

## Short contribution

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### The behaviour of secondary electrons from Ni (110) upon oxygen adsorption

To establish the cleanliness of nickel surfaces in an investigation of the interaction with oxygen we used Auger electron spectroscopy<sup>1</sup>. It is shown from the literature that AES spectra of a clean surface of several metals show a number of peaks in the secondary electron energy spectra in the range 100–400 eV, which cannot be associated with transitions in atoms at the solid surface. Becker and Hagstrum<sup>2</sup> measured these peaks on the Ni(100) surface at 121, 140, 173, 204, 241, 290 and 388 eV. McDonnell *et al*<sup>3</sup> previously reported analogous peaks on copper and cobalt. The position of the peaks appeared to be independent of the crystallographic orientation of the metal studied, but showed a regular shift over the whole range for different metals. McDonnell *et al* denoted these peaks as Temperature Dependent Peaks (TDP's) because they noted a strong dependence of the peak-heights on the sample temperature. From the temperature dependence they deduced that the phenomenon must be due to diffractions of secondary electrons leaving the sample. The surface ordering is hence directly correlated to the peak-height. Other authors have also reported on the appearance of such peaks<sup>4–7</sup>.

The AES spectra, recorded during the different stages of the cleaning procedure of a Ni(110) surface are represented in Figure 1. The different stages are described in the caption. Spectrum (e) shows the TDP's as reported by Becker *et al*<sup>2</sup>. The reason for a higher number of peaks is probably due to a higher sensitivity of our apparatus.

In this letter we report the change in the heights of the TDP's measured during the initial oxidation process of a Ni(110) surface. After cleaning the sample to a level below the Auger detection limit (at 740 K), the oxygen coverage of the sample was raised in small exposure steps ( $\sim 10^{-8}$  torr oxygen pressure during 10–15 s) till a saturation coverage was reached.

In Figure 2 the normalized peak-heights of the three most distinct TDP's  $h_X/h_{Ni,853\text{ eV}}$  where X is 141, 175 or 197 eV, are plotted versus the normalized oxygen peak-height,  $h_{O,515\text{ eV}}/h_{Ni,853\text{ eV}}$ . The latter is a measure for the amount of oxygen adsorbed at the surface, where  $h_{Ni,853\text{ eV}}$  is the height of the major nickel peak, which hardly varies on initial oxygen exposure.

In contrast with a contaminated surface where the TDP's are absent, the diffraction peaks, measured during the initial oxidation of a nickel surface, show a total different behaviour. The recorded peak-heights initially reduce in size after which they increase again. The behaviour of the peaks plotted in Figure 2 can be explained, as McDonnell *et al*<sup>3</sup> did for the temperature dependence of the peak-heights, by changes in the surface ordering. The differences in surface ordering can be derived from

the two stage model during initial oxidation of nickel, given by Mitchell *et al*<sup>8</sup>. A non-activated chemisorption is followed by nucleation of an oxide displaying a saturation coverage of two monolayers, where an ordered NiO layer is formed. The decrease in peak-heights is caused by a random chemisorption of oxygen during the first stage, the subsequent increase is due to the formation of an ordered NiO layer. The peaks at 141 and 197 eV are zero at an oxygen coverage of about 25% of the saturation coverage of the second stage of oxidation. Mitchell *et al*<sup>8</sup> as well as

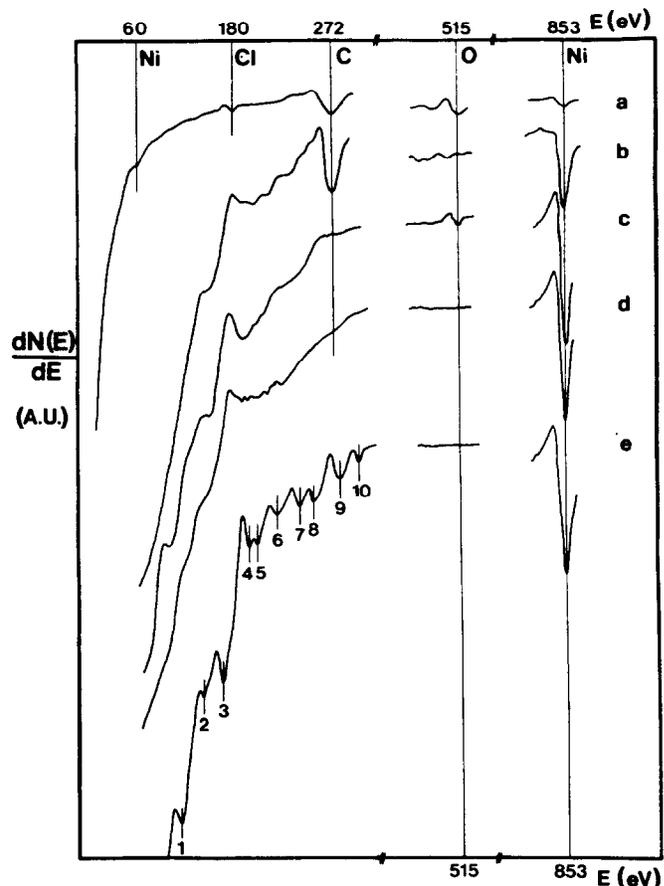


Figure 1. Secondary electron emission spectra from Ni(110). Spectrum (a) is recorded after mounting the sample, spectrum (b) after argon ion bombardment ( $6 \times 10^{-5}$  torr, 1 KeV, 2.5 h), spectrum (c) after the cycle oxygen exposure ( $10^{-6}$  torr, 0.5 min), evacuating, annealing (740 K, 0.5 h) and cooling to room temperature, spectrum (d) at 740 K after argon ion bombardment ( $6 \times 10^{-5}$  torr, 600 eV, 0.5 h) and heating (740 K, 0.5 h) and spectrum (e) after cooling to room temperature. The numbers 1–10 indicate the energies 141, 163, 175, 197, 204, 224, 239, 255, 277 and 293 eV.

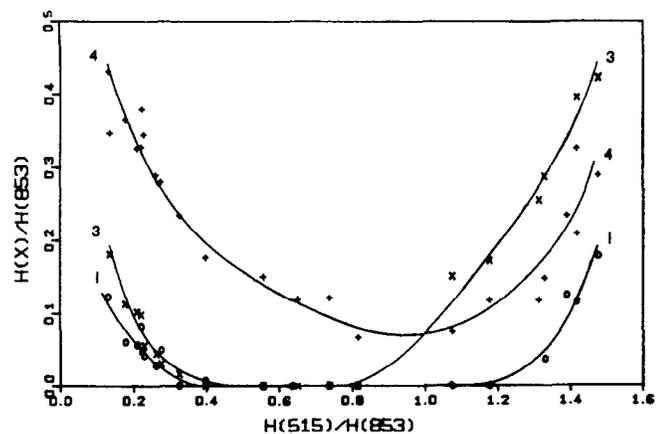


Figure 2.  $h_X/h_{Ni, 853 eV}$  vs  $h_{O, 515 eV}/h_{Ni, 853 eV}$  where X has the energy of 141(○), 175(+) or 197 eV(×). The numbers in the figure refer to the peaks in Figure 1, curve (e).

Smcenk *et al*<sup>9</sup> measured a same percentage of oxygen coverage at the end of the first stage of oxidation. Though not so extreme the peak at 197 eV shows the same tendency.

### References

- <sup>1</sup> L J Hanekamp, S J H Brader and G A Bootsma, *Surface Sci*, **135**, 383 (1983).
- <sup>2</sup> G E Becker and H D Hagstrum, *J Vac Sci Technol*, **11**, 284 (1974).
- <sup>3</sup> L McDonnell, B D Powell and D P Woodruff, *Surface Sci*, **40**, 669 (1973).
- <sup>4</sup> M A Chesters and J Pritchard, *Surface Sci*, **28**, 460 (1971).
- <sup>5</sup> E G McRae, *Surface Sci*, **44**, 321 (1974).
- <sup>6</sup> L McDonnell and D P Woodruff, *Surface Sci*, **46**, 505 (1974).
- <sup>7</sup> P A Hofmann, Thesis, Technische Universität München, Munich (1979).
- <sup>8</sup> D F Mitchell, P B Sewell and M Cohen, *Surface Sci*, **69**, 310 (1977).
- <sup>9</sup> R G Smeenk, R M Tromp, N F van der Veen and F W Saris, *Surface Sci*, **95**, 156 (1980).

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