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ABSTRACT

Results of an extended investigation of surface plasma-wave absorption and roughness-induced optical scattering from an interface of silver and air are described. In particular, the position of the surface plasma resonance minimum in reflectivity for a stochastically roughened metal silver surface has been studied as a function of a number of distinct roughness perturbations. In the case of a transparent liquid-silver boundary the frequency red shift of the resonance minimum has been determined and the location of the surface plasmon dip for various liquids is shown to agree well with a simple roughness theory. The additional interfacial properties due to the formation of a thin inhomogeneous oxide layer occurring either spontaneously or due to application of a small interfacial electrical potential are more complex. However, the optical constants of the interlayer have been determined from differential specular reflectance measurements at the boundary. Nodule size parameters determined from scattering and absorption measurements and features of both polarized and depolarized diffuse reflectance give further information on the state of the interface. Reflectance of ordered-corrugated surfaces are also described.

I. INTRODUCTION

This work is concerned with optical and structural properties of roughened silver foils. In some cases these have been covered by very thin metal-oxide overlayer residues. Rough silver surfaces which we have investigated are formed by evaporation of thin calcium fluoride layers on glass (0.1 to 0.3 μm) with subsequent evaporation of optically opaque silver overlayers. Inhomogeneous metal-oxide surface films which are formed on metals are corrosion products which can be produced in the liquid environment or also in vacuum plasma discharges. The study of such interfaces is fruitful, since a number of electronic processes depend on detailed information on the structure of this and related material boundaries.

In this study most of our measurements have utilized difference reflectance¹⁾ ($\Delta R/R \geq 10^{-3}$) and laser light scattering to measure both the surface absorption and scattering rates due either to metal roughness or due to adlayer corrections to a smooth metal surface. Concurrent scanning electron microscope analysis has served as an independent verification of these optical results, and generally the two methods give confirming surface topography information. In the experiments carried out to this time, difference reflectance measurements have been obtained in both liquid and air. In the liquid, a ring electrode arrangement¹⁾ has been helpful for forming thin oxide residues as well as in cyclic deposition (with varying interfacial voltage) of the thin oxide layers examined.^{2,3)}

These studies are essential and form a useful part of the investigation of both elastic and inelastic processes at boundaries. The somewhat limited understanding of these effects is evident in some enhanced

Raman scattering studies in the case of silver covered with certain thin-layer adsorbates.⁴⁾ A variety of different models^{5,6,7)} have recently been advanced to account for this behavior. The relative merits of these pictures will depend on agreement not only to Raman experiments on rough surfaces, but also to details of ordinary roughness-induced reflectance. Unfortunately ordinary differential reflectance studies may not directly influence the inelastic Raman scattering work, since it already seems well established that any possible anomalies in the differential reflectance are very much smaller⁸⁾ in magnitude than the observed Raman enhancements.⁹⁾ Nonetheless, the dependence of both the elastic and inelastic scattering on the interfacial roughness suggests that collective excitations of the metallic electron gas and details of such processes, as well as image dipole fields induced by adsorbed components, may be dominant mechanisms to explain the observed phenomena.

In conjunction with reflectance and scattering measurements on metals, the nature of adsorbed electrochemical oxides have been studied using difference reflectance. Oxides of silver are semiconductors and form interfacially as small surface nodules (of size comparable to the optical wavelength) at the boundary between silver metal and an electrolyte such as aqueous potassium hydroxide. We should note here that the structure of oxide residues of different metals are not the same. Thin oxide overlayers on aluminum, for example, tend to be much more uniform than similar layers on silver. In the present study, however, it has been possible to examine only what are thought to be mixed silver-oxide surface phases. Even though different electrochemical thresholds are believed to lead to different oxidation states, the optical properties determined¹⁰⁾ are similar enough so that distinct separation of the

properties of the various possible oxides has not been feasible. Yet such information could, of course, be helpful for a number of further investigations.

II. BACKGROUND AND EXPERIMENTAL RESULTS IN STUDY OF REFLECTANCE AND SCATTERING FROM SILVER AND SILVER OXIDE LAYERS

As stated in our introductory remarks, our present work in the area of reflectance and scattering spectroscopy of metal films has included investigation of residual silver oxide on silver^{2,3)} as well as roughness scattering from bare silver foils.^{11,12)} The inhomogeneous surface oxide clusters formed in the former instance have been of order tens to several hundreds of angstroms in linear size depending on the degree of the oxidation. The roughness structure on bare silver has been produced by evaporation of the metal onto calcium fluoride and also onto random dispersions of latex spheres on glass. It is also observed that the metal may be spontaneously roughened in certain cases by simply reducing the rate at which the metal is evaporated onto smooth substrates. In these various metal cases, the corrugation heights obtained have been of order 50 to 100 Å and the correlation widths of roughness have been determined to be of order 800 to 1500 Å. Examples of roughened surfaces examined in our present study are shown in Fig. 1.

The reflectance method used to study metal foils utilizes a difference technique allowing normalized reflectance differences $\Delta R/R$ of as little as 10^{-4} to be measured.¹⁾ In the case of metal oxidation the difference reflectance between a bare metal silver foil and a matched lightly oxidized foil can be used to obtain the optical constants^{3,10)} of the chemically deposited overlayer. In the case of metal reflectance, we have been more interested in determining reflectance values from measurement of differential quantities. Nonetheless, the same experimental configuration as for oxide layer studies may be used. In this case, we have found it convenient to compare the reflectance of silver to

FIGURE 1

readily-reproducible aluminum references. Assuming the aluminum optical constants to be known,¹³⁾ the difference reflectance measurements contain information primarily on silver since the Al reflectance is relatively structureless in the photon energy region between 1.0 eV and 6.0 eV. These measurements in turn can be used to check the self-consistency of the known silver dielectric function, a procedure we found to give agreement to the order of a percent. It should be noted that the silver reflectance can vary by this same order of magnitude simply because of varying conditions of deposition of the metal layer. As an example, such variations can be produced by film inhomogeneities, or grain-domain size effects which are mechanisms which can lead to roughness changes in the reflectance and which have, consequently, formed topics for further study. In this regard, the reflectance of a rough silver surface is shown in Fig. 2. This illustration includes the appearance of a surface plasma resonance dip at a frequency ω such that $\epsilon_R(\omega) = -1$ where ϵ_R is the real part of the metal dielectric function. This pole in surface wave metal scattering has formed a good deal of the basis of our current attention to this problem.

The air or vacuum scattering contribution of the metal foil or of the oxide-covered metal has been measured concurrently with the reflectance. The method employed in present work has utilized a normally-incident, polarized Ar^+ or He-Ne laser beam impinging on the interface under study. The scattered light is then collected with a polarized, rotating-arm photomultiplier having an acceptance solid angle of $\sim 3 \cdot 10^{-3}$ sr. Examples of measured metal scattering distributions obtained using this technique are shown in Fig. 3. As is evident from the curves shown, the s-wave (B-vector in scattering plane) and p-wave (E-vector in scattering plane) scattering are nondegenerate.

FIGURE 2

This is a characteristic of surfaces having inhomogeneity or roughness correlation lengths $a \ll \lambda$. In the limit that $a \gg \lambda$ the s and p wave scattering become degenerate. Such simpler spectra ($a > \lambda$) are also often observable, but really are only characteristic of surfaces which are wavy, rather than those which are truly rough. Thus, for the present purposes, they are somewhat uninteresting.

In our current investigation the correlation length has been defined by

$$\langle \zeta(\vec{R}) \zeta(\vec{R} + \vec{\rho}) \rangle = \zeta_0^2 e^{-\rho^2/a^2} \quad (1)$$

where the function $\zeta(\vec{R})$ is the metal surface topography function and ζ_0 is the r.m.s. roughness height. First order light scattering theories have recently been developed to account for surface inhomogeneities,¹⁴⁻¹⁶⁾ and we have made use of these models in the data analysis discussed in this report. This includes both vacuum diffuse reflectance having scattering cross sections $dR^{S,P}/d\Omega$ as well as the surface electronic-plasma wave contribution to the reflectance change which, in the zero damping limit, is

$$\Delta R_{sp} = \frac{16\pi^5 a^2 \zeta_0^2}{\lambda^4} \frac{\epsilon_R^2}{(-[\epsilon_R + 1])^{5/2}} \exp\left(-\left[\frac{\epsilon_R}{\epsilon_R + 1}\right] \frac{\pi^2 a^2}{\lambda^2}\right) \theta(-[\epsilon_R + 1]) \quad (2)$$

Here $\epsilon_R = \text{Re}\epsilon$, θ is the unit positive step function, and λ is the optical wavelength. Both the roughness scattering and absorption have been considered in analyzing our present data. Because of finite damping corrections ($\epsilon_I = \text{Im}\epsilon > 0$) numerical calculations have been necessary

FIGURE 3

in order to properly compare experiments to theoretical work. However, with the aid of previously known metal optical constants, this has yielded plausible values for metal roughness parameters. Moreover, it has also given one of the first self-consistent experimental determinations of roughness from both scattering and reflectance. The value for the power spectrum of roughness correlations obtained from both of these two experiments is in general agreement. However, the tendency for internal metal scattering is to require a slightly higher power spectrum of corrugations than for air scattering.¹⁷⁾ The numerical difference can be up to 25 to 50% and can be understood only with further investigation.

A more detailed examination of surface plasma wave scattering has also been conducted. The location of the surface plasmon resonance wavelength is dependent on a number of fine-structural corrections which are not included in the analysis summarized by Eq. (2). Thus, to ensure an accurate comparison to experimental reflectance and scattering measurements it is desirable to examine further theoretical extensions. In Table I we have listed a number of these contributions to the surface plasma resonance wavelength.¹⁸⁾

As a matter of summary in relation to this table, we can mention these corrections in turn. Using the real unscreened Drude expression $\epsilon = 1 - \omega_p^2/\omega^2$ for the dielectric function and $\omega_p = 13.85 \times 10^{15}$ Hz for the Ag bulk plasma frequency, we obtain the uncorrected surface plasmon pole of $\omega_{sp} = \omega_p/(2)^{1/2} = 9.79 \times 10^{15}$ Hz. Including the zero-frequency d-band (and atomic core) screening contribution gives $\omega_{sp} = \omega_p/(1 + \epsilon_0)^{1/2} = 6.54 \times 10^{15}$ Hz using $\epsilon_0 = 3.5$.²⁾ A further slight extension (third line of the table) is to include the frequency dependence of the d-band correction, which is equivalent to using the known optical

CONTRIBUTION	$\epsilon(\omega)$	ω_{sp}	λ_p^0 ($\lambda_p = 1360 \text{ \AA}$)	$\omega(10^{15} \text{ Hz})$	$h\nu(\text{eV})$	$\Delta\lambda$	$\Delta\omega(10^{15})$	$\Delta E(\text{eV})$
Bare resonance	$1 - \frac{\omega_p^2}{\omega^2}$	$\omega/p\sqrt{2}$	1924	9.79	6.32	920	-3.07	-2.02
Zero-frequency d-band correction	$\epsilon_0 - \frac{\omega_p^2}{\omega^2}$	$\omega/p\sqrt{1 + \epsilon_0}$	2880	6.54	4.30			
Self consistent d-band correction	$\epsilon_d(\omega) - \frac{\omega_p^2}{\omega^2}$	$\frac{\omega_p}{\sqrt{1 + \epsilon_d(\omega_{sp})}}$	3350	5.62	3.70	50	-0.08	-0.05
Damping due to electron-hole excitations and interband transitions	$\epsilon_R + i\epsilon_I$		3400	5.54	3.65	50	-0.08	-0.06
Correlation length cutoff	$a = 800 \text{ \AA}$		3450	5.46	3.59	80	-0.12	-0.08
Self-energy correction			3530	5.34	3.51			
TOTALS						1570	-4.27	-2.81

Experiment: 3550 Å

Table I: Summary of Corrections to the Surface Plasma Resonance Wavelength of Silver as Determined from Previous Work

dielectric function $\epsilon_R(\omega)$ in a self-consistent way to estimate the surface plasma frequency. This yields $\omega_{sp} = 5.62 \times 10^{15}$ Hz. Making allowance for damping which results from both electron-hole and interband excitations, we introduce an imaginary component to ϵ which rounds the pole in the surface plasmon dispersion relation, further shifting the resonance. The form for the dispersion relation in this case is

$$K^2 = \text{Re}\left(\frac{\epsilon}{\epsilon + 1}\right) k_0^2 \quad (3)$$

where $\epsilon = \epsilon_R + i\epsilon_I$. This form for the surface plasmon dispersion points to a further correction for stochastically-roughened surfaces which will again shift the resonance location. In the Gaussian correlation-length approximation the dominant surface wavevectors K are of order $2/a$ where a is the surface correlation length (approximately 800 to 1000 Å for our foils). Thus, we solve Eq. (3) for the corresponding values of K . Carrying this out gives the surface plasma frequency of $\omega_{sp} = 5.46 \times 10^{15}$ Hz. (3450 Å) which is close to the measured number of approximately 3550 Å.

At least part of the remaining discrepancy between calculated and measured numbers can be accounted for by considering the second order surface scattering of surface plasmons. This is perhaps the most technically complicated correction we have presently analyzed and may be considered a form of radiative self-energy effect. The usual surface plasmon dispersion relation in this case (Eq. 3) is modified to contain an additional additive term on the right hand side (call this Σ). The quantity Σ is understood to be a correction integral and its form follows recent calculations.^{19,20} The form of the dispersion relation Eq. (3) including the Σ correction is shown in Fig. 4. The observed effect of this term is to shift the surface plasma resonance to longer

FIGURE 4

wavelengths for correlation lengths a in the range 500 to 3000 Å. Below this range of correlation lengths the calculation does not apply, and above it the correction is small, approaching zero.

In including this quantity in our analysis we may ensure a self-consistent comparison of scattering and reflectance data from a given metal foil. However, the tendency found in carrying out this comparison is that measured correlation lengths are shorter than would be predicted in the absence of the self-consistent scattering correction. Thus, the inclusion of the effect might seem unjustified. This is not the case, however, since further examination of the trend in surface plasma wave resonance shifts as the surface correlation length is changed gives more accurate comparisons to measurement than when this correction is omitted.¹⁷⁾ This effect was studied by examining a series of different foils¹²⁾ though it is also suggestive to study this effect using annealing. However, we find that annealing in an inert Ar atmosphere changes the surface electronic properties in such a way as to compensate for roughness changes which could occur due to such heat treatment. In fact, measurement of the surface plasma resonance location shows that this peak shifts opposite to what is expected due to roughness changes on annealing. This assumes that annealing acts to average out higher Fourier roughness components. What may occur instead is that the optical constants of the metal may be modified slightly, particularly the electronic relaxation rate and thus ϵ_1 . If ϵ_1 is increased due to increased electronic surface scattering, then the observed surface plasmon frequency increase can be explained. In added support of this interpretation is the consistency of reflectance decreases also observed during the annealing process. This can be explained by changes in ϵ_1 of the same order of magnitude as for the surface plasma wave shift. The measured values

of the change in ϵ_1 , $\delta\epsilon_1$, are of order ± 0.25 . These produce surface plasma wave shifts $\delta\omega/\omega$ of ~ 0.01 and reflectance decreases ΔR of 0.01 to 0.02.¹⁷⁾

Our present oxide layer on metal study has been confined to thin oxide residues formed on the metal. These overlayers are formed by voltage cycling clean silver foils in a potassium hydroxide solution¹⁰⁾ to potential differences of order 0.3 V. The use of such a low limiting voltage is thought to yield predominantly Ag_2O rather than other oxidation states although this is presently uncertain. The reflectance changes $\Delta R/R$ introduced by formation of the inhomogeneous surface oxide on the metal are distinct for various situations as illustrated in Fig. 5. It is presently thought that the oxide as formed is deposited relatively smoothly on the surface and that it may roughen when the residue is formed during voltage cycling in the liquid. Unfortunately, it is currently difficult to distinguish whether any additional liquid polar layers may contribute to the in situ reflectance signal. In the case of the ex situ residue oxide the reflectance is also modified (Fig. 5) perhaps because of slight hydration or because of structural changes in the overlayer outside the liquid. The scattering from such layers is characteristic of an inhomogeneous composite having cluster size comparable to the wavelength ($a \sim \lambda$) as is suggested from the electron micrographs of Fig. 1. In this case the observed s- and p-wave scattering rates are very nearly degenerate, a condition not obtained in the case of roughness where $a < \lambda$. As one extension of this work we have examined the surface plasma metal resonance in the presence of various liquids. When spontaneous oxidation occurs, the peak is damped; however, in the case of a clean surface the approximate calculation given by Eq. (2) but including damping gives a good fit to the measured

FIGURE 5

lineshape. The same roughness parameters apply for various liquids with only a change of refractive index n . However, the peak shift is greater than that given by the condition $\epsilon_R = -n^2$. The reason is that the correlation length a becomes effectively longer in the liquid, approaching na . The dependence of the reflectance on the correlation length thus results in the extra wavelength shift.

III. RESEARCH PERFORMED DURING THE CONCLUDING PERIOD OF DOE CONTRACT SUPPORT

The efforts of the past several months have been directed to reviewing not only the surface plasmon behavior of stochastically roughened surfaces, but also to studying surfaces with either partial or complete order. In both of these cases we have been interested in the bare metal interface. As described previously, stochastic surfaces may be produced by evaporation of silver on calcium fluoride underlayers. In the case of lattice order the sub surfaces used are gratings or grating-like structures whose basic scattering properties have important applications to several scientific and technological areas. The present interest is in rather weakly periodic surfaces whose correlation functions are also periodic; thus we consider surfaces having $\langle \zeta(\vec{R}) \zeta(\vec{R} + \vec{\rho}) \rangle$ periodic in $\vec{\rho}$ and with a linear replication length of $\vec{L}$. This means that the correlation function is periodic in $\vec{\rho}$ with a repeat distance of $\vec{L}$. The criterion for weak scattering means that the rms corrugation height $(\langle \zeta^2 \rangle)^{1/2}$ is small; in particular much smaller than the optical wavelength λ .

During the time interval of this support we have examined a number of such corrugated surfaces, including some common linear gratings. The reflectance of these surfaces reveals a large variation in the surface plasma wave absorptance, depending on roughness, surface topographic order, or material layer combination. Scanning electron micrographs of some distinct cases are summarized in the electron micrographs of Fig. 6. Included here are a stochastic surface similar to those shown in Fig. 1 which is a calcium-fluoride roughened silver surface consisting of an opaque metal layer on approximately 0.25 μ of underlayer

FIGURE 6

(A). The case B is an optically opaque silver overcoat on a 600 L/mm grating. The cases C and D show opaque silver surfaces on a ruled holographic grating with a linear replication length of approximately 2800 Å. This is a grating with 3600 lines per mm. This sample is important because the replication distance is smaller than an optical wavelength and thus the cross-section for surface wave absorption is expected to be large. This is expected, since the grating wavevector for this metal silver substrate is larger than the optical wavevector k_0 and thus the normal component of the propagation vector for surface diffracted waves will be imaginary. This means a surface plasma wave component is expected to be large as is observed (see Fig. 8D, and discussion below).

In Fig. 7 we show a series of calcium-fluoride roughened optically-opaque silver surfaces showing reflectivity near the screened plasma edge for various stochastically-roughened silver-calcium fluoride combinations. The cases B and C are similar to those discussed earlier in this report and give a sizeable surface plasmon peak. The difference between these two instances lies in the nature of the deposited metal overlayer itself; namely in a small difference in the electron mean-free scattering rate and thus in ϵ_1 for the metal. The value of ϵ_1 is higher for case C than B. This means the reflectivity of this sample is lower than that of B. It also means that in the wavelength region between the bulk plasma frequency and the surface plasma frequency the reflectance of the surface will be lower. This is the region of the metal for which $0 > \epsilon_R > -1$ and is a roughness "restray region" in which surface plasma wave absorptance in the metal does not occur.¹²⁾ However, the magnitude of the reflectance in this same region depends on the value of ϵ_1 and decreases as ϵ_1 is increased. This behavior is very like the behavior

FIGURE 7

of an ordinary lattice restry region for ionic crystals. In case D the stochastic-roughened surface is stronger (higher rms roughness ϵ_0) than in the cases B and C. The correlation width a is inhomogeneously distributed yielding a broadened surface plasma wave minimum in this case as compared to the former cases. The application of the simple roughness considerations introduced earlier (Eq. 2) are appropriate in only a qualitative manner without a further modification of the theory to include higher-order effects expected with such larger roughness parameters.

In the next sequence of results we show reflectivity of some ordered surfaces again having optically opaque silver overlayers. We consider several cases. For silver on ruled plastic (case B) with 400 L/mm (available from Edmund Scientific Co.) the reflectivity near the screened plasma edge follows a linear ramp between the bulk plasma frequency and a lower frequency onset (indicated by an arrow). This result is somewhat surprising, but can perhaps be explained by an argument involving density of surface (grating) scattering states, since we approach the limit of low grating wavevectors (a quasicontinuum of diffracted wavevectors) for this sample. However, this analysis will not be carried out here. The silver on 600 L/mm grating (case C) is similar, although because of a larger grating step size the reflectivity is considerably reduced from that of the earlier case (B). The onset wavelength occurs for longer wavelengths than are observable with our current spectrometer. More importantly, however, the blazed grooves in our substrate in this case are large enough (500 to 1000 Å) so that further corrections to our roughness analysis is again necessary to deal with this case. It is surprising that the plasma edge absorptance is so strongly attenuated in this instance, since this optical property involves such a strong property of the silver metal.

FIGURE 8

The case D shows the silver reflectance for a holographic substrate for $\ell < \lambda$, as mentioned above. The substrate is available commercially.²¹⁾ The observed reflectance curve shows a rather broad surface plasmon minimum, with the valley occurring near 4200 Å. No splitting of the resonance minimum is obtained for this sample, such as has previously been suggested for plasma-wave scattering from ordered substrates.^{20,22,12)} Such splitting has previously been obtained in "accidental" induced roughness from potassium-vacuum interfaces²³⁾ and in other related experiments.

As a final set of sample cases whose reflectance has been examined we discuss a series of silver-composite layered materials. In example B in this sequence (Fig. 9) a rapidly evaporated magnesium sublayer has an opaque silver coating overlayer. This example shows some weak surface roughness as is evident from the reflectance notch observed in the reflectivity adjacent to the bulk plasmon. However, the roughness is much weaker than for analogous calcium-fluoride silver combinations. In cases C and D we examine some simple tri-layers consisting of partially transparent silver on a thin dielectric (TiO_x - C) or metal (Mg - D) underlayer evaporated on an opaque silver sublayer. The reflectance of each of these combinations again reveals a surface plasmon correction to the reflectance. No splittings of the surface plasma resonance are observed. Surprisingly, the silver-titanium oxide-silver sandwich shows a rather broad-range reflectance decrease near the surface plasma frequency. This reflectance structure appears rather like the linear ramp reflectance decrease observed for the opaque silver overcoat on the 400 L/mm grating of Fig. 8. This particular case would perhaps prove to be more interesting if a sequence of such layers were examined. Presently, however, the limits on precision of the evaporation technique

being used unfortunately restricts such a more sophisticated tri-layer study.

FIGURE 9

FIGURE CAPTIONS

- Fig. 1: Scanning electron micrographs of roughened surfaces examined in optical scattering studies presented in Section II of this report. The cases shown include:
- a) Calcium fluoride roughened silver having surface correlation lengths in the range $800 - 1000 \text{ \AA}$.
 - b) Silver surface having both rough and wavy components (i.e., regions with both short and long range surface correlations, respectively).
 - c) Latex spheres (diameter = $890 \text{ \AA}$) on glass.
 - d) Evaporated silver on latex sphere surfaces of preceding case C producing a macroscopically roughened silver surface.

Fig. 2: The reflectance of a rough silver surface in the neighborhood of the screened plasma edge. Roughness parameters for this spectrum are determined as shown to the left of the figure. The dip in the reflectance in the wavelength region near $3500 \text{ \AA}$ is due to resonant p-wave scattering of incident radiation into the metal and occurs at a frequency ω such that $\epsilon_R(\omega) \approx -1$. The rise in the reflectance at higher energy below the bulk plasmon onset ($-1 < \epsilon_R(\omega) < 0$) is due to a "restray" region in which no roughness absorption is possible.

Fig. 3: Metal roughness scattering distributions at various Ar^+ laser wavelengths for a rough silver surface. Four parameter

fits of the data have been made to calculated s- and p-wave polarized scattering distributions to include both rough and wavy contributions to the scattering, i.e. both short and long range surface correlations.

Fig. 4: Surface plasmon dispersion relation plotted in terms of surface wavevector K versus frequency ω in the physically interesting frequency region. Corrections to the dispersion curve due to self-energy corrections are included. The dominant K -vector in the surface scattering process for CaF_2 -roughened silver is of order $2/a$ where a is the surface correlation length (800 - 1200 Å). This yields dominant surface plasmon wavevectors in the range 1.6 to 2.5×10^{-3} .

Fig. 5: Difference reflectance signals for silver in the visible energy range for photon energies $h\omega$ between 1.5 eV and 5.5 eV showing reflectance changes under various oxidation conditions. In case (A) a quasi-equilibrated layer is shown when a cell voltage of 0.3 V is applied,²⁾ in case (B) the same layer is shown when the cell voltage has been cycled to zero, and in case (C) the residual layer is shown outside the liquid. The quantity $\Delta R/R$ is clearly distinct in all cases. The dip P is the surface plasmon which shifts to higher energy in air (C) compared to liquid (A) and (B).

Fig. 6: Electron micrographs of both disordered and ordered surfaces studied using difference-reflectance spectroscopy for the work discussed in Section III of this report. The cases

shown in this instance are

- A) Calcium-fluoride roughened silver having surface correlation lengths in the range 800 - 1200 Å similar to case a of Fig. 1.
- B) Silver-coated 600 L/mm grating showing lines spaced at 1.6 μ.
- C) Low-power scanning-electron micrograph of sample of case B illustrating surface blaze of this replicated ruled surface (to be compared to holographic grating).
- D) Holographic grating surface covered with an opaque Ag overlayer. The line density is 3600 L/mm yielding a grating spacing of 2800 Å. Magnification is 10^4 .
Note the distinction of case D to case B of the grating spacing being less than the wavelength in the former and greater in the latter. This affects the optical spectrum.

Fig. 7: A series of calcium-fluoride roughened optically-opaque silver surfaces showing reflectivity near the screened plasma edge for various example cases:

- A) Smooth silver on glass.
- B) CaF_2 ($\sim 0.25 \mu$ thick) on glass covered with opaque silver.
- C) CaF_2 ($\sim 0.25 \mu$ thick) on glass covered with opaque silver having higher electron scattering rate (higher ϵ_I) than for case B. This reduces the size of the restry hump (where $0 > \epsilon_R > -1$) between the surface-plasmon dip and the plasma edge.
- D) Thicker CaF_2 layer ($\sim 0.6 \mu$) showing broadened surface

plasmon absorption compared to former cases. Note also that the minimum in the reflectance is shifted to longer wavelength in this case indicating longer average surface correlation lengths.

Fig. 8: A series of reflectance spectra for opaque roughened-silver on ordered sublayers

- A) Smooth opaque silver on glass substrates.
- B) Opaque silver on ruled plastic having 400 L/mm.
- C) Opaque silver on a 600 L/mm ruled grating. The reflectance in this case is strongly reduced and the plasma edge of the metal is not strongly distinct. No surface plasma wave resonance is observed.
- D) Opaque silver on a holographic grating substrate showing a broad minimum (arrow) due to surface plasmon absorption near 4200 Å.

The cases C and D in this figure are those samples shown in the scanning electron micrographs of Fig. 6 in cases B and D respectively.

Fig. 9: Reflectance of a series of silver-composite layered materials near the screened plasma edge of silver

- A) Smooth opaque silver surface showing bulk plasma edge and serving as a comparison to material-layer combinations considered in following cases. Evaporation is on glass.
- B) Opaque silver layer on rapidly evaporated magnesium sublayer ($\sim 0.3 \mu$) showing a surface with a weak surface plasmon and thus some roughness.

- C) Tri-layer consisting of partially-transparent 500 Å of silver metal evaporated on a sublayer (800 Å) of TiO_x evaporated on an opaque silver underlayer. This combination shows some distributed surface plasmon absorption.
- D) Partially-transparent 400 Å layer of silver on 600 Å of Mg on an opaque layer of silver.

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V. LIST OF PUBLICATIONS RESULTING FROM RESEARCH
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Journal articles:

- P.D. Parry, T.R. Carver, S.O. Sari, and S.E. Schnatterly, "Optically pumped and monitored electron-nuclear double resonance in alkali halides," *Phys. Rev. Lett.* 22(8):326-329, 24 February 1969.
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- S.O. Sari, "Observation of Landau Levels at the E_1 edges of InSb and GaSb," seminar given at the Massachusetts Institute of Technology, July 1971.
- S.O. Sari and S.E. Schnatterly, "Optical spectroscopy in semiconductors in high magnetic fields using polarization modulation," Surface Science 37:328-339, 1973; invited paper at the First International Conference on Modulation Spectroscopy, Tucson, Arizona, November 1973.
- S.O. Sari, "Production of tunable picosecond pulses from dye lasers," colloquium given at Bell Laboratories, Holmdel, New Jersey, October 1974.
- S.O. Sari, "Surface effects in the magneto-optics of heavy semiconductors," Materials Science Center Colloquium, Massachusetts Institute of Technology, April 1975.
- S.O. Sari, "What we can learn about semiconductor surfaces from optical experiments," colloquia given at the Department of Applied Physics, California Institute of Technology, and at the Department of Physics, University of Washington, December 1975. Seminar given at the Optical Sciences Center, University of Arizona, July 1975.
- S.O. Sari, "Optical properties of semiconductors in the solar spectrum," Conference on Optical Properties of Semiconductors in Relation to Solar Energy, Tucson, Arizona, November 20-23, 1975, invited talk.
- S.O. Sari, "Properties of oxygen impurity states in disordered films of silicon," seminars given at Argonne National Laboratory, Argonne, Illinois, and at Oak Ridge National Laboratory, Oak Ridge, Tennessee, March 3-4, 1977.
- S.O. Sari, "Photoelectrolysis and the oxidation of metallic silver," given at the Review of the Materials Sciences Program of the Department of Energy, Ames Laboratory, Iowa State University, Ames, Iowa, 29-31 November 29-31, 1977.
- S.O. Sari, "Scattering and absorption due to roughness on clean and oxide covered silver surfaces," talk given to the Solid State Division of Argonne National Laboratory, March 16, 1979.
- S.O. Sari, "The reflectance of rough silver," colloquia given at the University of Utah and the Oregon Graduate Center June, 1979. Seminar given at Oregon State University, July 1979.
- S.O. Sari, "Scattering and Reflectance of Rough and Oxide-Covered Metal Layers," talks given at Brown University and at the GTE Advanced Technology Laboratory, Waltham, Mass. in March, 1980; talk given at the Tektronix Materials Colloquium, January, 1980.

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