DEUTSCHES ELEKTRONEN-SYNCHROTRON DESY

Optical Constants of Germanium
in the Region of the $M_{4,5}$ Edge

by

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Abstract

Reflectance measurements with polarized light for a germanium single crystal in the region between 310 and 550 Å (22.5 - 40 eV) for angles of incidence from 15° to 75° in steps of 7.5° are reported. The synchrotron radiation from DESY was used as a light source. Optical constants n, k, and thus ϵ_1, ϵ_2, and $|\text{Im } \epsilon^{-1}|$ were established by a least squares fit to the observed reflectances. The region of the present study includes the $M_{4,5}$ absorption edge from 29.5 to 32 eV. The results are discussed in connection with sum rules and the double plasma loss.

1. Introduction

The optical constants of a germanium single crystal have been determined in the region between 310 and 550 Å (22.5 - 40 eV) through reflection measurements. The highly polarized continuous spectrum of synchrotron radiation emitted by the 6 GeV electron synchrotron DESY was used as a light source.[1] Reflection measurements were carried out for angles of incidence from 15° to 75° in steps of 7.5° for both directions of polarization. Optical constants were established by a least squares fit to the observed reflectances.

The spectral region observed includes the leading part of the $M_{4,5}$ absorption band, namely the optical transitions from 3d to conduction band states. Hunter has made thin film transmission measurements to evaluate the extinction coefficient k in this region.[2,3] His result is essentially confirmed by our work but we find a slight difference near the edge. The spectral region of the present study partially overlaps with some previous reflection measurements of the optical constants. Recently Marton and Toots (M-T) published a result obtained with photon energies up to 25 eV for foils evaporated in high vacuum.[4] They demonstrated the effect of surface contamination on reflection measurements and the optical constants derived from them. The results discussed here have been obtained with the same specimen and preparation technique similar to that previously employed by Sasaki (S)[5]. The results are compared with S and M-T data in their region of overlap. The effect of surface contamination is apparently not as striking in this region

as it is for lower photon energies, but there are slight discrepancies from the high vacuum data, particularly for the refractive index n.

We give in section 2 a brief description of the experimental technique for reflection measurements with the synchrotron radiation as a light source. Problems concerning the determination of the optical constants in the extreme ultraviolet region where n approaches unity and k tends to be very small are discussed in section 3. The results are summarized and discussed in section 4.

2. Experiment

The continuous spectrum emitted by the Deutsches Elektronen-Synchrotron DESY served as the light source.[1] Throughout this measurement, the synchrotron was operated at 3.7 GeV. Our monochromator has been described elsewhere.[6] It is a 2 m normal-incidence type with a gold coated Bausch & Lomb grating. At the exit slit of the monochromator is attached a reflectometer which contains coaxial gear systems for driving the specimen and the detector separately.[7] The reflectance can be measured for angles from 15° to 82.5° in steps of 7.5°. The entire sample and detector assembly of the reflectometer can be rotated around the axis of the incident light beam. This allows reflection measurements to be made with light of any desired polarization. In this experiment the two states of polarization in which the plane of incidence is either parallel or perpendicular to the orbital plane of

electrons in the synchrotron have been measured. These two orientations we shall denote by subscripts p and s respectively. Since the synchrotron radiation is highly polarized parallel to the orbital plane, this nomenclature represents approximately the reflectance measured with 100 % polarized light.

Our detector, a Bendix M-306 electron multiplier, was used in a DC mode in connection with a Hewlett-Packard 419 A amplifier and an XY plotter. The voltage across the dynode was carefully controlled so that the output current remained below the region of nonlinear response. Throughout the measurements the overall voltage applied to the multiplier was 1800 V and the output current was less than 10^{-8} Amp.

The useful wavelength range spanned with this system extends from about 300 to 1000 $\mathring{A}$. At the shorter end of this region, there seemed to be difficulties arising from the stray light component of longer wavelengths. In order to eliminate the stray light, a thin unbacked foil of aluminum-tellurium was prepared by evaporation of 2000 $\mathring{A}$ Te onto an unbacked 1000 $\mathring{A}$ thick Al film. The spectrum obtained with the Al-Te filter is shown in Fig. 1. The effect of the filter on measurements near the $M_{4,5}$ edge of germanium is demonstrated by Fig. 2.

The specimen was prepared from the same single crystal of germanium that has been employed previously by one of the

authors[5] for measurements at longer wavelengths. Preparation of the surface was made nearly as before, the only difference being the annealing of the specimen in situ. Just after mounting in the reflectometer the crystal was heated for one hour at temperatures up to 250° C in a pressure of 2 x 10^{-5} torr.

3. Determination of Optical Constants

Reflectance spectra were obtained for angles of incidence from 15° to 75° in steps of 7.5° for both s and p polarization (Fig. 3a, b). No absolute determination of reflectance was attempted because neither the radiation field nor the sensitivity of the photocathode is homogeneous. Inhomogeneity may introduce some systematic errors into reflection measurements due to the inversion of the intensity distribution of the radiation upon the reflector. Therefore, determination of optical constants was made in a manner similar to that discussed by Hunter[8]: The ratio of any two recorded intensities, $I_s(\phi_i)/I_s(\phi_j)$, is compared with the ratio $R_s(\phi_i)/R_s(\phi_j)$ calculated from an input set of n and k, where ϕ_i and ϕ_j stand for the different angles of incidence, and R_s for the calculated "s" reflectance with the correction for some assumed degree of polarization. Then the error function, namely,

$$E = \sum_{i>j}^{N} \left(\{ I_s(\phi_i)/I_s(\phi_j) - R_s(\phi_i)/R_s(\phi_j) \}^2 / N(N-1) \right)^{1/2} \quad (1)$$

a sum for every set of two angles, is computed for the initial value (n_0, k_0) and the four neighboring points $(n_0 \pm \Delta n, k_0)$, $(n_0, k_0 \pm \Delta k)$ where Δn and Δk are stepping parameters[9]. If a point (n_1, k_1) other than (n_0, k_0) gives the minimum error, the procedure is repeated around the new central point (n_1, k_1). If (n_0, k_0) is the minimum, the steps Δn, Δk are reduced by a factor of 2/3 and the procedure is continued. The final value of (n, k) is determined either when E reaches a predetermined criterion or at the end of 26th iteration. The optical constants are determined in this manner assuming several different degrees of polarization. In the region covered by the present study, i.e. 300-550 Å, the theoretical value of the degree of polarization $P = (I_{||} - I_{\perp})/(I_{||} + I_{\perp})$ of the synchrotron radiation lies between 0.78 and 0.83 with the assumptions that the maximum electron energy is a little less than 4 GeV and that the radiation is accepted with an aperture 30 mm high at 40 m from the emitting electron. Since a recent measurement of P made by Rosenbaum et al.[10] has shown good agreement with the theoretical estimation, the above values should be quite reasonable. In the present measurement the beam is restricted further by an aperture 4 mm in diameter in front of the reflectometer, consequently the values of P should be somewhat higher than the above values. In computing the optical constants, the range of P between 0.82 and 0.88 was covered in steps of 0.02. The results suggest that a small error in P has no significant effect upon the final values of optical constants.

The above mentioned procedure was tried for three groups of data, i.e. $I_s(\phi_i)/I_s(\phi_j)$, $I_p(\phi_i)/I_p(\phi_j)$, and $I_s(\phi_i)/I_p(\phi_i)$. The data of the last group were studied only as a matter of reference, since these data may include additional errors due to inhomogenity introduced by taking the ratio of two measurements in different orientations of the system. After several test computations, it was soon recognized that these three groups of data lead to different results both for n and k. The results are normally within 10 % of each other, but in extreme cases the variations are as high as 100 %, particularly for k. Difficulties mainly come from the fact that we are in the region where n is nearly unity and k is very small. This situation is typical in the soft x-ray region. In this case, Brewster's angle is near 45° where the ratio $\rho \equiv R_p/R_s \approx 2k^2$. Since k is very small, ρ is extremely small. We have to measure a very weak signal for R_p at angles near 45°. That this is the case for "R_p" measurements in the present work is demonstrated by Fig. 3b. When k is smaller than 0.1 and P is in the range 0.8 to 0.9, a "p" measurement senses, in reality, only the small component of s light reflected. The observed ratio ρ should approximately represent P. That it does has been confirmed.

In the soft x-ray region where $\delta \equiv 1 - n \ll 1$, and $k \ll 1$, $R(\phi)$ may depend in general more strongly on δ than on k. If $\partial R/\partial k$ is very small, an accurate determination of k is difficult. Similar arguments can be made for n or δ. It is clear that any measurement includes some errors in $R(\phi_i)$ and thus in $R(\phi_i)/R(\phi_j)$. This leads to large deviations in k (or n)

if $\partial R/\partial k$ (or $\partial R/\partial n$) is small. A small value of $\partial R/\partial k$ also makes the convergence in the computer calculations described above very slow, because the small change of k makes no improvement in the error function. The values of $\partial R/\partial k$ are, of course, dependent upon n, k, ϕ, and P. One possibility of reducing the errors and improving the speed of convergence is to omit data lying in the region where these differentials are small. It should be done, however, during the process of computation, since it is impossible to know the exact situation previous to the computation. This will introduce time consuming complexity into the program, because the differentials change drastically in this region. As an example, the dependence of R_s on both δ (0.01 to 0.3) and k (0.001 to 0.1) at $\phi = 75^\circ$ is shown in Fig. 4. In this particular case, the dependence of R upon k is very weak except in a small region with $\delta < 0.02$, whereas $\partial R/\partial n$ is large except in the region $(k < 0.01, \delta > 0.1)$. In general, $\partial R/\partial n$ is larger than $\partial R/\partial k$ throughout the region studied in the present measurements. The poor consistency of the k values we have derived from our experiment is therefore not surprising. On the other hand, the accuracy of n is better than $\pm 5 \%$ throughout the region. The values of k may be improved by direct determination through transmission measurements on thin foils only if the thickness and structure of the foil are accurately controlled. To date no accurate transmission measurements have been attempted.

In order to determine which one of our three data groupings should yield the best values and the quickest convergence, test computations have been carried out. The test starts with a choice of (n, k) and calculation with Fresnel's formula of R_s (ϕ_i) and $R_p(\phi_i)$ for several angles and degrees of polarization. The first three significant figures of a reflectance calculated from the assumed (n, k) are taken as an input value of reflectance for the computer calculation. Then a different point (n, k) is given as a starting point and the computation by three different sets of input data are carried out. The speed of convergence, the magnitude of the error function E defined by Eq. (1), and the deviation of the final values from the correct ones have been compared for typical values of n and k in the range of interest here, namely, k = 0.01 - 0.1 and n = 0.8 - 1.1.

The test results show some deviations from the correct values for both n and k, partly due to the cutoff of the reflectance input and partly due to slow convergence. Errors introduced into n are usually less than 2 % but are sometimes as high as 5 %, whereas they are normally within 30 % for k but in some unfavorable cases are higher than 100 %. Among our three data groupings "s" gives best convergence, except in two small regions around 29.5 and 35 eV. In order to illustrate these points, the following sensitivity function is defined and plotted for several regions of the spectrum in the present study.

$$S_f^i \equiv \frac{f}{R_i} \frac{\partial R_i}{\partial f} \tag{2}$$

where i specifies s or p and f stands for n or k. Since this quantity is the ratio of relative error in reflectance to the relative error in optical constants, it serves as a measure of accuracy in determining optical constants from reflection measurements. As illustrated in Fig. 5, the depencence of S_f^i upon angle of incidence is critical for n. It should be noted that the advantage of high reflectance at large angles is sometimes negated by small values of S_f^i. The advantage of taking the "s" data group is also observed in the figure. These features are typical in the soft x-ray region. Sometimes, the values of S_k^p are quite low. Note, however, that a study of the accuracy in determining optical constants from reflection measurements made by Hunter[8] generally suggests a superiority of "p" polarization over "s". The present case should be considered as an exception. Where there are some discrepancies in our results, we have chosen those obtained with "s" polarization which we believe to be most dependable. The discussion given below is based upon the "s" results.

4. Results and Discussion

The spectral behavior of the reflectance in arbitrary units is shown in Fig. 3. The structure around 400 Å arises from the $M_{4,5}$ absorption edge. The minimum of reflectance at 15°

lies at about 420 Å and the maximum at 400 Å. In R_s these
values shift toward shorter wavelengths as the angle of
incidence increases. For R_p the behavior is a little more
complicated. The shifting results from the fact that the
onset of total reflection and its sharpness depend upon the
gradually changing optical constants. In R_p, $\phi = 45°$ gives
the minimum throughout this series of measurements, suggesting
that n is close to unity. The spectral behavior is quite
smooth except for the region of the $M_{4,5}$ edge and a small
shoulder appearing around 375 Å (33 eV). The data, especially
for R_p, below 330 Å are apparently influenced by stray light,
and the statistical fluctuation of the signal as indicated in
the figure is so high that the reliability of the data for R_p
is lower than that of the R_s data.

The optical constants, n and k, determined from the first
group of data, $R_s (\phi_i)/R_s(\phi_j)$, are shown in Fig. 6 together with
the previously published results of Marton and Toots and of
Sasaki. In the region of their overlap the value of n found
in the present measurement is a little lower than that of S
and higher than that of M-T. The variation of P from 0.82 to
0.88 in our data analysis gives a deviation of $\sim$ 0.3 % from
the central value for n and $\sim$ 3 % for k.

According to Marton and Toots, who made their measurements
with a foil prepared in high vacuum, the effect of surface
contamination is conspicuous for n, while k is less
sensitive.   Thus the difference in the value of n
found in the   various measurements

shown in Fig. 6 may be considered as a measure of surface contamination. The difference in n between S, which was obtained with the single crystal once exposed to the air, and M-T in Fig. 5. is quite large below 15 eV, but above 15 eV the difference gradually decreases with energy. Apparently the influence of surface contamination is less important in the region studied in the present measurement than at lower energies. A slight decrease of n at 23 eV from the previous data of S with the same crystal may be explained by the _in situ_ annealing after the crystal was mounted. Robin-Kandare[11] reported that reflectance is remarkably increased after annealing of a mechanically polished germanium single crystal. However, the values of n determined in the present work are still slightly higher than M-T in the region 22.5 to 25 eV, indicating that the influence of surface contamination has not been completely removed. The values of k in the same region are in agreement with M-T to within the experimental error. The spectrum of k evaluated from the transmittance measured with a thin evaporated foil of germanium, presumably exposed to air, has been reported by Hunter in the region 120 to 680 $Å^2$. His results are essentially in agreement with the present measurement except that he finds slightly higher values for k below the $M_{4,5}$ edge. Both our results and those of Hunter show a small hump in k just below the edge, the origin of which is not clear. It could not be predicted by a glance at the reflectance curves. On the contrary, the shoulder at 375 Å which is clearly seen in some of the reflectance curves is

reproduced neither in k nor in n. The spectral behavior of the dielectric functions $\epsilon_1(\omega)$, $\epsilon_2(\omega)$, and $|\mathrm{Im}\epsilon(\omega)^{-1}|$ is shown in Fig. 7. $\epsilon_1(\omega)$ nearly reproduces $n^2(\omega)$ because n >> k. The behavior of $\epsilon_2(\omega)$ is governed primarily by k(ω) with only minor modification by n(ω), since the variation of n in this region is $\lesssim$ ± 10 %. The loss function $|\mathrm{Im}\epsilon(\omega)^{-1}|$ is quite similar to $\epsilon_2(\omega)$, since ϵ_1^2 >> ϵ_2^2 and $\epsilon_1^2 \approx 1$, as is often the case in the soft x-ray region. ϵ_2 and $|\mathrm{Im}\epsilon^{-1}|$ exhibit peaks at 32 and 33 eV, respectively, which are close to $2\hbar\omega_p$. The value of $|\mathrm{Im}\epsilon^{-1}|$ at the peak is approximately 0.2. The values of $|\mathrm{Im}\epsilon^{-1}|$ at $\hbar\omega_p$ = 16 eV obtained by optical measurements are 1.7 in S and 3.5 in M-T, whereas the electronic energy loss data for crystalline thin foils give values between 3.9[14] and 5.4[12]. A consideration of these results suggests that there may be a competition between double plasmon excitation and the interband excitation of individual electrons in the characteristic energy loss spectra near $2\hbar\omega_p$. The competition should depend upon foil thickness and the primary electron energy. In some of the electron energy loss data for thick foils[13] the secondary peak of the loss spectrum at around 32 eV is as high as 20 or 30 % of the plasmon peak at 16 eV. On the other hand, the data for foils thinner than 500 Å give a much smaller peak at $2\hbar\omega_p$[14]. In the thin foils the peak at $2\hbar\omega_p$ was found to be only 5 % of the plasmon peak. This is in good agreement with the optically determined ratio of $|\mathrm{Im}\,\epsilon^{-1}|$ at 32 and 16 eV. In the latter case, the major part of the loss is probably caused by the interband transition. When the estimation

of the mean free path of inelastically scattered electrons is attempted for doubly scattered electrons[15], this background loss to the interband transition should be taken into account.

In Fig. 8 the number of electrons contributing to the optical transitions and also their contribution to the static dielectric constant $\varepsilon_{o,eff}$ is shown. These curves were evaluated with the aid of the following partial sum rules[16]

$$\int_0^{\omega_o} \omega \varepsilon_2(\omega) d\omega = \frac{2\pi^2 N e^2}{m} \cdot n_{eff} \qquad (3)$$

$$\int_0^{\omega_o} \frac{\varepsilon_2(\omega)}{\omega} d\omega = \varepsilon_{o,eff} \qquad (4)$$

where N represents the atomic density, while n_{eff} gives the number of electrons per atom. The results have been obtained from our measurements together with those of several other authors for lower energies. The data included in this evaluation are from Refs. 17 and 18 for $\hbar\omega \ll 1$ ev, P-T[19] for $1 < \hbar\omega \leq 8$ eV, and M-T for 8 to 25 eV as shown in Fig. 9. The evaluation of ε_2 ┤ by Phillip and Ehrenreich[20] with their last measurement (indicated as P-E in Fig. 9) gives higher values of ε_2 than P-T and M-T throughout the region of their overlap. n_{eff} is as high as 6 at 23 eV, which lies far from the onset of the $M_{4,5}$ excitation. Phillip and Ehrenreich ascribed this discrepancy from 4, the number of valence

electrons per atom, to the enhancement of the oscillator
strength f arising from the negative contribution to f
by 3d electrons[20].

Our results indicate that the oscillator strength is
saturating toward the onset of $M_{4,5}$ absorption. The value
4 is reached at 32 eV where there is a sharp rise of n_{eff}
corresponding to the onset of the core excitation. Thus,
there is apparently no enhancement of the oscillator strength
due to the d band. A recent result by MacRae et al.[21] leads
to a similar conclusion for white tin.

If we take P-E values instead of those of P-T in the region
between 1 and 8 eV, n_{eff} at 8 eV is 0.8 higher than our value
shown in Fig. 8 and n_{eff} reaches 4 at 9 eV. Further study
of an uncontaminated germanium crystal in this region is
desirable in order to settle unequivocally the question of
whether or not enhancement occurs.

The region between 30 and 40 eV contributes only a small
portion of the oscillator strength available for 3d electrons
in the $M_{4,5}$ absorption band. This fact suggests that the
major part of the $M_{4,5}$ absorption lies somewhere between
40 and 120 eV, where the $M_{2,3}$ edge is located. Note also
that $\delta\varepsilon_o = \varepsilon_o - \varepsilon_{o,eff}$ at 40 eV is still larger than 1.

The general behavior of the spectra could be interpreted
by comparing the curve for ε_2 in Fig. 7 with the theoretical
conduction band structure and the fundamental absorption
spectra in the infrared and visible regions. In Fig. 10
we have reproduced a portion of the band structure of
germanium derived by Brust[22]. Because the initial states
are of pure d symmetry, many singular points of primarily s
or d character in the conduction band states which play
important roles in the fundamental absorption spectrum of
germanium are less important in $M_{4,5}$ absorption, where the
most important singular point is Γ_{15}, a triply degenerate
p-type state. If one assigns the peak at 32 eV to Γ_{15}, the
width of the "edge" gives the distance of this point from the
bottom of the conduction band $\Gamma_{2'}$. The corresponding energy
difference 2.5±0.2 eV is consistent with the value 2.4 eV
from fundamental absorption data, while the theoretical band
calculation by Brust gives 2.9 eV and that of Bassani and
Yoshimine[23] 2.6 eV. If one attributes the small hump in the
reflectance spectra to L_3, the separation $L_1 - L_3$ is estimated
at 3.8±0.3 eV assuming L_1 lies 0.1 eV above $\Gamma_{2'}$, in good agree-
ment with the fundamental spectra (3.5 eV) and also the band
structures of Brust (3.7 eV) and Bassani (3.4 eV), whereas
the latter locates L_3 below Γ_{15}, which is not compatible with
$M_{4,5}$ absorption.

Acknowledgments

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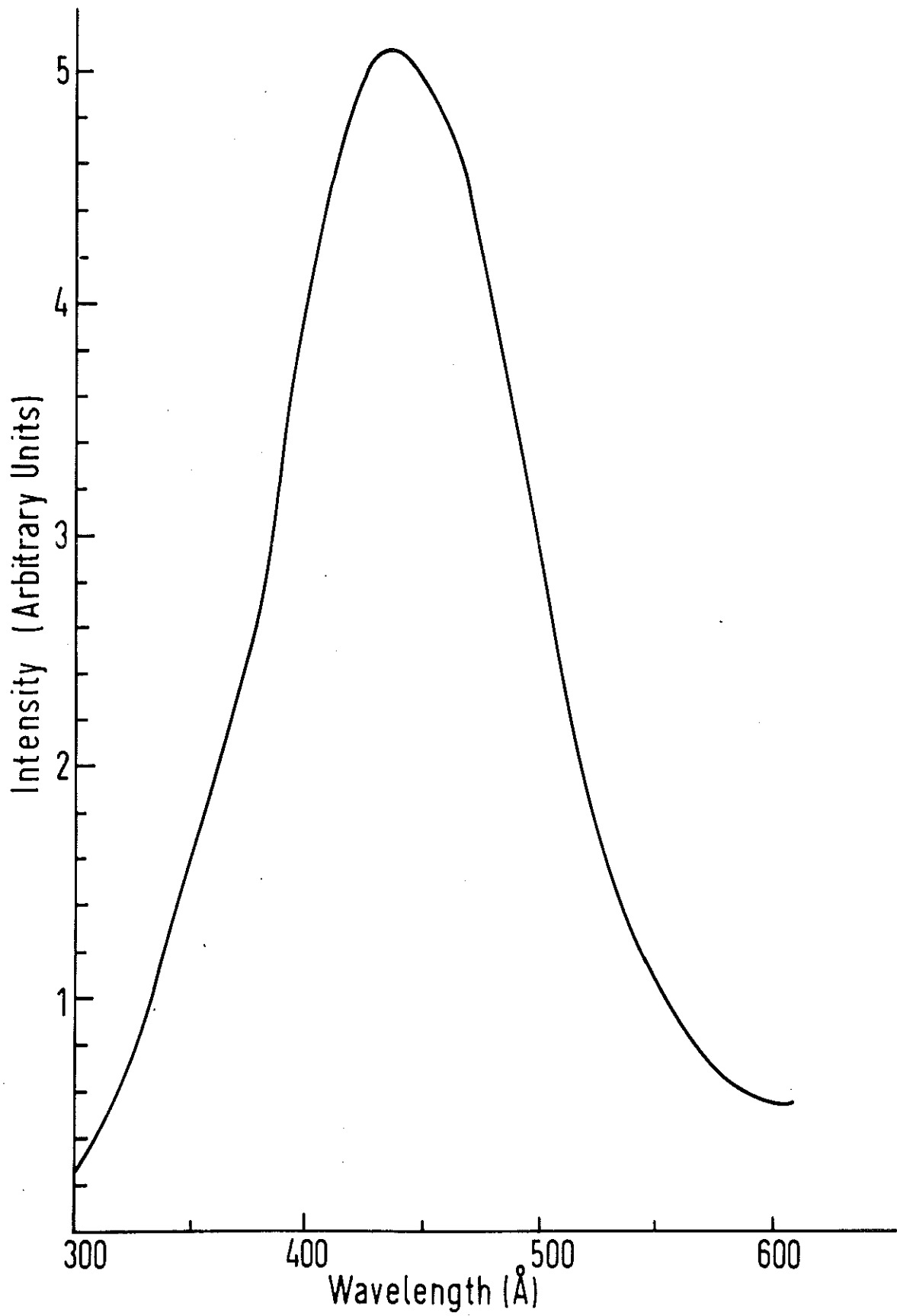23) F. Bassani and M. Yoshimine, Phys. Rev. 130, 20 (1963)
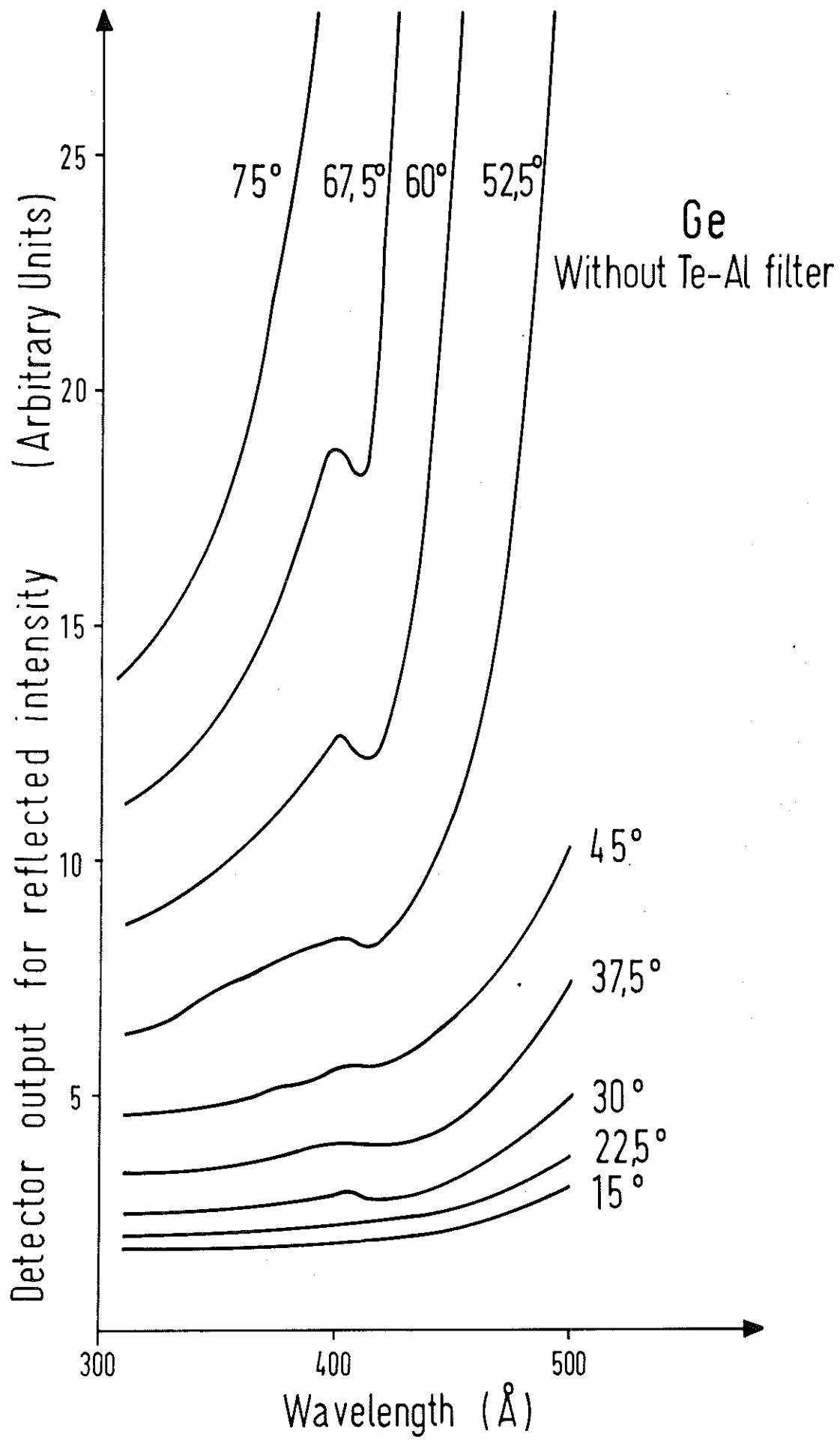
Footnotes

Figure Captions

Fig. 1   Spectrum of synchrotron radiation from DESY observed
with a normal incidence monochromator and Bendix
M-306 multiplier using an Al-Te filter.

Fig. 2   Detector output for reflection spectra R_s of germanium.
a) without Al-Te filter   b) with Al-Te filter.

Fig. 3   Spectral behavior of germanium reflectance for
various angles of incidence.
a) R_s b) R_p
Subscripts s and p indicate the major component
of polarization.

Fig. 4   Dependence of R_s (75°) upon $\delta = 1-n$ and k, where
both δ and k are small.

Fig. 5   Sensitivity function $S_f^i = \frac{f}{R_i} \frac{R_i}{f}$ (i = p,s; f = n,k)
calculated for some typical values of optical
constants studied in the present work. The values
in brackets are wavelengths.

Fig. 6   Optical constants n and k of germanium. Those from
the previous works by T. Sasaki (S) and by L. Marton
and J. Toots (M-T) in the region of lower photon
energies are also included for comparison.

Fig. 7   Dielectric functions ε_1, ε_2, and $|Im \frac{1}{\varepsilon}|$ of germanium
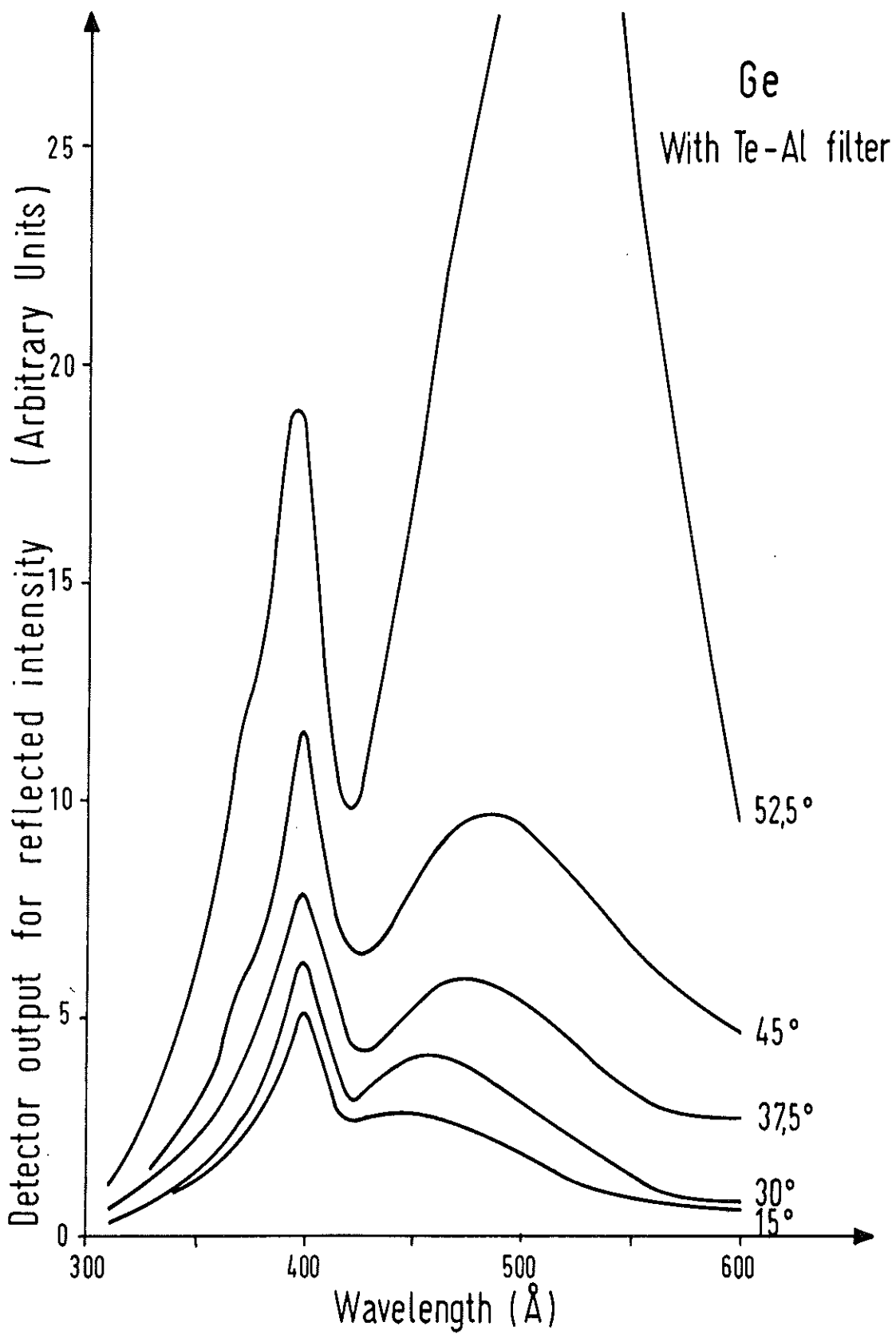as a function of photon energy.

Fig. 8 The effective number of electrons per atom n_{eff} and the contribution of the optical absorption to the static dielectric constant $\varepsilon_{o,eff}$ as functions of photon energy calculated from the data shown in Fig. 9.
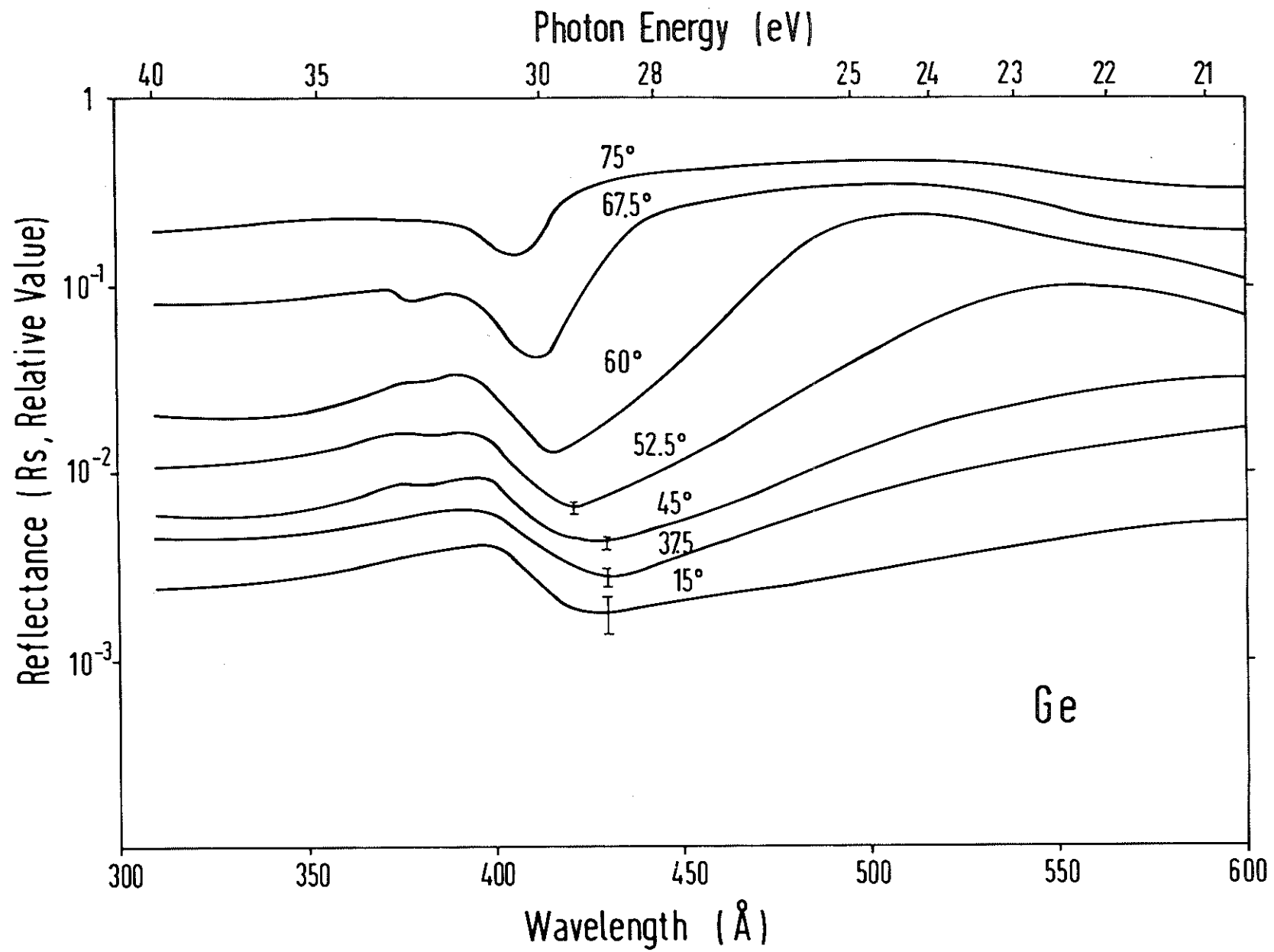
Fig. 9 ε_2, the imaginary part of the dielectric constant, as a function of photon energy derived from the works of various authors: DN-W.C. Dash and R. Newman, SV-C.D. Salzberg and J.J. Villa, PT-H.R. Philipp and E.A. Taft, PE-H.R. Philipp and H. Ehrenreich S-T. Sasaki, MT-L. Marton and J. Toots.
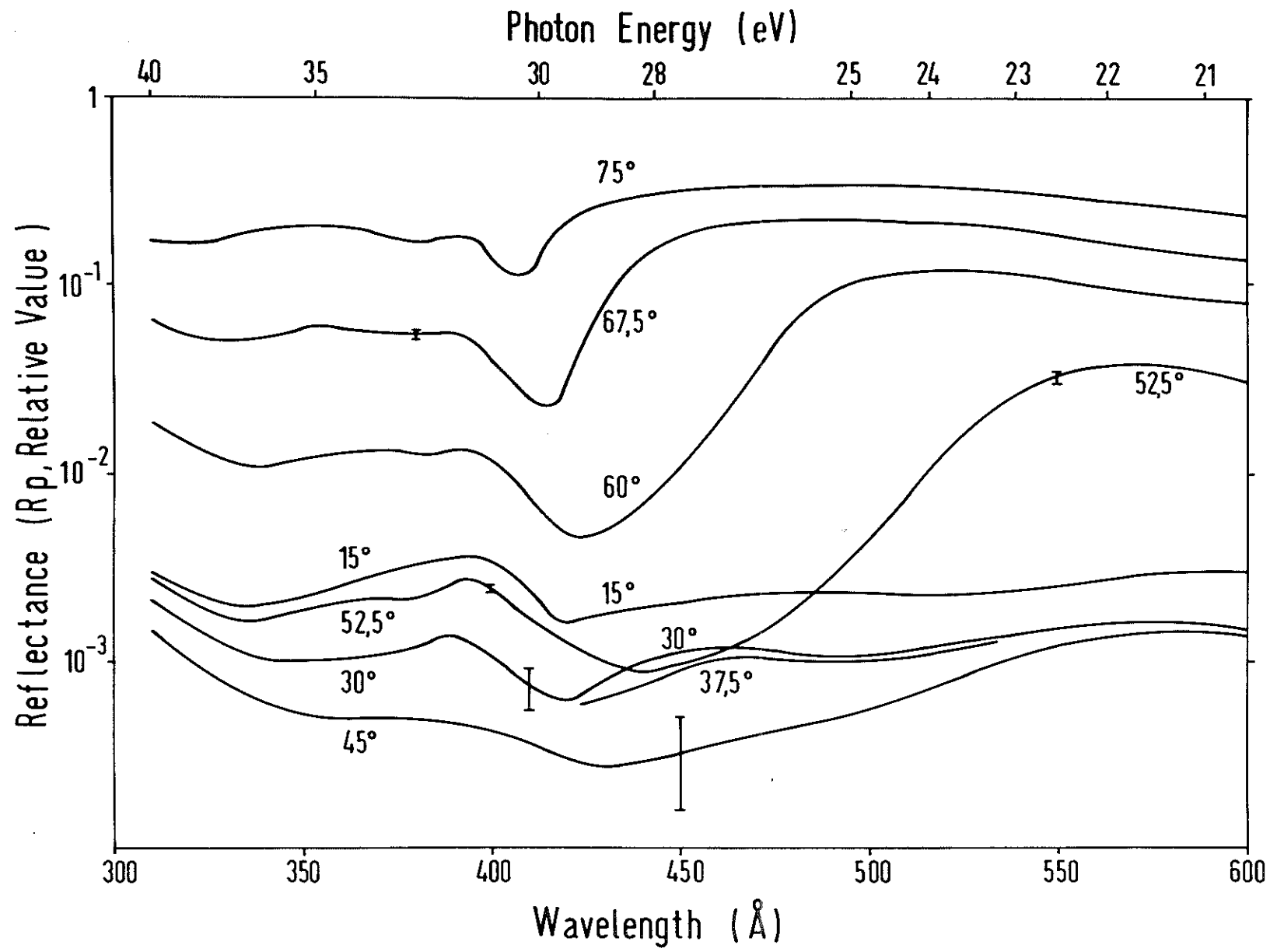
Fig. 10 A portion of the band structure of germanium according to Brust.

$\varphi = 75°$