Contents lists available at ScienceDirect

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Damage mapping with a degrading elastic modulus using piezospectroscopic coatings



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ARTICLE INFO

Article history: Received 24 March 2015 Received in revised form 12 July 2015 Accepted 13 July 2015 Available online 26 July 2015

Keywords: Piezospectroscopy Mechanical properties Multiscale mechanics Damage mechanics Non-destructive evaluation

1. Introduction

Detecting damage with NDE is an important field for the diagnosis of structural health with applications ranging from aerospace [1] to civil [2] structures. Conventional NDE techniques are based on the identification of the size and location of defects. Techniques such as ultrasound [3], thermography [4], shearography [5], and others [6] are technologies that each have their own set of advantages in this regard.

An alternative way of monitoring damage is by quantifying the reduced mechanical properties. The presence of micro-voids or cracks creates a softening effect which decreases the elastic modulus of the continuum [7]. When compared to a variety of damage measures, monitoring the degrading elastic modulus is a promising technique [8]. The degraded elastic modulus can be captured experimentally with the slope of an unloading curve or predicted with a damage model. The major weakness is that it lacks the capacity to detect local damage because a measure of the elastic modulus is conventionally a nominal or macroscopic measure.

In laboratory settings, determining nominal mechanical properties is straightforward with a load cell, strain gage, and standard test specimens. However, evaluating local mechanical properties of a structure would require a combination of stress and strain mapping. Strain mapping is available with a few techniques including electronic speckle pattern interferometry [9], Moiré interferometry [10], and

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ABSTRACT

The development of piezospectroscopic (PS) composites has enabled the creation of a non-destructive evaluation (NDE) technique which integrates piezospectroscopy, digital image correlation (DIC) and analytical multi-scale mechanics to map the elastic modulus of a coated material. The measured elastic modulus was represented as a normal distribution with a mean value (32.2 GPa) which is within 8% of the conventionally recorded modulus (35 GPa). Damage mechanics are applied to map elastic degradation *in situ* mechanical loading with an average uncertainty (\sim 10 GPa) that was sufficient in observing subsurface, progressive damage patterns which are unique to the coated material.

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digital image correlation (DIC) [11]. Stress mapping with the same flexibilities as the previously mentioned strain mapping techniques has not been available, until now, with piezospectroscopy. This work shows that piezospectroscopy, recorded over a field of view, can couple with strain maps to locally distinguish elastic moduli, thereby removing the weakness this damage measure is nominal.

Other approaches to estimate local mechanical properties are available such as atomic force microscopy [12,13] and ultrasonic [14] methods. These techniques rely on other physical principles to determine the elastic modulus rather than conventional stress/ strain relationships and are currently active fields of research [15,3]. Among these, however, piezospectroscopy stands out as the one technique suitable enough for integration with full-scale mechanical load frames for standard coupon testing [16,17].

Piezospectroscopy with Cr doped Al₂O₃ has been most widely used as a pressure sensor in diamond anvil cells [18], and as an NDE technique for monitoring the health of thermal barrier coatings [19]. The applications of piezospectroscopy have recently been expanded by the fabrication of alumina-epoxy nanocomposites with tailorable mechanical and PS properties [20]. This produced a material which can be applied as a compliant coating to structures for stress sensing [21–23]. In the process of holistically understanding the multi-scale mechanics of these new materials, the solution of an effective elastic modulus of the nanocomposite with the experimental PS response was discovered [24]. In this work, multi-scale mechanics are extended to a nanocomposite coating, to solve for an effective elastic modulus of the composite substrate *in situ* mechanical loading.

2. Experiment

The PS nanocomposite coating investigated in this work was manufactured by Elantas PDG, Inc. by mixing 150 nm, Cr doped α -Al₂O₃ nanoparticles (Inframat Corp.) with 99.8% purity in epoxy to achieve a 20% volume fraction of particles. The coating was applied to a composite substrate consisting of laminated IM7-8552 unidirectional tape (57.7% fiber volume) manufactured with a [45/-45/0/45/-45/90/45/-45/45/-45]_s layup resulting in transversely isotropic properties ($E_1 = E_2 = 35$ GPa) [25]. The coating has a very small thickness (300 µm) and low modulus (< 1 GPa) which ensures that it does not mechanically reinforce the composite substrate. The coated sample was machined and tested in accordance with composite open hole tension (OHT) ASTM standards [26]. The length, width, thickness and hole size of the coupon were 12, 1.5, 0.15, and 0.25 in respectively.

The sample was loaded at a rate of 0.05 in/min (0.127 cm/min) and held using displacement control using a hydraulic load frame at 10 load points throughout the experiment as marked in Fig. 1. This avoided creep during the higher loads, but prompted stress relaxation which was acceptable since the goal was to observe damage progression. During each hold, PS and DIC measurements were collected on the front and the back side of the substrate, respectively. Both measurements can be directly compared since the substrate has a symmetric layup, shown in Fig. 1.

PS data was collected with a prototype portable spectrometer system designed to be easily transported with a quick setup time [17]. Excitation was achieved using a low power, 1.5 mW laser of 532 nm wavelength in a back scattering configuration with a long working distance objective (28 mm). The photoluminescence (PL) spectra were collected on a 60×60 grid during a snake scan, implemented using a synchronized translation stage, with the accumulation time for spectra at 100 ms. The total measurement area was 25.4 mm squared, corresponding to a spatial resolution of 0.4 mm in both directions of the measurement plane. With these settings each map is collected within 8 min.

The DIC measurements were collected at the beginning of every hold and were relatively instantaneous. The speckle pattern, which was spray painted, had an average dot size of roughly



Fig. 1. Both PS and DIC tripod systems are measuring the stress and strain field respectively on opposing sides of the composite coupon in a mechanical load frame. The steps within the load history graph are displacement control holds, which were necessary to prevent creep in the sample at the high failure loads during mapping the PS response. This resulted in a softening of the material during and is observed as a reduction in force during the load steps.

0.2 mm. The DIC images were post processed to have a spatial resolution equal to that of the PS maps (0.4 mm). Furthermore, a post-processing algorithm was created to spatially match up the DIC and PS mapping data. In brevity, the algorithm interpolated data on a new coordinate system using the open hole as a reference. The accuracy of the pixel match up is worst in regions closest to the open hole (\leq 0.4 mm) but is negligible at a few pixels distance away (1–2) [27].

3. Multiscale mechanics to model the PS response

Spatial measurements of substrate strain and PS shift were obtained simultaneously for the first time in an novel experiment [23]. The PS relationships originate from the distortion of the Ligand field around the Cr^{3+} substitutional impurity within the Al_2O_3 lattice. As the lattice is deformed under mechanical load, the energy levels of the Ligand field change, resulting in a frequency shift of the photo-stimulated luminescence [28]. Usually, the PS relationships are dependent on crystallographic orientation, but for a polycrystalline a material whose grain size is an order of magnitude smaller than the probed volume (i.e. laser dot size) an averaging effect takes place and simplifies the PS relationship to be a function of the first stress or strain invariant [29]. In this study, the PS shift is considered as the mean shift of the R1 and R2 spectral lines, which has been associated to the first stress invariant [24,30].

Here, the PS response is taken as the substrate's DIC biaxial strain $(e_1^s + e_2^s)$ vs. PS shift $(\Delta \nu)$. When evaluating this relationship, a variety of mechanics have to be considered as illustrated in Fig. 2. For linear elastic behavior, a PS response is described by the composite's PS coefficient (Π_c) in $\Delta \nu = \Pi_c(e_1^s + e_2^s)$

This PS coefficient is experimentally measured for every pixel when the PS shift and DIC biaxial strain maps are combined. This experimental PS coefficient represents a combination of several mechanics that describe the process when load is transferred from the substrate to the coating to the particle. The series of ratios that describe this PS coefficient are shown in the following equation:

$$\Pi_c = \frac{\Delta\nu}{\epsilon_{ii}^p} \frac{\epsilon_{ii}^p}{\epsilon_{ii}^c} \frac{\epsilon_{ii}^c}{\epsilon_1^s + \epsilon_2^s} \tag{1}$$

The first ratio encompasses the relationship between the mean R-line shift with the particle's first strain invariant (e_{ii}^p) as shown in Eq. (2). This is a variation of the equation that commonly uses the first stress invariant (σ_{ii}) with the PS shift by the trace of PS tensor ($\Pi_{ii} = 7.6 \text{ cm}^{-1}/\text{GPa}$) [31]. The first stress and strain invariant is interchangeable with the bulk modulus of the particle (K^p) using the relation $3K\epsilon_{ii} = \sigma_{ii}$

$$\frac{\Delta\nu}{\epsilon_{ii}^p} = \Pi_{ii} K^p \tag{2}$$

The second ratio describes the relationship between the first strain invariant of the particle and coating. This equation was derived using Eshelby's inclusion mechanics [24] which assumed a spherical particle, and both phases are isotropic. The ratio is a function of the properties for the coating (E^c, ν^c) and particle (E^p, ν^p) shown in the following equation:

$$\epsilon_{ii}^{p} = \frac{-3E^{c}(2\nu^{p}-1)(\nu^{c}-1)}{(2\nu^{c}-1)(2E^{c}+E^{p}-4E^{c}\nu^{p}+E^{p}\nu^{c})}$$
(3)

The last ratio describes the relationship between the first invariant of the coating with the biaxial strain of the substrate. A number of steps are associated with obtaining this ratio which are outlined elsewhere [27] and are valid for a thin compliant coating. Briefly, the expression is obtained by equating interface strains $(\epsilon_1^s = \epsilon_1^c, \epsilon_2^s = \epsilon_2^c)$ between the isotropic coating and transversely isotropic substrate under plane stress $(\sigma_3^s = \sigma_3^c = 0)$. In addition,

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