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The Mean Displacement of Surface Atoms in Palladium and Lead Single Crystals

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Abstract

The intensity of specularly reflected (00-reflection) low energy (5-500 eV) electrons have been measured as a function of temperature for the (100) and (111) faces of palladium and for the (111) face of lead in the temperature ranges 25-600°C and 25-225°C, respectively. From the data the root mean square displacements, $\langle u_\perp \rangle$, of surface atoms perpendicular to the surface planes and the surface Debye temperatures have been calculated. The mean vibrational amplitudes of surface atoms are 40 → 100% larger than for bulk atoms. There is little difference between the surface mean displacements of different crystal orientations; $\langle u_\perp \rangle$ appears to be relatively insensitive to changes of surface structure or surface density. The experimental results correlate well with those obtained for platinum and silver single crystal surfaces.

It is believed that all monatomic face-centered cubic crystal surfaces should have mean displacements markedly larger than that in the bulk.

INTRODUCTION

It has long been postulated[1] that the lattice vibration spectra of atoms at the surface should be different from the bulk atoms. Calculations by Maradudin[2] and Wallis,[3,4] using realistic models have shown that the anisotropy of the atomic environment at the surface of face-centered cubic metals gives rise to increased root mean square displacement of surface atoms. This change was computed to be the largest for the displacement component which is perpendicular to the surface plane. Similar calculations for ionic[5] and molecular[6] crystals have also predicted surface mean displacements which are different from that in the bulk.

Since this predicted large vibrational amplitude is restricted to atoms in the surface plane, the vibrational amplitude should rapidly approach the bulk value within a few atomic planes.[2,3] Low energy electron diffraction appears to be the only experimental technique which can be used conveniently to detect this effect. Since low energy electrons (10-500 eV) back-scatter after penetrating only a few atomic layers, this technique has been eminently successful in monitoring surface phenomena on an atomic scale. The temperature dependence of the diffracted low energy electron beam intensity (Debye-Waller factor) can yield the root mean square displacement of surface atoms.[7]

Such measurements have been previously carried out using three low index faces of platinum[8] [(100), (111), and (110)] and the (111) face of silver[9] in the temperature ranges 25-700°C and 25-300°C, respectively. The root mean square displacements of surface atoms perpendicular to the surface planes were determined. For all of the faces which were studied a root mean square displacement $\langle u_{\perp} \rangle$ which was (40-100%) larger than the mean displacement of bulk atoms was measured.[8,9]

In order to determine whether these results are representative of all face-centered cubic crystal surfaces we have measured the root mean square displacement in the (100) and (111) surfaces of palladium and the (111) face of lead using back-diffracted low energy electrons (10-500 eV) in the temperature ranges 25-600°C and 25-230°C, respectively. We shall attempt to correlate the surface mean displacements obtained for the different face-centered cubic single crystal surfaces and shall discuss the experimental limitations which affect the accuracy of the results.

The vibrational amplitude of surface atoms was obtained from the temperature dependence of the specularly reflected electron beam (00-reflection).[8] For these specularly backscattered electrons the scattering vector, $(\vec{k} - \vec{k}_o)$ is perpendicular to the surface plane. Therefore, the data yields only the vertical component of the root mean square displacement, $\langle u_{\perp} \rangle$.

EXPERIMENTAL

The low energy electron diffraction system of the post-acceleration type[10] was used in these studies. The single crystals of the highest available[11] purity were x-ray oriented, cut, polished, and etched.[12] The palladium samples (5 mm diameter, 1-2 mm thick disks) were mounted on tantalum holders. During the experiments the crystals were heated by applying d.c. current across the holders. The lead crystals (5 mm diameter, 8 mm long cylinders) were placed in a high purity nickel enclosure. A thermocouple was spot-welded to the back face of the palladium samples to determine the temperature of the specimen. For the experiments with lead crystals the thermocouple was attached to the holder and was calibrated using the melting point of lead. The ambient pressure was in the range of 10^{-10}-10^{-9} Torr for most of the measurements. Diffraction patterns were obtained occasionally immediately after pump-down and bake-out of the diffraction chamber on the carefully prepared palladium crystals. Ion bombardment and subsequent annealing heat treatments were used, however, to obtain a more ordered surface with sharper diffraction features. Ion bombardment was necessary to obtain the diffraction pattern from the (111) face of lead crystals.

The (100)-face of palladium is known to undergo surface phase transformations in the studied temperature range.[12] The (2 ×1), (2 × 2), and c(2 × 2) surface structures are formed with increasing temperature above 200-250°C.[12] It was found that, in fact, the appearance of the (2 × 2) surface structure and then its partial transformation into the c(2 × 2) structure during the experiment could not be avoided. We have attempted to measure the Debye-Waller factor on the (100) face of palladium in the absence of any surface structure by heating the crystal to only 250°C, below the formation temperature of the surface structures. This way the diffraction pattern which is due to the substrate unit mesh (1 × 1) could be maintained throughout the experiment. The mean displacement calculable from the data obtained in this study agreed well with the values obtained in measurements which were carried out in the presence of surface structures. Therefore, most of the studies were carried out in the presence of these surface structures in order to extend the measurements to a wider temperature range and thereby minimize the experimental error. The reproducibility of consecutive measurements was uneffected by the extended heating cycles necessary to perform the experiments.

During work on Pd(111), a Pd(111)$\sqrt{3}$(1 × 1) R30° surface structure formed frequently during the course of the measurements. It appeared to result from adsorption of ambient gases, unlike the Pd(100) surface structures. Inasmuch as the heating to 600°C for the Debye-Waller runs easily removed the contaminant and the surface structure, it did not interfere with the experiments. However, its ability to form even after extensive heatings and/or ion bombardments upon overnight exposure to the ambient at $P \sim 2 \times 10^{-10}$ suggests the possibility for future study which we have not pursued at this time.

The (111) face of lead single crystals used in the experiments remained unchanged throughout the runs. No surface structure seems to form in the temperature range of this investigation (25-230°C).

All of the measurements of the temperature dependence of the specularly reflected electron beam (00-reflection) was carried out by the transient method.[8] The palladium crystal was heated to 600°C (230° for lead) and then the heating current was turned off. The intensity of the (00)-reflection was measured continuously using a small angle spot-photometer[13] as a function of temperature while the crystal was cooling to room temperature. The photometer output and the thermocouple emf were displayed simultaneously on an x-y recorder. This transient technique avoids the difficulties which arise from the interference of the heating current with the impinging electron beam, which make measurements below 50 eV electron beam energies very difficult to perform.

The angle of incidence of the impinging electron beam was (2.5-6°) with respect to the surface normal for most of the measurements. 2.5° is the smallest angle to the surface normal which can be used in the post-acceleration diffraction chamber. The intensity of the fluorescent screen background was monitored by either rotating the crystal so that the (00)-reflection was directed back to the electron gun (which is normal to the surface plane) or by scanning with the spot photometer about the (00)-reflection. The background intensity was found to be virtually independent of temperature at low beam voltages (<200 eV). However, for palladium, at higher electron energies, the background intensity was found to systematically increase slightly with increasing temperature. This intensity change was taken into account in calculating the root mean square displacements. The I_{00} vs. T curves were measured at different electron beam energies in the range of 10-400 eV. Those beam energies were selected which correspond to intensity maxima.

It may be useful to list some of the experimental difficulties which affect the accuracy of the measurements (5-10%). There is uncertainty in measuring the background intensity since it may vary markedly in the vicinity of the (00)-reflection. Also, the intensity of the (00)-reflection varies along the crystal surface due to the variation of crystal perfection along the surface, i.e., changes in the number of domains of ordered atoms in the crystal area hit by the electron beam flux ($\sim$1 mm^2). Therefore, care should be taken to standardize the conditions of the experiments.

It is advisable to use as large temperature range as possible in order to improve the accuracy of the Debye-Waller factor measurements. This is difficult when working with solids with low melting point or high vapor pressure.

RESULTS

In order to obtain the Debye-Waller factor from the experimental intensity curves the background intensity has been subtracted. This way the contribution of thermal diffuse scattering to the total intensity is removed.[14] The intensity of scattered electrons (neglecting multiple scattering events) is then given by

$$I = |F_{hkl}|^2 \exp[-(16\pi^2 \cos^2\phi/\lambda^2)\langle u_\perp^2\rangle] \qquad (1)$$

where the exponential term is the Debye-Waller factor λ is the electron wavelength, ϕ is the angle of incidence with respect to the surface normal, and $|F_{hkl}|^2$ is the scattered intensity by a rigid lattice. Using the Debye model of lattice vibrations in the high temperature limit[4,15] the mean square displacement is given by

$$\langle u^2\rangle = (3N\hbar^2/Mk)(T/\theta^\infty) \qquad (2)$$

where θ^∞ is the Debye temperature at the high temperature limit, M and T are the atomic weight and the temperature of the solid, respectively, N is Avogadro's number and k and $\hbar$ are the Boltzmann and Planck constants. Combining Eqs. (1) and (2) we have

$$I_{hkl} = |F_{hkl}|^2 \exp\left[-\frac{12Nh^2}{Mk}\left(\frac{\cos\phi}{\lambda}\right)^2 \frac{T}{(\theta^\infty)^2}\right] \qquad (3)$$

The logarithm of the intensity plotted as a function of temperature, T, gives a straight line. From the slope the root mean square displacement in the direction perpendicular to the surface plane, $\langle u_\perp \rangle$, can be calculated.

We have found, just as in the case of platinum and silver surfaces,[8,9] that the measured values of the root mean square displacements were strongly dependent on the electron beam energy. It is apparent that with increasing electron energy a larger fraction of electrons scatter from atomic planes which lie below the surface plane. Thus at higher electron energies the experimentally determined mean displacement approaches the bulk mean displacement value. The mean displacement which is characteristic of the surface atoms can be obtained from data taken using very low energy electron beam ($\geq$ 15 eV). At such low energies the largest fraction of the impinging electrons back-scatter from the surface atoms without penetrating deeper into the lattice. The effective root mean square displacement, $\langle u_\perp^{eff} \rangle$ calculated from the log I_{00} vs. T curves using Eq. (1) is plotted as a function of beam voltage for the (100) and (111) faces of palladium (Figure 1). It is apparent that the surface root mean square displacement is much greater than that of the bulk value (dotted line) for both orientations.[16]

The effective mean displacement measured for the (111) face of lead single crystals as a function of beam voltage is plotted in Figure 2. The highest beam voltage at which meaningful intensity measurements could be carried out was ~75 eV. The intensity of the diffracted beam decreases sharply with increasing beam voltage (decreasing electron wavelength) and approaches the background intensity at about 100 eV due to the large Debye-Waller factor for lead [$\theta_{bulk}(Pb) = 90°K$[18] compared with $\theta_{bulk}(Pd) = 274°K$].[17] Palladium yielded useful experimental information at beam voltages $\geqslant$400 eV.

The root mean square displacements which were obtained at the lowest electron beam energies were taken as values characteristic of the mean vibrational amplitude of surface atoms. These along with the corresponding bulk mean displacements, the calculated surface and bulk Debye temperature are listed in Table 1. For comparison, we have included the values obtained for the different platinum surfaces[8] and for the (111)-face of silver[9] single crystal.

DISCUSSION

The Debye-Waller factor measurements using several faces of four different face-centered cubic crystals indicate that the root mean square displacement of surface atoms is roughly 140 → 200% of the bulk value. The variation of the effective mean displacement with electron beam energy is clearly indicated by Figures 1 and 2. The effective mean displacement changes sharply as the contribution due to atoms in the surface plane starts to dominate.

A pseudo-kinematic model which takes into account the relative contributions of the surface and bulk atomic planes to the diffracted intensity assuming different Debye-Waller factors for the surface and for the bulk planes is discussed in the Appendix.

There seems to be little difference in the mean displacements of surface atoms in the different low index planes with respect to the large difference between the bulk and surface values. One would expect the highest density (111) plane to show the smallest increase from the bulk value while the sparsely populated (100) and (110) faces the largest change. Although there is some difference between the measured values they are still equal within the accuracy of the measurements. The insensitivity of the mean displacement of surface atoms normal to the surface plane to changes of surface density (number of nearest neighbors) can also explain why changes in the surface structure of palladium did not have observable effect on the experimental results. It is likely that the lateral displacement, $\langle u_{||} \rangle$, may show a more pronounced dependence on the surface density of atoms than $\langle u_{\perp} \rangle$.

We have found that the $\langle u_{\perp} \rangle$ vs. eV curves for the (111) faces of both palladium and platinum seem to change more sharply with decreasing beam voltage than for the other crystal faces and appear to cross over the effective mean displacement curves obtained for the other orientations.[8] It may indicate that either the fraction of electrons which penetrate below the surface plane decreases more rapidly with decreasing electron beam energy for the (111) face than for the other faces or, that the effect of the surface anisotropy falls off more rapidly along the (111) axis than along other crystallographic orientations.

The effective Debye temperature of the (111) face of silver seems to show little change below 100 eV.[9] Our measurements on lead and palladium surfaces indicate a continued change of the Debye temperature (or mean displacement) throughout the range 100 → 20 eV. In fact, all of the experimental information on the Debye-Waller factor of lead were obtained in this range of electron energies. It is apparent that the bulk atoms in these solids (Pd,Pb) are still contributing to the intensity scattered into the 00-reflection at these low electron energies.

In calculating the root mean square displacements we have not corrected the electron beam voltage, (eV), for the presence of the inner potential (ip). The attractive potential the electron experiences at the surface adds an energy increment to the electron beam energy which is of the order of 5-35 eV.[19] Such a correction would have little effect on the effective mean displacements which are calculated from measurements at high electron energies. It has large influence on the mean displacements, however, which are calculated from Debye-Waller factors determined at low electron beam energies. A root mean square displacement can be corrected for the presence of the inner potential according to the formula $\langle u_\perp \rangle$(corrected) = $\langle u_\perp \rangle$ (uncorrected)/$[(eV + ip)/eV]^{1/2}$. For example, at eV = 50 eV, and for ip = 20 eV, $\langle u_\perp \rangle$ (corrected) = 0.85 $\langle u_\perp \rangle$ (uncorrected). Since its value has not been determined accurately, all of the data are given without the inner potential correction. An inner potential correction would tend to decrease somewhat, the calculated mean surface vibrational amplitudes.

The scattering mechanism of low energy electrons by surfaces is not well understood at the present. There are indications, both experimental[20] and theoretical,[21] that multiple scattering of electrons between atomic planes may play an important role in low energy electron diffraction. Thus, the question arises how justified it is to use Eq. (1) in interpreting the temperature dependence of the diffracted intensity. Multiple scattering contributions may have a marked influence on the magnitude and/or the form of the Debye-Waller factor. There are several experimental informations, however, which indicate that multiple scattering effects do not have a marked influence on the Debye-Waller factor measurements. The surface mean displacement approaches that bulk mean displacement value with increasing electron beam energy, which was determined by independent heat capacity[17] or Mossbauer experiments.[22] Intensity measurements which were carried out at beam voltages which coincide with the appearance of "fractional order" or "secondary"-Bragg peaks yield effective mean displacements which fit well on the experimental curves as shown in Figures 1 and 2 within the accuracy of the experiments. Similar results were obtained in studies of the surface Debye temperature of platinum single crystal surfaces.[8] Figure 3 shows a set of representative I_{00} vs. eV plots at different temperatures which were obtained for the (100) face of platinum.[23] It is apparent that all of the diffraction peaks which are due to either primary or "secondary" Bragg reflections, show uniformly decreasing intensities with increasing temperature.

Debye-Waller measurements do not yield the "net" displacement of surface atoms. The large mean vibrational amplitude indicates, however, that the surface atoms either displace outward to new equilibrium positions and maintain a nearly harmonic motion, or they remain in their equilibrium positions similar to that for the bulk atoms and their vibration becomes strongly anharmonic. The phonon spectrum of surface atoms which is different from that for the bulk atoms is the likely reason for the formation of different surface structures.[24] The atoms in the surface plane may relax by moving to out-of-plane positions in a periodic manner. The free energy for out-of-plane relaxation of surface atoms which could lead to the formation of $(N \times 1)$-type surface structures [N is the periodicity of the new unit mesh of a surface structure with respect to the substrate $(N = 1)$ unit mesh] is comparable to that necessary to place the atoms in their in-plane equilibrium positions.[25,26]

Calculations using a pseudo-kinematic model were successful in reproducing many features of the I_{00} vs. eV curves for several solid surfaces by assuming that there is an "asymmetry" in the arrangement of atoms in the surface plane with respect to atoms in the underlying planes.[27] It should be pointed out that the larger Debye-Waller factor for surface atoms which is markedly different from that for the bulk atoms introduces such an asymmetry. The large difference in the Debye-Waller factors of the surface and bulk planes alone can generate fractional order Bragg peaks. The effect of the large mean displacement of surface atoms on the wavelength dependence of the specularly reflected electron beam will be discussed in detail elsewhere.

Correlation of the results of the Debye-Waller factor measurements using Pd, Pb, Pt, and Ag surfaces indicate that, (a) the surface vibrational amplitudes normal to the surface plane are between 40-100% larger than the corresponding bulk values for all of these metal surfaces; and (b) there is little difference between the mean displacements of the different crystal orientations, i.e., the perpendicular mean displacements appear to be insensitive to changes in surface density. It is believed that the mean displacement of surface atoms should be markedly larger than that in the bulk for all monatomic face-centered cubic crystals.

APPENDIX

Using a pseudo-kinematical theory[28,29] the intensity scattered by a set of atomic planes is given by[23]

$$I = |f_o|^2 A_o^2 (\beta + \alpha M)^2 \tag{A-1}$$

where f_o is the atomic scattering factor per plane, A_o is the amplitude of the incident beam, β is the Debye-Waller factor for the surface plane and α is the Debye-Waller factor for the bulk plane. M is given by

$$M = \exp i(2Dk) \frac{1 + T^2 \exp i[2(kD + \phi)]}{1 - T^2 \exp i[4(kD + \phi/2)]} \qquad \text{(A-2)}$$

where D is the interplanar distance, k is the scattering vector, ϕ is the phase angle and T is the transmission coefficient. At the intensity maxima predicted by the three-dimensional kinematic theory ($2Dk = 2\pi n$), M is equal to

$$M = \frac{1 + T^2}{1 - T^2} \qquad \text{(A-3)}$$

For high energy electrons which penetrate deep below the surface plane ($T \rightarrow 1$)

$$I \approx f_o^2 \, A_o^2 \, M^2 \, \alpha^2 . \qquad \text{(A-4)}$$

Thus, the effect of a different Debye-Waller factors for the surface and bulk planes is washed out. For low energy electrons which back-scatter mostly from the surface plane ($T \rightarrow 0$) the intensity is given by

$$I \approx f_o^2 \, A_o^2 \, (\beta + \alpha)^2 \qquad \text{(A-5)}$$

The observed effective Debye-Waller factor is therefore a mixture of the bulk and surface components. At the lowest beam voltage it yields the mean displacement of surface atoms while at high energies that of the bulk atoms. A realistic model which takes into account the fractional contributions of bulk and surface planes to the effective Debye-Waller factor may be useful to calculate the true surface mean displacement. The use of such a model would eliminate the errors due to the application of arbitrary inner potential corrections. It was found that using Eq. (7) the experimental $\langle u_\perp \rangle$ vs. eV curves for the different platinum surfaces could be reproduced by inserting the values listed in Table 1 and a mean inner potential of 17 eV.

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Table 1. The surface and bulk root mean square displacements and Debye temperatures of palladium, lead, platinum, and silver.

	Pd (100)(111)	Pb (100)	Pt (100)(110)(111)	Ag (111)
$\langle u_\perp \rangle$ (surface)(Å)	0.144	0.263	0.135	0.129
$\langle u \rangle$ (bulk)(Å)	0.074	0.160	0.064	0.089
θ_D (surface)(°K)	140±10	55±10	110±10	155
θ_D (bulk)(°K)	273.4	90.3	234	225

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FIGURE CAPTIONS

Fig. 1 The effective root mean square displacement, $< \mu_\perp >$, of surface atoms perpendicular to the surface plane in the (111) and (100) faces of palladium as a function of electron beam energy.

Fig. 2 The effective root mean square displacement, $< \mu_\perp >$, of surface atoms perpendicular to the surface plane in the (111) face of lead as a function of electron beam energy.

Fig. 3 The specular (00)-reflection as a function of electron beam energy at different temperatures for the (100)-face of platinum.

Fig. 1

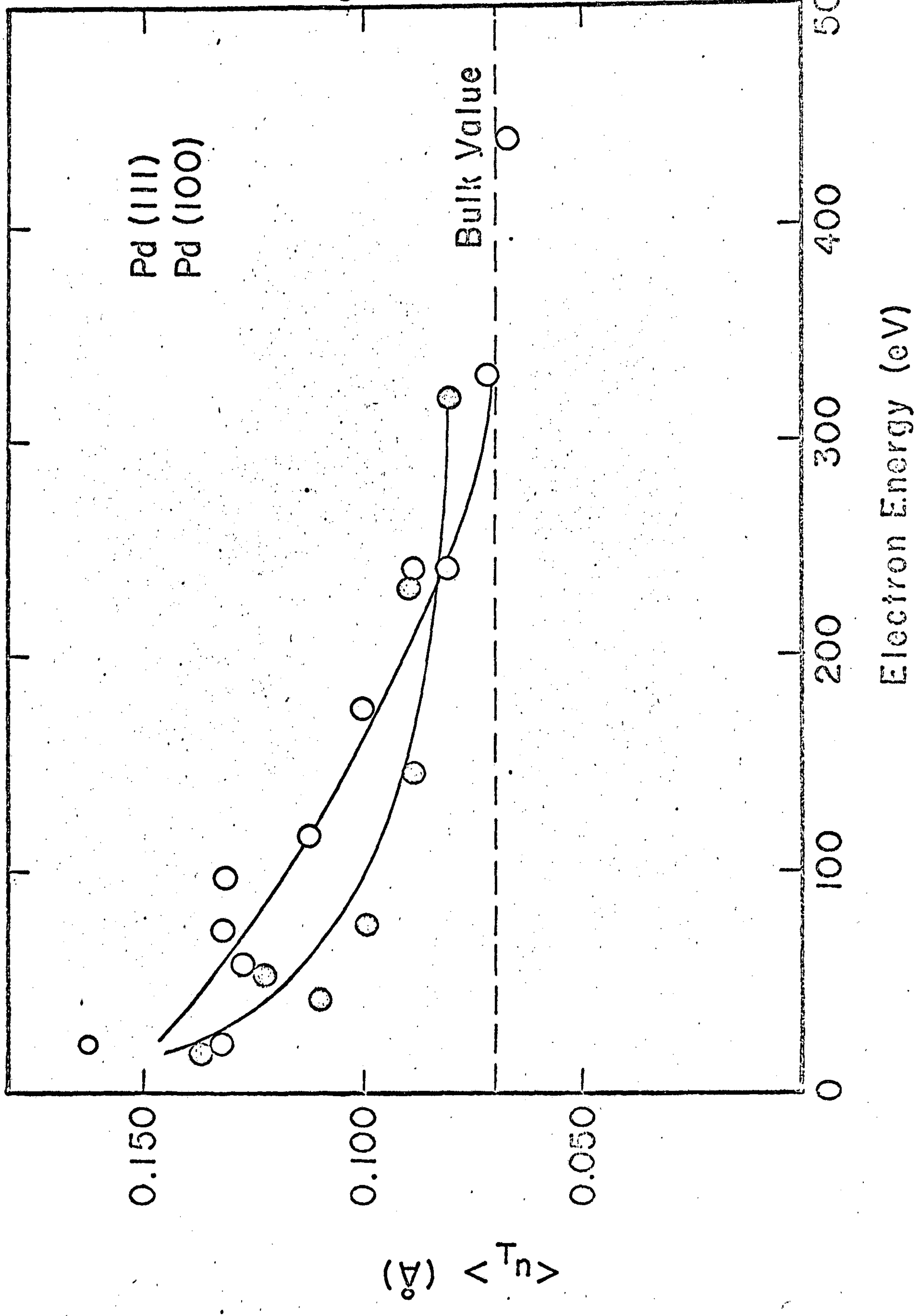

Pd (111)
Pd (100)
Bulk Value
0.150
0.100
0.050
0
100
200
300
400
500
Electron Energy (eV)
$\langle u_\perp \rangle$ (Å)

Fig. 2

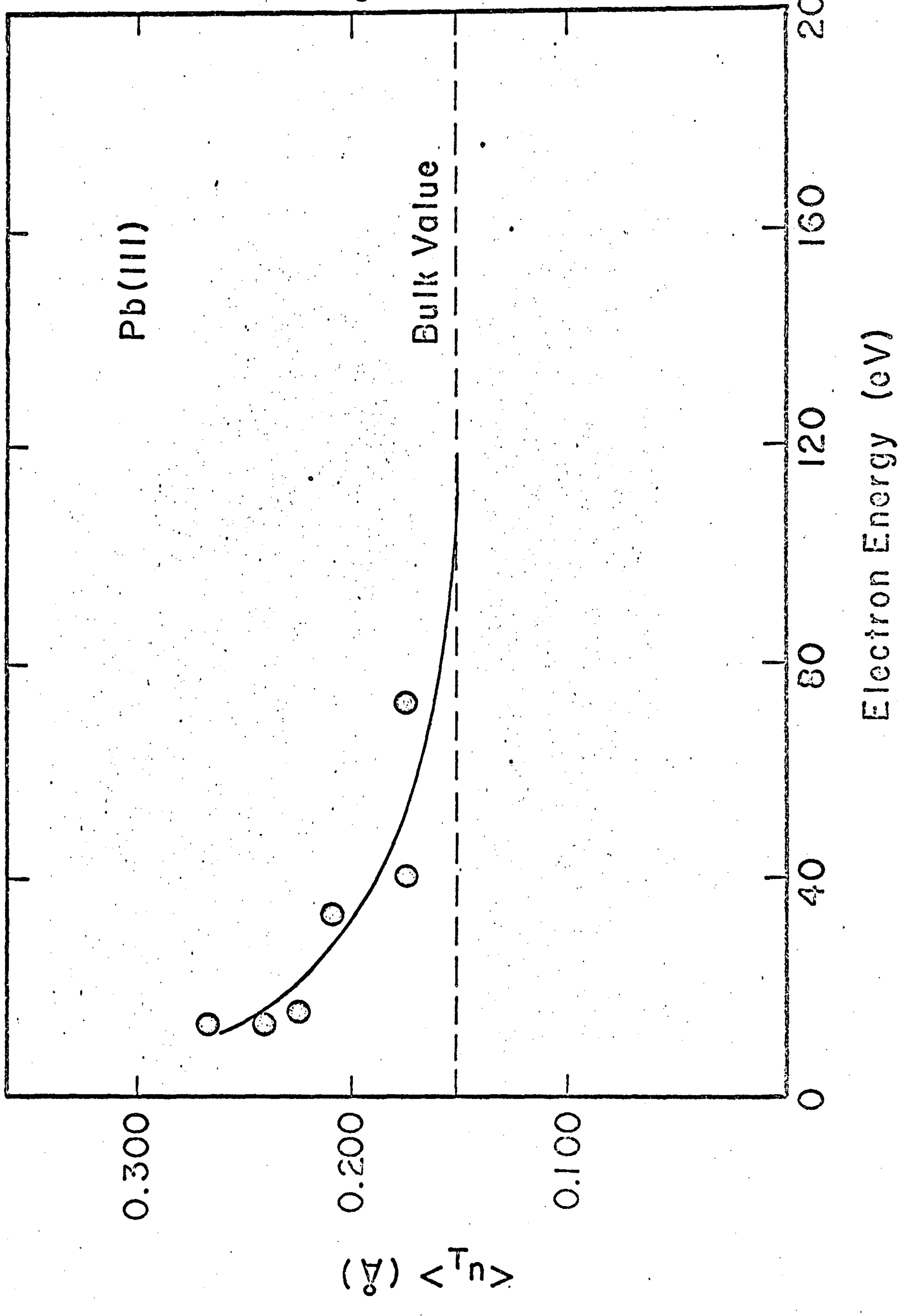
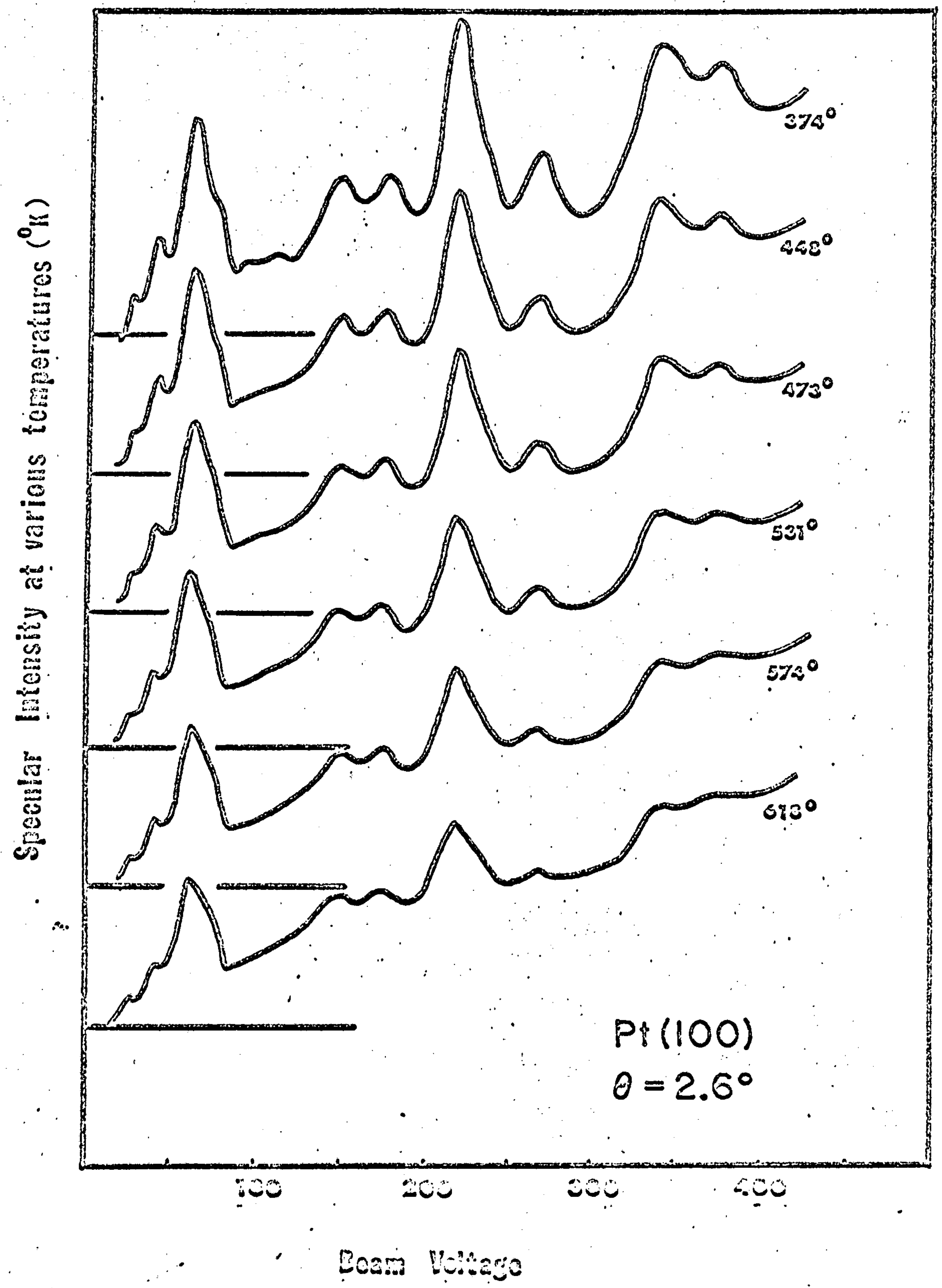

Pb(111)
Bulk Value
0.300
0.200
0.100
$\langle u_T \rangle$ (Å)
0
40
80
120
160
200
Electron Energy (eV)

Fig. 3


374°
448°
473°
531°
574°
613°
Pt(100)
θ = 2.6°
100
200
300
400
Beam Voltage
Specular Intensity at various temperatures (°K)