FULL PAPER – ALIGNMENT STUDY OF CNT VEILS AND THE INFLUENCE ON THEIR COMPOSITES

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ABSTRACT

A methodology was developed to measure the alignment of carbon nanotubes in pre-strained samples of carbon nanotube veils via image processing techniques. The methodology was used on samples of as-received and pre-strained carbon nanotube veils and calculated an increased Herman parameter for the pre-strained sample relative to the as-received. A number of image processing techniques, specifically high and low pass filters, were tested in order to increase the signal of the carbon nanotubes in the scanning electron micrographs, however it was found filtering the micrograph prior did not enhance nanotube signal and did not improve the calculated orientation distribution function.

1 INTRODUCTION

Pre-strained carbon nanotube (CNT) veils have shown promise in producing both CNT papers and CNT composites with unrivalled strength and stiffness as well as improved thermal and electrical conductivities [1, 2]. Many groups have demonstrated improvements in tensile properties of such composites when carbon nanotube alignment in veils was increased. The most common ways to align carbon nanotubes in veils is either by a winding approach, where veils are passed through a set of rollers which act to strain and densify the CNT veil (Figure 1-a) or a mechanical stretching approach where veils are stretched using a constant cross-head speed (Figure 1-b). However these composites are yet to reach their full potential in part due to difficulties in aligning the veils within the composite materials.

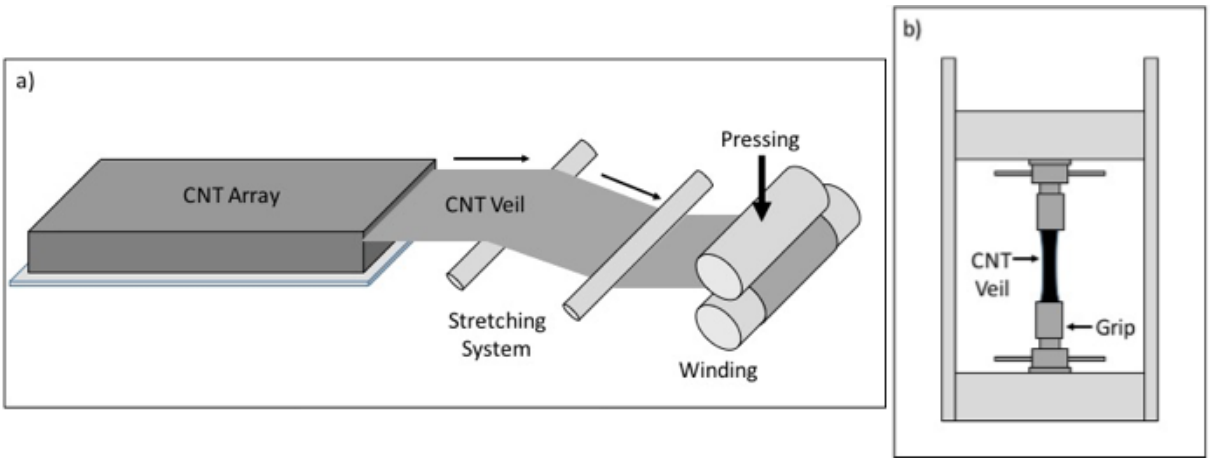

Figure 1 Schematic for straining CNT veils

3 EXPERIMENTAL

3.1 Materials

CNT veils were kindly provided by SiNano (China) and used as received. The CNTs in the veil were multiwall CNTs with a diameter approximately 10-30 nm, 10-15% iron catalyst, density 0.5 g/cm^3. The veils were produced from vertically aligned CNT forests that had been directly grown on predeposited catalyst film. The thickness of the veil was measured using micrometers (Mitutoyo, MDC Lite Digital Micrometer) and was 20μm.

3.2 CNT Veil Alignment

To align the carbon nanotubes in the veil, the veils were stretched using an Instron 4505 screw driven machine used with BlueHill3 software, at a stroke rate of 0.1 mm min^{-1}. The instron was used in combination with optical video gauge. Optical video gauge iMETRUN MG223B PoE E0022522 from iMETRUM Ltd (GB) using a iMETRUM general lens. The video gauge was triggered to work in sync with the BlueHill3 software. The video gauges lines of reference were dots made using a white tip-ex pen (Figure 2). For the analysis CNT veils were strained to 10%.

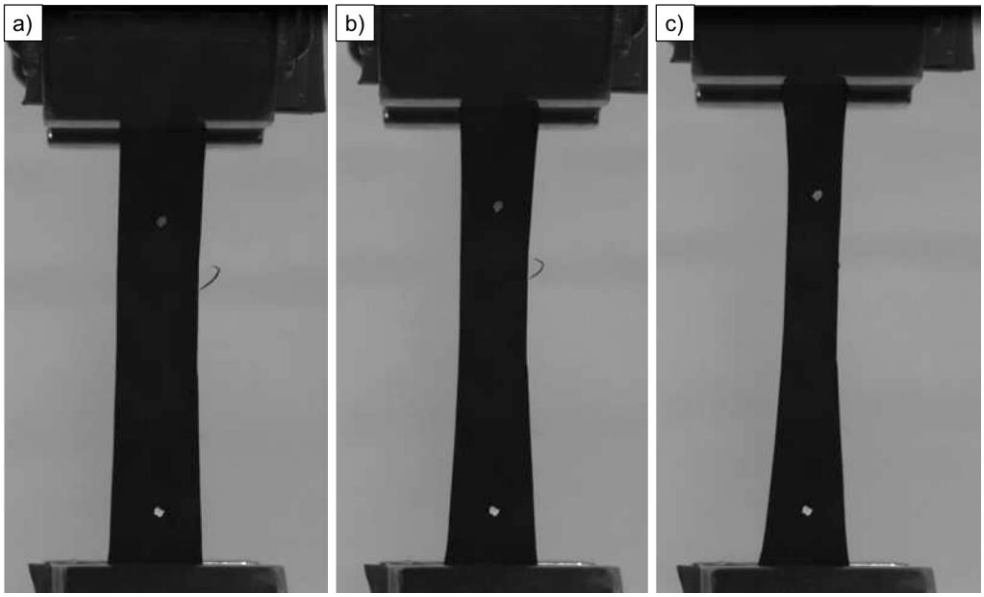

Figure 2 Straining of CNT veil

3.3 SEM Imaging

The CNT veil was imaged using a LEO Gemini 1525 FEG SEM (Carl Zeiss). The InLens detector was used to collect images at an accelerating voltage of 5kV. The working distance was 5-10 mm.

To create an orientation map, a high definition image of a large area of the CNT veil was required. To generate a high definition image of a large area several SEM images were stitched together. For the stitching the ImageJ plugin MosaicJ [3] was used. Starting from the top left corner images were captured sequentially with a 10% overlap per image in both the x and y direction. Within the MosaicJ plugin, the images were arranged into a ‘rough’ fit and then the plugin was used to adjust the images to find matching points and rearrange them to fit well, Figure 3.

The resulting image was a mosaic covering an area of 220 μm x 90 μm with the resolution of 52 pixels per micrometer (or 0.5 – 1 pixels per CNT diameter).

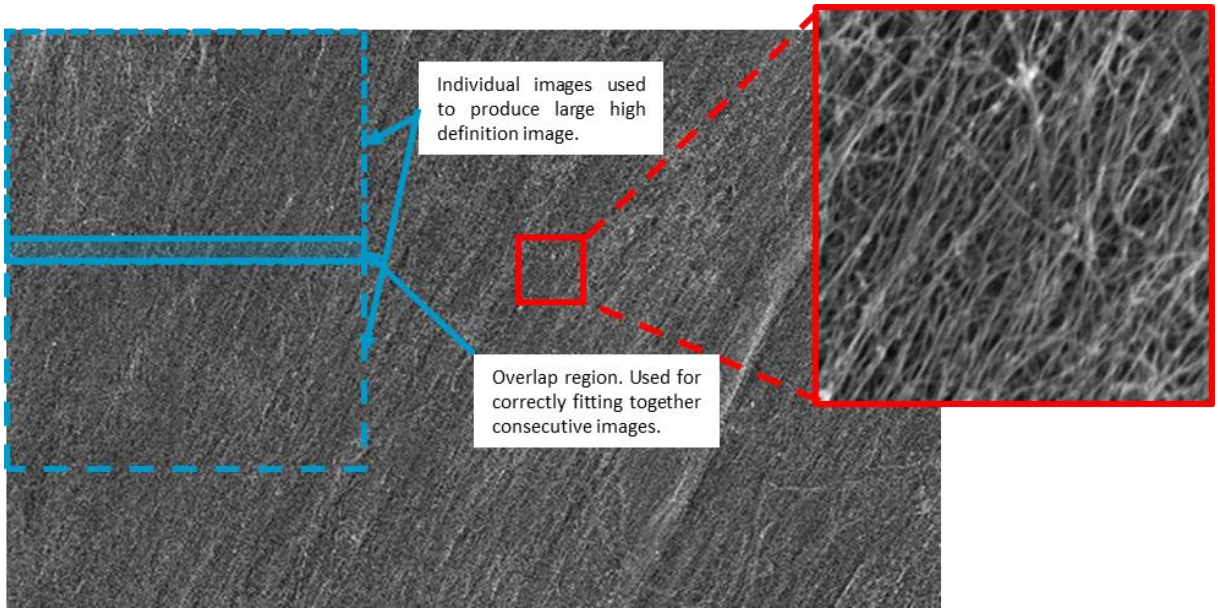

Figure 3 Example of production of large high definition mosaic.

4 RESULTS

4.1 Fourier Transform

Matlab was used for all image processing. All images processed were 8-bit grey scale images. The image crop function was used to split the large high resolution image into domains of 5μm x 5μm. The 2D-Fast Fourier Transform (2D-FFT) was computed using the inbuilt Matlab function FFT2, which returns the two-dimensional discrete Fourier transform (DFT) of the image, Figure 4. Integrating radially for all given angles θ around the Fourier transform produced an orientation distribution function from which the Herman Parameter was calculated (Equation 2 and 3). The Herman parameter is a well-known parameter which has been previously used to quantify the alignment of carbon nanotubes and calculated from Raman spectroscopy [5] and x-ray scattering [6].

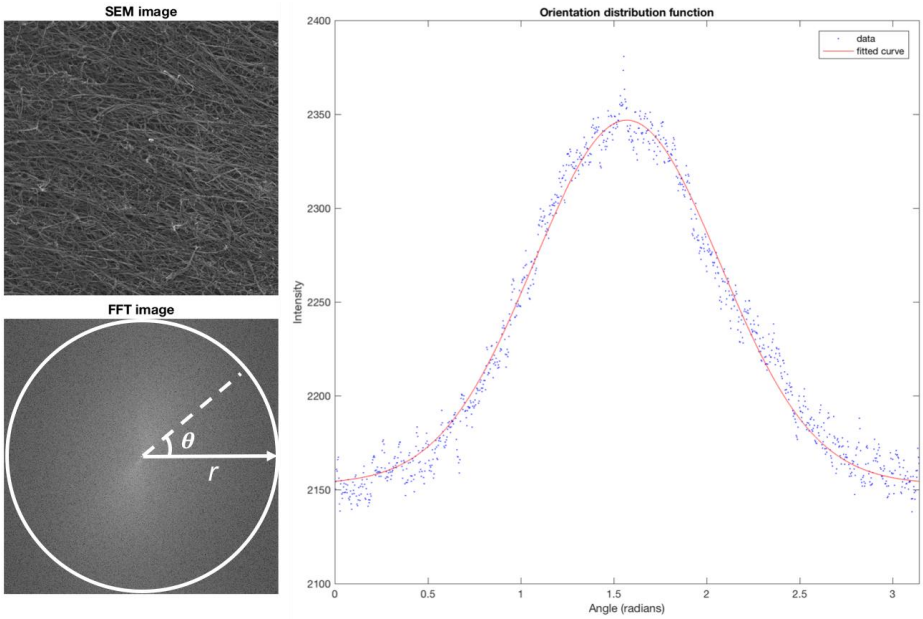

Figure 4 Original image with accompanying 2D-FFT and orientation distribution function

$$S = (3\langle\cos^2\theta\rangle-1)/\ 2 \qquad (2)$$

$$\langle cos^2\theta \rangle = \int I(\theta) cos^2\theta sin\theta d\theta / \int I(\theta) sin\theta d\theta \quad (3)$$

4.2 Image Filtering

Some image processing techniques can be applied to the original image before the 2D-FFT to remove noise from the image and enhance the key features of interest, i.e. the nanotubes and bundles. Image filtering has been useful for many applications including smoothing, sharpening and removing noise and edge detection. Filters used in this work included a Low Pass Filter (Figure 5), which acts to blur fine-scale details in the image, and High Pass Filter (Figure 6) and which raises the contrast of edges of the CNTs.

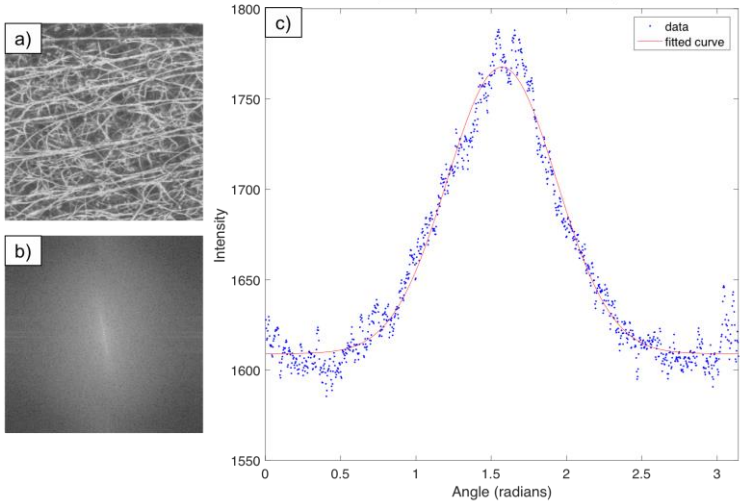

Figure 5 SEM image convolved with a low pass filter (a) the resulting FFT (b) Orientation distribution function (c)

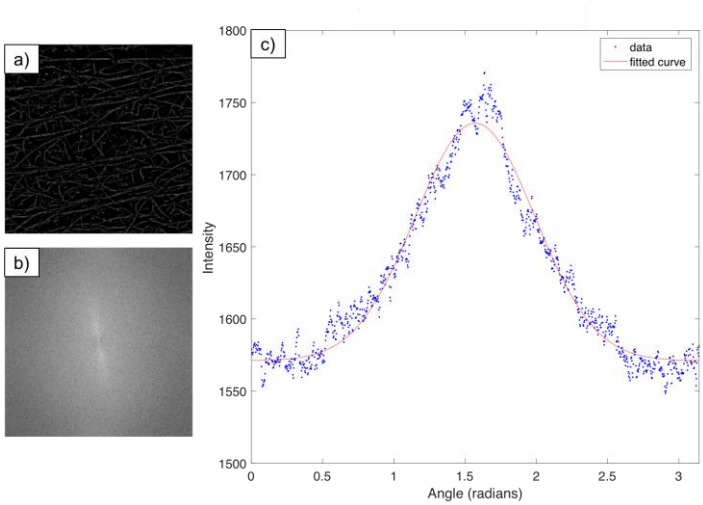

Figure 6 SEM image convolved with a high pass filter (a) the resulting FFT (b) Orientation distribution function (c)

A number of filters of varying cut off frequencies, were tested to minimize the least square rate of regression. Neither high nor low pass filters were found to significantly affect the orientation distribution function or lower the least square rate of regression. It was therefore decided not to apply a filter to the image before computing the 2D-FFT and orientation distribution function as it did not enhance the signal of the aligned CNTs

4.3 Initial Orientation Measurements

Previous studies have reported an increase in alignment (as measured by Polarized Raman spectroscopy) when veils are strained [7-8]. Therefore it was expected that veils strained to 10%, would be better aligned and have a greater Herman parameter than the as received sample. This can be seen qualitatively in the SEM micrographs and the orientation distribution function (Figure 7).

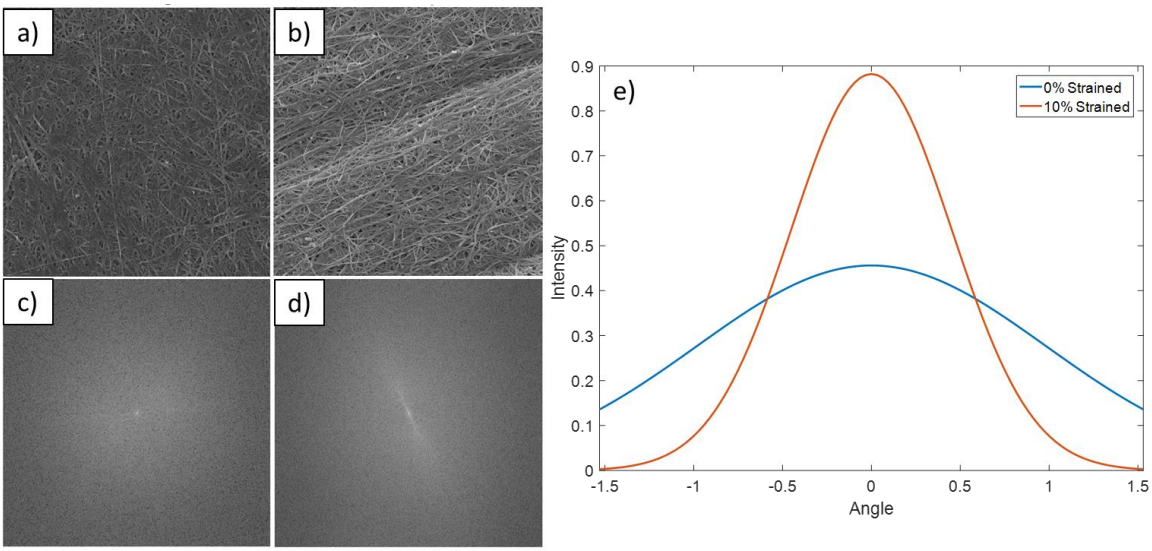

Figure 7 Two dimensional Fourier Transform and accompanying orientation distribution function for as-received and strained veils.

The white line in the power spectrum represents the spread of carbon nanotube orientation. The broader and duller this line, the greater the distribution, and therefore the less orientated the CNTs in the image are. It is this bright line that was quantified to produce the orientation distribution function and hence the resulting Herman parameter. As the CNTs in the image become more aligned the white line becomes more narrow and sharp and the calculated Herman parameter increases. The Herman parameter of the as received and strained samples was measured to be 0.09 and 0.55.

9 CONCLUSIONS

The use of a Fast Fourier Transform has been shown to be able to quantify the orientation of carbon nanotubes in veils. It was shown that taking a 2D-FFT of a scanning electron micrograph can quantify the orientation of carbon nanotubes and can be converted into an orientation parameter such as the Herman parameter. It was shown that filtering of the image prior to computing the 2D-FFT was not necessary as it did not act to increase the signal of the carbon nanotubes and bundles within the SEM micrograph.

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