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AUGER STUDY OF SURFACE CARBON AND OXYGEN ON THORIUM
FOLLOWING ION BOMBARDMENT

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ABSTRACT

The composition of a thorium metal surface has been monitored using Auger electron spectroscopy following Ar^+ bombardment at different temperatures. After extended Ar^+ bombardment, enough contaminated overlayers were removed to expose a surface region containing only thorium, bulk impurities, and imbedded argon. The main impurities, carbon and oxygen, differed in their behavior when the sample was annealed following bombardment. The amount of surface carbon either increased or remained constant during annealing depending upon the temperature of the sample during bombardment. The amount of surface oxygen decreased rapidly when the sample was heated above 500°C regardless of the sample temperature during bombardment. These experiments indicate that preparation of clean, annealed thorium surfaces requires ion bombardment at temperatures $\geq 400^\circ\text{C}$.

I. Introduction

The preparation of clean metal surfaces for surface studies often consists of argon ion bombardment followed by annealing of the sample. Argon ion bombardment is used to sputter away contaminated layers from the sample surface in order to expose a surface layer representative of the bulk material. Subsequent annealing serves two purposes by releasing implanted argon and by partly removing the surface damage caused by the ion beam. As a result of this procedure, the external impurities present on a metal surface can be eliminated. However, subsequent adsorption of gases or migration to the surface

of impurities present in the sample bulk may recontaminate the prepared surface. While the problem of gas adsorption can be reduced by maintaining an ultra-high vacuum environment around the sample, prevention of bulk to surface impurity migration is not always as straightforward. Even in extremely high purity samples, the remaining bulk contaminants may preferentially segregate to the surface and recontaminate it, especially at elevated temperatures.¹

One approach to reduce this impurity source is temperature control of the sample. Maintaining the sample at sufficiently low temperatures will reduce the diffusion rate of bulk impurities to the point that no migration to the surface occurs over reasonable periods. This approach, however, is incompatible with the annealing that is required following ion bombardment. An alternate method, which will be discussed in this paper, is to vary the temperature of the sample during ion bombardment so as to create a near surface region that contains implanted argon and is depleted in bulk contaminants. This method has been used to prepare a clean thorium metal surface. The major bulk contaminants in a sample of high purity thorium, carbon and oxygen, cannot be eliminated from the metal surface if the sample is kept at room temperature during argon ion bombardment. However, the amount of surface carbon and oxygen can be reduced to submonolayer quantities if the sample is heated during the bombardment.

II. Experimental

A wafer of high purity thorium metal that had been mechanically polished to a mirror finish was used as the starting material.^{2,3} Auger electron spectroscopy (AES) measurements of the surface composition of this wafer were made using a grazing incidence electron gun and a set of low-energy electron diffraction optics employed as a retarding field electron energy analyzer. The vacuum

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a residual gas analyzer attached to the vacuum chamber. The sample heating rate was $\sim 100^\circ\text{C}/\text{sec}$ and the maximum temperature attained was 700°C . The same sample was used in each experiment. Between runs, the sample was bombarded for at least two hours in order to remove the surface layers affected by the prior experiment.

III. Results and Discussion

Carbon and oxygen were the two main surface contaminants observed by AES. A representative spectrum of the sample surface is shown in Figure 1a. Surface carbon behaved differently than did oxygen when the sample was heated; its concentration either increased or remained unchanged depending upon the temperature of the sample during ion bombardment. If the sample was kept at room temperature during bombardment, the carbon AES signal intensity increased by 92% during annealing. When the sample was maintained at 400°C during bombardment, the carbon AES signal intensity decreased by 5%. The concentration of surface oxygen decreased when the sample was annealed. This decrease was observed both when the sample was bombarded at room temperature and at 400°C . This behavior is shown in Figures 2 and 3.

A residual gas analyzer attached to the vacuum chamber did not indicate that release of surface oxygen into the gas phase was responsible for the decrease in the surface oxygen concentration that occurred upon heating the thorium sample. Neither the O_2 nor the H_2O partial pressure in the chamber increased during sample annealing. An increase in the CO level was observed, but this was attributed to desorption occurring from the sample holder rather than from the sample itself because the carbon Auger signal intensity did not decrease in proportion to the oxygen signal decrease during sample heating.

The thorium Auger signal intensity was not significantly affected by the heating process. In each case, whether bombardment occurred at room

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The thorium Auger signal intensity was not significantly affected by the heating process. In each case, whether bombardment occurred at room

temperature or at 400°C, the thorium signal intensity decreased slightly with increasing temperature. Since the AES measurements indicated that the surface density of oxygen generally decreased during sample heating and that the surface density of carbon also decreased when the sample was annealed following bombardment at 400°C, it can be inferred that the reduction in the Th Auger signal intensity was not the result of signal attenuation by overlayer accumulation during the heating. If an increase in electron scattering accompanied heating of the sample, this process could be responsible for the observed temperature dependence of the thorium Auger signal intensity.

Release of implanted argon, which was imbedded during ion bombardment, was observed with the residual gas analyzer during sample annealing. The release occurred concurrently with the changes recorded in the surface density of carbon and oxygen. When the sample was heated at a rate of 100°C/sec, the maximum Ar release occurred at 553 and 643°C after bombardments at 25 and 400°C, respectively. The maximum rise in the partial pressure of Ar during annealing was 25% greater when the sample was maintained at 400°C during bombardment than when the sample was kept at room temperature. This result indicated that a greater amount of Ar was implanted at the higher temperature.

The surface composition of the thorium sample after annealing can be correlated with the amount of Ar implanted in the sample during bombardment. After annealing, the surface density of carbon and oxygen was smaller when increased amounts of Ar were implanted. It appears that the Ar implanted in the sample aided in producing a clean surface upon annealing. This suggests that implanted Ar effectively retarded recontamination of the surface by bulk impurities during annealing. The mechanism by which this is accomplished has not been established, but it is possible that the implanted Ar created a buffer region separating the surface layers from the bulk. The release of Ar

during the annealing may produce vacancies or alter interstitial sites in a manner that reduces effective segregation rate of bulk impurities to the surface.

The final surface composition of the thorium sample after ion bombardment at $>400^{\circ}\text{C}$ and annealing consisted primarily of thorium and contained only submonolayer quantities of carbon and oxygen, as shown in Figure 1b. This was evident from a comparison of the measured Auger signal intensities from these elements. Since the cross sections for Auger electron emission from carbon and oxygen are substantially greater than from thorium,⁵ the fact that the thorium Auger transitions had the largest signal intensities following the cleaning procedure indicated that thorium atoms predominated on the surface.

IV. Summary

Temperatures control during ion bombardment has been used to prepare a thorium surface containing only submonolayer quantities of carbon and oxygen. Implanted argon in the sample appeared to limit recontamination of the surface during annealing. These experiments indicated that the preparation of clean, annealed thorium surfaces requires ion bombardment at temperatures $\geq 400^{\circ}\text{C}$.

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Figure Captions

- Fig. 1a Auger spectrum of thorium following ion bombardment at room temperature and subsequent annealing.
- Fig. 1b Auger spectrum of thorium following ion bombardment at $>400^{\circ}\text{C}$.
- Fig. 2 Temperature dependence of carbon, oxygen, and thorium Auger signal intensities after ion bombardment at 25°C .
- Fig. 3 Temperature dependence of carbon, oxygen, and thorium Auger signal intensities after ion bombardment at 400°C .

Figure 1a

Figure 1b

Figure 2

Figure 3