from measurements of the internal friction at 9K using the Granato-Lücke theory, presented as a function of the annealing temperature T. The electron doses were chosen such that the amount of pinning due to each irradiation would be approximatively the same. The pinning stages I and III are exhibited and above 200K a depinning appears. The numbers of pinners n_I and n_{III} reaching a dislocation segment during stage I and III can be deduced and are presented in figure 6 showing the cross sections n_I/ϕ and n_{III}/ϕ for these two stages as a function of the maximum transfer energy T_m. Not too large a difference in the threshold energies appears between Stages I and III.

These experiments near threshold are crucial to testing the model and additional results are necessary in particular to separate intrinsic and impurities effects.

Rather than continue to develop theoretical models to explain the results of Thompson and Buck, [1] would like to present the arguments of experimentalists who have voiced quite early against a precautiousless quantitative interpretation of dislocation pinning.

Keefer [25] has shown clearly the influence of measurement temperature on the results deduced from experiment. In particular, an irradiation at 77K followed by anneal at 150K introduces a pinning effect if the measurement is made at 4K and a depinning effect if the measurement is made at 77K. This result is due to the fact that there are several dislocation types which intervene differently following the measurement temperature. Lücke et al. [7] have shown that the pinning rate of dislocations measured at 77K for very small strain, presents only a weak minimum as a function of irradiation temperature.

Lauzier and Minier [26] have extended the pinning rate measurements to very low temperatures (liquid helium temperatures range). Figure 7 shows that the pinning rate measured at 4K goes through a maximum near 70K with the ratio $(n_{70K}/n_{4K}) \sim 5$. Then, n decreases until around 150K, but the ratio (n_{80K}/n_{140K}) is $\sim 1,5$, being about ten times smaller than the ratio given by Thompson and Buck. They have also studied the influence of measurement temperature on the number of pinners. Figure 8 shows the ratio of the number of pinners deduced by measuring at 4K and at TK, following an irradiation performed at TK. The ratio is not too different from one, up to about 70K, but after, increases considerably. This confirms well the existence of two classes of dislocations of which one form becomes dominant in measurements above 80K, essentually the same temperature range as the Bordoni peak. In the second class of dislocations, the phenomenon of defect redistributioning has probably induced a reduction in the pinning rate or even depinning in the experiments of Keefer. These conclusions contest strongly all quantitative analyses of pinning rates obtained from measurements at different temperatures. Moreover, dislocation depinning by defect line diffusion must be also carefully considered. Until the present, line diffusion has always been neglected in the interpretations.

In the earlier pinning experiments of Keefer et al 27 a reduction in pinning takes place during isochronal anneal between 90 to 170K, particularly in samples having very small defect concentrations.

Our last results 20 show that depinning in this temperature range is more or less accentuated depending on the sample measured, with depinning more apparent from modulus measurements than from internal friction measurements. Figure 9 shows this clearly exhibiting for the internal friction a classic behavior where pinning is observed in Stages I and III during isochronal anneal and measurement at 4K. Small depinning occurs after 70K and larger scale depinning occurs after 220K. In contrast, the modulus results exhibit strong depinning between 120 and 170K.

These results may yet be explained by a model using two dislocation types. In one form, the point defects would be fixed in position, in the other, they may be allowed to migrate. Thompson and Buck 21 have shown that the internal friction measured may well be associated merely with the dislocation type having the weakest line tension while the modulus measurements appear to superimpose the phenomena taking place on the two types of dislocations.

IV. Conclusions

The study of point defect pinning of dislocations is certainly interesting because it allows observation at very low defect concentrations, but numerous experimental precautions must be realized before the results can be interpreted quantitatively. All the measurements must be performed on the same sample at very low strain amplitudes and at constant temperature for both the internal friction and the elastic modulus. It appears advantageous to work when possible on samples containing well characterized dislocations, for example as in the single crystal experiments of Paré et al 28 .

Depinning should be carefully considered since it can be observed in copper in the range 90-160K which is the temperature region where continuing controversy exists concerning various annihilation models.

The study of the dynamic creation of pinners on dislocations lines by electron bombardment in the liquid helium temperature range shows that in copper, the foccused collision mechanisms are not the most important.

The variation of the annealing of point defects as a function of the incident energy of the electron beam has not indicated in copper a large difference in the threshold energies of stages I and III, which is expected from the two interstitials model, including one dimensionnal migration in stage I. It may be noted that in nickel, a comparison of the internal friction due to interaction of interstitials with dislocations and Bloch walls has established that a three dimensionnal migration occurs during stage I.

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FIGURE CAPTIONS

Fig. 1 : Variation of the damping of a copper sample plotted for several amplitudes ϵ, as a function of irradiation time at (a) 160K, (b) 115K, (c) 75K, and (d) 60K.

Fig. 2 : Logarithmic plot of the inverse normalized modulus defect M_n versus inverse normalized damping δ_n of a copper sample for several strain amplitudes ε during irradiation at 9K.

Fig. 3 : Variation of the damping q^{-1} of an electron irradiated nickel sample measured at 4K as a function of the isochronal annealing temperature. ——— measurements before demagnetization, ... measurements after demagnetization.

Fig. 4 : Variation with the maximum transfer energy T_m, for the experimental creation cross section n_{ex}/ϕ, the theoretical cross section due to direct mechanisms n_D/ϕ, and focussed collisions n_F/ϕ.

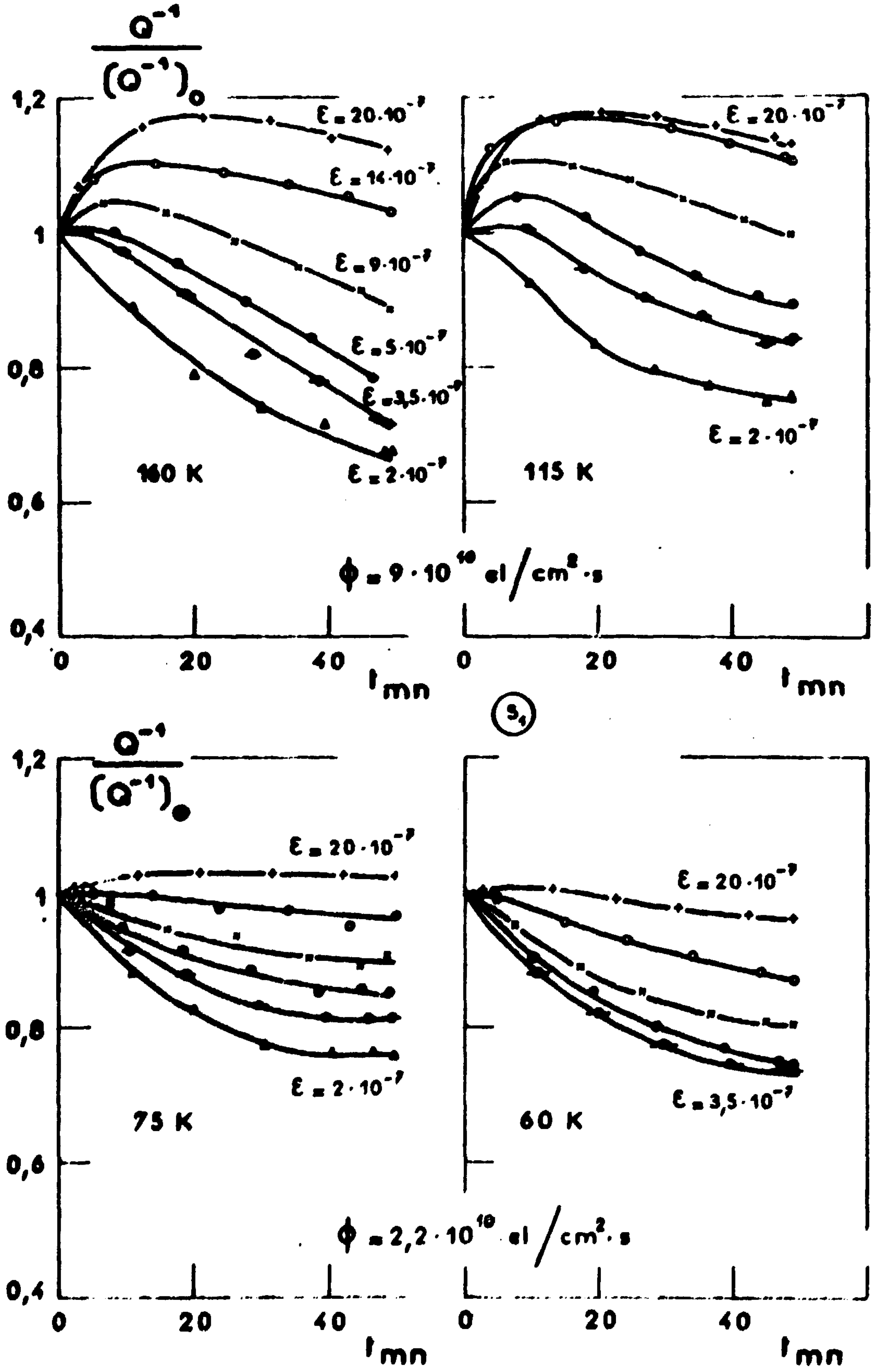
Fig. 5 : Variation of the number of pinners n during isochronal annealing for several electron energies E.

Fig. 6 : Variation of the cross sections corresponding to stages I and III as a function of the maximum transfer energy T_m.

Fig. 7 : Pinning rate measured at 4K versus irradiation temperature T : • from damping measurements n_{Z4} ; and • from modulus measurements n_{Y4}.

Fig. 8 : The ratios n_{Y4}/n_{YT} of the pinning rates from modulus measurements at 4K and TK and n_{Z4}/n_{ZT} from damping measurements versus the irradiation temperature T.

Fig. 9 : Variation of the frequency and the damping of an electron irradiated copper sample during isochronal annealing.

Q⁻¹/(Q⁻¹)₀
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0,6
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ε = 2·10⁻⁷
160 K
ε = 20·10⁻⁷
ε = 2·10⁻⁷
115 K
ϕ = 9·10¹⁰ el/cm²·s
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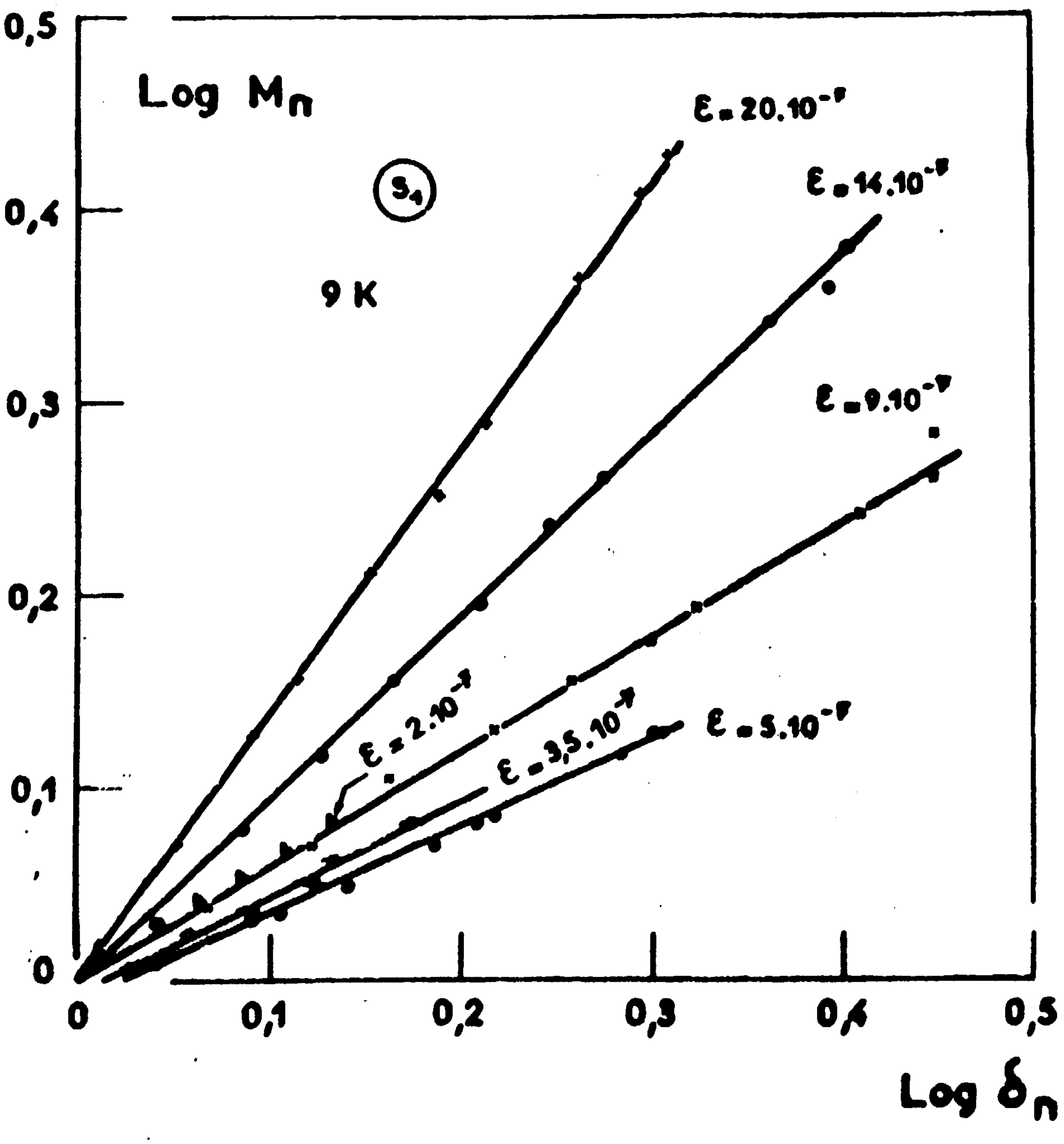
t mn

1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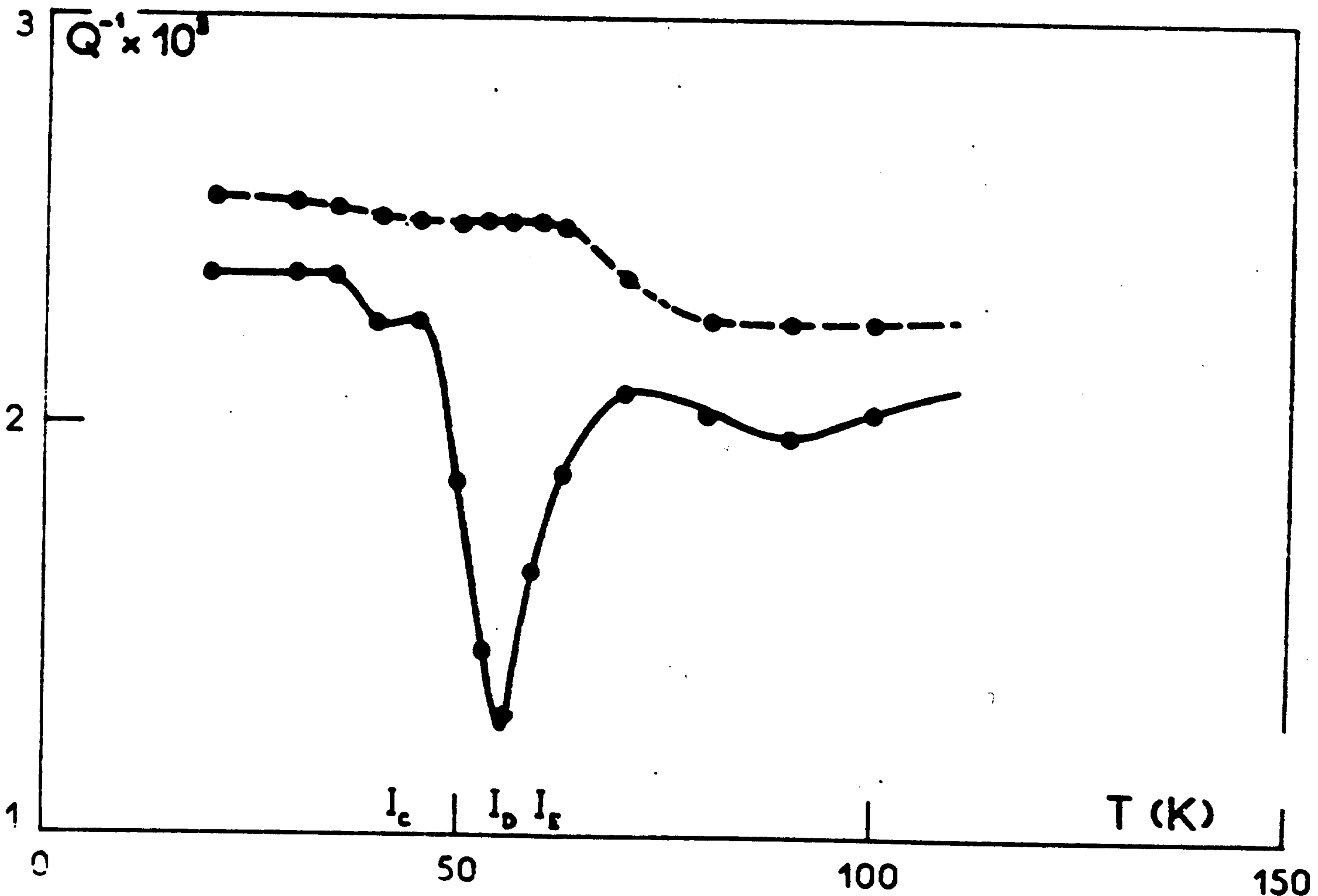

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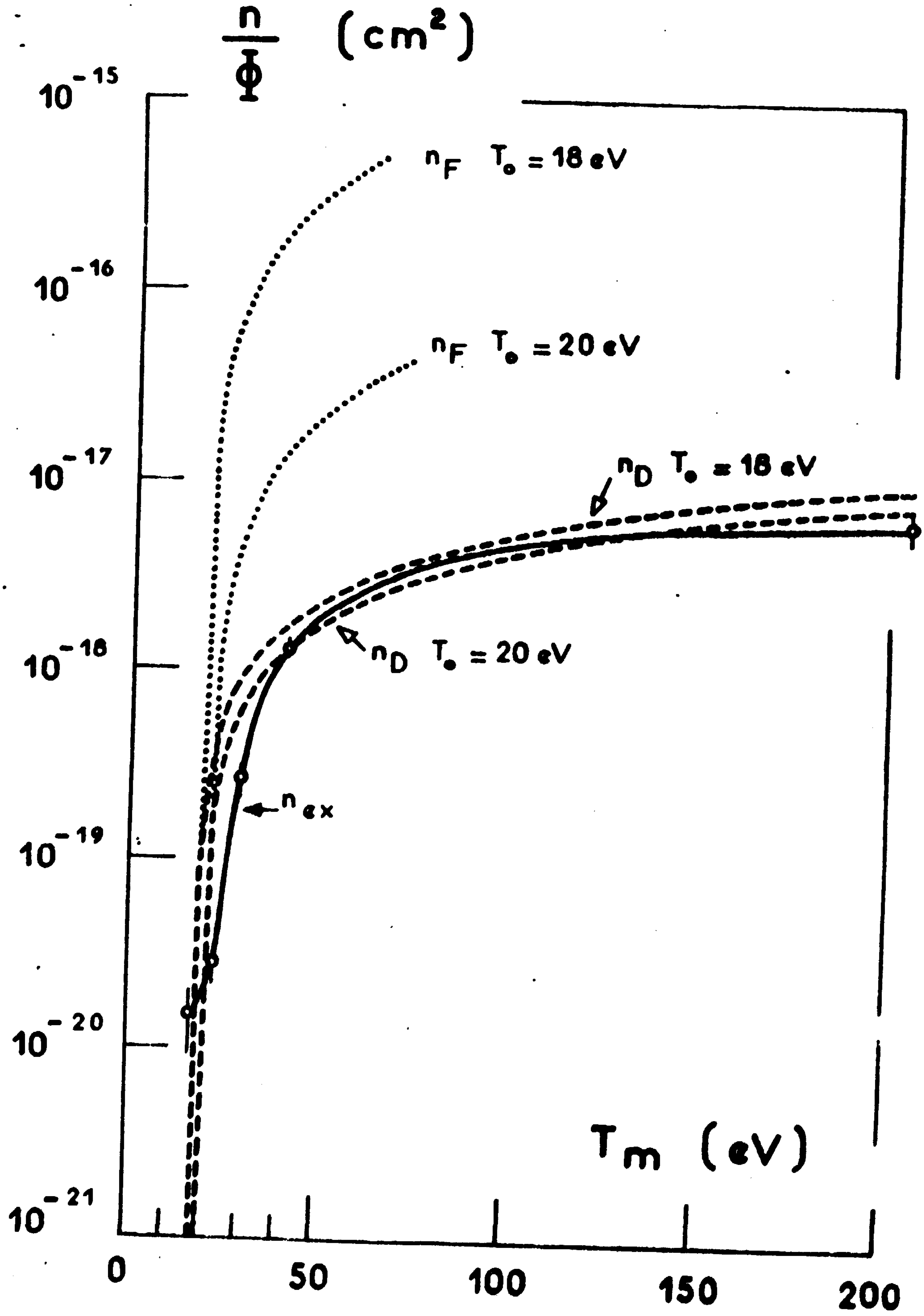
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10^{-17}
10^{-18}
10^{-19}
10^{-20}
10^{-21}
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n_F $T_o = 20$ eV
n_D $T_o = 18$ eV
n_D $T_o = 20$ eV
n_{ex}
T_m (eV)
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50
100
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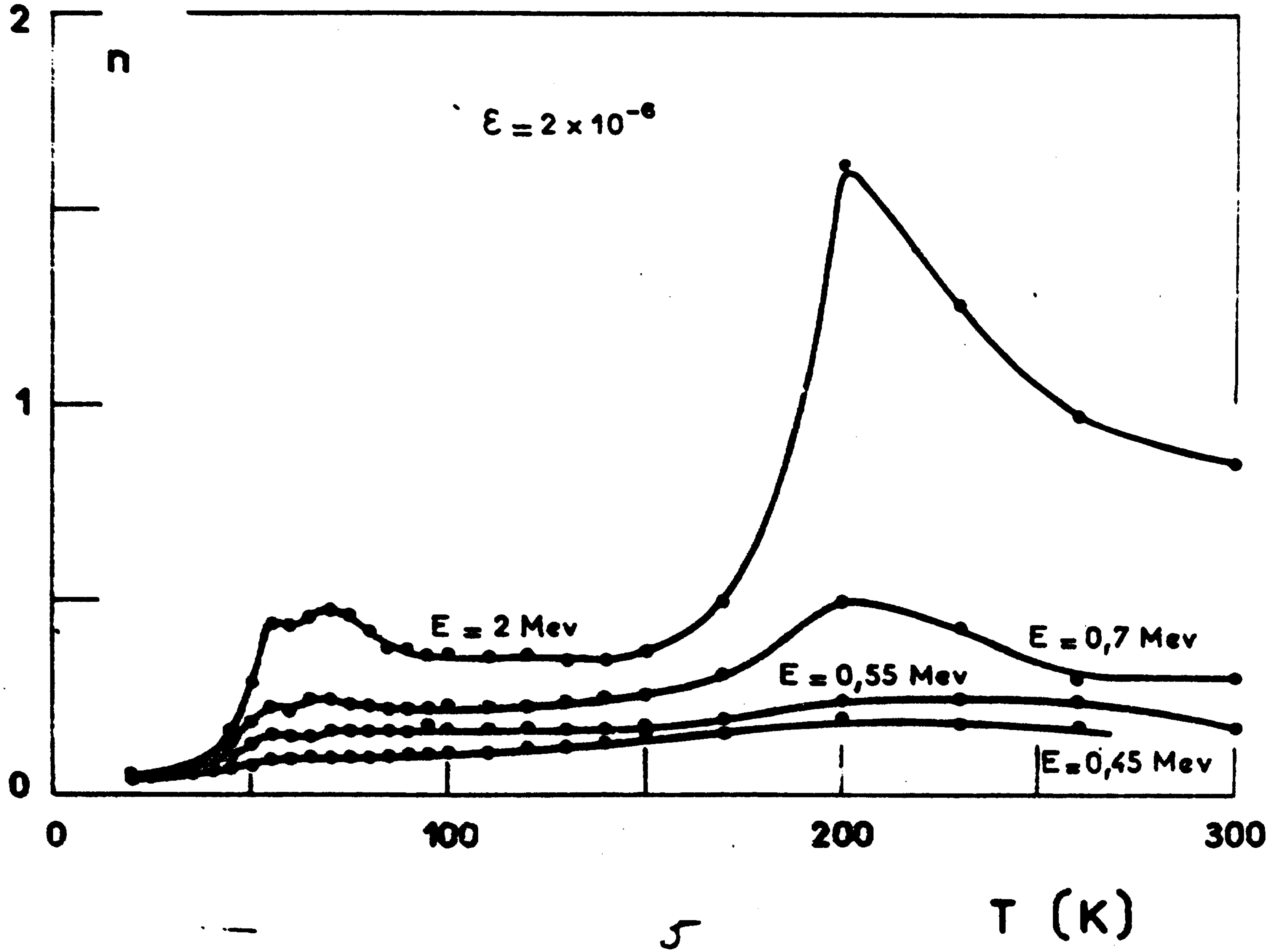
200

4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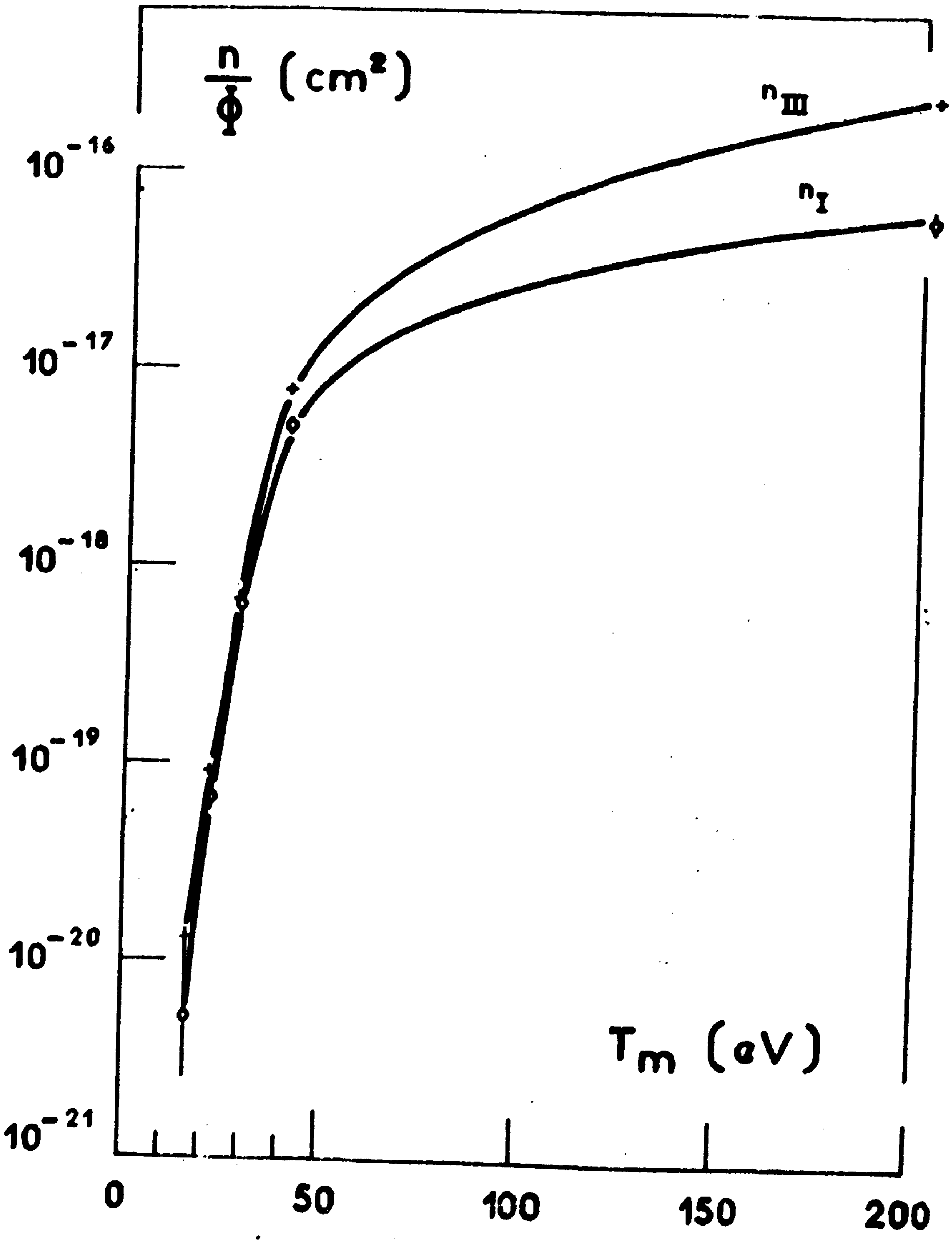
T (K)

5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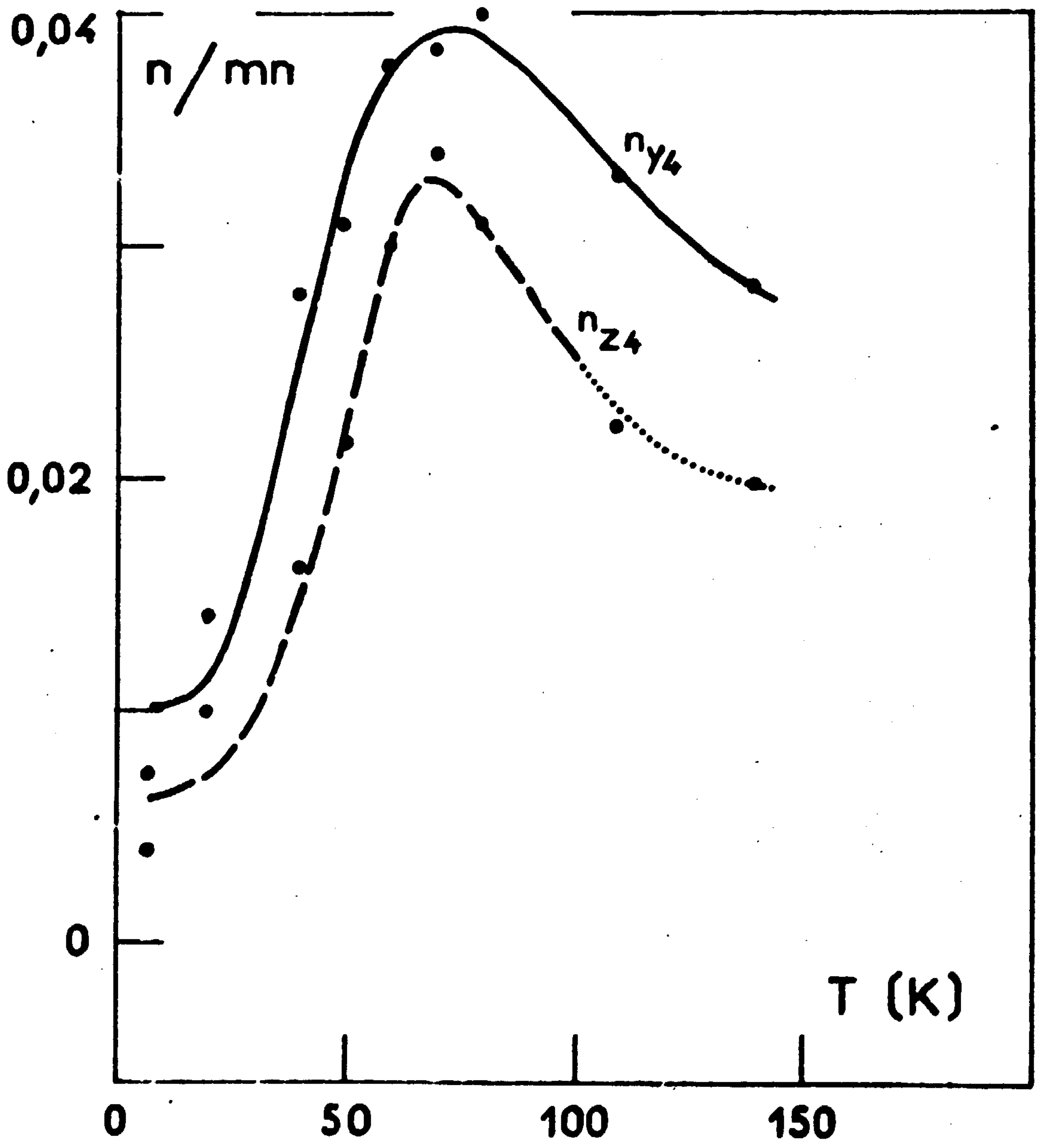
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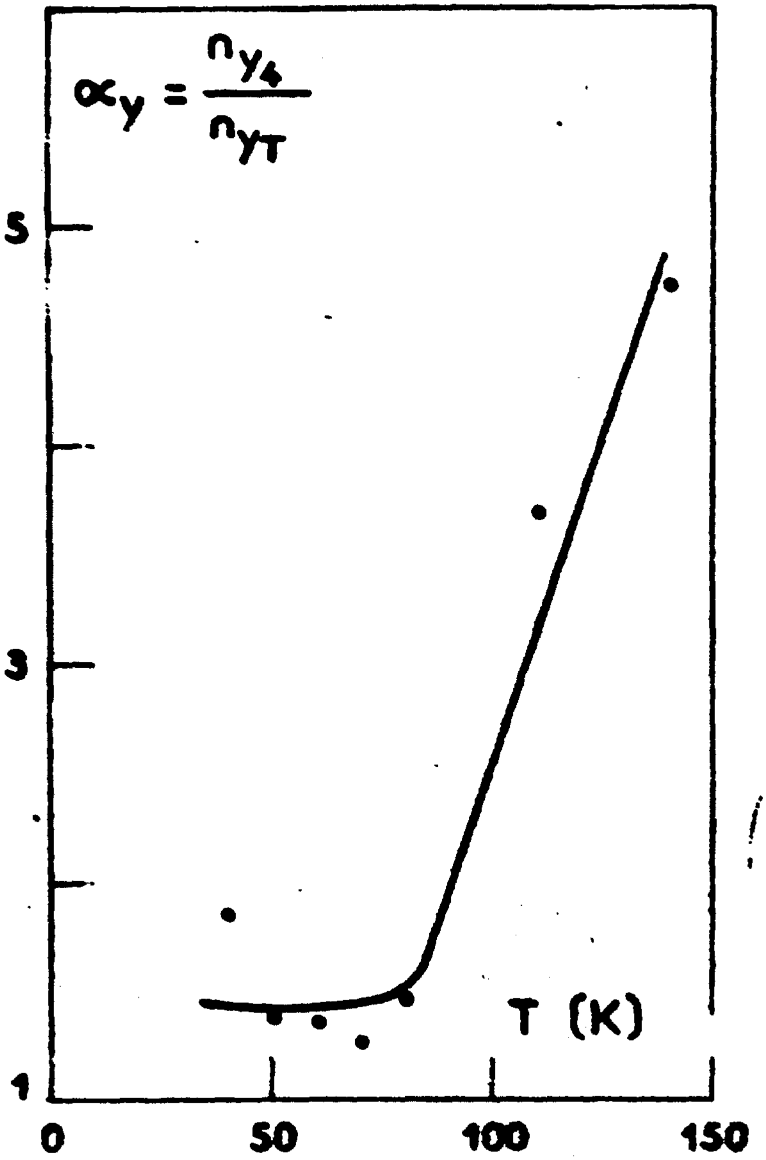
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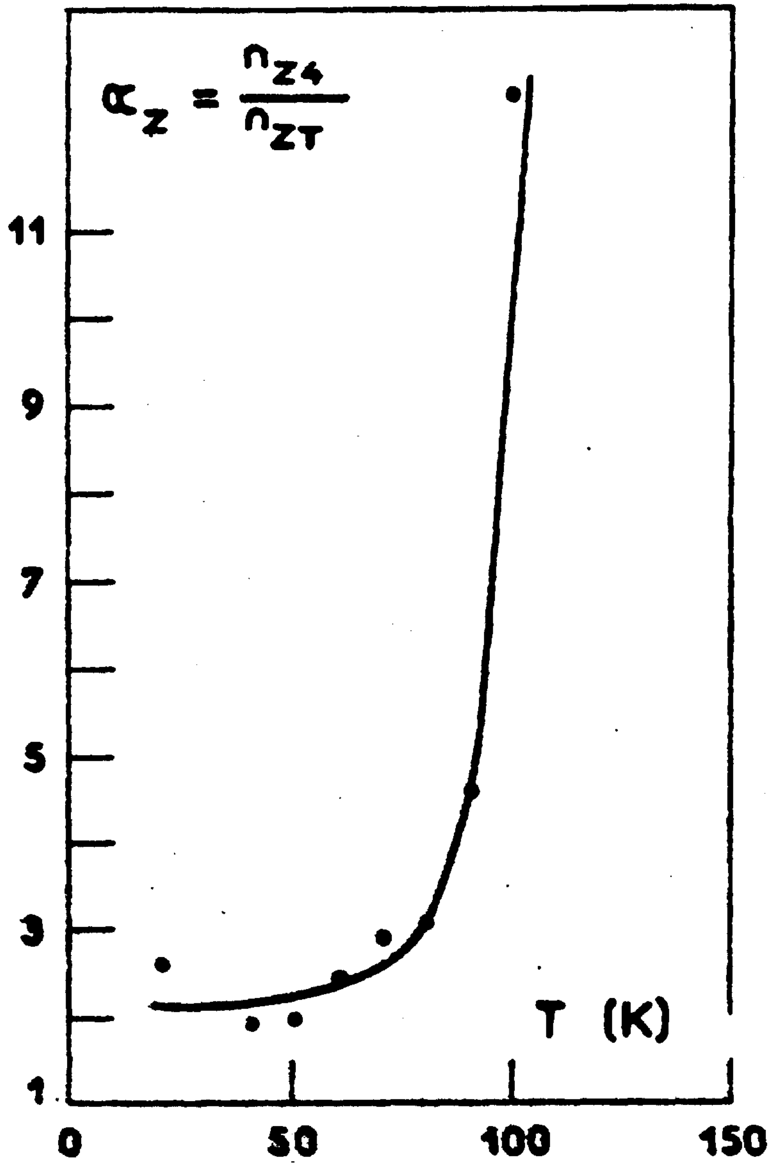
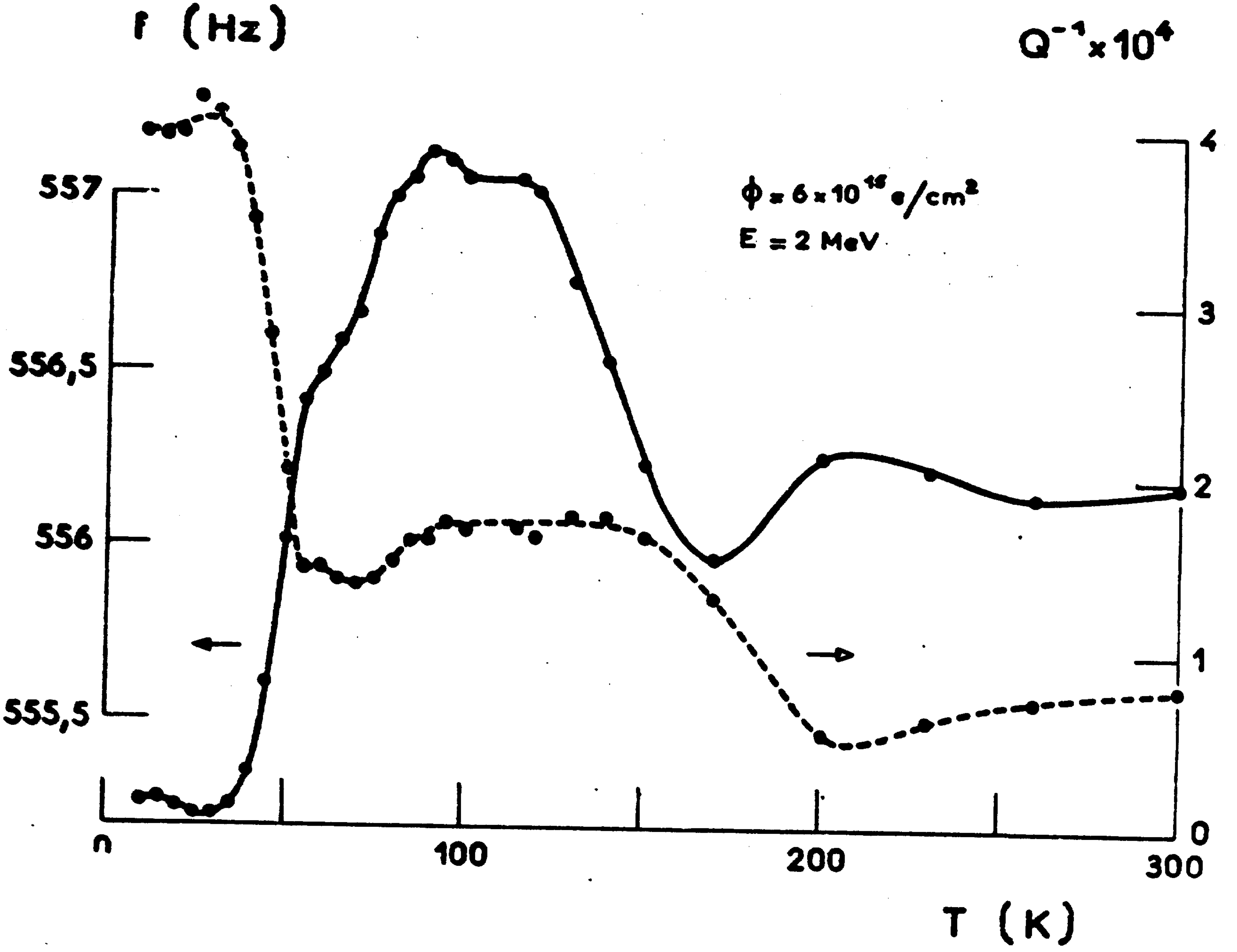
3
1
0
50
100
150
T (K)


$\alpha_z = \frac{n_{z4}}{n_{zT}}$
11
9
7
5
3
1
0
50
100
150
T (K)


f (Hz)
Q^-1 x 10^4
ϕ = 6 x 10^16 e/cm^2
E = 2 MeV
557
556,5
556
555,5
4
3
2
1
0
0
100
200
300
T (K)