POSITRON TESTING OF CARBON-FIBER COMPOSITES

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INTRODUCTION

Composites of various types are becoming more and more important as structural and engineered materials. The projected growth in worldwide use for fiber composites is 13% for the decade ending in 1995 [1].

Composites can be formed from many combinations of materials and in an equally vast number of configurations. A very simplified definition of a composite is that of a matrix with embedded fibers. Both the fibers and the matrix can be of metal, ceramic, or polymer material. The fibers can be long and continuous, short and chopped, or even small particles. The materials of interest here are composites of continuous carbon fibers in a brittle epoxy resin matrix.

Two common methods of forming polymer matrix carbon-fiber composites are hand-laying and filament-winding [2]. Hand-layed composites are formed from woven sheets of fibers that are impregnated with the uncured liquid polymer. These mats of wet fibers are stacked in a specific orientation and then compressed. The matrix polymer is solidified with a curing process, and the plate of composite is then ready for use. Filament-wound composites use continuous parallel lengths of fibers that are pulled or extended from a spinneret while being continuously coated with the liquid matrix polymer. This weft of fibers is usually wound around a mandrel, or form, in a specific interlocking pattern. These filaments can also be layered by hand in an interlocking weave into flat sheets without the use of a mandrel. These two patterns are shown schematically in Fig. 1.

Carbon-fiber composites of different types can vary greatly in strength and resistance to damage. As an example, the failure strengths in tension for two different configurations of the same epoxy matrix composites are 18.1 ksi (124.8 MPa) and 127.0 ksi (875.6 MPa) [3]. Barriers to even greater use of composites are the small number of available nondestructive test methods and a lack of standardized testing and strength standards.

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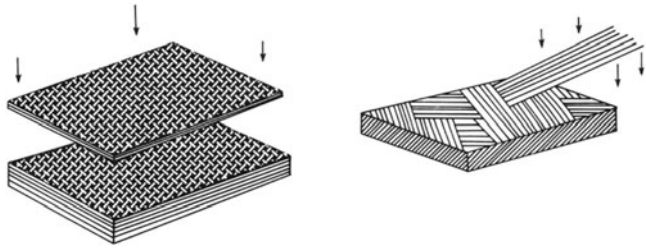

Figure 1. Schematic drawing of hand-layed (left) versus filament-wound (right) fiber composites.

Impact testing is one method in current use to quantify strengths of composites. Due to the variability of types of composites, there is no widely used absolute scale for strengths. Ultrasound scanning (C scan) is currently used as a nondestructive test method but does not lend itself easily to in-field use. Positron spectroscopy is being investigated here not only because of its nondestructive nature but also because it offers the possibility of portability of the equipment.

Positrons are antimatter particles which have the same mass as electrons but with a positive charge. Positrons are one decay product of nuclear beta decay [4]. A decay-ejected positron will possess a certain kinetic energy. As it enters and interacts with the structure of an object, it loses this kinetic energy in a process called thermalization. This thermalized positron is then an unstable particle with a very short lifetime and is subject to many different annihilation interactions [5].

When an electron and the positron come into close proximity, their opposite charges attract. As the two particles get within a critical distance, they will annihilate to give gamma rays. The dominant annihilation reaction is one electron and one positron combining to give two gamma rays propagating at approximately 180 degrees to one another [6]. These gamma rays would each have an energy of 0.511 MeV if the electron-positron pair center of mass had been stationary. If the subject electron possessed a significant kinetic energy, the gamma ray produced would be Doppler shifted from the 511 keV value. This Doppler shift can be measured and forms the basis of the technique of Doppler-broadened spectroscopy (DBS).

There are other types of spectroscopy of this dominant annihilation reaction. The length of time that the positron lives after thermalization is called the positron lifetime, and this becomes longer in a damaged crystalline material. If the electron has more than a certain threshold energy, an electron-positron pair can combine into a short-lived pseudo-atom. This pair of particles is called a positronium atom (Ps). The lifetime of the Ps atom is dependent on its chemical environment and the kinetic energy of the electron. The measurement of the lifetime of this pseudo-atom combination forms the basis of positronium lifetime spectroscopy (PLS). The variation of the propagation angle from 180 degrees of the two gamma rays is also dependent on the kinetic energy of the electron. This can be measured using angular correlation spectroscopy (ACS).

Positron spectroscopy of metals using DBS has been extensively investigated [7,8]. Also, positron and positronium chemistry using all three spectroscopic methods is a well-researched and ongoing topic [6]. The use of positron spectroscopy for the testing of carbon-fiber composites to our knowledge is a new area of research.

EXPERIMENTAL PROCEDURES

The positron source for this experiment was ^{68}Ge, an isotope of germanium with a half-life of 275 days. As the ^{68}Ge decayed, it produced positrons with a distribution of kinetic energies.

The source, a 2-mm diameter deposit of ^{68}Ge encased in a 1-cm diameter by 3-mm thick disk of metal-framed foil, was placed in a hole in a rigid stand and the specimen then taped against the source.

The gamma rays of the annihilation reaction were detected by an Ortec intrinsic germanium detector. The gamma rays entering the germanium detector were classified by a Nuclear Data multichannal analyzer (MCA). A digital spectrum stabilizer adjusted the centroid of the annihilation peak on the MCA to provide stability to the measurements. The information stored by the MCA was transferred to an IBM PC for analysis. The configuration of the experimental setup may be seen in Fig. 2.

The parameter used in this experiment was the peak-to-wings ratio (P/W) of the Doppler-broadened 511-KeV line of both damaged and undamaged samples. This parameter is both sensitive to change and easy to interpret. The IBM PC uses a program called POSITM to subtract background counts, calculate errors in the data, and give a (P/W) ratio. The (P/W) ratio of each specimen is the average of four sets of data. Each data set took approximately fifteen minutes to collect, resulting in about an hour for each final (P/W) value.

The specimens were placed directly in front of the detector with the ^{68}Ge source centered behind the specimen. A rate meter was used to position the sample each time. A stand was adjusted to a distance from the detector that gave 10,000 cps on the rate meter for each specimen. That distance was approximately 5 cm and only varied a few millimeters from sample to sample. Each data run was terminated after 1,000,000 counts were collected from a preset region of interest around the 511-KeV peak by the MCA.

The samples were received as large, irregularly cut panels of material. There were two types of plates received. One was a hand-layed, quasi-isotropic balanced laminate, and the other was a filament-wound balanced laminate. These sheets were approximately 0.125 inches thick and about one square foot in area. Both were 12 plys in thickness. The plys were oriented at 90 degrees to each other. The samples were cut from the plates using a hacksaw with a tungsten-carbide abrasive blade. The hand-layed plate yielded ten specimens while the filament-wound plate gave nine. Each final specimen measured three cm by five cm with a maximum of 10% variation in surface area. These were arbitrarily labeled A for the hand-layed and B for the filament-wound. They were marked with an indelible pen with numbers 1A thru 10A and 1B thru 9B.

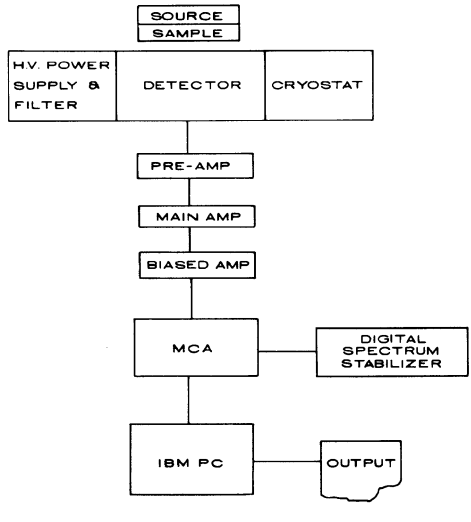

Figure 2. Schematic diagram of experimental arrangement.

Metals can easily be damaged in a controlled, quantitative manner by cold-rolling or other readily available methods. To damage a carbon-fiber composite in a quantified manner is not quite so easy. To damage the sample, a computer-controlled impact tester was used. The device used was a variable-height drop tower with changeable impact heads and masses. The drop speed and rebound speed of the impactor were measured by photoelectric means and along with the known impactor mass gave the kinetic energy of the head. The kinetic energy of the rebound was subtracted from the initial kinetic energy; this gave an amount of energy that was transferred to the specimen. The device used was built and provided by Dr. J. Nairn of the Materials Science and Engineering Department, University of Utah.

An *a priori* assumption was made that the kinetic energy transferred to the specimen resulted in structural damage to the specimen. At the higher levels of energy transferred, there was visible damage to the pieces, both on the impacted side and on the reverse side. It is presumed that at a low level of energy transferred there was a relatively small amount of damage done even in the absence of surface-visible damage.

An impactor mass of 580 grams was used for all samples. It had a 1-cm diameter and a slightly curved impact surface. The lowest height possible on the impact tester was 30 cm. This was used as a starting point for both A and B groups. The height was increased in 5-cm increments for each sample. The maximum height used, found by breaking one of the A group, was 60 cm. The B group proved to be much more resistant to damage, and the final data point for that group was three drops from 60 cm. Each sample was measured in the same manner for the DBS (P/W) ratio before and after damage. The impact data are summarized in Table 1.

RESULTS AND DISCUSSION

As seen in Fig. 3, there is a distinct difference in the ranges of the predamage DBS (P/W) ratio between the two types of composites. The (P/W) averages were 3.64 $\pm$ 0.02 for the 10

Table 1. Impact Energies

Sample Number	Drop Height (cm)	Number of Impacts	Energy Transferred (joules)
10A	30	1	1.00
2A	30	1	1.05
3A	35	1	1.24
4A	40	1	1.39
5A	45	1	1.67
7A	50	1	1.67
8A	50	1	1.90
9A	55	1	2.14
1B	30	1	1.06
2B	35	1	1.24
3B	40	1	1.25
5B	50	1	1.30
4B	45	1	1.56
8B	60	1	2.00
7B	55	1	2.02
9B	60	3	4.14

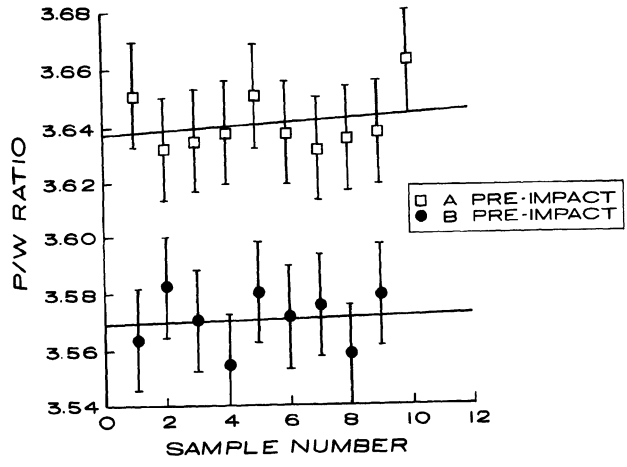

Figure 3. Before-damage (P/W) ratio of hand-layed and filament-wound samples.

samples of the hand-layed set (A group) and 3.57 $\pm$ 0.02 for the filament-wound set (B group). This is a 1.9% difference. This can be compared to (P/W) measurements in metals research where 1% difference is common [8]. Included in Fig. 3 is a linear least squares fit to the data and error bars of 0.5% of the data. This error reflects the percent difference in the maximum and minimum (P/W) measurements of each set. It may be noted that each data point is well within the error of its set.

It may be seen in Fig. 4 that as the energy imparted to the sample increased the trend in the (P/W) ratio decreased. The predamaged (P/W) ratio for each specimen is included for comparison. Errors bars are removed for clarity. A linear least squares fit is included for both pre- and postdamage to illustrate the general trends in the data.

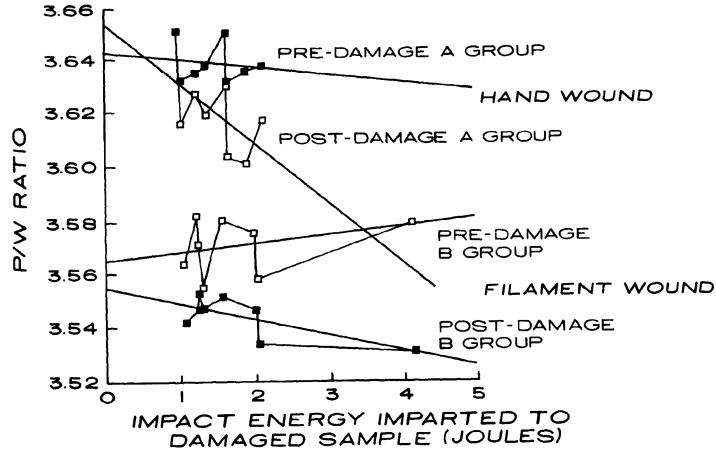

Figure 4. (P/W) ratio versus absorbed impact energy.

For metals, a decrease in the (P/W) ratio is indicative of less damage and a more perfect crystal. A cold-rolled sample will have a sharper peak and a corresponding increase in the (P/W) value relative to its pre-cold-worked value. This is the direct opposite of the trend currently observed with carbon-fiber composites.

The theories advanced for positron annihilation in metals invoke lattice defects and changes in the local balance between conduction and core electrons for explanations of the change in the annihilation peak shape. Since organic composites have few of the structures found in metals, a new theory must be found.

The existence of positronium in organic material could be involved in the explanation for the current changes in DBS line shape. In organic solids, the presence of voids leads to an increase in the concentration of positronium [9]. Also, the presence of voids in fiber composites has a large effect on the strength of the material. It is noted that a 1% increase in the volume fraction of voids in carbon-fiber composites will lead to a 7% decrease in the strength [2]. This is true for up to a 4% increase in void volume fraction.

Raw DBS data does not directly yield information about positronium concentration, but there is some evidence that deconvoluted DBS data can give such information [10,11].

CONCLUSIONS

It may be concluded from these experiments that:

1. There is a 2% difference in the peak-to-wings ratio of the Doppler broadened 511-KeV positron annihilation peak between filament-wound and hand-layed carbon-fiber/epoxy-matrix composites.
2. With an increase in the amount of damage to the specimens, there is a corresponding decrease in the trend of the peak-to-wings ratio of the Doppler peak. This is opposite to the trend of (P/W) with increasing damage in metals.

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