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Auger Electron Studies of Surfaces:
Uranium Dioxide, Uranium, Graphite,
300 Series Stainless Steel, and Niobium

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AUGER ELECTRON STUDIES OF SURFACES:

URANIUM DIOXIDE, URANIUM, GRAPHITE, 300 SERIES STAINLESS STEEL, AND NIOBIUM

by

Bruce D. Campbell*

ABSTRACT

The Varian low energy electron diffraction (LEED) system was successfully modified to produce Auger electron spectra from solid surfaces. The Auger spectra from cleaned (111) and (110) surfaces of UO_2 indicated that no large amount of surface contamination was present. When surface superstructures were formed by oxygen exposures, the Auger spectra indicated that oxygen was in fact the substance which caused the new structure. The Auger spectra from clean uranium metal resembled that from UO_2 , except for the greatly diminished intensity of the oxygen peak and different relative intensities of the four peaks between 50 and 100 eV. Also, a large sulfur peak at 150 eV was always present for the annealed uranium surface. Although the sulfur was removed by argon-ion bombardment, it diffused to the surface again when the sample was annealed. Polycrystalline samples of niobium and stainless steel also exhibited segregation of sulfur at the surface. As with uranium, the sulfur could be removed by ion bombardment but returned on heating. A sample of graphite exhibited only the expected carbon peak. However, after argon-ion bombardment, an intense argon peak was observed, indicating detection of argon imbedded in the lattice.

1. INTRODUCTION

An ionized atom may rearrange itself either through radiative or radiationless Auger transitions.¹ The radiative transitions form the basis for materials analysis by optical and x-ray spectroscopy. Recently, several investigators²⁻⁵ have shown that analysis of solid surfaces can be accomplished by particle spectroscopy based on the detection of Auger electrons with energies characteristic of the emitting atoms. In this technique, the surface atoms are ionized by electron bombardment and the Auger electrons are detected as peaks in the secondary electron distribution. This type of spectroscopy was originally suggested by Lander,⁶ and more recently by Scheibner and Tharp,⁷ but the value was considered questionable because of limited

sensitivity. Harris² showed that the sensitivity could be greatly enhanced by electronic differentiation of the secondary electron distribution, $N(E)$. Palmberg⁵ and Weber and Peria³ showed that the electron distributions and the derivatives could be produced with a standard three-grid low energy electron diffraction (LEED) system such as that made by Varian Associates. The combination of the LEED and Auger techniques thus allows determination of the surface structure and composition.

The Auger transition arises from the electrostatic interaction between two electrons in an initially singly ionized atom.^{1,8-10} For example, assuming that an atom is ionized in the K shell and an electron makes a transition from the L_I level to K while an Auger electron is ejected from the L_{II}

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level, the energy of the Auger electron, E_A , will be approximately $E_A = E_K - E_{L_I} - E_{L_{II}}$, minus a small correction for a chemical shift in the energy levels. In the common notation this transition would be denoted by $KL_{II}L_{II}$.⁸⁻¹⁰ Because the energies of the levels are known from x-ray data,¹¹ it should be possible to predict the energies of the Auger electrons for a given material. The Auger spectra are potentially more complicated than x-ray spectra because up to three levels are involved and radiative transitions forbidden by selection rules are not forbidden to the Auger process.

The x-ray fluorescence yield for ionization in the K shell is $\omega_K = 1 - a_K$, where a_K is the Auger electron yield. ω_K varies from 0.1 for atoms of atomic number $Z = 10$ to about 0.9 for $Z = 80$.⁸⁻¹⁰ Since the Auger yield is high for low- Z elements and the x-ray yield is high for high- Z elements, a combination of the two spectroscopies would be useful for studying the whole range of elements. The Auger technique is especially suited for surface studies since common surface contaminants are carbon, oxygen, nitrogen, and sulfur.

Another property of the Auger electron emission which makes it especially useful for analysis of surface composition is the very low escape depth that is involved. Palmberg and Rhodin⁴ have shown that 300-eV Auger electrons escape through only about four monolayers of silver. If the Auger electrons are produced any deeper below the surface, they lose too much energy while escaping and do not contribute to the peak. The short escape depth also accounts for the fact that, if the ionizing electron beam is at normal incidence, an increase in incident electron energy beyond a few kilovolts does not increase the yield. If the incident electrons penetrate too far into the sample, the Auger electrons do not escape. Palmberg¹² has also shown that an electron beam incident at about 15 degrees to the surface increases the sensitivity considerably by producing the Auger electrons closer to the surface.

2. EXPERIMENTAL

The principle of operation in the use of the LEED system for measuring secondary electron distribution can be shown with the aid of Fig. 1. Electrons of energy $eE_p = 1500$ eV are incident on the

Fig. 1. Diagram of experimental setup for using three-grid LEED system for Auger electron analysis.

sample and are scattered back into the system of grids. The sample is at the center of the spherical grids which subtend an angle of $4\pi/3$ steradians. Grid B is the suppressor grid and excludes all electrons below energy eE_0 , where E_0 is the voltage on the grid. Grid A serves as an electrostatic shield between the sample and the suppressor grid so that the electrons travel in a field-free region. Grid C serves as a capacitive shield between the suppressor and the collector D. The fluorescent screen of the LEED system serves as a collector, and the 300 V on this screen prevents it from charging up. If the current $I(E)$ to the collector is plotted as a function of the suppressor voltage E_0 , the integral of the energy distribution is obtained:

$$I(E_0) = \int_{E_p}^{E_0} N(E) dE. \quad (1)$$

Thus $N(E)$ is obtained from the first derivative of $I(E)$

$$N(E) = \frac{dI(E)}{dE}. \quad (2)$$

As Palmberg and Rhodin⁴ have shown, $I(E)$ can be expanded in a Taylor series about the point E_0 .

$$\begin{aligned}
I(E) = I(E_0) &+ \left[\frac{dI(E)}{dE} \right]_{E=E_0} (E - E_0) \\
&+ 1/2! \left[\frac{d^2I(E)}{dE^2} \right]_{E=E_0} (E - E_0)^2 \\
&+ 1/3! \left[\frac{d^3I(E)}{dE^3} \right]_{E=E_0} (E - E_0)^3 + \dots
\end{aligned} \quad (3)$$

If the voltage $(E - E_0)$ is made to vary sinusoidally by superimposing a small signal $k \sin \omega t$ on the suppressor grid voltage E_0 , the equation becomes

$$\begin{aligned}
I(E) = I(E_0) &+ k N(E_0) \sin \omega t + 1/4 k^2 \left(\frac{dN(E)}{dE} \right)_{E=E_0} (1 - \cos 2 \omega t) \\
&+ \frac{k^3}{24} \left(\frac{d^2N(E)}{dE^2} \right)_{E=E_0} (3 \sin \omega t - \sin 3 \omega t) + \dots
\end{aligned} \quad (4)$$

Thus, by placing a small sinusoidal signal on the suppressor grid and using a narrow band amplifier to detect the signal at the collector, $N(E)$ is detected by tuning the amplifier to ω and $dN(E)/dE$ is detected by tuning to 2ω .

3. RESULTS AND DISCUSSION

Sample secondary electron distributions, $N(E)$, of the (111) surface of UO_2 are shown in Fig. 2. The primary energies of the incident electron beams for the two curves were 300 and 500 eV. The sharp peaks at 300 and 500 eV indicate the elastically scattered electrons. The small peaks denoted by A

and B are characteristic loss peaks. They represent electrons which have lost a given amount of energy by exciting either plasmons¹³ or interband transitions.¹⁴ These peaks always appear at a characteristic distance from the elastic peak and thus are dependent on the primary energy. The small bumps, denoted by C and D, represent Auger electrons. These peaks are characteristic of the atoms at the surface and always occur at the same energy, no matter what the incident energy is, as long as it is

high enough to ionize the atom. It should be noted that the width of the elastic peak increases as the incident energy increases, indicating a decrease in resolution. Palmberg¹² has shown that the resolution can be increased by an order of magnitude by installing a fourth grid and by using the two center grids as the suppressor. The poor resolution with three grids is due to field penetration through the suppressor grid.

The effect of the modulating voltage on the resolution at low energies is shown in Fig. 3. These curves were taken with the same incident energy electrons but with two different modulating

Fig. 2. Energy distributions of secondary electrons from UO_2 for incident electron energies of 300 and 500 eV.

Fig. 3. Energy distributions for secondary electrons from graphite with two different modulating voltages.

voltages. Scheibner and Tharp⁷ have published similar curves for single crystalline graphite.

Above 200 eV in the $N(E)$ curve, a high modulating voltage did not appreciably affect the resolution but did increase the sensitivity. Therefore, the usual procedure was to use 11 VAC rms above 200 eV to obtain maximum sensitivity and 3 VAC rms below 200 eV to obtain the necessary resolution.

Figure 4 shows the effect of electronic differentiation of the distribution curve. The small shoulders below 100 eV on the $N(E)$ curve are now well defined maxima and minima in the $dN(E)/dE$ curve.

3.1 (111) and (110) Surfaces of UO_2

Interest in UO_2 arose from LEED studies made at Los Alamos Scientific Laboratory (LASL)¹⁵ with the same apparatus used in the present Auger studies. The absence of fractional-order diffraction beams from the cleaned $UO_2(111)$ surface indicated a clean surface. However an amorphous contaminant would not be detected by LEED, and the Auger spectra should be useful for verifying the cleanliness of this surface. On the $UO_2(110)$ surface, fractional-order beams or facet beams were usually present, no matter what cleaning procedure was used. Thus the Auger results should shed light on the type of material on the surface that produced these superstructures on $UO_2(110)$.

Surface damage caused by polishing the UO_2 was removed by forming a UF_4 film on the surface and then

removing this film by dissolving it in 0.2 M HNO_3 . Thus it should be possible to determine with the Auger technique whether the surfaces were contaminated with fluorine.

The Auger spectrum of the (111) surface of UO_2 , after the diffraction system was evacuated and baked but before the crystal was heated, is shown in Fig. 5. The large peaks at 275 and 525 eV correspond to the KVV transitions (V for valence band) in carbon and oxygen, respectively. As discussed below, the four small peaks between 50 and 100 eV appear to be characteristic of UO_2 . The $UO_2(110)$ surface also exhibited about the same curve before the crystal was heated.

Before proceeding further, it should be noted that the energies of the Auger peaks quoted in this report correspond to the points of maximum negative slope in the $N(E)$ vs. E curves. Because the actual position of an Auger peak is at the point of zero slope, there is an error of a few electron volts in locating the peak; but since the energies and designations of the levels from x-ray data are not precise, this error is of little importance. In addition, there are small unknown errors due to chemical shifts in the levels and the contact potential between the sample and the suppressor grid. Also, no importance should be placed on the differences of absolute magnitude of the peaks from one curve to another. Since there was no way to accurately

Fig. 4. Energy distribution, $N(E)$ (solid line), and derivative of energy distribution, $dN(E)/dE$ (dashed line), for secondary electrons from (111) surface of UO_2 .

Fig. 5. Auger spectrum from (111) surface of UO_2 before the crystal was heated.

measure and control the primary current, only the relative magnitudes of peaks within a given curve are important.

Heating $\text{UO}_2(111)$ for 40 min at 790°C produced the spectrum shown in Fig. 6. Relative to the 525-eV oxygen peak, the maxima between 50 and 100 eV are now much more intense. The peak at 275 eV is smaller and has shifted to 280 eV. As shown later, there is an approximate correspondence between the four peaks at 70, 80, 90, and 100 eV and peaks from uranium. The peak at 280 eV may be due to carbon, but since it shifted from the usual carbon position of 275 eV and always had about the same relative intensity as the four low voltage peaks, it may be from uranium. This 280-eV peak was also observed on the clean surfaces of U and $\text{UO}_2(110)$, whereas the contaminated surfaces of these materials exhibited a 275-eV carbon peak. Because the resolution of the present apparatus is not good enough to distinguish between two close peaks, the identity of this peak is still in doubt.

After ion bombardment and annealing, the $\text{UO}_2(111)$ exhibited the same spectrum as that shown in Fig. 6. Once this spectrum was formed, it was very stable and no changes were produced by ion bombardment or heating.

When oxygen was adsorbed on the $\text{UO}_2(111)$ surface, the magnitudes of the four low voltage peaks relative to oxygen were decreased as shown in Fig. 7.

Fig. 6. Auger spectrum from (111) surface of UO_2 after heating for 40 min at 790°C .

Once this spectrum was formed by the oxygen exposure, it could not be converted back to that shown in Fig. 6 by either heating or ion bombardment. A possible explanation for this change would be the presence of some excess uranium at the surface of the $\text{UO}_2(111)$ which was later combined with oxygen to form the spectrum in Fig. 7.

The spectrum from the heated $\text{UO}_2(110)$ surface was identical to that shown in Fig. 7. When this surface was first studied, the five peaks below 300 eV were less intense relative to oxygen, but after sufficient heating to induce faceting, the low voltage peaks increased to the magnitude shown in Fig. 7. A spectrum similar to that shown in Fig. 6 was never attained on the (110) surface. Again, the magnitude of these low voltage peaks may be a function of the relative concentrations of uranium at the surface. The fractional-order beams reported for $\text{UO}_2(110)$ surfaces¹⁵ were also observed in the present experiment. Auger spectra for surfaces with these superstructures did not show any large amount of a particular contaminant. We concluded that the fractional-order beams were due to excess oxygen at the surface.

The Auger spectra from $\text{UO}_2(111)$ and (110) surfaces were studied for any evidence of the presence

Fig. 7. Auger spectrum from (111) surface of UO_2 after an O_2 exposure of 10^{-8} Torr-min and then heating for 3 min at 790°C to remove oxygen.

of fluorine at the surface. The major fluorine Auger peak should occur at approximately 690 eV; however, we observed no peak at this energy. Thus, if fluorine was present, it was in very small concentration below the detection threshold of our apparatus.

3.2 Uranium Metal

The Auger spectrum from a polycrystalline sample of uranium metal, after the system was baked but before the sample was heated, is shown in Fig. 8. This spectrum is very much like that of UO_2 before heating. The four peaks below 100 eV are present, as well as carbon and oxygen peaks. In addition, there is a small peak at 148 eV which can be attributed to sulfur. The resemblance of the spectrum to that of UO_2 was expected because the uranium was covered with UO_2 before heating.

Heating the uranium at 1000°C for 30 min produced the spectrum shown in Fig. 9. The oxygen peak decreased significantly and the relative magnitudes of the peaks below 100 eV were changed. The sulfur peak at 148 eV increased considerably, indicating diffusion of sulfur from the bulk.

Argon-ion bombardment (100 μ A-min, 500 eV) produced the spectrum shown in Fig. 10. The sulfur peak was almost extinguished and the peaks below 100 eV were decreased in magnitude. When the ion-bombarded uranium was annealed for 1 min at 1000°C the spectrum shown in Fig. 11 was produced. The

Fig. 9. Auger spectrum from polycrystalline uranium after heating for 30 min at 1000°C.

Fig. 10. Auger spectrum from polycrystalline uranium after argon-ion bombardment (100 μ A-min, 500 eV).

Fig. 8. Auger spectrum from polycrystalline uranium before the sample was heated.

Fig. 11. Auger spectrum from polycrystalline uranium after annealing for 1 min at 1000°C.

oxygen peak almost disappeared and the peak at 275 eV was shifted to 279 eV. Also, the peaks at 72 and 88 eV increased considerably. An interesting factor is the large sulfur peak produced by heating for only 1 min at 1000°C, indicating that sulfur diffused from the bulk very easily at this temperature and segregated at the surface. After another ion bombardment, the crystal was annealed at lower temperatures to determine the temperature at which the sulfur began to accumulate at the surface. A 5-min anneal at 700°C was sufficient to cause the sulfur peak to appear. The impurities present in the uranium sample are shown below. The source of

Impurities in Uranium Sample in ppm.

Li < 0.1	Ca < 2	Cu 4
Be < 0.1	V < 20	Mo < 25
B < 0.2	Cr 2	Sn < 1
Na 1	Mn 3	Ba < 5
Mg 40	Fe 20	C 70
Al 15	Co < 2	S < 20
Si 20	Ni 6	

sulfur was the bulk of the sample, even though analysis indicated that the sulfur concentration was less than 20 ppm. A residual-gas analysis of the background gases showed that no sulfur compounds were present. The segregation of sulfur at the surface of uranium is another example of what appears to be a common phenomenon in metals. Harris¹⁶ has observed this segregation of sulfur on nickel, copper, niobium, molybdenum, tantalum, tungsten, titanium, vanadium, iron, and stainless steel.

Since there are several hundred possible transitions in uranium which could give rise to Auger electrons, we made no attempt to correlate the observed peaks with x-ray transitions. The only evidence that indicated that the four low voltage peaks on UO₂(111), UO₂(110), and U were due to uranium was the close correspondence between the spectra from the three samples.

3.3 Graphite, Stainless Steel, and Niobium

Graphite, stainless steel, and niobium were studied to test the experimental setup for taking Auger spectra and to compare the results of our experiment with those of other investigators.

The Auger spectra from a polycrystalline sample of graphite after argon-ion bombardment and after

annealing for a few minutes at 800°C are shown in Fig. 12. The peak at 273 eV is the expected carbon peak. In the argon-ion-bombarded sample, the peak at 220 eV corresponds to the IMM transition in argon. Although it has been known for some time that a considerable amount of argon is left imbedded in a lattice after ion bombardment, an argon Auger peak has not been generally observed from the bombarded surfaces of other materials. For instance, no argon peak has been observed after bombardment of uranium or UO₂. The strong argon peak from graphite may indicate a long penetration depth and long escape depth for the Auger electrons in this material.

Figures 13 and 14 show the Auger spectra from argon-ion-bombarded and annealed samples of 300 series stainless steel. After ion bombardment, a large carbon peak at 275 eV was present, in addition to the high voltage peaks from the various metals in the alloy. This curve agreed quite closely with a curve published by Harris² for an alloy steel. The small shoulder near 220 eV was probably due to imbedded argon since it disappeared when the sample was heated. The major changes which occurred when the sample was heated at 800°C for 25 min are shown in Fig. 14. A very large sulfur peak at 150 eV appeared and the carbon peak at 275 eV was almost extinguished. Again, this is an example of segregation of sulfur at the surface of a metal. The sulfur peak first began to appear after only 2 min at 800°C and grew to a

Fig. 12. Auger spectra from argon-ion-bombarded (solid line) and annealed (dashed line) graphite.

Fig. 13. Auger spectra from argon-ion-bombarded (solid line) and slightly annealed (dashed line) stainless steel.

Fig. 14. Auger spectrum from stainless steel after annealing for 25 min at 800°C.

maximum after about 20 min.

Results similar to those from uranium and stainless steel were obtained from polycrystalline niobium. Figure 15 shows the niobium spectrum after argon-ion bombardment. The most prominent peaks are the 275 and 525 eV peaks from carbon and oxygen, respectively. Presumably the appearance of these peaks was due to CO or CO₂ adsorption. Figure 16 shows the niobium spectrum after annealing for 30 min at 1000°C. A fairly intense sulfur peak appeared at 149 eV; five other peaks below 200 eV are appar-

Fig. 15. Auger spectrum from polycrystalline niobium after argon-ion bombardment.

Fig. 16. Auger spectrum from polycrystalline niobium after annealing.

ently due to niobium. These five peaks were more intense than those on the curve from the ion-bombarded sample. The spectrum could be cycled back and forth between those shown in Figs. 15 and 16 by ion bombardment and annealing, respectively. However, after a few cycles the sulfur peak did not come back as rapidly as when the crystal was first studied, perhaps indicating that the bulk was being depleted of sulfur.

One interesting effect that occurred on both uranium and niobium was their inability to adsorb CO on the annealed surfaces. Exposing the samples to 10⁻⁵ Torr-min of CO had no significant effect on the Auger spectra shown on Figs. 11 and 16. However, after ion bombardment, there was always evidence of CO adsorption on the two surfaces. This lack of sensi-

tivity of the well-annealed surface to CO agrees with previous studies of some other metals, such as nickel and palladium.¹⁷ However, if nickel and palladium surfaces were carefully prepared to avoid any diffusion of contamination to the surface from the bulk, the sticking coefficient was quite high. The cause of the low sticking coefficient may be traced to the presence of sulfur on the annealed surface.

SUMMARY

LEED studies of UO_2 at LASL had previously indicated that a clean (111) surface could be produced, but that the (110) surface was nearly always partially covered with some substance which formed surface superstructures. Auger electron spectroscopy from the two UO_2 surfaces indicated that both surfaces were relatively free of foreign contaminants. The superstructures on the (110) surface appeared to be due to excess oxygen at the surface. Although both surfaces had been treated with HF before study in the LEED system, no evidence of fluorine contamination was found.

To determine which peaks in the UO_2 spectra were due to uranium, a sample of this metal was studied. This study indicated that the series of four peaks between 50 and 100 eV was characteristic of both uranium and UO_2 . An Auger peak at 280 eV appeared on both materials, but this peak was very close to the normal position of the carbon peak of 275 eV and could be due to a carbon transition with a chemical shift. Diffusion to the surface and segregation of sulfur was also observed on the uranium. Although argon-ion bombardment removed the layer of sulfur from the surface, annealing for 1 min at 1000°C covered the surface again. Exposures of the sulfur-covered surface to CO did not cause adsorption, indicating that the presence of sulfur decreased the sticking coefficient.

Auger studies of three other materials (graphite, stainless steel, and niobium) were carried out to test the equipment and compare results with those of other investigators. The spectrum from graphite contained only the carbon Auger peak. After argon-ion bombardment, however, a peak from a 220-eV transition in argon was also present, indicating that argon was imbedded in the graphite near the surface. Results from Auger studies of stainless steel and niobium

were similar to those of uranium. The ion-bombarded surface was covered with CO or CO_2 , and the annealed surface was covered with sulfur due to diffusion and segregation at the surface. The Auger peaks from the metals compared very well with those published by Harris.^{2,16}

The use of the LEED system for making a qualitative analysis of the materials present at the surface was very successful. Auger electron spectroscopy in combination with LEED data should greatly enhance the reliability of surface studies by allowing a more accurate and sensitive means of determining surface cleanliness. By making the changes in equipment suggested by Palmberg,¹² one should be able to increase the sensitivity by a factor of 100 over that obtained in our experiment.

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