Overview of Nano-Composites Research Activities Conducted in ACE TeC/JAXA

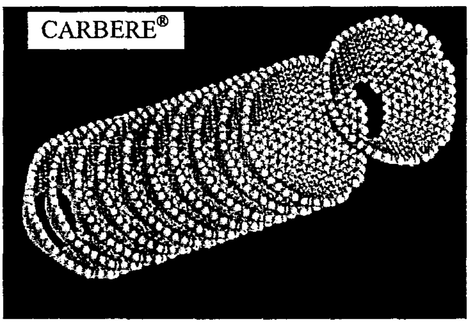
Takashi Ishikawa*, Yutaka Iwahori# and Toshio Ogasawara#
***: Director, #: Team Leader**
Advanced Composites Evaluation Technology Center (ACE TeC),
Institute of Space Technology and Aeronautics (ISTA: Used to be NAL of Japan)
Japan Aerospace Exploration Agency (JAXA)

Keywords: Carbon Nanofiber, Cup-Stuck Type, Compressive, Multi-Wall Nanotube, Heat Resistance Nanoclay, Gas Permeability

Abstract

A big boom in nanocomposites research has landed also in Japan. As a virtual "center of excellence" in composites technology there, ACE TeC of ISTA/JAXA has led pioneering portions of nanocomposites research particularly in mechanical properties oriented applications. An overview of research activities based on nano-technologies in ACE TeC /JAXA will be given first and some remarkable results will be introduced briefly.

The first example is a Carbon Fiber Reinforced Composite (CFRP) laminates using epoxy resin stiffened by carbon nanotubes (CNT). It is well known that CNT exhibits extremely high elastic modulus and strengths. One trend in CNT application as composites reinforcement is direct dispersion like chopped fiber in polymer with an alignment as parallel as possible, which is considered as two phases material of CNT and polymer. Although it looks like a proper way for obtaining CNT-reinforced composites, alignment of CNT with uniform spatial dispersion in resin is not an easy task. We pursue another route for CNT reinforced composites of three phases material, CNT, polymer and conventional carbon fiber (CF). In this idea, CNT can be regarded as a modifier of matrix resin for increasing its mechanical properties. In this trial, used CNT is a very unique one, which should be referred as carbon nanofiber (CNF): CARBERE®, made by GSI CREOS Corporation in Japan who refer this product as cup-stuck nanofiber (CSNF). A schematic illustration of such CSNF is shown in Fig.1 where conical graphene sheets are stuck like paper cups and their outer diameter is in the range of 80 to 100 nm rather larger than usual CNT. Mechanical Properties of this CSNF are compared in Fig.2 where common carbon fiber data are plotted. Two types of CSNF were employed in the difference of aspect ratios roughly of 10, i.e., fiber length of 500nm to 1μm (AR10) and 50, i.e., fiber length of 2.5 to 10.0 μm (AR50), respectively. These two types of CSNF are dispersed to



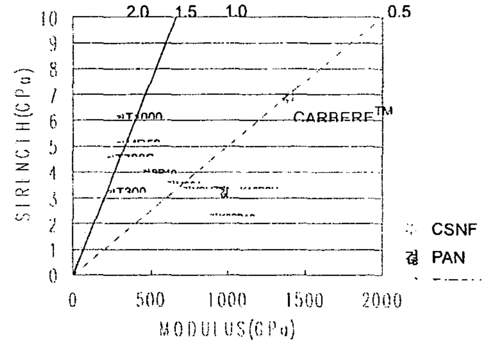


Fig.1 Schematic Illustration of CSNF:CARBERE® Fig.2 Comparison of Mechanical

by GSI-Creos Corporation, Japan Properties of CSNF with Carbon Fiber EPIKOTE® 827 epoxy resin by the company and they are supplying the dispersed compound of high fiber weight content to us. Although the final goal is to prepare the three phase composites, two phase (CSNF and epoxy) composites are also manufactured for checking the effect of resin modification. As our first processing step, fiber weight contents are adjusted by adding pure epoxy, i.e., diluted, into 5 and 10 % for both kinds of CSNF. The mixture is kept at 80 deg. C and stirred up for 30 minutes followed by hardener (Epicure W) addition. After extra stirring of 15 minutes, the mixture was evacuated for 30 minutes to degas. For two phase composites, this mixture is first cured for 75 minutes at 100 deg. C using mold. For three phase composites, the mixture is impregnated into carbon fiber fabrics (TORAYCA CO6343: T300/ plain weave) and they are laminated with 20 plies. The same curing conditions are employed, whereas a hot-press is used for this case. For two and three phase composites, control material without CSNF is also fabricated.

The essential experimental data for three phase composites are shown in Fig 3 (a) and (b) where normalized elastic moduli and strengths by control data are plotted, respectively. It can be seen that compressive strengths are improved considerably for three cases, whereas tensile strengths mainly governed by carbon fiber are not really improved. Although fully consistent and explanatory experimental data with the results of two phase composites have not been obtained yet, probably due to the effect of material defects such as voids, CSNF cluster and so on, the following data can be considered as sufficient to indicate the potential of the present CSNF for improving the mechanical, particularly compressive, properties.

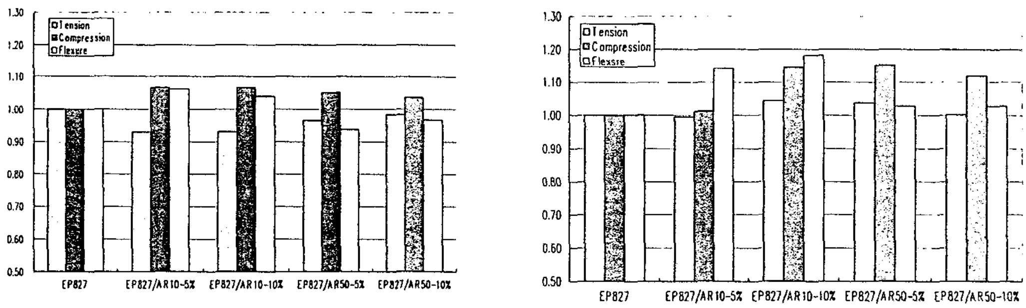


Fig.3 Comparison of Mechanical Properties of CSNF/Epoxy/CF Composites by Tensile, Compressive and Flexural Tests: (a) for Elastic Moduli, and (b) for Strengths

The second research topic in ACE TeC/JAXA is an improvement of heat resistance of high temperature polymer by loading of multi-wall carbon nanotube (MW-CNT). In this attempt, a baseline polymer itself is one of the ever-best high temperature polymer, Triple A Polyimide: TriA-PI, which shows much better heat resistance than NASA standard PETI-5. The final purpose here is to increase heat resistance such as glass transition temperature by adding MW-CNT to this polymer. The MW-CNT adopted in this attempt is fabricated through CVD technique by Carbon Nanotech Research Institute Inc. in Japan. SEM picture of this MW-CNT is shown in Fig.4 where its diameter and lengths show the scatter of 20 to 100 nm and several hundreds μm, respectively. Chosen loading weight fractions of MW-CNT are as follows: 3.3, 7.7 and 14.3%. Because the imid-oligomer is solid at room temperature, MW-CNT is added to imid-oligomer powder and mechanically blended by a ball mixer. Then the mixture is consolidated by using a hot-press for an hour at 370 deg.C. The obtained material is a two phase composite if we follow the previous terminology.

Dynamic viscoelastic properties were measured for obtained composite plates by a dynamic mechanical analyzer (DMA:Q800 of TA Instruments Corp.) in the single cantilever method and static tensile properties were measured ...

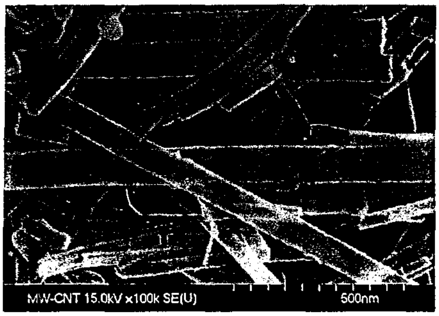


Fig.4 SEM Picture of Used MW-CNT

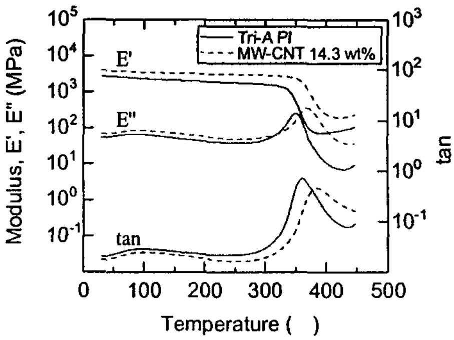


Fig.5 DMA Data (Neat Resin and Composites)

Measured data of dynamic mechanical analyzer are shown in Fig.5 where storage modulus: E', loss modulus: E'' and loss tangent: tan δ are plotted against temperature. Glass transition points (Tg) defined by initiation of E' reduction are 335 deg. C for neat resin (TriA-PI) and 357 deg. C for composite with 14.3 % weight MW-CNT, i.e., remarkable 22 deg. C increase in Tg was identified. The reason why dispersed MW-CNT increases the heat distortion temperature may be estimated as follows: the dispersed MW-CNT impedes the molecular motion in polyimide network at elevated temperature. However, more amount of research work should be required to prove that the estimated phenomenon is a true cause of higher Tg. Although static properties are obtained, discussions about them are skipped here. The final comment about the second research topic is that MW-CNT shows some potential for controlling electric conductivity and electro-magnetic wave absorbability. Some points will be disclosed at oral presentations.

The third topic about nanotechnology for composites conducted in ACE TeC/JAXA is nanoclay/polymer composites technology for increasing gas barrier property considering future space vehicle applications. However, because of the space, descriptions will be skipped in this abstract.

As closing comment, it can be pointed out that consistency and matching between nanotechnology and composites technology look very superb according to our recent experiences. We would like to increase our effort and investments to nanocomposites research area with the aid of circumstantial advantages of the implicit assistance from the Japan's annotate industries.