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Optical properties of the Au(110) surface

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We have used reflectance anisotropy spectroscopy to determine the surface dielectric anisotropy of Au(110) under ultrahigh-vacuum conditions. Additional angle-resolved photoemission spectroscopy measurements were carried out to correlate surface optical with surface electronic properties. The surface dielectric anisotropy mainly results from single-particle excitations involving the near surface bulk d and sp bands and free-electron contributions in the spectral region below ~ 2.4 eV. Surface-state contributions to the surface optical properties of Au(110) are discussed.

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I. INTRODUCTION

Reflectance anisotropy spectroscopy (RAS) has become a widely used technique to study surface optical properties. It measures the difference of the reflectivity along two perpendicular directions on the surface. The surface sensitivity of RAS results from the different symmetries of the bulk and the surface, e.g., the unreconstructed (110) surface of a face-centered-cubic (fcc) lattice with O_h symmetry has a C_{2v} symmetry. Since the difference in reflectivity along two directions of a cubic crystal for the bulk will be zero, remaining signals must be related to the surface. So far, RAS was mainly used to study cleaved (110) and reconstructed (100) surfaces of semiconductors,[1,2] however, more recently, investigations have been extended to metal surfaces.[3–12]

Comparably well understood is the optical anisotropy of the (110) surfaces of Ag and Cu, two materials of similar electronic properties as Au. On the former metal surfaces, different contributions to the surface optical anisotropy were identified.[9,12] On clean, (1×1) reconstructed Cu(110) and Ag(110) surfaces one source of optical anisotropy is due to electronic transitions between surface states (or resonances) at points of high symmetry in the surface Brillouin zone (SBZ). Apart from that, symmetry lowering induced by the surface gives rise to an optical anisotropy caused by electronic transitions involving the bulk d and sp bands. Finally, an anisotropy of the free-electron part may contribute to the surface optical properties.[7]

In recent years, the understanding of surface optical properties has advanced, particularly through calculations of the optical response based upon sophisticated numerical methods.[13] Unfortunately, to our best knowledge, the theory work so far has focused on semiconductor surfaces, while much less has been done on metal surfaces. RAS spectra of metals have been calculated within a semiclassical surface local-field theory developed by Mochán and Barrera in 1985.[14] The model is based on a phenomenological approach considering local surface dipoles and their interaction with the macroscopic dielectric response. It describes the intrinsic optical anisotropy of a clean bulk truncated crystal, but does not include microscopic surface properties such as the surface band structure. The model was successfully used to explain those parts observed in the reflectance anisotropy of Cu and Ag(110), which are not correlated with surface-state transitions.[3,10]

Investigations of the surface optical properties on Au(110) started a few decades ago. The electroreflectance measurements of Kofman *et al.*[15] yielded an anisotropic optical response. More recently RAS measurements were carried out in air[16] and in an electrochemical environment.[17] Also experiments under clean ultrahigh-vacuum (UHV) conditions have been performed recently.[18] The surface electronic properties of clean Au(110) surfaces, on the other hand, have been studied using photoemission and inverse photoemission. Occupied surface bands have been investigated by Heimann *et al.* with ultraviolet photoemission spectroscopy.[19] They reported an occupied surface state at the $\overline{Y}$ symmetry point of Au(110),[20] in analogy to the states at $\overline{Y}$ found on clean Cu(110) and Ag(110) surfaces. In refined photoemission experiments, however, the surface state at $\overline{Y}$ was not reproduced.[21] This was ascribed to the (1×2) missing row reconstruction of the clean Au(110) surface which is distinct from the (1×1) reconstruction of the (110) surfaces of Ag and Cu.[22] Moreover, several inverse photoemission studies have been published. They also disagree in the assignment of the experimental structures to unoccupied surface states.[23–26]

In this paper, we report an investigation of the surface optical properties of Au(110) under UHV conditions. Besides the RAS experiments, additional angle-resolved ultraviolet photoemission spectroscopy (ARUPS) measurements were performed in order to correlate the surface optical and electronic properties.

II. EXPERIMENT

The experiments were carried out at the SX700 beamline of the storage ring ASTRID in Århus. The base pressure of the ultrahigh-vacuum chamber was better than 5×10^{-11} mbar. The sample was cleaned using ion bombardment. A few cycles of Ar^+ sputtering (0.5 keV, 8 μA sample current) and annealing (to 400 °C) were sufficient to produce a good (1×2) low-energy electron-diffraction (LEED) pattern. The absence of contaminations was checked using x-ray photoemission spectroscopy.

The RAS spectrometer is a homebuilt system which is based on the original design by Aspnes.[27] The light from a 75 W Xe short arc lamp is focused onto the sample through a polarizer and a strain-reduced quartz window attached to the ultrahigh-vacuum system. After reflection under near-normal incidence the light contains a small elliptically polarized component, because of the different reflection coefficients of $r_{[1\bar{1}0]}$ and $r_{[001]}$. The state of polarization is detected using a combination of a photoelastic modulator (PEM) and an analyzer. A quartz PEM, a quartz Rochon prism as polarizer, and an α-BBO (barium oxoborate) Rochon prism as analyzer are used. Quartz is transparent between 0.44 and 6.9 eV and α-BBO between 0.35 and 6.5 eV. The system is equipped with a single grating monochromator containing two gratings of 600 lines/cm and 1200 lines/cm. Together with a Si photodiode as detector a spectral range of 1.3–5.5 eV is accessible. RAS measurements presented in this paper were restricted to 1.5–5 eV.

The RAS spectrometer records the real and the imaginary parts of the complex reflectance anisotropy defined by

$$\frac{\Delta r}{r}=2\frac{r_{[1\bar{1}0]}-r_{[001]}}{r_{[1\bar{1}0]}+r_{[001]}}. \tag{1}$$

If the bulk dielectric properties ε_b are known, $\Delta r/r$ subsequently can be converted to the surface dielectric anisotropy (SDA).

To calculate the SDA, a three-phase model is considered.[28] It assumes a homogeneous bulk with an isotropic dielectric function ε_b, a very thin biaxial surface layer (thickness $d\ll\lambda$) with a dielectric anisotropy defined by $\Delta\varepsilon=\varepsilon_{[1\bar{1}0]}-\varepsilon_{[001]}$, and a surrounding ambient with a refractive index of 1. The surface dielectric anisotropy can then be calculated from the reflectance anisotropy according to[28]

$$\Delta\varepsilon d=\frac{\lambda}{4\pi i}(\varepsilon_b-1)\frac{\Delta r}{r}. \tag{2}$$

The bulk dielectric function ε_b which is needed for this analysis was taken from Ref. 29.

The photoemission experiments at the synchrotron storage ring were carried out using a Vacuum Generators angle resolved spectrometer (ADES 400) for electron detection. The angular resolution was $\pm2°$ and the combined energy resolution of analyzer and beamline was about 180 meV in the energy range used for valence-band spectroscopy (around 30 eV). The angle of incidence of the photons with respect to the surface normal was fixed to 45°.

III. RESULTS AND DISCUSSION

A. RAS

Figure 1 shows RAS spectra (real part) of differently prepared Au(110) surfaces. The spectrum of a clean surface is shown together with spectra taken on potassium-covered surfaces and a model calculation. The spectrum labeled clean (1×2) was recorded after sputtering and annealing. Major spectral features are found around 1.9 eV, at 2.5, 2.8, 3.5, and 4.5 eV. The dominant features occurring for photon energies above 2.4 eV are related to transitions involving the bulk bands of the Au 5*d* states.

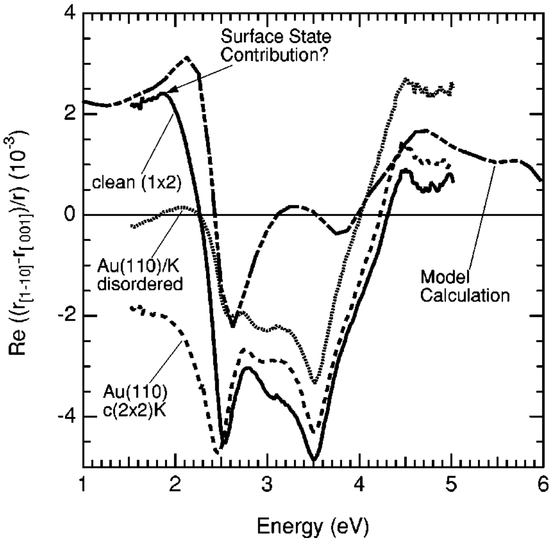

FIG. 1. RAS spectra of different Au(110) surfaces. Also shown is a spectrum calculated from Eq. (3) with $\Delta E=0.3$ eV, $\Delta\Gamma=0.7$ eV, and $d=1$ nm.

A sensitive test of surface-state-related features in the RAS spectra is usually the exposure to a small amount of reactive gas molecules. Since contaminants quench the electronic surface states, the according spectral features will vanish from the RAS spectra. In the cases of the Cu and Ag(110) surfaces a gas like oxygen or nitrogen was successfully used to alter the surface.[9,12] The Au(110) surface, however, is not reactive with oxygen or nitrogen. Therefore potassium was evaporated onto the sample (≈0.5 ML), in order to modify the surface electronic structure and to experimentally distinguish between bulk and surface contributions to the RAS spectra. Deposition of K at room temperature induced a disordered surface as evidenced by LEED. The optical spectrum also changed, as displayed in Fig. 1. In the real part of the anisotropy spectrum we observe a quenching of the signal below the onset of the Au 5*d* bands at 2.4 eV. The bulk *d*-band-related features at higher energies, however, are still visible but with reduced amplitude. Annealing of the K-covered surface to 400 °C resulted in a centered (2×2) LEED pattern. An array of alternating Au and K atoms is known to form this structure.[30] For this surface the RAS signal related to the Au 5*d* bulk interband contributions above 2.4 eV is rather similar to that of the clean Au(110) surface, whereas the anisotropy below the onset is negative.

Similar spectral features have also been found in RAS experiments performed on Au(110) in an electrochemical environment.[17] The strength of the minimum at 2.5 eV was found to decrease with increasing disorder of the surface. The anisotropy below 2.5 eV also varies with the order of the surface. The line shape of the RAS spectrum corresponding to the clean (1×2)reconstructed surface in Fig. 1 is similar to the (1×1)spectrum of Ref. 17. The difference in the anisotropy below 2.5 eV was explained with a difference in surface conductivity of the electrons across the atomic rows (the [001] direction) and perpendicular to it.[17] More collisions of the electrons lead to a shorter Drude relaxation time and a decrease in reflectivity in the [001] direction. This is consistent with the results presented here (see Fig. 1): On the potassium-covered, disordered surface there is no preferred direction for collisions, hence the anisotropy in the region below 2.5 eV is zero. In a similar manner the negative anisotropy observed on the potassium-induced centered (2×2) Au(110) surface could be related to distinct free-electron properties of that particular surface.

As already mentioned above, Mochán *et al.*[14,16] used a phenomenological surface-local-field model to calculate the surface optical anisotropy of *d*-band metals. Surface-state transitions are not considered in this approximation, but the bulk optical anisotropy may be reproduced. In Ref. 16 the optical anisotropy of the Au(110) surface was calculated assuming a bulk truncated (1×1) surface structure. In contrast to the examples of Cu(110) and Ag(110), for Au(110) the agreement between RAS data with the surface-local-field calculation is not satisfactory.[3,10,16] The calculated spectrum exhibits a peak at 2.5 eV but with a wrong sign, and the structure at 3.5 eV is not resolved. The calculated curve looks similar to the optical anisotropy of Cu(110) rather than Au(110).[10] Probably the surface structure is the crucial quantity leading to the discrepancy between calculated and experimentally determined optical anisotropy of Au(110). In contrast to the Cu(110) surface, Au(110) exhibits a (1×2) reconstruction which was not taken into account by Mochán *et al.*

Another qualitative description of the reflectance anisotropy related to bulk transitions can be attempted by the energy derivative of the bulk dielectric function. This approach is justified if peaks in the RAS spectra are close to the spectral structures of the bulk dielectric function (ε_b) of the material. It is assumed that surface-induced symmetry lowering leads to surface dielectric functions $\varepsilon_{[1\bar{1}0]}$ and $\varepsilon_{[001]}$ which are very closely related to ε_b, but with slightly different gap energies (E_g) and broadening parameters (Γ) for interband transitions in the $[1\bar{1}0]$ and $[001]$ directions. For the reflectance anisotropy it follows that[31]

$$\frac{\Delta r}{r} = -\frac{4\pi i d}{\lambda}\,\frac{(-\Delta E_g + i\Delta\Gamma)}{\varepsilon_b - 1}\,\frac{\partial \varepsilon_b}{\partial E}. \tag{3}$$

The dielectric function of gold used for the calculation was taken from the literature.[29] The result of this calculation is shown in Fig. 1 together with the experimental spectra. The parameters are $\Delta E = 0.3$ eV, $\Delta\Gamma = 0.7$ eV, and $d = 1$ nm. The calculation reproduces the major features of the bulk *d*-band-related transitions. Amplitude and line shape of this calculation, however, differ significantly from the experimental data. All structures in the model calculation are shifted to higher-energy values by 0.1 to 0.2 eV. The approximate reproduction of the data indicates that the main peaks in the RAS spectra of Au(110) result indeed from transitions involving bulklike electronic states, modified by the anisotropic surface.

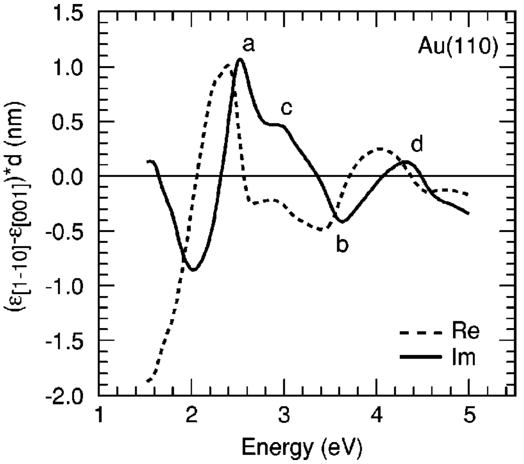

FIG. 2. Surface dielectric anisotropy (SDA) of the clean Au(110) surface. Main transitions involving the Au bulk bands are labeled a–d.

In Fig. 2 the surface dielectric anisotropy calculated according to Eq. (2) is shown. For the calculation, smoothed RAS spectra of the clean Au(110) surface and the dielectric function taken from Ref. 29 were used. The main features are clearly visible in the imaginary part of $\Delta\varepsilon$ and correspond energetically to the transitions found in the bulk dielectric function. The major peaks found in $\Delta\varepsilon$ are attributed to interband transitions from the Au $5d$ and Au $6sp$ bands into unoccupied states (surface modified bulk states). An inspection of the bulk band structure leads to the following tentative assignment: Transitions at the L symmetry point are responsible for the strong maximum (a) at 2.5 eV, the minimum (b) at 3.5 eV, and the maximum (d) at 4.4 eV. The shoulder (c) at 2.8 eV results from a transition at X.[32]

Transitions between surface states may also contribute to the reflectance anisotropy of Au(110), in analogy to the cases of Cu and Ag(110). Inspection of Fig. 1 yields a small positive feature at 1.9 eV which is absent on the potassium-covered surfaces. Apart from potassium deposition, annealing of the Au sample provides another possibility to prepare a disordered (110) surface. Corresponding RAS spectra recorded between 40 °C and 550 °C are shown in Fig. 3. The Au(110) surface should undergo a phase transition for temperatures above around 400 °C at which the LEED pattern changes from (1×2) to (1×1).[33] This pattern was interpreted as an indication of a disordered phase which forms at temperatures below that of surface roughening. The values given in the literature for the $(1\times2)\rightarrow(1\times1)$ order/disorder transition vary between 380 °C and 460 °C.[33] In our work the (1×1) reconstruction could not be verified by LEED, since the hot filament from the sample heater prevented the identification of any LEED pattern during annealing. However, RAS spectra could be recorded during annealing. The RAS spectra above 450 °C should be related to the disordered (1×1) phase. The spectra do not show an abrupt change in line shape as the temperature increases. Rather, the change observed in the RAS spectra indicates a gradual increase of surface disorder with temperature. The peaks are reduced in intensity and broaden at higher temperatures. At

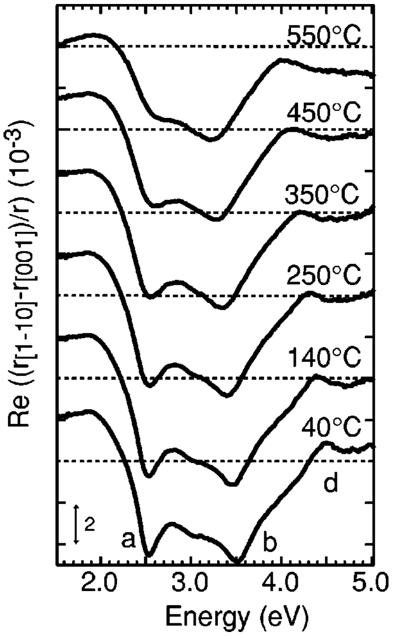

FIG. 3. RAS spectra of Au(110) at different temperatures. The spectra have been shifted on the *y* axis. Zero levels are indicated by the dashed lines.

550 °C the minimum at 2.5 eV and the positive anisotropy around 2 eV are nearly absent. This spectrum looks similar to the disordered Au(110)-K spectrum at room temperature in Fig. 1. The reduction of the anisotropy is therefore indicative of the disordered (1×1) phase.

Summarizing, the temperature dependence is in accordance with the interpretation of the potassium deposition experiments: In the spectral region around 2 eV, i.e., below the bulk *d*-band onset, optical transitions from surface states may contribute to the optical anisotropy of the ordered surface in addition to the Drude-like contribution due to an anisotropic surface conductivity.

B. ARUPS

As mentioned above, Cu, Ag, and Au have a similar electronic structure, but the surface structure of Au(110) differs from the other two due to its (1×2)reconstruction. On Cu and Ag(110), (1×1) surface states exist in the bulk L gaps. This gap also occurs in the band structure of gold. On Au(110) Heimann *et al.*[20] reported a corresponding surface state at $\bar{Y}$. The feature had a binding energy of about 0.1 eV. Another observation of this peak, achieved with a different excitation energy, supported the existence of the surface band on Au(110).[34] A more recent reinvestigation of Au(110), to the contrary, reported no surface states.[21] Also a first-principles calculation of the Au(110) surface, considering the (1×2) structure, did not reproduce these surface bands.[24] Inconsistent results were also reported from inverse photoemission. The early experimental identification of an unoccupied surface state at $\bar{Y}$ was later reassigned to an artifact by backfolding from other regions of the surface Brillouin zone.[23,25] More recently Smith *et al.*[26] have argued that the existence of surface states should be characteristic to the Au(110)(1×1) and (1×2) surfaces. According to the latter work the surface states on the (1×2) reconstructed surface will be modified such that both occupied and unoccupied surface states should exist at $\bar{Y}$.

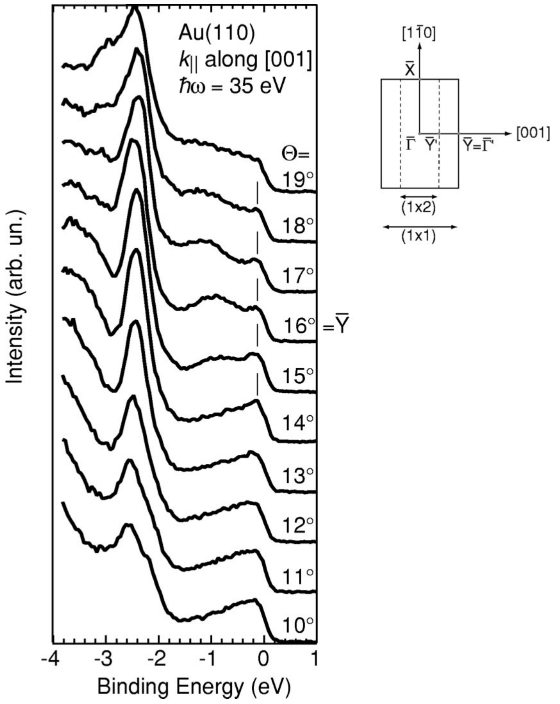

FIG. 4. Au(110) photoemission spectra in the vicinity of the $\bar{Y}$ point $(\Theta=16°)$. Additionally, the surface Brillouin zones for the (1×1) and the (2×1)structure are shown.

In order to address this issue we have performed photoemission experiments on the same surfaces as investigated by RAS. Figure 4 shows photoemission spectra of Au(110) (1×2) at room temperature for the clean surface around $\bar{Y}$. In a narrow range around the $\bar{Y}$ point, i.e., between 14° and 18°, there is indeed a small but distinct structure just below the Fermi level. The binding energy of the feature is between 0.1 and 0.2 eV. The disappearance of this structure for other emission angles could easily be explained by crossing the Fermi level since an upwards dispersion is expected for the surface state. Another property of a surface state is its independence of $k_{\perp}$. In Fig. 5, PES spectra for different photon energies at $\bar{Y}$ (left plot) and $\bar{\Gamma}$ (right plot) are shown. Indeed, the feature at $\bar{Y}$ is constantly visible for different photon energies. At $\bar{\Gamma}$ (right plot) we do not find a distinct peak of the Au(110)(2×1) surface structure as might be expected by backfolding from $\bar{Y}$. However, we would like to note that the backfolded surface state at $\bar{\Gamma}$ overlaps with bulk states and thus should rarely be observable, whereas at $\bar{Y}$ it falls into the L gap of the bulk bands.

An alternative explanation of the observed surface feature would be possible according to the observation of Bartynski *et al.*[23] They identified a surface feature occurring along the $\bar{\Gamma}$-$\bar{Y}'$ line in inverse photoemission experiments and associated it with an even surface resonance 0.2 eV above E_F

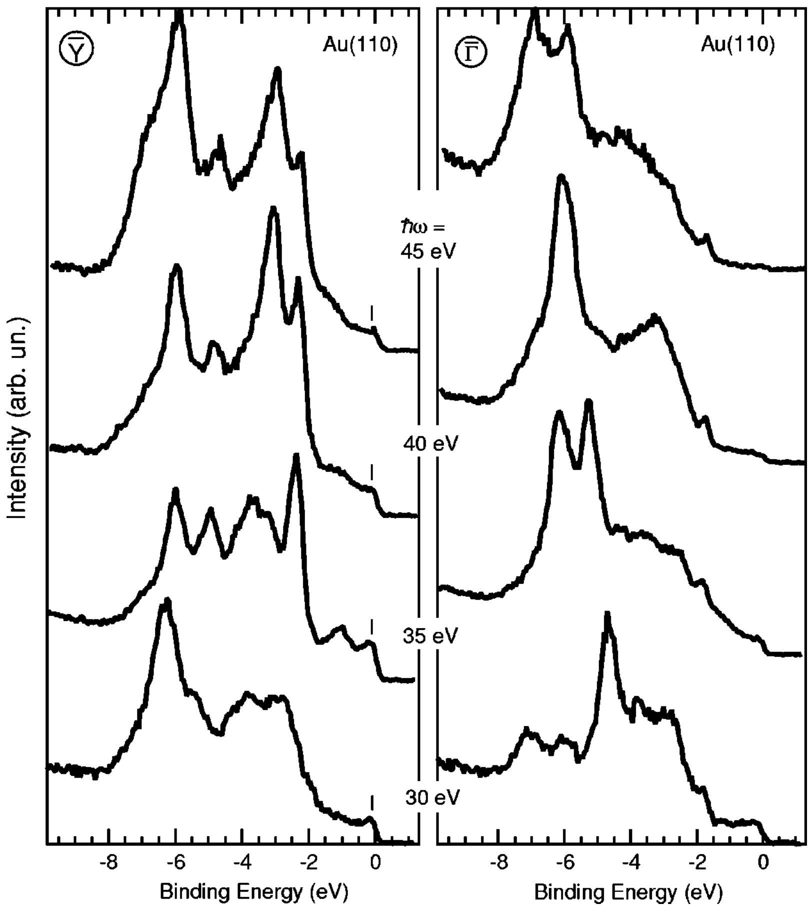

FIG. 5. Energy distribution curves from Au(110) obtained at different photon energies ($k_\perp$ scan). Left: at the $\bar{Y}$ symmetry point, right: at normal incidence.

which was revealed by band-structure calculations at $\bar{Y}$.[24] The same state should show up in photoemission as well if the linewidth of this resonance is larger than 0.2 eV. In this case, the peak found in the inverse photoemission experiment of Bartynski *et al.*[23] and the structure just below E_F found in our photoemission spectra at $\bar{Y}$ as well as in Refs. 20 and 34 would be assigned to a *p*-type surface resonance.[26]

A second empty surface band was consistently reported in inverse photoemission experiments at 2 eV above E_F along $\bar{\Gamma}$-$\bar{Y}'$. This state, in analogy to the surface band structure of Cu(110) and the Ag(110), was explained as an *s*-like Shockley state.[25,26]

Thus in accordance with the photoemission and inverse photoemission data, the small peak at 1.9 eV found in the RAS spectrum of the clean surface (Fig. 1) can indeed be interpreted as a transition between filled and empty surface states/resonances at the $\bar{Y}$ point located 0.1 eV below and 2 eV above E_F, respectively. The small difference of transition energy to the peak position in RAS would be accounted for by the finite resolution of the photoemission and in particular the inverse photoemission data. Due to the symmetry properties of the surface states or surface resonance the surface transition can only be excited with light polarized along [001]. Like in the case of Cu(110) and Ag(110), a positive peak would have to be expected in the RAS spectra which is indeed the case, as shown in Fig. 1.

There is a second mechanism involving surface states or resonances that can explain the feature at 1.9 eV found in the RAS. In a photoemission experiment, Hansen *et al.*[35] used a wide range of photon energies (to characterize bulk bands) and found a surface resonance 1.8 eV below the Fermi level at $\bar{\Gamma}$. This feature is also visible in our data as a small shoulder, see Fig. 5. It agrees with the first-principles calculations of Xu *et al.*,[24] who found a host of surface bands of *d*-type character located 1.8 eV or more below E_F. Thus, a transition between a *d*-type surface resonance below and the *p*-type surface state just above the Fermi level would match the selection rules and could account for the peak in the RAS. The second, empty surface state was found by Bartynski *et al.*[23] and discussed earlier. It also agrees with the theoretical work of Xu *et al.*[24]

Other interpretations of the photoemission data are discussed in the literature. Sastry *et al.*[21] conclude that the peak observed in ARUPS might be characteristic of disordered (1×1)surface regions, while the *well-ordered* Au(110)(1×2) surface would not exhibit any surface state. Photoemission experiments and investigations on the surface-plasmon dispersion on Ag(001) showed that the presence of a surface reconstruction changes the density of the outermost atomic layer, which causes an energetic shift of the surface states.[36,37] Moreover, it was suggested that the surface might contain (111) oriented facets giving rise to the surface state.[24] Based on our results we cannot completely rule out that the surface of a nominal (1×2) reconstructed Au(110) may be inhomogeneous to some extent. But we would like to note that our LEED images showed no signs of faceting or disorder.

IV. SUMMARY

We have investigated the surface optical properties of Au(110) using RAS. It could be shown that the main contribution to the surface dielectric anisotropy of Au(110) stems from transitions involving the bulk d and sp bands at the surface. A Drude contribution is found in the spectral range below the onset of the interband transitions.

On clean Au(110) a small structure at 1.9 eV is found in the RAS, which is absent on the adsorbate-covered surface. By comparison with photoemission data we assign this structure to a transition involving surface resonances or surface states. Corresponding surface states are identified by ARUPS at the $\overline{Y}$ point of the SBZ. We cannot resolve the question of whether the occupied surface state/resonance is characteristic for the Au(110)(1×2) surface structure or due to inhomogeneities on the surface. A second possibility is a transition involving surface states and resonances at the $\overline{\Gamma}$ point.

Moreover, we studied the temperature-induced disordering of Au(110) by RAS. A gradual change in the spectral line shape shows that there is no sharp order/disorder phase transition.

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