

*DETERMINATION OF MICROCHARACTERISTICS OF LEAD FROM MEASUREMENTS OF ITS OPTICAL CONSTANTS AND SPECIFIC CONDUCTIVITY*

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Submitted to JETP editor July 7, 1962

J. Exptl. Theoret. Phys. (U.S.S.R.) 44, 398-404 (February, 1963)

The optical constants of lead were measured in the range  $0.7-12\mu$  at room temperature and at liquid nitrogen temperature. The static conductivity and density of the same samples was determined. The results are analyzed taking into account the skin effect, quantum corrections for the frequency of collision of electrons with the lattice and electron-electron collisions. The concentration of conduction electrons, the velocity of electrons at the Fermi surface, and the electron collision frequencies are determined.

**I**N the present work the optical constants of lead were determined over the interval  $0.7-12\mu$  at room temperature ( $293^\circ\text{K}$ ) and over the interval  $0.8-11\mu$  at the boiling point of liquid nitrogen ( $77.4^\circ\text{K}$ ); the densities and temperature variation of the static conductivity of the samples were also measured.

Measurements at the low temperatures are particularly significant since they permit an extension of the range over which the microcharacteristics can be determined with the desired accuracy.

The previous measurements of the optical constants of lead were done only at room temperature and in the interval  $1-7\mu$ .<sup>[1]</sup> They were not supplemented by conductivity and density measurements, so that one cannot judge the quality of the metallic layer or determine the various microcharacteristics of lead. In addition, in treating the experimental results certain correction terms were not included which are large for the case of lead; their omission results in sizable errors in the determination of the microcharacteristics of the metal.

The optical constants  $n$  and  $\kappa$  (the complex index of refraction  $n' = n - i\kappa$ ) were measured using a polarization method based on the determination of the phase shift  $\Delta$  between the  $p$  and  $s$  components of the reflected light, and the azimuth  $\rho$  which is given by the ratio of the amplitudes of the two components. The measurement was done so that the phase shift after a series of reflections from the mirrors gave an odd multiple of  $\pi/2$ , i.e., so that the light became circularly polarized.

The measurements at room temperature were made on two different setups. In the first arrangement (cf. <sup>[1]</sup>) we used fourfold reflection of the

light from the mirrors. For each wave length  $\lambda$ , the angle of incidence  $\varphi$ , the phase shift  $\Delta$  and the azimuth  $\rho$  were measured. Measurements of  $n$  and  $\kappa$  for a given  $\lambda$  were made for several values of the angle of incidence  $\varphi$ . The results found for different angles of incidence coincided with one another.

In the second apparatus, described in <sup>[2]</sup>, an eightfold reflection of the light from the mirrors was used. In the measurements,  $\varphi$  was fixed and  $\lambda$ ,  $\Delta$ , and  $\rho$  were measured. For a fixed value of  $\varphi$  one could obtain circularly polarized light for several values of  $\lambda$ . Each value of  $\lambda$  corresponded to a total phase shift of  $\pi/2$ ,  $3\pi/2$ ,  $5\pi/2$ ,  $7\pi/2$ ... In this work we used shifts up to  $11\pi/2$ . To cover the range  $0.7-12\mu$  we used angles of incidence from  $57$  to  $80^\circ$ . The low temperature measurements were made only on the second apparatus.

For both equipments the light source was a dc carbon arc. The light was monochromatized using an NaCl prism monochromator. The polarization was gotten using an eight-layer selenium pile having a working diameter of 24 mm. The light detector was a germanium bolometer built by A. A. Shubin.

One important feature of the measurements at low temperatures is that different parts of the equipment are at different temperatures. This results in the appearance of a "parasitic" signal, which can seriously distort the results. To eliminate this effect, in the second arrangement the monochromator was placed between the analyzer and the detector. The effect of the "parasitic" signal was completely absent in the range  $0.8-8\mu$ . For measurements in the range  $8-11\mu$  this signal was noticeable. A special procedure for compen-

sating it had to be used.

Placing the monochromator as described above means that we must take account of the polarization which it produces. Special measurements were made of the degree of polarization produced by the monochromator over the whole spectral range used. For each wave length the polarization measurements were made with the same slits as used in the optical constant measurements. In the first apparatus, the monochromator was located between the light source and the polarizer. For this arrangement, with the method used for measuring the polarization, the effect of the monochromator is negligible. The results of measurements of the optical constants at room temperature using the two arrangements coincided, which shows that the degree of polarization produced by the monochromator in the second apparatus was correctly taken into account.

The measurements were made on layers of lead evaporated in vacuum onto polished glass plates. The deposition was done from tantalum boats at a pressure of  $(5-7) \times 10^{-6}$  mm Hg in the vacuum system. The purity of the lead was 99.999%.<sup>1)</sup> Lead deposits easily on a glass backing, so that getting good mirrors presents no special difficulties. The lead mirrors have a dark hue. The thickness of the layers deposited was  $0.6-1.6 \mu$ . Measurements were made immediately after deposition as well as after keeping the mirrors for several days in vacuum. Identical results were found in the two cases. The measurements lasted several hours. To study the effects of oxidation on the optical constants, control measurements were made after leaving the mirrors in air for a day. These measurements showed that the optical constants do not change within this period. Thus there is no effect of oxidation on the results of the measurements. Noticeable changes in the optical constants appeared only after several days.

At the same time as the mirrors were deposited, special samples for measuring conductivity and density were prepared.

For the conductivity measurements, a lead band was deposited onto a glass backing through a stencil, giving a definite length and width. The thickness was determined interferometrically. The re-

sistance was measured at room temperature, at the boiling point of liquid nitrogen and at the boiling point of liquid hydrogen.

For the density measurements, a sample of definite area was deposited. The layer thickness was again measured by an interferometric method.

The mirrors had the following characteristics: density  $11.1 \pm 0.6 \text{ g/cm}^3$  (the bulk metal density is  $11.3 \text{ g/cm}^3$ ), the conductivity at  $T = 293^\circ\text{K}$  is  $\sigma_{293} = (0.41 \pm 0.02) \times 10^{17} \text{ cgs esu}$ , and at  $T = 77.4^\circ\text{K}$ ,  $\sigma_{77} = (1.70 \pm 0.08) \times 10^{17} \text{ cgs esu}$ . (The bulk conductivity of the metal is  $0.41 \times 10^{17} \text{ cgs esu}$  at  $293^\circ\text{K}$  and  $1.70 \times 10^{17} \text{ cgs esu}$  at  $77.4^\circ\text{K}$ ). Thus to within the accuracy of the measurements, the conductivity and density of the deposited layers was the same as that of the bulk metal. To determine the residual resistance of our samples, we used the results of the measurements at liquid nitrogen temperature  $R_N$  and at liquid hydrogen temperature  $R_H$ . Comparing the ratio of the resistances at these temperatures with the resistance ratio for an ideal metal<sup>[4,5]</sup> having the same Debye temperature  $\Theta = 86^\circ\text{K}$ , it is easy to determine the residual resistance. For our layers it was 0.93% of the resistance at room temperature. The ratio of the room temperature resistance  $R_0$  to the resistance at liquid nitrogen temperature  $R_N$  was  $R_0/R_N = 4.14$  (for the bulk metal,  $R_0/R_N = 4.15$ ). For the ideal metal the ratio is 4.01. The somewhat more rapid change of resistance with temperature for the real metal can be partially explained by the presence of electron-electron collisions.

The results found for the optical constants of lead are given in Table I. The results given are averages over many series of measurements. The accuracy in determining the optical constants was 1% in the middle of the spectral region used and 1-2% at the ends. The main error arises in averaging different series of measurements. Within a single series the accuracy is still higher.

The values of  $n$  and  $\kappa$  in the spectral interval  $1-7 \mu$  found at room temperature in this work can be compared with the results in<sup>[1]</sup>. This comparison shows agreement for  $\kappa$  and a small discrepancy for  $n$ . The value of  $n$  in<sup>[1]</sup> was somewhat greater than here, which indicates better conductivity of the samples used in the present work.

A preliminary analysis of the results of the present work by the method proposed earlier<sup>[6]</sup> showed that at room temperature the mean free path of the electrons,  $l$ , is less than the skin depth  $\delta$ , and that in the wave length region considered  $\nu_{\text{eff}} \approx \omega$ , where  $\nu_{\text{eff}}$  is the effective elec-

<sup>1)</sup>From our measurements of the temperature variation of the resistivity of the polycrystalline deposited samples, it appears that the main cause of the residual resistance of the samples is usually not the presence of chemical impurities, but crystalline boundaries and structure defects. There is therefore no special need to use such pure metals.

Table I. Optical constants of lead at  $T = 293^\circ\text{K}$  and  $77.4^\circ\text{K}$ 

$\lambda, \mu$	$T = 293^\circ\text{K}$		$T = 77.4^\circ\text{K}$		$\lambda, \mu$	$T = 293^\circ\text{K}$		$T = 77.4^\circ\text{K}$	
	$n$	$\kappa$	$n$	$\kappa$		$n$	$\kappa$	$n$	$\kappa$
0.7	1.63	3.78	—	—	5.0	9.03	24.8	4.10	29.5
0.8	1.48	4.34	1.04	4.12	6.0	11.7	28.1	5.75	35.5
0.9	1.41	4.94	0.946	4.64	7.0	14.1	30.9	7.50	40.3
1.2	1.44	6.60	0.655	6.43	8.0	16.4	33.6	9.35	44.4
1.5	1.66	8.30	0.595	8.25	9.0	18.7	35.8	11.1	48.2
2.0	2.27	11.1	0.78	11.3	10.0	21.0	37.4	12.8	51.5
2.5	3.19	13.7	1.10	14.4	11.0	23.2	39.2	14.4	54.7
3.0	4.26	16.4	1.45	17.5	12.0	24.6	40.5	—	—
4.0	6.57	20.8	2.50	23.6					

tron collision frequency and  $\omega$  is  $2\pi$  times the light frequency. In analyzing the data at room temperature, we therefore used the formulas for the normal skin effect without expanding in powers of  $\nu_{\text{eff}}/\omega$ . We also included the contribution to the attenuation of surface losses in the same way as in [7].

At liquid nitrogen temperature,  $l > \delta$ , while  $\nu_{\text{eff}} < \omega$ , so that one can use the more precise formulas for the anomalous skin effect, in which the relation between the optical constants and the microcharacteristics is given in the form of a series in the variables  $\nu_{\text{eff}}/\omega$  and  $v/\omega\delta$ , where  $v$  is the velocity of the electrons at the Fermi surface.

1. Analysis of experimental results for room temperature. Using the relation of the quantity  $1/\epsilon' = 1/(n - i\kappa)^2$  to the microcharacteristics, we get the following relations<sup>2)</sup>

$$\lambda^2 \frac{\kappa^2 - n^2}{(n^2 + \kappa^2)^2} = \frac{\pi mc^2 \cdot 10^8}{e^2} \frac{1}{N} = \frac{0.112 \cdot 10^{22}}{N}, \quad (1)$$

$$\lambda \frac{2n\kappa}{(n^2 + \kappa^2)^2} = \frac{mc \cdot 10^4}{2e^2} \frac{\nu_{\text{eff}}}{N} = 5.93 \cdot 10^5 \frac{\nu_{\text{eff}}}{N}. \quad (2)$$

From now on,  $\lambda$  is the wave length of the light in microns,  $m$  is the mass of the free electron,  $c$  is the velocity of light in vacuum,  $e$  is the charge on the electron, and  $m$ ,  $c$ , and  $e$  are in cgs esu.

These relations are derived on the assumption of a spherical Fermi surface.<sup>[8]</sup> The actual Fermi surface for lead differs from a sphere. But as a first approximation, in deposited metals having a fine-grained polycrystalline structure, one may assume that there is an average Fermi surface which is close to a sphere. Thus the characteristics appearing in (1) and (2) are averaged quantities.

<sup>2)</sup>In formulas (1) and (2) unity is neglected compared to  $\epsilon'$ . For lead, in the range used for determining the microcharacteristics (2-12 $\mu$ ),  $1/\epsilon' = 8 \times 10^{-3} - 5 \times 10^{-4}$ , and this neglect is entirely justified.

It is easy to determine the value of  $N$  from the first relation. Figure 1 shows the dependence of  $\lambda^2(\kappa^2 - n^2)/(n^2 + \kappa^2)^2$  on  $\lambda$ . We see that over the interval from 2 to 12 $\mu$  this quantity is practically independent of  $\lambda$ . In the interval 0.7-1.5 $\mu$  a dependence on  $\lambda$  is observed indicating that one is close to the threshold for the internal photoeffect. Using the optical data for the range 2-12 $\mu$ , we get  $N = 4.0 \times 10^{22} \text{ cm}^{-3}$ .

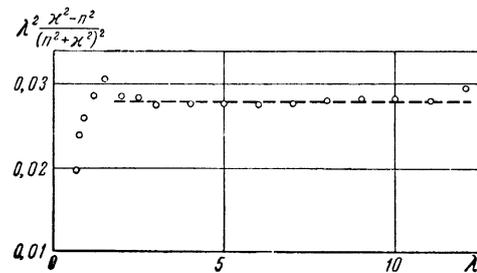


FIG. 1. Dependence of  $\lambda^2(\kappa^2 - n^2)/(n^2 + \kappa^2)^2$  on  $\lambda$  for  $T = 293^\circ\text{K}$ .

In formula (2),  $\nu_{\text{eff}} = \nu^{\text{ep}} + \nu^{\text{ee}} + \nu^{\text{i}} + \nu^{\text{e}\Sigma}$ , where  $\nu^{\text{ep}}$  is the electron-phonon collision frequency,  $\nu^{\text{ee}}$  is the electron-electron collision frequency,  $\nu^{\text{i}}$  is the frequency of collision of electrons with impurities. The last term takes into account the additional collisions of electrons with the surface of the metal produced by the light, which for the case of diffuse reflection of electrons from the surface gives a surface absorption

$$A_{\Sigma} = 0.75 \beta = 3.54 \cdot 10^{-5} \frac{\nu^{\text{e}\Sigma}}{\sqrt{N}}; \quad \beta = \frac{v}{c}.$$

Considering that for lead at room temperature  $T \gg \Theta$ , we get  $\nu^{\text{ep}} = \nu_{\text{cl}}^{\text{ep}}$ , where  $\nu_{\text{cl}}^{\text{ep}}$  is the classical frequency of collision of electrons with the lattice. Furthermore  $\nu^{\text{ee}} = \nu_{\text{cl}}^{\text{ee}} [1 + (\hbar\omega/2\pi kT)^2]$ , where  $\nu_{\text{cl}}^{\text{ee}}$  is the classical frequency for electron-electron collisions.<sup>[10]</sup> Thus the quantity  $2\lambda n\kappa/(n^2 + \kappa^2)^2$  has the form  $a + b/\lambda^2$ . Constructing its dependence on  $1/\lambda^2$ , we get a straight line,

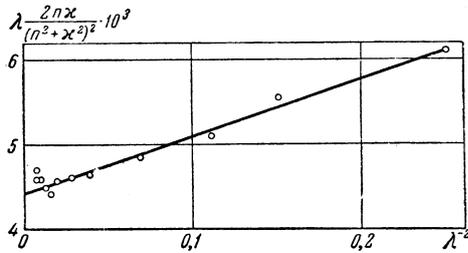


FIG. 2. Dependence of  $2\lambda n\kappa/(n^2 + \kappa^2)^2$  on  $\lambda^{-2}$  for  $T = 293^\circ\text{K}$ .

from whose slope we determine  $\nu_{\text{cl}}^{\text{ee}}$ , and whose intercept gives the sum  $\nu^{\text{ep}} + \nu_{\text{cl}}^{\text{ee}} + \nu^{\text{i}} + \nu^{\text{e}\Sigma}$ . The dependence of  $2\lambda n\kappa/(n^2 + \kappa^2)^2$  on  $1/\lambda^2$  for lead in the interval  $2-12\mu$  actually is linear. In the interval  $0.7-1.5\mu$  there is an additional absorption, increasing toward the short-wave end, which, as in the case of Fig. 1, indicates that one is close to the threshold for the internal photoeffect.

Figure 2 shows this dependence for the interval  $2-12\mu$ . From the slope of the line,  $\nu^{\text{ee}} = 0.08 \times 10^{14} \text{ sec}^{-1}$ . The conductivity measurements give  $\nu^{\text{ep}} + \nu_{\text{cl}}^{\text{ee}} + \nu^{\text{i}} = 2.53 \times 10^8 \text{ N}/\sigma_{293} = 2.47 \times 10^{14} \text{ sec}^{-1}$ . From the temperature variation of the resistance, we find  $\nu^{\text{i}}/(\nu^{\text{ep}} + \nu_{\text{cl}}^{\text{ee}} + \nu^{\text{i}}) = 0.93 \times 10^{-2}$ . We then get  $\nu^{\text{ep}} = 2.37 \times 10^{14} \text{ sec}^{-1}$ ,  $\nu^{\text{i}} = 0.023 \times 10^{14} \text{ sec}^{-1}$ . The intercept on the ordinate axis gives  $\nu^{\text{ep}} + \nu_{\text{cl}}^{\text{ee}} + \nu^{\text{i}} + \nu^{\text{e}\Sigma} = 2.97 \times 10^{14} \text{ sec}^{-1}$ , from which  $\nu^{\text{e}\Sigma} = 0.5 \times 10^{14} \text{ sec}^{-1}$  and  $v = 3.5 \times 10^8 \text{ cm/sec}$ .

From the data one can easily determine the mean free path of the electrons,  $l$ . For  $\lambda = 5\mu$ ,  $l = 1.1 \times 10^{-6} \text{ cm}$ . The skin depth for this same is  $\delta = 3.2 \times 10^{-6} \text{ cm}$ . We see that at room temperature  $l$  is a factor of three less than  $\delta$ .<sup>3)</sup>

2. Analysis of experimental data for liquid nitrogen temperature. The value of  $N$  was determined from the expression

$$\lambda \frac{4\kappa}{n^2 + \kappa^2} = \frac{1.34 \cdot 10^{11}}{\sqrt{N}} (1 + p^2 G), \quad (3)$$

where the notation is the same as in [6],  $p^2 G$  is a correction term which is small at liquid nitrogen temperature and was not included. Figure 3 shows the dependence of  $4\lambda\kappa/(n^2 + \kappa^2)$  on  $\lambda$ . We see that at short wave lengths there is a deviation from the

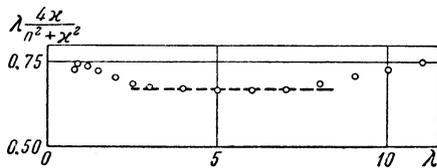


FIG. 3. Dependence of  $4\lambda\kappa/(n^2 + \kappa^2)$  on  $\lambda$  for  $T = 77.4^\circ\text{K}$ .

<sup>3)</sup>The values of  $l$  and  $\delta$  depend slightly on wavelength  $\lambda$ .

horizontal line which is also apparently due to the nearness of the threshold for the internal photoeffect. We have no explanation for the deviation from the horizontal at the long wave length end. Using the values of the optical constants in the interval  $2.5-8\mu$ , we get  $N = 3.95 \times 10^{22} \text{ cm}^{-3}$ .

To determine the other parameters we used the expression

$$\frac{4n}{n^2 + \kappa^2} = 0.75\beta + \frac{3.54 \cdot 10^{-5}}{\sqrt{N}} (\nu_{\text{cl}}^{\text{ep}}\varphi + \nu_{\text{cl}}^{\text{ee}} + \nu^{\text{i}}) + \frac{1.83 \cdot 10^2 \nu_{\text{cl}}^{\text{ee}}}{\sqrt{N} T^2 \lambda^2} - p^2 \beta D, \quad (4)$$

where  $\varphi$  is the quantity computed by Gurzhi.<sup>[11]</sup> For our case,  $\varphi = 1.15$ . The last term is a correction; it is small at low temperatures and was not considered by us. The dependence of  $4n/(n^2 + \kappa^2)$  on  $1/\lambda^2$  is linear in the interval  $2.5-11\mu$ . As was the case at room temperature, the short wave length part shows an additional absorption. In Fig. 4 we give the dependence of  $4n/(n^2 + \kappa^2)$  on  $1/\lambda^2$  in the range  $2.5-11\mu$ . Analysis of the optical results in this interval, supplemented by conductivity data, gives

$$\begin{aligned} v &= 2.3 \cdot 10^8 \text{ cm/sec} \\ \nu_{\text{cl}}^{\text{ep}} &= 5.7 \cdot 10^{13} \text{ sec}^{-1} \\ \nu_{\text{cl}}^{\text{ee}} &= 6 \cdot 10^{10} \text{ sec}^{-1} \\ \nu^{\text{i}} &= 0.23 \cdot 10^{13} \text{ sec}^{-1} \end{aligned}$$

After determining all the microcharacteristics, one can verify that the parameters in which the expansion was made are less than unity: for  $\lambda = 5\mu$ ,  $\nu_{\text{eff}}/\omega = 0.18$ ,  $v/\omega\delta = 0.22$ .

Let us determine the values of  $l$  and  $\delta$ . For  $\lambda = 5\mu$ ,  $l = 3.3 \times 10^{-6} \text{ cm}$ ,  $\delta = 2.7 \times 10^{-6} \text{ cm}$ . Using the values of  $\nu^{\text{i}}$  and  $v$ , we can estimate the size of the crystallites in the layers used:  $L \approx v/\nu^{\text{i}} = 1.0\mu$ . We see that the crystallite size is of the same order as the thickness of the layers.

A summary of the results is given in Table II.

From the table it follows that the concentrations of conduction electrons are the same at the two temperatures. This indicates that  $N$  is reliably determined by the optical method. The values of

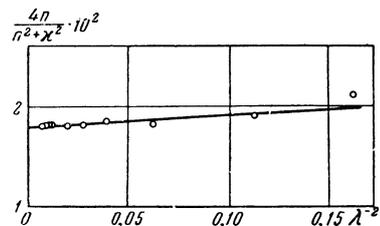


FIG. 4. Dependence of  $4n/(n^2 + \kappa^2)$  on  $1/\lambda^2$  for  $T = 77.4^\circ\text{K}$ .

**Table II.** Microcharacteristics of lead from measurements of optical constants

	293° K	77,4° K
$N$	$4.0 \cdot 10^{22} \text{ cm}^{-3}$	$3.9_5 \cdot 10^{22} \text{ cm}^{-3}$
$N/N_a$ *	1,2	1,2
$v$	$3.5 \cdot 10^8 \text{ cm/sec}$	$2.3 \cdot 10^8 \text{ cm/sec}$
$v_{cl}^{ep}$	$23.7 \cdot 10^{13} \text{ sec}^{-1}$	$5.7 \cdot 10^{13} \text{ sec}^{-1}$
$v_{cl}^{ee}$	$0.8 \cdot 10^{13} \text{ sec}^{-1}$	$0.006 \cdot 10^{13} \text{ sec}^{-1}$
$v_i$	$0.23 \cdot 10^{13} \text{ sec}^{-1}$	$0.23 \cdot 10^{13} \text{ sec}^{-1}$

\* $N_a$  is the concentration of atoms.

the electron velocity  $v$  at the Fermi surface differ by a factor of approximately 1.5. Such a large difference indicates the poor accuracy in the determination of  $v$  from optical data. This is connected with the smallness of the surface loss compared to the bulk losses. The values found at the low temperature should be more reliable.

According to the theory, the frequency of electron-electron collisions should vary  $\sim T^2$ . In our case the variation was much stronger ( $\sim T^3$ ).

The results can be compared with data on the electronic specific heat  $C_e = \gamma T$ . At present there are no measurements of electronic specific heat for the samples used in the optical measurements. We must therefore make the comparison using results for the metal prepared differently. According to Eisenstein,<sup>[12]</sup>  $\gamma = 1.59 \times 10^3 - 1.70 \times 10^3 \text{ erg/cm}^3 \text{ deg}^2$ , from which  $v/\sqrt{N} = 0.36 \times 10^{-3} - 0.35 \times 10^{-3} \text{ cm}^{5/2} \text{ sec}^{-1}$ . The optical measurements give  $v/\sqrt{N} = 1.2 \times 10^{-3} \text{ cm}^{5/2}/\text{sec}$ . The results disagree by a factor of three.

The high values of  $v$  found from the optical data may be connected with the incomplete inclusion of the interaction of the electrons with high frequency oscillations of the lattice in the optical measurements. In the work of Gurzhi<sup>[9,10]</sup> which we used, the dispersion law is taken to be a linear dependence of phonon frequency on momentum, which holds only for low frequency vibrations. One should include this effect.

The assumption made that the Fermi surface is spherical for metals of group IV is only a very

crude first approximation. The problem is not so much that this surface is anisotropic but rather that it is multiply connected. The Fermi surface for lead is located in the second, third, and fourth zones.<sup>[13]</sup> The electrons which are in the fourth zone constitute a small fraction and cannot have any large effect on the optical constants. We suggest that a more detailed analysis be made of the influence of multiple connectivity on the optical constants. The metals of the fourth group are of particular interest for explaining this point, and we suggest that they be studied.

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Translated by M. Hamermesh