Experiments on the Testing Sensitivity for Microstructure Evolutions of Coatings by Electro-Mechanical Impedance Method in kHz and MHz Frequencies

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Abstract: Two kinds of plasma sprayed Cr2O3 coatings are characterized by electro-mechanical impedance (EMI) method with PZT patch in kHz and MHz frequencies. Comparing frequency shift of impedance peaks f and root mean square deviation (RMSD) of PZT electric impedance signatures, the influence of frequency on the testing sensitivity is investigated in some frequency bands among 10-238 kHz and 5.45-6.33 MHz. The results show that in the kHz frequency range, the frequency shifts f increase with testing frequency and the RMSD values first increase and then decrease. The latter may be attributed to the decreasing of peak number and the decline of impedance magnitude. In MHz frequency range, the frequency shifts f become more significant, and a prominent frequency shift of 206.1 kHz is observed near 6 MHz. The RMSD values are also much larger than that in kHz frequency range.

Keywords: Electro-mechanical impedance method; Frequency band; Testing sensitivity; Coating; Structure health monitoring

1 Introduction

Real-time monitoring microstructure evolutions of thermal barrier coatings with structural health monitoring method is significantly important for gas-turbine engines used in aircraft propulsion, power generation, and marine propulsion. Electro-mechanical impedance (EMI) method which has many advantages such as high sensitivity, anti-interference ability, real-time monitoring of damage development, has been successfully used in aerospace, civil engineering, precision machinery,

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etc. [Yang and Divsholi (2011); Shanker et al (2010); Shin and Oh (2009); Bhalla et al (2009); Park et al (2008)]. The monitoring of TBCs with EMI method has attracted the interest of researchers. Optimizing of testing frequency band, excitation voltage and other experimental parameters is the basis and an important part of this subject. The testing frequency band is generally an important factor for EMI sensitivity [Park et al (2003)]. In high frequency ranges, the acoustic signals excited by PZT patch have shorter wavelength, and are more sensitive to small damages theoretically. While the relationship between sensitivity and frequency is not monotonous, since the PZT is predominantly capacitive leading to the decreasing of electric impedance with frequency. For example, in the study of one dimensional beam structure, Park et al (2004) reported the detection of crack damage on steel beam with the size of $1000 \times 100 \times 6 \text{ mm}^3$. The results show that the frequency shifts $f$ in 2-5 MHz frequency range are much larger than that at 200-500 kHz, and the MHz frequency band has a higher testing sensitivity. On the other hand, Tseng and Naidu (2002) tested the hole damage on aluminum beam structure with the size of $1250 \times 100 \times 3 \text{ mm}^3$, and founded that the sensitivity at 400-450 kHz frequency band is lower than that at 100-150 kHz. Peairs et al (2007) tested the mass changes of aluminum beam structure with the size of $1219 \times 31.8 \times 6.4 \text{ mm}^3$, and founded that in the frequency range of 0.1-200.1 kHz, the changing trend of sensitivity is nonlinear with the increasing of frequency. Comparing with the cracks and holes on one dimensional beam structure, microstructure evolutions of on-line coating materials are more difficult to be tested with EMI method, and very little work has been reported on this subject.

In the frequency ranges of 10-238 kHz and 5.45-6.33 MHz, the electric impedance signals of plasma sprayed Cr$_2$O$_3$ coatings before and after irradiation with high intensity pulsed iron beam (HIPIB) are measured with WK 6500B precision impedance analyzer in several frequency bands, in which there are large number of dominant peaks. The relationship between testing frequency band and EMI sensitivity is quantitatively analyzed from two aspects, the frequency shift of electric impedance peaks $f$ and the root mean square deviation (RMSD) value. The results show that the MHz frequency band has a higher testing sensitivity to the microstructure evolutions of coatings.

2 Experimental specimen

The Cr$_2$O$_3$ coatings are directly air plasma sprayed on 2 pieces of cleaned and grit blasted heat-resistant steel substrates (50 mm $\times$ 30 mm $\times$ 3.5 mm) using a plasma spray equipment (MeTco-Plasma 9MB, USA). The depth of the sprayed Cr$_2$O$_3$ coatings is about 50 $\mu$m. The HIPIB irradiation of the second piece of sample is carried out in a TEMP-6 type HIPIB apparatus at the iron current density of 300A/cm$^2$, with a
Figure 1: Cross-sectional morphology of the plasma sprayed Cr$_2$O$_3$ coatings: (a) as-sprayed; (b) 1 shot by HIPIB.

Fig. 1 presents the cross-sectional SEM images of the as-sprayed and irradiated Cr$_2$O$_3$ coatings by HIPIB, respectively. The typical morphology of as-sprayed coating reveals an obvious lamellar structure with many cavities [Fig.1(a)]. After HIPIB irradiation with 1 shot, a thin discontinuous remelted layer of about 1.5 µm near the surface was observed, which was generated by the locally reformed splats [Fig.1(b)]. And an apparently compact structure in the matrix coating was formed due to an impact effect of HIPIB irradiation. The number of pores and microcracks decreases obviously. Both the density and stiffness of Cr$_2$O$_3$ coating increase after irradiation. For this study, the mechanical impedance of coating structure is changed, which may lead to some changes of PZT electric impedance spectroscopy.

3 Electric impedance measurement results

The electric impedance signals of PZT patch bonded on the upper surface of Cr$_2$O$_3$ coatings are measured with WK 6500B precision impedance analyzer, as shown in Fig.2. In order to select a suitable frequency range for acquiring the impedance signals, the experiment are investigated over a wide frequency range of 100 Hz-10MHz [Fig.3(a)]. It can be observed that the resonant peaks in the electric impedance spectroscopy of Cr$_2$O$_3$ coatings before and after irradiation are mainly distributed in 10-238 kHz [Fig.3(b)] and 6 MHz.

To thoroughly investigate the relationship between testing frequency and sensitivity, 5 frequency bands are selected for quantitative analysis in kHz frequency range, which are 10-55, 55-100, 100-145, 145-190 and 193-238 kHz, respectively,
over a wide frequency range of 100 Hz-10MHz [Fig.3(a)]. It can be observed that the resonant peaks in the electric impedance spectroscopy of Cr$_2$O$_3$ coatings before and after irradiation are mainly distributed in 10-238 kHz [Fig.3(b)] and 6 MHz.

To thoroughly investigate the relationship between testing frequency and sensitivity, 5 frequency bands are selected for quantitative analysis in kHz frequency range, which are 10-55, 55-100, 100-145, 145-190 and 193-238 kHz, respectively, as shown in Fig. 4. To ensure the comparability, the bandwidths of 45 kHz and the data points of 451 data are fixed for each selected bands. All the significant resonant peaks in the frequency range of 10-238 kHz are included in the 5 frequency bands. Similarly, in the MHz frequency range, 5.45-6.33 MHz is selected to analyze, as shown in Fig. 5.

It is observed in Figs.4 and 5 that the impedance signatures from Cr$_2$O$_3$ coatings before and after irradiation show sharp peak in various frequencies. The peaks correspond to the vibration modes of the local structure around the PZT bonding position. After irradiation by HIPIB, all the resonant peaks almost shift to lower frequency and the amplitudes of peaks and valleys are also changed.
4 Results and discussion

Firstly, the frequency shift of impedance peaks $f$ is calculated as an indicator to correlate the relationship between excitation voltage and the testing sensitivity of EMI method, quantitatively. The frequency shift $f$ is an independent and explicit parameter. It is defined as the frequency difference of corresponding peaks in the PZT electric impedance spectroscopy of $\text{Cr}_2\text{O}_3$ coatings before and after irradiation. Fig. 6 shows the frequency shift $f$ versus the peak frequency. It can be observed that in the kHz frequency range of 10-238 kHz, the whole trend of $f$ increases monotonously with the increase of testing frequency. In the MHz frequency
193-238 kHz, and \( i \) are signatures obtained from the PZT bonded to the upper surface of sample 1, 2, 3

is the number of data points in the sampled impedance signatures, (continuous function).

### 4. Results and discussion

Firstly, the frequency shift of impedance peaks \( \Delta f \) is calculated as an indicator to correlate the relationship between excitation voltage and the testing sensitivity of EMI method, where

\[
\Delta f = \sum_{i=1}^{N} i \cdot \Delta f_i
\]

and correlation coefficient (CC), etc. In particular, the RMSD value can reflect the changes of characteristic parameters in statistical techniques are further employed as recognition indicators to quantitatively analyze the changes of electric impedance spectroscopy.

The RMSD is mathematically defined as

\[
RMSD = \sqrt{\frac{1}{N} \sum_{i=1}^{N} (x_i - \bar{x})^2}
\]

mean square deviation (RMSD), mean absolute percentage deviation (MAPD), covariance (Cov) etc. The changes of other data adjacent peaks and the amplitude in the electric impedance spectroscopy are not included. Thus, the frequency difference of corresponding peaks in the PZT electric impedance spectroscopy of Cr\(_2\)O\(_3\) coatings before and after irradiation. Fig. 6 shows the frequency shift \( \Delta f \) versus peak frequency.

The microstructure changes of Cr\(_2\)O\(_3\) coating after irradiation can be intuitively identified by \( f \) values. However, only the shifts of resonant peaks along the direction of frequency are considered. The changes of other data adjacent peaks and the amplitude in the electric impedance spectroscopy are not included. Thus, the characteristic parameters in statistical techniques are further employed as recognition indicators to quantitatively analyze the changes of electric impedance spectroscopy. At present, the indicators commonly used in EMI method consist of root

![Figure 5: The electric impedance signals in MHz frequency range](image)

range of 5.45-6.33 MHz, with the increase of peak frequency, the \( f \) decreases from 206.1 kHz at 5.88 MHz to 138.4 kHz at 6.05 MHz. It’s also noticed that the \( f \) values in MHz frequency range are much larger than that at kHz frequency range.

![Figure 6: The frequency shift \( \Delta f \) versus peak frequency](image)
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mean square deviation (RMSD), mean absolute percentage deviation (MAPD), covariance (Cov) and correlation coefficient (CC), etc. In particular, the RMSD value can reflect the changes of electrical impedance spectroscopy in both frequency and amplitude directions, and it is very sensitive to the property changes of Cr$_2$O$_3$ coatings. The RMSD is mathematically defined as [8]

$$\text{RMSD} (%) = \sum_{i=1}^{n} \sqrt{\frac{(y_i - x_i)^2}{x_i^2}} \times 100$$ (1)

where $n$ is the number of data points in the sampled impedance signatures, $x_i$ and $y_i$ ($i = 1, 2, 3 \cdots N$) are signatures obtained from the PZT bonded to the upper surface of Cr$_2$O$_3$ coatings before and after irradiation, respectively.

Fig. 7 presents the RMSD values of the PZT electric impedance signatures in different testing frequency ranges. It can be observed that the RMSD values can give obvious indications on the microstructure changes of Cr$_2$O$_3$ coatings after irradiation. In the kHz frequency range of 10-238 kHz, the RMSD values first increase and then decrease with the increase of testing frequency. They first increase from 7.0 at 10-55 kHz to 11.6 at 100-145 kHz, and then decrease gradually to 4.2 at 193-238 kHz. The reason may be attributed to the comprehensive interactions of frequency shift $\Delta f$, peak number and impedance amplitude. In the MHz frequency range of 5.45-6.33 MHz, the RMSD values decrease from 69.29 at 5.45-5.89 MHz to 64.34 at 5.45-6.33 MHz. In MHz frequency range, the calculated RMSD values are much larger than that at kHz frequency range. It seems that higher testing sensitivity can be obtained in the MHz frequency range for monitoring evolutions of coating structures.
The RMSD value is a more comprehensive parameter since it is concerned with the frequency shift $f$, the differences of impedance spectroscopy, the amplitude of impedance and other factors. Because the PZT is predominantly capacitive, the electric impedance amplitude of PZT decreases with testing frequency. In MHz frequency range, the calculated RMSD value may be smaller than that at kHz frequency range. The frequency shift $f$ and RMSD value quantify the change of PZT electric impedance spectroscopy from different aspects. Combination of the above analyses, the RMSD value and frequency shift $f$ are proposed to be used in kHz and MHz frequency ranges, respectively, when EMI method is employed to evaluate evolutions of coating structures.

5 Conclusions

This paper investigates the microstructure changes of plasma sprayed Cr$_2$O$_3$ coatings before and after irradiation with HIPIB by EMI method. The relationship between testing frequency and sensitivity of EMI method is quantitatively analyzed on 7 frequency bands among 10-238 kHz and 5.45-6.33 MHz. The results show that the microstructure evolutions of Cr$_2$O$_3$ coatings after irradiation can be effectively tested by EMI method. In MHz frequency range, the frequency shift $f$ and RMSD value are obviously larger than that at kHz frequency range. In the actual testing process, the sensitivity of EMI method is influenced by many factors. The testing frequency band and other influence factors such as PZT excitation voltage, the distance between damage and sensor etc. should be considered carefully.

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