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CHARACTERIZATION OF STATIC- AND FATIGUE-LOADED CARBON COMPOSITES BY X-RAY CT*

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ABSTRACT

The development and improvement of advanced materials is strictly connected to the understanding of the properties and behavior of such materials 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 applied CT to study a set of aerospace grade carbon fiber/thermoplastic matrix composites. Samples of APC-2 (PEEK/AS4) were subjected to either static or high-stress fatigue loading in tension. Both notched (central circular hole) and unnotched specimens were examined. We are investigating a high-temperature thermoplastic polyimide composite sample by acquiring CT data sets before, during (at set intervals), and after full-reversal (tension-compression), low-stress fatigue loading at the upper use temperature. The CT scanner employed and the results obtained in the analysis of 3D CT data sets to study the defects and other features within the different composites are presented in this report.

I INTRODUCTION

Computed Tomography methodologies were investigated to better understanding their possibility to improve the knowledge and a correct understanding of the behavior of thin Carbon-Polymer composites when static or fatigue loaded to failure.

The main goal of this work was to demonstrate the feasibility of using Computed Tomography to characterize the internal defects and microstructural damages for such materials. Our tasks included studying the typical load size composite specimens (on the order of 20 x 2 x 200 mm), identifying the optimum scan parameter values for these samples, to correlate the defects with the corresponding loading situation in order to evaluate the agreement of the real data with the mechanical models of failure proposed in previous studies [1-3].

II MATERIAL DESCRIPTION

The samples used for this study are Graphite-Epoxy [3], Graphite-Peek [2] and Graphite Polyimide[1] composites. A list of the samples and their material, geometric and loading characteristics are included in Tables 1-3, respectively.

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TABLE 1 Material Characteristics

Name	CT Scan Session	Fiber-Matrix	Layers Sequence	Density (g/cc)
SU7/2/P10	SS-A	Graphite-Epoxy	[±45/0/90]4s	~1.6
P35P.20	SS-B	Graphite-Peek	[±35]4s	~1.6
QIBH.9	SS-C	Graphite-Peek	[±45/0/90]2s	~1.6
P15H.11	SS-C	Graphite-Peek	[±15]4s	~1.6
SU7/1/A	SS-C	Graphite-Epoxy	[±45/0/90]4s	~1.6
QPS1	SS-D	Graphite-Polyimide	[+45/90/-45/0]2s	1.6

TABLE 2 Geometric Characteristics

Name	Geometric characteristics	Geometric cross section size (mm)	Hole Diameter (mm)	Scan Aspect Ratio
SU7/2/P10	unnotched	20 x 2.2	NA	~4.5
P35P.20	unnotched	20 x 2.2	NA	~9
QIBH.9	circular hole	50 x 2.2	8	~7.6
P15H.11	circular hole	50 x 2.2	8	~7.6
SU7/1/A	circular hole	50 x 2.2	8	~7.6
QPS1	unnotched	25.4 x 2.286	NA	~5.5

TABLE 3 Loading Characteristics

Name	Loading Test	Total number of cycles	σ_{max} % fract. load	Temperature (°C)
SU7/2/P10	static/tens-tens			20
P35P.20	fatigue/tens-tens	7000	65	20
QIBH.9	fatigue/tens-tens	277000	70	20
P15H.11	fatigue/tens-tens	500000	88	20
SU7/1/A	fatigue/tens-tens	750000	80	20
QPS1	fatigue/tens-comp	961401	0.2	180

The CT study for all the Epoxy and PEEK samples occurred after the loading tests done at the University of Cagliari. The Graphite-Polyimide sample was studied before and after four tensile-compression low-stress fatigue loading tests at LLNL. CT data after loading were acquired every 0.2 Mcycles at 180° C. The fourth loading test was interrupted before completing its 0.2 Mcycles (total load of 961401 cycles) because excess deformation due to reduction in sample stiffness, exceeded 0.02 mm.

III CT ACQUISITION SYSTEM

The CT scanner used in this study, called PCAT, is a area-array (2D) rotate only (third generation) system. It basically consists of a 450 kVp x-ray machine source manufactured by Phillips and a detector system that couples a thermoelectrically cooled Photometrics Astronomy grade CCD camera to a high-density scintillating glass by a photographic lens. A schematic of PCAT is shown in Fig. 1. 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. Data preprocessing, image reconstruction and analysis are typically done on another Sun or Silicon Graphics computer (for this specific study we used a Silicon Graphics ONYX machine).

PCAT was used to acquire CT projections or Digital Radiographs (DR). It was configured to obtain a reconstructed pixel size of about 60.5 μm for the Peek and Epoxy samples and about 41.7 μm for the Polyimide one. In order to have the 41.7- μm pixel size we needed to acquire two different CT scans to image most of the height of the sample (about 200 mm) for each load situation. All data were acquired at 100-kVp peak energy and 9-mA current, over a range of 180°; each scan session includes 360 equispaced projections in form of digital radiographs.

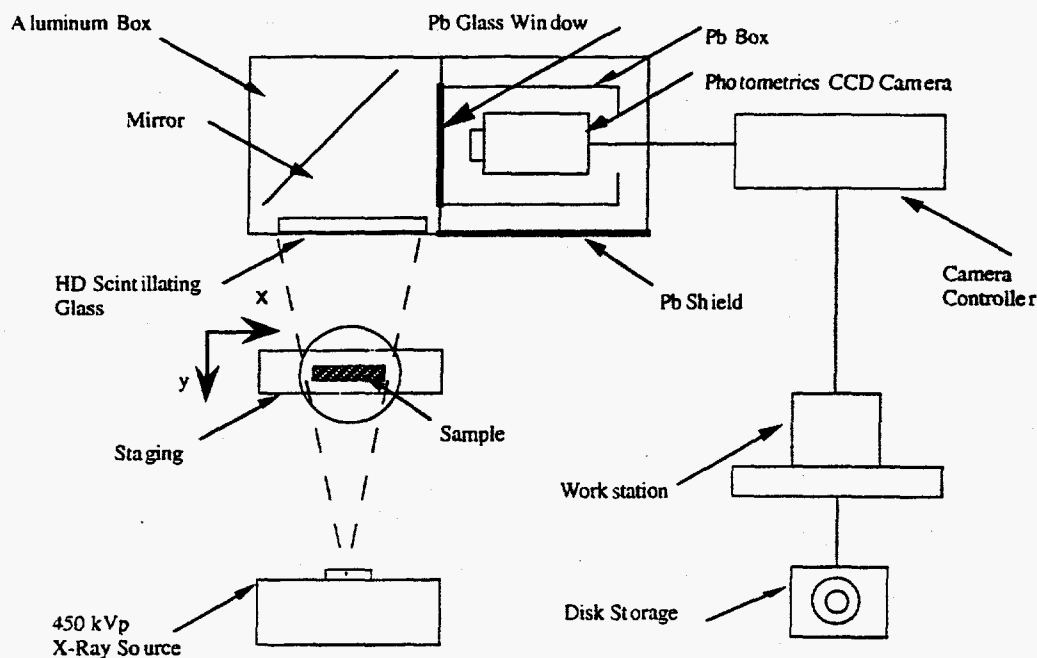


Fig.1 Schematic of the PCAT System.

All the studied samples have a large-aspect ratio (see Table 2), i.e. high value of width to depth in the cross section, which causes a strong variation in the x-ray absorption in the directions along the x and y axes. Therefore we decided to scan the specimens in three different configurations (see Table 1):

- Scan-Session A (SS-A) two pieces of the same sample together for SU7/2/P10;
- Scan-Session B (SS-B) one single sample for P35P.20;
- Scan-Session C (SS-C) three different samples together for QIBH.9 , P15H.11 and SU7/1/A;
- Scan-Session D (SS-D) one (test only) and two (test and reference) pieces of the same material for QPS1;

to study the influence of the value of the aspect ratio in the reconstruction process for the Carbon-Polymer samples.

The scanner output was preprocessed in order to remove some source of errors that may affect the reconstructed images. We used both the Filtered Backprojection and the Convolution Filtered Backprojection reconstruction algorithms [4], available inside the RECON package at the Lawrence Livermore National Laboratory [5], with a Butterworth filter of order 35 and cutoff frequency 0.45.

IV RESULTS AND DISCUSSION

The data resulting both from DR and CT reconstructed images may be analyzed from different points of view [4,6,7]. First of all we can visually compare the DR x-ray absorption coefficient or CT relative x-ray attenuation coefficient variations within each image and from one image to another¹. Such analysis assume the use of the same display scale and reveals gross anomalies inside the composite samples. The CT data provides the possibility to view internal features within the sample along the three different axes in the 3D space. This allows us to locate several defects in the studied samples, such as cracks, inclusions, voids, delaminations, and to calculate its spatial extent.

¹The basic relationship for DR and CT is represented by $I = I_0 e^{-\mu_r \ell}$ where : I is the photon intensity of the transmitted beam; I_0 is the photon intensity of the incident beam; ℓ is the path length; $\mu_r \ell$ is the relative x-ray absorption; and μ_r is the relative attenuation coefficient, that is a non linear function of the energy, E, the density, ρ , and the effective atomic number, Z_{eff} .

A more detailed analysis is performed by extracting and plotting 1D profiles of the data. These profile can spot small variations within each image. This technique allows to confirm and quantify the anomalies revealed with the previous mentioned analysis and to locate smaller variations between close points. 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 [6,7].

The first two techniques were used for all the scanned samples, the third only for the Graphite-Polyimide one. Here we only describe the results from the SU7/1/A, QIBH.9 and QPS1 samples. The results concerning the other samples are given in [8]. Several interesting topics were identified and analyzed in all the considered samples.

Peek and Epoxy Samples

For the notched QIBH.9 piece, we know from the mechanical model of failure [2] 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. Radiographically we could not determine the number of -45° plies damaged. In the CT data, we observed that the triangular damaged area appears in the plies near the outer edge of the sample (see Fig. 2), but is not detectable in the inner plies.

After the loading test 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 [2,3]. Even after the samples were left inside a water recipient for almost half an hour to eliminate all the remnant of the contrast liquid, we located some strong variations of the attenuation coefficient (up to 6 to 1 with respect to the mean value of the signal inside the sample). This is more likely due to a change in the effective atomic number than in the density value. This result is shown in Fig.3 for the SU7/1/A sample.

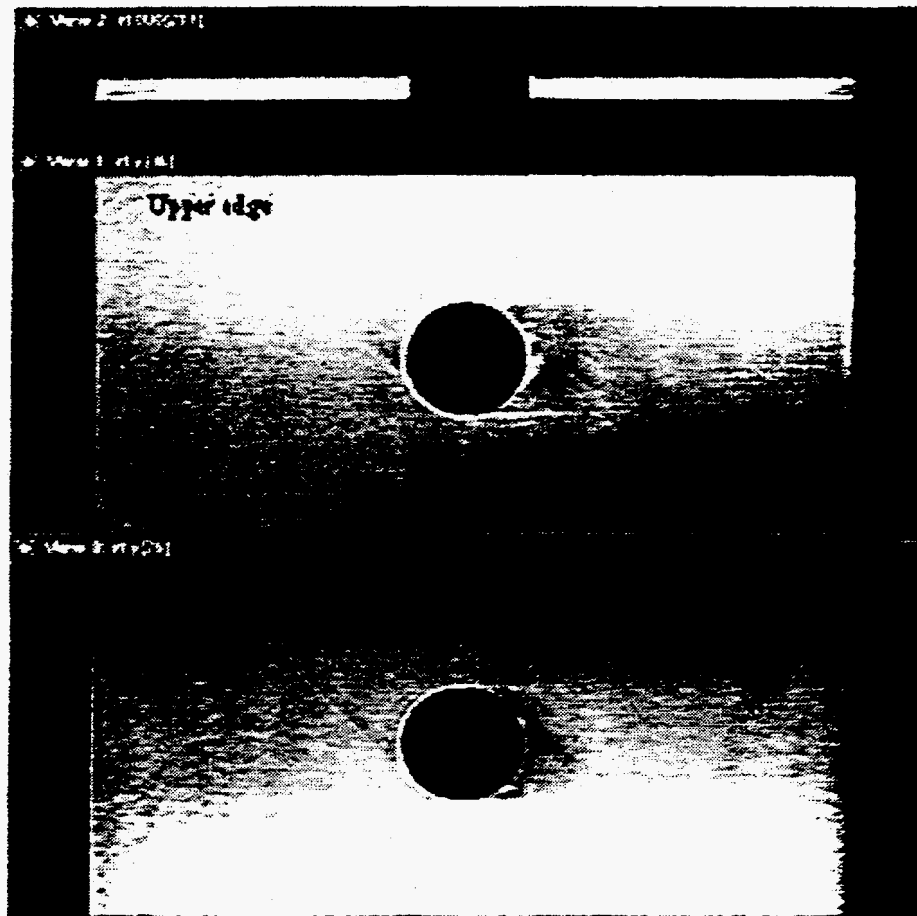


Fig. 2 A reconstructed slice (top) of the QIBH.9 sample with two longitudinal sections (middle and bottom) in the x-z plane. The damaged triangular area around the hole corresponds to the -45° plies near the outer edge of the sample.

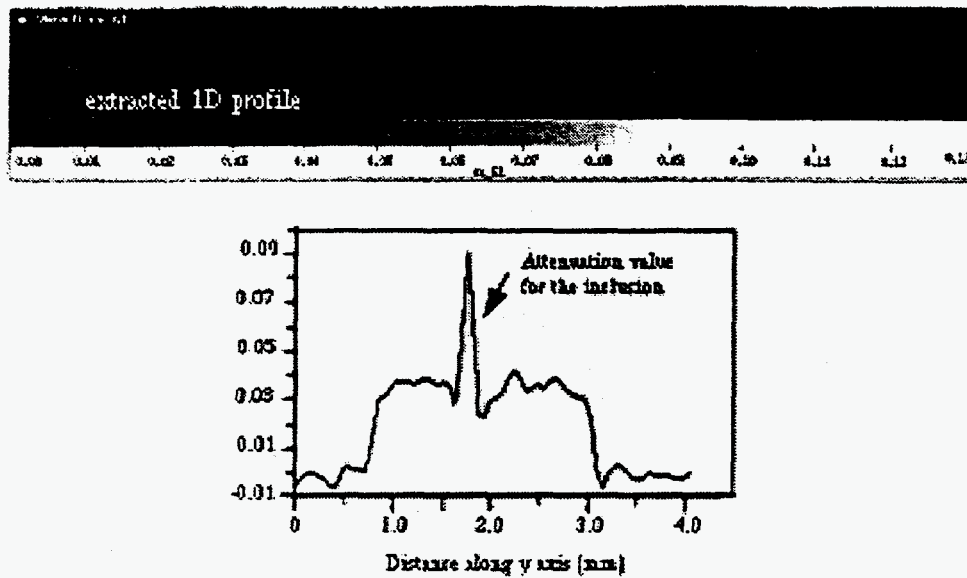


Fig. 3 The SU7/1/A sample: a representative CT cross section (top) and 1D profile (bottom) plotted for the line shown in the top CT image.

Graphite-Polyimide Sample

The Digital Radiograph at 90° reveals two interesting vertical regions (see Fig. 4-left) characterized by a higher value of the relative absorption coefficient, and several low absorption vertical areas; both these regions are most likely due to a variation of the density value. Preliminary analysis revealed that we may correlate the low-density regions with the different ply orientations, see Fig. 4-right. Because all these features appear in the untested sample yet, we may affirm that are due to the manufacturing process.

Comparing the tomograms of a same region with respect to the different loading situations we found some interesting topics :

- a crack, located in the top grip area, appears after the first loading cycle, and seems to be closing in the last considered situation (see Fig. 5);
- an inclusion, identified in the unloaded situation yet, doesn't change both shape and position with the increase of the number of the cycles;
- several delaminations appear near the edges after the last fatigue test, for both the gauge and the grip areas (see Fig. 6-a and b);
- a void was clearly located in the ~ 0.9 Mcycles situation (see Fig. 6-c).

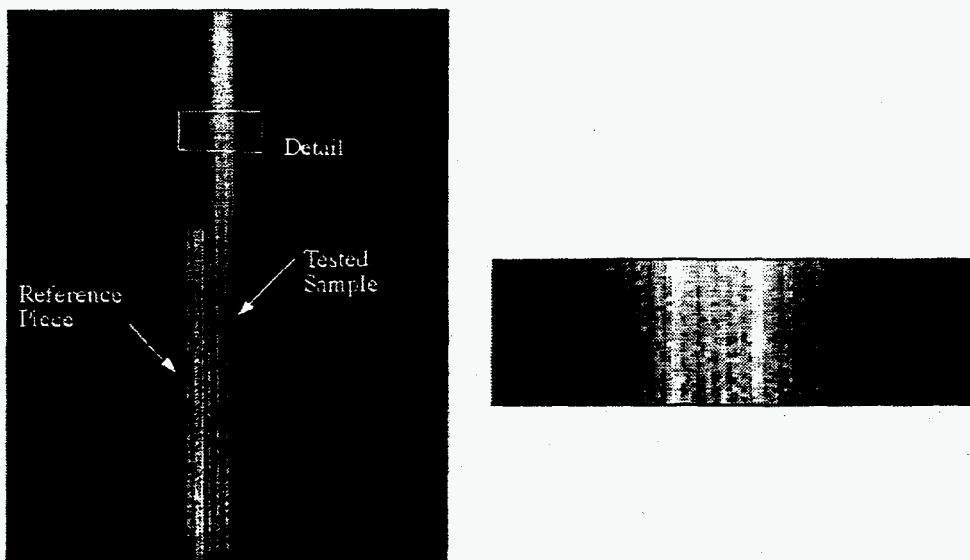


Fig.4 A Digital Radiograph at 90° (on the left) for the Graphite-Polyimide sample and a detail is shown (on the right) with a magnification factor of 4.

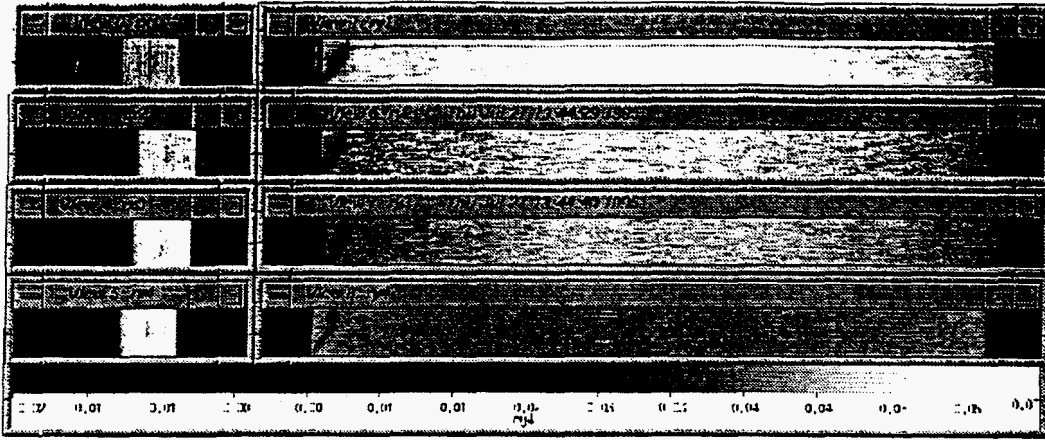
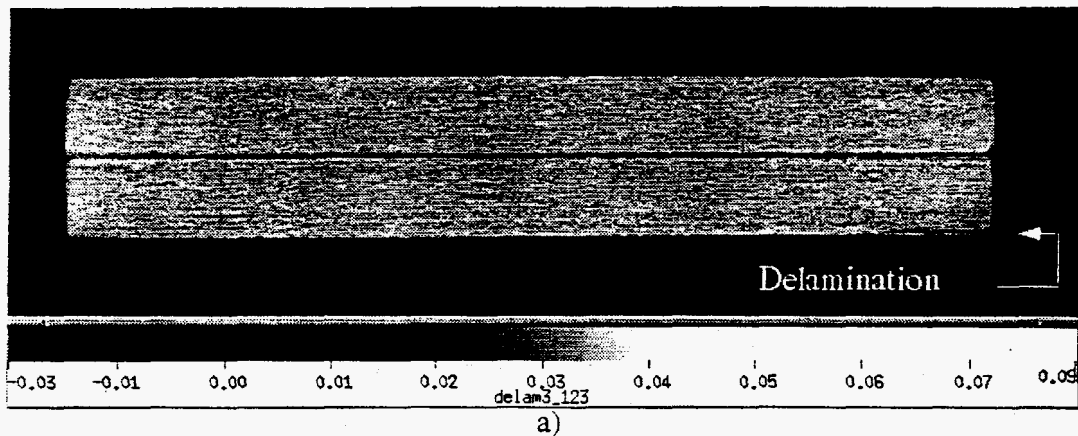


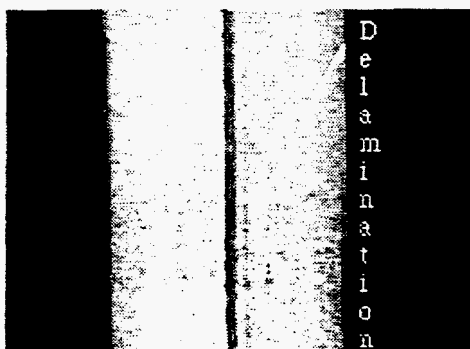
Fig.5 Crack development as a function of the tensile-compression test(the load increases from the top to the bottom) .

We were also able to visualize all the different orientations of the fibers in four contiguous sections along the x-z plane, even if the capability of the system doesn't allow us to solve fiber by fiber (whose diameter is about $6 \mu\text{m}$).

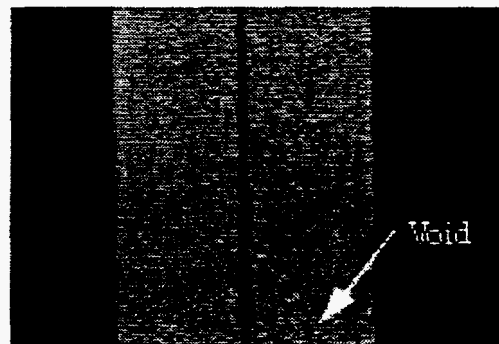
A histogram (Fig.7-a) related to an extracted region containing an inclusion shows clearly the different peaks corresponding to the background, the scotch tape used for binding the two sample pieces, the reconstructed region inside the sample, the two high-density regions (see Fig. 4) identified from the beginning in the untested sample and the small inclusion.



a)



b)



c)

Fig. 6 After the last loading test several defects appear : a) some delaminations in a cross section; b) a transversal section shows the vertical extension for one of the delamination; and c) a void.

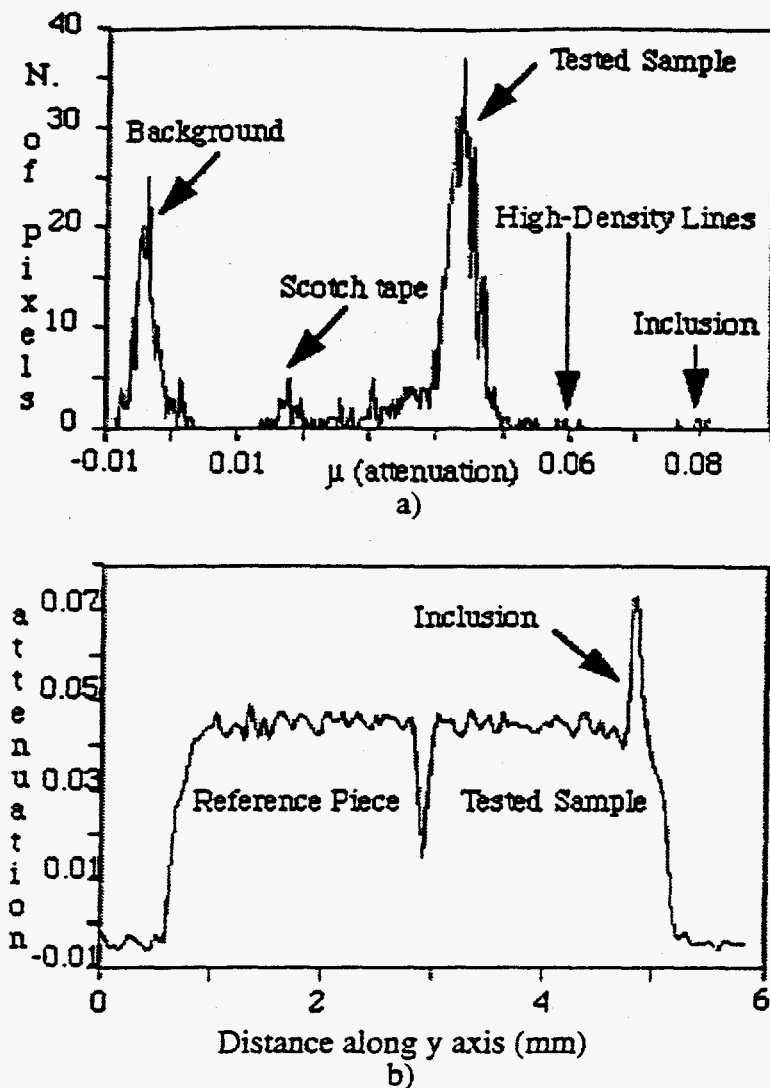


Fig.7 = A histogram (a) and 1D profile (b) characterized by an inclusion .

The attenuation corresponding to the inclusion is shown in Fig.7-b. The ratio between the inclusion and the mean attenuation for the sample is about 1.6. We noticed that the attenuation inside the two samples (reference and test) isn't regular. This fact is probably due to either the different orientation of the fibers in each ply, the matrix or both.

V SUMMARY

In the present work, we studied six different Carbon-Polymer Composite samples with different matrix composition, geometric characteristics and static or fatigue loaded to failure. As above mentioned, five of the six considered samples were only studied after the loading test. Their previously unknown situation is a factor of uncertainty with respect to the origin of some observed features inside them.

The study of the Graphite-Polyimide sample allowed us to ascertain some interesting characteristics. First of all we had the possibility to locate from a spatial point of view the defects inside the sample, to establish the moment they appeared, their evolution along the loading test and the relationship between such defects and the mechanical characteristics of the material itself. The use of different investigation techniques permitted us to look at several different aspects of the same phenomena.

One of the main purposes of this study was to determine if the X-ray CT Non Destructive Inspection method can be used to evaluate composite materials characterized by a low-contrast sensitivity between fibers and matrix components without the use of any

enhancing solution and with fiber dimensions of the order of few microns. We also showed that x-ray data are different in physical characteristic identified and complementary to other NonDestructive methods. The X-Ray CT results obtained are encouraging with respect to the study of thin Carbon-Polymer Composite Materials.

VI FUTURE WORK

Our interest is actually focused on studying the performance of other LLNL CT systems applied on the Carbon-Polymer composites, using linear and area array detectors, different energy values and resolution, to further investigate other methodology in order to study the defects and various features for such materials. An interesting test will be the possibility to compare the results obtained studying the same phenomena with different NonDestructive Techniques (e.g. UT and IR) in order to evaluate the specific capabilities of the proposed method. The development of a CT Scan System in situ for the acquisition in real time both of Digital Radiographs and CT Projections during loading tests using typical load-size sample may represent an important improvement of the proposed method along the way of a more complete knowledge of the behavior of such materials from a spatial and a mechanical point of view.

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