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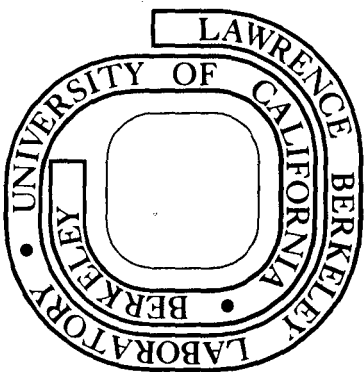
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A LEED-AES STUDY OF THE RECONSTRUCTED Pt(110) SURFACE
AND THE EFFECT OF OXYGEN TREATMENT

by

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Abstract

The Pt(110) crystal face has been studied by low energy electron diffraction (LEED) and Auger electron spectroscopy (AES). The stable reconstructed (2x1) surface structure transforms, reversibly, to the (1x1) structure upon heating in vacuum to $830 \pm 30^\circ\text{C}$. Ion bombardment at 25°C also stabilizes the metastable (1x1) surface structure which converts, irreversibly, to the stable (2x1) structure upon heating in vacuum at $\leq 500^\circ\text{C}$. The diffraction beam intensity vs. electron energy curves ($I(hk)$ vs. eV) exhibit complex behavior when monitored as a function of temperature. Heat treatments in oxygen at high crystal temperatures ($>800^\circ\text{C}$) result in the appearance of new (3x1), (5x1) and ("7x1") surface structures while oxygen remains undetectable by AES. Adsorbed CO causes the (2x1) \rightarrow (1x1) transformation while it does not effect the (3x1) surface structure.

1. Introduction

The nature of reconstruction of the (110) and (100) crystal faces of Pt, Au and Ir has been the subject of many experimental investigations.¹⁻¹⁴ It has been established that (a) impurities are not present on the reconstructed surfaces within the limit of detection of AES, 0.1%-1% of a monolayer and (b) under proper conditions the metastable (1x1) structure may be stabilized that, upon heating, converts irreversibly to the more stable reconstructed surface structure.

The reproducible presence of the reconstructed surface structures without detectable surface impurities indicate that they are the properties of the noble metal crystal faces. However, the role of impurities that are located below the surface in stabilizing or destabilizing the surface reconstruction is less clear. Since the concentration of oxygen, hydrogen or carbon below the topmost layer of atoms can only be detected with poor sensitivity and with utmost difficulty by the various surface diagnostic techniques, the role of these impurities in influencing the surface phase transformations cannot be ascertained readily.

The purpose of this paper is to investigate in some detail the properties of the Pt(110)-(2x1) surface structure as a function of temperature and heat treatments in oxygen. The diffraction beam intensities as a function of temperature, exhibit complex behavior indicating that several structural rearrangements occur

while the (2x1) surface unit cell remains unaltered. A metastable (1x1) surface structure can be produced by ion bombardment. Oxygen located in the near surface region but undetectable by AES induces the formation of new surface structures ((3x1), 5x1), and ("7x1")). These have been detected occasionally by other authors.^{15,16}

2. Experimental

The experiments were performed in a UHV system equipped with low energy electron diffraction (LEED) and Auger electron spectroscopy (AES). This apparatus is currently being used for molecular beam experiments and has already been described in detail.¹⁷ The platinum crystal has been spark cut from a Pt rod of 99.999% purity. Prior to insertion in vacuum the crystal was polished by the standard metallographic techniques. In the course of our experimental work this crystal has been repolished several times. In some cases, a final etching in aqua regia was performed. These treatments, however, had no detectable effect on the behavior of the crystal. The crystal specimen were mounted on a Ta foil that served to heat the sample from room temperature (25°C) up to ~1500°C. The crystal temperatures were measured by an infrared pyrometer which was previously calibrated with a thermocouple spot-welded to a Pt crystal. The accuracy in the temperatures is of $\pm 30^\circ\text{C}$. In the LEED studies the intensities of the diffraction beams were measured by means of a spot-photometer. The composition of the background gases was monitored with a quadrupole mass spectrometer. The main components were, normally, H₂ and

CO, while H₂O, CH₄, Ar and CO₂ were also detectable at a total pressure of 10⁻⁹ torr.

3. Results

3.1. The Thermal Stability of the (2x1) Surface Structure.

The commonly observed surface structure of the clean Pt(110) is the (2x1). It was obtained after removing carbon from the platinum surface by oxygen at $\sim 700^\circ\text{C}$ and 10⁻⁷-10⁻⁶ torr in approximately 10 minutes or just the time it takes to remove the carbon as monitored by AES. We shall call this a mild O₂ treatment. The importance of not overdosing the crystal with O₂ will be made clear in Section 3.2. Also Ar ion sputtering was used to remove small amounts of Ca, followed by annealing to $\sim 800^\circ\text{C}$, which produced again the (2x1) structure. This surface structure is observed in surfaces which do not show any contaminant in the Auger spectrum.

Closer examination of the variation of the diffraction beam intensities as the crystal temperature is changed, reveals complex changes (which we shall examine below) and finally, at 830 $^\circ\pm$ 30 $^\circ\text{C}$ the half-order spots disappear completely giving rise to a (1x1) structure. At certain electron energies the transition to the (1x1) structure is accompanied by an increase in the intensity of some of the integral order spots. The complete disappearance of the half-order spots is also preceded by streaking along the [001] direction as the temperature is increased. This transition is illustrated in

the series of pictures of Fig.1.

Cooling the crystal below 830°C restores the (2x1) structure, while reheating again yields the (1x1) structure. The reproducibility of this structural transformation during several heating and cooling cycles indicates the complete reversibility of the process.

The intensities of the various diffraction beams were measured as a function of crystal temperature. These are shown in Fig.2 at an electron energy of 41.5 eV. The behavior of the various diffraction beams is quite complex, with various breaks appearing in the $\log I(hk)$ vs. T_s curves. The breaks occur around 250°C, 450°C, 660°C, 720°C and 770°C. At 720°C some of the diffraction beams show a sharp intensity minimum and finally at $\sim 830^\circ\text{C}$ the half-order spots disappear as mentioned previously. This complex $I(hk)$ vs. T behavior was also observed at many other electron energies. In order to check that it was not due to any artifact in the measurement we have repeated the experiment with diffraction beams from a Pt(332) crystal which was mounted on the same manipulator. In that case, the $\log I$ vs T curves were straight lines confirming that the effect observed on the (110) crystal was intrinsic to that surface.

3.2. The Effect of Heat Treatments in Oxygen.

The heat treatment of the crystal in oxygen (a procedure used routinely in many laboratories to remove carbon may have important consequences which were particularly detectable in the Pt(110) crystal surface. Following a heavier oxygen heat treatment ($\text{PO}_2 \gtrsim 10^{-6}$ torr, and crystal temperatures above 800°C for 1/2 hour or more) a

(3x1) surface structure appeared (Fig.3(B)). After this oxygen treatment the crystal was flashed to high temperature ($\sim 1000^\circ\text{C}$) so that no trace of oxygen or any other impurity could be detected by AES. This (3x1) structure is stable in the same temperature range as the (2x1) structure and it undergoes a transition to the (1x1) at $T \sim 830^\circ\text{C} \pm 30^\circ\text{C}$. This transition is illustrated in the series of diffraction patterns in Fig.4. Also as with the (2x1) \leftrightarrow (1x1) transition was observed to be completely reversible.

After the initial heavier oxygen heat treatment, O_2 was no longer used to clean the crystal. The Pt surface was cleaned only by Ar sputtering to avoid a further increase of the amount of oxygen dissolved into the Pt crystal. For days, however, the (3x1) pattern persisted as the clean surface structure. Many attempts to restore the original (2x1) pattern were unsuccessful. These included high-temperature annealing (up to 1200°C), Ar ion sputtering and high-temperature heat treatment in hydrogen.

However, after many days of continuous exposure to background gases, with long periods of annealing at an average temperature of $\sim 400^\circ\text{C}$, the LEED pattern showed streaks in the [001] direction and the appearance of (2x1) regions in a mostly (3x1) surface. There are other regions on the crystal surface that exhibit (5x1) and ("7x1") surface structures, as shown in Fig.3(C) and (D). These latter structures, however, were never observed to cover the whole crystal surface and always coexisted with the (3x1) and sometimes with the (2x1) structures. When the crystal was heated above 800°C the new structures and also the (2x1) regions, if present,

transformed to the (1x1) but not reversibly this time; upon cooling the whole surface was again covered with the (3x1) pattern. The appearance of (2x1), (5x1) and ("7x1") patches was also commonly observed after prolonged exposures to the background gases at 25° or 400°C. All of these patterns could be observed while the platinum surface was clean as judged by AES. The ("7x1") pattern is probably not a (7x1) surface structure because the central doublet and the two barely visible beams beside the integral spots are not perfectly aligned along the [001] direction. Also, only the two central spots are clearly visible over the whole electron energy range until at temperatures approaching 800°C, they became a continuous streak that disappeared at 830°C \pm 30°C.

After extended heating in vacuum the (2x1) surface structure could be easily produced again and persisted even after a short heating to 800°C. In this last stage the (3x1) pattern could only be observed after a high-temperature treatment of up to 1000° to 1200°C.

3.3 The Effect of Various Adsorbates on the Surface Structures

Common to the observations made by many authors that the adsorption of certain impurities on an initially clean (2x1) surface stabilizes the (1x1) surface structure. We have seen this with carbon and carbon monoxide. Particularly striking is the adsorption of CO on the two structures: (2x1) and (3x1). On the (2x1) surface a short (4 minute) exposure to CO (10^{-7} torr) at 25°C rapidly transformed the surface to the (1x1) structure (Fig.5(A)), in agreement

with the findings of many authors. For the (3x1) structure, however, exposure to CO (of up to 10^{-5} torr for 1/2 hour) resulted only in a decrease in the intensity of the one-third-order spots, but the transformation to the (1x1) could not be observed.

3.4 The Effect of Ar Sputtering

The bombardment of the (2x1) surface structure with Ar ions at 500 V resulted in the appearance of a (1x1) structure (Fig.5(B)). Surprisingly, however, the diffraction pattern showed a very low background, as can be seen in the picture of Fig.5(B) indicating good atomic order on the sputtered surface. This structure was maintained up to $\sim 500^\circ\text{C}$ where an irreversible transition to the stable (2x1) surface structure has occurred (Fig.5(C)).

4. Discussion

The results of Section 3.1 indicate that the Pt(110)-(2x1) surface has a very complex structure in spite of its simple LEED pattern. The various breaks observed in the $I(hk)$ vs. T curves indicate the occurrence of structural changes within the (2x1) unit cell induced by the changing temperature. It is not possible to ascertain the nature of these changes in the atomic location without a complete analysis of the $I(hk)$ vs. eV curves at various crystal temperatures. $I(V)$ curves have been obtained for this (2x1) structure by Ducros et al. ⁽¹⁶⁾ for the specular beam, probably at 25°C since the crystal

temperature was not specified. They observed an extreme sensitivity of the $I(V)$ curves to the angle of incidence and also the absence of any kinematic Bragg peaks. Lambert, et al⁽¹⁸⁾ have attempted to reproduce their experimental $I(V)$ curves by calculation using various models of surface reconstruction without obtaining any reasonable fit to the experimental curves. All of these observations seem to indicate that the (2×1) surface has a very complex structure. Davies et al⁽¹⁹⁾ studied the ion backscattering and channeling of He in this and other surfaces of Pt. They found that the number of scattering centers at normal incidence was about a factor of 2 higher than that expected for a (1×1) structure. This result implies that either there is a lateral displacement of the compact rows of Pt atoms or that an extra layer of Pt is present in a position not coincident with the (110) plane. An example of the latter possibility is obtained by rocking a compact $[110]$ row to the threefold sites of the 'valley' sides and adding an extra row between this one and the opposite side of the valley. The result is an undulated '100' face on top of the (110) surface. An undulated (111) overlayer on top of the (100) surface was proposed by various authors^(3,4) to explain the (5×1) pattern observed on the (100) face of Pt, Ir and Au.

The possibility that the stability of the clean (2×1) structure is influenced by an impurity, perhaps at 3 to 5 layers below the surface, cannot be ruled out. As suggested by the results of Section 3.2 subsurface oxygen below the Auger detectability limit causes important changes in the structure of the surface. Some authors⁽¹¹⁾ have indeed suggested the stabilizing effect of subsurface

oxygen in the case of the clean Pt(100)-(5x1) surface.

Reversible transformations from the reconstructed (100)-(5x20) faces of Au and Pt to the (1x1) structure have been observed by many authors at temperatures around 800°C^(7,8,11,20). Also the (110)-(2x1) surface of gold was observed to undergo such a transformation at 580°C.⁽²⁰⁾

The (3x1) surface structure that was judged to be clean by AES studies has previously been reported by Berthier et al.⁽¹⁵⁾ following a heavy oxygen treatment of their crystal. Also Ducros et al.⁽¹²⁾ report patches of (3x1) structure observed following oxygen treatments of the Pt(110) crystal face. This and the larger periodicity structures (5x1) and ("7x1") are indeed stabilized by oxygen that has been dissolved into the crystal during the high-temperature exposures. The depth of oxygen penetration will depend on the conditions of the oxygen treatment. After flashing the crystal to high temperature in vacuum, the first few layers (3 to 5) are sufficiently depleted of oxygen as to be undetectable by AES. Under these conditions, the surface periodicity is (3x1) as long as there is enough oxygen in the bulk.

At temperatures above ~830°C the stable surface structure is (1x1) in both the initially (2x1) and (3x1) surfaces. It is not clear whether this structural transformation is influenced by variation of the oxygen concentration in the near surface region. At these high temperatures the solubility and diffusion rates of many impurities, including oxygen, are quite large¹⁷. As a result, oxygen may diffuse deeper into the bulk,

away from the surface, The effect of H_2 and CO to remove the weakly bound surface oxygen from platinum is well documented.

It has been observed that the reconstructed (100) and (110) surfaces of Au, Pt and Ir transform to the (1x1) periodicity upon adsorption of various types of foreign atoms^(3,5,7,8,12,20). In the case of the Pt(100)-(2x1) crystal we have also observed this effect in agreement with these other observations. The ease with which the structure transforms seems to favor those reconstruction models involving only small displacement of the surface atoms. However, the behavior of the (2x1) structure during CO adsorption is different from that of the oxygen-induced (3x1) surface structure which remains stable up to high CO exposures, indicating a higher stability of this surface for CO adsorption.

Finally, Ar sputtering of the (2x1) surface produces a low-background (1x1) structure. This metastable structure is transformed rapidly back to the original (2x1) upon heating to $\sim 500^\circ\text{C}$ when the (2x1) structure is restored irreversibly. This transformation is similar to that found for the metastable Pt(100)-(1x1) structure by Bonzel *et al.*^{7,8}, which also transformed irreversibly to the reconstructed (5x20) structure upon heating.

5. Conclusions

a. The reconstructed Pt(110)-(2x1) surface exhibits complex structural changes as a function of crystal temperature

which are reflected in the appearance of breaks in the $\log I(hk)$ vs. T curves.

b. There is a reversible transformation from the (2x1) to the (1x1) structure at $830^\circ\text{C} \pm 30^\circ\text{C}$.

c. The dissolution of oxygen into the Pt(110) crystal in sufficient amounts causes the surface structure to change to (3x1), (5x1) and perhaps still larger periodicities.

d. The (3x1), (5x1) and ("7x1") structures transform to the (1x1) structure at $\sim 830^\circ\text{C} \pm 30^\circ\text{C}$. Upon cooling the (3x1) structure reappears if there is sufficient oxygen in the bulk.

e. An unreconstructed (1x1) surface may be obtained by CO adsorption on the (2x1) structure. The (1x1) structure may also be obtained in a metastable form by Ar sputtering. In this case the surface structure transforms irreversibly to the (2x1) at approximately $\sim 500^\circ\text{C}$.

The adsorption of CO on the (3x1) structure does not cause the transformation to the (1x1) periodicity but results only in a small decrease in the intensity of the one-third order spots.

Acknowledgement

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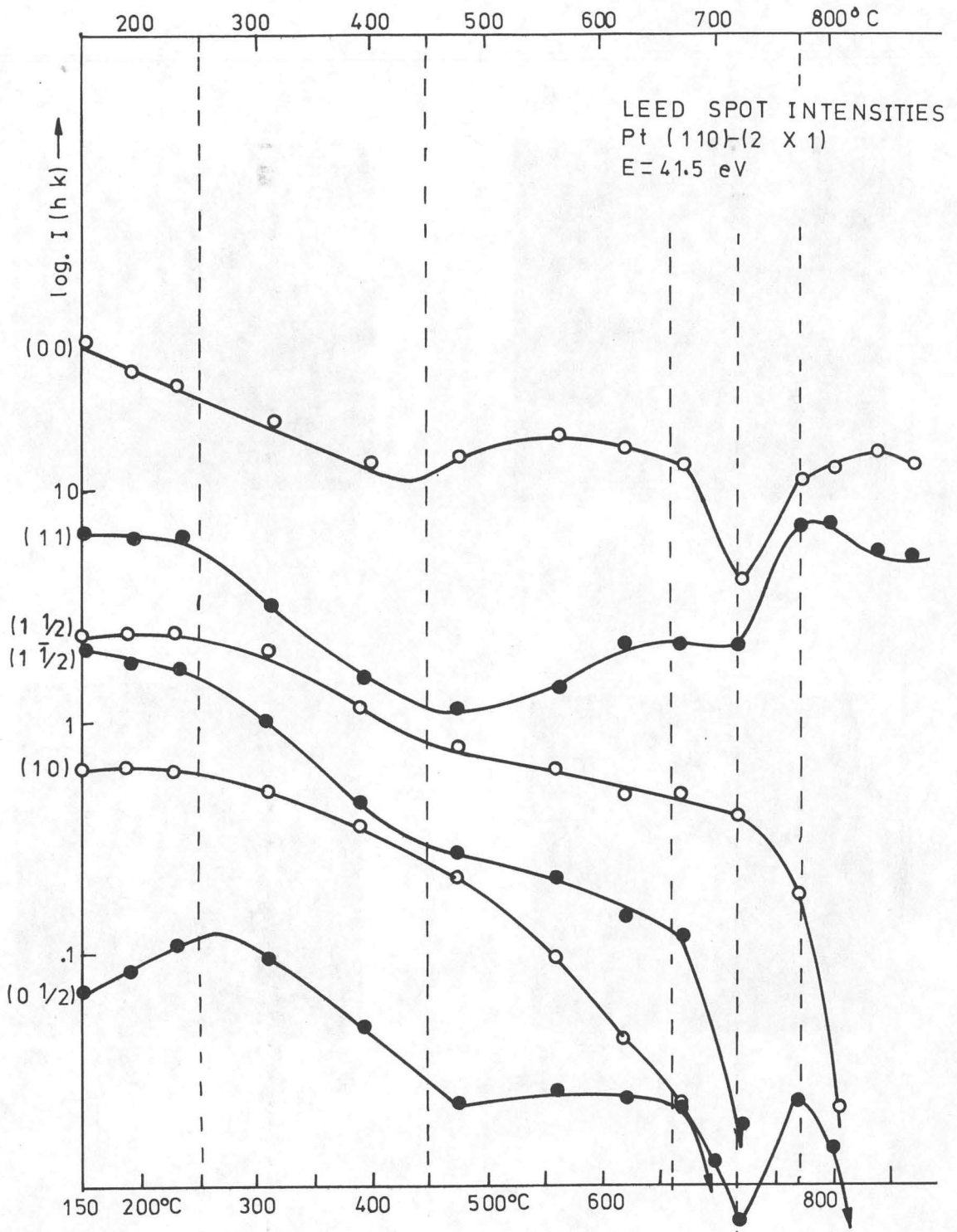
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Figure Captions

- Fig. 1 Surface transition in Pt(110) observed with LEED at $E=50$ eV. At $T < 830^\circ\text{C}$ the structure is (2x1), at $T_s \gtrsim 830^\circ\text{C}$ it is (1x1). A blue filter was used in front of the objective lens of the camera, in pictures B, C and D, to block the light emitted by the hot crystal and Ta support.
- Fig. 2 Semilog plot of the intensities of various diffraction spots from the Pt(110)-(2x1) surface as a function of crystal temperature. The curves have been displaced vertically for convenience only.
- Fig. 3 Surface structures of reconstructed Pt(100). A) Crystal has not been treated with oxygen; B) crystal has been treated with oxygen above 10^{-6} torr and 800°C for at least 30 minutes; C) and D) several days after the above oxygen treatment, during which the crystal has been exposed to the background gases (H_2 and CO mainly) at pressures between 10^{-9} and 10^{-8} torr. In most cases the (5x1) and ("7x1") structures can be found in different areas of the same crystal surface.
- Fig. 4 Surface transition in Pt(110) observed by LEED at $E=80$ eV. For $T_s \gtrsim 830^\circ\text{C}$ the structure is (3x1). This structure appears after oxygen treatments at $T_s \approx 800^\circ\text{C}$ and $P_{\text{O}_2} \gtrsim 10^{-6}$ torr for about 30 minutes or more. No oxygen is detected by AES, however. Above $T_s \gtrsim 830^\circ\text{C}$ the structure is (1x1). Pictures B, C and D were taken with a blue filter in front of the objective of the camera.
- Fig. 5 (A) Pt(110)-(1x1) structure resulting from exposure of the (2x1) structure to CO at 2×10^{-7} torr for ~ 4 minutes at 25°C ; (B) Pt(110)-(1x1) structure obtained by Ar ion bombardment at 500 eV of the (2x1) structure. Notice the low background of the pattern; (C) Pt(110)-(2x1) structure resulting from heating the structure of picture (B) at $\sim 500^\circ\text{C}$ for a few minutes.

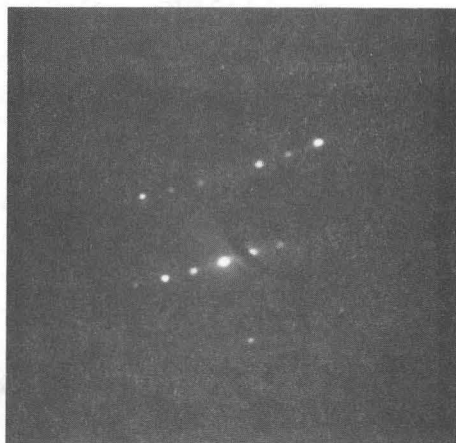
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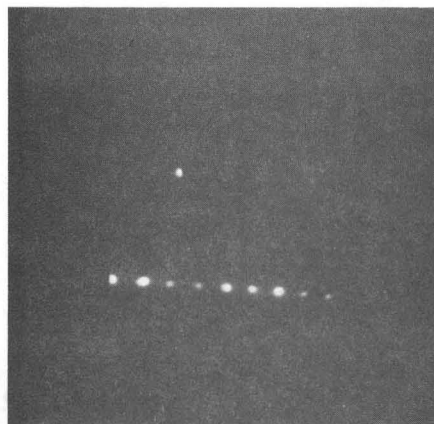


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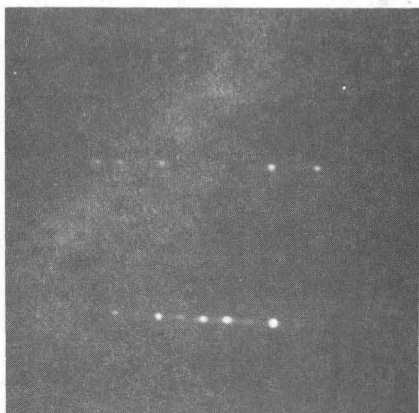
Fig.2



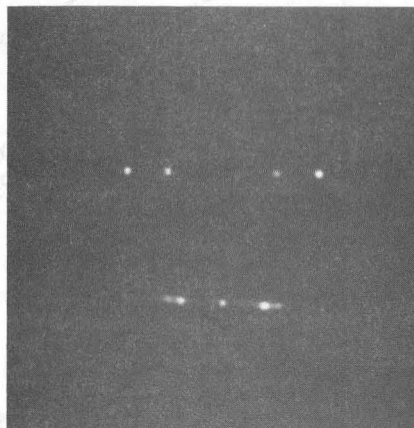
A. Clean Pt(110)-(2x1). E = 123 eV.



B. Clean Pt(110)-(3x1). E = 80 eV.



C. Clean Pt(110)-(5x1)". E = 52 eV



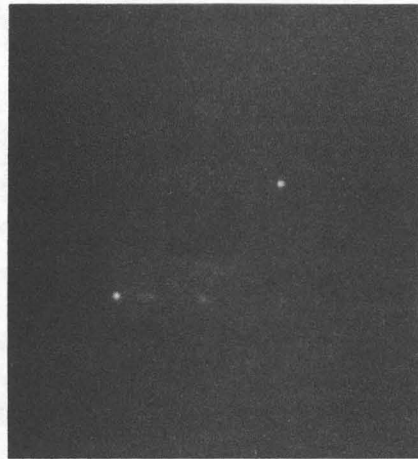
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D. Clean Pt(110)-(7x1)". E = 46 eV.

Surface structures of reconstructed Pt(110). A) Crystal has not been treated with oxygen B) Crystal has been treated with oxygen above 10^{-6} torr and 800°C , for at least 30 minutes. C and D) Several days after the above oxygen treatment, during which the crystal has been exposed to the background gases (H_2 and CO mainly) at pressures between 10^{-9} and 10^{-8} torr. Unlike the 2×1 and 3×1 structures, the structures of pictures C and D appear as islands on the surface, in the process of degradation of the 3×1 to the 2×1 structure. In most cases the 3×1 , " 5×1 ", and " 7×1 " structures can be found in different areas of the same crystal surface.

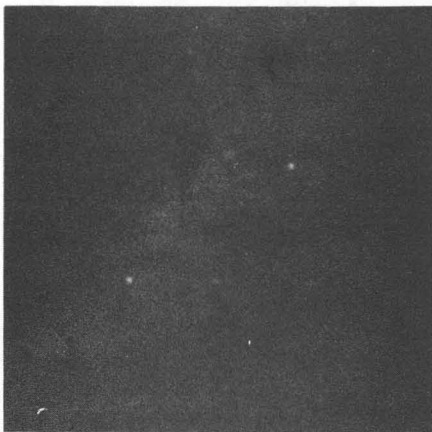
Fig.3



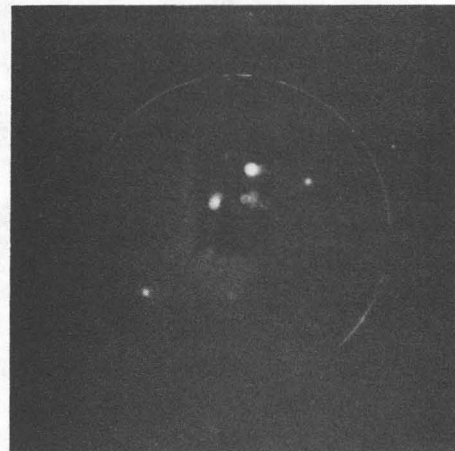
A. Pt(110)- (3X1). $T_s = 640^\circ\text{C}$



B. Pt(110) $T_s = 720^\circ\text{C}$
10 minutes exposure



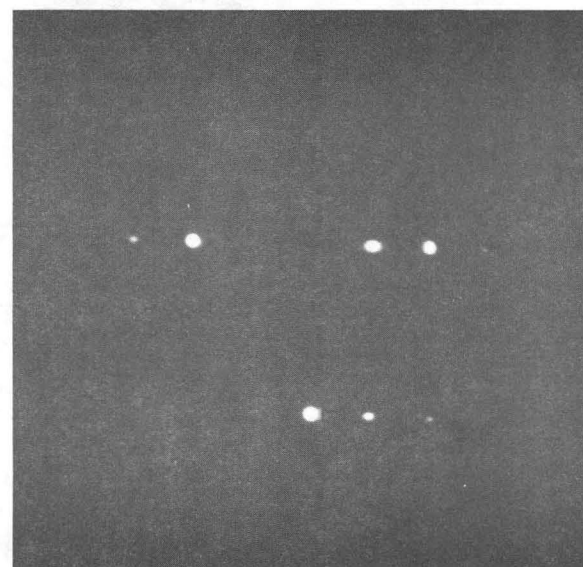
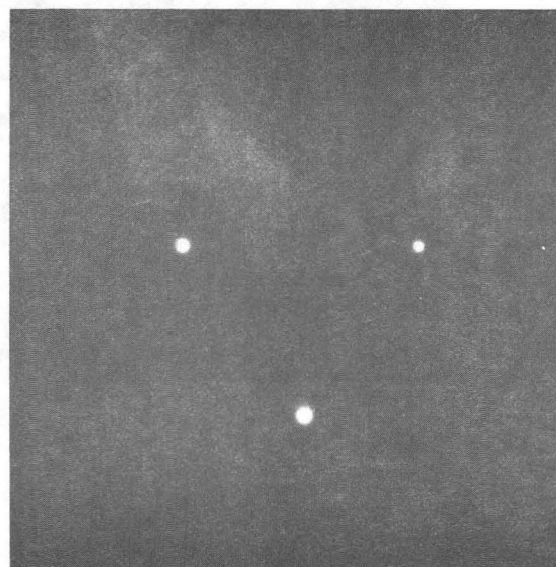
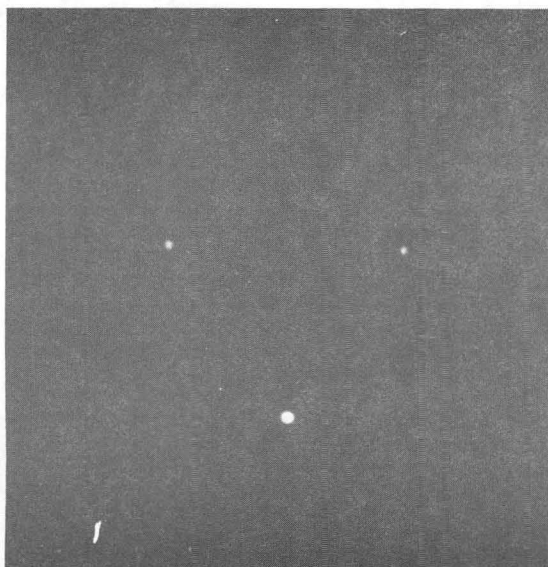
C. Pt(110) $T_s = 800^\circ\text{C}$
10 minutes exposure



D. Pt(110) $T_s = 850^\circ\text{C}$
15 minute exposure
XBB 776-5848

Surface transition in Pt(110) observed by LEED, at $E = 80$ eV. For $T_s \leq 800^\circ\text{C}$ the structure is 3X1. This structure appears after oxygen treatments at $T_s \approx 800^\circ\text{C}$ and $P_{O_2} \approx 10^{-6}$ torr for about 30 minutes or more. No oxygen is detected by AES, however. Above $T_s = 800^\circ\text{C}$ the structure is 1X1. Pictures B, C, and D were taken with a blue filter in front of the objective of the camera.

Fig.4



XBB 780-14923

Fig. 5

(A) Pt(110)-(1 x 1) structure resulting from exposure of the 2 x 1 structure to CO at 2×10^{-7} torr for ≈ 4 min. at room temperature.

(B) Pt(110)-(1 x 1) structure obtained by Ar ion bombardment at 500 eV of the (2 x 1) structure. Notice the low background of the pattern.

(C) Pt(110)-(2 x 1) structure resulting from heating the structure of picture (B) at $\approx 500^\circ\text{C}$ for a few minutes.

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