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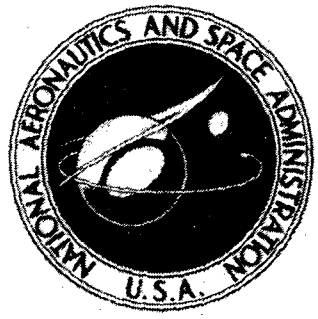

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OBSERVATION OF OXIDE PARTICLES BELOW THE APPARENT OXYGEN SOLUBILITY LIMIT IN TANTALUM

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION • WASHINGTON, D. C. • SEPTEMBER 1973

1. Report No.	2. Government Accession No.	3. Recipient's Catalog No.
NASA TM X-2889		

4. Title and Subtitle	5. Report Date
OBSERVATION OF OXIDE PARTICLES BELOW THE APPARENT OXYGEN SOLUBILITY LIMIT IN TANTALUM	September 1973
	6. Performing Organization Code
7. Author(s)	8. Performing Organization Report No.
Stephan Stecura	E-7530
	10. Work Unit No.
9. Performing Organization Name and Address	502-21
Lewis Research Center National Aeronautics and Space Administration Cleveland, Ohio 44135	11. Contract or Grant No.
	13. Type of Report and Period Covered
12. Sponsoring Agency Name and Address	Technical Memorandum
National Aeronautics and Space Administration Washington, D. C. 20546	14. Sponsoring Agency Code

15. Supplementary Notes

16. Abstract

The apparent solubility of oxygen in polycrystalline tantalum as determined by the X-ray diffraction lattice parameter technique is about 1.63 atomic percent at 820° C. However, oxide particles were identified in samples containing as low as 0.5 atomic percent of oxygen. These oxide particles were present at the grain boundaries and within the grains. The number of oxide particles increased with increasing oxygen concentration in tantalum. The presence of oxide particles suggests that the true solubility of oxygen in the polycrystalline tantalum metal is probably significantly lower than that reported in the literature.

17. Key Words (Suggested by Author(s))

Tantalum
Oxygen
Solubility
Oxides

18. Distribution Statement

Unclassified - unlimited

19. Security Classif. (of this report)	20. Security Classif. (of this page)	21. No. of Pages	22. Price*
Unclassified	Unclassified	17	$3.00

* For sale by the National Technical Information Service, Springfield, Virginia 22151

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SUMMARY

The distribution of oxygen in high purity polycrystalline tantalum containing up to 3.0 atomic percent of oxygen was studied by X-ray diffraction, microhardness measurements, extraction in methanol-bromine solution, polarized light optical microscopy, and electron microscopy.

X-ray diffraction and microhardness measurements indicated a uniform oxygen distribution in all oxygen-doped samples. From the X-ray diffraction lattice parameter study, it was concluded that the apparent solubility of oxygen in polycrystalline tantalum at 820° C is about 1.63 atomic percent. Neither method disclosed the presence of tantalum oxides on the surface or in the matrix of the tantalum metal.

However, tantalum oxides were extracted from the samples containing less than 1.63 atomic percent oxygen. The extracted oxides were identified by X-ray diffraction as Ta_6O, Ta_4O, Ta_2O, TaO_2, and Ta_2O_5. Particles assumed to be oxides were seen by electron microscopy in the samples containing about 0.5 atomic percent oxygen. Electron microscopy investigations further indicated that these particles were present at the grain boundaries and within the grains. Furthermore, investigations by polarized light optical microscopy indicated that the number of particles in tantalum increases with the increase of oxygen concentration in tantalum.

The presence of tantalum oxides below the oxygen solubility limits reported in the literature and determined in this study suggests that the true solubility of oxygen in the polycrystalline tantalum metal may be significantly lower than reported in the literature. Segregation of oxygen at the grain boundaries is believed to be primarily responsible for the observed behavior. The determination of the solubility of oxygen in the tantalum metal might be determined by the X-ray diffraction technique provided that the amount of oxygen combined in the form of oxides can be determined.

INTRODUCTION

The presence of oxygen in tantalum affects many of its properties. Thus the uses of tantalum may be limited or enhanced by the presence of dissolved oxygen. One case where the usefulness of the tantalum is detrimentally affected by oxygen is in high-temperature heat pipes. Tantalum has been used for heat pipes up to 1600° C with lithium as a working fluid. It has been reported that these heat pipes failed at high temperatures, the failures being attributed to the presence of oxygen in the tantalum (refs. 1 and 2). Tantalum has also been used to contain gettering agents such as yttrium in lithium in the T-111 tantalum alloy (Ta 8 wt. % W - 2 wt. % Hf) heat pipes.

A review of the literature showed that the reported solubility values for oxygen in tantalum vary. For example, the solubility of oxygen at 820° C has been reported at 2.8 atomic percent (ref. 3), 1.9 atomic percent (ref. 4), and 1.0 atomic percent (ref. 5). It is possible that these differences might be due to the presence of tantalum suboxides and/or oxides at these low oxygen concentrations. The presence of these suboxides or oxides would not be detected by the methods used in determining the oxygen solubility in polycrystalline tantalum if these oxides are present as widely dispersed, small particles. If such oxides are present in tantalum, they would affect not only the compatibility of tantalum with other metals but also such properties as ductility and electrical and thermal conductivities.

As part of a larger study involving lithium corrosion of oxygen-doped tantalum, the distribution of oxygen in tantalum was examined. This study discusses the distribution of oxygen in tantalum at 820° C. The data were obtained by X-ray diffraction, microhardness measurements, extraction in methanol-bromine solution, electron microscopy, and polarized light optical microscopy.

MATERIALS, APPARATUS, AND PROCEDURE

The data in this study were obtained as follows. High purity tantalum cups and coupons were doped with high purity oxygen to various oxygen concentration levels. These samples were then examined by chemical analyses, X-ray diffraction analyses, microhardness measurements, extraction in the methanol-bromine solutions, electron microscopy, and polarized light optical microscopy.

Materials

Tantalum cups were machined from high purity, 1.27-centimeter-diameter, polycrystalline tantalum rod and coupons from high-purity polycrystalline tantalum sheet.

Cups and coupons were cleaned in the manner described in reference 6. After cleaning, both cups and coupons were annealed at 1200° C for 2 hours at 10^{-8} torr pressure. Two randomly selected cups and four coupons were submitted for analyses of impurities. As indicated in table I, maximum total impurities in the tantalum metal were less than 0.05 weight percent. Oxygen, nitrogen, carbon, and hydrogen were determined by inert-gas fusion, modified Kjeldahl (ref. 7), combustion, and hydrogen extraction methods, respectively. Metallic impurities were determined by either mass or optical spectrographic methods. The original oxygen concentrations in the cups and coupons were about 12 and 50 weight ppm, respectively. Annealed cups and coupons were doped with oxygen to various oxygen concentration levels. After doping, some of the oxygen-doped cups and coupons were annealed at 1200° C for 2 hours then cooled to about 820° C and annealed at this temperature for 3 hours. The dimensions of the cups and coupons used in this study are given in figure 1.

The oxygen gas used in the doping experiments had the following impurities in parts per million by weight: nitrogen, 5; argon, <1; krypton, 8; xenon, <1; nitrous oxide (N_2O), <1; and water, <1.

Apparatus

The doping of tantalum cups and coupons was done in an apparatus described by Barrett (ref. 8). Normally, four cups or four coupons were doped at one time to a particular oxygen concentration level. The estimated accuracy of temperature measurement in this resistance heated quartz tube furnace was ±10° at 820° C.

Procedure

Tantalum cups or coupons were hung by platinum wires on a quartz sample holder and placed into the furnace. The furnace was evacuated to about 10^{-7} torr pressure and then slowly heated to 820° C. During heat up, the pressure in the furnace did not exceed 5×10^{-6} torr. After 1 hour at this temperature, oxygen was introduced into the furnace. All doping runs were conducted at 2×10^{-4} to 3×10^{-4} torr oxygen pressure.

After completing the doping experiments, half of the samples were annealed at 1200° C for 2 hours at 10^{-8} torr pressure followed by annealing at 820° C for 3 hours. The rest of the samples were evaluated in the as-doped condition. Afterward, some of the samples were subjected to X-ray diffraction analyses, microhardness measurements, extraction in methanol-bromine solution, electron microscopy, and polarized light optical microscopy.

RESULTS AND DISCUSSION

To determine the presence of oxides in tantalum, samples were doped at 820° C to various oxygen concentration levels. The oxygen concentration levels varied between 0.02 atomic percent of oxygen in as-received tantalum to about 3.0 atomic percent. The oxygen concentrations were determined both by weighing the samples before and after doping with oxygen and by inert gas fusion analysis. The differences between the oxygen concentrations as determined by the two methods were negligible (table II). The data in table II also show that the carbon concentrations did not change with the increase in the oxygen concentration in the samples. However, the nitrogen concentrations in the samples increased slightly with the increase in oxygen concentrations.

The X-ray diffraction data confirmed that no oxide layers were formed on the surfaces of any of the samples. Complete X-ray diffraction patterns obtained of the oxygen-doped sample surfaces showed that only tantalum X-ray diffraction peaks were present. The calculated lattice parameter values for 211, 310, and 321 Miller indices showed about the same increase due to increase of oxygen concentration in the tantalum. This then indicates that there was no preferential distribution of oxygen in the tantalum lattice.

The X-ray data in figure 2 show a break in the lattice parameter against oxygen content curve at about 1.63 atomic percent of oxygen. This value should be the solubility value for oxygen in tantalum at 820° C. The 1.63 atomic percent value is in very good agreement with the solubility data reported by Gebhard (ref. 4), but is in disagreement with the solubility values reported in references 3 and 5 (fig. 3). The data in figure 2 also show that the annealing of oxygen-doped samples at 820° C had no significant effect on the lattice parameter values, indicating that an equilibrium condition in the samples was established during doping with oxygen.

From the data in figure 2 it can be concluded that tantalum suboxides and/or oxides should have been formed above 1.63 atomic percent oxygen level. However, no such oxides were detected by the X-ray diffraction technique. This could be due to the small amount of oxide phase present. Since the presence of oxides above 1.63 atomic percent of oxygen was not detected, it is also possible that tantalum oxides might have been formed below this value but were not detected. This implies that the true solubility of oxygen in tantalum could be lower than reported in references 3 to 5 and determined in this study.

The absence of tantalum oxides in these oxygen-doped samples is also inferred from the microhardness data (fig. 4). Microhardness measurements were used by Kofstad (ref. 9) in support of X-ray diffraction to establish a uniform distribution of oxygen in tantalum. Brehm (ref. 10) used microhardness measurements to determine the distribution and concentration of oxygen in niobium. The microhardness was observed in this study to increase linearly with the increase in oxygen concentration (fig. 4). These data

are about the same as those reported in reference 11. This linearity is observed even in samples in which the oxygen concentrations have exceeded the apparent solubility limit. Furthermore, it was found that annealing samples at about 820° C for 3 hours at 10^{-8} torr pressure had no significant effect on the microhardness of oxygen-doped tantalum. The X-ray diffraction and microhardness data indicate that the oxygen was probably distributed uniformily since no oxides were detected. However, the fact that no oxides were detected at concentrations above the solubility limit of oxygen in tantalum suggests that the oxides were present in very small amounts and as very small particles.

To resolve this problem it was decided to extract the oxides, if they were present, in a methanol-bromine solution and also to study these oxygen-doped samples by electron microscopy and polarized light optical microscopy. Six tantalum samples containing about 0.02, 0.5, 0.7, 1.2, 1.4, and 1.9 atomic percent oxygen were dissolved in methanol - 15-volume-percent bromine solutions. No residue was observed from the sample containing 0.02 atomic percent oxygen. Although residues were observed from the samples containing 0.5 and 0.7 atomic percent oxygen, the amounts of residues were insufficient for X-ray difraction analysis. Sufficient amounts of residues were recovered from the remaining three samples. The amount of residue was greater for the sample containing 1.9 atomic percent oxygen than for the samples containing 1.4 and 1.2 atomic percent oxygen.

The results of optical spectrographic analyses of the 1.2, 1.4, and 1.9 atomic percent oxygen sample residues showed that the residues contained tantalum. The recovered residues were identified by X-ray diffraction analyses on the basis of the X-ray diffraction data reported in references 12 to 16. The suboxides and oxides found in these residues were Ta_6O, Ta_4O, Ta_2O, TaO_2, and Ta_2O_5. The X-ray diffraction data for 1.4 and 1.9 atomic percent oxygen sample residues are given in table III. The X-ray diffraction data for the 1.2 atomic percent oxygen sample residue were similar to those from the 1.4 atomic percent oxygen sample residue. The data in table III also show that only the strongest X-ray diffraction peaks for various tantalum oxides were observed.

The recovery of these oxides from samples containing less than 1.63 atomic percent oxygen suggests that the true solubility in tantalum at 820° C is less than 1.63 atomic percent. Furthermore, the recovery of these oxides points out that the usefulness of the X-ray diffraction technique in determining the true solubility of oxygen in the polycrystalline tantalum is very limited.

The presence of oxides below 1.63 atomic percent of oxygen is also supported by electron and optical microscopy observations. The four samples investigated contained 0.02, 0.5, 0.92, and 1.9 atomic percent of oxygen, respectively. Particles were observed by electron microscopy in all samples and are believed to be tantalum oxides, based on the identification of oxides in the extracted residues. In the tantalum sample containing 0.02 atomic percent of oxygen, only one small particle was found after a very

extensive and careful investigation of most of this sample. Many particles were found in the sample containing 0.5 atomic percent of oxygen. The number of particles increased with the increase of the oxygen concentration in tantalum. Optical microscopy observations gave similar results as is apparent from the photomicrographs shown in figures 5 to 7. These photomicrographs were obtained by polarized light optical microscopy. The photomicrograph of the sample containing 0.02 atomic percent of oxygen shows no second phase (fig. 5). The photomicrograph (fig. 6) of the 0.5 atomic percent oxygen sample shows second phase, represented by the white dots in the photomicrograph. The number of white dots is even greater in the photomicrograph of the sample containing 1.9 atomic percent of oxygen (fig. 7). Thus, the polarized light optical microscopy observations confirm that the number of particles increases with the increase of oxygen concentration in tantalum.

The electron microscopy investigation also indicated that in the sample containing 0.50 atomic percent oxygen, most of the oxide particles were present at the grain boundaries. Only a few particles were found within the grains. All of these particles were similar to the particles shown in figure 8. Similar oxide particles were observed in the samples containing 0.92 and 1.9 atomic percent oxygen. It was observed that as the oxygen concentration increased the number of oxide particles within the grains increased. Furthermore, larger particles were observed with increasing oxygen concentration. This is readily apparent from a comparison of the particles shown in figure 8 with those shown in figures 9 and 10.

The presence of oxide particles in the tantalum sample containing about 0.50 atomic percent of oxygen suggests that the true solubility of oxygen in tantalum metal at 820° C is significantly lower than indicated by the lattice parameter measurements in this study and reported in references 3 to 5. Indeed, it might be concluded that the true solubility of oxygen in polycrystalline tantalum at all temperatures is significantly lower than reported in references 3 to 5.

The extraction of oxides, electron microscopy, and polarized light optical microscopy investigations suggest that it would be very difficult to determine the true solubility, distribution, and concentration of oxygen in polycrystalline tantalum by X-ray diffraction and microhardness measurements. Both of these methods have been previously used for such purposes (refs. 3, 4, 9, 10, 17, and 18). The X-ray diffraction method could be used to determine the true solubility of oxygen in polycrystalline tantalum provided a method is found for determining quantitatively the amount of oxygen in the form of oxides. The use of single crystals would eliminate grain boundaries and should make it possible to obtain oxygen solubility values that would be closer to the true solubility values.

CONCLUSIONS

High purity polycrystalline tantalum samples were doped with oxygen at 820° C up to 3.0 atomic percent level. These samples were studied by X-ray diffraction, microhardness measurements, extraction in methanol-bromine solution, electron microscopy, and polarized light optical microscopy.

It was determined that the apparent solubility of oxygen in polycrystalline tantalum at 820° C is about 1.63 atomic percent, which is in good agreement with the values reported in the literature. However, this is not the true solubility of oxygen in tantalum because tantalum oxide particles were found at oxygen concentrations as low as 0.5 atomic percent. Segregation of oxygen to grain boundaries is believed to be primarily responsible for the observed behavior. The true lattice solubility of oxygen in tantalum is significantly lower than that reported in the literature and probably cannot be determined by using polycrystalline samples.

The formation of small oxide particles at the grain boundaries and within the grains limits the usefulness of the X-ray diffraction technique in determining the true solubility of oxygen in polycrystalline metals.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, June 20, 1973,
502-21.

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TABLE I. - IMPURITY CONTENT OF TANTALUM SAMPLES

Impurity	Concentration of impurity, ppm by weight		Impurity	Concentration of impurity, ppm by weight	
	Cup	Coupon		Cup	Coupon
Carbon[a]	5	22	Lithium	<1	<1
Oxygen[a]	15	50	Magnesium	<1	<1
Nitrogen[a]	22	18	Manganese	<1	<1
Hydrogen[a]	5	6	Molybdenum	10	15
Aluminum	<1	<1	Nickel	20	20
Antimony	<1	<1	Niobium	100	100
Arsenic	<1	<1	Osmium	<1	<1
Barium	<1	<1	Palladium	<1	<1
Berilium	<1	<1	Platinum	10	10
Bismuth	<1	<1	Potassium	10	10
Boron	<1	<1	Rhenium	---	---
Bromine	<1	<1	Rhodium	<1	<1
Cadmium	<1	<1	Rubidium	<1	<1
Calcium	<1	<1	Ruthenium	<1	<1
Cesium	<1	<1	Silicon	5	10
Chlorine	10	15	Silver	<1	<1
Chromium	5	8	Sodium	<1	<1
Cobalt	<1	<1	Strontium	<1	<1
Copper	5	5	Tantalum	High	High
Fluorine	5	5	Tellurium	1	<1
Gallium	<1	<1	Thalium	<1	<1
Germanium	<1	<1	Thorium	50	50
Gold	20	20	Tin	<1	<1
Hafnium	<1	5	Titanium	8	10
Indium	<1	<1	Tungsten[a]	4	5
Iodine	<1	<1	Vanadium	<1	<1
Iridium	<1	<1	Zinc	3	5
Iron	20	30	Zirconium	<1	<1
Lead	<1	<1			

[a]Results obtained by chemical analysis. All other data were obtained by optical spectrographic analyses.

TABLE II. - OXYGEN, NITROGEN, AND CARBON CONCENTRATIONS IN TANTALUM BEFORE AND AFTER DOPING WITH OXYGEN

Sample number	Concentration, at. %			
	Oxygen		Nitrogen[c]	Carbon[d]
	By weight difference[a]	By chemical analysis[b]		
[e]Ta-1	-----	0.020	0.028	0.008
[e]Ta-2	-----	.060	.023	.033
Ta-3	0.188	.202	.045	.029
Ta-4	.375	.402	-----	-----
Ta-5	.438	.462	.072	.024
Ta-6	.738	.774	-----	-----
Ta-7	1.406	1.446	.079	.018
Ta-8	2.398	2.350	.089	.015

[a]Determined by weighing the sample before and after the oxygen doping.

[b]Determined by inert-gas fusion method.

[c]Determined by a modified Kjeldahl method (ref. 7).

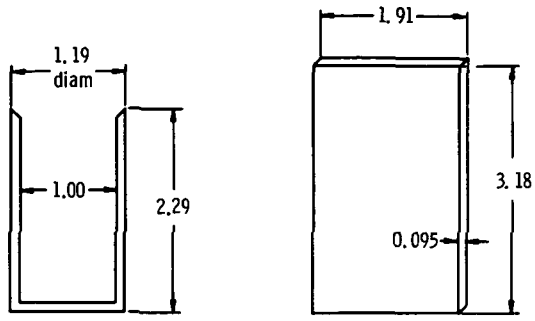
[d]Determined by combustion method.

[e]Ta-1 is an undoped crucible and Ta-2 is an undoped coupon.

TABLE III. - X-RAY DIFFRACTION PATTERNS FOR THE RESIDUES EXTRACTED FROM OXYGEN-DOPED TANTALUM IN METHANOL-BROMINE SOLUTIONS

1.4 at. % oxygen			1.9 at. % oxygen		
d-spacing, m	Intensity (a)	Compound	d-spacing, m	Intensity (a)	Compound
4.72×10^{-10}	M	Ta_6O or TaO_2	4.72×10^{-10}	VW	Ta_6O or TaO_2
3.870	VW	Ta_2O_5	3.870	W	Ta_2O_5
3.600	VW	Ta_4O	3.149	W	Ta_2O_5
2.722	VS	Ta_2O	2.721	VS	Ta_2O
2.579	VW	TaO_2	2.600	W	TaO_2
2.446	VVW	Ta_2O_5	2.447	W	Ta_2O_5
2.395	M	Ta_4O or Ta_6O	2.394	W	Ta_4O or Ta_6O
2.354	M	Ta_2O	2.355	M	Ta_2O
2.327	W	Ta_6O	2.286	W	Ta_4O
2.288	W	Ta_4O	2.111	W	Ta_2O
2.110	VW	Ta_2O	1.928	W	Ta_2O
1.929	W	Ta_2O	1.795	S	Ta_4O
1.795	S	Ta_4O	1.668	S	Ta_2O
1.674	S	Ta_2O or Ta_6O	1.653	W	Ta_2O_5
1.654	VW	Ta_2O_5	1.576	S Ta	Ta_2O_5
1.574	M	Ta_2O or Ta_2O_5	1.423	W	Ta_2O
1.424	VW	Ta_2O	1.398	W	TaO_2
1.403	W	Ta_4O	1.366	W	TaO_2 or Ta_6O
1.364	W	Ta_6O or TaO_2	1.335	VW	Ta_4O
1.339	VW	Ta_6O	1.299	W	Ta_2O_5
1.058	VW	Ta_6O	1.114	W	Ta_2O

[a]Symbols: M = medium; S = strong; V = very; and W = weak.



(a) Cup. (b) Coupon.

Figure 1. - Dimensions of samples used in this study (all measurements are in cm).

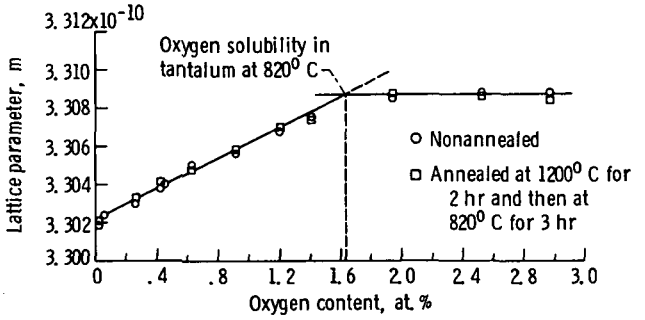


Figure 2. - Tantalum lattice parameter as function of oxygen content.

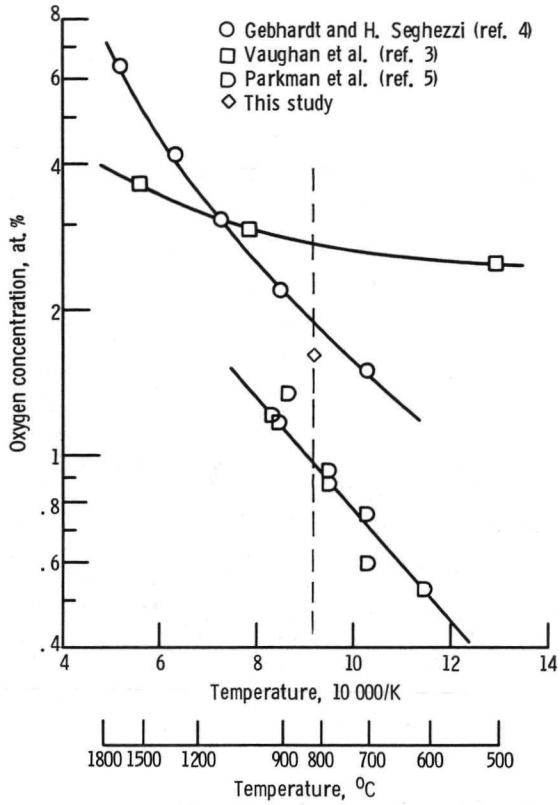


Figure 3. - Solubility of oxygen in tantalum.

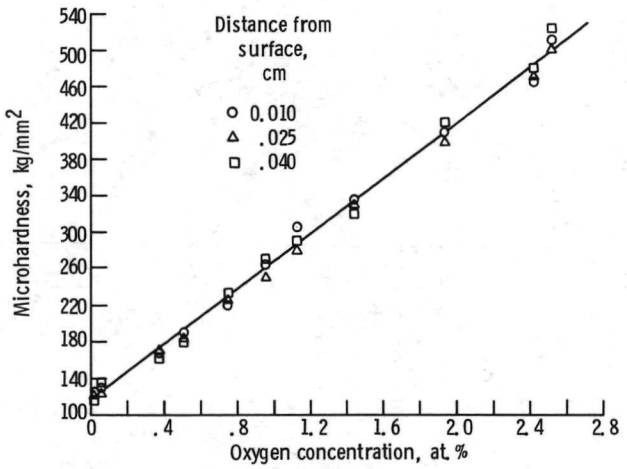


Figure 4. - Cross sectional microhardness of 0.095-centimeter-thick, unannealed, oxygen-doped tantalum.

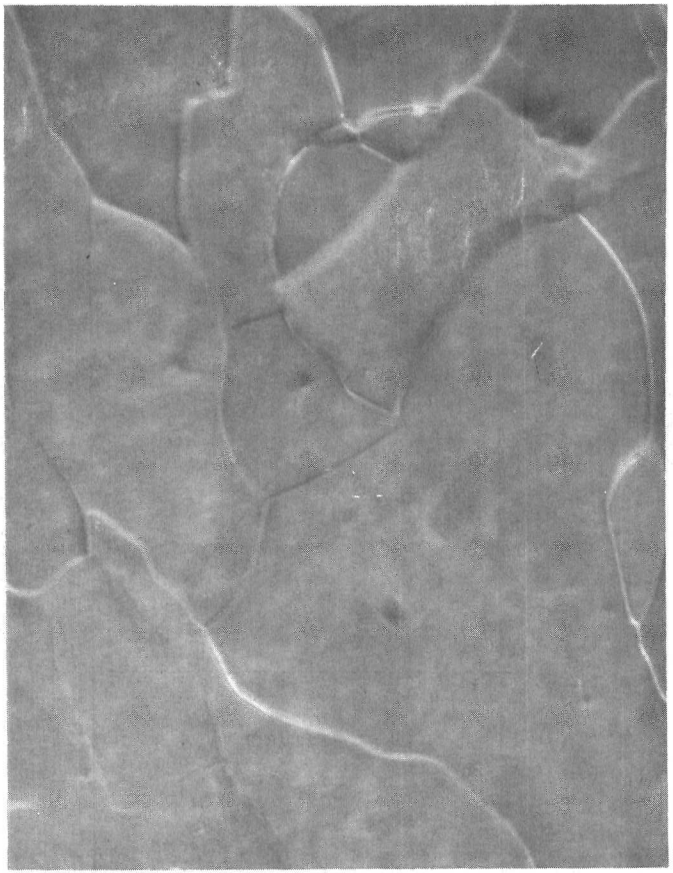


Figure 5. - Optical-microscope-polarized light photomicrograph of tantalum containing 0.02 atomic percent oxygen. X1000.

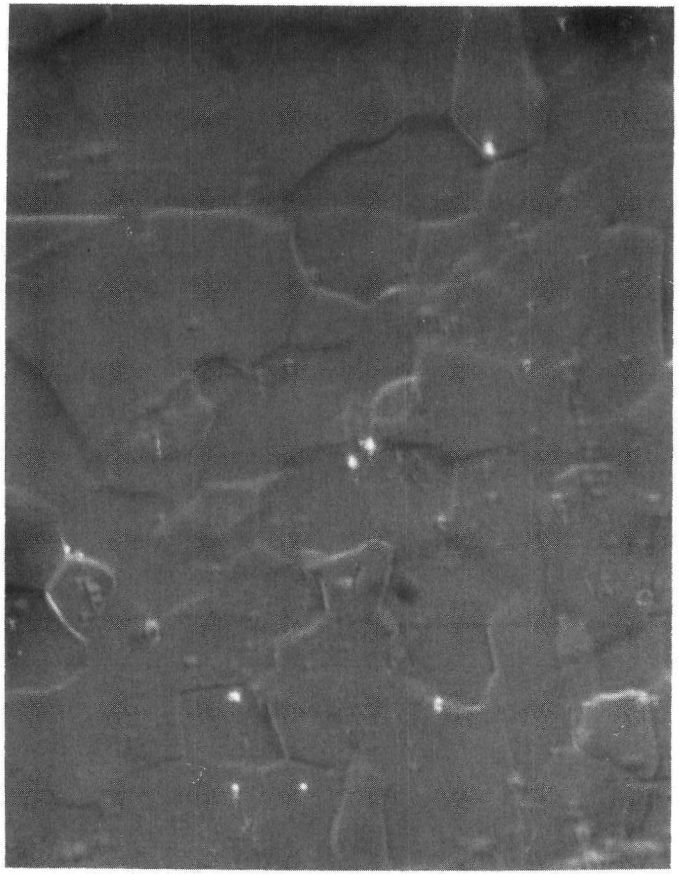


Figure 6. - Optical-microscope-polarized light photomicrograph of tantalum containing 0.5 atomic percent oxygen. X1000.

Figure 7. - Optical-microscope-polarized light photomicrograph of tantalum containing 1.9 atomic percent oxygen. X1000.

Figure 8. - Electron transmission photomicrograph of oxide particles found at the grain boundary and within the grain in tantalum containing 0.50 atomic percent oxygen. X28 000.

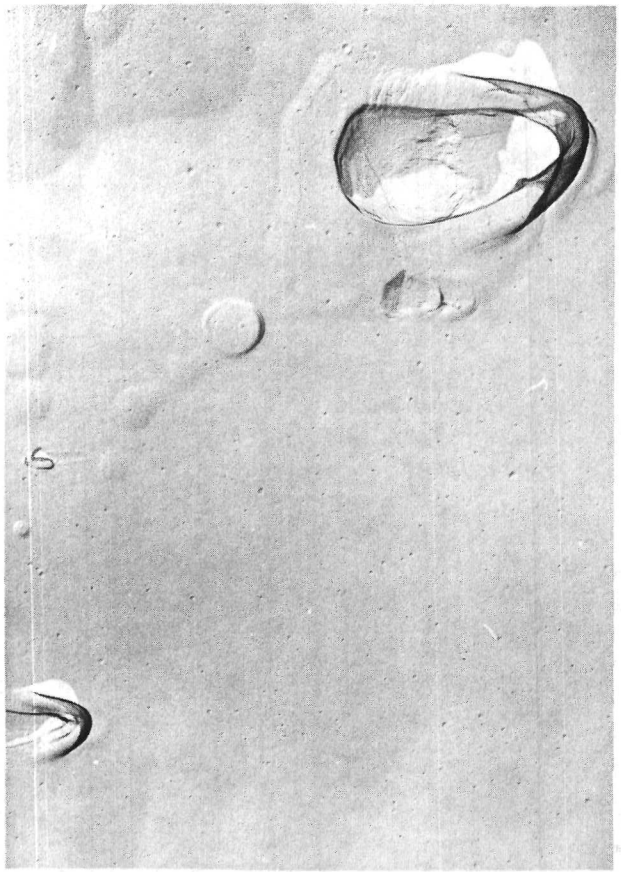


Figure 9. - Electron transmission photomicrograph of oxide particles in tantalum containing 0.92 atomic percent oxygen. X28 000.

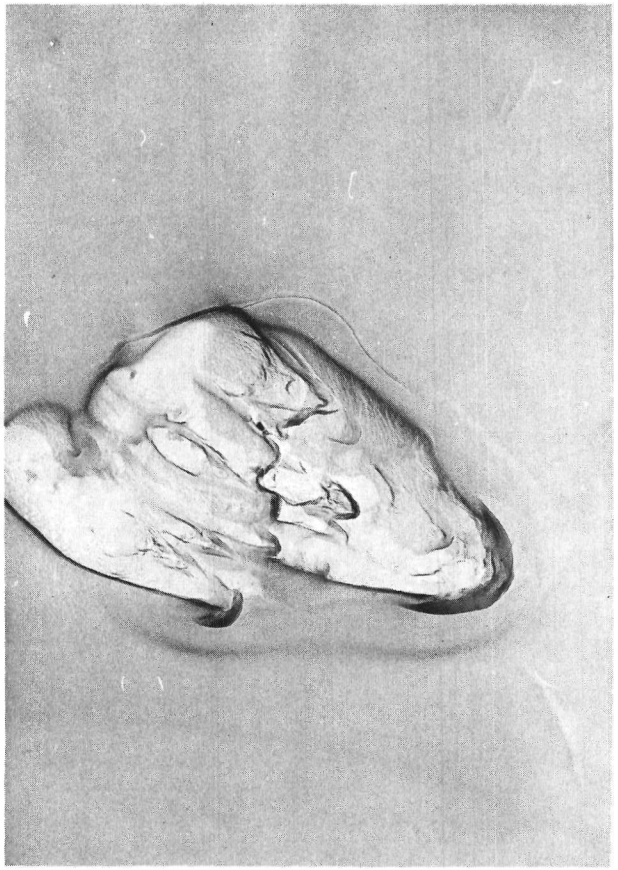


Figure 10. - Electron transmission photomicrograph of an oxide particle in tantalum containing 1.9 atomic percent oxygen. X28 000.

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