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SELF-CONSISTENT SWEPT FREQUENCY EDDY CURRENT MEASUREMENTS FOR CHARACTERIZATION OF NEAR SURFACE MATERIAL CONDITIONS

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ABSTRACT. This paper reports on a self-consistent, swept frequency eddy current (SFEC) technique for characterizing surface and sub-surface conditions of materials, with specific applications to detecting residual stresses in shot-peened Ni-base superalloys and surface oxidation in engineering components. The technique involves measuring lift-off normalized vertical component signal to suppress lift off noise and instrumentation effect. Theoretical study shows that the vertical component signals are insensitive to coil dimensions, thus enabling EC measurements in separate frequency bands using multiple coils, while yielding continuous broad-band data so that both the bulk conductivity and near-surface conductivity profile can be determined by model-based inversion. We demonstrate the technique on two surface-modified materials, namely Inconel 718 samples shot peened at 4A to 8A, and an Ag-1.5at%Al alloy which was used as a model material for a fundamental study of internal oxidation. For each sample set, the vertical component signals measured using two dissimilar sets of coils and instruments were found to overlap, confirming that the signals are insensitive to coil dimensions and instrumentation. The bulk conductivities of the samples were determined by inverting the low frequency data. The results were then used to constrain model-based inversion of the high frequency data to obtain near-surface conductivity profiles, from which the residual stress profile of the shot-peened Inconel 718 and the oxidization depth of the Ag-Al alloy can be inferred.

Keywords: Swept Frequency Eddy Current, Residual Stress, Shot Peening, Oxidation
PACS: 81.40.Ef, 81.40.Rs, 81.65.-b, 81.65.Mq, 81.70.Ex, 81.70.Jb,

INTRODUCTION

This paper reports on a self-consistent, swept frequency eddy current (SFEC) technique for characterizing surface conditions of materials by means of model-based inversion of electrical conductivity depth profiles without requiring any prior knowledge of their baseline conductivity. The technique, which is based on the measurements of lift-off normalized vertical component signals [1], was initially developed for residual stress measurements on surface-treated aerospace components in particular shot-peened nickel-base superalloys [2, 3]. For such application, high frequency operations up to 50 MHz or

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more are needed in order to achieve a small enough skin depth (preferably of tens of microns) for residual stress profile measurements due to the relatively low conductivities of the engine materials. The measurement technique offers the advantages that it suppresses the lift off noise and instrumentation effect [4], and at the same time allows direct comparison of experimental data with theoretical calculations for model-based inversion of conductivity depth profiles [5]. Specifically, the experimental lift off normalized vertical component signal V_{EX} is defined as

$$V_{\mathrm{EX}} \equiv \mathrm{Im}\left(\frac{V_T^O - V_R^O}{V_L^O - V_R^O} \right) \tag{1}$$

where V^0_{R}, V^0_{L}, and V^0_{T}, denotes the reference signal, the lift off signal and the signal from a surface-treated material, respectively. For direct comparison with experiment, a theoretical vertical component signal V_{TH} is introduced as

$$V_{\mathrm{TH}} \equiv \mathrm{Im}\left(\frac{Z_T - Z_R}{Z_L - Z_R} \right) \tag{2}$$

where Z_{R}, Z_{L}, and Z_{T} are the coil impedances for the reference, lift off, and test configurations, respectively. Given a coil impedance deviation (i.e. Z_{T} - Z_{R}) caused by different conductivity profiles of the reference and test samples, the instrument output voltage shows a corresponding deviation (V^0_{T} - V^0_{R}). The proportionality factor (i.e. the multiplicative transfer function) is instrument-dependent but material-independent. If the instrumentation conditions are kept the same for the three measurements, the transfer function cancels out in the ratios defined in Eqns. (1) and (2), and therefore $V_{\mathrm{EX}} = V_{\mathrm{TH}}$.

It is clear from Eqns. (1) and (2) that the vertical component signal measurement technique is primarily for characterizing the near-surface conditions of a test sample relative to a reference material, which is typically chosen to be the same material or even from the same batch with similar baseline conductivity as the test object. Nevertheless, any difference in bulk conductivity between the test and reference samples arising from, for example, different processing, heat treatment or service-induced changes in the bulk material conditions of the test object, also contributes to the detected vertical component signals. This needs to be taken into account when inverting SFEC data for near-surface conductivity profiles. In this paper, we report on a self-consistent approach for surface characterizations by utilizing broad-band SFEC data to account for any bulk conductivity difference between the test and reference samples. Our theoretical study has shown that the vertical component signals are insensitive to coil dimensions, thus enabling EC measurements in separate frequency bands using multiple coils to yield continuous broad-band data. We demonstrate the technique on two surface-modified materials, namely a series of Inconel 718 samples shot peened at 4A to 8A, and an Ag-1.5at%Al alloy subjected to internal oxidation. The latter was used as a model material in a fundamental study of internal oxidation, as the alloy exhibits a well-defined surface oxide layered structure and the oxide layer thickness can be controlled by adjusting the oxidation time and temperature [6]. For each sample, we found that the vertical component signals measured by using two dissimilar sets of coils and instruments overlap, confirming that the signals are insensitive to coil dimensions and instrumentation. The bulk conductivities of the samples were determined by inverting the low frequency data. The results were then used in model-based inversion of the high frequency data to obtain near-surface conductivity deviation profiles, from which the residual stress profile of the shot-peened Inconel 718 and the oxidization depth of the Ag-Al alloy can be inferred.

EXPERIMENTAL DETAILS

The Inconel 718 samples used are 3”×2”×0.5” (76 mm×51 mm×13 mm) blocks from the same batch. The samples’ surfaces were polished to mirror finish before shot peening. The polished surfaces were found to have different baseline conductivities using a conductivity gage operated at 60 kHz (Table 1). For each sample one polished surface was retained for reference and lift off measurements, while the other surface was shot peened at various Almen intensities from 4A to 8A. Swept frequency EC measurements were carried out over two different but overlapping frequency bands using (i) a differential pair of 12 mm, 14-turn spiral coils fabricated on a printed circuit broad (PCB) and a network analyzer (Agilent E5061A) for SFEC measurements from 300 kHz to 65 MHz, and (ii) an air-core 244-turn pancake coil together with an impedance analyzer (Agilent 4292A) for frequencies between 100 kHz and 2 MHz (Fig. 1). For each experimental setup, three sets of measurements were carried out to obtain vertical component signals V_{EX} (Eqn. (1)) which were directly compared with model predictions (Eqn. (2)) recursively to obtain the conductivity profiles up to a depth of 500 μm.

A series of drop-cast Ag-1.5at%Al discs 3/4” (19 mm) in diameter were used in the fundamental study of internal oxidation. The baseline conductivities of the samples were measured using the conductivity gage before oxidation. The samples were oxidized at 600°C in air for various time periods (Table 2). SFEC measurements were performed on the samples before and after oxidation using the impedance analyzer together with a 244-turn coil from 100 kHz to 1 MHz, and a 63-turn (or 50-turn) coil from 1 MHz to 5 MHz.

TABLE 1. Measured and inverted substrate conductivities of the Inconel 718 samples.

Sample	**Side**	**Surface condition**	**rms roughness (μm)**	**Conductivity gage reading for as-polished surfaces (%IACS)**	**Estimated substrate conductivity σ_{Sub} of shot-peened surfaces (%IACS)**
S1	Top Bottom	Polished Peened 4A	0.07 0.44	σ^{Top}_0 = 1.400±0.004 σ^{Btm}_0 = 1.393±0.003	1.392
S2	Top Bottom	Polished Peened 6A	0.04 0.62	σ^{Top}_0 = 1.375±0.003 σ^{Btm}_0 = 1.383±0.003	1.384
S3	Top Bottom	Polished Peened 8A	0.05 0.69	σ^{Top}_0 = 1.378±0.004 σ^{Btm}_0 = 1.384±0.002	1.388

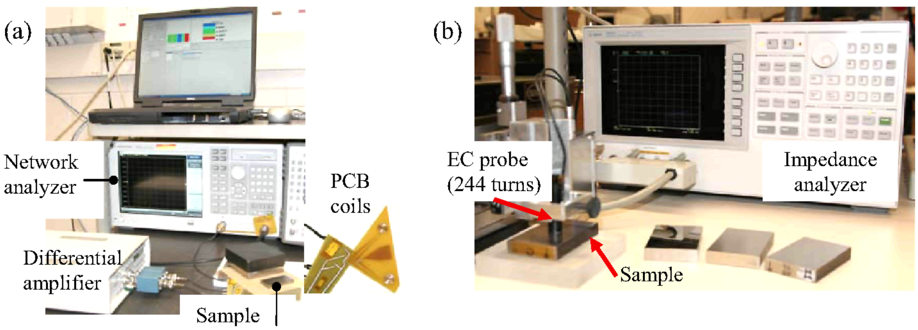

FIGURE 1. Experimental setups for SFEC measurements using (a) a differential pair of 12 mm, 14-turn spiral coils fabricated on a printed circuit broad (PCB) and a network analyzer (Agilent E5061A), and (b) an air-core 244-turn pancake coil together with an impedance analyzer (Agilent 4292A).

TABLE 2. Oxidation conditions, baseline conductivities measured using a conductivity gage, as well as the inverted thicknesses and conductivities of the surface oxide and subsurface Al-depleted layers for the Ag-Al samples.

Sample	Oxidation condition	Surface	Baseline conductivity * (%IACS)	Oxide layer		Al-depleted layer	
				Thicknes s (μm)	Conductivity (%IACS)	Thickness (μm)	Conductivity (%IACS)
O1	600°C 32 mins	Top	40.1	43.9	35.4	33.7	46.4
		Bottom	35.6	39.6	35.0	11.0	52.1
O2	600°C 130 mins	Top	47.1	76.6	38.9	4.7	106.7
		Bottom	45.0	56.7	36.2	6.8	57.6
O3	600°C 293 mins	Top	43.7	84.6	41.3	58.6	53.4
		Bottom	43.0	91.9	41.3	22.4	62.6
O4	600°C 293 mins	Top	40.4	95.1	36.3	14.0	84.8
		Bottom	40.7	103.0	35.6	11.9	65.8

RESULTS OF SWEPT FREQUENCY EC MEASUREMENTS

Shot-peened Inconel 718

As shown in Fig. 2, the vertical component signals measured from the shot peened Inconel 718 samples using the two different set of coils and instruments show continuous curves over the entire frequency band. The samples exhibit significantly different trends at low frequencies (< ~1 MHz), due to the fact that each of the shot peened surfaces has a different substrate conductivity relative to the reference surface. Such effect was accounted for when performing model-based inversion of the near-surface conductivity profiles.

Oxidized Ag-Al Alloy

The vertical component signals are shown in Fig. 3 for two of the oxidized Ag-Al samples. The vertical component signals measured using two different coils show continuous curves throughout the entire frequency range. Of special note is that a peak is observed at low frequencies (~ 300 kHz) for some of the oxidized samples, suggesting the presence of a more conducting sub-surface layer underneath the surface oxide layer. This is attributed to Al depletion at the oxidation front, where Al was consumed to form aluminum oxide precipitations during oxidation. Subsurface Al-depletion has been found in an oxidized Ni-Al alloy by measuring its composition depth profiles. The Al-depleted layer is rich in Ag and is therefore more conducting than the substrate.

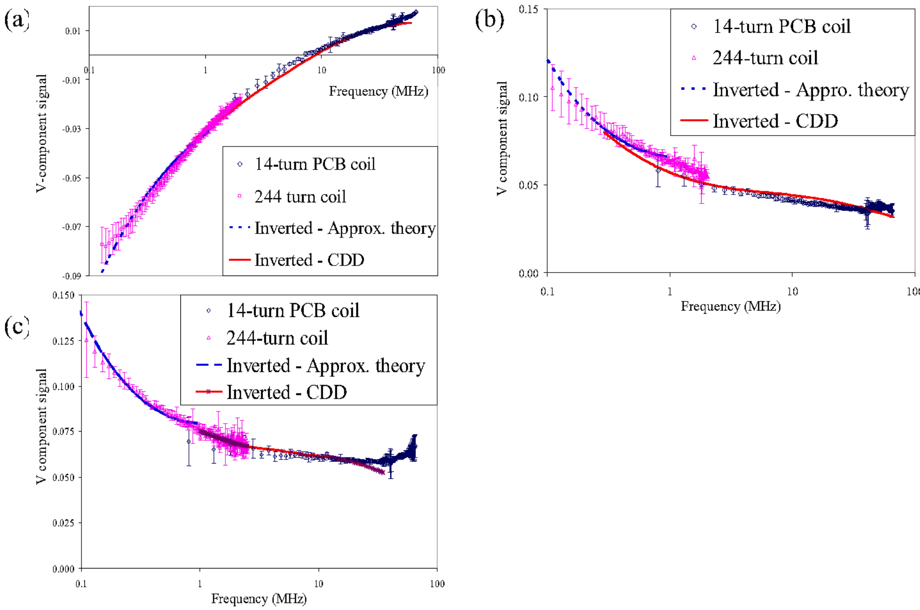

FIGURE 2. Experimental swept frequency vertical component signals V_{EX} (from 100 kHz to 65 MHz) measured using a 12mm, 14-turn PCB coil and 244-turn coil from a series of Inconel 718 samples shot peened at (a) 4A, (b) 6A and (c) 8A. The dotted and solid lines correspond to the modeled vertical component signals V_{TH} at low and high frequencies using the approximation theory and the multi-layer model of Cheng-Dodd-Deed, respectively.

MODEL-BASED INVERSION OF CONDUCTIVITY PROFILES

Shot-peened Inconel 718

The effects of bulk conductivity difference on the vertical component signals measured from the shot-peened Inconel 718 samples can be described by means of a perturbation theory. The theory provides a simplified expression to describe the vertical component signal for materials with a depth profile of conductivity deviation $\Delta\sigma(z)$ which is much smaller than the bulk conductivity σ_{Ref} of the reference material (i.e. $\Delta\sigma(z)/\sigma_{Ref}$ <<1). In the simplest case where a surface-treated material is represented by a single surface layer of thickness d and a conductivity σ_{Surf} overlaying an homogeneous substrate with conductivity σ_{Sub} (where $|\sigma_{Surf} - \sigma_{Sub}| << \sigma_{Sub}$), the vertical component signal can be expressed as function of frequency f as

$$V_{TH} = \sqrt{\pi\mu_0\sigma_0}\,\frac{d^2}{l}\frac{\Delta\sigma_S}{\sigma_0}\sqrt{f} + \frac{1}{4l\sqrt{\pi\mu_0\sigma_0}}\frac{\Delta\sigma_0}{\sigma_0}\frac{1}{\sqrt{f}} \qquad (3)$$

where $\Delta\sigma_S = \sigma_{Surf} - \sigma_{Sub}$ and $\Delta\sigma_0 = \sigma_{Sub} - \sigma_{Ref}$, and d is the surface layer thickness. Here, all σ_{Ref}, σ_{Surf}, and σ_{Sub} deviate slightly from the nominal sample conductivity σ_0 that we measured to be 1.38%IACS (0.800MS/m). The first term, which arises from conductivity deviation in the surface layer, becomes increasingly important at high frequencies. The second term describes the contributions of any bulk conductivity difference between the test and reference samples to the detected vertical component signals at low frequencies. Eqn. (3) provides a means to determine the bulk conductivity σ_{Sub} of the test object (e.g. a

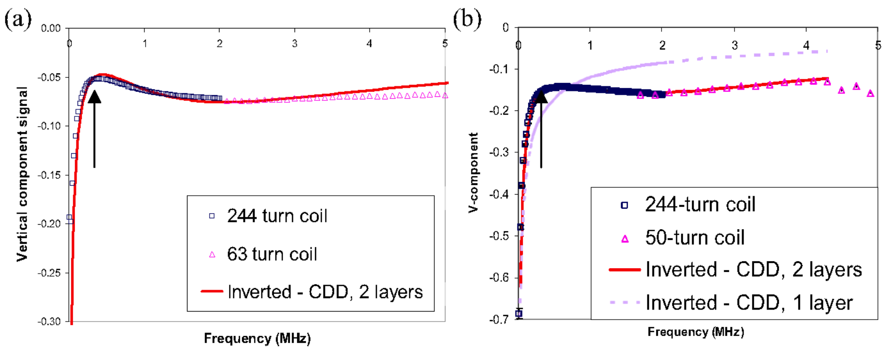

FIGURE 3. Swept frequency vertical component signals V_{EX} (from 100 kHz to 5 MHz) measured using a 244-turn coil and a 63-turn (or a 50-turn) coil from the (a) bottom surface of sample O3 and (b) the top surface of sample O4. The peaks at about 200 kHz indicate the presence of a more conductive subsurface layer in the samples. The solid lines in (a) and (b) correspond to the calculated vertical component signals V_{TH} using the CDD model by assuming two surface layers overlaying on the non-oxidized substrate. The dotted line in (b) corresponds to the calculated vertical component signals V_{TH} by assuming a single surface layer. The arrows highlight the signal peak at around 300 kHz.

peened surface) from the low-frequency vertical component signals without requiring any priori knowledge of its baseline conductivity.

The vertical component signals from 100 kHz to 1 MHz was fitted to determine the substrate conductivity σ_{Sub} of the peened samples using Eqn. (3). As shown in Table 1, the inverted substrate conductivities agree reasonably well with the values directly measured from the samples before shot peening using a conductivity gage. The estimated substrate conductivities were used to determine the near-surface conductivity profiles of the samples using the analytical multi-layer EC model of Cheng-Dodd-Deed (CDD) [7]. The conductivity profile is parameterized as a function of depth z as [5]

$$\left(\sigma(z)-\sigma_{Sub}\right)/\sigma_{Sub}=e^{-z/\lambda}(a_0+a_1z+a_2z^2+a_3z^3+a_4z^4). \qquad (4)$$

This empirical function is chosen to capture typical residual stress profiles of shot-peened materials.

Oxidized Ag-Al Alloy

For model-based inversion, the oxidized Ag-Al samples are represented by a simplified layered structure which consists of two surface layers overlaying on a homogeneous, non-oxidized substrate. This material model was used to account for the possible occurrence of a more conducting, sub-surface Al-depleted layer as indicated by the signal peaks in the SFEC data (Fig. 3). The fitting parameters, including the thicknesses and conductivities of the oxide layer and the Al-depleted layer, were determined by model-based inversion of SFEC data using the CDD multi-layer model.

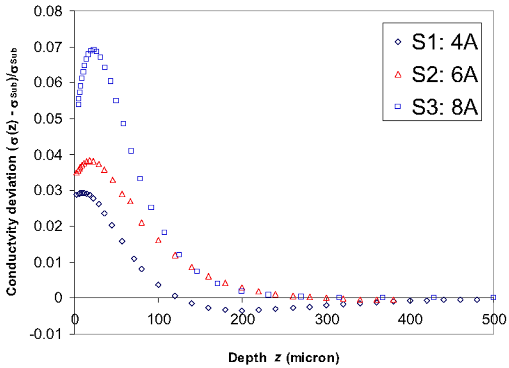

FIGURE 4. Inverted conductivity profiles for the Inconel 718 samples shot-peened at 4A, 6A and 8A.

INVERSION RESULTS

Shot-peened Inconel 718

The inverted conductivity profiles of the shot-peened Inconel 718 samples are shown in Fig. 4. The modeled vertical component signals are shown as solid lines in Fig. 1 for comparison with the experimental data. The shot peened samples show increased conductivity in the surface layer relative to the substrate value, presumably due to the shot-induced residual compressive stresses which have been found to increase conductivity via the piezoresistivity effect [2]. The peak conductivity value tends to increase and the peak position shifts to a larger depth with increasing Almen intensity. Similar trend has been observed in our previous study of another series of Inconel 718 samples shot peened at higher Almen intensities [5].

Oxidized Ag-Al Alloy

The inverted thicknesses and conductivities of the oxide and subsurface Al-depleted layer of the oxidized Ag-Al samples are shown in Table 2. The modeled vertical component signals (solid lines in Fig. 3) closely follow the experimental data, despite the simplicity of the double-layer model to represent the oxidized samples. The inversion results show that the oxide layer thickness increases monotonically with time for a given oxidation temperature, and the subsurface layer is more conducting than the substrate for all samples. In order to evaluate the validity of the double surface layer structure, inversion was repeated by assuming a single surface layer only. As shown in Fig. 3 (b), the best-fitted vertical component (dotted line) based on the single layer model cannot satisfactorily describe the experimental results, indicating that it is necessary to include the subsurface layer to capture the conductivity profiles. The inversion can be further improved by parameterizing the conductivity profile using physic-based or empirical functions, such as the error function which represents a solution to diffusion processes. Destructive characterizations of the oxidized Ag-Al samples by electron microscopy and other analytical techniques are underway in order to verify the inversion results.

CONCLUSIONS

This paper presents a self-consistent, swept frequency eddy current technique for characterization of surface-treated materials. The method is based on measurements of lift-off normalized vertical component signals over a wide frequency band extending nearly to three decades by using multiple coils and instrument, so that both the bulk and near-surface conductivity deviations can be consistently determined by means of model-based inversion. The technique was applied to characterize the near surface conditions of shot peened Inconel 718 samples, and a model Ag-1.5at%Al alloy subjected to internal oxidation. The liftoff-normalized vertical component signals measured from these samples using multiple coils and instrument in separate frequency bands yield continuous broad-band data. The bulk conductivities of the samples inverted from low frequency EC data agree with the values directly measured using a conductivity gage. For the shot peened Inconel 718, the inverted near-surface conductivity deviation profiles show a single peak which increases in magnitude and shifts to larger depth with increasing Almen intensity due to shot-induced compressive residual stresses. Inversion results show that the oxidized Ag-Al samples can be adequately described in terms of a double layer model comprising a less conducting surface oxide layer whose thickness increases with oxidation time, and a subsurface layer which is more conducting than the material matrix due to Al depletion.

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