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X-ray tomography characterization of density gradient aerogel in laser targets

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Abstract. The low-density solid laser target characterization studies begun with the SkyScan 1074 computer microtomograph (CMT) [1, 2] are now continued with higher resolution of SkyScan 1174. The research is particularly focused on the possibility to obtain, control and measure precisely the gradient density polymers for laser target production. Repeatability of the samples and possibility to obtain stable gradients are analysed. The measurements were performed on the mm-scale divinyl benzene (DVB) rods.

1. Introduction

Low-density micro- and nanostructured polymers – foams and aerogels correspondingly, are widely used in high-power and high-intensity laser physics. However, structures with smooth and known density profile were not yet achieved as a stable technology and would be preferable, for example, for Rayleigh-Tailor instabilities mitigation, laser energy smoothing, laboratory astrophysics studies and as a substitute for step-density targets to generate plasma profile close to the one of the corona of compresses shock-ignition target [3].

The inner structure of an aerogel consists of thin polymer fibres (or walls for foams) forming an open-cell quasiperiodic microstructure. The mean density understood as averaging the wall/fibre solid density multiplied by the solid elements volume then divided by the aerogel volume no less than several pore sizes. Experimentally the density of a solid low-density structure is mainly measured by weighting. This only provides the overall average density of the sample and gives no information on spatial changes.

Wet gel structures with density gradients were examined in [4] and the X-ray microtomography diagnostics was applied for density profile analysis of semitransparent polymer materials. Here we propose and perform precise characterization of mm-size divinyl benzene (DVB) aerogel which is close to laser target scale and requirements.

2. Samples

The low-density divinyl benzene (DVB) rods are chemically synthesized as gel structures inside capillary glass tubes then subjected to solvent exchange. Finally the samples are dried in the Polaron®

critical point dryer (CPD dryer). The height of the rods varied from 3 to 10 mm with the diameter of around 1 mm. Here chemical synthesis was fulfilled for a large bath of similar samples, so as to model gradient targets mass production. Then CMT measurements were performed to yield certain statistical data. Method of production of these density gradient foams is described in previous publication [1].

Self-standing DVB aerogel cylinders display the density gradient as visibly differing optical transparency in low- and in high-density regions. Inner microstructure and surface were examined by SEM in [1]. The material turns out to be homogeneous on the scale of several microns which increases precision of X-ray diagnostics with the voxel dimensions of around 10 um.

Besides density-gradient samples which are the main goal of our work, we needed the constant-density samples for calibration of density measurements. The procedures of chemical synthesis, solvent exchange and CPD drying for constant-density aerogels, excluding the conditions to produce gradient, remain strictly identical with gradient-density samples.

3. X-ray microtomography for density measurements

X-ray tomography is a non-destructive penetrative method for volumetric visualization and material's characterization based on the absorption properties of the material. Mathematically reconstructed flat X-ray images show absorption in each spatial point of the sample:

$$I_{image} = I_{initial} \cdot e^{-\mu_{abs} \cdot x},$$

where I_{image} intensity is read from CCD as grayscale index, μ_{abs} – absorption coefficient for the specific material, x – optical path through the sample.

3.1. SkyScan 1174

The microtomograph SkyScan 1174 used in this work allows the maximum resolution of 6.9 um per pixel. However for DVB rods scanning a 17.5 um resolution was chosen to fit the view window. Tungsten cathode of the X-ray tube provides broad-spectrum X-ray radiation in cone beam geometry. The sample is placed onto a rotating table B (Figure 1a) on the way of X-rays to form multiple flat absorption images (Figure 1b) by CCD camera. The rotation step could be as small as 0.6 degree. The set of images is reconstructed to 3D model using special software by Feldkamp [5] algorithm. The reconstruction gives a number of horizontal cross sections with absorption recalculated to internal computer tomography units (CT number) in each voxel of each cross section. The obtained CT number in each voxel corresponds to grayscale index of the 2D image and thus through X-ray absorption in voxel to the local density.

Figure 1a. SkyScan 1174 inner view. A – X-ray tube window; B – rotating table with a DVB sample; C – *Be* filter of the CCD camera window.

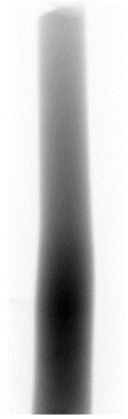

Figure 1b. Flat X-ray image of DVB rod.

3.2. Absolute calibration

The reconstruction with the built-in SkyScan 1174 software does not give information on absolute density of the sample. However one can only analyse comparatively the exposed density-gradients with the obtained statistics on CT numbers. This is not enough for purposes of laser target characterization. Therefore the absolute calibration of the CMT measurements was performed using the samples with initially known average densities. The average densities were calculated from the solution concentration and checked by weighting.

For the calibration samples of pre-known density separate CMT scanning was done. Averaging over each horizontal cross section we got a distribution of CT numbers over the height of each sample (Figure 2). CMT-measured density variation via height for cylindrical samples of 4 constant nominal densities are plotted in solid line, average CT numbers of constant-density derived from real curves are plotted in dash lines.

Averaging was carried out by the following method: the cross section was scanned for non-zero pixels and the remaining objects were eroded by several pixels to minimize the boundary artefacts. Then, the resulting group of pixels was used to calculate the average CT number.

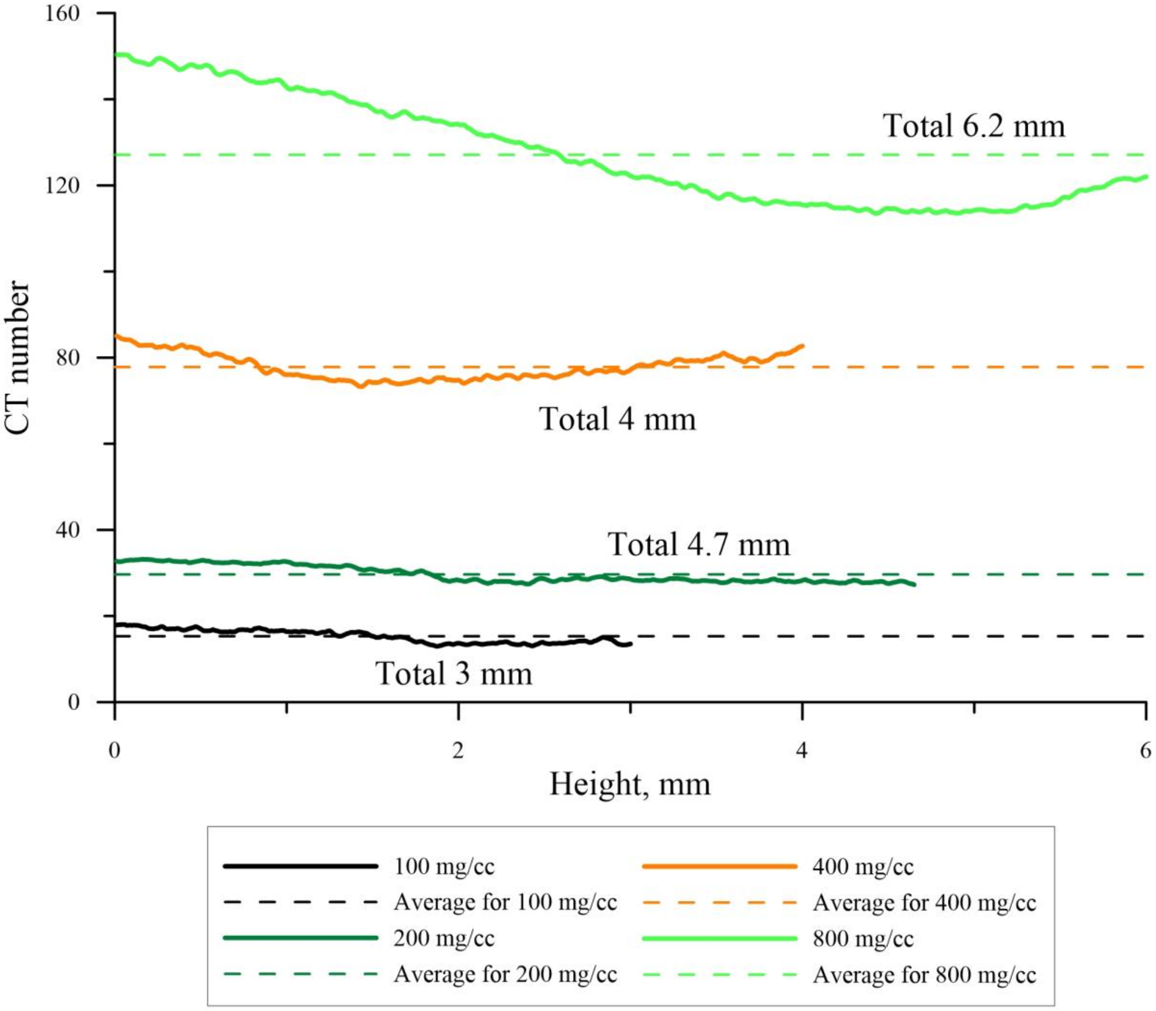

Figure 2. Density variations (in units of CT numbers) over height of the sample for the constant nominal density DVB rods. Dashed constants are calculated average values.

Absorbing properties of materials in X-rays strongly depend on the nuclei charge Z. Thus for each atomic composition the calibration is to be performed independently.

The calibration curve for DVB aerogel is shown in Figure 3 and defines the conversion coefficient from CT numbers to density of 0.16 ±0.01 units/(mg/cc). The aerogels with density gradient are further analysed in terms of reconstructed and recalculated density.

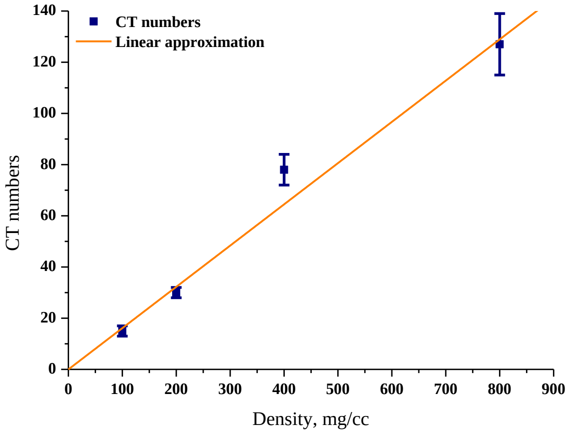

Figure 3. CT numbers to density calibration curve.

4. Density gradient in DVB aerogel rods: realisation and measurements

The density gradient DVB rods were examined on SkyScan 1174 with the same scanning parameters and resolution as the calibration samples. The data was reconstructed with the built-in software, averaged CT numbers were calculated over each cross section and then the obtained distribution was recalculated in terms of density in mg/cc.

To diminish the noise and for stricter curve analysis the density function was plotted after application of moving average filter over 11 points (Figure 4a left, Figure 4b left). To visualize the density changes over the height 1st derivative was calculated and is further referred to as gradient function or gradient. The gradient was plotted and analyzed with Fast Fourier Transform (FFT) filter over 15 points to cut off the high-frequency noise.

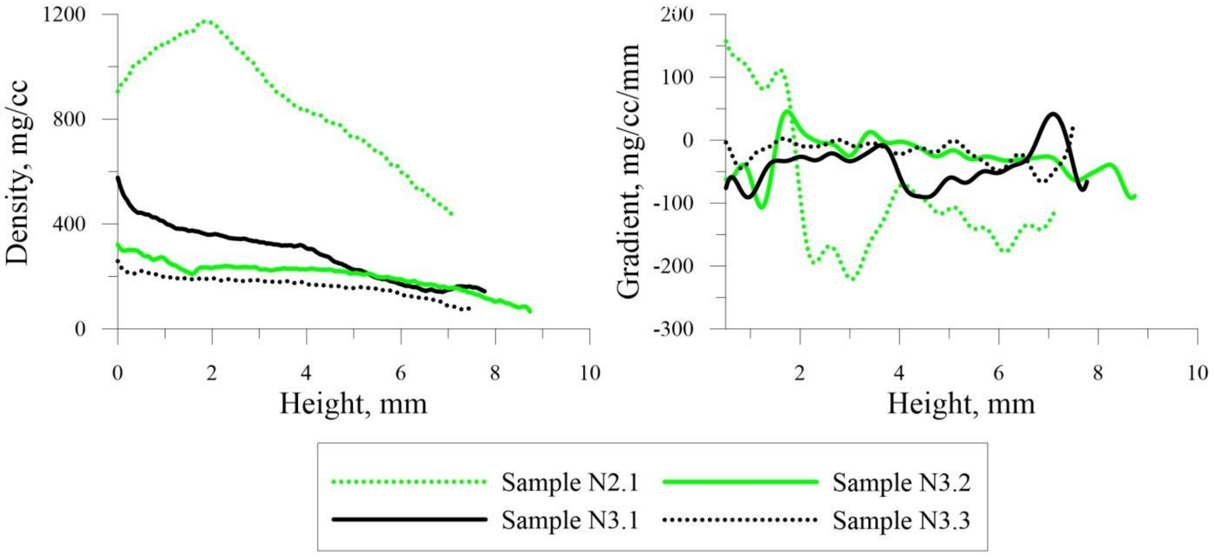

Figure 4a. Density (left) and gradient (right) function via height for old samples.

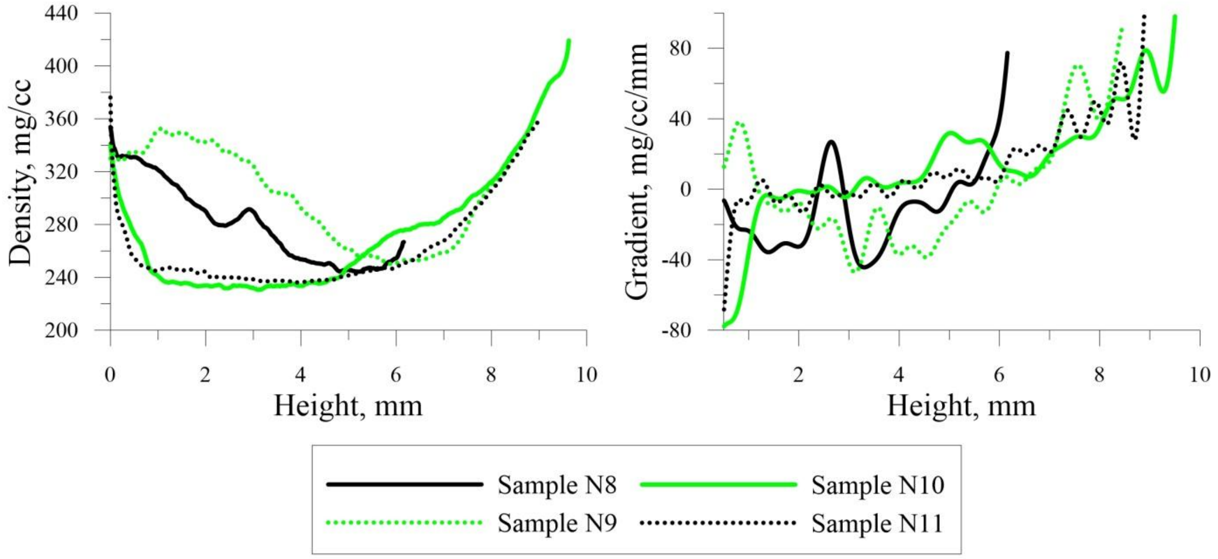

Figure 4b. Density (left) and gradient (right) function via over height for new samples.

We used 2 batches of samples with almost two year gap in the time of fabrication. The Figure 4a refers to the earlier done samples, the same as described in [1]. After 2 years of storage they were measured once more, now together with newly fabricated samples of Figure 4b for comparison, production studies and more profound characterization with next-generation CT-scanner SkyScan 1174.

Comparing the old (Figure 4a) and the new (Figure 4b) samples we found no significant difference. The samples are stable in time and can be stored and transported.

For previous results [1] we used practically manual calculations (averaging and part of filtering procedures). Thus the precision was less than possible but the route and algorithms for future standard measurements were justified. For example, the observed waves of density to some extend were due to calculation inconsistences. Larger pool of experimental data processed at present with higher accuracy for every gradient sample, provides reliable data and this is still comparable with previously measured results.

On Figure 4 we observe the regions of almost constant average gradient of 10-20 mg/cc/mm on the samples N3.1 (from 2 to 5 mm from the bottom), N3.2 (from 3.5 to 7 mm), N3.3 (from 1.5 to 4 mm), N9 (from 6 to 7 mm) and N11 (from 4 to 7 mm). Hence such gradient is reproducible on the scale of up to 3 mm.

The maximum gradients obtained (sample N2.1 on Figure 4b) are 150-200 mg/cc/mm and are realized within 1.5 mm length.

5. Results and discussion

The microtomography technique was proposed and used in this work for study and characterization of density gradient plastic aerogels. Self-standing mm-scale aerogel rods are a good approach for further analysis of laser targets. Standard procedures are worked out for profound studying of separate samples during technological research and for routine monitoring of cylinder-shape samples.

Obtained and precisely analyzed density and density gradient distributions over height of the DVB rods showed stable gradient regions of up to 4 mm. Maximum gradients of 150-200 mg/cc/mm were observed on 1.5 mm length interval. The average gradient was calculated 20 mg/cc/mm. The gradients are reproducible on the scale of 3 mm although no dependence of the gradient sharpness on the length of the sample was observed.

The DVB samples with density gradient are stable in time (years) and the technique of density gradient aerogel production is promising for laser target development. More importantly, it has been demonstrated that the production method for density gradient foams is reproducible and precise positioning of necessary gradient interval for further isolation is possible and easy with CMT.

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