Characterizing Tantalum Sputtered Coatings on Steel by Using Eddy Currents

Yaron Danon, Changqing Lee, Chris Mulligan, and Greg Vigilante

***Abstract*—With the goal of building a system for fast inspection of coatings, we have developed a method that uses induced eddy currents to characterize tantalum alpha and beta phases in a layer of thin sputtered tantalum on steel. The detection of the tantalum phases is based on the large difference in electrical conductivity between them. Measurements based on the method agree well with values based on theoretical calculations. We applied the method in a two-probe differential system having higher sensitivity and less noise than a one-probe system. The probe uses pulsed eddy currents with a pulsewidth of 1 μs, allowing us to scan at rates of up to 10^5 pulses per second on a computer-controlled XY table for fast data acquisition. When the system was used to scan steel samples coated with 12.5–30 μm of tantalum, a clear difference between alpha and beta phases was observed. The system was also used to measure the conductivity of the alpha and beta phases. We present here a conductivity map of the sample.**

***Index Terms*—Differential system, eddy-current testing, pulsed eddy current, tantalum coatings.**

I. Introduction

THE eddy-current method can be applied to measure conductivity and thickness of thin coatings on metallic material [1], [2]. Eddy-current techniques are widely used for detection of corrosion in airplane structures and for detection of defects in metals [3]–[5]. The eddy-current method is based on the induction of magnetic fields in a sample. These magnetic fields created by a coil will induce currents in the metal, which can be detected by a probe above the metal surface [6]. In the work described here, we use the same probe to induce the magnetic field and detect the eddy currents. The net effect of this process is to change the impedance of the probe when it is coupled to the metallic surface under test. The eddy-current technique has several advantages; it is a nondestructive and noncontact method, as a result, no damage is done to the surface even when the scan is performed at very high speeds. Thus, it does not rely on good contact with the surface like other conductivity measurement methods. The method can accurately determine the conductivity and in some configuration also measure the coating thickness. Another advantage is that the instrumentation required for these types of testing is relatively simple and thus reliable and inexpensive.

The deposition of protective coatings is necessary in the bore of large-caliber gun tubes to mitigate wear and erosion during gun firing. The current protective coating for the interior diameter of large caliber tank and howitzer gun tubes is electroplated Cr. However, the current drive for higher energy propellants have reduced the utility of Cr as a protective coating in gun tubes. In addition, electrodeposition of Cr is associated with the production of large quantities of hazardous waste. Based of these two issues, alternatives to this material and process are being pursued. One alternative is the sputtering of high melting point refractory metals such as tantalum. The sputtering process does not produce any hazardous chemicals or waste and is thus environmentally friendly [7]. When Ta coatings are deposited by sputtering, an undesirable beta phase can be induced. Generally speaking, the formation of the metastable beta phase is most often related to the energetics of the sputtering plasma. The beta phase can be a factor of five times harder than the stable alpha phase and is thus much more brittle. Under the high thermal loads that exist in a gun bore, the beta phase would tend to crack and spall readily [7].

Our goal was to try to detect the undesirable beta phase and provide a fast method that can be used as a part of the quality control of the manufacturing process. One method used to distinguish between alpha and beta phases is X-ray diffraction, by which direct information on the crystalline structure can be obtained [7]. However, this method is relatively slow and much more difficult to execute than the method proposed by the authors.

In order to distinguish between the Ta phases we need to rely on some unique physical property that is different in the two crystalline structures. Measurements of the properties of alpha and beta phases of tantalum were done by Catania *et al.* [8], Clevenger *et al.* [9], and also by Kazuhide *et al.* [10]. They found that the different crystalline structure of the alpha and beta phases give rise to a large difference in the electrical conductivity, Clevenger *et al.* reports that the conductivity for the alpha phase $\sigma_\alpha = 7.1$ MS/m, and the conductivity for the beta phase $\sigma_\beta = 0.48$–0.59 MS/m. This large disparity in conductivity can be used to distinguish between the different phases. Conductivity measurement is not a direct indication of the crystalline structure as obtained from X-ray diffraction, however, for a uniform coating it can be very effective in determining the coating phase. A measurement of the conductivity of the tantalum coating on the gun steel will result in a value between σ_α and σ_β; this value can be used to determine how much beta contamination exists in the alpha phase. Also, a predominantly alpha coating will have a uniform conductivity. Any sudden drop in the conductivity across the surface indicates the existence of the beta phase.

The pulsed eddy-current technique, which uses a step function voltage to excite the probe, is a promising approach in the field of eddy-current testing. The advantage of using a step func-

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tion voltage is that it contains a continuum of frequencies; as a result, the electromagnetic response to several different frequencies can be measured with just a single pulse. Since the skin depth of penetration is dependent on the frequency of excitation, information from a range of depths can be obtained simultaneously. For example, Tai *et al.* [2] showed that pulsed eddy current could be used to simultaneously determine the conductivity and thickness of coatings on nonmagnetic substrates. However, for the work presented here, we chose to simplify the procedure and measured the thickness of the tantalum coating with the magnetic induction method. Another advantage of applying pulsed eddy currents for the purpose of repetitive scanning is that the low duty cycle of the pulses puts less average power through the small probe coils, which allows one to operate at high instantaneous current during the pulse itself.

We present a study on the feasibility of this method for coating characterization in this paper. The research includes theoretical calculations and experiments for detection of the crystalline phases of 12.5–30 μm thick tantalum coating on gun steel that contains both alpha and beta phases. In order to use the eddy-current method for rapid scanning of the surface, we explored the pulsed eddy-current method. The pulsewidth used is in the order of 1–5 μs wide, the information can be collected in a time interval of 10 μs from the start of the excitation pulse this results in the ability to collect up to 10^5 pulses per second. This high pulses rate can be translated to high scanning speeds.

II. Theory

Consider an air coil positioned above a one layer coated substrate as illustrated in Fig. 1.

The coil inner radius is noted by r_1 and the outer radius is noted by r_2. The bottom and top distance of the coil edges from the coating are h_1 and h_2 respectively such that the coil length is given by $L = h_2 - h_1$. The number of turns of the coil is denoted by n. σ_1 and σ_2 are the conductivity of the coating and the substrate, respectively. μ_1 and μ_2 are the permeability of coating and the substrate, respectively. The thickness of the coatings is denoted by d.

The analytical solution for the coil impedance of such a system was given by Cheng *et al.* [11]

$$Z_L = Kj\omega \cdot \int_0^\infty \frac{I^2(\alpha, r_1, r_2)}{\alpha^5} \cdot \left(2(h_2 - h_1) + \frac{1}{\alpha}[2e^{-\alpha(h_2-h_1)} - 2 + A(\alpha)\phi(\alpha)]\right) d\alpha \quad (1)$$

where ω is the angular frequency ($\omega = 2\pi f$), also

$$K = \frac{\pi\mu_0 n^2}{(h_2 - h_1)^2(r_2 - r_1)^2} \quad (2)$$

where μ_0 is the permeability of free space, and

$$\phi(\alpha) = \frac{(\mu_2\alpha+\mu_0\alpha_2)(\mu_1\alpha_2-\mu_2\alpha_1)+(\mu_2\alpha-\mu_0\alpha_2)(\mu_1\alpha_2+\mu_2\alpha_1)e^{2d\alpha_2}}{(\mu_2\alpha-\mu_0\alpha_1)(\mu_1\alpha_1-\mu_2\alpha_1)+(\mu_2\alpha+\mu_0\alpha_2)(\mu_1\alpha_2+\mu_2\alpha_1)e^{2d\alpha_2}} \quad (3)$$

$$I(\alpha, r_1, r_2) = \int_{\alpha r_1}^{\alpha r_2} x J_1(x) dx \quad (4)$$

$$\alpha_{1,2} = \sqrt{\alpha^2 + j\omega\mu_{1,2}\sigma_{1,2}} \quad (5)$$

$$A(\alpha) = (e^{-\alpha h_1} - e^{-\alpha h_2})^2. \quad (6)$$

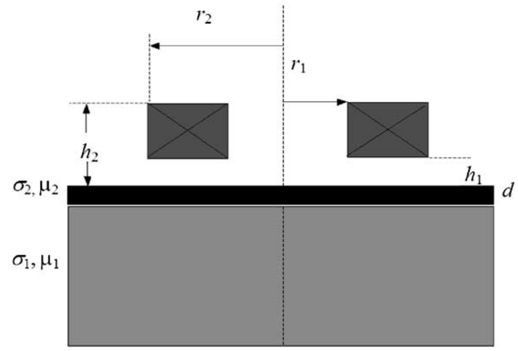

Fig. 1. Coil over one-layer coating.

In (4), $J_1(x)$ is first-order Bessel function of first kind. The coil impedance above the substrate can be obtained by setting $\sigma_1 = \sigma_2$, and is given by

$$Z_H = Kj\omega \cdot \int_0^\infty \frac{I^2(\alpha, r_1, r_2)}{\alpha^5} \cdot \left(2(h_2-h_1)+\frac{1}{\alpha}\left[2e^{-\alpha(h_2-h_1)}-2+A(\alpha)\frac{\mu_1\alpha-\mu_0\alpha_2}{\mu_1\alpha+\mu_0\alpha_2}\right]\right) d\alpha. \quad (7)$$

The expression for the impedance in (1) and (7) is applicable for a single excitation frequency. A method that is better suited for fast sampling of the impedance is the pulsed eddy current, in which the coil will be excited by a square pulse of duration t_p during the pulse that we apply constant voltage V_0 to the coil. This is illustrated in Fig. 2.

In order to apply the theory developed for the impedance calculations of a coil excited by a single sinusoidal frequency, a Fourier transformation of the pulse is used and is given by

$$V(t) = V_0 \left[\frac{t_p}{2T} + \sum_{n=1}^{N} (a_n \cos(\omega_n t) + b_n \sin(\omega_n t))\right] \quad (8)$$

where the parameters an a_n and b_n are given by

$$a_n = \frac{\sin(\omega_n t_p)}{n\pi} \quad (9)$$

$$b_n = \frac{1 - \cos(\omega_n t_p)}{n\pi}. \quad (10)$$

The angular frequency and frequency are given by $\omega_n = n\pi/T$ and $f_n = \omega_n/(2\pi)$, respectively.

To calculate the coil impedance, consider the circuit in Fig. 3.

Applying the relation $I = V(t)/Z(\omega)$ for each frequency, we get

$$V_{L,H}(t) = V_0 R_L \cdot \left[\frac{t_p}{2TR_T} + \sum_{n=1}^{N} \cdot \left(\frac{a_n\cos(\omega_n t-\theta_{L,H}(\omega_n))+b_n\sin(\omega_n t-\theta_{L,H}(\omega_n))}{\mathrm{Mag}_{L,H}(\omega_n)}\right)\right] \quad (11)$$

R_T is the total resistance in the circuit, including the load resistance R_L, the coil resistance R_C, and the output resistance of the pulse generator R_O. In this case, the complex impedance of the coil and the real resistance of the other components were represented in polar coordinates with magnitude

$$\mathrm{Mag}_{L,H}(\omega_n) = \sqrt{\mathrm{Im}(Z_{L,H}(\omega_n))^2 + [\mathrm{Re}(Z_{L,H}(\omega_n)) + R_t]^2} \quad (12)$$

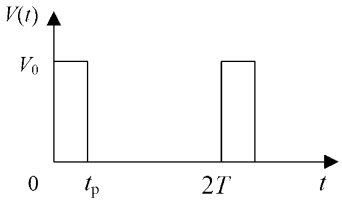

Fig. 2. Square excitation pulse with width t_p, height V_0 repeating every $2T$.

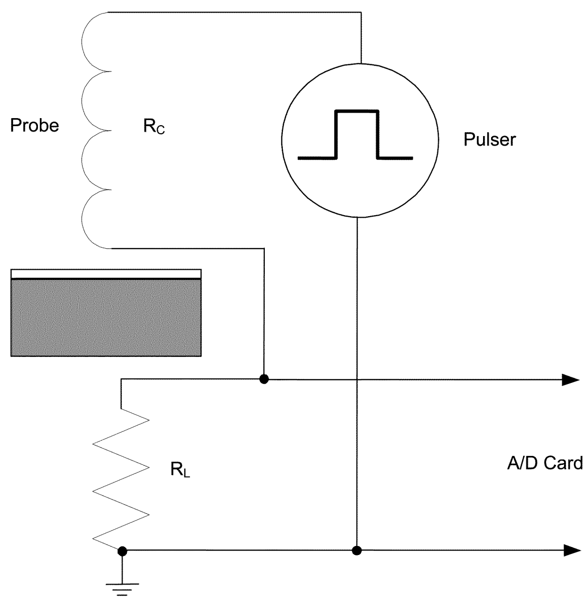

Fig. 3. Experiment setup for a one-coil system.

and phase

$$\theta_{L,H}(\omega_n) = \tan^{-1}\left(\frac{\mathrm{Im}(Z_{L,H}(\omega_n))}{\mathrm{Re}(Z_{L,H}(\omega_n)) + R_t}\right) \tag{13}$$

$$\Delta V(t) = V_L(t) - V_H(t). \tag{14}$$

One advantage of such pulse excitation function is that one pulse provides impedance information in many frequencies. This is important because each frequency has different penetrability, and thus we can obtain a response signal with information from different depths. The information is of course weighted by the Fourier coefficients a_n and b_n. Another advantage is that energy delivered to the coil per pulse could be higher than ac excitation because of the lower repetition rate. This provides a stronger measured response during and immediately after the pulse.

III. Experimental Setup

In our experiments, a differential system with two coils noted in Table I as coils 2a and 2b was used. Both of these coils are wound on plastic bobbins. In winding the coils, we attempted to obtain a zero response in the differential system. This yielded slightly different number of turns in the coils. Also, the coil bobbins purchased from Cosmo Cooperation has some variability in the radius.

The experimental setup is illustrated in Fig. 4. The differential system was driven by a Tabor-8024 waveform/function generator. The values of the load resistors R_1 and R_2 were 100 Ω. The differential amplifier is a wide-band amplifier that was designed by the authors for this purpose. The gain of the amplifier

TABLE I
Parameters of the Coils

Coil	2a	2b
number of turns, n	110	112
inner radius, r_1(mm)	1.19	1.21
outer radius, r_2 (mm)	2.00	2.10
height, h_2-h_1 (mm)	2.85	2.84
resistance, R_c (Ω)	1.96	2.03
inductance[a], L (mH)	0.02289	0.02256

[a]The inductance of the coils was measured with Instek LCR meter at a frequency of 1kHz.

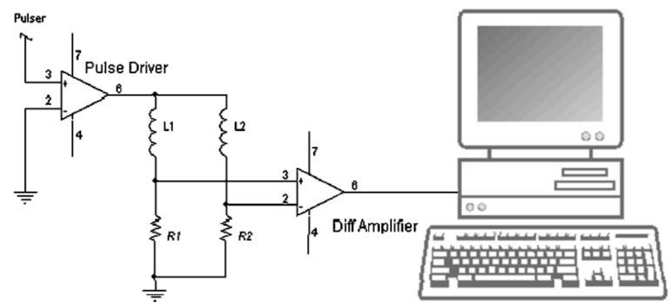

Fig. 4. Experimental setup for the differential system.

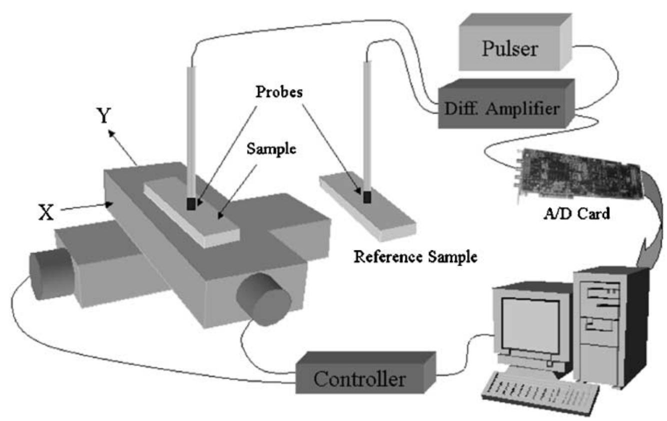

Fig. 5. Illustration of the XY eddy-current scanner.

is changeable and a factor of 5 is applied in the experiment. The output of the amplifier was connected to CompuScope 14100 card, which is a 14-bit A/D card from Gage Applied.

The pulse driver was designed to provide the system with constant voltage with a very low internal resistance, less than 1 Ω. In order to map the conductivity of a sample, an eddy-current scanner was designed and constructed.

The scanner uses the data acquisition system shown in Fig. 5. One sample was positioned on a computer controlled XY table and another reference sample was used as illustrated in Fig. 5. The two coils are stationary and the sample being scanned is moved under the scanning probe. This arrangement allows the system to scan the conductivity of the sample relative to the conductivity of the magnetic gun steel. In order to reduce inaccuracies from small differences between the two differential channels, a measurement of the system's response when the two probes are placed over stainless steel was done first. This measurement is treated as background and was subtracted form the signal obtained for each pulse. This background signal is typically about one-tenth of the scan signal.

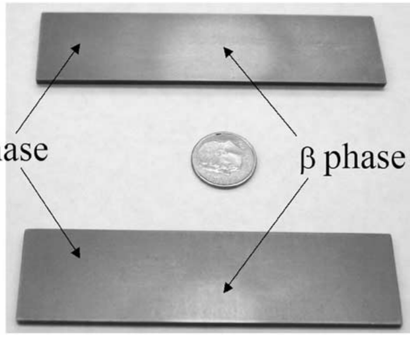

Fig. 6. Samples obtained from Benét Laboratories.

The typical height of the probe above the surface $h_1 = 0.5$ mm.

IV. Results

An experiment to determine the conductivity of the tantalum alpha and beta phases was performed. We obtained two samples of gun steel coated with tantalum from Benét Laboratories. A picture of the samples is shown in Fig. 6; the brighter color in the center of the samples corresponds to the tantalum beta phase region, while the darker gray surrounding it is the tantalum alpha phase.

Several measurements of different foils with different thickness placed over the gun magnetic steel (the back side of the samples shown in Fig. 6) were taken.

The only prior information on the substrate is that it is a magnetic gun steel metal with properties most closely associated with commercial 4340 steel. A calibration procedure was developed in order to find the relation between the digitized pulse coil response and the conductivity of the coating. The method used was to place various known conductivity foils on the SST substrate and generate curves of the response as a function of the thickness of the coating. A series of plastic foils was used to simulate a nonconducting coat. The average of three points at the peak of the measured response is plotted in Fig. 7.

The foils include plastic, SST-304, Ti, and Ta. Fig. 7 includes the experimental data points and solid lines that are the theoretical calculation. When performing the calculations, the only unknowns are the conductivity and permeability of the substrate SST. These values were found by obtaining best fits of the calculations and the measurements for the variety of coating conductivities and thickness. Note that all the coating materials used are nonmagnetic. The value obtained for the conductivity and permeability of the gun steel substrate are $\sigma_1 = 0.5 \pm 0.02$ MS/m and $\mu_1 = (50 \pm 2) \cdot 4\pi \times 10^{-7}$ H/m.

In order to add the alpha and beta phases to this curve, an accurate thickness measurement was done using a coating thickness instrument from DeFlsko. The method used by the DeFlsko instrument is magnetic induction, which does not depend on the conductivity of the coating. The measurements of the thickness and conductivity were taken at the same location on the sample. The thickness of the alpha phase was found to be 12.7 ± 1.0 μm (0.50 ± 0.04 mil) and the thickness of beta phase is 33.0 ± 1.5 μm (1.30 ± 0.06 mil).

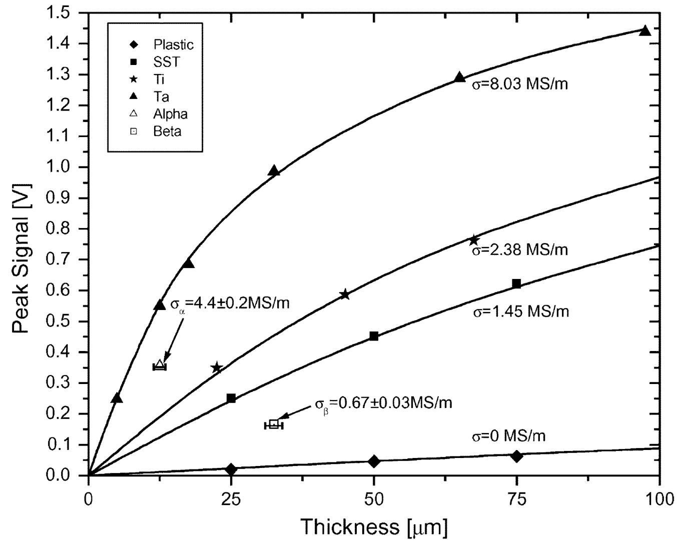

Fig. 7. Measurements and calculations for different thickness and material foils on the gun steel. The scattered points are measurements, and the solid lines are the calculated values for the substrate of the gun steel.

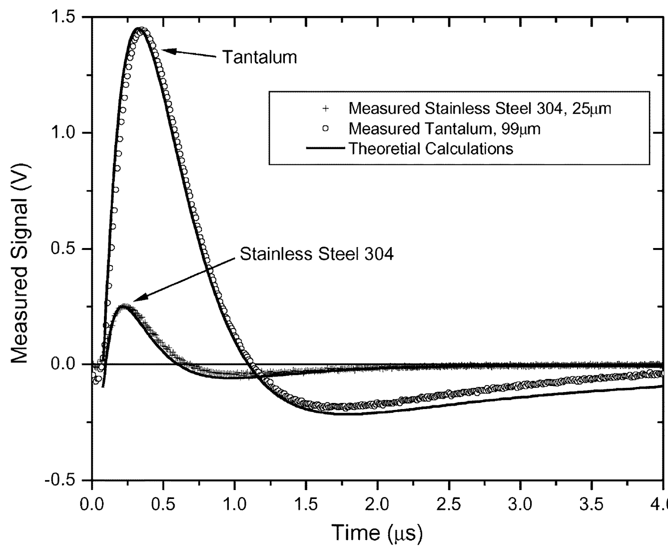

Fig. 8. Measured calculated pulse responses for 25-μm SST304 and 99-μm Ta on the gun steel. The pulsewidth used was 5 μs.

An example of the experimental results and theoretical calculations for one pulse is shown in Fig. 8. The pulsewidth is 5 μs.

Having found the gun steel's conductivity and permeability, the experimental data could be fitted to solve for the conductivity of the coating. The fitted curves for the alpha and beta phases are shown in Fig. 9.

Overall, the fits look good although there is some disagreement with the experiment. This is mostly related to imperfections in the coil winding that cannot be modeled and some systematic errors due to nonuniformity of the coating thickness.

The results of the fitting show that the conductivity of the alpha phase is $\sigma_\alpha = 4.4 \pm 0.2$ MS/m and the conductivity of the beta phase is $\sigma_\beta = 0.67 \pm 0.03$ MS/m. The uncertainty of the measurement was calculated from the uncertainty in the thickness measurement. Converting these values to resistivity, one obtains $R_\alpha = 22.7$ $\mu\Omega$/cm and $R_\beta = 149$ $\mu\Omega$/cm, which is in agreement with the values of Clevenger *et al.* [9] who ob-

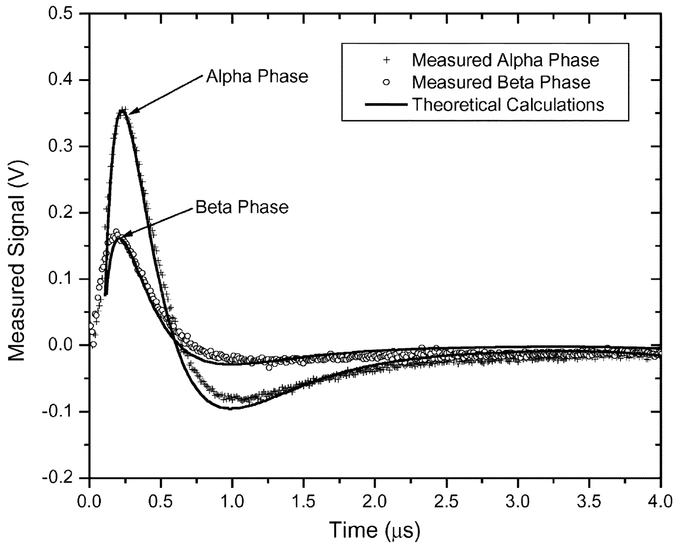

Fig. 9. Measured (points) and calculated (solid lines) of Ta coating on gun steel used for fitting the conductivity for the alpha and beta phases.

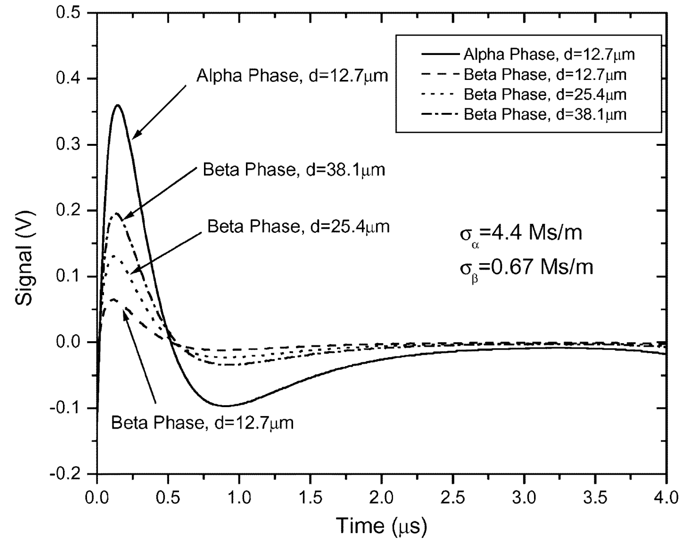

Fig. 10. Coating thickness effects on conductivity.

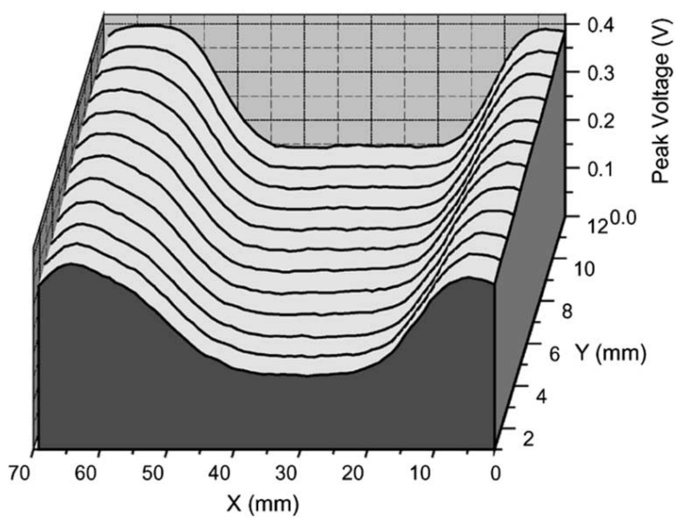

Fig. 11. Example of the raw data obtained in one scan. For this case, $\Delta x = 0.008$ mm and $\Delta y = 1$ mm.

tained $R_\alpha = 14\ \mu\Omega$/cm and $R_\beta = 170$–$210\ \mu\Omega$/cm. It is important to note that the conductivity of the alpha phase depends on the sputtering conditions and normally the lower resistivity indicates a pure alpha phase. This demonstrates the application of the eddy-current method to characterize the coating thickness.

From these measurements shown in Fig. 7, it is clear that some degree of uniformity of the coating thickness is required in order to measure its conductivity. One way to overcome this problem was to simply measure the thickness using the magnetic induction technique. However, this requires an additional probe. Using the eddy-current pulsed method, the information on the thickness and conductivity are obtained in the same measurement and a method to separate the two is still under development.

In order to estimate the effect of the thickness on the measured signal, several calculations of Ta beta phase coating with different thickness were done. Another calculation was done with Ta alpha phase with the thickness of 12.7 μm (0.5 mils); the results are plotted in Fig. 10.

The data in Fig. 10 indicate that the method is sensitive to changes in the coating thickness. However, even for changes of a factor of 3 (12.7–38.1 μm) in the coating thickness, the Ta alpha and beta phases are still easily distinguishable. In order to improve the sensitivity of this instrument, it will be desirable to find a way to distinguish the changes in thickness and conductivity.

The sensitivity to thickness can be advantageous when using this method for inspection of coated parts where coating uniformity and phase integrity are required.

The eddy-current scanner was used to scan the samples provided by Benét Laboratories. The software allows the user to set the step size dX and dY. The scanner moves the sample at a constant speed while pulsing the probe at a rate of 1 kHz with a pulsewidth of 2 μs and sampling the probe at a constant rate. The data is collected at a time resolution of 10 ns. An example of such a scan is shown in Fig. 11. The scan region was only a part of the actual sample surrounding the beta phase in the center (see Fig. 6).

The thickness of the Ta coating is about 25.4 μm (1 mil) (13.0 ± 1.0 μm for alpha phase, 33.0 ± 1.5 μm for beta phase). For the purpose of calibration, we assume that the coating is of uniform thickness and the only difference between the phases is conductivity. For the coating thickness change in our sample, this assumption is expected to give an error of about 1 MS/m in the measured conductivity.

We could assume that the conductivity is linearly proportional to the peak voltage as shown in Fig. 12 and thus

$$\sigma(V) = A + B \cdot V. \tag{15}$$

The parameters of A and B are determined by

$$\sigma_\alpha(V) = A + B \cdot V_\alpha \tag{16}$$

$$\sigma_\beta(V) = A + B \cdot V_\beta \tag{17}$$

where V_α and V_β are the measured peak voltage for the alpha and beta phases.

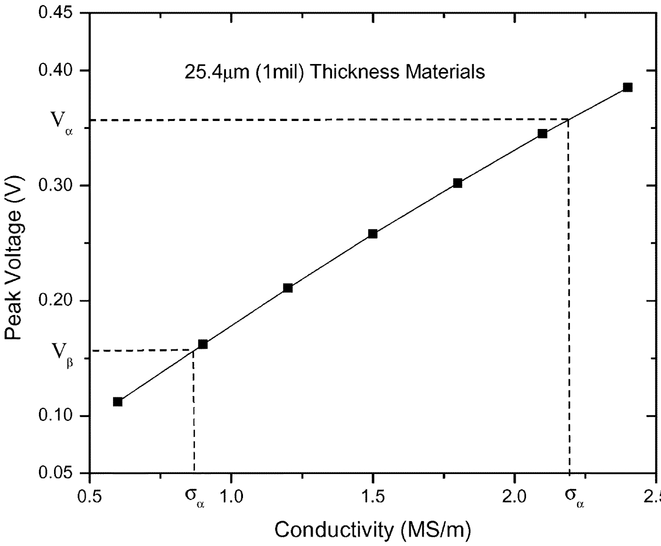

Fig. 12. Peak voltage versus conductivity for a coating thickness of 25.4 μm.

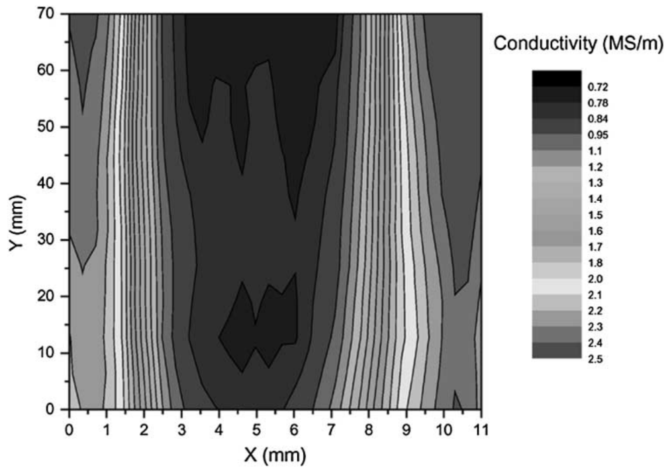

Fig. 13. Contour lines showing the conductivity map of the Ta coating. The center regions shows the beta phase and the outer regions are the alpha phase.

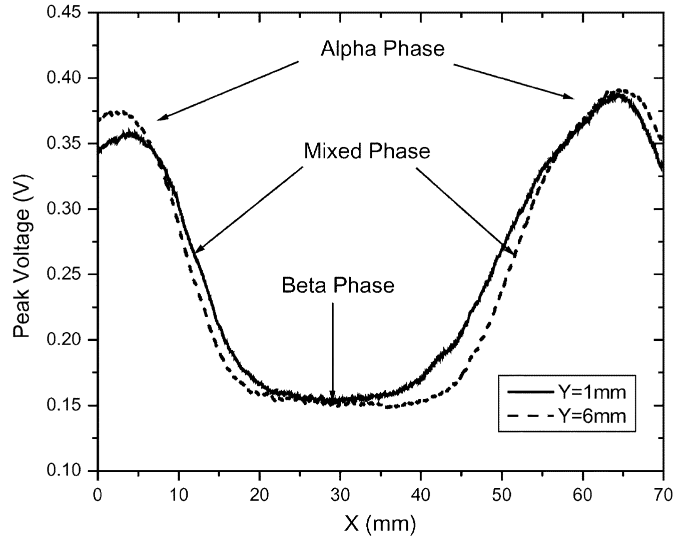

Fig. 14. A cut along the x axis for two locations in the y direction showing the details of the phase transition region.

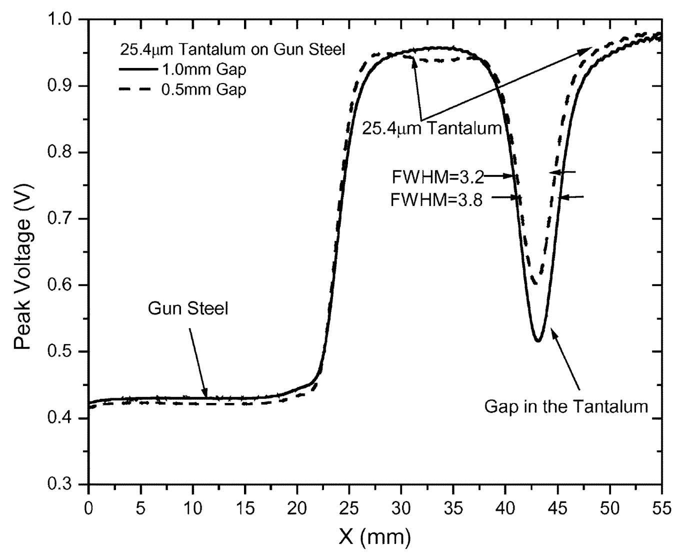

Fig. 15. One line from a scan across a 0.5-mm and 1-mm gaps in Ta on the gun steel substrate.

Based on the suppositions of the uniform thickness of Ta coating and the linear relationship between the peak voltage and the conductivity, we obtained the following false-color conductivity map by converting the raw data of Fig. 11 to conductivity (see Fig. 13).

A cut of raw signal along the axis of x for two locations in the y direction can give more information on the uniformity in the beta region and on the dimension of the transition region (see Fig. 14). The transition from alpha to beta phases occurs along a distance of about 10–15 mm and depends on the y location on the sample.

In order to estimate the resolution of the system, two Cu foils with thickness of 25.4 μm (1 mil) were placed on the gun steel substrate with a gap of 1 mm between them.

The thickness of the gun steel substrate is 2.5 mm. The line along the Ta foils is perfectly straight, which indicates that the system locates each sampling point exactly.

Lines from a scan of the configuration with a gap of 1 mm and 0.5 mm are shown in Fig. 15. For 0 mm $< x <$ 20 mm, only the substrate is scanned. The gap in the Cu coating is clearly visible. The full-width at half-maximum (FWHM) of signal from the gap is used as a measure of the resolution. This gives a value of 3.2 mm for the 0.5-mm gap and 3.8 mm for the 1-mm gap. The resolution is mostly a function of the coil diameter, which for this case is about 2 mm (see Table I).

The imperfect resolution will cause the inferred conductivity in the gap to be higher than that of the substrate. However, this test demonstrates that even with a coil diameter of 2 mm, we can easily detect features with dimensions of 0.5 mm.

V. Summary

A conductivity scanner based on eddy currents was constructed and used to determine the conductivity of tantalum coatings. The large difference between the conductivity of the alpha and beta phases in tantalum ($\sigma_\alpha \approx 10\sigma_\beta$) allows this method to be used in order to distinguish between the phases.

The feasibility of eddy-current methods to measure the conductivity and distinguish between the phases was demonstrated. Furthermore, an accurate measurement of the conductivity of the alpha and beta phases of one of the samples was done. The measurements were done by calibration of the system to known conductivities of thin foils over magnetic gun steel. The results obtained are in good agreement with other published measurements. The conductivity of the coating depends on the sputtering process and thus provides a method to characterize the quality of the coating.

An eddy-current scanner was designed, constructed, and used to scan planar samples. The pulsed eddy-current method was used to develop a technique that allows very fast scan rates with up to 10^5 samples per second. Such scanning capability makes this method useful for coating diagnostics during the manufacturing process in the field.

The spatial resolution of the system depends on the size of the probe. For the experiment reported here, a resolution of about 3 mm was obtained and features with dimensions of 0.5 mm were resolved. It is feasible to construct much smaller probe coils that can provide a resolution of about 0.1 mm; however, this requires more research to optimize the size of the probes.

The measurement reported here indicates that detection of mixed alpha and beta phases is possible but more research to actually quantify the sensitivity is required.

The measurement theory of the pulsed eddy current is well understood and our calculations agree well with the measurements. The ability to accurately predict the probe response for different coating thickness or conductivity is very important in interpreting the results and optimizing the measurements. In order to further improve the method, we would like to investigate several ideas. As indicated in this paper, the probe response depends both on the coating conductivity and thickness. The contribution of the thickness is relatively small but when trying to detect small variations in the conductivity, this could be a limiting factor. The fact that relative contribution of the thickness and conductivity varies with the probing frequency can be used as a method to measure both the conductivity and thickness of the coating. Theoretical calculations and measurements will be used to study this method. The same methodology can be used to try and reduce the effects of coil liftoff. Again, this is a small effect, but elimination of the effect will increase the sensitivity of the scanner.

The conductivity measured with the pulsed eddy-current technique is the weighted average conductivity of the coating where the weight is exponentially decaying from the surface of the coat. The penetration of the magnetic field to the sample is a function of the frequency. By using several frequencies, we can obtain information from different depths, which are weighted differently. This redundancy of information can be used for determination of the coating conductivity as a function of depth. The pulsed eddy-current method provides all these frequencies in one pulse. This frequency domain information can possibly be used to add a depth dimension to this scanner and thus provide the three-dimensional conductivity map of the coating. This information is valuable to the study the coating characteristics because it provides information on the interface between the coating and the substrate.

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