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# Fatigue response of hybrid magnesium/APC-2 nanocomposite laminates at elevated temperature



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### ABSTRACT

AS-4/PEEK APC-2 nanocomposite laminates and Mg/APC-2 hybrid nanocomposite laminates were fabricated. The mechanical properties and fatigue response of laminates were obtained due to tensile and cyclic tests at room and elevated temperatures. The surface treatment of chemical etching on Mg sheets resulted in strong bonding with APC-2 laminates. The nanoparticles SiO<sub>2</sub> of optimal weight were uniformly spread on the faces of APC-2. From the tensile test the mechanical properties, such as ultimate strength and stiffness, of cross-ply APC-2 nanocomposite laminates were found higher than those of quasi-isotropic nanocomposite laminates. The ultimate strength of hybrid nanocomposite laminates was predicted satisfactorily well with experimental data by using the rule of mixtures.

As for cyclic tests we received the data of applied stress vs. cycles and plotted the S-N curves. Also, the failure mechanisms were observed. It was found APC-2 quasi-isotropic nanocomposite laminates possessed better fatigue resistance than that of the cross-ply nanocomposite laminates. Through the regression of fatigue data we proposed the semi-empirical model for APC-2 nanocomposite laminates and the theoretical model for hybrid nanocomposite laminates. Both models predicted the results of durability/ life in good agreement with experimental data. The theoretical model for hybrid nanocomposite laminates model for hybrid nanocomposite laminates.

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#### 1. Introduction

Recently, aerospace researchers have endeavored to manufacture the ongoing demands of both military and commercial aircrafts to fly faster, longer, and more safe and comfortable than ever before. Such an advancement is contingent upon structural materials ability for operating at long lives and higher temperatures due to fatigue resistance and damage tolerance. Various hybrid composite laminates have been developed and designed for specific purposes since 1980 [1]. Commonly, hybrid composite laminates, consisted of varied layers of metal alloy sheets alternately bonded with layers of polymeric matrix composites, possess high performance and fatigue-resistance that retaining the high strength without incurring a significant weight increase. Kaufman [2] proved that a laminate of adhesively bonded aluminum plies had nearly twice the fracture toughness of a single aluminum plate. Johnson et al. [3] demonstrated that a laminated aluminum structure greatly improved the fatigue crack growth resistance, and Johnson [4] also found the laminated titanium increased

\* Corresponding author. E-mail address: jmhr@mail.nsysu.edu.tw (M.-H.R. Jen). hybrid composite laminate called ARALL. Wu et al. [6] conducted the tensile tests and finite element analysis of two types of ARALLs. After the initial success of ARALL, Young et al. [7] developed GLARE to use R and S2 glass fibers instead of aramid fibers. These materials were useful in many designs. In addition, the researchers investigated the hybrid laminates with different constituents. Kawai et al. [8] studied the effect of stress ratio on the off-axis fatigue behavior of the unidirectional fiber-metal hybrid GFRP/Al laminate at room temperature. Rhymer et al. [9] conducted tensile and fatigue tests in single edge notched hybrid titanium composite laminates to measure the crack growth rate and to compare the data with the three-dimensional finites element model predictions. Pärnänen et al. [10] investigated the impact resistance of AZ31B-H24 magnesium composite laminates and compared the properties with those of GLARE.

damage tolerance capability. Verbruggen [5] developed the first

As for the fillers in matrix, Rajkovic et al. [11] discussed the effect of  $Al_2O_3$  particle size and fine grain structure on reinforcing the copper matrix. Wang et al. [12] determined the effects of minor Zr and Sc elements on the microstructures and mechanical properties of AZ31 and found that the ductility of AZ31 is strongly dominated by the grain size. Bartolome et al. [13] successfully





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fabricated the multi-scale and multi-phase hybrid composite ceramic-metal materials Al<sub>2</sub>O<sub>3</sub>-nZrO<sub>2</sub>-Nb, and found their mechanical properties are better than those of single-phase alumina and conventional alumina-Nb. Wu et al. [14] investigated the nanocalcium carbonate composite particles synthesized by the soapless emulsion polymerization technique of double monomers and their applications. Kim et al. [15] improved the fracture toughness of carbon black and nanoclay mixed with epoxy resin and measured the fracture toughness using the single edge notched bend specimens at the room (25 °C) and cryogenic (-150 °C) temperatures. Quaresimin et al. [16] presented a survey of the effect of three different commercially available nano-modifiers on the mechanical properties of epoxy/anhydride unidirectional carbon fiber reinforced laminates and showed that the tensile modulus was slightly different from that of the unmodified laminates: whilst, a modest decrease in the tensile strength. Su et al. [17] studied the friction and wear properties of carbon fabric composites with nano-Al<sub>2</sub>O<sub>3</sub> and nano-Si<sub>3</sub>N<sub>4</sub> and indicated that the incorporation contributed to the ultimate strength of the composites. Kuo et al. [18] fabricated low density and high performance Mg-based laminated composites by means of sandwiching the AZ31 Mg foils with the carbon-fiber/PEEK(polyether-ether-ketone) prepregs. Jen et al. [19] developed a methodology to fabricate AS-4/PEEK aromatic polymer composite (APC-2) nanocomposite laminates and found the optimal content of nanoparticles  $(SiO_2)$  was 1% by total weight. Jen et al. [20] also fabricated the high performance Ti/APC-2 hybrid nanocomposite laminates to find the kink angle of Ti sheets in the stress vs. strain curves and the enhancement effect on mechanical properties by adding the optimal nanoparticles. Sutton and Varvani [21] performed fatigue analysis on Al-Al<sub>2</sub>O<sub>3</sub> composite samples with different volume fractions of alumina particles. Li et al. [22] studied the dispersion of carbon nanotubes in metal matrices for improving the properties of carbon nanotube/magnesium alloy composites. It demonstrated that the degraded matrix properties was described by the appropriate micromechanics equation [23-25]. Hawileh et al. investigated the temperature effect on the mechanical properties of several fiber reinforced polymer (FRP) laminates and presented models for predicting the mechanical properties o f carbon, basalt and hybrid carbon-basalt FRP laminates at elevated temperatures [26,27]. Analytically, the prediction results based on the approximation of hygrothermo-mechanical response models for composites were correlated well with experimental data [28]. Until now, the related research work and application of hybrid composites have been still immature. Thus, the investigation of mechanical responses in the AS-4/PEEK aromatic polymer composite (APC-2) and Mg/APC-2 with SiO<sub>2</sub> nanoparticles composite laminates at elevated temperatures due to tensile and fatigue loadings is necessarily needed to understand their complicated behaviors thoroughly.

#### 2. Experimental

The twelve-inch wide unidirectional APC-2 prepregs, from Cytec Industries Inc. (USA), were cut and spread the solution of nanoparticles on the faces of cross-ply  $[0/90]_{ns}$  and quasi-isotropic  $[0/45/90/-45]_{ns}$  lay-ups, and stacked into nanocomposite laminates. The fiber volume fraction of APC-2 was 61%. The glass-rubber transition temperature ( $T_g$ ) of PEEK was 143 °C and the melting temperature ( $T_m$ ) 343 °C. The nanoparticles SiO<sub>2</sub> supplied by Nanostructured & Amorphous Materials, Inc. (USA) possessed the average diameter 15 ± 5 nm, specific surface area 160 ± 20m<sup>2</sup>/g, spherical crystallographic and amorphous powder. According to Jen et al. [19] the optimally total weight percent of nanoparticles was 1 wt% of the weight of APC-2 laminate. The procedure of making nanoparticle solution was first to dilute nanoparticles in

alcohol (50 ml alcohol:  $2 \text{ g SiO}_2$ ) and stirred uniformly. Then, SiO<sub>2</sub> solution was spread on the prepregs, i.e., the interfaces of APC-2 laminate, in a temperature-controlled box to evaporate the alcohol.

The AZ31 (Al: 2.5–3%, Zn: 0.6–1.4%, Mn: 0.15–0.7%, Mg: balance) Mg sheets were supplied by Grandmont Co., Ltd., Taiwan. The slimmed Mg-AZ31 sheets were first subjected to abrasion with # 40 SiC abrasive papers, and to chemical etching by the solution of  $CrO_3/Na_2SO_4$  (50 g  $CrO_3$ , 4 g  $Na_2SO_4$  & 1000 g  $H_2O$ ). The surface treatment was to create the rough surfaces of Mg sheets for strong bonding with the APC-2 prepregs [18]. The fabricated hybrid APC-2 cross-ply and quasi-isotropic nanocomposite laminates were Mg/ [0/90]<sub>S</sub>/Mg/[0/90]<sub>S</sub>/Mg and Mg/[0/45/90/–45]/Mg/[0/45/90/–45]/Mg. Both the pure APC-2 and hybrid APC-2 nanocomposite 1aminates were cured by the modified diaphragm curing process [20]. The geometry and dimensions of specimens were made according to the standards of ASTM-D3039 as shown in Fig. 1.

An MTS 810 servohydraulic computer-controlled universal material testing machine was used to conduct both the tensile and cyclic tests. At the tensile tests the strain rate was 2 mm/ min. The strain was monitored by a 25.4 MTS 634.1 1F-25 extensometer at room and elevated temperatures. Each set had at least 3 specimens for tensile tests to receive the mechanical properties such as ultimate strength and longitudinal stiffness. The constant stress amplitude T-T cyclic tests were performed in a loadcontrolled mode with sinusoidal waveform at stress ratio  $R(\sigma_{min}/\sigma_{max}) = 0.1$  and frequency = 5 Hz. The T-T tests were stopped at 10<sup>6</sup> cycles as run-outs according to ASTM E606-92. Each S-N curve was constructed by  $10 \sim 12$  samples. Then, we obtained the fatigue properties, i.e., fatigue strength and life. Finally, an MTS 651 hot chamber was installed to control the specimen inside at specified temperatures such as room temperature (25 °C), 75, 100, 125 and 150 °C, for static tensile and T-T cyclic tests. Generally, it took about 30 min to warm up the chamber evenly. If the operating temperature is higher than PEEK  $T_g$  (143 °C), the mechanical properties and fatigue response of pure APC-2 laminates will be degraded in rubbery state. Then, the visco-elasticity is appropriately adopted to handle the problem. Thus, to avoid complexity we chose the highest temperature 150 °C, slightly over PEEK T<sub>g</sub>, in the study.



**Fig. 1.** (a)The geometry and dimensions of a specimen, (b) the schematic picture of a hybrid APC-2 laminate.

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