TECHNICAL NOTE

EDDY CURRENT METHODOLOGY FOR NONDESTRUCTIVE ASSESSMENT OF THICKNESS OF SILICON CARBIDE COATING ON CARBON-CARBON COMPOSITES

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Abstract

Eddy current (EC) methodology has been developed for non-destructive assessment of thickness of Silicon carbide (SiC) coating on carbon-carbon (C/C) composite specimens. For coating, polymer precursor route has been employed and approximate coating thickness has been determined by weight gain method. EC probe signal amplitude has been measured before and after the coating. Despite scatter in the baseline data due to surface roughness on C/C specimens, EC methodology could identify undercoated (thickness <20 μm) specimens. A calibration graph has been established between EC signal amplitude and coating thickness (weight gain) to evaluate the performance of the EC methodology. The methodology has evaluated the coating thickness with error less than ±5 μm. Studies confirm that it is possible to assess the efficacy of coating process and to readily identify thinly coated regions using eddy current imaging.

***Keywords**: Carbon-carbon composites, Silicon carbide, aerospace, eddy current, NDE, coating*

Introduction

Composite materials find widespread use in aerospace industry as structural material for components such as tails, wings, fuselages and propellers due to their lightweight, high strength and ability to withstand harsh loading conditions. Carbon composite is a key material in today's launch vehicles and spacecraft. The spacecrafts and reentry vehicle components such as nose cap and wing leading edges experience high temperatures exceeding 1200^{o}C during the atmospheric entry. For thermal protection of these parts, Carbon-Carbon (C/C) composites are preferred. C/C composites are known for their high strength to weight ratio, excellent mechanical properties at high temperatures (as high as 2500^{o}C) and their ability to retain these properties under extreme conditions [1, 2]. However, they are oxidized on exposure to oxidizing atmospheres above 500^{o}C and this limits their use as high temperature structural material. As a solution, C/C composites are coated with oxidation resistant materials such as Silicon carbide (SiC). In air, SiC forms a protective Silicon oxide coating at ~1200^{o}C and this oxide coating is stable up to 1600^{o}C. It is essential to ensure that SiC coating on C/C composite structures is uniform and more than 20 μm thick. Hence, utmost care should be taken during manufacturing stage to ensure the quality and thickness of SiC coating. Nondestructive evaluation (NDE) techniques play a vital role in this regard.

Traditionally, various NDE techniques are used for characterization of coatings and for assessment of coating thickness. These include laser scattering, pulse thermography, ultrasonic, beta back scattered, eddy current, magnetic Barkhausen noise, X-ray fluorescence, scanning acoustic microscopy and atomic force microscopy [3]. As the working principle and detected response to coatings

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are different for these techniques, the range of thickness, sensitivity and measurement accuracy are different. Table-1 gives the range of thickness and the measuring accuracy of these techniques [3]. For non-destructive characterization of SiC coatings on C/C composites, among different NDE techniques, eddy current (EC) technique is preferred, essentially because the substrate and coating are electrically conducting and the depth of interrogation can be controlled in this technique. This technique is useful for fast, reliable and cost effective assessment of quality of coatings, especially for ensuing uniformity and adequacy of the coating process. This technique is undoubtedly superior to the traditional weigh gain, sampling based techniques and other surface NDE techniques which will not ensure 100% examination, especially sub-surface. In this technique, depth of coverage can be changed by varying the excitation frequency. This technique is non-contact and can be used on components at elevated temperatures. This technique measures changes in impedance of coil placed over a coated surface and correlates to variations in the coating. There are several studies reported in the literature on the application of EC technique to detection and assessment of damage in C/C composites and SiC coatings [4-7]. However, studies on SiC coating thickness measurements using EC technique are very limited.

This paper discusses eddy current methodology developed for nondestructive assessment of SiC coating on C/C composite substrate. The developed methodology is first of its kind and similar studies are not reported earlier to the best of the authors' knowledge. The coating is made at VSSC, Trivandrum by polymer precursor route. The objectives for eddy current methodology are two fold: (1) identification of undercoating (SiC coating thickness less than 20 μm) and (2) quantitative assessment of SiC coating thickness. This paper discusses the details of SiC coating on C/C specimens, EC methodology developed and the results.

Table-1 : Capabilities of Various NDE Techniques for Characterisation of Coatings

NDE Technique	Range of Thickness, μm	Measuring Accuracy, %
Acoustic microscopy	> 10	≤ 2
Atmoic force microscopy	< 100	≤ 10
Beta back-scatter	0.2-800	1-10
Eddy current	$2\text{-}10^3$	0.5-10
magnetic adhesive force	$5\text{-}10^4$	≤ 10
magnetically inductive	$5\text{-}10^4$	≤ 10
Laser scattering	< 100	≤ 10
Thermography	$1\text{-}10^3$	1-5
Ultrasonic pulse-echo	> 100	≤ 10
Ultrasonic back-scatter	> 500	100
X-ray flurescence	0.1-200	1-10

Coating of SiC on C/C Composite

SiC coating over C/C is made by polymer precursor route [8]. In this method, polymeric resin based precursor slurry is prepared. The main ingredients of the slurry are PF resin, Si metal, SiC additive and ethanol as solvent. These ingredients are taken in a container and rotated in a three-roll mill for 12 h. A spray coating of precursor slurry is given over the cleaned and dried surface of the C/C substrate. Spray coating of precursor slurry was done by two to three passes so as to get a uniform coating of ~25 μm. The spray coated specimen is cured at 175^{o}C for 2 h followed by sintering at 1650^{o}C under inert gas atmosphere. Conversion of slurry indgredients into SiC takes place at this stage of sintering. This process is repeated two or more times to produce of SiC coating of desired thickness on the C/C substrate. The polymer precursor route is cost effective, easy to implement and fast as compared to Chemical Vapor Deposition (CVD) technique. Five numbers of 60x 60 x 5 mm^3 C/C specimens have been prepared and varied thickness of SiC coating has been given.

Principles of Eddy Current Technique

Eddy current technique works on the principle of electromagnetic induction [9, 10]. Fig.1 shows the schematic of basic principle of EC technique. In this technique, change in impedance of a coil excited with sinusoidal current is measured when it is placed over an electrically conducting material surface. The primary magnetic field setup by the coil induces eddy currents in the material which in turn, setup secondary magnetic fields. According to Lenz's law, the secondary magnetic fields oppose the primary fields of the coil. This results in the reduction of flux linkage in the coil and manifests as change in coil impedance which is a complex quantity with resistance (real component) and inductive reactance (imaginary component) axes. As variation in the thickness of SiC coating affects the distribution of eddy currents in the coating and substrate, there will be an associated change in the coil impedance.

Frequency of excitation is important in EC testing as it controls the depth of penetration of eddy currents depending on electrical conductivity and thickness of SiC coating and C/C substrate. Usually, lift off (distance between the specimen and the probe coil) is kept as minimum as possible and signal phase angle is rotated such that signals from desired variables fall along the vertical axis of the impedance plane. When shallow penetration of eddy currents is required, higher excitation frequencies are used following the classical skin-depth equation given by

$$\delta = \frac{1}{\sqrt{\pi f \mu \sigma}}$$

where δ is depth of penetration, f is excitation frequency, μ is magnetic permeability and σ is electrical conductivity. In the present case, both coating and substrate are non-magnetic and their electrical conductivities are in the range of 0.02-0.05% IACS (International Annealed Copper Standard).

EC Methodology for Assessment of SiC Coating Thickness

Figure 2 shows the setup used for developing the EC methodology for assessment of SiC coating on C/C composites. It consists of a PC based single frequency eddy current instrument connected to a 5 mm diameter absolute probe coil. An excitation frequency of 300 kHz has been experimentally chosen such that the phase angle between EC signals for lift-off and coating thickness is maximum and the EC signal from coating thickness is predominantly along the vertical axis of impedance plane i.e. imaginary component. For EC testing of specimens, probe is balanced on an uncoated region (reference) and change in vertical amplitude is measured upon placing the probe on a coated region.

EC measurements of C-C composites are expected to be influenced by the surface roughness of both the substrate and the coatings. In order to differentiate the contributions from these two, baseline EC measurements were taken on C-C composites prior to the SiC coating. After coating, the coating thickness is evaluated based on weight gain, assuming that the coating is uniform throughout the coated surface. A calibration graph is established between EC signal amplitude and coating thickness for the central portion of the specimens and this is used for assessment of coating thickness everywhere. Thus, the methodology consists of the following steps:

- Preparation of specimens
- Baseline EC measurements on 5x5 grid locations
- SiC coating on specimens
- Thickness evaluation using weight gain method
- EC measurements on coated specimens
- Establishing calibration graph between EC amplitude and coating thickness (weight gain)
- Assessment of coating thickness

Five specimen viz. C0, C1, C2, C3 and C4 as shown in Fig.3 have been prepared. Baseline EC amplitude measurements have been performed on 5x5 grid locations using 5mm dia. probe as shown in Fig.4. SiC coating has been given on specimens C1, C2, C3 and C4 by polymer precursor route and assuming uniformity in coating process, coating thickness has been evaluated by weight gain method using

$$Coating\ thickness = \frac{(W_2 - W_1) \times 10000}{Area \times Density},\ \mu m$$

where W_1 and W_2 are the weights of specimen before and after coating. Against the theoretical density of 3.21 g/cc, the density of SiC has been found to be 2.8 g/cc due to the porous nature (around 10%) Table-2 gives the SiC thickness evaluated by the weight gain method. As can be noted, the SiC thickness range is found to be between 11.87 and 29.54 μm and this covers both undercoated and correctly coated category of specimens. After coating, EC amplitude measurements have been repeated using the same test conditions as the baseline measurements.

Results and Discussion

Identification of Undercoating

Figure 5 shows the mean and maximum EC amplitude for uncoated specimens as dotted lines. As can be seen the EC amplitude of uncoated specimens varies between 0-1.25 Volts. This variation in amplitude is mainly due to the surface roughness noticed on the C/C composites. Thus, by ensuring good surface finish, it will be possible to reduce the scatter in EC measurements. Fig.5 also shows the variation in EC amplitude with coating thickness evaluated by weight gain method. An increase in EC amplitude with increase in coating thickness is clearly observed from Fig.5. The scatter in measurements is ±0.8 V. However, the EC amplitude after coating is higher as

compared to the mean as well as maximum baseline amplitudes of uncoated specimens. Thus, it is possible to clearly identify undercoating i.e. coating thickness less than 20 μm from the EC amplitude measurements. Typical undercoated region is highlighted as shaded region in Fig.5.

Quantitative Assessment of Coating Thickness

Figure 6 shows coating thickness (weight gain) as a function of EC amplitude for the central portion of the coated specimens. As can be seen, a linear correlation is observed between the EC amplitude and the coating thickness. The correlation coefficient and standard deviation are 0.98 and ±2.4 μm respectively. Fig.7 shows the performance of the EC methodology for the 5x5 grid region. Here, the coating thickness (eddy current) is compared with coating thickness (weight gain). As can be observed, a linear correlation exists between weight gain method and EC method and the maximum error by EC methodology is ±5 μm. The measured EC amplitude and the EC evaluated SiC coating thickness of all the specimens are also summarized in Table-3. Thus, this study reveals that quantitative assessment of thickness of SiC coating on C/C composite is possible using the EC methodology developed.

The coating thickness on specimen No.4 measured based on weight gain has been confirmed using sectional metallography and the coating thickness was measured at five different locations to estimate the coating thickness. The mean coating thickness measured by this method has been found to be 29.48 μm against 29.54 μm determined using the weight gain method.

It is possible to assess uniformity of coating in the specimens using eddy current images formatted by raster-scanning of EC probe over the coated specimens. Typical EC images for C0 (uncoated), C3 (SiC thickness, 17.30 μm) and C4 (SiC thickness, 29.54 μm) are shown in Fig.8. As can be observed, in specimen C3 the coating is uniform at the center as compared to specimen C4. The EC image of C4 specimen clearly shows non-uniformity of coating which has resulted in large scatter (refer Fig.6) and associated error in thickness assessment (refer Fig.7). EC images can also be used to assess the suitability of a coating process as the images reveal variations within a coated specimen. It is also possible to generate thickness images (using calibration graph of Fig.6) of coated specimens to assess the variations in coating thickness, enabling easy identification of thinly coated regions on the coated surfaces. Further, by using an array of EC probes, rapid screening of coated surface is possible for automated

Table-2 : SiC Coating Thickness Evaluated by Weight Gain Method

Specimen No.	Weight Before Coating (W_1), g	Weight After Coating (W_2), g	W_2-W_1, g	Coating Thickness, by Weight Gain, μm
C0 (Uncoated)	26.5896	26.5896	0	0
C1	26.3248	26.4445	0.1197	11.87
C2	26.3225	26.4869	0.1644	16.30
C3	26.5524	26.7268	0.1744	17.30
C4	26.5014	26.7992	0.2978	29.54

Table-3 : Measured Mean EC Amplitude and the EC Evaluated Coating Thickness

Specimen No.	Coating Thickness, by Weight Gain, μm	Measured Mean EC Amplitude, Volts	EC Evaluated Coating Thickness, μm
C0 (Uncoated)	0	0.52	2.1309
C1	11.87	1.44	11.9504
C2	16.30	2.20	16.7054
C3	17.30	2.40	18.9587
C4	29.54	4.05	33.4618

identification of under and over coated regions, for corrective action.

Conclusion

Eddy current methodology has been developed for non-destructive assessment of SiC coating on C/C composite specimens. Using this methodology, it is possible to readily distinguish undercoated (thickness < 20 μm) specimens. There exists a linear correlation between coating thickness evaluated by weight gain method and that of by eddy current methodology. Quantitative assessment of thickness of coating is possible using eddy current methodology with an error less than ±5 μm. Studies reveal that EC raster-scan imaging can be used to verify the suitability of coating process, to assess the uniformity of coating thickness and to identify thinly coated regions.

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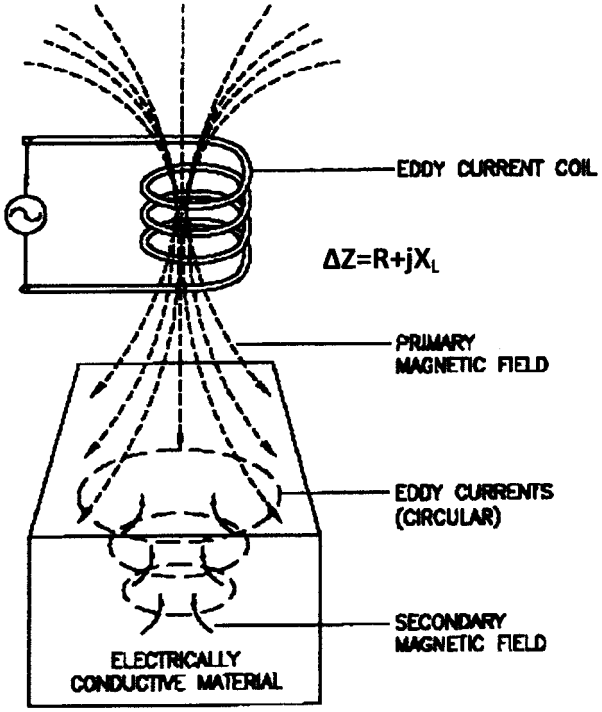

Fig.1 Principles of Eddy Current Nondestructive Testing

Fig.2 Eddy Current Test Set-up Used for Assessment of Thickness of SiC Coating on C/C Composite Specimens

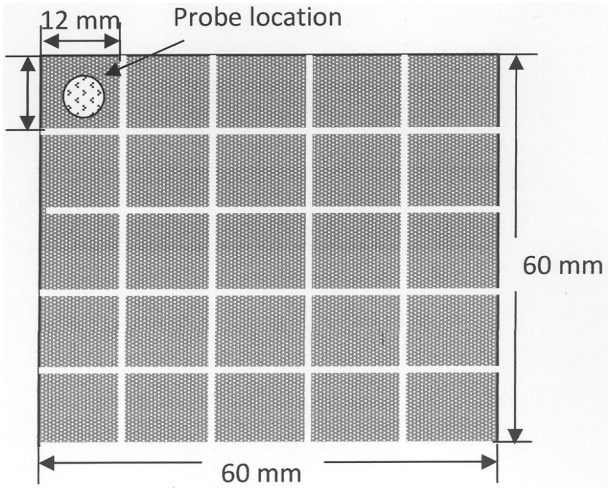

Fig.4 Schematic of 5x5 Grid Used for EC Measurements on SiC Coated C/C Composite Specimens

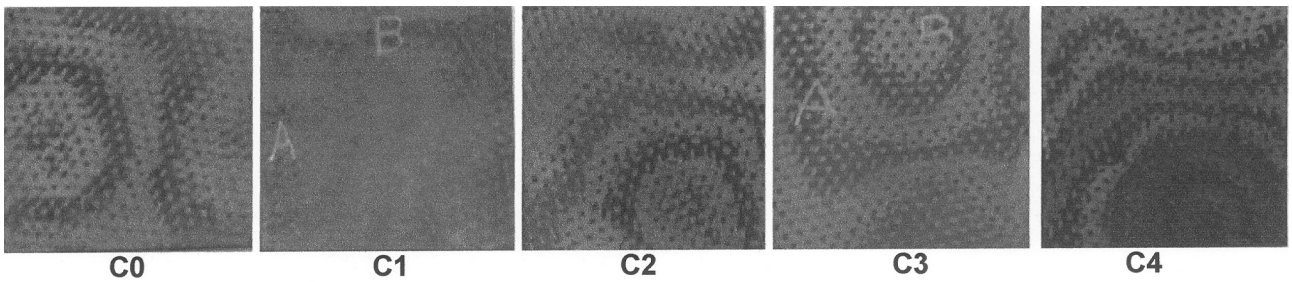

Fig.3 Five Uncoated Composite Specimens

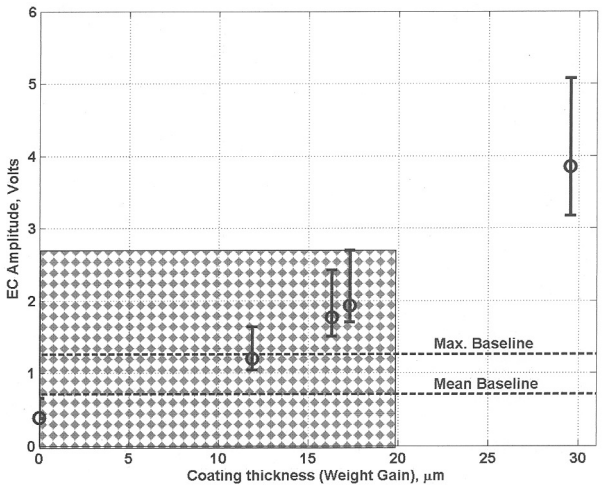

Fig.5 The Measured EC Amplitude After SiC Coating Shown along with the Mean and Maximum Baseline Amplitudes (dotted lines) on Uncoated Specimens. The Shaded Region Represent Typical EC Amplitude Values for Undercoating (i.e. Thickness Less than 20 μm)

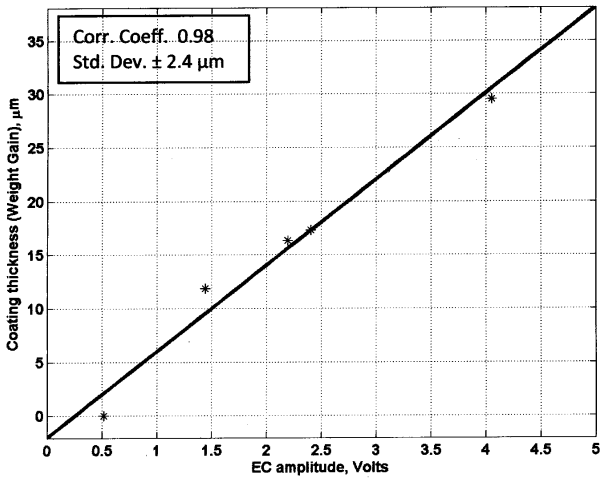

Fig.6 Calibration Graph Generated for Assessment of SiC Coating Thickness

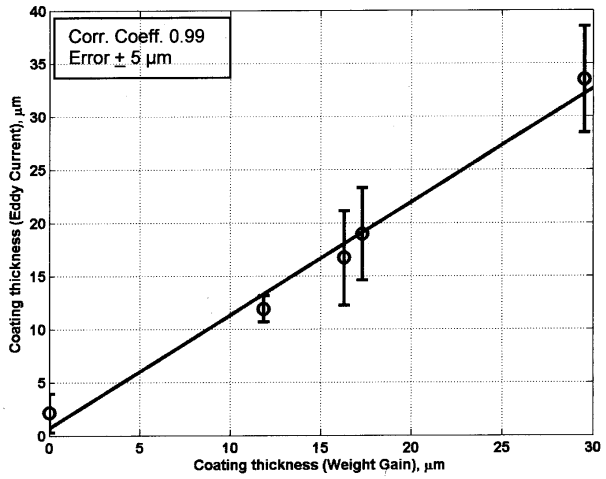

Fig.7 Performance Evaluation of EC Methodology Developed for Assessment of SiC Coating Thickness

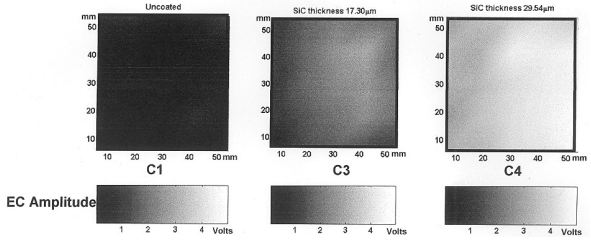

Fig.8 EC Amplitude Raster-scan Images of Specimen C1 (Uncoated), C3 (Thickness, 15.10 μm) and C4 (Thickness, 25.77 μm) Generted for Mapping Variations in SiC Coating