QUALITY ASSESSMENT OF REFRACTORY PROTECTIVE COATINGS USING MULTI-FREQUENCY EDDY CURRENT MWM-ARRAYS

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ABSTRACT. Demands for increased range, rate of fire, and muzzle velocity have prompted development of new refractory metal coatings. Nondestructive measurement of coating electrical conductivity and thickness is crucial to the process development and statistical process control. This paper presents absolute property coating characterization results for Ta coatings obtained with a Meandering Winding Magnetometer (MWM®) eddy-current sensor and MWM-Array sensor. The measured coating conductivity indicates the ratio of the intended α-Ta to the undesirable β-Ta.

Keywords: coatings, eddy-current sensor, gun tubes, Meandering Winding Magnetometer (MWM), nondestructive inspection

INTRODUCTION

The development of more aggressive propellant formulations and firing scenarios has resulted in a significant reduction to large caliber cannon life. As a result of the increased wear and erosion problems in gun tubes there has been a drive to introduce improved bore surface coatings to mitigate these problems. Additionally, the current aqueous electroplating process used to deposit protective Cr bore coatings creates a hazardous waste stream and the associated high environmental clean-up costs. Cylindrical Magnetron Sputtering (CMS) is an environmentally benign PVD process being used to deposit high temperature refractory metal coatings, such as tantalum, for improved wear and erosion protection. In support of this developmental work, implementation of quantitative nondestructive evaluation techniques to rapidly characterize the structure and properties of coatings is needed to advance the next generation of coatings for gun tubes. This paper describes the use of a Meandering Winding Magnetometer (MWM®) eddy-current sensor and an MWM-Array to measure the coating thickness and absolute electrical conductivity of these advanced coatings, as well as the permeability of the underlying steel substrate. Some of the original work on the sensors and measurement methods is described in the 1993 paper by Goldfine [1] and in early patents [2, 3]. These sensors have found numerous applications including but not limited to characterization of coatings [4-7].

Conductivity measurement is a particularly useful tool for coatings due to the strong structure property relationships. For example, the conductivity of coatings is very sensitive to changes in density and morphology. The presence of porosity in coatings and/or interstitial impurities such as H, O, C, and N can result in orders of magnitude decreases in conductivity when compared to bulk values [8]. In this respect, electrical conductivity measurements via four-point probe have been very effective for research in process control in fabrication of micro-electronic circuits [9].

In PVD coatings, multiple phases of some materials may form with varying conductivities. This is the case for the Ta coatings deposited by CMS. Tantalum can form two phases during PVD**:** the alpha (α) phase, which is the stable bcc phase, and beta (β) phase, which is a metastable phase. The presence of β-Ta is problematic in that it is hard, brittle, prone to cracking, and leads to premature coating failure. Due to the difference in structure, β-Ta has a conductivity an order of magnitude lower than that of α-Ta [9,10]. Therefore, conductivity can be directly correlated to the presence of the detrimental β-Ta phase.

The current practice to identify and quantify concentrations of beta tantalum is through X-Ray diffraction, by which direct information on the crystalline structure can be obtained, and/or destructive characterization. However, for a successful quality control tool a more rapid technique to characterize the conductivity over large areas is needed.

Past work has been completed on analyzing relationships between coating properties and eddy current measured coating conductivity. The relationship between coating phase, impurity level, and structure/morphology of Ta, Nb, and Cr coatings was studied using pulsed eddy current methods [10,11]. This proof-of-principle testing lead to the adoption of the MWM eddy-current array sensor as a robust tool to rapidly quantify these properties along the full surface of the gun tube.

MWM SENSORS AND MWM-ARRAYS

Several examples of MWM and MWM-Array eddy current sensors are provided in Figure 1 (a) through (c). Each sensor has a single drive winding, consisting of one, two or several rectangular loops, and a number of rectangular inductive sensing loops. For these sensors, the transimpedance (sensing element voltage divided by drive current) is measured independently for each sensing element.

These sensors are carefully designed to enable accurate and rapid modeling from basic physical principles and to minimize unmodeled contributions to the sensor response. Each sensing element response at one or more input current frequencies is used by a multivariate inversion algorithm to determine absolute property values (e.g., electrical conductivity or magnetic permeability) at the location of the sensing element on the test specimen or component.

The sensor in Figure 1(a) averages measured properties over the 7 mm x 9 mm footprint area and is typically used for point-by-point measurements at selected locations, as in Figure 1 (b). Multi-channel MWM-Array sensors such as shown in Figure 1(c) are often used for scanning complex geometry regions. During scanning, data is taken at each sensing element as it traverses a part to produce an image of each unknown property of interest. These images reveal spatial variations of measured properties/material conditions.

Databases of precomputed sensor responses, known as measurement grids, lattices, and hypercubes, are used with an algorithm to convert complex impedance data into two or more unknown property estimates at each sensing element. In the case of three unknowns, 3-D lattices, which can be visualized as a collection of 2-D measurement grids, are used (see Figure 2). Databases of responses for four or more unknowns are referred to as hypercubes.

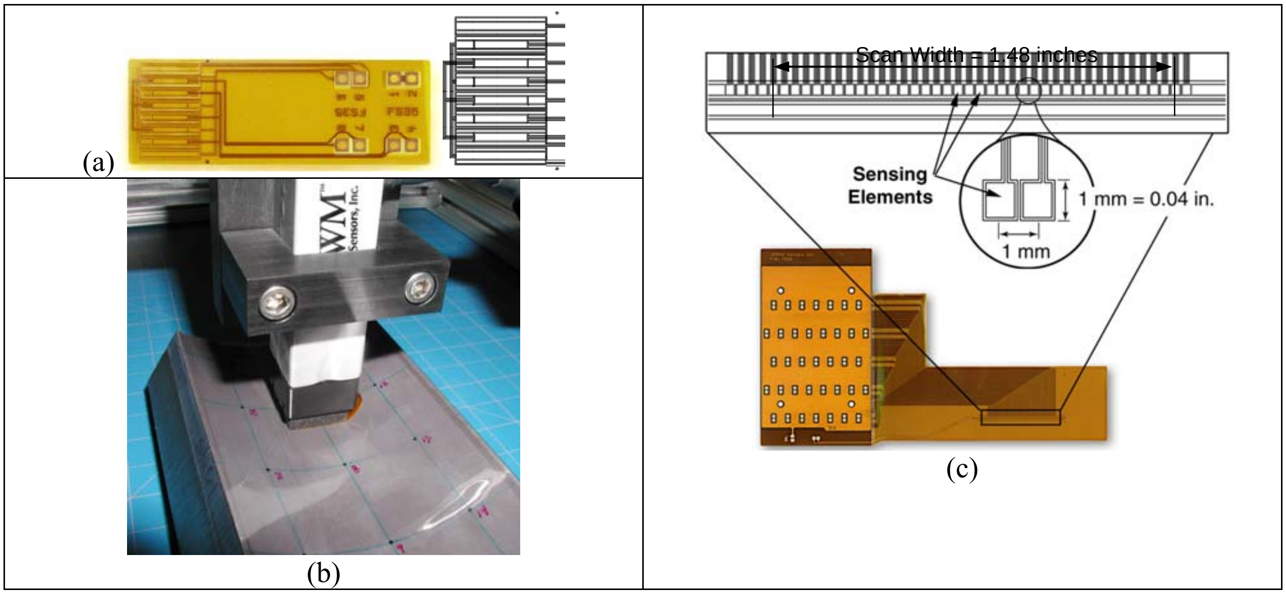


FIGURE 1. (a) Single-channel FS35 MWM sensor; (b) FS35 probe fixture used in this study; and (c) FA28 MWM-Array sensor used for acquiring data on the gun barrel.

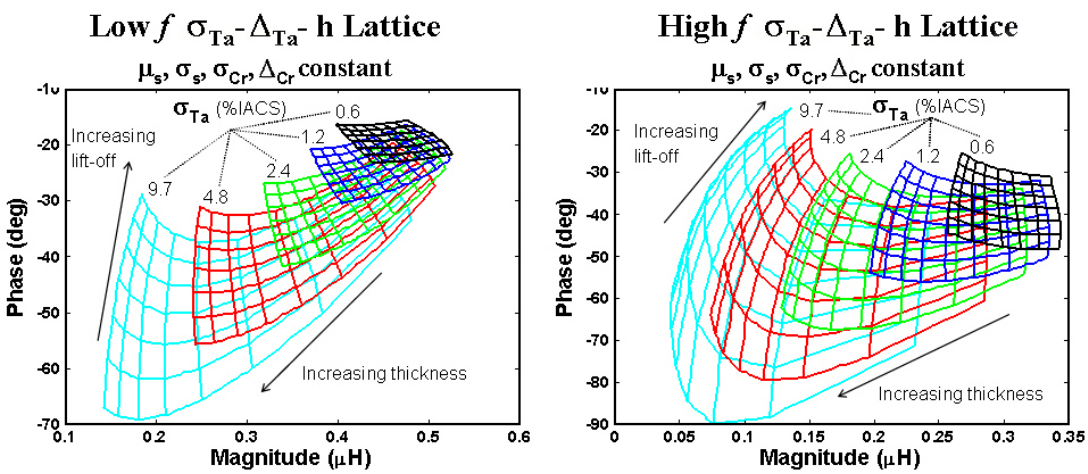


FIGURE 2. Measurement grid lattices for 3 unknowns (patents issued and pending).

SAMPLES

Eight longitudinally sectioned coated gun barrel samples and one flat noncoated steel sample were made available for this study by Benet Laboratories. Seven of the eight coated samples had tantalum coating sputtered either directly on steel or on a sputtered chromium interlayer. The coating phase composition based on metallography, and the sputtering gas for the coated samples are provided in Table 1. Tantalum coating thickness data obtained previously by an independent method at 15 locations per sample were also provided.

APPROACH

The key objective of this study was to demonstrate the ability to detect and locate regions within the tantalum coating that contain significant amounts of the undesirable β-phase. In addition to detecting and mapping of β-phase tantalum regions, simultaneous tantalum coating thickness measurements were sought from the same inspection scans.

TABLE 1. Description of samples provided by Benét Labs

Sample #	Description	Sputtering Gas	% Alpha Phase in Ta Coating
1	99% Alpha-Ta	Ar	99
2	Beta-Ta Surface, Alpha-Ta Interface	Kr	78
3	99% Beta-Ta	Ar	1
4	99% Alpha-Ta with sputtered Cr Interlayer	Kr	99
5	100% Alpha-Ta with Cr Interlayer	Kr	100
6	Alpha/Beta Mix with Cr Interlayer	Kr	73
7	Alpha/Beta Mix with Cr Interlayer	Kr	77
8	100% Sputtered Cr Coating (no Ta Coating)	Ar	
9	No coating (bare gun steel)	n/a	

Generally speaking, this is a multiple-unknown problem, with up to seven unknowns in the case of tantalum coating sputtered over a chromium interlayer. Practical considerations required that reasonable assumptions are made about some of the properties to reduce the number of unknowns and, yet achieve satisfactory robustness of the method. Figure 3 shows a schematic representation of the layered constructs with four unknowns. In addition to the two key unknowns such as tantalum coating electrical conductivity and thickness, two other unknowns are included: (1) lift-off, or proximity of the sensor to the conductive surface and (2) magnetic permeability of the steel substrate. The latter had to be included after preliminary MWM measurements on the noncoated steel sample revealed significant spatial variations of magnetic permeability as illustrated in the Results section.

A proprietary 4-unknown inversion method was used to characterize the properties of the tantalum coating and steel substrate. The two 4-unknown Material Under Test (MUT) constructs shown in Figure 4 were used for the physics-based model generation of the hypercubes. These hypercubes were then used in a multivariate inversion process to estimate the four unknowns: (1) lift-off, (2) tantalum coating conductivity, (3) tantalum coating thickness, and (4) steel substrate permeability. The value for the electrical conductivity of the steel substrate was obtained from MWM FS35 single-channel sensor measurements of the substrate specimen. In Construct B (Figure 3, right), the thickness of the chromium interlayer was assumed based on information provided by Benét Labs, and the conductivity of the chromium interlayer was determined by analysis of FS35 measurement data.

SENSORS AND MEASUREMENT PROCEDURES

The single-channel FS35 sensor and the 37-channel MWM-Array used in this study are shown in Figure 1. The FS35 sensor with a sensing footprint of 7 x 9 mm (0.28 in. x 0.36 in.) was used for a preliminary assessment of the properties. This assessment aided in better understanding of the problem and in refining the procedure used for MWM-Array measurements.

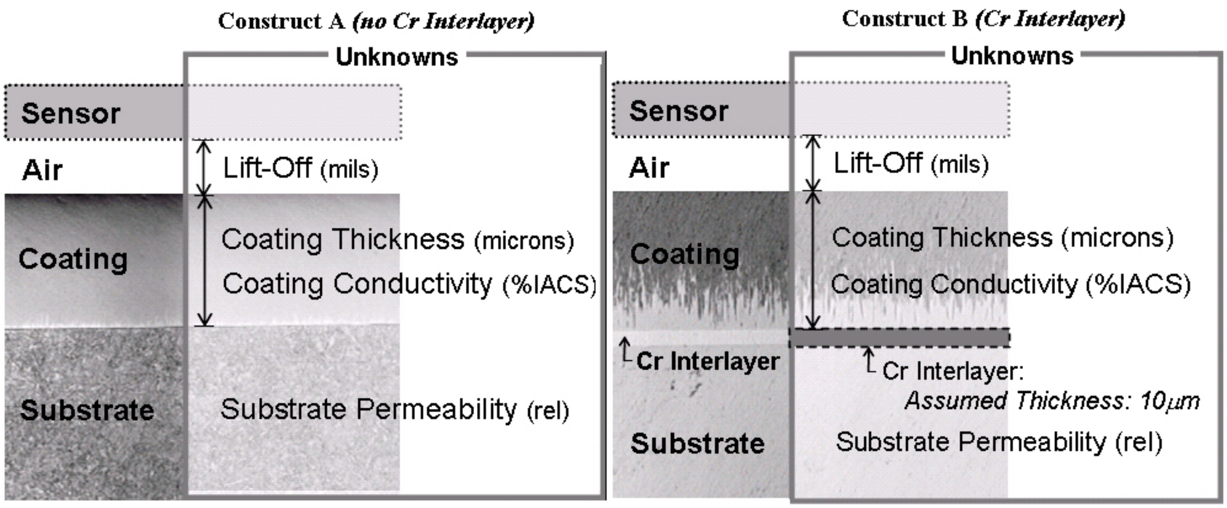
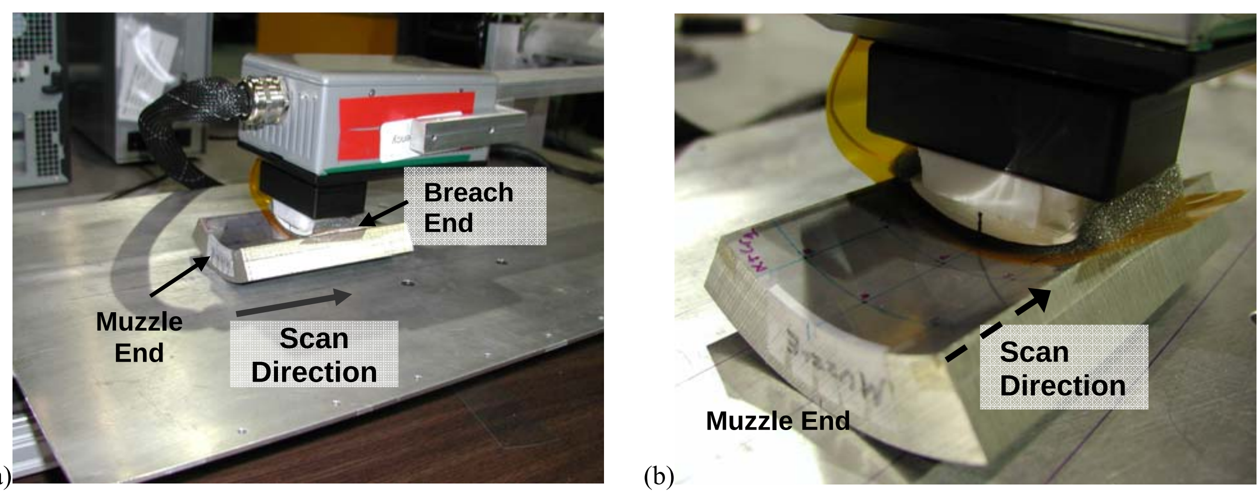


Figure 3. Schematic of the two 4-unknown constructs used for analysis. Construct A assumes no chromium interlayer and Construct B accounts for the chromium interlayer.

The calibration procedure performed prior to the measurements did not require any standards. In the case of the scanning MWM-Array, this unique procedure included an Air/Shunt calibration by taking measurements in air with the FA28 sensor followed by measurements in air with the corresponding shunt. The MWM-Array measurements were performed at frequencies from 100 kHz to 25 MHz. Figure 4 shows an experimental setup used for scanning of the samples. A 125-μm thick protective shim was placed on top the samples during scanning. The scan rate during data acquisition was 1.5 mm/sec (3.5 in/min) at a measurement rate of 125.7 ms/measurement. Significantly faster scan rates can be used during scanning of actual gun barrels. For some other applications, scan rates of 1.5 to 4.5 m/min (5 to 15 ft/min) have been demonstrated. In selecting a scan rate, one should consider the trade-off between throughput and image resolution/detection requirements. In this case, the scan rate would depend on the size of the smallest low conductivity β-phase region, within the otherwise α-phase coating, which must be detected.



(a) (b)

FIGURE 4. FA28 MWM-Array scanning setup: (a) Overall view of the setup, (b) close-up view of the MWM-Array sensor fixture.

RESULTS

MWM sensor and MWM-Array examinations of the uncoated steel plate indicated significant spatial magnetic permeability variations that necessitated permeability measurements at each measurement location. MWM-Array scans of Samples 1 through 7 produced effective electrical conductivity and thickness images for the tantalum coating. These images were obtained via the fast multivariate inversion for estimation of the four unknowns. Figure 5 shows examples of MWM-Array generated images of tantalum coating conductivity and thickness. Analysis of the electrical conductivity data obtained for the tantalum coating on Samples 1 through 7 by the MWM-Array indicated significant differences between the tantalum coating sputtered in krypton vs. the coating sputtered in argon. This was true for both coatings sputtered directly on the gun steel substrate and those sputtered over the chromium interlayer. Figure 6(a) shows the average MWM-Array measured electrical conductivity of the sputtered tantalum coating vs. percent of α-Ta in the coating. The latter was estimated by visual inspection of the micrographs of the coating layers provided by Benét Labs. Figure 6 (b) shows a plot of the local thickness values for the tantalum coatings measured with an FA28 MWM-Array vs. the thickness values provided by Benét Labs based on previous measurements by an independent method. The dashed line represents an ideal 1:1 correlation.

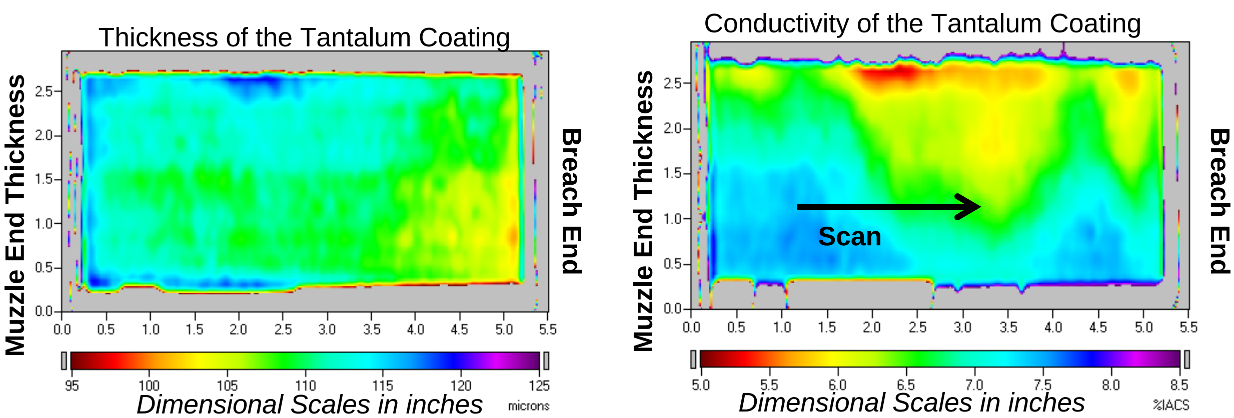
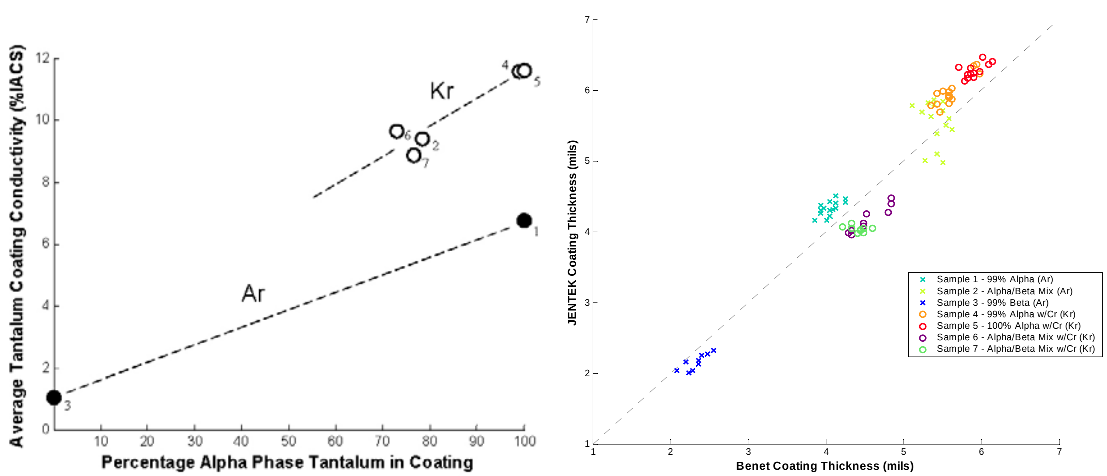


FIGURE 5. Examples of MWM-Array generated thickness of tantalum coating and conductivity of tantalum coating.



(a) (b)

FIGURE 6. (a) Coating conductivity vs. the percentage of alpha phase tantalum in the coating, and (b) MWM-Array measured coating thickness values vs. coating thickness values reported by Benét Labs.

DISCUSSION

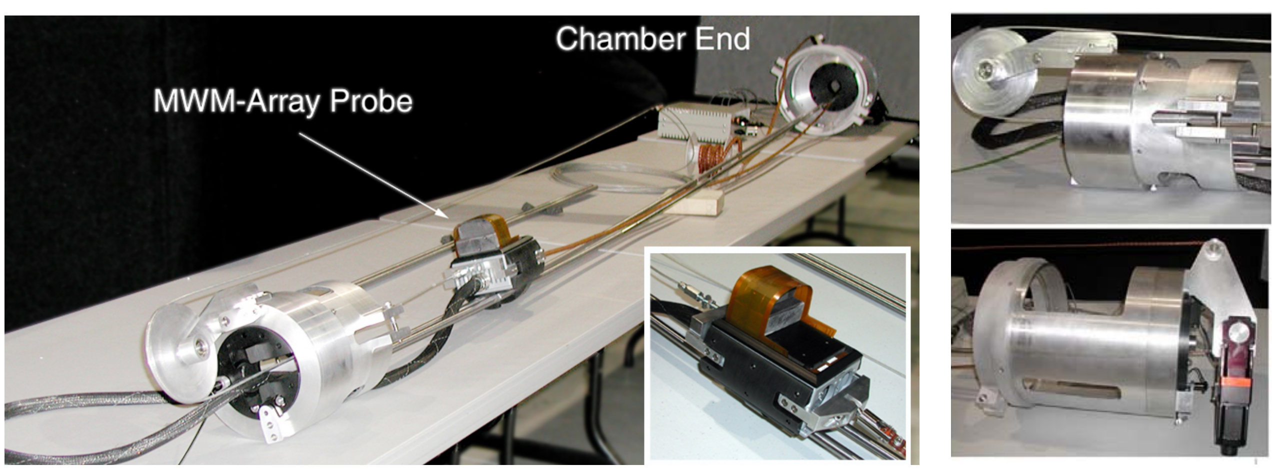
The two distinctly different relationships between tantalum coating phase composition and conductivity revealed by the MWM-Array measurements suggest that conductivity of the tantalum coatings is strongly affected by the sputtering gas. The carrier gas used in the sputtering deposition process plays a prominent role in determining the microstructure and therefore the electrical properties of the deposited coating. Based on the MWM-Array measurements, pure α-Ta has a conductivity of about 11.6 %IACS when sputtered in krypton and 6.74 %IACS when sputtered in argon.

The correlations between tantalum coating conductivity and composition should be verified using a significantly larger well characterized sample set. This should include samples with pure β-Ta coating sputtered in krypton gas or samples with very low α fraction in tantalum coating.

The interdependence of processing, microstructure and properties coupled with the ability of the MWM-Array to successfully measure absolute materials properties presents an opportunity for improved quality control. Potential future applications for coating degradation assessment and life management performance may also result.

The results show that MWM-Arrays can determine the extent of lower conductivity regions containing β-phase. MWM-Array imaging capability provides a means for visual assessment of the distribution of absolute materials properties and coating thickness. The ability to spatially characterize the occurrence of undesirable conditions and detect coating property gradients may lead to improved process control as well as development of more robust processes.

The procedures developed in this study are being implemented for scanning of actual large caliber gun barrels. Figure 7 shows a prototype fixture that is used for scanning of gun barrels during the evaluation stage of this inspection method.



(a) (b)

FIGURE 7. (a) Inspection fixture with an inset photograph of the MWM-Array probe; (b) close-up photographs of the muzzle end (left) and breach end (right) of inspection fixture.

CONCLUSIONS

MWM sensors and MWM-Arrays provide a means of assessing tantalum coating phase composition via measurements of electrical conductivity of tantalum coatings in gun barrels. MWM-Array images offer an easy visual assessment of lower conductivity regions containing β-phase. The sensors and MWM-Arrays can measure electrical conductivity of the tantalum coatings on gun barrel steel substrate with and without a chromium interlayer.

Relative conductivities of pure α-phase and mixed (α+β) phase regions depend on the sputtering gas. Pure α-Ta conductivity values are significantly different for coatings sputtered in argon vs. those sputtered in krypton, e.g., 6.7% IACS in the case of argon and about 11.5% IACS in the case of krypton. It is possible to distinguish between coatings sputtered in argon and coatings sputtered in krypton when β-phase content is less than 50% (see Figure 6). Moreover, the sensors can quantify β-phase content, assuming a priori knowledge of process gases. In addition, these sensors can measure thickness of tantalum coatings sputtered on gun barrel steel.

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