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EVIDENCE FOR PSEUDO BRIDGE BONDING OF c(2x2)-O on Ni(100)

by

J. E. Demuth, N. J. DiNardo, and G. S. Cargill III

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Evidence for Pseudo Bridge Bonding of  $c(2 \times 2)$ -O on Ni(100)

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(Received 24 February 1983)

New low-energy electron-diffraction intensity measurements and dynamical calculations indicate that oxygen in a  $c(2 \times 2)$  overlayer resides  $0.80 \pm 0.025 \text{ \AA}$  above the top layer of Ni atoms and is displaced  $0.30 \pm 0.1 \text{ \AA}$  from the fourfold hollow position along a  $\langle 110 \rangle$  direction. This produces a structure having  $C_2$  symmetry with two inequivalent nearest-neighbor Ni-O bond distances of  $1.75 \pm 0.05$  and  $2.14 \pm 0.08 \text{ \AA}$ .

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The chemical nature of chemisorbed oxygen on metal surfaces is an important fundamental question which has recently attracted considerable attention and controversy. In particular, Upton and Goddard<sup>1</sup> have proposed an "oxide state" for the  $c(2 \times 2)$ -oxygen phase on Ni(100) which has an unusually short Ni-O bond distance ( $d_{\text{Ni-O}} = 1.78 \text{ \AA}$ ).<sup>2</sup> Their results also provided a natural interpretation for the unusually low oxygen-metal vibrational frequency<sup>2</sup> as well as a detailed calculation and agreement with the low-frequency phonon spectrum.<sup>3</sup> However, the Ni-O bond length of this new phase was in contradiction to an early low-energy electron-diffraction (LEED) structural analysis<sup>4</sup> ( $d_{\text{M-O}} = 1.97 \pm 0.05 \text{ \AA}$ ) as well as a more recent surface extended x-ray absorption fine structure (SEXAFS) analysis<sup>5</sup> ( $d_{\text{M-O}} = 1.96 \pm 0.03 \text{ \AA}$ ). Photoelectron diffraction studies<sup>6</sup> and recent LEED calculations<sup>7</sup> offered support for a short-bond-length "oxide" species, while all results to date having only considered high-symmetry sites have favored a fourfold hollow bond site.

We have performed new LEED intensity measurements and calculations which provide evidence that the fourfold hollow site is a "saddle point" and that a new, low-symmetry bonding site is preferred. This new structure has oxygen displaced  $\sim 0.3 \text{ \AA}$  from the fourfold hollow site along the  $\langle 110 \rangle$  direction and implies pseudo bridge bonding of oxygen to two neighboring nickel atoms with  $d_{\text{Ni-O}} = 1.75$ . We also show that this structure is consistent with recent SEXAFS measurements.

These experiments were performed in an ion-pumped UHV vacuum chamber (base pressure  $6 \times 10^{-11}$  Torr) with facilities for sample sputter cleaning and LEED, Auger, high-resolution electron energy-loss (EELS), and uv photoemission spectroscopies. Surface cleanliness and relative oxygen coverages were monitored by Auger electron spectroscopy while LEED intensity meas-

urements and EELS were used to isolate and characterize the  $c(2 \times 2)$  structure. The purest, most well ordered  $c(2 \times 2)$  structures were prepared by exposures at  $2 \times 10^{-8}$  Torr with the sample at  $150 \text{ }^\circ\text{C}$  or slightly higher followed by momentary heating to  $250 \text{ }^\circ\text{C}$  for a total of 25 L (langmuir).<sup>8</sup> EELS revealed no oxide vibrations nor are oxide islands visible with LEED. AES indicates a concentration of oxygen  $\sim 1.8$ -2 times that of the "best"  $p(2 \times 2)$  structure produced by a 1.2-L exposure and momentary heating.

LEED measurements were performed with the sample at  $T \approx 160 \text{ K}$  by use of a magnetically shielded, four-grid Varian LEED optics and a Dataquire video LEED detection system. Intensity-energy,  $I(E)$ , measurements were made between 20 and 260 eV for the  $11$ ,  $10$ ,  $\frac{1}{2}\frac{1}{2}$ , and  $\frac{1}{2}\frac{1}{2}$  beams at normal incidence ( $\theta = 0$ ) as well as the  $00$  and  $\frac{1}{2}\frac{1}{2}$  beams for nonnormal incidence ( $\theta = 5^\circ$  and  $10^\circ$ ) and were all considered in this analysis. LEED  $I(E)$  calculations were performed with the layer-Korringa-Kohn-Rostoker method<sup>9</sup> and the bulk Ni parameters established from a previous study.<sup>10</sup> All final calculations used eight phase shifts and a top layer containing both Ni and oxygen with 58, 90, and 138 beams considered above 60, 128, and 196 eV, respectively. Geometry searches were generally done in  $0.05\text{-\AA}$  steps. Several oxygen scattering potentials were investigated: the original atomic superposition potential,<sup>4</sup> an  $X\text{-}\alpha$  potential for NiO,<sup>11</sup> and a self-consistent-field potential for bulk NiO.<sup>12</sup> All gave nearly identical spectra, with the self-consistent-field NiO potential only marginally better. Ni and O mean square vibrational amplitudes ( $\sigma^2$ ) of  $0.009 \text{ \AA}$  were initially used and later changed to  $0.009$  and  $0.012$ , respectively, as determined from temperature-dependent measurements.

In Fig. 1 we show a comparison of calculated spectra to our experimental spectra for the  $\frac{1}{2}\frac{1}{2}$

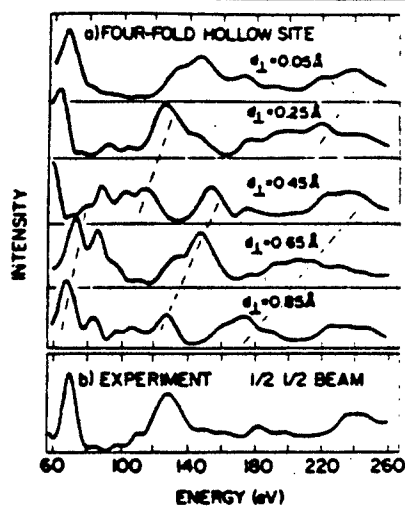


FIG. 1. Calculated and measured  $\frac{1}{2}$ - $\frac{1}{2}$ -beam LEED intensity-energy spectra of  $c(2 \times 2)$ -O on Ni(100) at  $T = 160$  K for different vertical heights of oxygen ( $d_1$ ) above the fourfold hollow. All calculations have a subsurface expansion of  $0.05 \text{ \AA}$  between the first and second nickel layers. The dashed lines in (a) indicate the dispersion of peak positions with  $d_1$  as determined from smaller  $d_1$  variations.

beam. As a result of the greater sensitivity of the calculated spectra to uncertainties in the surface barrier and scattering potentials below  $60 \text{ eV}$ ,<sup>13</sup> we consider results only above  $60 \text{ eV}$ . The calculations (a) represent oxygen bonded in the fourfold hollow site at the indicated vertical displacement  $d_1$  above the top Ni atoms. The best agreement for oxygen in a fourfold hollow site occurs for  $d_1 = 0.85 \pm 0.05$  where a relatively flat minimum in the  $R$  factor<sup>14</sup> of  $0.24$  is found for the fractional-order beams. We note that the agreement we observe in Fig. 1 for  $d_1 = 0.05$ – $0.25 \text{ \AA}$  appears to be due to a multiple-scattering coincidence,<sup>15</sup> and does not occur for several other beams. However, in view of this we have considered other nearly coplanar ( $-0.15 \leq d_1 \leq 0.35$ ) oxygen/nickel structures with a wide range of substrate atom distortions ( $-0.1$  to  $+0.15 \text{ \AA}$ ); reconstruction structures with oxygen atoms replacing every other Ni atom in the top layer; and mixed layers having interstitial oxygen atoms between the first and second layers of the normal Ni lattice or with a top Ni layer reconfigured to give  $d_{\text{Ni-O}} = 1.95 \text{ \AA}$ . These all proved unsatisfactory ( $R > 0.3$ ).

In our geometry search we have found markedly improved agreement if the oxygen is displaced

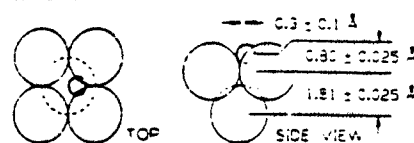


FIG. 2. The location of oxygen (small circle) in the asymmetric bonding site.

off the fourfold hollow site along a  $\langle 110 \rangle$  direction as shown for the optimized structure in Fig. 2. The best overall agreement we have achieved occurs for this asymmetric bond site which is shown in Fig. 3 and compared to the best agreement for the fourfold hollow position. We note that the relatively large uncertainty in the parallel displacement of  $\pm 0.1 \text{ \AA}$  occurs because some beams favor  $0.2 \text{ \AA}$  while others  $0.4 \text{ \AA}$ . This as well as more detailed analysis of  $\sigma^2$  for O suggests strong anisotropic vibrational motion and/or some small residual static disorder for oxygen in the  $c(2 \times 2)$  mesh.

The improvement in agreement for the asymmetric site over a fourfold hollow site occurs for all our data and appears to be independent of

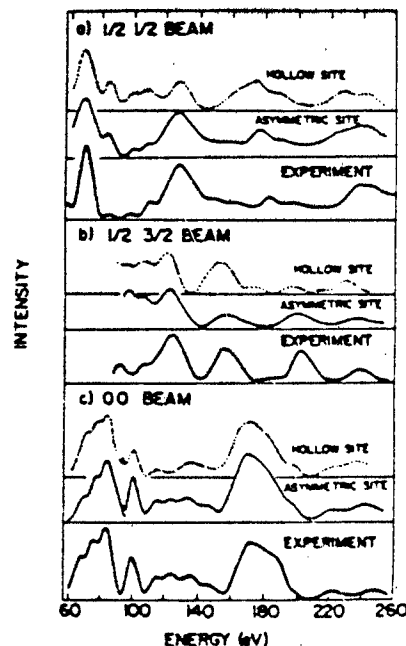


FIG. 3. Comparison of calculated and experimental fractional-order beam spectra for the asymmetric site shown in Fig. 2 and a fourfold hollow site (dotted curve) with  $d_1 = 0.85 \text{ \AA}$  and a subsurface expansion of  $0.05 \text{ \AA}$ . Both rational-order ( $\theta = 0$ ) and a 00 beam spectra ( $\theta = 10^\circ$ ,  $\phi = 45^\circ$ ) are shown.

the detailed choice of Debye temperature, electron damping, oxygen scattering potential, or inner potential,  $V_0$ . For a constant  $V_0$  determined from our clean-surface results, the fractional-order beam  $R$  factors are  $\sim 0.07$  better for the asymmetric site than our best fourfold hollow site. The integral-order beams produce agreement comparable to that achieved in Fig. 3 for the asymmetric structure but show weaker changes and are less sensitive to the location of oxygen. The agreement we achieve over a substantial energy range and for a large number of beams supersedes any previous LEED study of this system and strongly argues for this new structure.

The new structure we determine produces two nearest-neighbor Ni-O bond lengths of  $1.75 \pm 0.05$  Å and  $2.14 \pm 0.08$  Å, and contradicts the single bond length of  $1.96 \pm 0.04$  Å proposed from recent SEXAFS measurements.<sup>5</sup> In investigating the sensitivity of SEXAFS to such structural differences, we have found that the SEXAFS signal  $k\chi(k)$  for a two-bond-length structure can be nearly identical to the single-bond-length structure over a limited energy range when the zero of the energy scale is chosen as described by Stöhr, Jaeger, and Brennan.<sup>16</sup> Examples of  $k\chi(k)$  for one-bond-length (1.96 Å) and two-bond-length (1.88 and 2.36 Å) structures, shown as insets in Fig. 4(b), were calculated with Teo and Lee backscattering amplitudes<sup>17</sup> and the phase shifts empirically determined from bulk crystalline NiO.<sup>5</sup> The  $\sim 9\%$  larger Ni-O distances for the two-bond-length structure compared with those determined in our LEED analysis are required to match the experimental  $k\chi(k)$  spectra and may reflect the inadequacy of the bulk Ni-O phase shifts which differ significantly from those calculated.<sup>17</sup> For the two-bond-length structure the beats in  $k\chi(k)$  which distinguish it from the single-bond-length structure occur outside the experimentally accessible range. As shown in Fig. 4, the Fourier transform of  $k\chi(k)$  for either structure over the experimental range produces a single peak in  $|f(r)|$ . Polarization effects for the two structures are nearly identical and both are consistent with the experimental SEXAFS results. Thus, given the possible uncertainties in the Ni-O phase shifts, the SEXAFS results cannot rule out our  $C_{2v}$  symmetry, two-bond-length structure.

The structure we determine indicates a relatively short NiO bond distance and an unusual local bonding geometry relative to NiO.<sup>18</sup> At the sur-

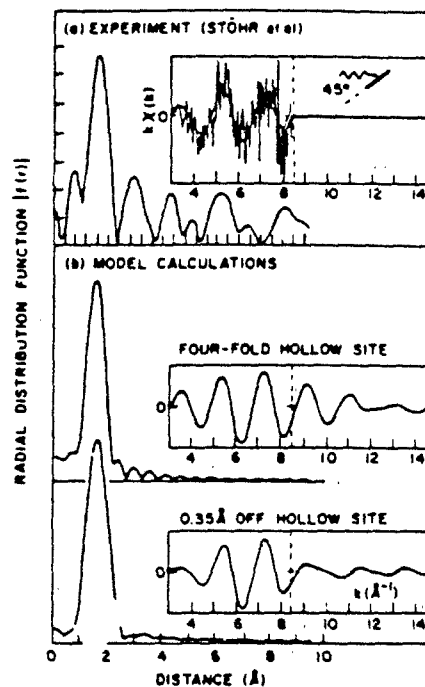


FIG. 4. Comparison of (a) experimental (Ref. 5) and (b) model calculations of the radial distribution function,  $|f(r)|$ , and the SEXAFS signal,  $k\chi(k)$ . The calculated results are for the single-bond-length structure with oxygen in a fourfold hollow site ( $d_1 = 0.85$  Å) and for a two-bond-length structure as described in the text. (The polarization is indicated in the experiment.)

face, the absence of octahedral symmetry as occurs in the oxide, lateral adsorbate interactions, or a tendency to form a  $Ni_2O$ -like surface compound may allow preferential bonding of oxygen to two Ni atoms. Although Upton and Goddard's original "oxide" bond distance of 1.78 Å (Ref. 1) is consistent with our result, more recent calculations (again for oxygen in the fourfold hollow site) suggest a longer bond length of  $\sim 1.89$  Å.<sup>19</sup> If this latter result is correct, our asymmetric bond sites may reflect a Jahn-Teller distortion in chemisorption bonding. However, we also note that a low-oxygen-density compound,  $Ni_3O$ , is reported with  $d_{NiO} = 1.80$  Å (Ref. 18) which is consistent with the bond length we find.

Finally, we briefly mention some further experimental evidence which supports a  $C_{2v}$  site symmetry. Our angle/energy-dependent EELS measurements show a weak, dipole-excited loss at 53 meV for the  $c(2 \times 2)$  structure which can be associated with a nearly parallel vibrational

mode.<sup>20</sup> Our uv photoemission results also show two broad O<sub>2s</sub> peaks at ~5 and 6 eV which from more complete angle- and polarization-dependent studies<sup>21</sup> are consistent with our proposed bonding geometry. Finally, domain-boundary misfits for this low-symmetry structure can account for the difficulty of c(2×2)-O to yield a stoichiometric coverage relative to the p(2×2) unless heated when dosed.

In summary, we have performed new LEED measurements and calculations which indicate that oxygen in a c(2×2) ordered array on Ni(100) has a C<sub>2v</sub> site symmetry, is pseudo bridge bonded to two Ni atoms, and has a relatively short nearest-neighbor NiO bond length.

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