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Multiscale mechanics to determine nanocomposite elastic properties with piezospectroscopy

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Abstract

The piezospectroscopic (PS) properties of chromium-doped alumina allow for embedded inclusion mechanics to be revisited with unique experimental setups that probe the particles' state of stress when the composite is under applied load. These experimental investigations of particle mechanics will be compared to the Eshelby theory and a derivative theory. This work discovers that simple nanoparticle load transfer theories are adequate for predicting PS properties in the low to intermediate volume fraction range ($\leqslant$20%). By applying the multiscale mechanics to a PS response, the inverse problem was demonstrated to reveal the elastic modulus of the composite. The implications for this technique are damage monitoring through observation of reduced mechanical properties in addition to a method to assist with engineering nanomaterials.

Keywords: Particulate mechanics; Piezospectroscopy; Alumina; Load transfer

1. Introduction

Piezospectroscopy is a technique to measure stress using the spectral emissions of a material. This has been commonly applied to ruby as pressure sensors for diamond anvil cells [1], and indicators of residual stress build up in the alumina oxide layer of thermal barrier coatings [2]. Recent efforts that introduced photoluminescent alumina nanoparticles as a constituent into a matrix material have shown them to enhance the stress sensitivity of the resulting nanocomposite [3]. Such non-invasive measurements of microscale stresses with multiscale spatial resolution assist in the engineering of advanced nanocomposites. Although such PS measurements are only possible when there are embedded inclusions which exhibit intrinsic PS properties, the insights gained from measurements using these materials can be used to obtain a greater understanding of particulate composites mechanics. Similar works studied the strength of alumina and sapphire fiber bundles by quantifying the volume of damaged to undamaged fibers using piezospectroscopy [4]. Here, a unique combination of piezospectroscopic (PS) and particulate mechanics is presented that enables the determination of nanocomposite elastic properties.

Particulate nanocomposites have shown great promise for future applications because the intrinsic size effect produces enhanced mechanical properties [5]. One of the many types of nano- or microinclusions that have been used in developing nanocomposites is ceramic particles such as alumina, which has high hardness and excellent retention of strength at high temperatures [6]. The trace amounts of Cr^{3+} impurities in alumina afford excellent PS properties [7]. In general, when the particles are added to a matrix they increase the fracture toughness, wear resistance and elastic modulus with respect to unfilled matrix. However, there are many microstructural factors

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to consider when evaluating the performance. These factors include particle size [8–11], particle shape [12,13], surface treatment [11,12,14,15] and dispersion [9]. These factors are not limited just to alumina composites but also apply to particulate composites in general.

The application of piezospectroscopy could supply an enhanced understanding of the micromechanics of particulate composites. The ability to experimentally probe the stresses and strains of the particle phases has been limited to less accessible high energy X-ray [16] and neutron [17] diffraction techniques. Therefore, much of the design of these composites depend on the bulk strain response of the composite from strain gage or computational simulations of the nanocomposite. Previous results have shown that the complex micromechanics of alumina plasma-spray coatings under in situ mechanical loading was revealed with piezospectroscopy in great detail when compared to a conventional bulk strain measurement [18]. In addition, piezospectroscopy can point out micron-level structural defects in such materials with its capability of high spatial resolution [19].

This investigation begins with an effort to analytically replicate the experimental PS coefficients of alumina–epoxy nanocomposites from a combination of previous works [3,20] where increasing the volume fraction of alumina resulted in enhanced sensitivity of the PS properties. The state of stress obtained by the PS measurements will be discussed along with the approach of comparing it with theoretical calculations. A generalized method of calculating stresses of the embedded inclusions will be presented which was inspired by the works of Kim [21]. Then the application of multiscale mechanics will be shown to convert nanoparticle stresses from piezospectroscopy into elastic property calculations of the bulk composite.

2. Experimental background

When under compressive load, the spectral emissions of the Cr^{3+} doped polycrystalline alumina have been shown to exhibit negative wavenumber shifts [7,22]. This trend is also observed when the alumina is distributed as nanoparticles within an epoxy matrix, as illustrated in Fig. 1a and b. The rate of this wavenumber shift is much higher for the nanocomposites, indicating increased PS sensitivity. This was quantified directly by the experimental works of Stevenson [3], and then later it was substantiated by testing additional volume fractions [20]. The PS sensitivity of the composite can be quantified by the PS coefficient (Π_c) and defined as the rate of PS shift per applied uniaxial external stress to the composite, as illustrated in Fig. 1c.

The PS coefficient for bulk polycrystalline alumina under uniaxial compression has been recorded to be 2.54 cm^{-1}/GPa [7]. Recent work has established the PS coefficients for alumina–epoxy nanocomposites for different volume fractions of alumina, and these are shown in Table 1 [3,20]. The experiments were conducted with alumina nanoparticles (150 nm) embedded in an EPON 826 epoxy matrix. The samples were manufactured as parallelepipeds, using ASTM standard D695 [23].

The wavenumber shifts of the PS response are usually very small ($\leq 1 cm^{-1}$) for conventional mechanical tests. However, pseudo-Voigt curve-fitting algorithms can resolve even smaller changes in peak position with a standard deviation less than 0.01 cm^{-1} [3,24]. A systematic uncertainty with a stress measurement of 200 MPa gives an error of 0.05 cm^{-1}/GPa for the composite PS coefficients in Table 1. Details on the experimental setup used to obtain results in Table 1 are found elsewhere [25].

Table 1
Experimental PS coefficients (Π_c) for R1 and R2 peaks [3,20].

Volume fraction %	R1 cm^{-1}/GPa	R2 cm^{-1}/GPa
5	−3.16	−2.60
20	−3.34	−3.19
25	−3.65	−3.42
34	−4.10	−3.88
38	−5.63	−5.08

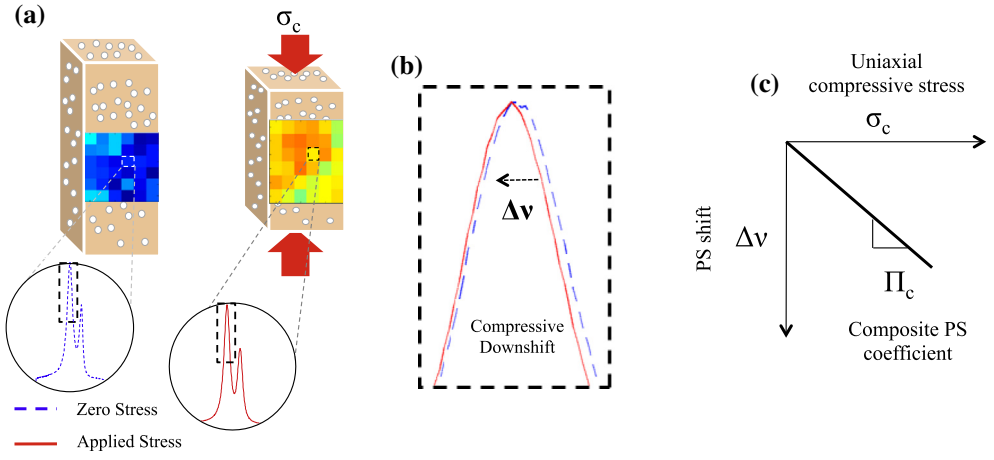

Fig. 1. (a) A schematic representing hyperspectral imaging of a PS nanocomposite under compressive loading, (b) the corresponding peak position downshifts with compressive loading and (c) the composite PS coefficient (Π_c) relating a peak position shift with applied stress.