ANALYSIS OF CERAMIC COMPOSITES BY 3D MICROTOMOGRAPHY

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ABSTRACT

In the first part we describe the use of 3D X-ray microtomography for the analysis of density distributions inside and between cold pressed green state whisker reinforced Si_3N_4 / Si_3N_4 bending test specimens. The differences between a changing content of whisker volumes (0 to 35 %) show a sharp density reduction between 10 and 15 %. This agrees with previous research which shows that maximum density is achieved between 10 and 15 % whisker loadings. Since density is directly related to fracture strength, microtomography may be useful as strength prediction.

In the second part we describe the use microtomography combined with image processing, especially a 2D FOURIER transform, for the analysis of cloth direction distributions in multidirectional reinforced chemical vapor infiltrated (CVI) SiC / SiC test components. The SiC fibers were NICALON™ and were oriented 30, 60, and 90°. In laying up the cloth, orientations may not be maintained and this impacts the mechanical properties. Use of microtomography with image processing may allow mechanical property variations to be established.

INTRODUCTION

The use of 2D or 3D computed tomography for the inspection of ceramics was reported earlier [1 - 3]. Often, however, the primary aim of these works was to find flaws or irregularities and according to the nature of ceramic materials, microtomographic systems were used [4 - 5]. In terms of spatial resolution the today's possibilities are in the region of 50 μm or even less. With 3D cone beam tomographs this resolution is also to be achieved in the third direction, however, not without more sophisticated and therefore time consuming algorithms and only for a very small aperture [6]. According to the setup of 3D cone beam tomographs in terms of contrast discrimination, 2D tomographs are superior for the moment. The coming

design of data acquisition and especially computing hardware could change this situation. The recent works in 3D NMR imaging of ceramics [7] could emphasize this effort.

METHODS AND MATERIALS

3D Computed Tomographic System

Measurements were carried out on a third generation 3D cone beam computed tomographic system which schematic setup is given in figure 1. The source is a 160 kV microfocus X-ray tube (IRT HOMX 161) with a spot size of 10 to 20 μm diameter. The detector system contains of a trifold (9, 6, 4.5 ") image intensifier and a Sony XC 57 512 x 512 CCD-camera (8 bits). Using a third generation CT-method this setup e.g. would allow a pixelsize of 50 μm for an object of 20 mm diameter (geometric magnification 10 x). For the motor control and the data acquisition we used an IBM PC/AT with an accelerated frame grabber board. The next calculations up to the rough images were made either on a VAX 8700 or on a SUN 4 workstation. For the image processing and display, finally, we used a Macintosh IIfx.

Figure 1. Schematic setup of the used 3D tomographic system.
For reasons of clarity, the aperture is shown too large.

Specimens

The first set consisted of Si_3N_4 / Si_3N_4 whisker reinforced bending-test specimen (about 50 x 6 x 3 mm) in green state. The whiskers had a diameter of 0.6 to 1.0 μm and a length of about 10 to 15 μm . Their distribution was more or less randomly but certainly influenced by the cold pressing manufacturing process. The volume content of the whisker loadings ranged from 0 to 35 %. For this preliminary work, where we had to learn how to handle these delicate samples, we took already broken ones. But the aim of this method certainly is to do this examination before mechanical tests are made.

The second set, however, consisted of multidirectional cloth reinforced SiC / SiC composites. These samples have been produced at Oak Ridge National Laboratory, using a chemical vapor infiltration (CVI) technique which combines the thermal gradient and pressure gradient processes [8]. The SiC fibers were NICALON™ and were oriented 30, 60, and 90°. The objects had a shape like a disk with a diameter of about 45 mm and a height of approximately 12 mm.

In each case the materials of the matrix and of the reinforcement are the same, so that resulting gray levels are pretty much related to the density.

RESULTS

Si_3N_4 / Si_3N_4 Samples

For these samples the gray level differences between different whisker loadings was examined. Figure 2 shows one slice of each sample. Already here a sharp reduction between 10 and 15 % is clearly visible.

Figure 2. Differences in gray levels for different whisker loadings.

To save calculation time, the images were reconstructed with 256 x 256 pixels (each with two samples), so that a pixelsize of 40 μm is resulting.

To avoid edge effects, the gray levels were measured in the central region of the slices, covering about 50 % of the total area. The standard deviation for one slice is always about 3 to 4 grays, the differences of the mean values of different slices of the same sample are about the same. No differences of these statements are to be found between the unloaded (0 %) and the loaded samples. The reduction between 10 and 15 % is significant; the lowest gray level found in all slices of the 10 % sample is still 15 grays higher than the highest of the 15 % sample (figure 3).

Figure 3. Mean gray levels over seven slices, highest and lowest mean value of the single slices and maximum and minimum gray level of single pixels for 10 and 15 % (dotted line).

SiC / SiC Samples

These objects were much larger and in this case the best possible pixelsize would have been about 100 μm . For the examination of the reinforcement structure, however, a pixelsize of about 200 μm was sufficient. Figure 4 shows a slice through two different samples (261 and 262). Four effects are recognizable: in both cases the density in the center of the samples is higher than at their periphery (the usual beam-hardening effect would show just opposite characteristics). From the gray level curves through the center of the objects, it is to assume that the density gradient is pretty linear but different for both samples. The same curves also show a support structure due to the reinforcement.

Figure 4. Variations of the density inside of two SiC / SiC samples.
Each tomogramm contains 256 x 256 pixels, the air is cut.

It is not possible to make statements about the reinforcement structure looking at the slices in the time domain. After a 2D fast FOURIER transform (FFT), however, this structure becomes clearly visible (figure 5). As the height of each slice is about 1 mm, all six directions (30, 60 and 90° and perpendicular to these, respectively) are represented. In the frequency domain they are represented as points at a corresponding frequency, in this case 1.5 mm per cycle. As figures in the frequency domain are radially symmetric, a total of twelve points is to become detected.

Figure 5. 2D FFT of a slice; the points represents the reinforcement structure at about 1.5 mm per cycle, the upper and lower part of the image is cut.

Examining the structure of the reinforcement using this kind of image processing, several effects are to be found. The radial extension of the points and therefore differences in the directions of the cloths are not always the same; for some slices even separations in one direction are found (figure 6). To be able to measure these radial extensions equally, the images were transformed into binary versions.

Figure 6. Two basic types of 2D FFTs; left side: normal reinforcement, difference always about 30° ; right side: separation in one direction.

The results of these measurements for seven slices are given in figure 7, the direction of 0° is arbitrary as it is influenced on the orientation of the object on the CT system. The trend of these graphs shows that the directions of the layers are not strictly maintained and that the relation between one direction and its perpendicular direction is not always very strong (e.g. sample 261, directions 60° and 150° , respectively).

Figure 7. Directions of the reinforcement structure of the multidirectional cloth reinforced samples; each slice has a height of about 1 mm.

DISCUSSION

In the first examination a clear density reduction which for similar materials is also reported by other authors [9] was found using 3D microtomography. Inside each sample certain density variations can be found which are due to the cold pressing manufacturing process. Because of that and of small density differences we can clearly distinguish between a high density region (0 - 10 % whisker loading) and a low density region (≥ 15 % whisker loading) but only hardly inside of these. The clear difference between measurements on the 10 % and the 15 % samples leads to the statement that there are no whisker enrichments with dimensions nearly the examined voxel (1000 x 40 x 40 μm).

In the second examination it was possible to find variations in multidirectional cloth reinforcements in several heights of the samples. Even if the radial accuracy is not more than 5°, differences are clearly shown. The demonstrated weak relation between perpendicular directions of the reinforcement let us assume that certain cloths are under residual stress which would impact their mechanical properties. For flat objects this technique can also be used directly with radiographic images, for larger object, however, the information in the third dimension would be missing and separations or variations would not become detected.

CONCLUSIONS

X-ray microtomographic systems may not only be used for inspection of ceramics with the aim to find flaws or irregularities but also as an aid for process control. Even if the contrast discrimination of a todays 3D system is lower than that of a 2D system it is suitable, especially for the second purpose.

In two examples we have shown the use of 3D microtomography for strength prediction and, combined with sophisticated image processing, for establishing mechanical property variations.

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