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Study of Carbon-Polymer Composite Samples using an Area-Array CT Scanner

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ABSTRACT

The development of advanced materials and their improvement is strictly connected to the understanding of their properties and behavior as a function of both their macro- and micro-structures. The application of X-ray computed tomography (CT) to these materials allows for a better understanding of the materials properties and behavior on either macro- or micro-structure scales. We studied five Carbon-Polymer composite samples, static and fatigue tensile-tensile loading tested, characterized by different matrix composition (Epoxy and Peek), different fibers orientation and layer sequences. We considered both unnotched and notched (by a circular hole) samples too. We scanned these material with the Lawrence Livermore National Laboratory PCAT area array system, in order to investigate the feasibility of CT to study the defects and other features for such materials.

I INTRODUCTION

A general understanding of the damage and failure mechanism is essential for predicting the evolution of mechanical properties in composite materials when subjected to a static or fatigue loading test. Thus it is very important to be able to describe in as much detail as possible the damage situation, using different observation techniques.

One of the main tasks of this work was to demonstrate the capability of X-ray Computed Tomography when applied to the study of this specific class of material, characterized by poor contrast between their different components, typical loading size, notched or unnotched and to compare the results obtained with the observations due to other Destructive and NonDestructive laboratory techniques (e.g., Film Radiographs, Stereoscopic X-ray, Optical Microscope and Ultrasonics). This specific study was carried out analyzing the data from an area-array detector Computed Tomography scan system developed at Lawrence Livermore National Laboratory.

II MATERIAL DESCRIPTION

The samples used for this preliminary study are Graphite-Epoxy and Graphite-Peek composites. The graphite fibers are GRAFIL 6KXAS fibers for the Fibredux 914 epoxy resin samples and AS4 graphite fibers for the peek resin ones [AYM92].

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A list of the samples and their characteristics are included in Tables 1 and 2.

TABLE 1 : Graphite-Epoxy Samples

NAME	SU7/2/P10	SU7/1/A
LAYERS SEQUENCE	[$\pm 45/0/90$] _{4s}	[$\pm 45/0/90$] _{4s}
DENSITY (g/cc)	~1.6	~1.6
GEOMETRY	Unnotched	Circular Hole
CROSS SECTION (mm ²)	20 × 2.2	50 × 2.2
HOLE DIAMETER (mm)	NA	8
ASPECT RATIO	~9	~9
LOADING TEST	Static	Fatigue
N.OF CYCLES	NA	750000
MAX STRESS (% σ_{fract})	100	80

TABLE 2 : Graphite-Peek Samples

NAME	P35P.20	QIBH.9	P15H.11
LAYERS SEQUENCE	[± 35] _{4s}	[$\pm 45/0/90$] _{2s}	[± 15] _{4s}
DENSITY (g/cc)	~1.6	~1.6	~1.6
GEOMETRY	Unnotched	Circular Hole	Circular Hole
CROSS SECTION (mm ²)	20 × 2.2	50 × 2.2	50 × 2.2
HOLE DIAMETER (mm)	NA	8	8
ASPECT RATIO	~7.6	~7.6	~7.6
LOADING TEST	Fatigue	Fatigue	Fatigue
N.OF CYCLES	7000	277000	500000
MAX STRESS (% σ_{fract})	65	70	88

CT was used to study the composite samples only after the loading tests. All the loading tests were carried out at the University of Cagliari (Italy) in a servohydraulic ± 250 kN MTS machine under load control. Fatigue loading was sinusoidal tension-tension with $R = \sigma_{min}/\sigma_{max} = 0.1$ and frequency 3 Hz. All the sample were immersed in a radio-opaque zinc iodide solution¹ in order to infiltrate the damaged area and enhance the contrast of film radiographic x-ray images.

III. CT SCANNER

The system used in this study, called PCAT, is a area-array (2D) third generation CT scanner. This system uses a 450 kV x-ray machine source manufactured by Phillips. The area-array detector system consists of a scintillating material lens coupled to a CCD camera. PCAT (see Fig. 1) is used to acquire digital radiographic or projection images. The set up used in the experiments presented here is as follows:

- a 63 × 63 mm² high-density scintillator, that converts the x-ray beam into a visible-light image of the scanned object;

¹ The composition of the used zinc iodide solution was: 60 g of zinc iodide, 1 ml of Kodak Photoflo, 10 ml of distilled water and 10 ml of isopropyl alcohol.

- a mirror, that removes the CCD from the direct path of x-rays (the x-rays are a possible source of damage and hot pixels);
- a Pb glass window, to reduce scattered x-rays entering the CCD camera;
- a thermoelectrically cooled CCD camera (with a dynamic range of 14 bits), whose sensor contains 1024 rows and 1024 columns of detector elements, each $\sim 15 \times 15 \mu\text{m}^2$.

The PCAT staging consists of three degrees of freedom: rotational, and x- and y-translations. These are mounted on a computed-controlled system that provides the movements along all three axes remotely. PCAT uses a Sun 3/260 workstation for data acquisition. The PCAT system was configured to obtain a reconstructed pixel size of about $61.7 \mu\text{m}$. All data were acquired at a peak energy 100 kV and current 9 mA, over a range of 180 DEG; each scan session includes 360 equispaced projections in form of Digital Radiographs (DR).

All the sample have a large aspect ratio, i.e. high value of width to depth in the cross section, which causes a strong variation in the x-ray attenuation in the two directions along the x and y axes. We decided to scan the specimens in three different ways :

- one single sample for P35P.20;
- two pieces of the same sample together for SU7/2/P10;
- three different samples together for QIBH.9, P15H.11, & SU7/1/A;

to compare these different combinations and to find the best manner to study such samples.

The scanner output was preprocessed in order to remove some source of errors that may affect the reconstructed images; these error may be due to:

- the effect of background and dark current;
- beam hardening (BH) effects due to the polychromaticity of the x-ray radiation;
- local pixel noise;
- image-to-image variations;
- detector-to-detector imbalances.

We omitted the BH correction in the data corresponding to the P35P.20 CT scan session, in order to evaluate the influence of this kind of error on the final reconstruction results for the Carbon-Polymer Composite samples.

The data preprocessing include the calculation pixel-by-pixel of the x-ray linear absorption coefficient (i.e. $\ln I_0/I$ or $\mu_r \ell$), according to Beer's law :

$$I = I_0 e^{-\mu_r \ell}$$

where:

- I = photon intensity of the transmitted beam;
- I_0 = photon intensity of the incident beam;
- $\mu_r \ell$ = relative x-ray absorption;
- μ_r = relative linear attenuation coefficient;
- ℓ = path length.

For details of the physical x-ray interactions see Barrett and Swindell. [BAR81]

In order to obtain further information concerning the acquired data, the data are rebinned to create sinograms from the available DRs; the same sinograms were used as input data for the reconstruction process.

The best results were obtained using the Filtered Backprojection and Convolution Backprojection image reconstruction algorithms, available inside the RECON package at the Lawrence Livermore National Laboratory.[BRA95] Typically we used a Butterworth filter of order 35 and cutoff frequency of 0.45.

IV RESULTS AND DISCUSSION

The data resulting both from Digital Radiographs and CT reconstructions may be analyzed from different points of view. First of all we can visually compare the x-ray relative absorption coefficient (DR) or x-ray linear attenuation coefficient (CT) variations within each image and from image to image. Such analysis assumes the use of the same color or gray scale for all the considered images; this technique reveals gross anomalies inside each image and between different images. A more detailed analysis is performed by extracting and plotting 1D profiles of the data; these profiles can be used to reveal small variations from image to image and within each image.

Another detailed technique deals with the entire object or 2D regions belonging to it; this analysis involves the study of the histograms, the mean, the variation and the standard deviation of the CT data set. The histogram of a CT image (generally a 2D one) gives a representation of the number of pixels whose value is within a particular range of the attenuation coefficient. It can be used as qualitative (for non-energy discriminating detector scanners) and a quantitative (for energy discriminating detector scanners) measure of the total uniformity within the object, or a portion of it.

One measure of the total uniformity within the object is accomplished by measuring the Full Width at Half Maximum (FWHM) of the resulting peak in the histogram; furthermore we can measure the CT scanner contrast sensitivity by calculating the FWHM and the distance between two peaks representative of two regions with different linear attenuation coefficient (CT slices). The mean value can be regarded as a measure of the centroid of the histogram or as the mean attenuation coefficient within the sample at the considered slice plane. The standard deviation is related to the FWHM of the histogram or more importantly the attenuation coefficient variance within the sample for a particular slice plane.

The variance is the measure of the variation in the pixel-wise attenuation coefficient within the considered region, and it is a function of three components:

- the CT scanner measurement errors due to statistical and systematic noise sources (e.g. photon statistical noise, electron noise, etc.);
- reconstruction errors due to the algorithm properties (e.g. point-spread function or PSF, and noise amplification);
- deviation in uniformity due to variation in Z and ρ (i.e., atomic number and volume density, respectively).

We used all the previous mentioned techniques, except for the calculation of the mean and the variance. The existence of zinc iodide remnants determines in fact strong local variations of the attenuation coefficient that invalidate the study of the variation of the mean and the variance along the vertical z axis.

DIGITAL RADIOGRAPHS AND CT RESULTS

Unnotched Samples

SU7/2/P10 SAMPLE

The Digital Radiographs of Fig. 2 show some visible areas characterized by a higher absorption value (especially near the right vertical edge of the sample), possibly due to zinc iodide remnants;

* FWHM = 2.354 σ for a Gaussian distribution.

the breakage section is clearly represented and it's also possible locate the different ply orientations within the sample.

The CT reconstructed slices presented in Fig. 3 allow us some interesting observations; first of all we can notice clearly the paper label along the lower edge of the lower sample; this fact gives an idea of the goodness of the resolution adopted with the PCAT scan system. Moreover all the images show some higher x-ray attenuation regions near the right edge, corresponding to the same location where we noticed the strong absorption line in the radiographs; we suppose the existence of higher absorption materials in the specimen, in the specific situation a remnant of the zinc iodide contrasting agent. After the loading test in fact all the samples were immersed in a zinc-iodide solution to improve the contrast in the Radiographic Films used to study the internal damage due to the static or fatigue test [AYM92 & AYM93]. Even though the samples were left inside a water recipient for almost half an hour to eliminate all the remnants of the contrast liquid, we located some strong variations of the attenuation coefficient (with an order of 10 to 1 with respect to the mean value of the signal inside the sample) due more likely to a change in the effective atomic number than in the value of the volume density.

We can notice some brighter horizontal lines too, near the central section of the sample, where the relative attenuation coefficient is higher; the reason would be the existence of one (or more) layer(s) characterized from an higher value of the material density and/or effective atomic number in the matrix or in the percentage of the graphite fibers, or also it could be due to a different (less) damage of the central sample area, or again most likely zinc iodide. Extracting three signals along the x axis averaged over 2 pixels width, in correspondence to the slice 112, as shown in Fig. 4, we can observe inside an high-attenuation area a value of the signal five times the outside signal value. Through the VIEW signal image processing software we can visualize (see Fig. 5) longitudinal and/or transversal 2D sections normal to the x or y axis as well; these images allow us to see more detailed features inside the specimen.

P35P.20 SAMPLE

This sample is characterized by a graphite-peek material composition, with a sequence of $\pm 35^\circ$ fiber orientated plies; it was fatigue loaded (for further detail see Table 1). A series of 45° equispaced preprocessed Digital Radiographs is shown in Fig. 6; the top high-absorption area corresponds to some of the glue used for the connection between the specimen heads and the aluminum reinforcement.

We took into consideration several algorithms for reconstructing the internal cross sections of the sample; we found the Convolution Back-Projection (CBP) the most efficient. Using the images of Fig. 7 we can visually compare the tomogram corresponding to the slice at $z = 2$ when reconstructed using the CBP and the FBP (Filtered Back-Projection) algorithms and the image difference between the first and the second, respectively. The main differences could be noticed in the bottom edge and around the right bottom corner, so even if the CBP is more expensive in terms of computational time (about 3.5-4 times longer than FBP), we decided to adopt it for the reconstruction process.

The study of all the tomograms reveals the existence of several damaged areas near the outer edges of the sample for all its vertical development (see Fig. 8), but a relative homogeneity of the attenuation value inside it. This homogeneity is confirmed from the analysis of the signal value for different slices. Fig. 9 shows two different plots (each for one value of the average width) of the attenuation value extracted along the x axis corresponding to different position inside the tomograms or cross sections. The parabolic curve corresponding to the signal inside the sample is due to the beam hardening effect. In the tomogram corresponding to the slice at $z = 649$ we can notice also voids inside the extracted regions.

Further details may be noticed observing the internal structures of the sample in the x,z and y,z planes. The vertical extension and the length (along x) of the damaged areas are clearly detectable

in the x,z planes (see Fig. 10, section a-a and b-b); the depth and other detail along the y axis are instead noticeable in the y,z planes (compare with sections c-c , d-d and e-e on Fig. 10). No zinc iodide remnants were found in this sample.

Notched Samples

The three notched samples were scanned simultaneously; for this reason both the Digital Radiographs and the tomograms contain the information related to all three notched samples. By any way we'll summarize our observations for each one.

QIBH.9 SAMPLE

For the notched QIBH.9 piece we know from the mechanical model of failure [AYM93] that the ultimate sequence is characterized by the fracture of the 0° fibers near the hole and large matrix cracks in the adjacent -45° plies, noticeable in a typical triangular distribution around the hole. We determined that the triangular damaged area around the hole appears in the x,z plane sections near the outer edge of the sample, but is not detectable inside (see Fig. 11).

SU7/1/A SAMPLE

The reconstructed slices of this graphite-epoxy sample reveal several wide damaged regions along the whole studied length; two representative tomograms are shown in Fig. 12 ; we can notice the existence of some areas characterized by a high value of the relative attenuation coefficient, in some cases 6.5-7 times the average attenuation value inside the sample (see Fig. 13). Also in this situation we interpret this strong changing in the attenuation value as due to the infiltration in the damaged areas of the radio-opaque zinc iodide solution. In Fig. 14 we can notice the different fiber orientation along the plies and a higher concentration of the damage in the right part of the sample. We also can notice some zinc iodide remnants around the hole.

P15H.11 SAMPLE

This piece appears to be less damaged than the other two, as shown in Fig. 14. We didn't find any particular feature inside it moreover than the knowledge about failure detectable with other Destructive and NonDestructive methods. Moreover we were not able to detect zinc iodide remnants inside the sample, except for a small area around the hole. This fact could be considered as an index of the low level of damage.

V SUMMARY

In the present work, we studied five Carbon-Polymer Composites samples with different matrix composition, geometric characteristics, and static or fatigue loaded to failure. These circumstances and the unknown situation of the internal feature of the sample before the tensile loading tests do not allow us to ascertain exactly the origin of some observed characteristics inside the sample. However we do know that all the samples were immersed in a zinc iodide solution for enhancing the contrast of film radiographic images. This fact helped us to interpret some strong variations of the x-ray attenuation detectable in the reconstructed slices. We considered that all the samples were subjected to a loading test that could have affected the internal physical properties, such as density. However the x-ray absorption variations are too high to be due to a change in the density of the graphite or epoxy/peek resin. These variations are more likely caused by a change in the effective atomic number. There are two possible explanations. The first, may be due to inclusions (high Z

materials) being introduced into the sample during the manufacturing process. The second possibility is due to remnants of the zinc iodide contrast agent. The latter is most likely.

The CT scan spatial resolution ($\sim 61.7 \mu\text{m}$) allow us to resolve internal features and the thickness of each layer with about two times the pixel size accuracy ($\sim 124 \mu\text{m}$). The CT images produced with this pixel accuracy are very revealing as to identification of external and internal features. However, this spatial resolution limits the size of features that can be resolved and our understanding of layer delaminations. Furthermore, the low contrast between the fiber and the matrix material doesn't allow us to resolve the exact location of either. The possibility to study the sample with higher resolution may allow us to investigate more detailed internal features and to understand their origin.

VI FUTURE WORK

We applied CT to study a set of Carbon-Polymer composites, characterized by a thermoset or thermoplastic matrix, when subjected to either static or high-stress fatigue loading tension. Our future interest will be focused on studying the performance of other LLNL CT systems applied on the present samples. For example, using linear-array detectors, different energy values and different spatial resolutions, to investigate the best methodology to study the defects and other features for such materials.

We plan to investigate some high-temperature thermoplastic polyimide composite samples too, before, during (at set intervals) and after full-reversal (tension-compression), low-stress fatigue loading at the upper use temperature.[SAV95]

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IX FIGURE CAPTIONS

- Fig. 1 Schematic of the PCAT acquisition system.
- Fig. 2 Two representative Digital Radiographs (on the left the radiograph corresponding to 0° and on the right to 30°) of the static loaded SU7/2/P10 sample; along the vertical right edge of the sample we can observe an higher value of the absorption coefficient ($\mu_r \ell$).
- Fig. 3 Six reconstructed cross sections (or tomograms) of the SU7/2/P10 sample, starting at $z=50$ and with $\Delta z=25$ (from top left to bottom right moving along a vertical line as labeled in brackets). Each slice includes two pieces of the sample (the top one was scanned in correspondence of the breakage section). We can notice: in the bottom piece (lower edge) the change in depth due to the paper label; the high attenuation area near the right edge; and the horizontal high attenuation area(s) in the central part of the sample.
- Fig. 4 Three reconstructed slices of the SU7/2/P10 sample (on the left) and a plot of three signals extracted along the x axis, as shown in slice 112 or the second tomogram. We can notice the lower value of the attenuation coefficient corresponding to the breakage area of the sample (~ 0.024 , s0 plotted line) and the peak corresponding to the zinc iodide remnant; the signals inside a less damaged part of the sample (s1 and s2) show an average attenuation value ($\sim 0.48-0.5$) higher than in the previous situation; again we can see the peak on the right corresponding to the high attenuation region along the right edge of the sample.
- Fig. 5 A tomogram of the SU7/2/P10 sample (on the top) with three longitudinal sections (as labeled) in the plane x,z (bottom). The different orientations of the fibers are clearly shown as well as other kinds of features.
- Fig. 6 A series of five equispaced Digital Radiographs of the P35P.20 graphite-peek unnotched sample, from 0° to 179° with an angular step of 45° ; the top area in each radiographs is characterized by a high absorption value due to the glue used to connect the specimen heads with the aluminum reinforcement.
- Fig. 7 Two images of the same slice performed with the Convolution Backprojection (top left) and the Filtered Backprojection (top right) reconstruction algorithms for the P35P.20 sample. The bottom image shows the difference pixel-by-pixel between the results of the two reconstruction methods proposed.
- Fig. 8 A digital radiograph of the P35P.20 sample at 0° (left) and five reconstructed slices with their location along the z axis as shown. Several damaged areas near the edges, corresponding to cracks and delaminations are easily shown in the tomograms.
- Fig. 9 A reconstructed slice corresponding to $z=649$ for the P35P.20 sample (middle); A plot of the signal extracted along the s0 line for two different pixel widths (black line 1-pixel and red line average of 10-pixels) is shown at the top; in the bottom the same representation,

but for the s1 extracted signal. The parabolic curve corresponding to the attenuation coefficient inside the sample is due to the beam hardening effect.

- Fig. 10 The reconstructed slice at $z=190$ (bottom right) with five sections in the x,z or y,z plane. These images show more detail about the internal damage of the sample P35P.20.
- Fig. 11 Representative tomograms or cross sections containing all the three notched samples (top) and three sections in the plane x,z for the QIBH.9 piece as labeled. Note that the triangular damaged area appears near the outer border (sections s_0 and s_2) but not inside the piece (section s_1).
- Fig. 12 Two representative tomograms for all three notched samples at $z=19$ and $z=21$; we notice that the SU7/1/A epoxy sample (third piece from the top) appears to be more damaged than the peek and it contains several remnants of the zinc iodide contrasting agent or inclusions.
- Fig. 13 The reconstructed slice at $z=19$ (top) with a plot (bottom) of the attenuation value extracted along the x axis for the SU7/1/A sample. The profile was chosen to include some high attenuation regions; the black line represents the attenuation coefficient with a 1-pixel width, the red one, is calculated for a 10-pixel average width.
- Fig. 14 A reconstructed cross section containing all three notched samples (top) for $z=59$ and three sections in the plane x,z for the SU7/1/A sample only; we see that the triangular damaged area appears near the outer border (sections s_0 and s_2) but not inside the piece.

Fig.1

Fig. 2

Fig. 3

Fig. 4

Fig. 5

Fig. 6

Fig. 7

DIGITAL RADIOGRAPH and RECONSTRUCTED SLICES

Fig. 8

TWO DIFFERENT 1D PROFILES EXTRACTED FROM THE 649th SLICE WITH TWO DIFFERENT AVERAGE WIDTH

SECTIONS ALONG Y- and X- AXES

Fig. 10

Fig. 11

Fig. 12

Fig. 13

Fig. 14

Fig. 14

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