INL/CON-16-38345
PREPRINT

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43rd Review of QNDE (2016)

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July 2016

Determining Confounding Sensitivities In Eddy Current Thin Film Measurements

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Abstract. Eddy current (EC) techniques are widely used in industry to measure the thickness of non-conductive films on a metal substrate. This is done by using a system whereby a coil carrying a high-frequency alternating current is used to create an alternating magnetic field at the surface of the instrument's probe. When the probe is brought near a conductive surface, the alternating magnetic field will induce ECs in the conductor. The substrate characteristics and the distance of the probe from the substrate (the coating thickness) affect the magnitude of the ECs. The induced currents load the probe coil affecting the terminal impedance of the coil. The measured probe impedance is related to the lift off between coil and conductor as well as conductivity of the test sample. For a known conductivity sample, the probe impedance can be converted into an equivalent film thickness value. The EC measurement can be confounded by a number of measurement parameters. It was the goal of this research to determine which physical properties of the measurement set-up and sample can adversely affect the thickness measurement.

The eddy-current testing was performed using a commercially available, hand-held eddy-current probe (ETA3.3H spring-loaded eddy probe running at 8 MHz) that comes with a stand to hold the probe. The stand holds the probe and adjusts the probe on the z-axis to help position the probe in the correct area as well as make precise measurements. The signal from the probe was sent to a hand-held readout, where the results are recorded directly in terms of liftoff or film thickness. Understanding the effect of certain factors on the measurements of film thickness, will help to evaluate how accurate the ETA3.3H spring-loaded eddy probe was at measuring film thickness under varying experimental conditions. This research studied the effects of a number of factors such as i) conductivity, ii) edge effect, iii) surface finish of base material and iv) cable condition.

This work was performed with support from the Department of Energy under the United States National Nuclear Security Administration (NNSA) at the Idaho National Laboratory (INL).

BACKGROUND AND MOTIVATION

The Hot Fuel Examination Facility (HFEF) at Idaho National Laboratory(INL) houses a unique hot-cell where hot fuel can be analyzed, either destructively or non-destructively. A characterization system called the plate checker has been installed in HFEF at the INL to measure oxide film thickness on newly developed, irradiated fuel samples. These novel fuels are designed as part of the nationwide effort to reduce the enrichment of Uranium fuels used in research reactors. By analyzing the irradiated surface of the new, lower enriched fuels, we can better understand how they will perform in an actual reactor.

Fuel thermal conditions are calculated by coupling plate power conditions with oxide thickness measurements. The oxide layer thickness on the fuel plate can grow with increasing the surface temperature and is used to determine plate fuel performance and estimate fuel burnup. This increases the fuel meat centerline temperature and causes low thermal conductivity of the oxide. It also reduces the reliability and lifetime of the fuel plate. EC sensors are utilized for testing due to their compatibility in hot cells environments. Eddy current measurements of oxide thickness are also used estimate thermal conditions of the fuel. The fuel meat temperature profile is very sensitive to the thermal conductivity of the oxide layer. The effective thermal conductivity of this material can be estimated by treating it as a two-phase system using a modified Hashin and Shtrikman correlation. [1] These parameters are important in determining the performance of the fuel during irradiation. The eddy current sensors are non-contact and robust with respect to irradiation and hence will not alter the oxide layer. Sensor measurement accuracy or the determination of measurement uncertainty is crucial because the findings will determine whether a fuel will be suitable for reactor use.

Eddy current measurements utilizing historical data and current INL equipment are used to estimate the sensitivity of the oxide layer thickness to confounding factors. This study documented the results of a model-based approach, using the ETA3.3H eddy current probe, to conduct a systematic parametric study of potentially confounding factors to understand the effect of conductivity and oxide layers on EC measurement. The parameters considered: base material conductivity, edge effect, surface finish and cable condition. This report focuses on material conductivity and surface finish. EC measurements can also be affected due to the instrument's sensitivity, accuracy, resolution and penetration.

Each project that uses the plate checker measurement bench will need to understand the measurement system's sensitivity to confounding influences, as each measurement campaign will be significantly unique. Although this report shows that most of the confounding influences tested have a noticeable effect on a measurement, it will ultimately be the responsibility of each project to define what the acceptable uncertainty is. Once this acceptable uncertainty has been defined, then the appropriate mediation action or inaction can be made based on the sensitivity testing. Fundamentally, this EC study shows that confounding effects will affect the eddy current measurements.

Eddy Current System and Experimental Plan

The eddy current test is performed using a commercially available, hand held eddy current probe (ETA3.3H spring loaded eddy probe running at 8 MHz) that comes with a stand to hold the probe. The stand holds the probe and adjusts the probe on the z-axis to help position the probe in the correct area as well as make precise measurements, see Figure 1. The signal from the probe is sent to a hand held readout that displays thickness only. The measurements from the display are recorded directly in terms of liftoff or film thickness. The EC system can handle thickness measurements on non-conductive films and metal substrates. Calibration standards include base material AL T6 (in general) and Mylar Film Thicknesses: from 5.46 μm to 77.10 μm.

The probe was placed in a stand, so the probe was perpendicular to the surface of the metal test plate. Figure 1(a) shows the initial set up with the Aluminum T6 – Test Plate on the stand. The lever was pulled in order to lower the probe onto the Aluminum T6 – Test Plate pressing the probe against the Aluminum Test Plate as indicated in Figure 1(b). The probe was pressed against the plate 10 times (or as many times as the experiments required) and the resulting data was collected and processed. The data collected was from two types of tests designed to assess the sensitivity of the EC measurement to material composition and surface roughness. The measurements were made on Mylar films placed on various metallic substrates or a stainless steel block with varying surface finishes.

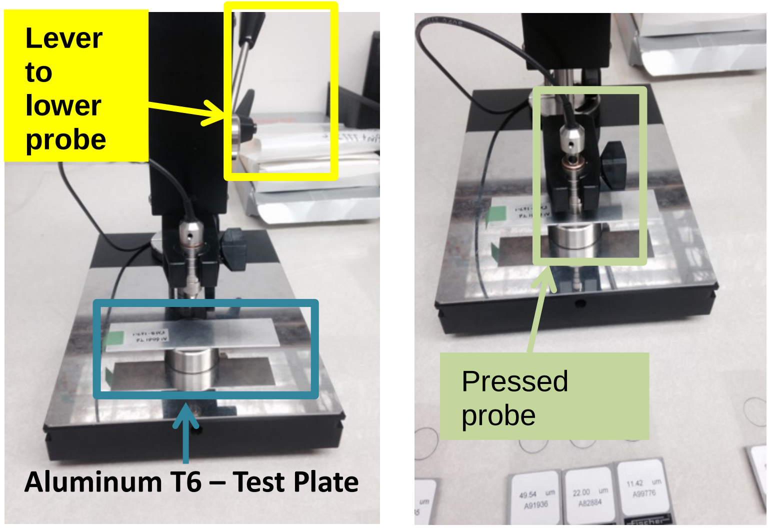

FIGURE 1. Experimental setup showing probe and Al T6 test plate with (a) probe raised (b) pressed on top of sample.

Composition & Microstructure Results

Substrate Composition

Material and microstructure changes can significantly affect EC measurements [2]. The materials tested and their conductivity values are shown in Figure 2.The material effects on EC measurements can be seen in Table 1. The measurements of film thickness on top of copper and the stainless steel are in significant error. Stainless steel has lower conductivity value than that of the calibration sample and the measured thickness values are seen to be consistently larger than the true film thickness. Alternatively, copper has a higher conductivity value than aluminum and the measured thickness values are consistently smaller than the true film thickness. This was expected since higher conductivity value of base material results in higher EC probe signal that can be translated as lower liftoff and hence smaller film thickness and vice versa.

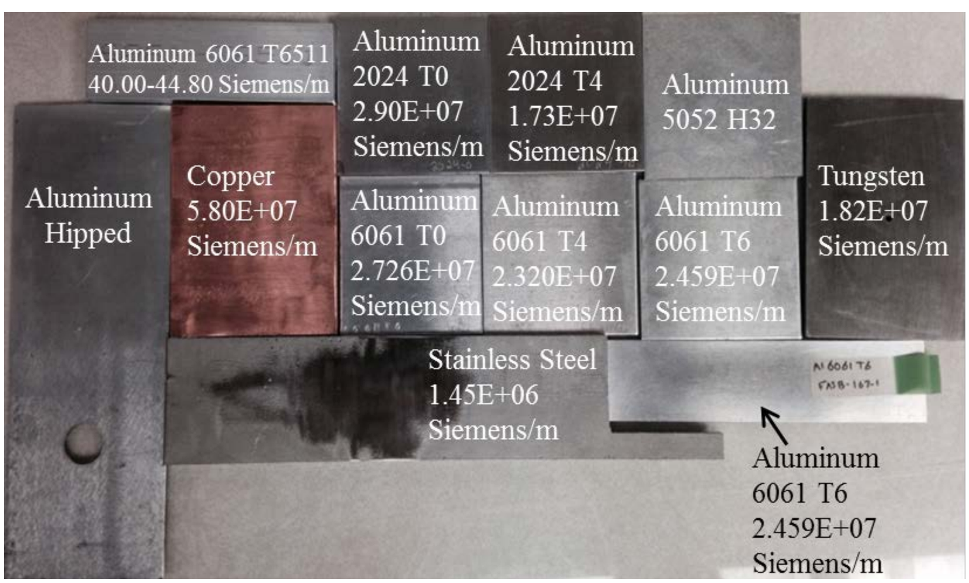

FIGURE 2. Materials with different conductivity values [3] affect the EC probe measurement.

TABLE 1. Mean values of measured film thickness on materials with different conductivity. The probe was calibrated on Al 6061-T6 plate.

		Film Thickness 11.42 (μm)	Film Thickness 22.00 (μm)	Film Thickness 49.54 (μm)
Alloy	Temper	Mean (μm)	Mean (μm)	Mean (μm)
Al 6061 – Test Plate	T6	11.49	22.62	49.20
Copper		9.53	20.19	48.10
Stainless Steel		19.73	30.10	58.11
Tungsten		10.35	21.85	48.69
Alloy	Temper	Standard Deviation (μm)	Standard Deviation (μm)	Standard Deviation (μm)
Al 6061 – Test Plate	T6	0.15	0.05	0.05
Copper		0.79	0.28	0.75
Stainless Steel		0.21	0.09	0.07
Tungsten		0.06	0.24	0.16

Alloy and Temper

Aluminum (Al) temper can affect the measurement more than composition changes. The results in Table 2 can be at odds with the data in Table 1. The errors shown in both tables are relatively large. Some of the base material conductivity values that are largely different from that of the calibration sample are seen to have larger errors. However there was no observable correlation between conductivity difference and error in predicted film thickness.

TABLE 2. Mean values of thickness measured on each material with different conductivity value along with percent error. The probe was calibrated on Al 6061-T6 plate.

Alloy	Temper	Mean (μm)	Standard Deviation (μm)	Percent Error (%) (a)
Al 6061	T0	9.96	0.69	18.49
Al 2024	T0	11.55	0.29	5.48
Al 5052	H32	11.28	0.06	7.69
Al 2024	T4	11.69	0.07	4.33
Al 6061	T4	11.76	0.06	3.76
Al 6061	T6	11.75	0.85	3.84
Al 6061 – Test Plate	T6	11.78	0.21	3.60
Al 6061	T6511	14.80	0.84	21.11
Al 6061	Hipped	11.91	0.73	2.53
Tungsten		11.01	0.06	9.90

On the first row, the alloy Al 6016-T0 has the largest error namely, 18%, whereas the conductivity difference between this sample and the calibration sample was roughly 10%. The randomness of observations in Table 2 left concerns that will need to be investigated further since it is not a clear depiction of substrate conductivity. The conductivity [4] percentage compared to the percent error can be seen in Figure 3.

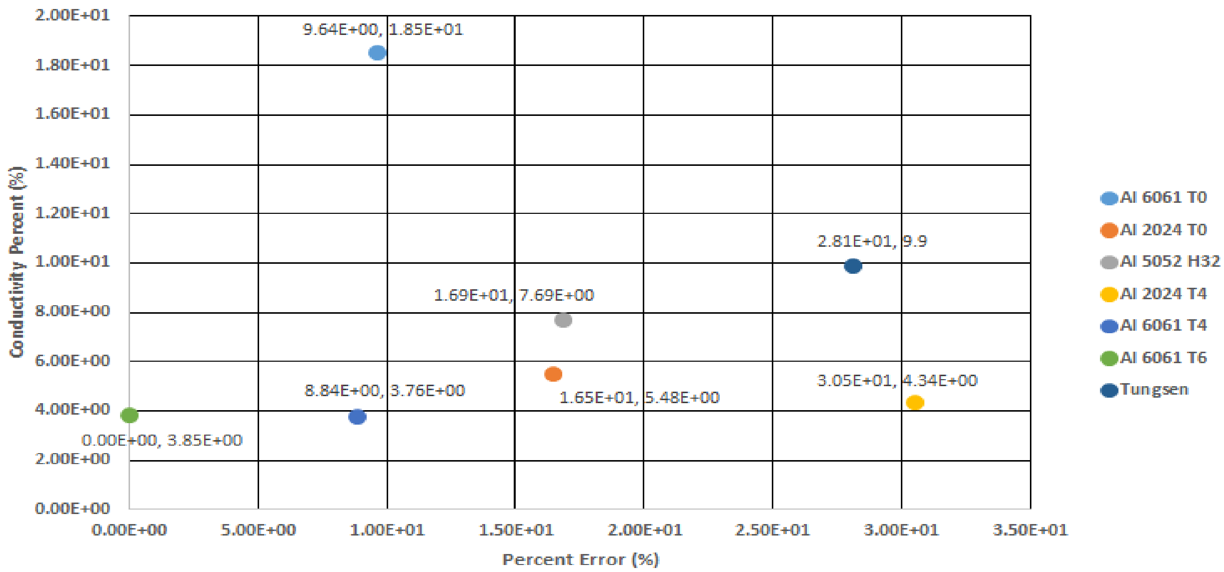

FIGURE 3. Conductivity Percent (%) vs Percent Error (%).

Measurement Anomaly Explanation

An anomaly occurs in the hot cell when using EC testing on an irradiated plate that can produce small negative values. The anomaly about recording negative thickness measurements in the hot cell with the installed eddy current system for small oxide thicknesses has been reproduced in this study. If the calibration sample has conductivity lower than the base material conductivity, the instrument can output negative thickness values as such in Table 3.

TABLE 3. Mean values of measured film thickness on base materials of different conductivity values. The probe was calibrated on Stainless Steel plate.

		Film Thickness 5.52 (µm)	Film Thickness 5.52 (µm)
Alloy	Temper	Mean (µm)	Standard Deviation (µm)
Stainless Steel		5.64	0.03
Al 6061 – Test Plate	T6	-0.25	0.08
Copper		-2.00	0.63

The reason for that this anomaly occurring in the hot cell is that irradiation changes substrate conductivity. In the lab the anomaly can be reproduced by calibrating the EC probe with a lower conductivity value (stainless steel). While the thickness measurements are made on substrates with larger conductivity value (Al or Cu). For that reason, film thickness measurements can come out negative or lower for larger thicknesses. The exact behavior will be an algorithm specific from EC vender.

Surface Roughness Measurement

This section documents the results of conducting the thickness measurement test on samples with different surface finishes. The objective of this test was to determine whether the surface finish affects the measurements of film thickness. The experimental surfaces used for the tests are shown in Figure 4.

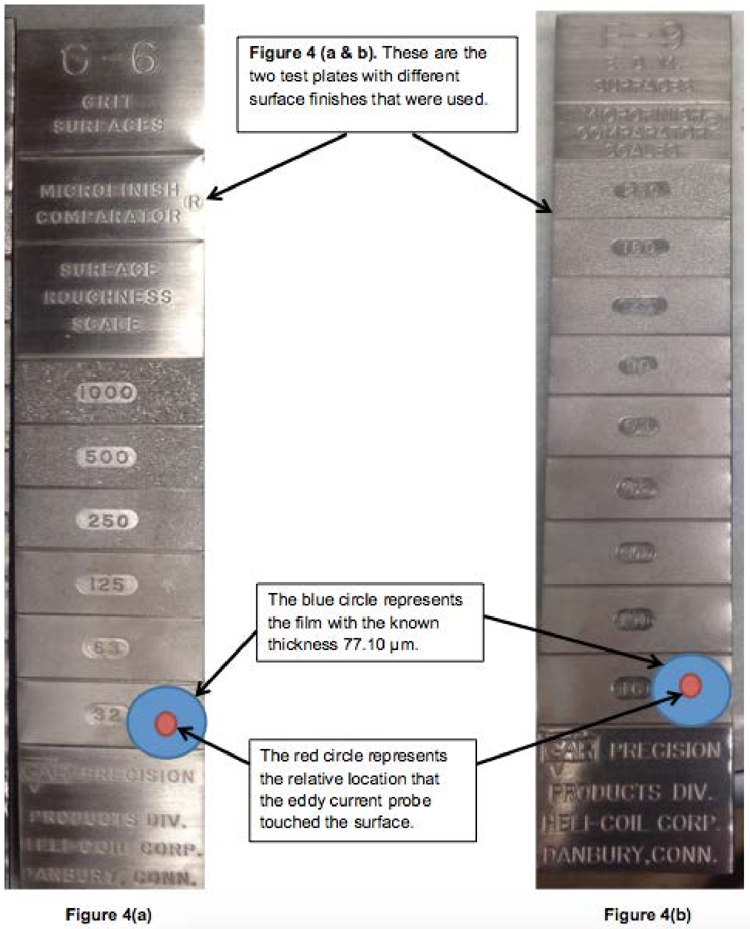

FIGURE 4. Blue circle represents the film with the known thickness 77.10 μm. Red circle represents the EC probe.

Surface roughness will affect EC measurements and the degree of measured thickness increases with roughness. Table 4 presented the mean value of the thickness obtained with different surface finish in the two metal test plates. The true film thickness was 77.10 (μm). Also the percent error was calculated using the formula $\frac{|\text{Actual} - \text{Meausured}|}{\text{Actual}} \times 100\%$, where "actual" value was 77.10 μm and "measured" was the output of the instrument. The percent error in film thickness measurement for each surface finish was provided in the last column.

TABLE 4. Measured values of film thickness on different surface finish along with standard deviation, and the percent error of measurements. (Actual Value = 77.10 μm).

Surface Roughness Scale (μin)	Mean (μm)	Standard Deviation (μm)	Percent Error (%)
1000	115	8.27	49.15
500	93.79	0.87	21.64
250	84.83	0.69	10.02
125	83.69	1.4	8.54
63	81.24	0.17	5.36
32	78.28	1.04	1.53

Table 5 presented the corresponding film thickness values measured (average of 10 measurements, standard deviation and percent error) on the second metal plate.

TABLE 5. Measured values of film thickness on different surface finish along with standard deviation, and the percent error of measurements.

Microfinish Comparator Scale (µin)	Mean (µm)	Standard Deviation (µm)	Percent Error (%)
250	88.48	3.02	14.76
180	85.30	3.12	10.63
125	81.23	1.09	5.35
90	79.96	0.22	3.70
63	86.30	1.8	11.93
45	84.30	3.17	9.33
32	78.46	0.26	1.76
24	78.73	0.55	2.11
16	78.05	0.35	1.23

The surface finish on which the thickness measurement was made had significant effect on the output of ETA3.3H EC probe system. Table 4 indicated that as the surface finish became coarser, the error in measured film thickness value grew larger. Table 5 also indicated that the error was small for finer surface finishes and increased with coarser grain finish. Hence, for the instrument to provide an accurate estimate of the oxide layer thickness, a good surface finish should be ensured. This conclusion is to be expected since the measurements are made at very high frequency (8MHz) and EC skin depth being very small at this frequency carries more information on the surface condition. Clearly, the instrument was very sensitive to surface condition of the sample on which measurement was made.

The performance of the instrument with respect to edge effect was consistent with the physics of eddy currents [5]. At 8 MHz, the fields are fairly localized and the footprint of the probe was very small. Unless the probe was on top of an edge or < 2 mm from edge, the results are not affected by edge. The effect of surface condition of the material was consistent with physics of eddy currents. At very high frequency, the probe was very sensitive to surface condition and hence thickness measurements. The capacitive effects at 8 MHz seem to be much smaller and did not produce significant error when the cables were kinked and coiled.

CONCLUSIONS

The experiment's objective was to determine the performance of the EC instrument in predicting the known thickness of films with respect to accuracy and repeatability of measurements by the introduction of confounding factors. The findings of this work indicate three major opportunities to improve upon measurement uncertainties with current equipment, and technique:

1) sensitivity of conductivity of substrate to that of calibration sample
2) sensitivity to surface condition of substrate.

The conductivity of the calibrated sample in relation to the base material is expected to affect thickness measurement. This was the case when the base materials chosen for calibration were Aluminum alloys, copper and stainless steel. When differently treated alloys of Aluminum were chosen as base material, no consistent correlation was observed, however some alloys did cause significant variations. This raises the possibility that the conductivity values used from the handbooks may differ from true conductivity of the material.

The anomaly about recording negative thickness measurements in the hot cell with the installed eddy current system for small oxide thicknesses has been reproduced in this study. If the calibration sample has conductivity lower than the base material conductivity, the instrument can output negative thickness values.

In conclusion, potential confounding effects with EC measurement uncertainty span irradiated fuel plates, composition changes, microstructure changes and surface roughness. EC testing has shown that these effects change substrate properties and prove that the calibration standard is less than a perfect match due to material components, temper, surface finish, etc.

FUTURE WORK

Another parameter that can vary was temperature of sample, coil and cable. Temperature affects the conductivity of a sample and also the performance of the probe, which includes the effects of the cable and the probe coil. These variations can be fully characterized a priori using experimental data and applied for a given measurement temperature.

If adequate data is not available, temperature variations can be compensated by using invariance transformation using multiple frequencies.

At a high operating frequency (≈8Mhz), the skin depth is very small and measurements should be relatively insensitive to conductivity, but studies conducted in this work show otherwise. Consequently we have unknowns, namely, oxide layer thickness and conductivity of substrate material. It is required to estimate the oxide thickness in a manner that is independent of conductivity since the irradiation effects will be unknown. The solution of such a problem will require multiple measurements, which can be obtained at taking measurements at multiple frequencies.

Work in the use of multi-frequency, along with additional information in EC probe impedance to estimate conductivity and oxide thickness, will refine the information obtained from EC probe. The temperature effect can be compensated with consideration of temperature variations during the measurement, or by utilizing the 3rd frequency measurement to compensate for fluctuation in temperature as part of the processing algorithm. Multi-frequency measurements, using a single probe coil, is commonly used in many EC testing applications [6]. One example is Steam Generator tube inspection in nuclear power plants, where the measurements are taken at 4 frequencies with the same probe coil in a single scan using time multiplexing. Advanced signal processing algorithms based on invariance transformation can be used to make the data insensitive to unwanted parameters such as base conductivity, temperature and surface roughness, while retaining sensitivity to parameters of interest which is the oxide layer thickness [7,8]. The ability to use the same coil for the multiple frequencies will minimize the scanning time of a measurement bench. This also means that installed EC benches would be able to use the in-cell equipment with minor modifications.

ACKNOWLEDGEMENTS

This research could not have been possible without the assistance of Francine Rice, Steve Marschman, Adam Robinson, and Walter Williams at the INL. This work was supported by of Idaho National Laboratory and the Department of Energy.

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