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POINT DEFECT DISLOCATION PINNING IN IRRADIATED COPPER

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ABSTRACT

We investigate the problems of the quantitative analysis of dislocation pinning at very low temperatures and stress the importance of strain amplitude, measurement temperature, and eventual depinning. The variation in pinning rates between 9 K and 140 K are compared with theories and the influence of electron energy is discussed.

INTRODUCTION

The proposal by Koehler[1] and Granato-Lücke[2] (K-GL) that dislocation motion may be impeded by discrete pinning defects has provoked considerable analysis and discussion. Despite the successes of this vibrating string model, application in the Hz to kHz frequency range remains problematical and open questions have arisen experimentally[3,4], Lücke et al[5] have especially expressed the importance of the influence of measurement strain amplitude. We have performed detailed internal friction and modulus measurements at low frequency (∿ 700 Hz) on well-annealed copper samples during electron irradiation to further clarify existing problems.

STRAIN AMPLITUDE STUDIES

In an attempt to verify to very low irradiation temperatures the squared-functional dependence predicted between the modulus defect and the dislocation damping in the (K-GL) model, we have confirmed for our experiments the importance of applied strain amplitude[6]. As seen in Fig. 1 the slopes of a log-log plot of the normalized modulus defect and normalized damping changes in a low temperature irradiated sample, exhibit a decrease in value toward an expected theoretical value of 1/2 with decreasing strain amplitude.

We have also established that during electron irradiation a peak in the damping can occur while the modulus defect decreases continuously, the presence of which is dependent on strain amplitude and temperature. Fig. 2 summarizes these results. The peak is largest at high strain amplitudes, diminishes and disappears at the lowest applied strain amplitudes. These effects are in contrast to the experimental and theoretical results of Simpson and Sosin et al (SS)[7,8] of a peaking effect during irradiation which they attribute to point defect dragging by moving dislocations and report as essentually strain amplitude-independent.

No peaking effect is observed for irradiation temperatures below 60 K, even at large applied strain amplitudes. Although the defect dragging model of (SS) does not explicitly include a temperature dependence treatment, it suggests a peaking effect in the damping at all irradiation temperatures. Seeger[9,10] and Hornung[10] have presented further analysis in which they attribute the "peaking effect" in irradiated metals and its strong temperature dependence to a "dislocation-enhanced Snoek effect".

PINNING-RATE MEASUREMENTS AT LOW TEMPERATURES

Many questions have been raised in trying to understand and verify the various (one-interstitial and separate conversion-two-interstitial) models. Important among attempts to resolve these questions are the experimental results of Thompson and Buck[11] in irradiated copper, who show that the pinning-rate measured above 77 K goes through a minimum near 160 K. The minimum is taken as evidence of a thermal conversion mechanism in the two-interstitial model. The ratio of pinning rates in their study obtained between 80 K and the minimum n_{80}/n_{160} is ≈ 15. Our pinning-rate measurements at very low temperatures, have shown that the effect of measurement temperature is important when interpreting pinning-rate results [12]. From Fig. 3, the pinning-rate measured at 4 K exhibits a maximum near 70 K where the ratio n_{70}/n_4 is ≈ 5. Then it decreases to a minimum near 150 K and subsequently increases. The ratio of pinning-rates at the minimum ($n_{70}/n_{140} \approx 1.5$) is about ten times smaller than the Thompson-Buck result. In Fig. 4 we show the ratio of numbers of pinners $n_{y4,z4}/n_{yT,zT}$, deduced from modulus and damping measurements respectively, by measuring at 4 K and at T K following an irradiation at T K. The ratio is not too different from one up to about 70 K, after which it increases abruptly.

Thus the quantitative analysis of pinning-rate data taken at various measurement temperatures is questionable, and further suggests the existence of two classes of dislocations, of which, one form becomes dominant during measurement above 80 K (the Bordoni peak range) and along which perhaps line diffusion may take place.

Separate quantitative analyses of the pinning-rate minimum have been proposed based on the conversion-two-interstitial model, differing directly in restrictions on migration. An analysis presented by Simpson and Sosin[13] assumes that Stages I and III defect forms migrate three-dimensionally. This model fits the Thompson-Buck pinning-rate minimum results quite well above 80 K by accounting for impurity effects. We have extended this calculation to lower temperatures using their parameters and find agreement through the maximum in pinning-rate near 70 K down to 40 K, below which the values determined theoretically become several orders too small. This difference below 40 K indicates the importance of other processes, namely, the direct creation of point defects at dislocations which we will discuss later.

Concurrently, Frank and Seeger[14] have proposed a conversion-two-interstitial model based on the presence of Stage I defect configurations which migrate one-dimensionally. Two types of crowdion defects are postulated, the "on-line", migrating in the direction of its self-vacancy and having a small chance of pinning dislocations, if it is not converted ; and the "off-line", which has a greater possibility of intercepting dislocations before converting. The threshold energy for the creation of on-line crowdions is given as about 9 eV and for off-line crowdions as about 19 eV. This energy difference has received support experimentally by Roth and co-worked[15], but is not confirmable by experiments performed as a function of variable incident electron energy, which we discuss next.

INFLUENCE OF ELECTRON ENERGY

The defect production and annihilation mechanisms for different values of the incident electron energy were studied at 20 K, followed by isochronal measurements up to room temperature. The electron doses were chosen such that the magnitude of pinning during irradiation at each energy studied would be approximately the same. The sample was well annealed before each change in electron energy. Fig. 5 summarizes these results where the number of pinners n, per dislocation length, as deduced by the Granato-Lücke models from damping measured at 9 K, is plotted as a function of anneal temperature. Two pinning regions appear. Stage I near 40-70 K, and Stage III in the range of 140-200 K. Depinning is evident above 200 K, particularly in the results obtained following irradiation at 0,7 MeV and above.

We may then distinguish three numbers experimentally : those defects created on dislocations by irradiation at 20 K, n_c ; arriving during Stage I, n_I ; and n_{III} during Stage III. In Fig. 6 the corresponding effective cross-sections n/ϕ are plotted as a function of maximum transfer energy. The values of threshold energies for Stage I and III appear not very different, thus not confirming the model of Frank and Seeger. Additional experimental studies at lower energies are in progress to clarify this crucial point. These additional experiments are hoped also to elucidate the small evidence of lower threshold energy for creation occurring at dislocations exhibited in curve n_c.

STABILITY OF DEFECTS ON DISLOCATIONS

The possibility of defects diffusing along dislocations has always been neglected in the interpretation of pinning-rates. In the earlier pinning studies of Keefer et al[16] a reduction in pinning takes place during isochronal anneal between 90 to 170 K, particularly in samples containing very small defect concentrations. We 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 damping measurements. Fig. 7 shows this clearly for a sample exhibiting the largest amount of depinning. The damping exhibits pinning in Stages I and III. Small depinning occurs above 70 K with a larger amount after 200 K. By contrast, the modulus results show strong depinning between 120 and 170 K. Such a discrepancy between derived pinning point numbers may be explainable by a two-dislocation model incorporating different line tensions for the separate dislocation types. Thus the internal friction being measured may well correspond to dislocations with apparently the weakest line tension since the modulus defect is probably influenced by a superposition of the contributions of both dislocation types as discussed by Thompson and Buck[11]. Defect mobility along one dislocation type would then be fixed or restricted, while along the other, defects would be allowed to migrate.

CONCLUSIONS

We have demonstrated that numerous experimental precautions must be realized before the usual interpretation of dislocation pinning experiments can be done quantitatively.

Measurements of the pinning-rate at very low temperatures show that it goes through a maximum at 70 K.

The variation of the pinning with the electron energy is of importance in identifying the defect species, we have shown only a small difference exists between Stages I and III threshold energies, and are thus not in agreement with the crowdion version of the conversion-two-interstitial model.

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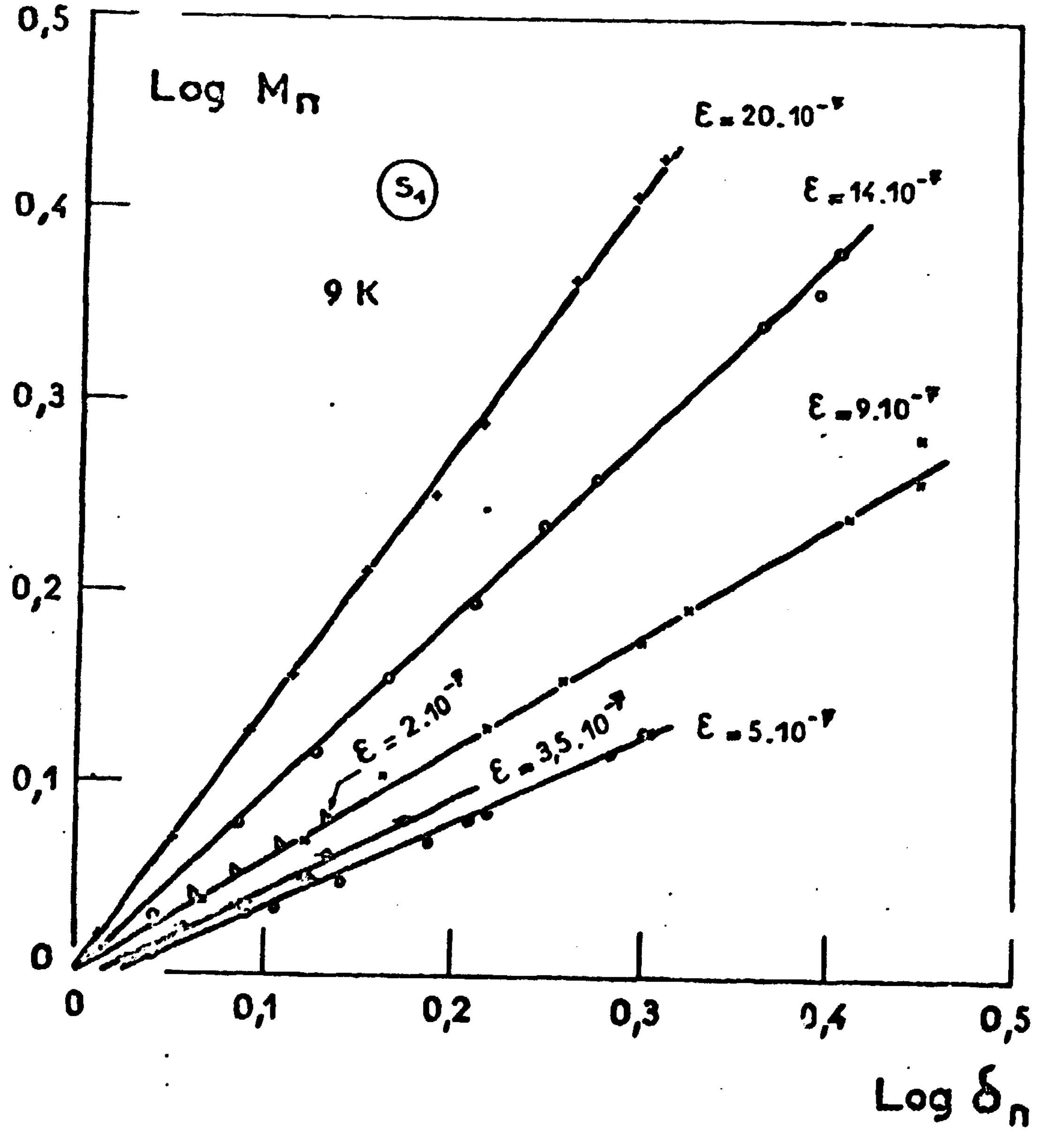
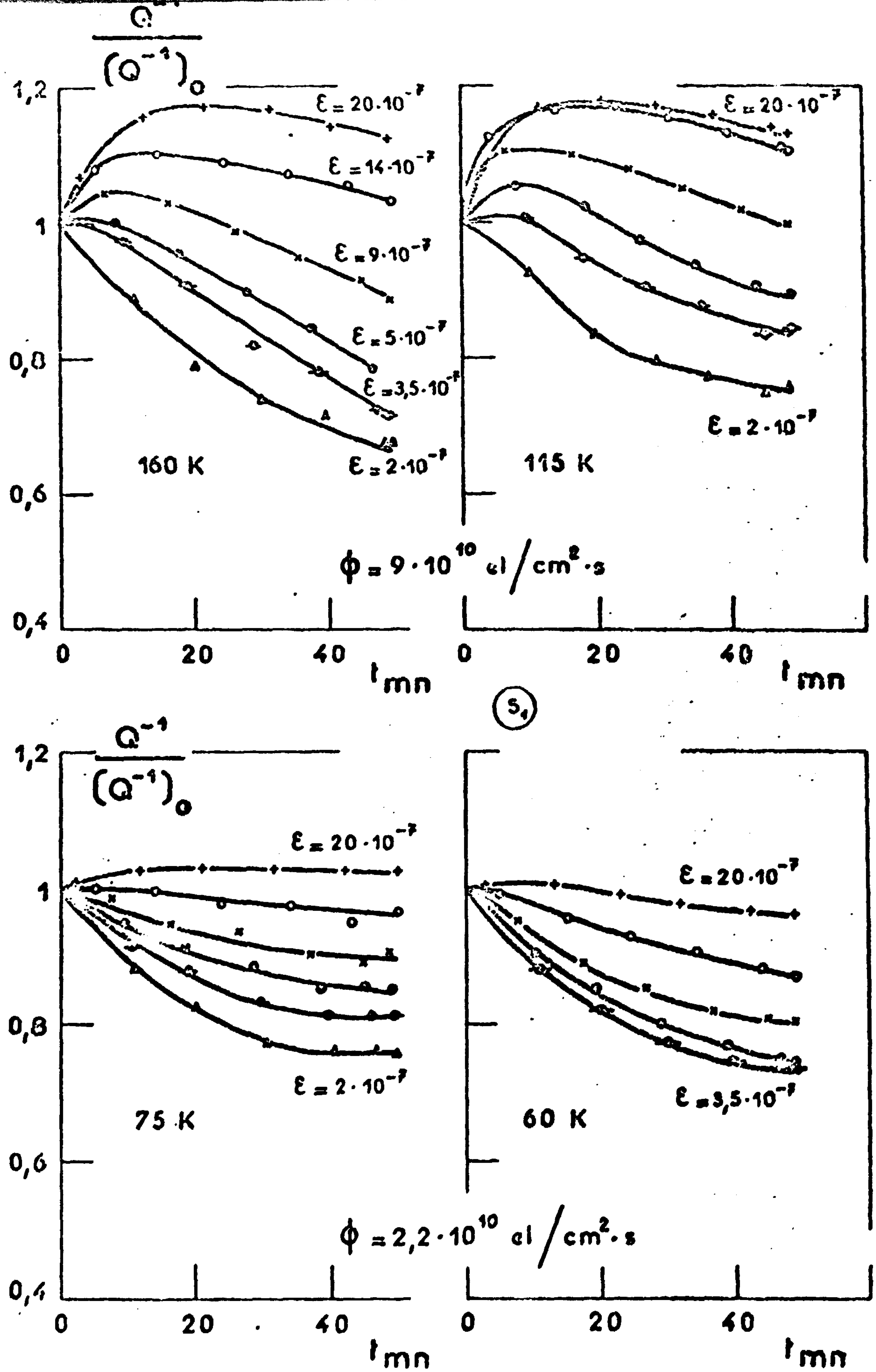
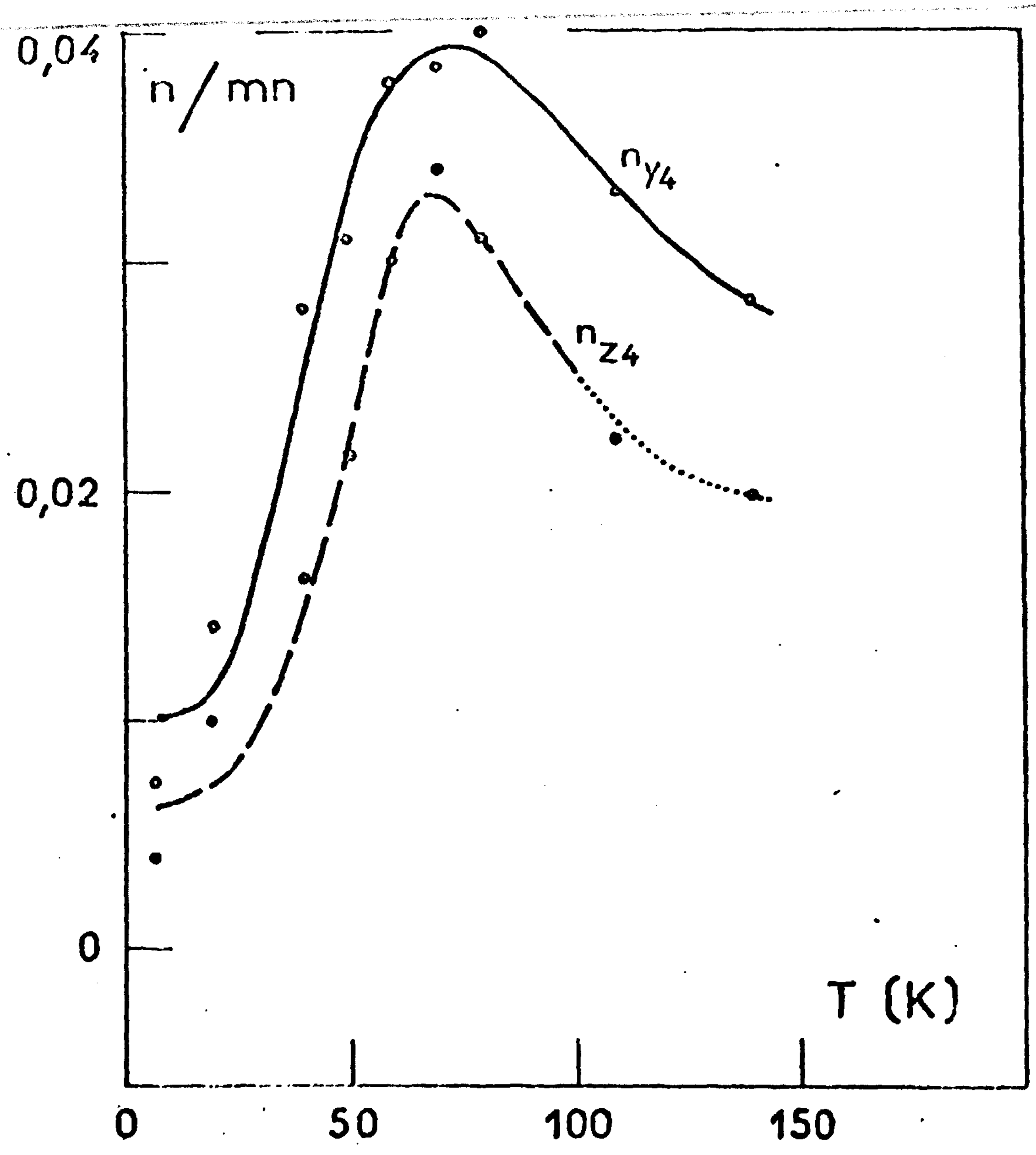
Log M_n
Ⓢ$_1$
9 K
$\varepsilon = 20.10^{-7}$
$\varepsilon = 14.10^{-7}$
$\varepsilon = 9.10^{-7}$
$\varepsilon = 2.10^{-7}$
$\varepsilon = 3{,}5.10^{-7}$
$\varepsilon = 5.10^{-7}$
0,5
0,4
0,3
0,2
0,1
0
0
0,1
0,2
0,3
0,4
0,5
Log δ_n

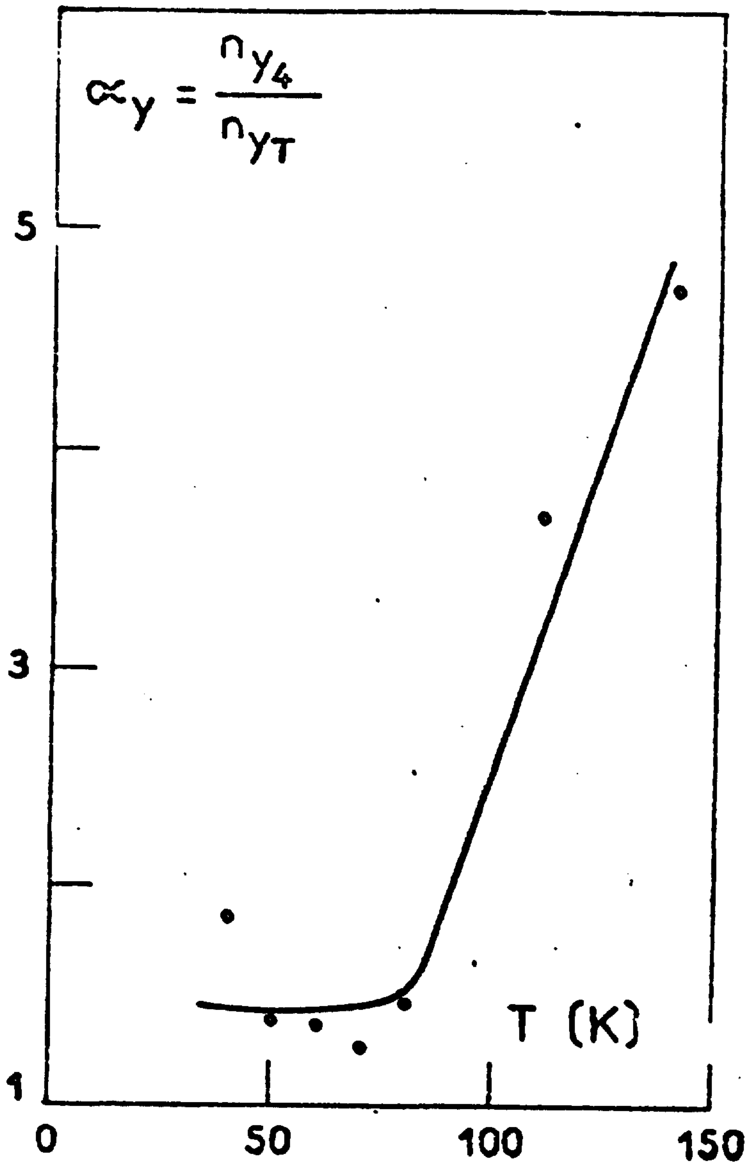
2

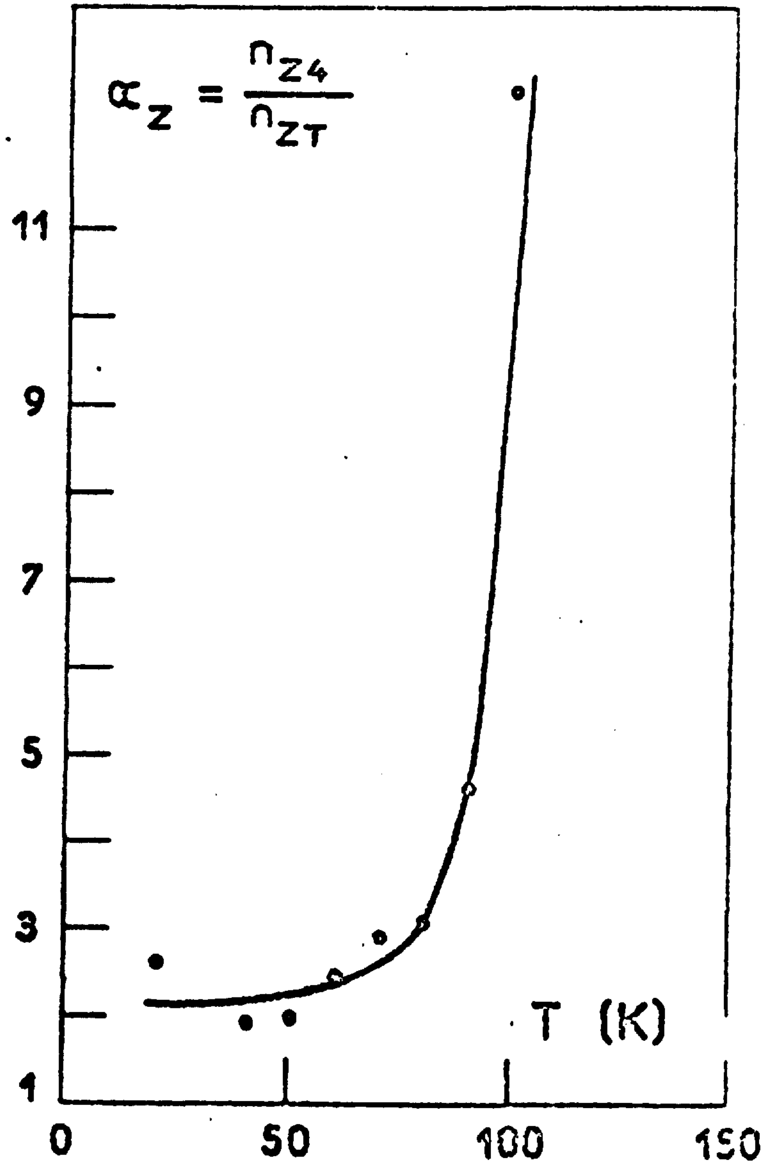

$\frac{Q^{-1}}{(Q^{-1})_0}$
1,2
1
0,8
0,6
0,4
0
20
40
t mn
ε = 20·10⁻⁷
ε = 14·10⁻⁷
ε = 9·10⁻⁷
ε = 5·10⁻⁷
ε = 3,5·10⁻⁷
ε = 2·10⁻⁷
160 K
115 K
φ = 9·10¹⁰ el/cm²·s
S1
ε = 20·10⁻⁷
ε = 2·10⁻⁷
ε = 3,5·10⁻⁷
75 K
60 K
φ = 2,2·10¹⁰ el/cm²·s

2

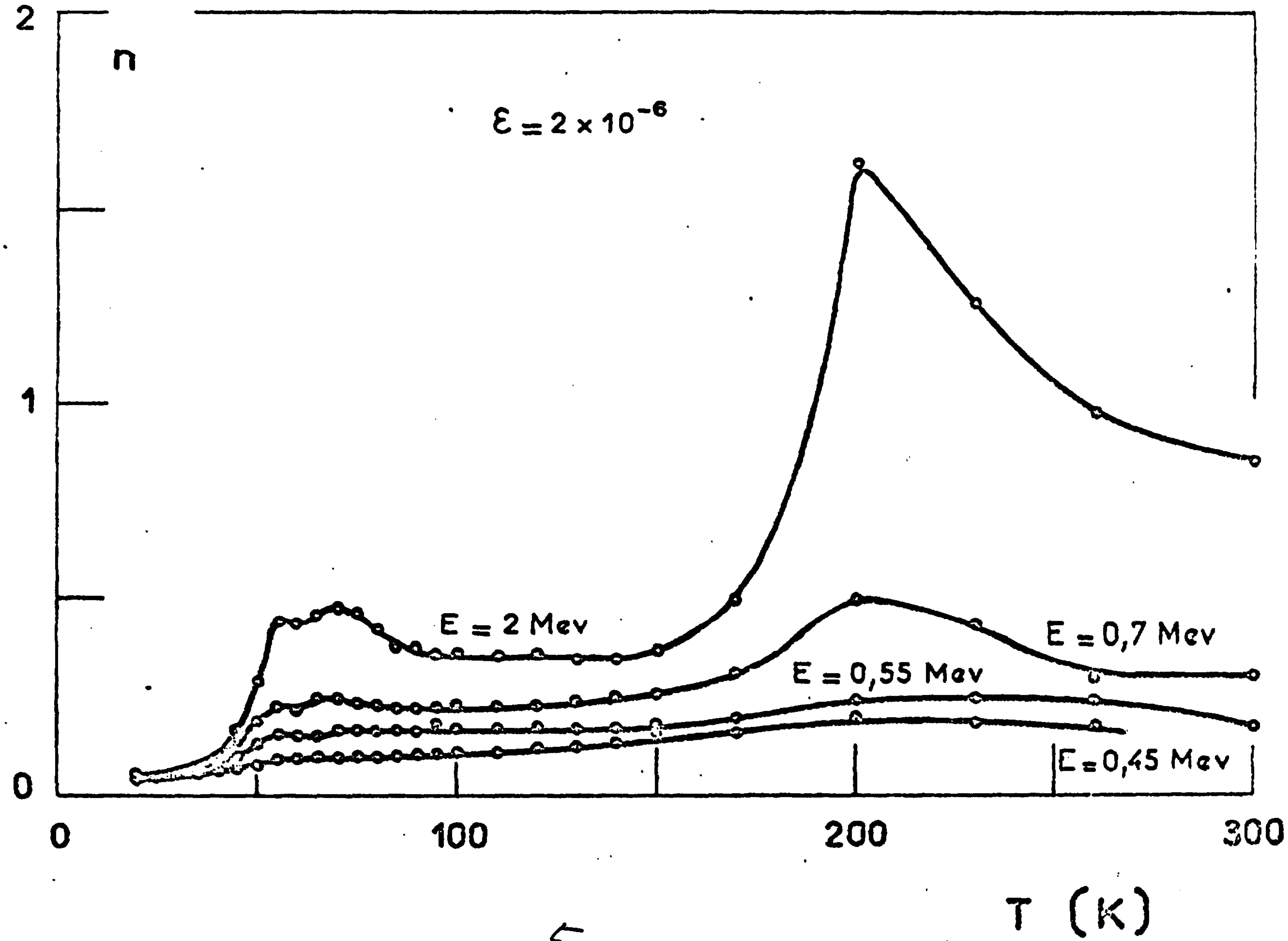


3

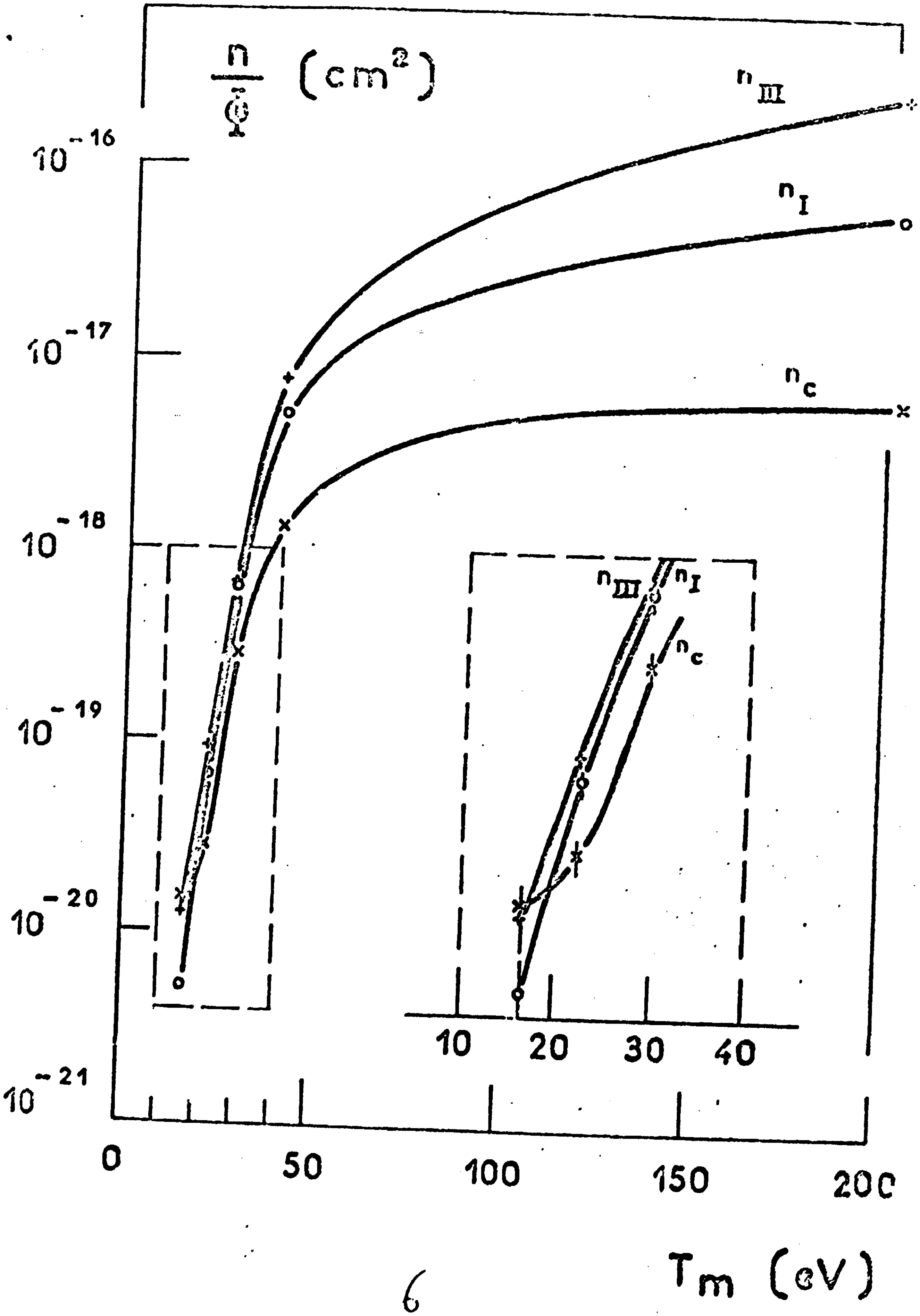

$\alpha_y = \frac{n_{y_4}}{n_{y_T}}$
5
3
1
0
50
100
150
T (K)


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

4

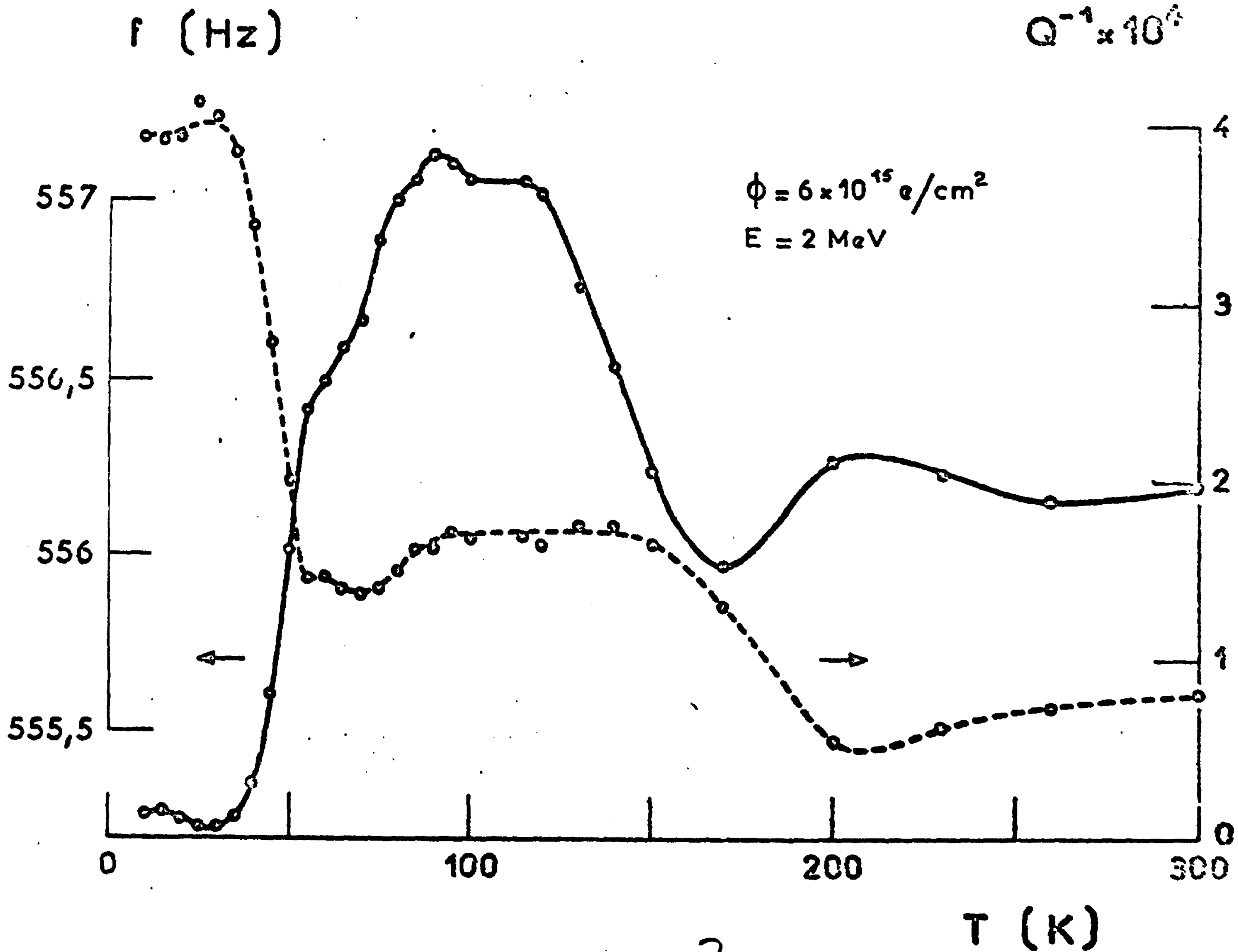

2
1
0
n
$\varepsilon = 2 \times 10^{-6}$
E = 2 Mev
E = 0,7 Mev
E = 0,55 Mev
E = 0,45 Mev
0
100
200
300
T (K)

5



$\frac{n}{\bar{\Phi}}$ (cm²)
10^{-16}
10^{-17}
10^{-18}
10^{-19}
10^{-20}
10^{-21}
n_{III}
n_I
n_c
n_{III}
n_I
n_c
10
20
30
40
0
50
100
150
200
Tm (eV)

6


f (Hz)
$Q^{-1} \times 10^{4}$
557
556,5
556
555,5
4
3
2
1
0
$\phi = 6 \times 10^{15}$ e/cm^2
E = 2 MeV
0
100
200
300
T (K)

7