Effect of Nano Fillers on the Joint Strength of High Performance Polymeric Nanocomposite

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***Abstract*—** The main purpose of using high performance adhesive bonded polymeric composites in the automotive / aeronautics industry is to decrease energy consumption by reducing the weight of the structural parts. However, the use of adhesive bonded polymeric composite in primary structures of an automobile and aircraft is still limited, as their properties have been found to degrade drastically due to aerodynamic friction heating of the structure as it moves through the air. Therefore, striving for the development of polymeric nanocomposites is a major area of research for high engineering applications. This paper investigates the influence of carbon nano fiber and silicate nano powder on the joint strength of high performance polymeric nanocomposite based on PEEK through high performance nano adhesive bonding using ultra high temperature resistant epoxy adhesive.

***Keywords*—** Polymeric nanocomposite, PEEK, carbon nano fiber, silicate nano powder, epoxy adhesive.

I. Introduction

AUTOMOTIVE and aerospace industries are constantly striving for lighter materials especially polymer based composites in order to maximize performance while minimizing weight and cost. In recent years, a remarkable growth in the use of polymeric composites has been observed for high engineered applications in automotive and aerospace industries. The rationale behind the choice of polymeric composites lies in the superior physical, mechanical and chemical performance along with better resistance to corrosion and structural weakening from irradiation [1]. High performance polymers such as poly ether ether ketone (PEEK) with its light weight, outstanding tribological performance combined with excellent chemical, thermal and mechanical properties is increasingly used in high engineered applications. However, the harsh and difficult conditions in space demand such materials to be durable, stiff and strong and have dimensional stability to withstand large temperature variations in space. Due to these short comings of polymeric composites, research activities towards the development of nanocomposites are essential especially for the aviation and space industries. It is expected that with the inclusion of nano fillers, the adhesion characteristics of the adhesive will increase manifold owing to their extremely high surface area to volume ratios.

The use of nanocomposites in vehicle parts and systems is expected to improve manufacturing speed, enhance environmental and thermal stability, and promote recycling [2]. Thus, nanocomposites open more numerous applications in structural panels, ultra-light weight thin-walled space structures and high stiffness-to-weight space mirror substrates. Presently, investigation on polymer-clay nanocomposite for applications in aerospace has become a very important research area because it is established that thermal and mechanical properties of polymer-silicate nanocomposite are far superior to those of conventional polymer or polymeric micro composites [3, 4, 5]. A consistent small improvement in the onset temperature of the nanocomposite was noticed which suggested that clay served as a catalyst and acted as a barrier in the decomposition of the epoxy matrix which was the reason for its thermal stability [6].

Unidirectional carbon fibre reinforced epoxy straps have been proposed as fatigue crack growth retarders for aircraft construction [7]. The influence of nickel nanostrand loading level, mode of their incorporation into the epoxy resin and magnetic orientation on mechanical and electrical properties of the composite were also examined [8]. Several methods have been studied such as the incorporation of carbon nanotubes (CNT) and indium tin oxide (ITO) nanoparticles in the polysiloxane matrix, with the objective of attaining a high transparency, a high emissive, and an antistatic resin [9]. Conductive multifunctional polymer nanocomposite “NanoSphalt” is a carbon nanofibre and fibreglass composite material [10]. The nanofibres impart new property like electrical conductivity which opens the door for new applications.

Successful incorporation of nanoparticles in thermoset resins is a desirable technology for the development of high performance polymer nanocomposite for applications in aerospace [11]. But, the polymers exhibit poor adhesion characteristics due to low surface energy. To overcome this problem, Bhatnagar et al. [12] have modified the surface of PEEK by low pressure plasma and atmospheric pressure plasma and have reported in their study that plasma, especially atmospheric pressure plasma results in the significant increase in the lap shear tensile strength of the polymeric composite based on PEEK. The main focus of the present investigation has been on the improvement of high performance polymeric nanocomposite based on untreated and plasma treated PEEK through high performance nano adhesive bonding by

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dispersing carbon nano fiber and silicate nano powder in the ultra-high temperature resistant epoxy adhesive.

II. Experimental Methodology

A. Materials

In this investigation, PEEK sheets with service temperature ranging from - 250oC to + 300oC, tensile strength of 140 MPa (as reported by the manufacturer, Victrex, Lancashire, UK) were used as substrate material. Ultra high temperature resistant epoxy adhesive, DURALCO 4703, supplied by Cotronics Corp., Brooklyn, NY) with service temperature ranging from - 260°C to + 370°C was used as adhesive. CNFs with diameter ranging from 70 to 200 nm and length 50 to 200 µm, provided by Pyrograf Products Inc. Cedarville, Ohio with trade name PR-19-XT-LHT and unmodified silicate nano powder of 50nm particle size, manufactured by Glassven, La Victoria, Argua 2121 United States were used as nano particles for dispersion in to the adhesive. The mixing weight ratio of resin to hardener, curing temperature and time of this adhesive was: 1:0.22, 120 0C and 4 hours, respectively as given by the manufacturer for the best condition.

B. Methodology

The following samples were prepared and mixed thoroughly using high speed mechanical stirrer at a speed of 2000 rpm.

- Epoxy adhesive having calculated amount of resin and hardener (100/22 parts)
- Epoxy adhesive dispersed with calculated amount of 1%, 2 % and 3 % CNF
- Epoxy adhesive dispersed with calculated amount of 1 % SNP

In all the cases degassing of the adhesive was carried out in a vacuum chamber under a pressure of 100Pa.

Rectangular specimens, having dimensions length x width x thickness: 150 x 25 x 3 mm^3 were used for tensile lap shear testing. The specimens were bonded to perform single tensile lap shear tests. Prior to the preparation of an adhesive bonded joint, degassing of the adhesive was carried out under a pressure of 1 Pa for 10 min. The tensile lap shear specimens were prepared by applying high temperature resistant epoxy adhesive. Any excessive adhesive present at the interface was expelled by mechanical pressing of the joint, which resulted in a joint having an adhesive of uniform thickness about 0.20 mm. Pressure was applied to the lap joint during the curing cycle by two binder clips. The bonded specimens were cured at 120°C for 4 hours. Four types of PEEK joints were prepared and tested (i) atmospheric pressure plasma treated PEEK joint with 25 mm overlap length (ii) 1 %, 2% and 3% Carbon Nano Fiber (CNF) dispersed epoxy adhesive joint of atmospheric pressure plasma treated PEEK, and (iii) 1 % Silicate Nano Powder (SNP) dispersed epoxy adhesive joint of atmospheric pressure plasma treated PEEK.

Similarly, tensile specimens having same dimensions as above were prepared with butt joint configuration by applying high temperature resistant epoxy adhesive. The bonded specimens were cured at 120°C for 4 hours. Four types of PEEK joints were prepared and tested (i) untreated PEEK butt joint, (ii) atmospheric pressure plasma treated PEEK butt joint (iii) 1 %, 2% and 3% Carbon Nano Fiber (CNF) dispersed epoxy adhesive butt joint of atmospheric pressure plasma treated PEEK, and (iv) 1 % Silicate Nano Powder (SNP) dispersed epoxy adhesive butt joint of atmospheric pressure plasma treated PEEK.

Lastly, lap shear test of adhesive and nano adhesive bonded joints prepared with and without surface modified PEEK is carried out to determine the joint strength of the polymer-polymer joint. Similarly, tensile testing of the butt joints of untreated, atmospheric pressure plasma treated and nano particles dispersed epoxy adhesive butt joint of atmospheric pressure plasma treated PEEK was also carried out.

Both the Lap Shear testing and tensile testing of butt joints were carried out using computer-controlled testing machine, ZWICK 2010 under a load cell of 50 kN. The specimens were loaded in tension at a test speed of 5 mm/min. Five specimens of each sample were tested and the mean value is reported in the results. All the tests were performed at room temperature of 25 ^{0}C and at 50 % humidity.

III. Result and Discussion

Lap shear tensile strength of 1 %, 2% and 3% Carbon Nano Fiber (CNF) dispersed epoxy adhesive joint of atmospheric pressure plasma treated PEEK, and 1 % Silicate Nano Powder (SNP) dispersed epoxy adhesive joint of atmospheric pressure plasma treated PEEK are shown in figure 1. The figure demonstrates the comparison of lap shear tensile strength of atmospheric pressure plasma treated PEEK bonded joint and nano particles dispersed epoxy adhesive joint of atmospheric pressure plasma treated PEEK bonded joint. The figure reveals that the lap shear tensile strength of atmospheric pressure plasma treated adhesive joint increases to 10 MPa, 12.5 MPa and 12 MPa respectively with dispersion of 1%, 2% and 3% CNF in to the epoxy adhesive, resulting in considerable improvement in the adhesion properties due to dispersion of CNF in to the epoxy adhesive.

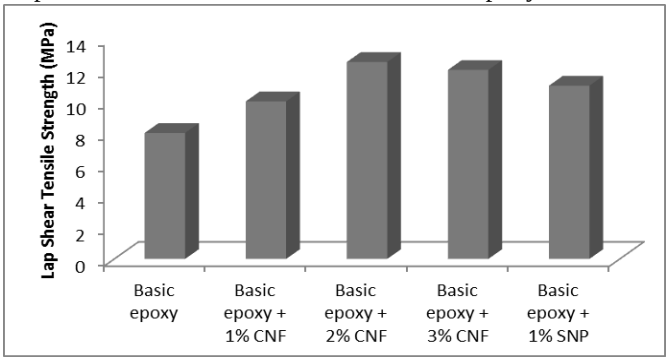

Fig. 1 Comparison of the lap shear tensile strength of Atmospheric Pressure Plasma (APP) treated PEEK, with basic epoxy adhesive joint, 1%, 2% and 3% CNF dispersed epoxy adhesive joint and 1% SNP dispersed epoxy adhesive joint

The figure also reveals an improvement in the lap shear tensile strength from 8 MPa to 11MPa after dispersion of silicate nano powder in to the adhesive.

Figure 2 shows a comparison of the tensile strength of the butt joints of untreated, atmospheric pressure plasma treated PEEK for 30 seconds, 2% CNF dispersed epoxy adhesive butt joint of atmospheric pressure plasma treated PEEK, and 1 % SNP dispersed epoxy adhesive butt joint of atmospheric pressure plasma treated PEEK. The figure clearly reveals that with atmospheric pressure plasma treatment, there is a considerable increase in tensile strength from 1 MPa to 20 MPa and shows a further improvement in tensile strength of the atmospheric pressure plasma treated joints to 25 MPa and 27 MPa with the dispersion of 2% CNF and 1% SNP respectively in to the epoxy adhesive.

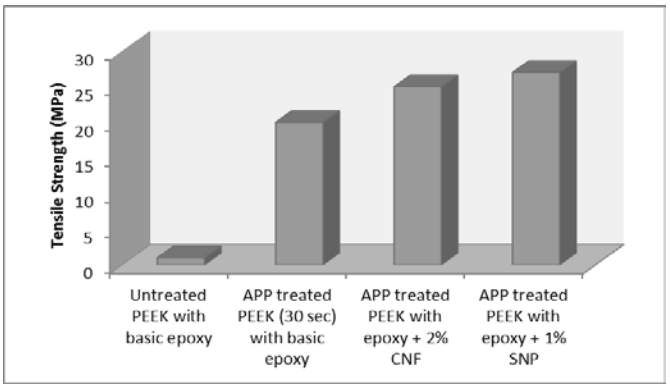

Fig. 2 Comparison of the tensile strength of untreated with basic epoxy, Atmospheric Pressure Plasma (APP) treated PEEK for 30 seconds, APP treated PEEK for 30 s with basic epoxy + 2% CNF and APP treated PEEK for 30 s with basic epoxy +1% SNP

The incorporation of nano particle like carbon nano fiber and silicate nano powder in the epoxy adhesive results in an increase in lap shear strengths from 8 MPa to 12.5 MPa and 11 MPa respectively as evident from figure 1. The tensile strength of the different joints prepared with the butt joint configuration is shown in figure 2. The figure clearly reveals that due to atmospheric pressure plasma treatment, there is a considerable increase in tensile strength from 1 MPa to 20 MPa and shows a further improvement in tensile strength to 25 MPa and 27 MPa with the dispersion of 2% CNF and 1% SNP respectively in to the epoxy adhesive.

IV. Conclusion

It is observed that when 1%, 2%, 3% carbon nano fiber and 1% silicate nano powder are dispersed in to the epoxy adhesive, the lap shear tensile strength of the atmospheric pressure plasma treated adhesive joint increases to 10 MPa, 12.5 MPa and 12 MPa respectively resulting in considerable improvement in the adhesion properties due to dispersion of CNF in to the epoxy adhesive. The result also reveals an improvement in the lap shear tensile strength from 8 MPa to 11MPa after dispersion of silicate nano powder in to the adhesive. The tensile strength of the butt joints of untreated PEEK increases from 1 MPa to 20 MPa, 25 MPa and 27 MPa respectively with atmospheric pressure plasma (APP) treated PEEK and APP treated butt joints of carbon nano fiber and silicate nano powder dispersed epoxy adhesive.

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