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INFRARED AND STRUCTURAL ANALYSES OF
URANIUM OXIDATION PRODUCTS: PRELIMINARY RESULTS

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ABSTRACT

Water vapor oxidation of uranium follows a cyclic process: (1) rapid oxidation to form a thin dense, adherent layer of uranium dioxide, (2) slow diffusion of H_2O and H_2 through this oxide, until (3) a thickness of 0.2 to 0.3 nm is achieved in a strained state that (4) relaxes from the surface allowing further (1) contiguous oxide formation and finally (5) the shards of oxide comprised of oxide laminae spall completely free of the metal surface. The oxide thus formed is hydrated on the surface. Diffuse reflectance infrared Fourier transform spectroscopy shows that these are several distinct modes of water binding delineated with respect to energetics and reversibility. Scanning electron microscopy aids immensely in defining the nature of the laminae. Implications and suggestions for further work are given to aid in a better understanding of corrosion processes.

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INTRODUCTION

Uranium metal oxidizes readily in air and the mechanism and structure of the oxide product depends upon the environmental conditions that prevail.^{1 2} This preliminary report is the result of a study made at the Oak Ridge Y-12 Plant(a) to comprehensively define the oxidation mechanism in terms of the molecular entities involved in and/or produced in the oxidation processes. The project is designed to aid in other studies related to the kinetics and mechanisms of the oxidation processes.³

(a) Operated by the Union Carbide Corporation, Nuclear Division, for the Department of Energy.

EXPERIMENTAL

MATERIALS AND TECHNIQUES

The oxide used in this program was formed by water vapor oxidation of high purity uranium at 80°C. The black powder which spalled off of the metal ingot (1 cm x 1 cm x 10 cm) was collected in a Pyrex reaction chamber and transferred under pure argon to the infrared diffuse reflectance cell. The evacuable diffuse reflectance cell was designed and built by N. J. Harrick^(b). The cell will be described, along with some improvements, in a subsequent report.⁴ The spectra were transformed as the signal average of 2000 co-added interferograms (for each case shown in Figure 1) by using an infrared Fourier transform interferometric spectrometer. Several changes have been made in the original configuration of the instrumentation described in this report: the Data General Nova 2 computer has been replaced by a Nova 3 unit and a second Digilab Model 15C interferometer unit with a side-mounted GC/IR accessory has been added to run in tandem with the original Model 15B unit. A second 1.2 megaword Diablo disk drive has also been added to the system.⁵

The spectra are sequentially labeled for incrementally increased exposure to laboratory air (A through E) where E corresponds to full exposure through open valves to the cell. The intensity of the band at ca 2360 cm^{-1} is a measure of the amount of carbon dioxide gas in the chamber (and in turn a measure of the amount of water introduced from the ca 60% relative humidity of the laboratory). The spectrum labeled E (Figure 1) was acquired for the subsequent vacuum state.

INFRARED SPECTRAL ANALYSES

The integrated reflectance intensities for the bands labeled I, II, and IV are given in Table 1. Band I is due to a wide range of hydrogen-bonded hydroxyl (O-H) entities where the range of hydrogen bond strength gives rise to the broadband from 2400 to 3700 cm^{-1} .⁶⁻⁹ The complete lack of sharp vapor state bands at ca 3750 and 3650 cm^{-1} indicates that virtually all of the water introduced into the cell has been sorbed by the sample. Band II is a measure of the molecular water content as a result of the H-O-H bending mode of vibration which is noted at ca 1599 cm^{-1} for an isolated water molecule in the gas phase.¹⁰ This band is comprised of two components (1635 and 1565 cm^{-1}) equally displaced from the reference value 1599 cm^{-1} . This indicates that the molecular water is involved in two modes of hydrogen bonding: (1) electron donor from the oxygen (1635 cm^{-1}) and (2) proton donor from the hydrogen (1565 cm^{-1}).

The increase in water sorption for increasing exposure is noted in Figure 2, where the integrated intensity of Band I is linearly correlated to the carbon dioxide concentration. The results show markedly that the sorbed water can be categorized into two types: (a) reversibly sorbed water which can be

(b) Harrick Scientific Company, Ossining, N.Y.

Figure 1. Diffuse Reflectance Spectra of Uranium Oxide Powder. A through E were acquired for increasing extent of exposure to air (ca 65% relative humidity). R'_{∞} is the ratio of reflectance from the sample, $R(\text{samp})$, to the reflectance from a mirror, $R(\text{ref})$. Spectra F was acquired for the subsequent vacuum state.

Table 1

Integrated Intensities for Reflectance From
Uranium Dioxide Powder
(arbitrary units)

	CO ₂ (g)	I	II	IV
A	95.0	11083	1058	2113
B	181.0	11988	1545	2251
C	430.5	13689	1832	2193
D	623.5	14694	1866	2241
E	747.8	16816	1894	2042
F	16.6	10646	1480	2335

Integration Limits (cm ⁻¹)				
max	2400.0	3700	1750	1120
min	2275.0	2400	1450	940

removed in vacuum at room temperature and (b) energetically retained water that can be removed in vacuum. However the complex behavior of the intensity of Band II is indicative of a restructuring of the bound water accompanying the cyclical sorption-desorption. Such restructuring has been noted for water sorbed on other oxide systems such as catalysts¹¹, lunar soils¹², and thorium dioxide¹³. The general statistical and entropic analyses of such phenomena has been undertaken¹⁴. No attempt is made to resolve the two overlapping components of Band II. More detailed studies are underway to better define the nature of the restructuring of the bound water in terms of the relative amounts of the two bound species.

Comparative analyses of the spectra are shown in Figure 3 for Band I. These results show to a good approximation that the distribution of hydrogen bond types is virtually the same for the tightly bound water (vacuum state, Spectrum F), and the loosely bound water spectrums E through A, a measure of the vacuum labile component. There is also evidence that the oxide agglomerates are flexing in the sorption-desorption cycle as manifested in the altered "background scattering" of the electromagnetic radiation in the realm of 3650 to 4000 cm⁻¹. The major feature here is that the amount of sorbed water varies systematically with the exposure conditions but that the nature of the hydrogen bonds (as reflected in the perturbed O-H stretching frequency) is not grossly different. The distinguishing feature here is primarily noted in population and not in speciation.

In contrast, Bands II and III due to the H₂O bending are not as systematically variable and/or reversible as noted in Figures 2 and 4. Increased exposure does indeed lead to an enhanced sorbance indicative of an increase

Figure 2. Reflectance Relationships for Characteristic Bands for Infrared Spectra of Uranium Dioxide. Band notation is noted in Figure 1 and Table 1.

Figure 3. Stretching Frequency Band for Sorbed Water. Virtually no discernable difference is noted for the chemical species (hydrogen bond types) for the reversibly bound component.

Figure 4. Bending Frequency Band for Sorbed Water. Very similar environments are noted for both the inherent (bound) water and the reversibly bound water.

in species with little variation as to type (environment of the sorbed water). However, subsequent evacuation removes only a small portion of this ordered molecular water, and this component is that characterized by the vibrational frequency of ca 1635 cm^{-1} as shown in Figure 5. An alternate presentation is given in Figure 6 as an even more conclusive verification of the aforementioned mechanisms.

Band III seems to be indicative of a carbonate structure by analogy to the bands at 1356 and 1397 cm^{-1} observed for sodium carbonate aqueous solutions¹⁵. The insensitivity of these bands to varied exposure does not confirm this assignment and further investigation is warranted. Data are not available to eliminate the possibility that this band may be due to surface vibrational modes for these submicroscopic uranium dioxide crystallites¹⁶. The intensity variations for Band III virtually paralleling those of Band II (see Figure 4) leads us to suspect that this is a surface mode in a perturbed state (nonvacuum) involving an interaction of sorbed water with a surface oxide (or, more realistically, surface hydroxide) entity.

Band IV (Figure 7) is possibly due to solid-state vibrations for the UO_2 system (ca 765 cm^{-1} for matrix isolated UO_2) perturbed by the ligand fields of the solid. X-ray diffraction shows only weak broadbands characteristic of UO_2 in finely divided form. No indication of other oxides (i.e., U_3O_8) was detected for either the original material or after exposure to air. The intensity of Band IV is virtually independent of the exposure conditions as one would expect for UO_2 lattice vibrations. This is confirmation that the sorbed water is on the surface of the crystallites and/or in the interparticulate void space, and does not penetrate the substrate to form a bulk hydration entity. Furthermore, no alteration is noted from exposure to the oxygen in the air. This assignment is tentative in view of the 1017 cm^{-1} bands observed by others for bicarbonate and carbonate, respectively, in studies on the $\text{CO}_2\text{-H}_2\text{O}$ system¹⁵. In aqueous solutions there are also bands at 1600 and 1400 cm^{-1} which are not discernable in our spectra for the sorbed entities.

Band V (Figure 7) is most assuredly due to the U-O vibrational modes for bulk UO_2 . Matrix isolated UO_2 spectra¹⁷ show pronounced bands at 776 cm^{-1} . Corresponding results for ThO_2 molecules in solid argon matrix show bands¹⁸ at 786.8 , 734.5 , and 124 cm^{-1} . Bates¹⁹ has noted vibrational frequencies for single crystal ThO_2 at 735 , 877 , 1020 , and 1176 cm^{-1} . Further experimental and theoretical analyses are warranted to better define our system which is intermediate between the molecular state studied in matrix isolation and the classical state of "infinitely large" crystals.

MICROSCOPIC ANALYSES

Optical microscopic analyses show that the powder is composed of optically opaque angular fragments as shown in Figure 8. Reflected light shows that there are some facets that reflect an appreciable fraction of visible light. Electron microscopic analyses show that these angular fragments are composed of two different fine structures. Figure 9 sequence shows that the spall

Figure 5. Direct Comparative Analyses for the High Coverage (E) State and the Vacuum State (F). The labile water content is virtually all in the state characterized by the 1635 cm^{-1} vibrational mode.

B AND II

Figure 6. Indirect Comparative Analysis of the High Coverage (E) State and the Subsequent Vacuum State (F). The species desorbed are virtually entirely from the state characterized by the 1635 cm^{-1} vibrational mode.

Figure 7. Solid State Vibrational Bands for Uranium Dioxide. The virtual independence of frequency and intensity is consistent with the assignment of these bands to solid state modes.

Figure 8. Optical Micrographs of Uranium Dioxide. The black slivers of material are opaque to transmitted light. Light is reflected from facets revealing that the adequate order exists in these regions to preclude destructive interference in this wavelength (400 to 800 nm) regime. Photomicrograph was taken combined transmitted light (defining particle size and shape) and reflected light (defining the ordered facets with high reflectivity). Full scale = 965 μm .

Figure 9a. Scanning Electron Micrograph of Uranium Dioxide. The angular fragments vary in size and shape over quite a large range.
Full scale = 575 μm .

Figure 9b. Magnification of 9a. The striated nature of the fragments extend over quite a large scale. Full scale = 282 μm .

Figure 9c. Magnification of 9b. The striations often show discontinuities with interlacing for the larger splinters. Full scale = 57.5 μm .

Figure 9d. Magnification of 9c. Distorted amorphous material is seen to adhere to the outermost striated material. Full scale = 23.0 μm .

Figure 9e. Magnification of 9d. The amorphous material seen to be rather loosely adherent to the striated substrate. Full scale = 12.8 μm .

Figure 9f. Magnification of 9e. The amorphous component is noted to be composed of nodules or globules of uranium oxide. Full scale = 7.67 μm .

splinters have a somewhat striated composition with fine, open structure. Detailed analyses show that the rod-like structure is composed of rather loosely agglomerated nodules. Figure 10 depicts the other common morphology where a laminar structure is noted. The laminae are composed of extremely fine submicroscopic particles. One general conclusion is evident: The low-temperature process has formed extremely fine crystallites which comprise the larger shards that spall off of the parent contiguous oxide layer adhering to the metal surface. The reaction seems to proceed at an appreciable rate until the thickness of the oxide layer becomes great enough to inhibit the reaction and subsequent strain relief allows the more facile reaction to occur. The average laminae thickness of 0.2 to 0.3 μm is associated with the periodic nature of the oxidation process.

Figure 10a. Scanning Electron Micrographs of Uranium Dioxide. Angular fragments are seen to be comprised of laminar composites. Full scale = 575 μm .

Figure 10b. Magnification of 10a. Splinters, slabs, and blocks of the laminar structure are noted in the spallation products.
Full scale = 288 μm .

Figure 10c. Magnification of 10b. Details of the interplanar structure of a fragment are noted as contrasted to the interplanar edge structure. Full scale = 57.5 μm .

Figure 10d. Magnification of 10c. The stepped nature of the transplanar fracture is quite evident. Full scale = 23.0 μm .

Figure 10e. Magnification of 10d. The fine grain structure of the interplanar oxide is quite evident in this delaminating fracture zone. Full scale = 12.8 μm .

Figure 10f. Magnification of 10e. Fine structural features are noted contrasting three distinct morphologies: (1) Fine grained material in interplanar zones rather randomly agglomerated with compact zones surrounding irregularly shaped void regions; (2) Rather compacted linear regions on the splinter edges with relatively uniform periodicity in the translaminar coordinate; (3) Amorphous irregularly shaped, curvilinear fragments adhering to the splinter surfaces. Full scale = 7.67 μm .

DISCUSSIONS AND RECOMMENDATIONS FOR FURTHER STUDY

SPECTROSCOPIC ANALYSES

As noted in the experimental section, there is some ambiguity associated with the interpretation of the spectra. These can be readily resolved by systematic analytical procedures. For instance, Bands III and IV possibly arise from inadvertent exposure to CO_2 initially as reflected in Spectrum A. If indeed the carbonate species exist they must be rather strongly and intimately associated with the UO_2 surface species. Additional studies at higher temperatures will aid in the unambiguous interpretation. Solid-state vibrations are relatively insensitive to thermal desorption effects. Carbonate species are generally thermally labile and can be thermally desorbed. Surface vibrational modes of UO_2 will be thermally labile if and only if the thermal treatment involves sintering (crystal growth) which can and will be verified by microscopic and vapor sorption techniques.

The evaluation of the structure of the sorbed water is based on detailed evaluation of Bands I and II. Further details of the structure are forthcoming based on three additional experiments:

1. Thermal treatment to further evaluate the thermodynamics of water binding. This involves incorporation of a high-temperature control on the sample stage and detailed evaluation of both temperature and pressure effects on the concentration of the specific hydrogen-bonded entities. Subsequent experiments will involve pure water vapor (rather than air exposure) with a pressure transducer directly connected to the sample cell. Additional evaluation of synergetic relationships between water and carbon dioxide will be explored. In situ studies will also allow direct determinations of the kinetics and mechanisms of these reactions.
2. Exchange studies with deuterated water (D_2O) will further elucidate the nature of the water sorption-desorption reactions. Combined with the thermal and pressure variations these exchange reactions will aid in a better understanding of the kinetics, mechanisms, and thermodynamics.
3. Studies in the overtone (shorter wavelength) region have been an additional informative endeavor for many hydrogen-bonded systems. In addition to providing more information, such analyses will assure us that the broadband (2400 to 3700 cm^{-1}) is free of any combination bands and that this band truly represents a distribution of hydrogen band strengths due to the asymmetry association with the electrostatic field inherent at the UO_2 surface and/or micropores formed by juxtaposition of adjacent UO_2 surfaces.

STRUCTURAL ANALYSES

A comprehensive analysis of the system must include a concurrent microscopic analysis at the maximum resolution (magnification) attainable. The porous structure and particle size of the oxides will vary markedly for various preparative techniques and one must monitor alterations induced by thermal

treatment and/or substrate restructuring induced by sorption-desorption cycling. Crystallite size effects should be monitored also by diffraction techniques (line breadth analyses) to positively correlate grain size and particle size effects.

The quantitative evaluation of particle size effects and porosity effects can and will be monitored by inert gas sorption analyses as related to the chemical and structural nature of the oxide and its sorbed constituents. Details of the capabilities of the technique to investigate submicroscopic systems are described elsewhere.^{13,20,21} In addition, we will use refined mercury intrusion porosimetry measurement to help in defining the amount, size, and shape of pores inherent in the oxide, and any changes wrought in chemical and thermal treatments.²²

OTHER ANALYSES

As described earlier, this material was produced by water oxidation of high-purity metal. Our techniques are equally applicable to the study of the more practical systems of lower purity, uranium alloys, or strained systems. For instance Figure 11 shows a case of the modifications inherent in machining a surface. The roughness induced by the tool comprises a more reactive surface and small fragments of metal which adhere and are rapidly oxidized. The upper surface of the machined "curl" has been subjected to compressive stresses which induce strains that may or may not alter the metal reactivity. The effect of excess exposed surface can be modeled by the oxide formed by water oxidation of finely divided (powdered) uranium metal. The increased rate allows production of much more oxide for analysis in a conveniently reasonable period of time.

Additional studies are warranted for other metal-oxide systems. For instance oxidized thorium exists solely as ThO_2 with virtually no analog of U_3O_8 , etc. known to exist under the conditions of interest here. Other studies of oxides of multivalent character (i.e., FeO , Fe_2O_3 , Fe_3O_4 , etc.) may aid in understanding the U/O system as well as understanding the important iron-rusting problem itself.

In all cases, it will be beneficial to study the initial stages of oxidation of the metal to better understand the oxidation process per se. This can be done in the spectrometer cell which allows an in situ study of the oxidation that leads to the strain release process of spalling. Simultaneous sorption analyses and electron microscopy will also aid appreciably in this study.

Figure 11a. Electron Micrograph of Tooled Surface of Uranium Metal Turning. This underside of the metal turning shows the tool marks and some adherent fine oxide particles. Full scale = $443 \times 83 \mu\text{m}$.

Figure 11b. Electron Micrograph of Upper Surface of a Uranium Metal Turning. The compressive forces of the machining operation have led to a markedly distorted (folded) structure. Full scale = $880 \times 152 \mu\text{m}$.

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