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Variations in Optical Reflectivity of Sputter Deposited Metal and Semimetal Films

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FOREWORD

The research described in this effort was performed and funded by the Engineering Department during fiscal year 1975. It is part of a continuing effort to develop techniques and applications of thin-film coatings for infrared weapons systems and/or infrared missile detector systems.

This report has been reviewed for technical accuracy by G. H. Turner.

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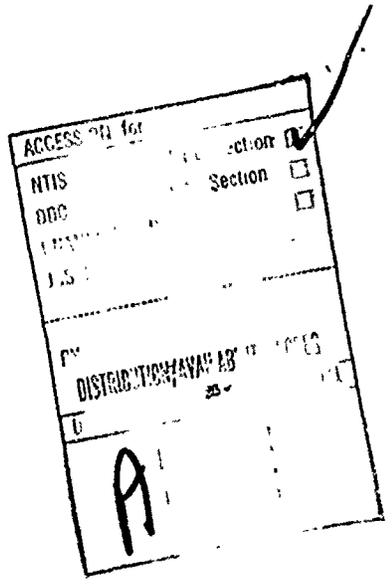
(U) The surface structure of vacuum sputter deposited metal and semimetal films can be varied by changing the substrate bias potential and the total time of deposition. The corresponding change in the optical reflectivity can be related to enhanced scattering and absorption effects. Reflectivity measurements over the infrared range from 1 to 7 microns are presented and discussed for titanium, antimony, bismuth, and copper films. Results for the metal films, copper and titanium, are in agreement with those obtained by Bennett for aluminum films. The results for antimony and bismuth indicate the influence of enhanced absorption.



INTRODUCTION

The surface structure of vacuum sputter deposited metal and semimetal films can be varied by changing either the substrate bias or the total time of the deposition. Reflectivity data for titanium, bismuth, antimony, and copper films are examined and features of these curves related to surface scattering and possible enhanced absorption effects.

DEPOSITION TECHNIQUE

Films of titanium, bismuth, antimony, and copper were bias sputtered in a Varian Model 980-2404 chamber mounted on a standard Inovac pumping station. Initially films were deposited using a combination of various bias settings and deposition times. The objective was to determine which particular set of parameters produced films that scattered or absorbed very strongly in the visible spectrum. Once this was determined, a set of samples was prepared for reflectivity measurements over the range of 1 to 7 microns. The substrates were polished quartz. The smooth films were prepared by sputtering a continuous film about 500 to 800 angstroms thick. Argon was used as the sputtering gas. Figure 1 is a schematic of the arrangement used to obtain the films. Scanning electron micrographs were taken of the samples. Figures 2 through 6 illustrate the structures obtained for bismuth films.

The bismuth film shown in Figures 2 and 3 was sputter deposited on polished quartz at zero potential bias and with an argon pressure of 3×10^{-2} torr. The average size of the smaller protuberances is 1.2 microns and the larger ones average 15 microns in width. The appearance of small particles surrounded by rings of larger particles suggests that a diffusion process may be taking place in which the larger particles grow at the expense of the smaller particles. The film shown in Figures 4 and 5 verifies this growth characteristic. The larger protuberances average 127 microns in diameter. Preparation of this film differed from that shown previously only in length of deposition time. The deposition time in this case was doubled.

The influence of a crystalline substrate on this growth characteristic is illustrated in Figure 6. Here the deposition was similar to that for the bismuth film shown in Figure 2 except that the substrate is single-crystal sapphire. The effect of the substrate structure is to promote crystalline growth of the bismuth within the individual protuberances. The average width of these projections is 12 microns, very nearly the same as the average width of the larger particles grown on the quartz substrate.

REFLECTIVITY DATA

Reflectivity data for aluminum films deposited onto intentionally roughened substrates obtained by Bennett is shown in Figure 7.[1] This data indicates that for rougher films the point at which reflectivity becomes apparent shifts to longer wavelengths and a reduction in reflectivity occurs. These characteristics are attributed to scattering by the surface protuberances. A shift to longer wavelengths and reduction in reflectivity is also seen in the data obtained (by the authors) for bias sputtered bismuth and copper shown in Figures 8 and 9 and Tables 1 and 2. Reflectivity of titanium and antimony also display these characteristics (Figures 10 and 11 and Tables 3 and 4). The data indicates that a rather abrupt change in the relative reflectance occurs between 3.0 and 4.0 microns for the titanium and between 3.5 and 4.5 for antimony (Figures 12 and 13). Note that for bismuth the relative reflectance curve is a smooth function (Figure 14).

TABLE 1. Relative Reflectance of Smooth and Rough Sputtered Bismuth.

λ	R_{smooth}	R_{rough}
1.0	0.22482	0.00000
1.5	.39548	.01633
2.0	.47071	.01740
2.5	.49830	.04192
3.0	.51854	.07244
3.5	.53485	.12104
4.0	.54538	.17219
4.5	.55352	.21533
4.7	.55688	.22590
4.75	.55695	.22972
4.8	.55712	.23319
5.0	.56028	.24480
5.5	.56623	.27171
6.0	.57207	.29517
6.5	.57314	.31396
7.0	0.57429	0.32568

TABLE 2. Relative Reflectance of Rough Copper.

λ	R_{rough}
1.0	0.00000
1.5	.00000
2.0	.00000
2.5	.02514
3.0	.06108
3.5	.17740
4.0	.29728
4.5	.37421
4.7	.39604
4.75	.40516
4.8	.40854
5.0	.42441
5.5	.45789
6.0	.46618
6.5	.47504
7.0	0.48564

RESONANT DIPOLE ABSORPTION

This additional change in reflectivity of titanium and antimony films may possibly be associated with enhanced absorption due to a resonant dipole mechanism (surface plasmon effect). The resonant dipole model as described by Decker considers polarizable oblate spheroids elevated above a reflecting plane.[2] These spheroids are an approximation to the actual protuberances observed on a real film.

[1] H. E. Bennett, "Specular Reflectance of Aluminized Ground Glass and the Height Distribution of Surface Irregularities," *J. Am. Opt. Soc. 53* (1963), p. 1389.

[2] D. Decker, "High Energy Laser Mirrors and Windows," Advanced Research Project Agency Order 2175, Semiannual Report No. 4, Sept. 1973 to March 1974.

TABLE 3. Relative Reflectance of Smooth and Rough Sputtered Titanium.

λ	R_{smooth}	R_{rough}
1.0	0.45115	0.11687
1.5	.49670	.18930
2.0	.53000	.26342
2.5	.56545	.31464
3.0	.63006	.40325
3.5	.69428	.47608
4.0	.74109	.52141
4.5	.77979	.58181
4.75	.79424	.61080
5.0	.80524	.63948
5.5	.82832	.68451
6.0	.84689	.72857
6.5	.85709	.75627
7.0	0.86824	0.78383

TABLE 4. Relative Reflectance of Smooth and Rough Sputtered Antimony.

λ	R_{smooth}	R_{rough}
1.0	0.59409	0.00000
1.5	.60812	.03414
2.0	.61856	.10993
2.5	.62318	.19481
3.0	.62906	.26579
3.5	.63405	.31912
4.0	.63949	.34696
4.5	.64681	.37690
4.7	.64900	.38531
4.75	.65007	.38684
4.8	.65113	.38817
5.0	.65492	.39520
5.5	.66674	.40629
6.0	.67797	.41814
6.5	.68815	.43001
7.0	0.69830	0.44249

They have an aspect ratio given by the ratio of the semiminor axis to the semimajor axis, a/b, shown in Figure 15. The analysis requires two physical conditions: (1) the polarizable oblate spheroid must have a large aspect ratio, and (2) the minor axis must lie in the direction of propagation of the incident light. These two factors are readily obtained in the rough sputtered films as seen by examination of the scanning electron micrographs.

The analysis proceeds by finding the amplitude of the incident and reflected intensities at the top of the oblate spheroid, see Figure 12 and Appendix A. The expression for the fractional decrease in reflectivity, Figure 16, is obtained after additional conditions are imposed and is given by

$$\frac{\Delta R}{R} = \frac{R_{\text{incident}} - R_{\text{reflected}}}{R_{\text{incident}}}$$

or

$$\frac{\Delta R}{R} = \frac{-32kap}{3} \quad \frac{\epsilon_2 (L - 1)}{\left\{ [L(\epsilon_1 - 1) + 1]^2 + L^2 \epsilon_2{}^2 \right\}}$$

From the analysis in the appendix the condition which maximizes $\Delta R/R$ is

$$L(\epsilon_1 - 1) + 1 = 0$$

Imposing the condition that $| \epsilon_1 | >> 1$ leads to $\epsilon_1 = -1/L$, where L is the depolarization factor for the oblate spheroid and may be regarded as the slope, a/b. The appearance of a maximum is consistent with the spectral dependence of ϵ_1 and ϵ_2 for a free-electron metal, that is ϵ_1 is

negative for wavelengths greater than the bulk resonance wavelength and $|\epsilon_1| >> \epsilon_2$ in this region. These conditions generally prevail in the region where $\lambda > 1$ micron. For silver the surface plasmon effect has been reported to occur at ultraviolet wavelengths (Figure 17).[3] The shift to longer wavelengths for titanium and antimony may be associated with greater effective masses for these materials since

$$\epsilon_1 = 1 - \frac{4\pi Ne^2}{m^*} \quad \frac{1}{\left(\omega^2 + \frac{1}{\tau^2}\right)}$$

or

$$\epsilon_1 = 1 - \frac{\omega_p{}^2}{\left(\omega^2 + \frac{1}{\tau^2}\right)}$$

for a free-electron metal, a larger effective mass will shift the ϵ_1 versus ω curve to lower values of ω, hence to longer wavelengths. The condition of maximum reflectance for the resonant dipole model,

$$\epsilon_1 = \frac{-1}{L}$$

then occurs at longer wavelengths.

The magnitude of the $\Delta R/R$ for the sputtered titanium and antimony films is expected to be greater than the values determined by Decker for aluminum films, Figure 18. An argument for greater magnitude can be made on the basis of a broad resonant dipole. Following Decker's analysis the depolarization factor L is associated with the slope of the oblate spheroid. Consideration of the effect of a distribution in slope, $D(L)$, leads to an expression for the average value of the change in reflectivity

$$\left\langle \frac{\Delta R}{R} \right\rangle = \int_0^\infty D(L) \frac{(\Delta R)}{R}\, dL.$$

From this one can say that when $D(L)$ becomes large due to a large number of particles having slopes which contribute to the integration, then $\langle \Delta R/R \rangle$ can be expected to be large. For rough sputtered films the number of particles having a distribution of slopes is considerable, as evidenced by the scanning electron micrographs for bismuth. For relatively smooth films the distribution in slopes is small and the number of particles contributing to the integration is small, so that $\langle \Delta R/R \rangle$ is relatively unenhanced.

[3] T. Huen, G. Irani, F. Wooten. "Scanning Ultrahigh Vacuum Reflectometer," *Appl. Opt. 10* (1971), p. 552.

The computation of the change in reflectivity from basic materials properties for rough films remains very intractable; however, characteristics associated with a broad resonant dipole model indicate the possibility of enhanced absorption in rough sputtered films.

CONCLUSION

The decrease in reflectivity of bias sputtered titanium, bismuth, and antimony films shows a behavior that is explainable by scattering effects. In addition, an enhanced absorption in antimony and titanium in the intermediate infrared is possibly due to a broad resonant dipole mechanism.

Bias-sputtered films with rough surface texture appear to provide a means for studying absorption by surface plasmons since the effect may be enhanced in these films.

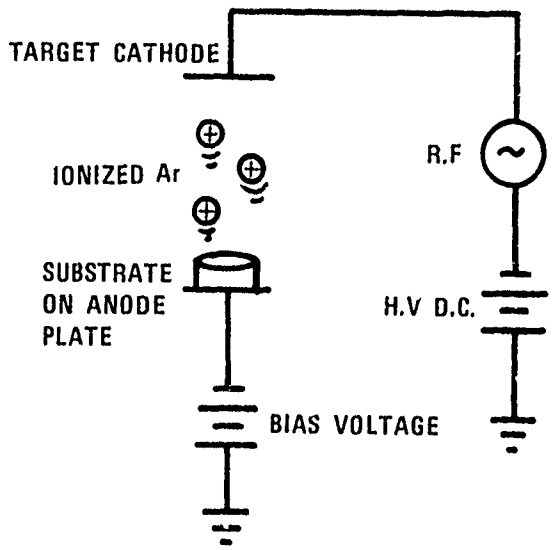


FIGURE 1. Schematic of Sputter Bias System.

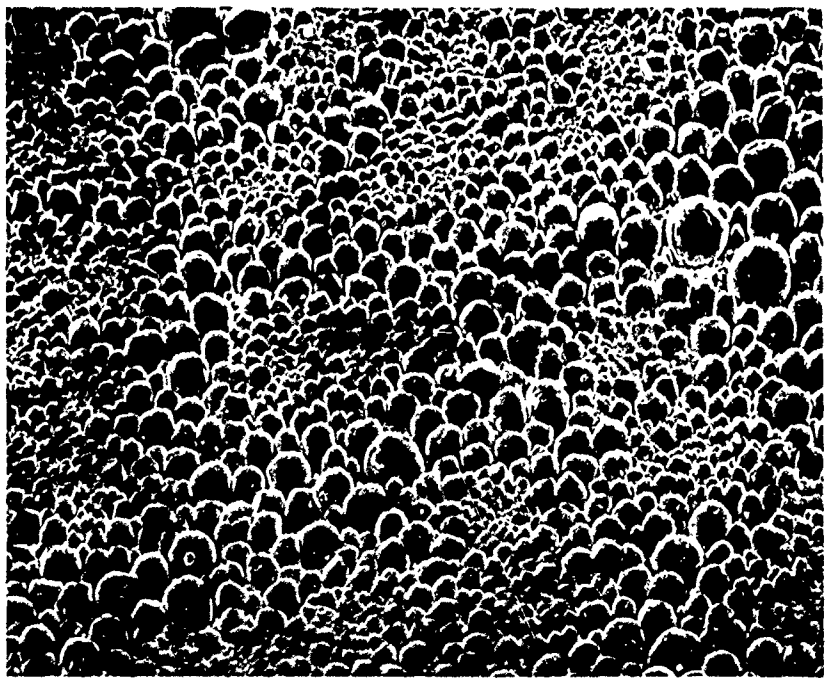


FIGURE 2. SEM Micrograph of Sputtered Bismuth Film, 400X. Bias voltage was zero and pressure 3×10^{-2} torr of argon. Average size of particle is 15 microns.

FIGURE 3. SEM Micrograph of Sputtered Bismuth, Same Film as Figure 2, but at 2100X.

FIGURE 4. SEM Micrograph of Sputtered Bismuth Film 100X. Bias voltage was zero and pressure 3×10^{-2} torr of argon. Average diameter of larger particles is 127 microns.

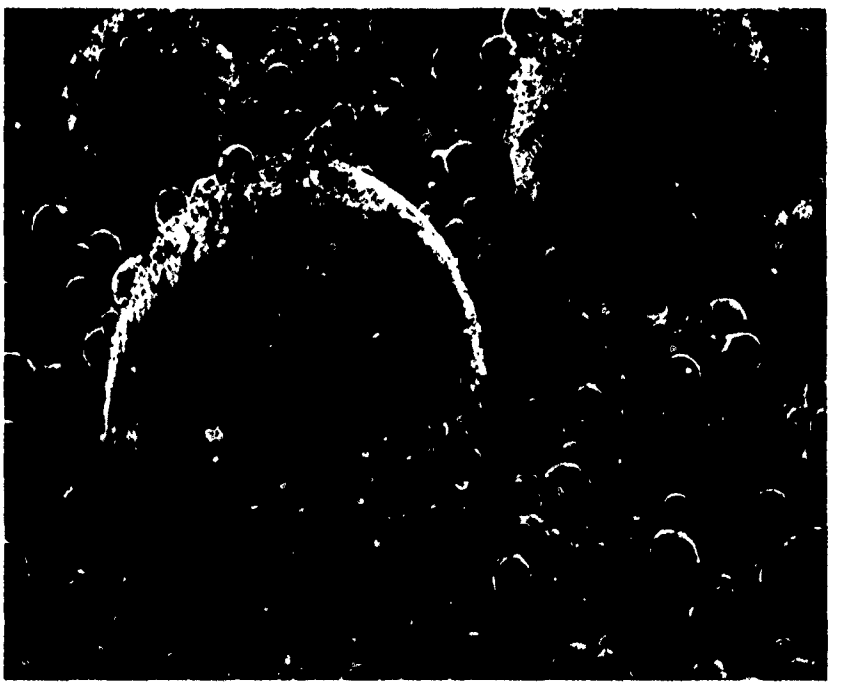


FIGURE 5. SEM Micrograph of Film Shown in Figure 4, but at 400X.

FIGURE 6. SEM Micrograph of Sputtered Bismuth on Sapphire Substrate, 400X. Average width of columnar protuberances is 12.5 microns.

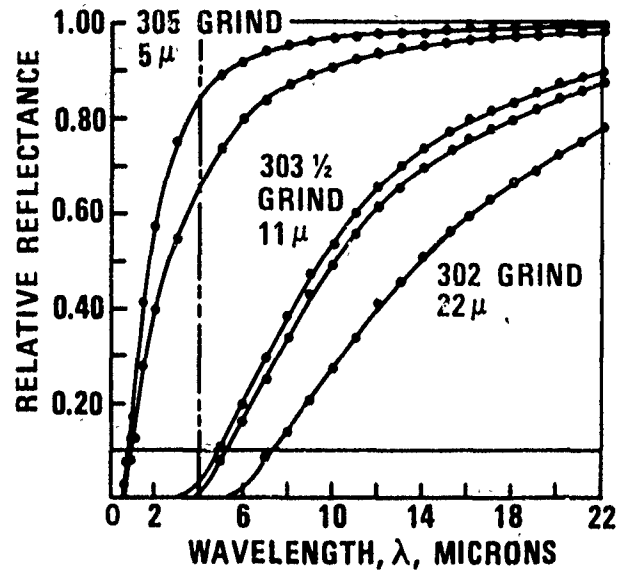


FIGURE 7. Effect of Intentially Roughened Surface on Reflectivity of Aluminum.

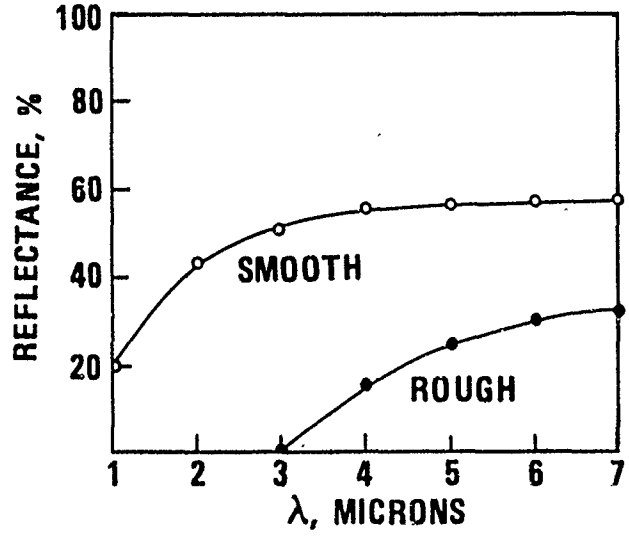


FIGURE 8. Reflectivity of Sputtered Bismuth.

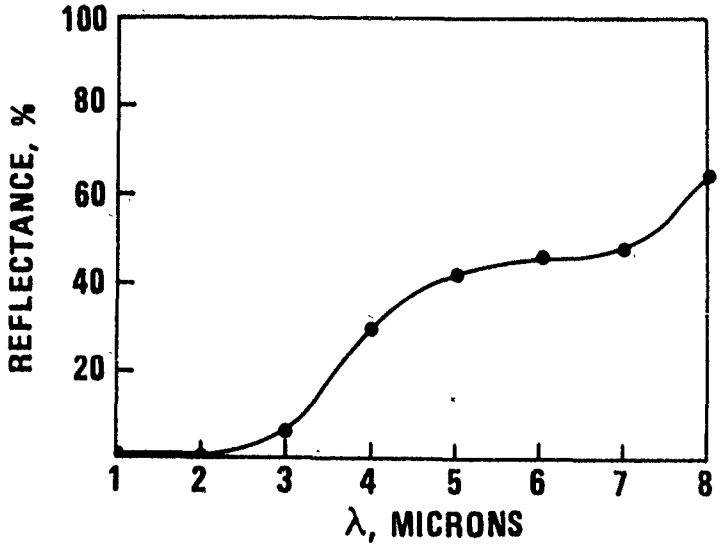


FIGURE 9. Reflectivity of Sputtered Copper.

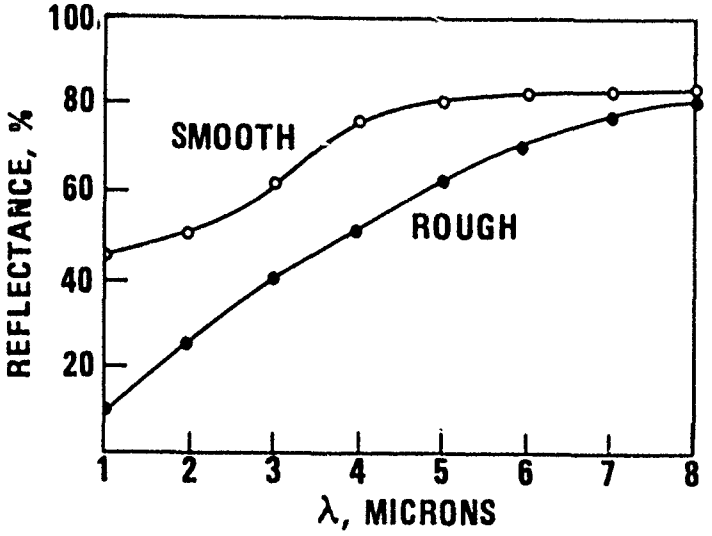


FIGURE \0. Reflectivity of Sputtered Titanium.

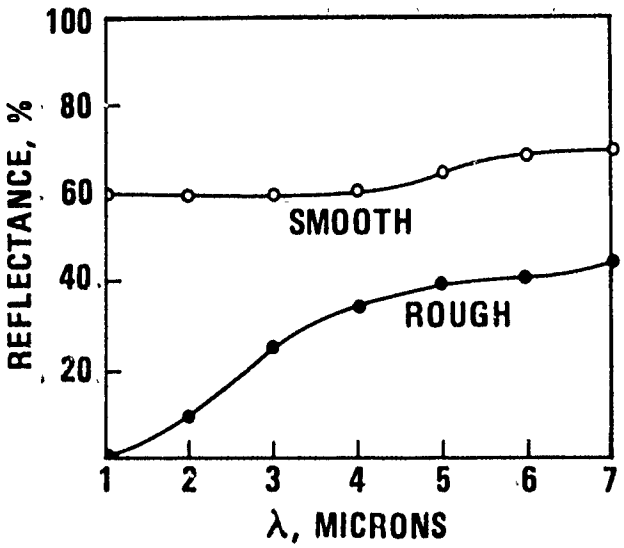


FIGURE 11. Reflectivity of Sputtered Antimony.

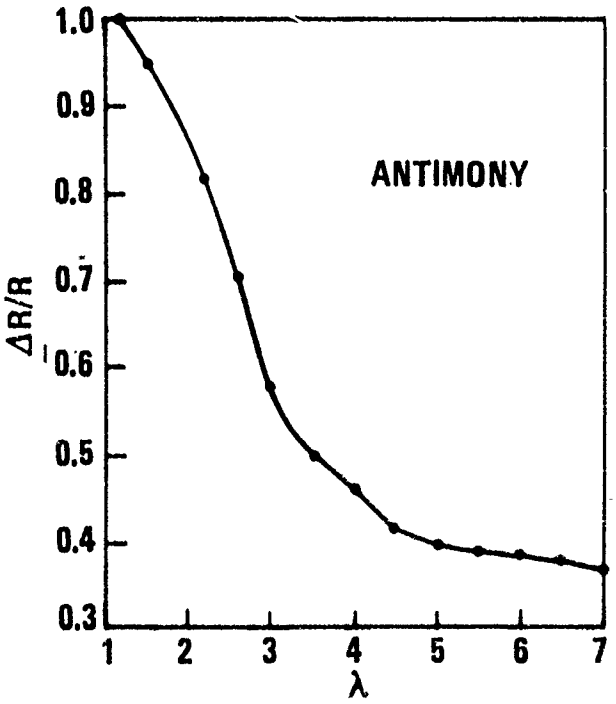


FIGURE 12. Relative Reflectance of Antimony.

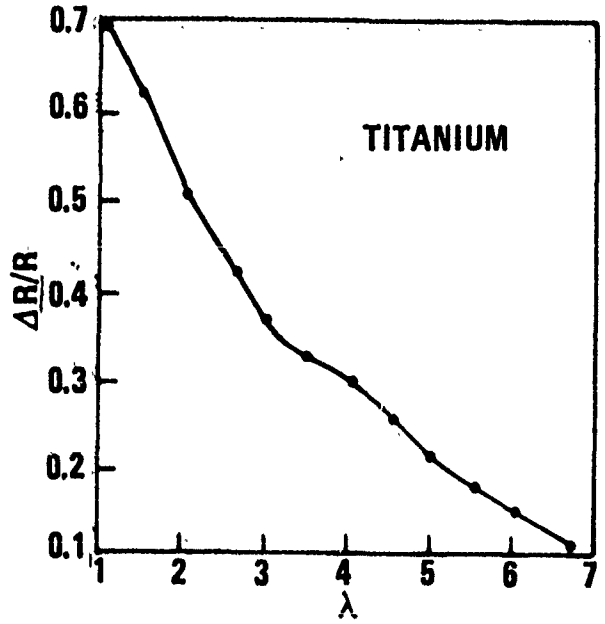


FIGURE 13. Relative Reflectance of Titanium.

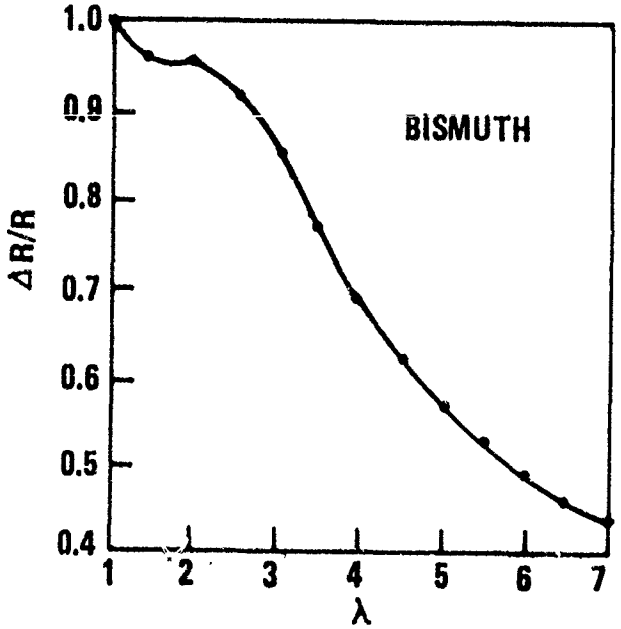


FIGURE 14. Relative Reflectance of Bismuth.

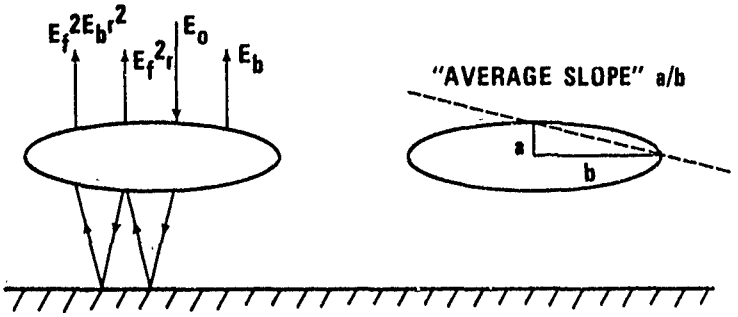


FIGURE 15. Oblate Spheroid of Resonant Dipole Absorption Model, After Decker.

$$E_f = E_0 \left[1 - \left(\frac{2\pi}{k^2}\right) n\, S_{(0)}\right]$$

$$E_b = -E_0 \left(\frac{2\pi}{k^2}\right) n\, S_{(180)}$$

$$\frac{\Delta R}{R} = \frac{|r_0|^2 - |r|^2}{|r_0|^2} \qquad \text{(RELATIVE CHANGE IN REFLECTIVITY)}$$

$$\frac{\Delta R}{R} = -32\,\frac{kap}{3}\,\frac{\epsilon_2\,(L \perp 1)}{\left\{[L(\epsilon_1 - 1) + 1]^2 + L^2\,\epsilon_2^2\right\}}$$

WHERE

$$L \cong \frac{\pi}{4}\,\frac{a}{b} \qquad \left\{\begin{array}{l}\text{DEPOLARIZATION FACTOR FOR}\\ \text{OBLATE SPHEROID I.E. SLOPE OF}\\ \text{POLARIZABLE ELEMENT OF}\\ \text{SURFACE}\end{array}\right\}$$

$$p = \frac{\pi b^2 n}{2}$$

$$\frac{\langle \Delta R \rangle}{R} = \int_0^\infty D_{(L)} \left(\frac{\Delta R}{R}\right) dL$$

FIGURE 16. Expressions Derived in Decker.

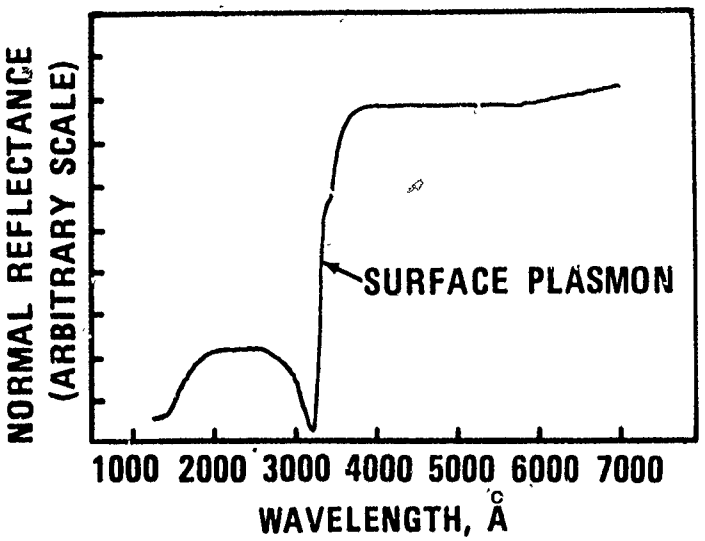


FIGURE 17. Surface Plasmon Effect in Silver.

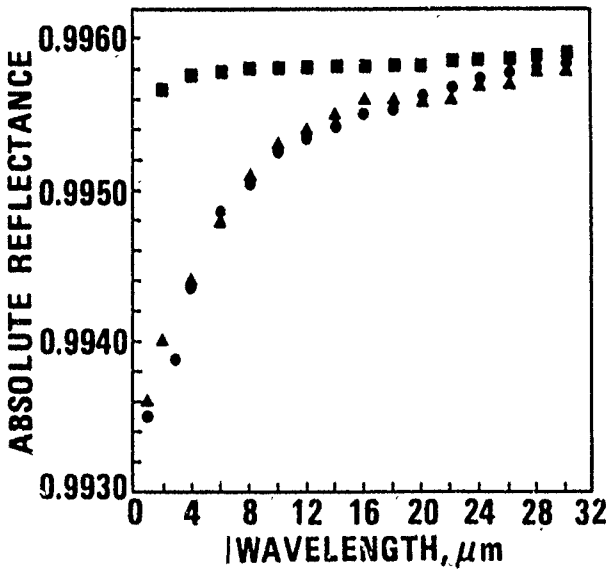


FIGURE 18. Reflectance for ■ Drude Model of Free-Electron Metal, ▲ Experimental Data Taken on Silver at NWC, and ● Drude Model With Addition of Resonant Dipole Absorption, From Decker.

Appendix A
OUTLINE OF THE BROAD RESONANT DIPOLE MODEL

From Figure 15, which shows the incident and reflected electric field vectors for a single polarizable oblate spheroid, the electric fields due to a collection of n particles are given by

$$E_f = E_o \left[1 - \left(\frac{2\pi}{k_2} \right) n S_{(o)} \right]$$

for the forward direction and

$$E_b = -E_o \left(\frac{2\pi}{k_2} \right) n S_{(180)}$$

for the backward direction, where k is the wave vector, $2\pi/\lambda$, and S is the scattering amplitude factor.

Next the amplitude reflectance is computed and, imposing the condition that $S_{(o)} = S_{(180)}$ when a is much less than the exciting wavelength, is reduced to

$$r = r_o \left\{ 1 - \frac{2\pi}{k_2} n S_{(o)} \frac{(1 + r_o)^2}{r_o} \right\}$$

The fractional decrease in reflectance due to resonant absorption is then expressed by

$$\frac{\Delta R}{R} = \frac{|r_o|^2 - |r|^2}{|r_o^2|}$$

Using the condition that the depolarization factor L_1 for an oblate spheroid of large aspect ratio with the minor axis in the direction of the propagating electric field can be expressed as,

$$L = \frac{\pi}{4} \frac{a}{b}$$

and letting

$$p = \frac{\pi b^2 n}{2}$$

then after an appropriate amount of algebraic expansion and manipulation, keeping only terms to first order in ka, the fractional decrease in reflectance is expressed by,

$$\frac{\Delta R}{R} = \frac{-32kap}{3} \frac{\epsilon_2(L - 1)}{\left\{\left[L(\epsilon_1 - 1) + 1\right]^2 + L^2 \epsilon_2^2\right\}}$$

Now $\Delta R/R$ becomes maximum when

$$[L(\epsilon_1 - 1) + 1]^2 = 0$$

Let

$$\left| \epsilon_1 \right| \gg 1 \quad \text{then} \quad G_1 - 1 \cong G_1 \text{ and } L\epsilon_1 + 1 = 0$$

Now the condition for maximum $\Delta R/R$ is $L = -1/\epsilon_1$. Note that ϵ_1 is a negative quantity for free-electron metals in the region of interest; hence L is a positive quantity, as it should be.

For a metal, the complex dielectric constant is given by $\tilde{\epsilon} = \epsilon_1 + i\epsilon_2$

where

$$\epsilon_1 = 1 - \frac{\omega_p{}^2\tau}{(1 + \omega^2\tau^2)}$$

$$\epsilon_2 = \frac{\omega_p{}^2\tau^2}{\omega(1 + \omega^2\tau^2)}$$

$\tau \equiv$ mean free time between collisions of free electrons in the metal

$$\omega_p{}^2 = \frac{4\pi Ne^2}{m^*} \quad \text{and}$$

$m^* \equiv$ effective mass of the electrons in the metal

From these relationships it can be shown that as m^* is increased the value of ω at which $L = -1/\epsilon_1$ decreases, and hence resonance occurs maximizing $\Delta R/R$ at longer wavelengths.

The total absorption due to all elements acting in resonance is expressed by considering the average fractional change in reflectance, $<\Delta R/R>$. When there is a distribution of slopes among these particles, this can be accounted for by considering a slope distribution function, $D_{(L)}$.

Then

$$< \frac{\Delta R}{R} > = \int_0^\infty D_{(L)} \left(\frac{\Delta R}{R}\right) dL$$

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