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Conf-91132--15

PNL-SA--19598

DE92 006489

HIGH RATE SPUTTER DEPOSITION
OF WEAR RESISTANT TANTALUM
COATINGS

D. W. Matson
M. D. Merz
E. D. McClanahan

November 1991

Presented at the
38th Annual AVS Symposium and
Topical Conference
November 11-15, 1991
Seattle, Washington

Work supported by
the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830

Pacific Northwest Laboratory
Richland, Washington 99352

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ABSTRACT

The refractory nature and high ductility of body centered cubic (bcc) phase tantalum makes it a suitable material for corrosion- and wear-resistant coatings on surfaces which are subjected to high stresses and harsh chemical and erosive environments. Sputter deposition can produce thick tantalum films but is prone to forming the brittle tetragonal beta phase of this material. Efforts aimed at forming thick bcc phase tantalum coatings in both flat plate and cylindrical geometries by high-rate triode sputtering methods are discussed. In addition to substrate temperature, the bcc-to-beta phase ratio in sputtered tantalum coatings is shown to be sensitive to other substrate surface effects.

I. INTRODUCTION

Tantalum metal, because of its refractory nature and high ductility, is being investigated as a protective material for the inner surfaces of cylindrical reactors exposed to high velocity gases at elevated temperatures and pressures. An attractive approach to applying tantalum coatings on cylindrical surfaces is sputter deposition. Two other common processes used to produce coatings of this material, chemical vapor deposition and electrodeposition from fused salt baths, typically involve heating of the substrate to temperatures of 800°C or higher. Consequently, these methods have the potential disadvantage of exceeding the tempering temperature of steel substrates. Sputtering does not require substrate heating to form the coating, and heat produced by the process itself (e.g., from exposure to the plasma discharge and kinetic energy of the sputtered neutrals) can be removed from thermally conductive metal substrates by external cooling.

Sputtered tantalum is typically observed to be deposited in one of two distinct crystallographic forms.^{1,2} The body centered cubic (bcc) structure, which is the

form of bulk tantalum, has the physical properties that make it attractive as a material for protective coatings. A metastable second phase (referred to as beta tantalum) was identified in sputtered tantalum films over 25 years ago.¹ The beta phase has a tetragonal structure and distinctly different physical and electrical properties from those of bulk tantalum. Beta tantalum spontaneously reverts to the bcc phase at temperatures between 750 and 800°C. Sputtered tantalum deposits typically contain a mixture of the two phases.

Primarily as a result of interest in thin (<1 micron) tantalum films for use in electronics applications, considerable effort has been made to identify the specific conditions required to control the formation of one sputtered tantalum phase preferentially to the other.²⁻⁴ A number of processing parameters have been identified which appear to influence the structure of sputtered tantalum. These include the sputtering gas purity,^{1,5-12} the nature of the substrate surface,^{3,4,10,13-15} substrate bias during deposition,^{8,11,12,16} length of presputtering time,^{6,11-13} and the substrate temperature.^{5,17} There remains, however, considerable controversy regarding the specific effects of each of these variables, primarily because of the difficulty in fully isolating each variable from the effects of all others. Also, little or no work has been done to investigate the phase characteristics of sputtered tantalum films greater than one micron in thickness.

Our goal in the present work was to investigate and identify conditions required to sputter deposit thick (>25 microns), adherent, low defect coatings of bcc tantalum at high rates onto interior surfaces of cylindrical substrates.

II. EXPERIMENTAL

Coating Deposition

Two demountable high-rate triode sputtering systems having flat plate and cylindrical sputtering geometries were used to deposit tantalum onto metal substrates

to evaluate the effects of sputtering conditions on the formation of bcc and beta tantalum in 35 to 130 micron thick coatings. Both systems were typically pumped to between 1.0 and 4.0×10^{-7} torr using a liquid nitrogen trapped, oil diffusion pumped vacuum system and were helium leak tested prior to operation. Krypton (99.99%) at a 3×10^{-3} torr pressure was used as the sputtering gas in all runs.

The first experiments used a flat plate to flat plate configuration to deposit approximately 130 micron thick films on substrate coupons. The tantalum targets used for these experiments were fabricated from bar stock (99.9% Ta) and were circular in shape (42 mm dia.) with a smaller diameter boss on the underside which was closely fitted to a water cooled heat sink. 25 mm diameter substrates were machined from AISI 4340 steel bar stock and were welded to a cooling jacket. The target to substrate gap was roughly 19 mm.

The substrate coupons used for the flat plate experiments were polished on a Lapmaster with alumina No. 1700, followed by 6 micron and 0.25 micron diamond paste. Ultrasonic cleaning in isopropyl alcohol and an alcohol rinse preceded installation of the substrates in the sputtering chamber. A thermocouple was spot welded to the back side of the substrates to monitor temperatures during deposition. Warm water was circulated behind the substrates for deposition temperatures below 100°C and was replaced by nitrogen gas for deposits made at 350°C and 450°C .

After establishing the krypton pressure, a thermionic discharge was ignited and maintained at 20 amps for 30 minutes by applying a 40 V potential between the heated tungsten filament and the anode. The substrate was then ion etched at a current density of 6 mA/cm^2 for 5 minutes by applying a -100 V potential. On completion of the etch, the target potential was increased to -1500 V and the target current density was raised to 15 mA/cm^2 by increasing the electron flow in the discharge. The typical tantalum deposition rate for this system was 0.6 microns per minute.

The second set of experiments were performed using a cylindrical sputtering apparatus shown schematically in Figure 1. This system was specially designed for coating interior surfaces of 25 mm internal diameter right circular cylinders. Substrates used for the cylindrical sputtering apparatus were fabricated from 4340 steel or Incoloy-909 (International Nickel Corp.) stock and were roughly 21.3 cm long. The tantalum sputtering target, which consisted of 0.965 cm O.D. by 0.15 cm wall thickness tantalum tubing (99.98%, NRC, Inc., Newton, MA) ran along the axis of the substrate cylinder. Substrates were slip fit within a water-cooled aluminum holder that constituted the outer wall of the vacuum chamber.

The difference between the inside diameter of the aluminum holder and the outside diameter of the substrate cylinder was calculated on the basis of thermal expansion data for the appropriate materials such that the substrate temperature could be limited to a pre-determined temperature range during sputter deposition. Once the substrate had heated and expanded to the point where a thermal equilibrium was established between the heat input by the sputtering process and that removed through the cooling jacket, the substrate temperature could be maintained to within a few degrees Celsius during sputtering operation. Substrate temperatures were continuously monitored using a spring loaded thermocouple fed through the holder wall with a 1/16 inch Swagelok fitting outfitted with PTFE ferrules. The thermocouple tip was inserted into a shallow hole drilled in the outside surface of the substrate cylinder.

A thermionic discharge was ignited and raised to between 20 and 25 amps over a 30 minute period by applying a potential of 60 volts between the heated tantalum ribbon filament and the anode. The discharge was maintained at that current level for 30 to 90 minutes to raise the substrate to its equilibrium temperature as determined by the substrate/holder gap (for deposits made at 175 to 225°C), or to some intermediate temperature for runs in which the deposits were being made at

higher substrate temperatures. Upon completion of the substrate heating by exposure to the plasma discharge, the substrate was etched by ion bombardment at between -100V and -200V and an ion current density of 5 to 10 mA/cm². Immediately following the substrate etch, the target voltage was raised to -1500 V over a 5 minute period while maintaining the substrate bias, and the plasma current was adjusted to attain the desired target current density. During coating runs in which substrate temperatures in excess of 400°C were used, the substrate temperature continued to rise during the substrate etch and initial tantalum deposit until the point at which thermal equilibrium was attained. Deposition rates on the order of 0.3 microns/min were typical for the cylindrical sputtering system.

Coating Evaluation

Structural evaluation of selected tantalum deposits were made with scanning electron microscopy (SEM) and x-ray diffraction on the growth surfaces using copper K-alpha radiation and a Phillips diffractometer. Microhardness measurements were made on both transverse and growth surfaces of selected samples with loads varying between 50 and 500 grams. Optical metallography was performed on transverse sections of the coatings for the purpose of measuring deposit thicknesses, obtaining a qualitative evaluation of the phase distribution, and for studying grain size, shape, and growth defects. Trace impurities were analyzed with a Kevex x-ray energy spectrometer and a Materials Analysis Corp. electron microprobe.

III. RESULTS

In general, the tantalum deposits produced in both sets of experiments had a bright metallic appearance but most were roughened as a result of protruding growth defects on the coating surface. With two exceptions noted below, all of the

tantalum deposits remained bonded to the substrate surfaces after sectioning the samples with a silicon carbide cut off wheel.

Flat Plate Deposits

A summary of the sputter deposition parameters used to produce the six coatings using the flat plate sputtering apparatus, and the results of surface phase analysis and coating microhardness values are summarized in Table 1.

Photomicrographs showing representative polished cross sections of these samples are provided in Figure 2.

X-ray diffraction analysis of the sputtered coatings on the flat plate samples was performed on the growth surfaces and yielded only reflections corresponding to the bcc and beta phases of tantalum. Relative intensities of the reflections corresponding to the two phases were used to derive growth surface fractions of the phases in the deposits. No attempt was made to correct for grain orientation effects,¹⁸ and these results should be interpreted as semi-quantitative.

A polished cross section of the coating on sample FP-003 (Figure 2a), shows that the tantalum deposit on this sample consisted almost entirely of closely packed columnar grains of the lighter colored beta phase extending from the substrate/coating interface to the growth surface. The bcc phase present in this sample appears as fingers of darker material extending in the direction of coating growth. Specimen FP-003 was the only one of the six flat plate samples in which the coating showed signs of separation. Cracks in the deposited tantalum running parallel to the substrate/coating interface were observed in many areas of this sample.

Both bcc and beta tantalum were also observed in the cross sectional areas of the other planar samples produced in this study, although the growth characteristics of the phases varied as a function of the deposition parameters (Fig. 2, Table 1). In specimen FP-004 (Fig. 2b), produced at a 69°C substrate temperature, the coating was

predominantly bcc phase. However, continuous cones of lighter colored beta tantalum can be seen to extend from the substrate interface to the growth surface, where they appear as bumpy growth defects. Knoop hardness measurements taken on the conical growths indicated values as high as 1500-1800 KH using a 50 g load. Beta tantalum was also observed in higher temperature deposits (Figs. 2c and 2d) where it appeared primarily as an irregular layer located near the substrate/coating interface. The beta layer in specimen FP-006, which was deposited on a 450°C substrate, was less than 4 microns in thickness (Fig. 2d).

The purity of the tantalum grown in the bcc and beta phases using the flat plate sputtering apparatus was investigated in several samples using electron dispersive spectroscopy (EDS) and electron microprobe analysis. Neither phase was found to contain concentrations of carbon, nitrogen, or oxygen in excess of 5 or 6 atom percent (the detection limit of the microprobe), or greater than 100 to 1000 ppm metallic contaminants.

In all of the as-polished cross sections of the coatings produced using the flat plate geometry there was visual evidence of particles embedded in the surface of the steel substrates. SEM examination of a 4340 steel substrate prepared in the same manner as those used for tantalum coating runs indicated that the lapping procedure left small pits in the substrate surface that were filled with an aluminum-containing material. Others have suggested that beta phase tantalum is preferentially nucleated on oxide or hydroxylated surfaces,³ and this effect may be compounded at low substrate temperatures.^{5,17} Many of the conical beta tantalum growths observed in the cross sections of specimen FP-004 (Fig. 2b) appeared to have originated at particles embedded at the substrate surface.

Cylindrical Geometry Deposits

Deposition conditions and coating results for runs utilizing the cylindrical sputtering geometry are summarized in Table 2. Photomicrographs of cross sectioned tantalum coatings produced using this apparatus are shown in Figures 3 and 4.

All of the coatings produced using the cylindrical apparatus consisted primarily of bcc tantalum and all except sample cyl-005 had areas in which there was a layer of beta phase tantalum produced at the substrate/coating interface (Fig. 3). Thickness and continuity of the beta layer varied with the sample and location on the substrate. Conical-shaped growth defects were observed in several of the cross sectioned coatings. These defects were either 100% beta phase (similar to those seen in Fig. 2b) or were bcc phase, but were separated from the bulk coating by a porous boundary. In nearly all cases a "grain" of beta phase material was found at the base of the bcc-type conical defect structures and apparently initiated the defect nucleation and growth.

Polished cross sections of coated samples taken from the upper, lower, and middle sections of the cyl-002 substrate indicated the presence of a thin (<3 microns thick), irregular interfacial deposit of beta tantalum covered by the bcc phase which extended to the growth surface (Fig. 3a). The phase distribution in the coating on sample cyl-001 was similar, but the beta layer was roughly 3 times thicker. X-ray analysis of the growth surface on the cyl-002 deposit indicated the presence of no beta tantalum.

Cross sections of samples taken in upper, middle, and lower areas of the cyl-003 deposit indicated a more varied distribution of the beta phase in this specimen than in cyl-001. Some areas of coating had almost no beta tantalum (Fig. 3b), while others had a thin interfacial area (similar to Fig. 3a), or fingers of beta extending parallel to the direction of film growth (Fig. 3c).

Interpretation of the tantalum deposit produced during run cyl-004 was complicated by the introduction of small amounts of water vapor into the sputtering system when the water-cooled tantalum target developed a leak midway through the run. The leak was not large enough to produce a noticeable change in discharge or krypton gas flow characteristics, but was detected upon helium leakchecking the target after completion of the run. The coating produced during the cyl-004 run had a relatively high concentration of the beta phase, which in some areas extended to the growth surface. In other areas of the cyl-004 sample the beta phase comprised all or nearly all of the coating volume. The coating also contained many cracks which ran parallel to the growth surface and which were associated with the beta structure.

The coating deposited on sample cyl-005 consisted of 100% bcc tantalum and was produced by precoating the tantalum target with a thin layer of sputtered niobium metal prior to the run. A thin (~2.5 micron), uniform niobium layer was produced between the substrate and the tantalum deposit by sputtering from the precoated target onto a 4340 steel substrate (Fig. 4). No beta phase tantalum was observed in any of three cross sections sampled from this deposit. The coating deposited in run cyl-005 also had an extremely small number of surface defects compared to the other tantalum coatings produced using the cylindrical apparatus.

IV. DISCUSSION

If sputtered tantalum is to be used effectively as a corrosion resistant barrier, the deposited material must be continuous, have relatively few defects, and be adherent to the substrate it is intended to protect. Furthermore, protective coatings subjected to stresses in the presence of high temperature, high pressure gases must be capable of withstanding repeated operating cycles without debonding. Tantalum coatings containing a large percentage of the beta phase will be harder than bcc-

only coatings, but are less ductile, and cracks resulting from intrinsic stresses (e.g., Fig. 2a) clearly limit their use for corrosion protection applications. Coatings consisting of continuous bcc tantalum with an interfacial layer of the beta phase (e.g., Figs 2c and d, and 3a) should provide an adequate protective barrier in the absence of mechanical or thermal stresses. However, the brittle beta layer is a potential site for crack generation and may promote debonding of the coating under conditions of thermal or mechanical stress cycling. In instances where the beta phase nucleates and continues to grow as "fingers" from point sources (e.g., Figs. 2b, 3c) a rough surface texture can develop and the interphase boundaries provide paths for corrosive agents to attack the substrate. Kashu et. al.²⁰ showed that high concentrations of defects in their sputtered tantalum coatings on stainless steel plates led to high corrosion rates of the substrates when they were exposed to concentrated sulfuric acid.

The beta tantalum phase had a tendency to nucleate at or near the substrate surface in nearly all of the deposits produced in this study regardless of the sputtering geometry used. This suggests that substrate conditions during initial sputter deposition determine the tantalum phase characteristics at the interface. Two parameters which have been specifically addressed in this study are the effects of substrate temperature and modification of the substrate surface by introducing an intermediate layer of material between the substrate and sputtered coating. Independently, both factors were observed to influence the growth characteristics of the sputtered tantalum, with the latter being the more pronounced. The small bias voltage variations (-34 to -50 V) investigated in the flat plate studies appeared to have little effect on the beta-to-bcc ratio. The cylindrical geometry deposits, which were made using higher substrate biases (-110 to -150 V), appeared to have less beta phase than the flat plate deposits, even at the intermediate sputtering temperatures (Table 2). This observation is consistent with the findings of other workers^{11,12,16}

that higher substrate biases favor formation of the bcc phase in sputtered tantalum films. However, biases to -150 V were not adequate in themselves to eliminate beta phase formation in our coatings at deposition temperatures approaching 250°C.

As has been noted by others previously,^{5,17} high substrate temperatures favored the formation of bcc tantalum in our deposits. Unfortunately, not all substrate materials can be subjected to the temperatures ($>600^{\circ}\text{C}$ ¹⁷) which would ensure the deposition of 100% bcc tantalum. The two deposits produced at the lowest substrate temperatures (FP-003 and FP-004) both exhibited undesirable beta tantalum growth characteristics. Higher substrate temperatures (including those in the 175 to 250°C range in the cylindrical geometry) promoted an initial deposition of the beta phase followed by transition to the bcc phase, which continued to grow until termination of the run. Hieber and Mayer²¹ have noted similar phase transition characteristics in evaporated tantalum films and suggested that the thickness of the beta layer is dependent on the substrate temperature. This is consistent with the differences in the thicknesses of the beta layers in films deposited at 350°C and 450°C substrate temperatures in the flat plate configuration (Figs 2c, 2d). Similarly, we may account for differences in the thickness of the the beta layers in samples cyl-001 (~10 microns) and cyl-002 (~3 microns) by a lower substrate temperature at initiation of the deposit in the former (~135°C vs. ~220°C for the latter), despite otherwise similar deposition conditions. It is difficult to account for the range of beta phase distribution seen in sample cyl-003 (Figs. 3a, 3c) unless some as yet unidentified effect related to the geometry of the cylindrical sputtering system can be invoked. The large amounts of beta tantalum in the coating on sample cyl-004 were almost certainly related to the incorporation of hydrogen and oxygen from the water which leaked into the system during the course of this run.

The most promising tantalum coating, both because of its low surface defect concentration and lack of an interfacial layer of the brittle beta phase, was the

sample produced in run cyl-005. This sample was distinguished from the others produced in this study by the presence of a thin niobium underlayer between the substrate and tantalum coating. Face and Prober⁴ recently demonstrated that underlayers of niobium as thin as 1.0 nm could be used to promote exclusive nucleation of the bcc phase in thin tantalum films deposited by ion beam sputtering on room temperature silicon and amorphous carbon substrates. They attributed the growth of bcc tantalum on niobium to the close lattice match between bcc tantalum and niobium, which is deposited only as a bcc phase. In addition, the niobium underlayer acts to cover and neutralize oxide and hydroxide species present on the substrate surface which are thought to promote the preferential nucleation of beta tantalum in sputtered films.³ In an independent series of experiments we found that a sputtered niobium underlayer would not promote exclusive growth of bcc tantalum if the niobium was first exposed to the atmosphere before deposition of the tantalum. The dramatic reduction in growth defect concentration observed in sample cyl-005 relative to other samples produced in this series can likewise be attributed to suppressed beta phase nucleation as a result of the niobium underlayer.

V. CONCLUSIONS

The results presented here indicate that it is possible to sputter deposit thick, adherent tantalum films at growth rates in excess of 0.3 microns per minute with substrate temperatures as low as 200°C. All of the films deposited directly onto 4340 steel and Incoloy-909 substrates at these conditions were characterized by the presence of at least a few percent of the more brittle beta tantalum, which was concentrated at the substrate-coating interface. Sputtering the tantalum coating immediately following the deposition of an underlayer of niobium, however, prevented the formation of the beta phase, even at substrate temperatures well below the beta/bcc transition temperature.

ACKNOWLEDGEMENT

The authors gratefully acknowledge the assistance of J.W. Johnston and A.T. Owen in preparing the tantalum deposits. This work was partially supported by Benet Laboratories under a Related Services Agreement with the U.S. Department of Energy (DOE) under contract DE-AC06-76RLO 1830. Pacific Northwest Laboratory is operated for DOE by Battelle Memorial Institute.

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FIGURE CAPTIONS

Figure 1. Schematic diagram of the cylindrical geometry sputtering apparatus used for deposition of tantalum coatings on the interior surfaces of 25 mm inside diameter 4340 steel and Incoloy-909 cylindrical substrates.

Figure 2. Photomicrographs of sputter deposited tantalum on 4340 steel using a flat plate configuration. a) FP-003, b) FP-004, c) FP-002, and d) FP-006

Figure 3. Photomicrographs of sputter deposited tantalum on 4340 steel and Incoloy-909 using a cylindrical geometry sputtering system. a) cyl-002, b) and c) cyl-003.

Figure 4. Photomicrograph of sputter deposited tantalum on 4340 steel (run cyl-005) using a cylindrical geometry sputtering apparatus. A 2.5 micron underlayer of niobium was presputtered onto the substrate prior to initiation tantalum sputtering by coating the tantalum target with a thin niobium layer.

TABLE 1

DEPOSITION PARAMETERS AND PROPERTY MEASUREMENTS FOR
130 MICRON THICK Ta FILMS SPUTTER DEPOSITED ON 4340 STEEL
USING FLAT PLATE CONFIGURATION

SPECIMEN	DEPOSITION	SUBSTRATE BIAS (V)	SUBSTRATE TEMP. (°C)	SURFACE PHASE COMPOSITION		MICROHARDNESS*	
	RATE (microns/min.)			(% bcc)	(% beta)	GS	TS
FP-001	0.51	-34	450	72	28	394	-
FP-002	0.55	-50	350	87	13	387	367
FP-003	0.59	-50	73	5	95	1341	1411
FP-004	0.65	-50	69	71	29	455	361
FP-005	0.79	-50	350	88	12	341	-
FP-006	0.56	-38	450	92	8	396	334

*GS = growth surface TS = transverse surface

TABLE 2.
SPUTTERING PARAMETERS FOR TANTALUM COATINGS ON
25 mm CYLINDRICAL SUBSTRATES

SPECIMEN	SUBSTRATE TEMPERATURE (°C)	SPUTTERING CONDITIONS ¹			TIME (hrs)	AVG. COATING THICKNESS (microns)
		V _t (V)	I _t (mA/cm ²)	V _s (V)		
cyl-001	472 ²	-1500	10	-110	3.0	50
cyl-002	460 ³	-1500	11	-110	4.5	75
cyl-003	245	-1500	7.5	-150	4.1	70
cyl-004	177	-1500	7.5	-150	2.0	35
cyl-005	200	-1500	7.5	-150	2.0	35

1 V_t = target voltage

I_t = target current density

V_s = substrate bias voltage

2 substrate temperature at initiation of sputtering was roughly 135°C

3 substrate temperature at initiation of sputtering was roughly 220°C

MATSON et al, Fig 1

MATSON et al. Fig 2

MATSON et al. Fig. 3

bcc Ta Coating

Nb Underlayer

Substrate

30 μm

MATSON et al Fig 4

END

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