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Reflection anisotropy spectroscopy of clean and adsorbate-covered Ni(110) surfaces

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Abstract

The reflection anisotropy (RA) spectra from clean Ni(110), Na/Ni(110)(1 × 2), and CO/Ni(110) surfaces have been measured between 1.5 eV and 4.0 eV. Electronic transitions involving exchange-split d bands to an unoccupied surface state contribute a broad structure to the RA profile of Ni(110). The Na-induced surface reconstruction gives rise to a change in the RA profile that is related to changes in the unoccupied surface state. RA spectroscopy is found to be sensitive to the exposure of Ni(110) to CO.

1. Introduction

Reflection anisotropy spectroscopy (RAS) probes as a function of energy the response of a surface to linearly polarized light by measuring the difference in normal-incidence reflection of two in-plane orthogonal directions (Δr) normalized to the mean reflection (r). For cubic crystals of isotropic bulk optical properties, RAS achieves surface sensitivity by the cancellation of contributions involving the bulk, and the spectrum arises from the electronic structure of the surface. The reflection anisotropy (RA) is defined in terms of complex Fresnel reflection amplitudes [1] and for Ni(110) we define the RA as

$$\frac{\Delta r}{r} = \frac{2(r_{[1\bar{1}0]} - r_{[001]})}{r_{[1\bar{1}0]} + r_{[001]}} = \frac{2i\omega d}{c} \left(\frac{\tilde{\epsilon}_s^{[1\bar{1}0]}(\omega) - \tilde{\epsilon}_s^{[001]}(\omega)}{\tilde{\epsilon}_b(\omega) - 1} \right) \quad (1)$$

where $\hbar\omega$ is the photon energy, c is the speed of light, d is the surface layer thickness, and $\tilde{\epsilon}_s$ and $\tilde{\epsilon}_b$ are the surface and bulk dielectric functions, respectively.

RAS has been used to study polarization-dependent optical transitions in the noble metals Cu(110) [2–4], Ag(110) [5–7], Au(110) [8, 9] and the transition metal W(110) [10]. In each case, features observed in the RA spectrum of the clean surface have been identified with transitions involving surface electronic states. The ability to observe the behaviour of surface states is of importance, as surface states can be used as a monitor of the physical and chemical

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state of the surface [11]. The RAS technique has been used to monitor the response of surface states to reconstruction induced by molecular [2,3] and alkali metal [4] adsorption and also to temperature-induced disorder [12].

In this work we present the results of RA measurements of the Ni(110) surface, the Na-induced (1×2) surface reconstruction, and CO adsorption on Ni(110). The results indicate that the RAS profile of Ni(110) is sensitive to transitions between exchange-split d bands near E_F and an unoccupied surface state. We relate changes in the RA profile to known changes [13,14] in the unoccupied surface state accompanying the Na-induced surface reconstruction. To complement the RAS study of the reconstruction, we investigate the effect of molecular adsorption on the RA spectrum of Ni(110) without inducing a rearrangement of the Ni surface atoms by exposure of Ni(110) to CO.

2. Experimental procedure

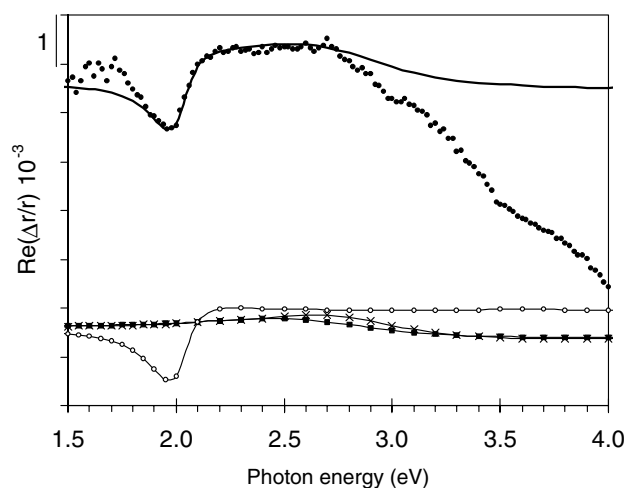
The experiments were carried out in an ultrahigh-vacuum (UHV) chamber with a base pressure in the 10^{-11} mbar region. The Ni(110) single-crystal specimen was aligned to 0.5° and mechanically polished to one-quarter-micron standard. A clean Ni(110) surface was prepared in UHV by cycles of Ar-ion bombardment ($10 \mu\text{A cm}^{-2}$, 1 kV, 300 K) and annealing to 1000 K. Surface order was confirmed by a sharp (1×1) low-energy electron diffraction (LEED) pattern and cleanliness was monitored using x-ray photoelectron spectroscopy (XPS). Na was deposited from a fully outgassed commercial dispenser onto the Ni(110) surface at room temperature and Na coverage was determined by LEED analysis with reference to published data [15–17] and with support from analysis of the relative intensities of the Na 1s and Ni 2p core-level photoelectron lines. We define Na coverage as the ratio of Na atoms to Ni atoms in the unreconstructed surface layer; i.e. $\theta = 1$ ML corresponds to 1.1×10^{15} Na atoms cm^{-2} . The clean Ni(110) surface was exposed to CO at room temperature for varying pressures and exposure times. CO coverage was determined by LEED analysis with reference to the work of Behm *et al* [18]. CO overlayer formation of the $c(4 \times 2)$ phase and the higher-coverage (2×1) $p2mg$ structure on the clean Ni(110) surface were achieved by varying the partial pressure of CO between 1×10^{-8} mbar and 1×10^{-6} mbar.

The RA spectrometer of the Aspnes design [19] projected and received light through a low-strain window on the UHV system. Experimental artifacts were removed from the spectra using a correction function obtained by measuring spectra with the sample in two orthogonal positions. Spectra of the real part of the complex RA were taken over a photon energy range of 1.5 eV to 4.0 eV.

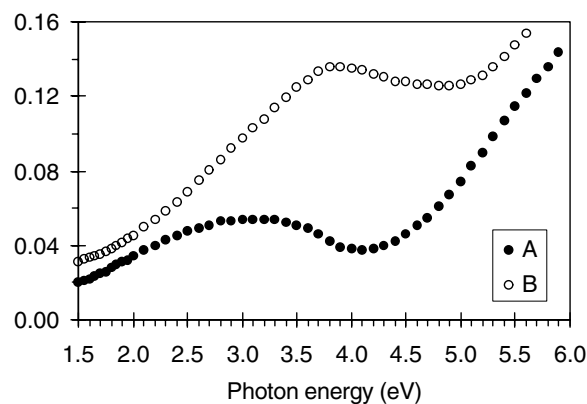
3. Results

3.1. The Ni(110) surface

The RA spectrum of the clean Ni(110)(1×1) surface is shown by the solid circles in figure 1(a). A broad spectral profile is observed with a relatively sharp valley located at 2.0 eV. A slight variability in the intensity of the valley at 2.0 eV was observed and we found indications that this variability was dependent upon the number of cleaning cycles and thus likely to be dependent upon surface order. The steady fall in the RA signal which begins at ~ 2.7 eV correlates with a steady decrease in the reflectivity r although the difference signal Δr in this region remained strong, negative, and significantly above the noise level as indicated by the low scatter in the data points in figure 1(a). The characteristic RA spectrum of Cu(110) [2–4]



(a)



(b)

Figure 1. (a) Experimental (dots) and simulated (solid line) RA profiles of Ni(110). The three components of the fit are shown with transition energies of 2.01 eV (open circles), 2.60 eV (crosses), and 2.80 eV (solid circles). (b) Calculated $A(\omega)$ and $B(\omega)$ functions related to the bulk dielectric function of Ni(110).

was obtained under the same experimental conditions and confirmed that the fall in the RA signal above 2.7 eV was a property of the Ni(110) surface.

3.2. The Na/Ni(110)(1 × 2) surface reconstruction

The effect on the RA profile of Ni(110) upon increasing Na deposition is shown in figure 2. For a surface exhibiting a LEED pattern characteristic of an Na coverage of $\theta \sim 0.05$ ML, no significant difference was observed in the RAS profile from that of the clean surface (figure 2(a)). A difference in the RAS response was observed for $\theta \sim 0.16$ ML in the form of a reduction in anisotropy over the photon energy region 2.0 eV to 4.0 eV (figure 2(b)). A (1 × 2) LEED pattern with well-defined streaks in the [001] direction at the positions ($\pm 1/2, 0$) was observed from the surface associated with the RA spectrum of figure 2(c). This LEED

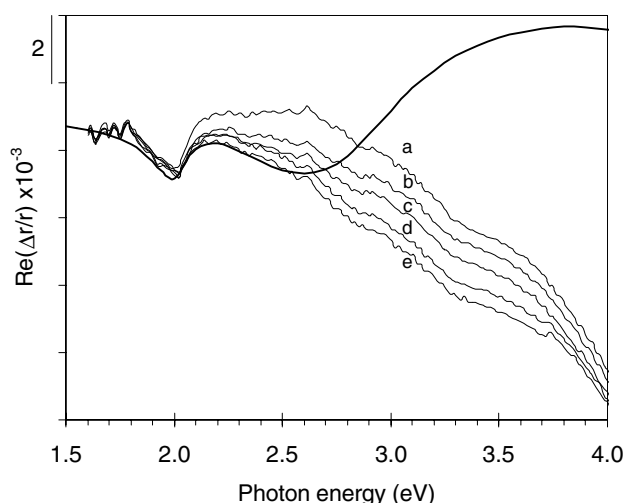


Figure 2. RA spectra of Na adsorption on Ni(110): (a) clean Ni(110), (b) 0.16 ML Na coverage, (c) 0.25 ML, (d) 0.47 ML, and (e) 0.66 ML. The simulated RA profile of 0.66 ML Na coverage is shown (thick line).

pattern indicates Na coverage of 0.25 ML and corresponds to a well-ordered (1×2) surface reconstruction that has been assigned to a (2×2) 1D structure [17] to reflect the nature of the Na overlayer. On the perfect reconstructed surface, the Na overlayer consists of rows of adatoms located in the missing-row troughs along the $[1\bar{1}0]$ direction [16, 17]. The streaks observed in the $\theta = 0.25$ ML LEED pattern arise from diffraction from the Na overlayer, in which the Na atoms possess some long-range order within each row and suffer discrete random positional shifts between adatom rows [15–17]. Increasing the Na coverage to 0.47 ML (figure 2(d)) and to 0.66 ML (figure 2(e)) produced further decreases in the RA signal. For all the coverages investigated, the spectrum at energies below 2.0 eV appeared insensitive to the developing reconstruction.

3.3. The adsorption of CO on Ni(110)

CO adsorption at room temperature on the Ni(110)(1×1) surface is known to result in several CO overlayer structures. We prepared the $c(4 \times 2)$ and the (2×1) $p2mg$ structures, the latter being formed at near-saturation coverage. Figure 3 shows the RA profile of the clean surface (curve a), the CO/Ni(110) $c(4 \times 2)$ surface (curve b), and the (2×1) $p2mg$ surface (curve c). As was found for the Na-induced reconstruction, the RA response above 2.0 eV was observed to change upon CO exposure with a decrease in RA intensity over a broad energy range. In contrast to the results of Na deposition, the RA response below 2.0 eV was found to change upon CO adsorption, with a greater difference observed from that of the clean surface for higher CO coverage (figure 3).

4. Discussion

To characterize the RA profile of clean Ni(110) (figure 1(a)) we model the system in terms of a Fresnel-based three-phase model [1, 20] of vacuum, biaxially anisotropic surface layer, and isotropic bulk. This model has been used to simulate the RAS response of a number of

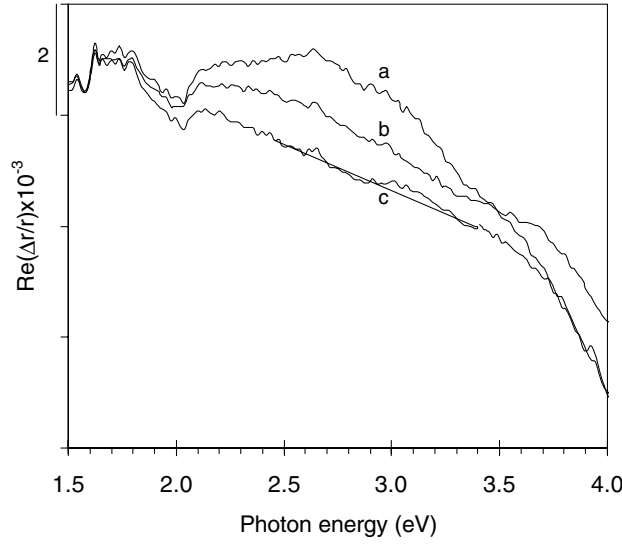


Figure 3. RA spectra of (a) clean Ni(110), (b) CO/Ni(110)c(4 × 2), and (c) CO/Ni(110)(2 × 1) p2mg.

surfaces [20]. The vacuum, surface, and bulk media have associated dielectric functions of unity, a surface dielectric anisotropy (SDA) defined as $\Delta\tilde{\epsilon}_s(\omega) = \tilde{\epsilon}_{[1\bar{1}0]}(\omega) - \tilde{\epsilon}_{[001]}(\omega)$, and $\tilde{\epsilon}_b(\omega)$ respectively. The bulk dielectric function $\tilde{\epsilon}_b(\omega)$ may be represented in terms of the functions $A(\omega)$ and $B(\omega)$ defined by

$$A(\omega) - iB(\omega) = \frac{1}{1 - \tilde{\epsilon}_b(\omega)}. \quad (2)$$

Bulk dielectric function, $\tilde{\epsilon}_b(\omega)$, data for Ni were obtained from experimentally determined tabulated data [21] and the resulting functions $A(\omega)$ and $B(\omega)$ are plotted in figure 1(b). The real part of (1) may be expressed in terms of $A(\omega)$ and $B(\omega)$ by

$$\text{Re}\left(\frac{\Delta r}{r}\right) = \frac{-2\omega d}{c} [A(\omega) \Delta\epsilon_s''(\omega) + B(\omega) \Delta\epsilon_s'(\omega)] \quad (3)$$

where $\Delta\epsilon_s'(\omega)$ and $\Delta\epsilon_s''(\omega)$ denote the real and imaginary parts of the SDA respectively. To simulate the experimental RA spectrum, a SDA is calculated from model parameters representing the energy ω_t , strength S , and linewidth Γ of transitions in the surface layer for light polarized along the [001] and [1 $\bar{1}$ 0] directions. Each transition has a Lorentzian form given by

$$\tilde{\epsilon}^{[ijk]} = 1 + \sum_{n=1}^m \frac{S_n/\pi}{\omega_{tn} - \omega + i\Gamma_n/2}. \quad (4)$$

To model the SDA we take note of inverse photoemission (IPE) studies of the unoccupied surface electronic structure of Ni(110) [22–25]. The results of these studies indicate that the bulk band gaps occurring at the \bar{X} and \bar{Y} points of the surface Brillouin zone (SBZ) contain surface states lying above E_F . We focus on the state located at 2.5 eV above E_F at \bar{Y} . This surface state has been found to exhibit spin splitting by ~ 0.2 eV [26]. Exchange-split bulk bands exist at \bar{Y} with a maximum density of states for minority-spin electrons at E_F and a maximum for majority-spin electrons ~ 250 meV below E_F [24]. Thus transitions from the

bulk d bands to the unoccupied surface state at \bar{Y} will have a range of energies and it is likely that such transitions give rise to the broad RA profile between 2 eV and 3 eV. The symmetry of the unoccupied surface state is predominantly p_z in character with respect to the surface layer atoms [25] and so dipole selection rules ($\Delta l = \pm 1$) are obeyed for such transitions.

Transitions involving the exchange-split d bands around E_F and the unoccupied surface state at 2.5 eV above E_F at \bar{Y} have been found to generate a strong resonance in magnetic second-harmonic generation (MSHG) spectra of Ni(110) [27]. The transitions occurring at \bar{Y} that result in the MSHG resonance range in energy from 2.6 eV to 2.8 eV [27]. On the basis of these results, we simulate the SDA using two transitions of energy 2.6 eV and 2.8 eV. In order to produce the best fit to the experimental RA profile we require both transitions to be generated by light polarized along the [001] direction. In addition to these transitions, we find it necessary to introduce a narrow transition at 2.01 eV for light polarized along $[1\bar{1}0]$ in order to reproduce the sharp valley in the RA profile in this region. The simulated RA spectrum is shown by the solid line in figure 1(a) and the contributions to the total spectrum made by each component are plotted separately below the main fit. The parameters of the fit are listed in table 1. We note that this simple model fails to account for the experimentally measured fall-off in signal beyond ~ 3 eV or for the response from 1.6 eV to 1.9 eV.

Table 1. The parameters used to calculate the fit displayed in figure 1. The error values are best estimates.

	Transition 1	Transition 2	Transition 3
Direction	$[1\bar{1}0]$	[001]	[001]
ω_i/eV	2.01 ± 0.01	2.60 ± 0.10	2.80 ± 0.10
Γ/eV	0.20 ± 0.01	1.00 ± 0.30	0.90 ± 0.20
S	0.40 ± 0.02	0.20 ± 0.06	0.20 ± 0.06

We now seek an explanation of the need to include a transition at 2.01 eV in our model of the RA spectrum. No structure is observed at ~ 2 eV in either the $A(\omega)$ or $B(\omega)$ functions (figure 1(b)) representing the bulk optical response. This suggests that the 2 eV feature is associated with the electronic structure of the surface. A comparison of IPE data [22] with band-structure calculations [28] suggests that a composite structure of bulk band and surface resonance exists at \bar{Y} close to the bulk band gap edge whose lower gap edge is located ~ 0.1 eV above E_F [13]. Such a transition between surface resonance and empty surface state would have an energy of ~ 2 eV—just what is required. However, the bulk band of the composite structure has sp-like character [23] and if the resonance also shares this symmetry character then dipole-allowed transitions into the empty p_z surface state at \bar{Y} would be unlikely.

In addition to the surface states discussed above, further electronic states have been found on the Ni(110) surface. The existence of up to three occupied surface states or resonances has been reported at the \bar{S} point [29, 30] lying between $\sim E_F$ and 0.4 eV below E_F . These states have predominantly d-like character [31] and it is unlikely that transitions from these surface states into unoccupied d bands at \bar{S} are involved in the RAS results since no bands are located at \bar{S} at the desired energy above E_F [22] and transitions between such states would be forbidden by dipole selection rules.

An alternative view of the origin of the 2.01 eV peak is that it arises from the surface local-field effect. The model, originally developed by Mochán and co-workers [32, 33], has been applied to Ag(110) and Cu(110) [34]. The SLFE model has been found to produce a good fit, particularly around 2 eV, to the RA spectrum of Cu(110) at high temperature where the surface state contribution is absent from the spectrum [12]. The model simulates the surface region by means of a lattice of polarizable cores, each with localized d electrons, and a screening Drude

electron gas describes the delocalized s–p electrons. The RA response results from a difference in surface conductivity, due to polarization processes in the presence of an oscillating electric field between the two principal directions in the surface plane. The model is well suited to the noble metals Ag and Cu; however, for Ni(110) modifications to the model may be necessary and we hope the experimental work presented here will stimulate interest in such a model.

4.1. The Na/Ni(110)(1 × 2) surface reconstruction

The behaviour of the unoccupied surface states of Ni(110) at \bar{Y} in response to Na-induced reconstruction has been investigated using IPE [13, 14]. With increasing Na deposition a significant reduction is observed in the intensity of the IPE peak associated with the surface state at ~ 2.5 eV above E_F . A shift in the surface state energy away from E_F towards the vacuum level is observed with the energy of the state residing at ~ 3.0 eV at a Na coverage of 0.3 ML. Upon further deposition, the state returns towards the original energy found on the clean surface.

The effect on the RA profile of Ni(110) upon increasing Na deposition is shown in figure 2. An Na coverage of $\theta \sim 0.05$ ML produced no significant difference in the RAS profile from that of the clean surface (figure 2(a)). A difference in the RAS response was observed for Na coverage $\theta \geq 0.16$ ML in the form of a reduction in anisotropy over the photon energy region 2.0 eV to 4.0 eV (figure 2(b)). The reduction in RA intensity with increasing Na coverage correlates with the strong attenuation of intensity of the surface state observed in IPE spectra [13, 14] and supports the identification of the RA spectral response in the 2.0 eV to 3.0 eV region of the clean Ni(110) surface with transitions that involve this state.

As a result of the broad RA profile of Ni(110) it is difficult to relate changes in the RAS upon Na adsorption to the energy-dependent behaviour of the surface state involved in the RAS transition. However, the Fresnel-based model used to simulate the clean Ni(110) surface (figure 1(a)) can reproduce some of the changes observed upon Na adsorption. The thick line in figure 2 is the simulated RA spectrum for 0.66 ML Na coverage (curve e).Suppressions of the transitions at 2.6 eV and 2.8 eV are not enough to reproduce the experimentally observed decrease in intensity above 2 eV for Na coverage greater than 0.16 ML. Instead, to simulate the spectrum in terms of our model it is necessary to change the symmetry of these two transitions into activation by light polarized in the $[1\bar{1}0]$ direction and increase their intensities to produce a simulated curve that follows the RA profile (e) up to ~ 2.6 eV, although above this energy the simulation diverges significantly from experiment. The parameters of the fit shown in figure 2 are listed in table 2. Upon Na adsorption, the symmetry of the surface state is expected to gain an appreciable p_y character [13] but, considering the nature of the RAS transitions from d orbitals to a p_y orbital, it is not clear how this would cause a RAS signal. The parities of the p_z and p_y states with respect to orthogonal planes perpendicular to the surface are the same for one such plane and opposite for the other. If, for example, a RAS signal exists due to one allowed and one disallowed transition to a p_z state for two orthogonal polarizations of the light, then

Table 2. Parameters used to calculate the fit displayed in figure 2. The error values are best estimates.

	Transition 1	Transition 2	Transition 3
Direction	$[1\bar{1}0]$	$[1\bar{1}0]$	$[1\bar{1}0]$
ω_i/eV	2.03 ± 0.01	2.60 ± 0.10	2.80 ± 0.10
Γ/eV	0.20 ± 0.03	1.00 ± 0.20	1.00 ± 0.10
S	0.40 ± 0.02	0.40 ± 0.06	1.60 ± 0.06

the transitions to a p_y state will be *either* both allowed *or* both disallowed. In either case, no RAS signal will be produced. The current situation is clearly unsatisfactory and highlights the need for further characterization, by photoemission and inverse photoemission, of the surface electronic structure of Ni(110).

4.2. The adsorption of CO on Ni(110)

The system CO/Ni is of interest in hydrocarbon synthesis and as such has been studied extensively by a variety of techniques. It is known that CO molecules are adsorbed on top of Ni atoms in the close-packed $[1\bar{1}0]$ rows. In the $c(4 \times 2)$ structure CO molecules have their molecular axis oriented perpendicular to the surface whereas in the higher-coverage (2×1) $p2mg$ phase the reduced intermolecular separations force a tilting of the molecular axis forming a zigzag structure, with CO alternately tilted away from the direction normal to the (001) plane [18, 35, 36]. Photoemission and inverse photoemission studies have observed changes in electronic structure upon CO adsorption. Angle-resolved photoemission [37] has been used to study the Ni d-band region upon CO exposure and transitions from states in this energy region could generate the observed RA spectrum. With increasing CO adsorption, emission from the d bands is rapidly attenuated and on the (2×1) $p2mg$ surface the Ni d-band emission is replaced by CO 2π -Ni 3d bands shifted to higher binding energy by ~ 1 eV [37]. The fall in RA observed above 2.0 eV with increasing CO coverage (figure 3) is consistent with our assignment of the RA response in this region to the Ni d bands.

In contrast to the Na/Ni(110)(1×2) reconstruction, the RA profile below 2.0 eV appears sensitive to CO adsorption.

5. Conclusions

RA spectra from the clean Ni(110), Na/Ni(110), and CO/Ni(110) surfaces have been measured between 1.5 eV and 4.0 eV. Electronic transitions involving exchange-split d bands to an unoccupied surface state contribute a broad structure to the RA profile of Ni(110). The Na-induced surface reconstruction gives rise to a change in the RA profile that is related to changes in the unoccupied surface state. RAS is found to be sensitive to the exposure of Ni(110) to CO. Simulations of the RA of Ni(110) and Na/Ni(110) show some success and highlight the need for further characterization of the surface electronic structure of Ni(110).

Acknowledgments

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