ABLATION MEASUREMENTS IN THICK COMPOSITE MATERIALS

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INTRODUCTION

X-ray Computed Tomography (CT) has been used for several years as an inspection technique in the rocket nozzle industry, but only recently has it been examined as an instrumentation technique. Boeing pioneered the use of CT in the rocket nozzle industry when it began to use medical CT systems to evaluate exit cones [1]. Recently, Boeing has conducted experiments using CT to instrument high temperature testing of composite parts. We have successfully conducted real-time tests of ablating thick (>25 mm) carbon phenolic (C-P) material using a high framing rate CT system. Ablation, charring, and thermal cracking can be quantified over the time of the burn, at scan times as short as 0.06 sec as allowed by special purpose medical systems developed to image the human heart.

High performance composites are currently finding applications throughout the aerospace industry for extreme temperature and/or stress environments. Ablation for high temperature nozzle applications is one particular example. The rate of degradation of the composite material during a burn is critical for the overall performance of the nozzle but depends on complex physical interactions, including radiation transport, convection and chemical reaction rates. While the design of high temperature structures uses thermal analysis codes to predict material performance (ablation, pyrolysis and charring) as a function of time, verification of the accuracy of the complex analytical models used by the codes can be quite difficult. Boeing is now investigating the use of CT as a tool for high temperature analysis of composites. High framing rate ("Real Time") CT was used to monitor the ablation of C-P test pieces while they were being subjected to heating from an oxyacetylene flame.

BACKGROUND

The complete thermal response analysis of an ablating system is rather complicated. The results of such analysis depend upon many interrelated factors. Each phase of the analysis chain is subject to uncertainty. System design often entails spacers, gaps, bonds, and multimaterials that have different ablation characteristics. When ablative materials such as C-P are heated above a certain temperature, thermochemical decomposition results. Char begins to form, and pyrolysis gases are generated. As the material continues to be heated, the pyrolysis zone moves toward the interior and the char layer thickens. Ablation of the char layer occurs as the surface temperature exceeds certain values [2]. The phenomena in both the pyrolysis and char zones are quite complex

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and not fully understood. Pyrolysis of the matrix causes a reduction in the material density [3]. The pyrolysis gases form microscopic pores which grow and allow the flow of the gases toward the surface. Reactions and energy exchanges between the char matrix and the passing pyrolysis gases can occur. The char can become further eroded, or the char passage can become narrow because of coke deposition [4]. Cracks may form allowing escape of the pyrolysis gases, thus altering the thermodynamics of the gas-to-char energy transfer.

The mathematical models do not accurately represent these actual physical phenomena. Measurement of ablation and internal decomposition can be very difficult, and access to the part has often been limited to pre- and post-test evaluations.

Instrumentation has been developed in the last 15 to 20 years to measure ablation rates for reentry vehicle nosetips. This instrumentation includes optical, acoustic, resistive, and radiative gauges [5]. These techniques provide only very limited thickness data at selected locations on the part. In addition, with the possible exception of the ablation source radiation gauge, these techniques are not applicable to the rocket nozzle environment [6]. Other techniques which are now being explored are flash CT and real time radiography.

The objective of this work was to demonstrate the feasibility of using high framing rate CT to monitor the ablation of carbon fiber based composites.

TEST SET UP

Because our goal was to demonstrate a concept, a rigorous quantitative test was not attempted. Instead, a relatively simple demonstration was designed which would contain the basic ingredients of any ablation test.

An oxyacetylene torch, providing temperatures above 2500^{o} F, was chosen as the heating source. The composite chosen for the demonstration was a carbon phenolic made by Fiberite and donated to us by NASA Marshall Space Flight Center.

The tests were conducted at Imatron, Inc. in South San Francisco, CA on their C-100 Ultrafast CT Scanner. In this system, an electron beam that sweeps along fixed target rings replaces the mechanically rotating x-ray tube of conventional medical scanners. This unique method allows for very high speed scanning. At present, up to 17 scans/sec for a 256 X 256 image, or 9 scans/sec for a 512 X 512 image are possible. The current RAM storage permits forty consecutive scans to be acquired without pausing. Both the speed and the storage are limitations of the computer and not the electron beam technology.

Several pre-tests were conducted without CT instrumentation to determine the important test variables. These pre-tests were done under a fume hood at Boeing's Radiation Effects Laboratory Machine Shop. A C-P sample 1/4 inch thick was hung from a load cell (connected to a chart recorder) and heated from the bottom with an oxyacetylene torch. The time necessary to burn a hole through the sample allowed us to estimate the rate of erosion we could expect when heating the thicker blocks and cylinders (approximately 3 inch on a side) in the actual test. The load cell was intended to give us a quantitative measure of the material loss throughout the burn. Oscillations caused by the convection currents around the burning part rendered the data useless. However, we were able to make a qualitative measure of the amount of smoke produced during the burn. This information helped us design our method of heat and smoke extraction for the actual test. A second pre-test was conducted with a C-P sample with the same dimensions as the ones which would be heated in the CT system. In this pre-test, we tested the thermal board insulation box and heat extraction system which is described below. Slight modifications to the cooling duct to allow heat dissipation were made as a result of this test.

The configuration chosen for the actual test within the CT machine is shown in Fig. 1. This orientation allows progressive ablation to be observed in the CT scan plane without having the torch intersect the scan plane as well.

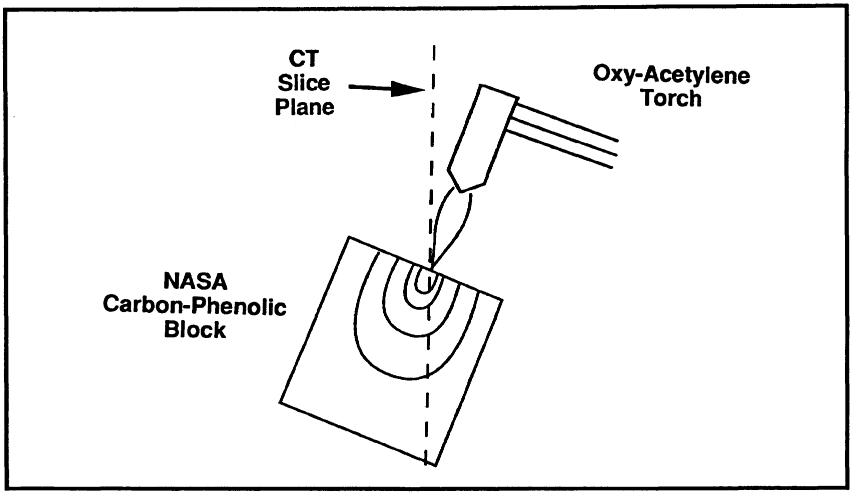


Figure 1. Real Time CT ablation test configuration.

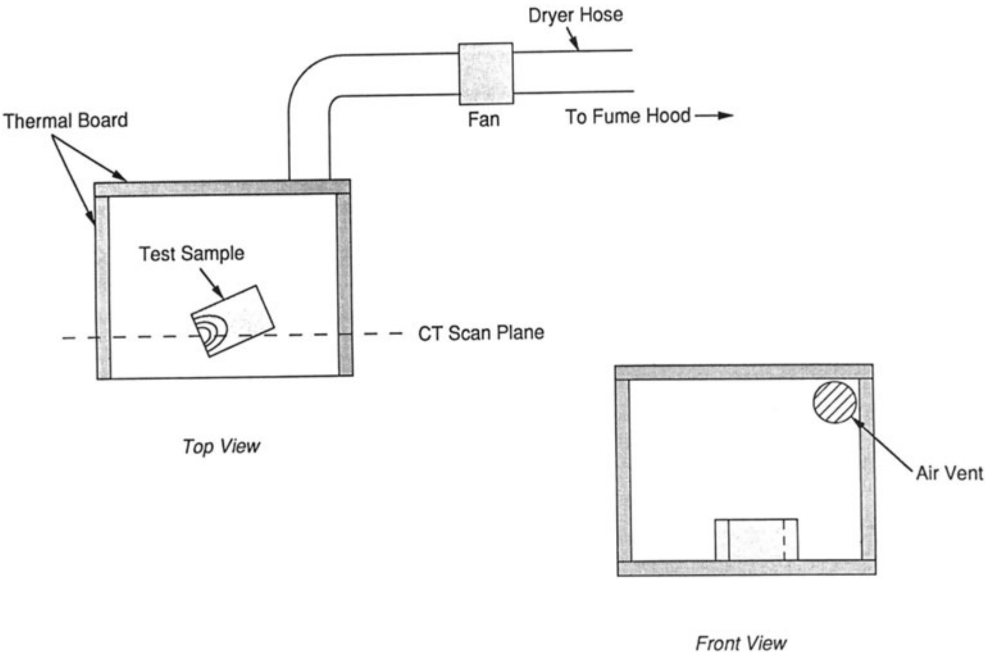


Figure 2. Test chamber for Real Time CT ablation studies.

Two primary considerations on this test were the safety of the operator and the safety of the CT equipment. The operator's safety was the easiest to resolve by wearing a lead apron, lead gloves, and welding goggles. Protection of the CT equipment required that we design and build a special test chamber. A top and front view of the final design of this chamber is shown in Fig. 2. It was designed to protect the CT system from excess heat by insulating the test and drawing away the product gases. The test chamber was made of Magnaboard, a thermal insulating board that can withstand continuous exposure to a flame of 2600^{o} F. The chamber has three sides plus the top and the bottom. The back end contains a 3 inch hole for the escape of exhaust gasses, and the front is open. This chamber was placed on top of a bigger piece of the insulating board to protect the patient table. The exhaust gasses from this test were drawn through metal dryer venting for the first 50 feet from the test chamber using a 60 cubic ft./min. exhaust fan. The gasses were blown through another 100 feet of standard plastic dryer vent hose which emptied into an exhaust hood. The metal dryer venting was used first since it could not only stand the higher temperatures but bends in the hose increased the heat transfer from the gasses to the hose wall, thus cooling down the gasses substantially before they encountered the plastic section of the hose. Trial runs were made before the system was put near the CT equipment and the temperature of the gasses at the outlet of the exhaust fan did not exceed 150^{o} F.

A block of the insulating board with a semi-circle cut out at the top was used as a stand for the torch tip to keep it from being moved during the burn. The test articles had been machined into cylinders approximately 3 inches in diameter and were placed into an open ended box made from the insulating board. Before a sample of C-P was burned, the sample was placed inside the test chamber in the approximate position and the torch was placed in its rest and digital radiograph (DR) was taken. The DR was used to position the test article and torch for the best slice. The scan plan was then developed. This covered the time delay from the time the torch's flame first touched the part until scanning began and included the time between slices and the number of slices to be taken.

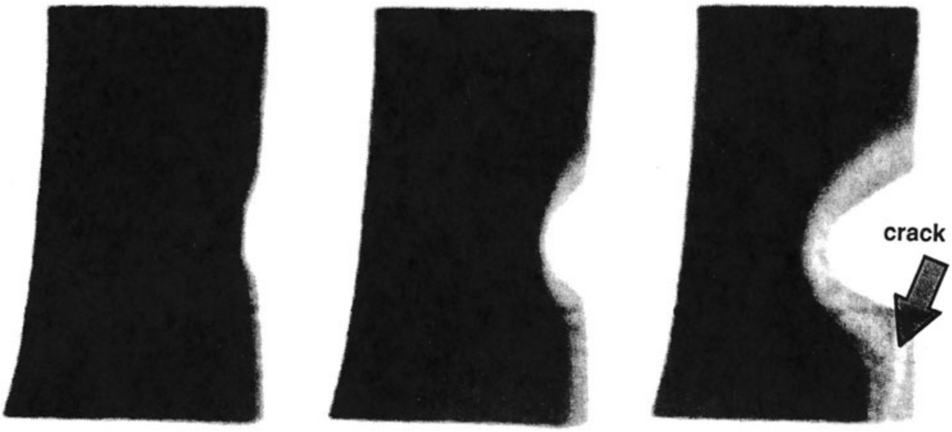
RESULTS

A series of CT images were produced for each C-P sample tested. These images provided both qualitative visual information about the ablation process and an immense amount of digital density data. An example of what can be done with such high framing rate CT information is given below using scans taken on one C-P sample.

Selected images of the ablation of block #7 are shown in Fig. 3. One scan every 0.8 seconds was taken, 12 to 25.6 seconds into the burn, and one scan every 2.5 seconds, 26.5 to 82 seconds into the burn. Thus, a total of 40 scans (the maximum allowed on the system's RAM) were taken over the time of the burn.

Images of this type were able to reveal several features of the ablation process. The density gradation in front of the eroding material is clearly visible in the images. Two small bands can be observed which apparently represent fronts separating different temperature related reactions. Figure 4 is a graph of CT density versus time for a selected region of the C-P block. Small steps can be observed on this curve as the separate chemical reactions associated with the burn pass through this particular area. These steps produce distinct density bands in the images which can be observed in the original display but are lost in the reproduction. Another feature that is immediately recognized from the images is the increase in density in the material adjacent to the eroding front. Figure 5 is a graph of CT density versus time for a selected area where this phenomena is occurring. To the best of our knowledge, these scans mark the first time that these density changes in ablating C-P have been observed during an actual burn. In fact, until this point in time, it has not been known if the density upturn previously measured at the completion of a burn happened during cooling or during the burn itself [7].

Thermal cracking was also observed in this specimen. A crack became visible late in the burn, and widened upon cool-down. However, we were able to select data in the



T = 12 sec T = 32 sec T = 82 sec

Figure 3. Selected CT images of ablating carbon phenolic block.

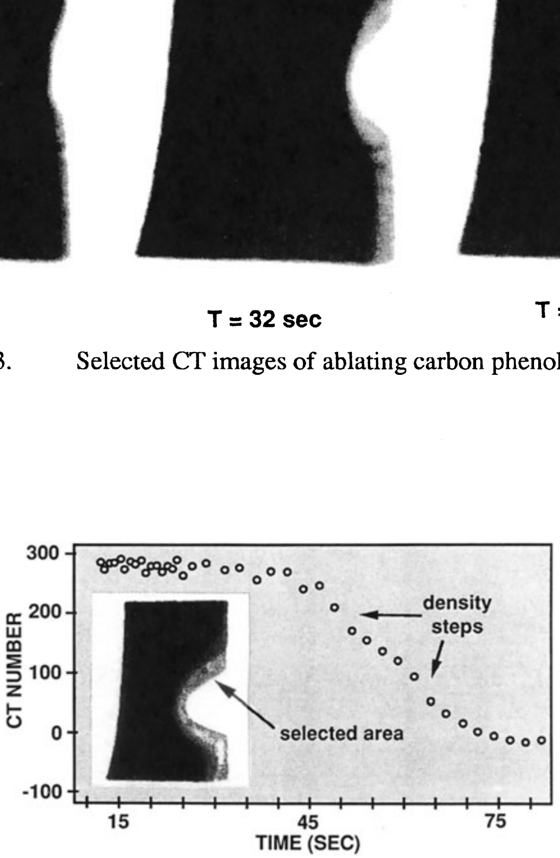


Figure 4. CT density versus time for selected region of C-P block. The small "steps" in the curve are caused by separate chemical reactions associated with the burn.

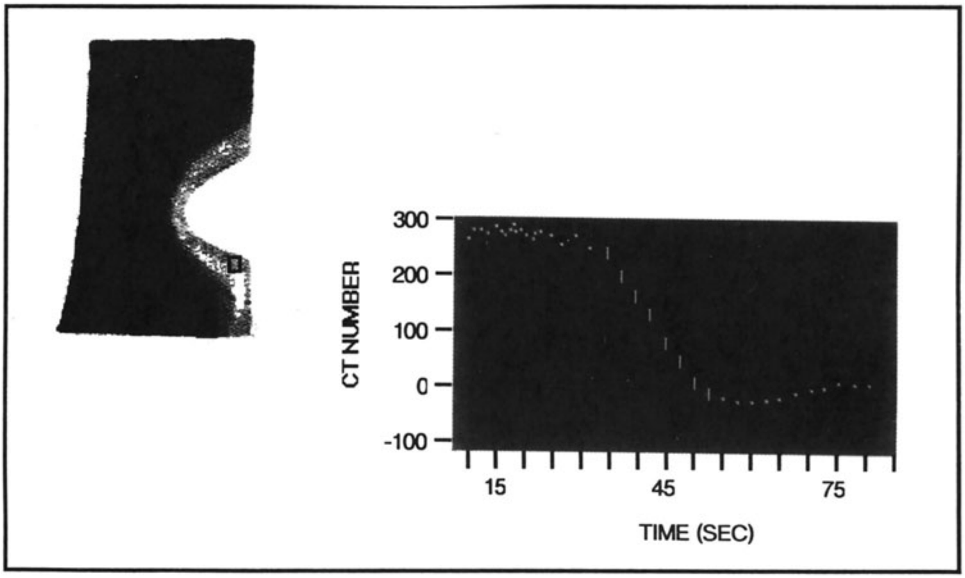


Figure 5. CT Density versus time for a selected region of ablating C-P block. The density at this location actually drops and then increases as the burn passes.

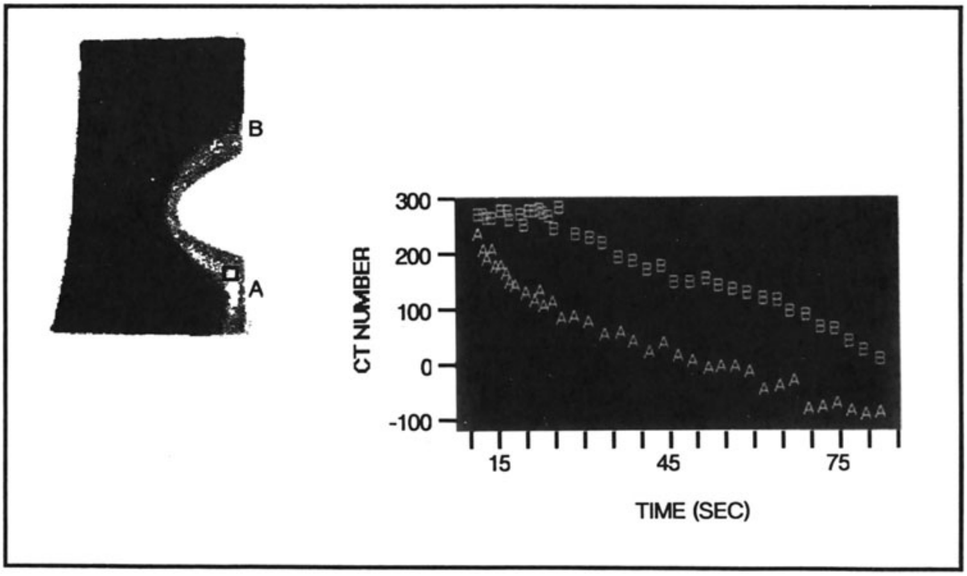


Figure 6. CT density versus time for two comparable areas of an ablating C-P block. Region A was chosen at the location of a crack. Region B was chosen at the same relative distance from the burn front, but on the opposite side.

area of the crack and determine that it started much earlier into the burn. A major benefit of Real Time CT is that it gives one the capability to go back in time and analyze any area where data was taken.

Figure 6 is a graph of CT density versus time for two comparable areas on the sample (shown as regions A and B). Region A was chosen at the crack itself, and region B, in a similar area where no crack formed. The growing crack produced a density change in the pixels at A long before it could be observed in the image.

There are many other diagnostic software tools available for CT images which will directly assist in material property and state analysis. For example, the following tools would be very useful:

1) Statistics such as the mean, standard deviation, error in the mean, and number of pixels can be shown for any chosen image area. For the multiple images from Real Time CT, these statistics can be shown versus time, providing for time-density analysis.

2) A histogram which plots the number of pixels in the CT image versus the CT number will provide a type of fingerprint of the image. Real Time CT will give a measure of the change in this histogram over time.

3) Point-to-point distance measurements allow for static and real time dimensional analysis.

4) Multiplanar reformatting allows image display and processing in a variety of views.

5) Images can be aligned and subtracted from one another. An application of this feature is the measurement of small strains or material recession (on the order of a pixel size).

6) Contiguous scan plane imaging provides volumetric data.

7) Combining of successive multiple images allows for real time playback in movie format.

All these real time CT capabilities can support thermostructural analysis of ablating materials by providing information into the process kinetics. These results show that Real Time CT can be an effective tool for ablation measurement and analysis. We recommend that Real Time CT be considered for standard instrumentation for ablative rocket motor material testing and analysis.

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