The future of aerospace structures is highly dependent on the advancement of reliable and high performance materials such as composite materials, ceramics, and metals. The application of photo-luminescent $\alpha$-alumina nanoparticles in coatings allows for various structural materials to be evaluated nondestructively for stress and damage. The capabilities of these coatings and their ability to monitor the underlying substrate were measured by using photoluminescence piezospectroscopy (PLPS). Different volume fractions of $\alpha$-alumina nanoparticles in the piezospectroscopic (PS) coatings were studied for determining the sensitivity of the coatings on 2024 aluminum tensile substrates with subsurface notches. Here, it was found that the 10 vol% PS coating better captured the location and the extent of the subsurface notch than the 1 vol% PS coating. Application of the PS coating can provide potentially invaluable information to monitor structural integrity of aerospace structures and allows for preventive measures to be taken before failure.

I. Nomenclature

\[ \Delta \nu = \text{average frequency shift} \]
\[ \Delta \nu = \text{frequency shift} \]
\[ \Pi_{ij} = \text{PS coefficients (or PS tensor)} \]
\[ \sigma_{ij} = \text{stress tensor} \]
\[ \Pi_{ii} = \text{trace of the PS tensor} \]
\[ \sigma_{jj} = \text{trace of the stress tensor} \]

II. Introduction

Nondestructive evaluation (NDE) methods are utilized in the aerospace industry for aircraft maintenance, quality assurance, and investigation of the properties of newly developed materials. Metals are commonly used in aerospace structures, but they tend to age over time and become susceptible to major flaws, such as creep and corrosion \[1\]. These flaws can lead to structural failure. Thus, there is a need for the advancement of reliable and high performance NDE methods. Stress sensing coatings can provide a viable method for NDE of structures. Figure 1 shows a schematic of a photo-luminescent coating for stress sensing of an aerospace structure.

Using photo-luminescent $\alpha$-alumina nanoparticles in coating material allows for stress and damage sensing on aerospace structures. These $\alpha$-alumina nanoparticles consist of chromium ions. Thus, laser excitation of these nanoparticles reveals characteristic spectral emissions in the form of R-lines, which consist of R1 and R2 peaks. The stress sensing method, known as piezospectroscopy (PS), involves the concept of photoluminescence (PL) spectroscopy, which is a non-equilibrium emission of radiation due to photon excitation \[2\]. When the $\alpha$-alumina nanoparticles within the coating material undergo stress along with the underlying substrate, PS correlates the changes in stress with shifts of the R1 and R2 peaks from the unstressed state. This phenomenon, called the PS effect, is demonstrated in Figure 2.

The change in stress can be quantified by the frequency shift based on the PS coefficients, as shown in Equation \[1\] \[3\]:

\[ \Delta \nu = \Pi_{ij} \sigma_{ij} \]
Alumina-epoxy coating

Laser excitation

Emission from alumina

Stress and damage on the surface and subsurface

Fig. 1  A schematic of photo-luminescent coating for stress sensing

![Fig. 1 A schematic of photo-luminescent coating for stress sensing](image)

Wavenumber(cm\(^{-1}\))

Intensity (a.u.)

Stressed state

Unstressed state

R1 Δν

R2 Δν

Fig. 2  Stress state characterized by shifts of the R1 and R2 peaks from the unstressed state

![Fig. 2 Stress state characterized by shifts of the R1 and R2 peaks from the unstressed state](image)

where Δν is the frequency shift, Π\(_{ij}\) represents the PS coefficients (or PS tensor), and σ\(_{ij}\) is the stress tensor. Equation 1 can be reduced to Equation 2 which assumes that the stress state the α-alumina nanoparticles is uniaxial. In this equation, \(\overline{Δν}\) is the average frequency shift, Π\(_{ii}\) is the trace of the PS tensor, and σ\(_{jj}\) is the trace of the stress tensor.

\[
\overline{Δν} = \frac{1}{3} Π_{ii}σ_{jj}
\]

(2)

PS was used in the past in diamond-anvil pressure cells to monitor pressure based on the R-line fluorescence of rubies [4], for computing residual stresses in ceramic oxides [3], and monitoring the stresses in the thermally grown oxide (TGO) layer inside thermal barrier coatings (TBCs) [5]. The stresses in the TGO layer decrease during the lifetime of the TBC and could be used as a technique to estimate the remaining life and damage of turbine engines. However, the applicability of PS materials can be further improved to perform more precise measurements and extended to new applications.

Previous works from our group have shown that the sensitivity of PS can be tailored [6, 7]. It was found that distributing the α-alumina in nanoparticulate form within a matrix forms a nanocomposite material that is sensitive to changes in stress. It is known that stress on the particles in a composite depends on the particle size, shape, dispersion and volume fraction. Varying the parameters of the reinforcing particles allows for tunability of the stress sensing coating. Increasing the particle volume fraction up to a certain level increases the sensitivity of the coating to applied loads [7]. However, it was reported that 38 vol% of α-alumina is the maximum limit to be added to a polymer matrix to improve its mechanical properties [8]. Frehofer et al. [9] demonstrated the capability of PS coatings in monitoring stress on a composite laminate substrate when it is subjected to load and detecting damage before failure occurs. An open-hole tension (OHT) carbon fiber reinforced polymer (CFRP) substrate with a PS coating consisting of 20 vol% of α-alumina nanoparticles in epoxy matrix was manufactured and tested. The coating detected internal ply damage at 76...
% failure load, which was well before it surfaced at 92 % failure load as measured by digital image correlation (DIC) [9].

This study aims to investigate the capability of the PS coating to sense stress and damage concentrations when applied onto metallic substrates with subsurface notches. Here, spectral data from two samples with PS coatings, consisting of 1 vol% and 10 vol% of α-alumina, on 2024 aluminum tensile substrates are compared for their stress and damage sensing capability.

III. Experimental Setup

Two 2024 aluminum tensile substrates were coated with PS coatings consisting of 1 vol% and 10 vol% α-alumina nanoparticles, with an average particle size of 150 nm, in epoxy matrix. The substrates were machined and prepared in accordance with ASTM E8-04 [10]. In order to capture and monitor the development of the subsurface damage, a 0.25" × 0.16" × 0.08" notch was introduced in both substrates on the face opposite of where the coatings had been applied.

Figure 3 shows the experimental setup for measuring stress on an aluminum sample using the PS method. A servohydraulic MTS universal testing machine was used to apply uniaxial tensile load to both samples until failure load was reached, using a crosshead displacement rate of 1 mm/min. PL scans of the coatings were conducted at every 4 kN increment while the load was held constant and images were taken between each increment.

The PL scan was performed using a portable piezospectroscopy system developed in-house [11]. This system consists of a Princeton Instrument Pixis 100 charge coupled device (CCD), Acton SP2150 spectrometer, a 532 nm continuous laser source, and an InPhotonics Inc. RPB Raman probe. An XYZ stage was used alongside the probe and CCD to collect measurements over a designated area. A laser power output of 30.1 mW and 10.6 mW for the 1 vol% and 10 vol% samples, respectively, were used to excite the α-alumina nanoparticles in the PS coatings of both samples during loading. The laser power was chosen based on the spectral emission of the PS coatings, which is dependent on the amount of α-alumina nanoparticles present within those coatings. The PL scans were taken at each hold with a map size of 18 mm × 30 mm with spatial resolution of 200 µm.
IV. Results and Discussion

Figure 4 shows the tensile response of 2024 aluminum tensile specimens with 1 vol% and 10 vol% PS coatings. Similar stiffness was measured from both tests, which indicated that the coating had no affect on how the aluminum specimens responded to the applied tensile load. The results presented here are focused on the photo-luminescent measurements from the coatings during the tensile tests. The signal response, in the form of R-lines, from each sample are shown in Figure 5. The R-lines correspond to the PL scan of one point on each sample at zero applied load. The R1 peak positions from both PS coatings are at 14,402 $cm^{-1}$ at zero load. The peak positions are used as a reference to determine the peak shifts in response to the applied tensile load on the samples. The intensity count was found to be affected by the volume fraction of $\alpha$-alumina nanoparticles in the coating. Specifically, the intensity from the coating with 10 vol% $\alpha$-alumina nanoparticles had almost double the intensity from the coating with 1 vol% of $\alpha$-alumina nanoparticles.

![Fig. 4 Load-displacement curves for each sample](image)

The peak shift maps obtained from the PL scans on the coated surface, opposite of the notch on each sample, are shown in Figure 6 for each load step until failure. These maps were plotted from 90 $\times$ 150 point-wise scans to cover an area of 18 $\times$ 30 $mm^2$ at a spatial resolution of 200 $\mu$m. They indicate the full field stress state of the coating (and thereby the substrate) for each area that was mapped. A higher peak shift indicated larger tensile stress. By comparing the maps as they changed with load, the peak shift maps of both coatings showed signs of gradually increasing tensile stress. It was observed that the stress is uniform on the surface up to 8 kN of load. However, the peak shift map from the 10 vol% PS coating better captured the stress concentration associated with the subsurface notch compared to the 1 vol% PS coating, which can be observed in the peak shift maps at 12 kN load. Down shifts were shown in the peak shift maps starting from the 8-kN load until post failure due to the stress concentration arising from the subsurface notch. This observation indicated that PS is capable of detecting the location of subsurface damage initiation. Furthermore, the size of the stress concentration area due to the subsurface damage showed more prominently on the peak shift maps from the 10 vol% PS coating than the maps from the 1 vol% PS coating.

V. Conclusions and Future Work

PS coatings with 1 vol% and 10 vol% $\alpha$-alumina nanoparticles in epoxy matrix were applied to 2024 aluminum tensile substrates for stress sensing and damage detection. PL scans were taken during the tensile tests using a custom-made portable piezospectroscopy system. The coatings were capable of determining full-field stress, including the stress concentration, due to the subsurface notch on the aluminum substrates. Also, the PS coating with higher volume fraction (10 vol%) of $\alpha$-alumina nanoparticles showed higher stress sensitivity than the PS coating with lower volume fraction (1 vol%) of $\alpha$-alumina nanoparticles. To conclude, the PS coating can provide high spatial resolution images of stress fields and damaged zones. For future work, full field deformation measurements obtained via DIC and finite element analysis will be used to validate the presented stress sensing method for detecting the stress concentration and damage initiation site due to the subsurface notch.
Fig. 5  R-lines obtained from each sample at zero applied load

Fig. 6  R1 peak shift maps with increasing tensile load.

VI. Acknowledgements

This material is based upon work supported by the National Science Foundation (NSF) under Grant No. 1701983.

References


