Evaluation of fibers dispersion of cement-based sensors using scanning electron microscope

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INTRODUCTION

Cementitious sensors are obtained by reinforcing the cement paste with conductive materials such as carbon fibre, carbon nanotubes [1], carbon nanofibers [2] and/or carbon black [3]. The performance and sensitivity of the sensors mostly depend on the proper dispersion homogeneity of the conductive material within the cementitious matrix. Researchers have obtained different levels of electrical conductivity (an important property of cementitious sensors) by using various combinations of physical, chemical and mechanical dispersion techniques. The homogenous dispersion of fibres in the cementitious matrix allows the formation of an optimum conductive network and minimize the required volume fraction of fillers to achieve stable conductivity. Meanwhile, longer fibre length is more effective in providing a continuous conductive path [4]. Owing to their brittle nature, carbon fibres tend to break into smaller length, during the dispersion procedure. The general conductivity of CFRC sensors is influenced by the post dispersion lengths of the carbon fibres. Therefore, to maximize CFRC sensor conductivity and average fibre length, the dispersion method and duration should be refined. The three-dimensional alignment of carbon fibres is another important aspect in CFRC sensor performance [4]. In this study, several image processing techniques were implemented on scanning electron microscope (SEM) images to assess the quality of fibres dispersion in CFRC sensors.

EXPERIMENTAL PROCEDURE AND ANALYSIS TECHNIQUES

Materials and processing

Two sets of cylindrical CFRC sensors having a diameter of 50 mm were prepared with the mix proportions given in Table 1. The mixtures were designed with 0.5% and 10% volume fraction of carbon fibres. Thin sections were cut from Mixture #2 and prepared to be examined by Petrographic microscope and SEM. The main purpose of casting

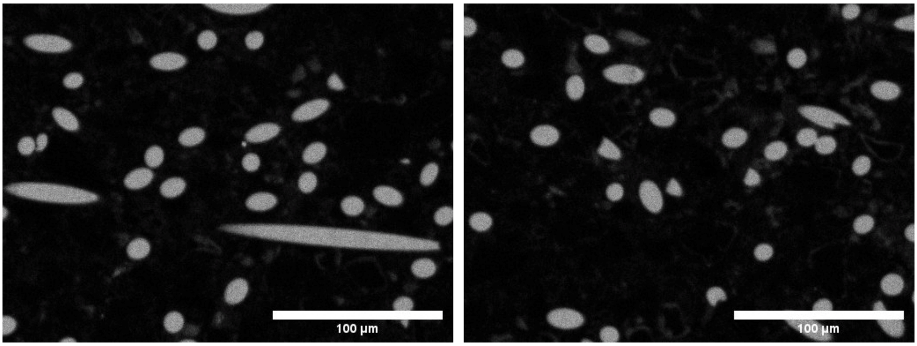
Mixture #1 was to investigate a methodology for evaluating the length distribution of carbon fibres right after mixing. The initial length of the carbon fibre used in the development of both mixtures under consideration is 6 mm.

Table 1: Mixture proportions

Mixture number	Volume fraction of carbon fibre	Quantity (g)			
		Cement	Water	Super-plasticizer	Carbon fibre
1	0.5%	3204	962	2	21.2
2	10%	2730	850	30	386.0

Evaluation of fibre distribution

The distribution characteristics of carbon fibre can be quantitively evaluated by interaction analysis. To detect the fibres in the image, elemental mapping was done for different areas using SEM (see Figure 1). Then, binarization was performed by the application of a threshold to the grey value of the image using an open source software (ImageJ). The nearest distances of the fibres to the edge of each other was finally obtained and represented in a form of probability density.



(a) (b)

Figure 1: Elemental maps for carbon: (a) area 1 and (b) area 2

Evaluation of fibre orientation distribution

The fibre orientation is the angle of the fibre inclined to the two-dimensional (2-D) thin section. 2-D image analysis technique may result in a significant systematic error in orientation measurements. To avoid this error, the orientation of the 2-D slice relative to the reference plane needs to be determined. To improve the evaluation precision of fibre orientation distribution, two images were taken from the same area on the thin section (both backscattered (Figure 2a) and optical images). Then, the images were aligned using Esri ArcMap software (see Figure 2b) and the position of individual fibres were studied. The dip angle of the fibres was simply calculated by Eq. 1:

$$\theta = Arccos\left(\frac{d}{L}\right) \tag{1}$$

Where θ, d and L are the dip angle, diameter and major axis length of the fibres, respectively. The bearing angle, based on the long axis of the individual fibre cross-sectional ellipse exposed at the surface is either θ or 180 + θ (Figure 3). The correct bearing angle selection was based on the aligned transmitted light thin section image; depending on which direction the fibre was pointing at depth through the thin section, the correct θ or 180 + θ bearing was identified. This was done by checking the orientation of each fibre in Figure 2b and by considering the right side of the image as north (0°, 360°) and the left side as south (180°).

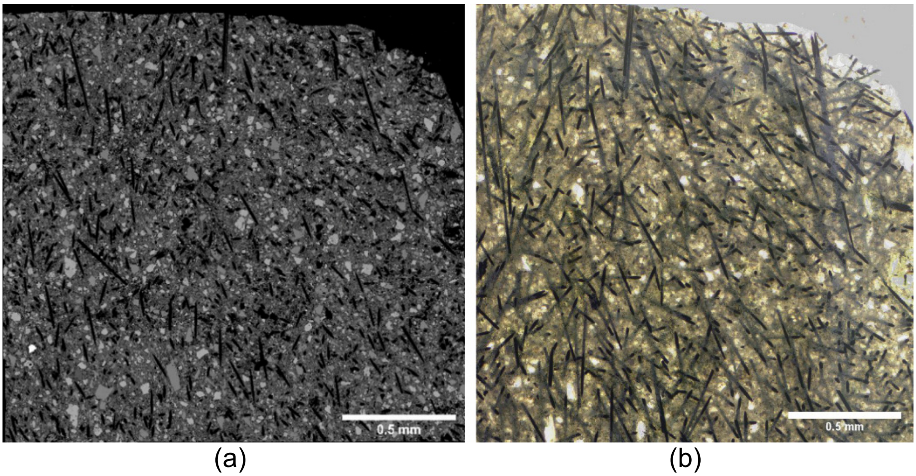


Figure 2: CFRC thin section (a) SEM backscattered image and (b) alignment of backscattered and optical image.

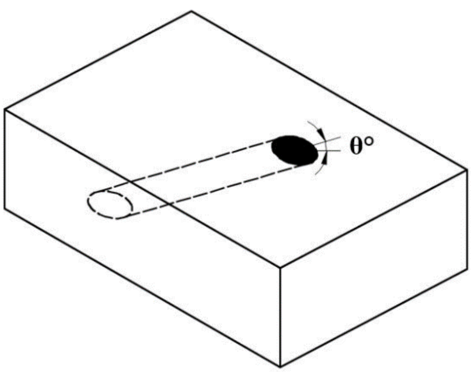
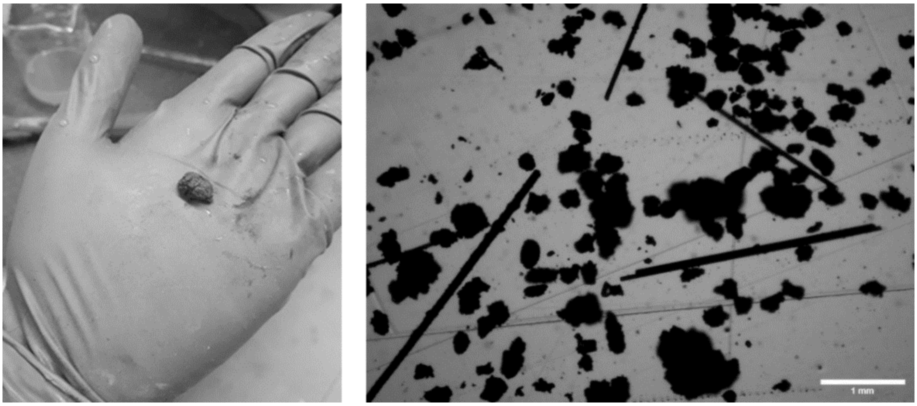


Figure 3. 3-D view of the bearing angle of a typical carbon fibre

<u>Evaluation of fibre length distribution</u>

A sample of 0.2 grams from the fresh Mixture #1 (see Figure 4a) was placed on a filter paper. Water was added gradually until most of the cement particles were washed off.

The remaining material on the filter were allowed to dry under normal condition. Then, images were taken from the sheet using an optical microscope (Figure 4b). Similar approach has been used by Li and Obla (1994) for studying the effect of fibre length variation on tensile properties of carbon fibre cement composites [5]. The individual fibre length after mixing was analyzed using the ImageJ software.



(a) (b)

Figure 4. Evaluation of fibre length: (a) sample containing 0.5% carbon fibre and (b) image of carbon fibres retained on filter paper

RESULTS AND DISCUSSION

Evaluation of fibre distribution

Figure 5 compares probablity density versus the nearest edge distance of the fibres at the two areas shown in Figure 1. As can be observed in Figure 5 the probability density function for area 1 and 2 was found to be very similar (bandwidth and maximum probability density). For instance, the maximum probability density of the fibre distribution corresponds to approximately 18 µm and 22 µm in area 1 and area 2, respectively. This observation reflects the adequate dispersion of the carbon fibres in both areas within the same thin section (Mixture #2). It should be mentioned that, the two areas in Figure 1 are from the same thin section (same volume of fibers and same dispersion techniques) which resulted in the similar probability density distribution. However, this analysis could also be useful in comparing the fibre distribution of specimens made with variable dispersion techniques.

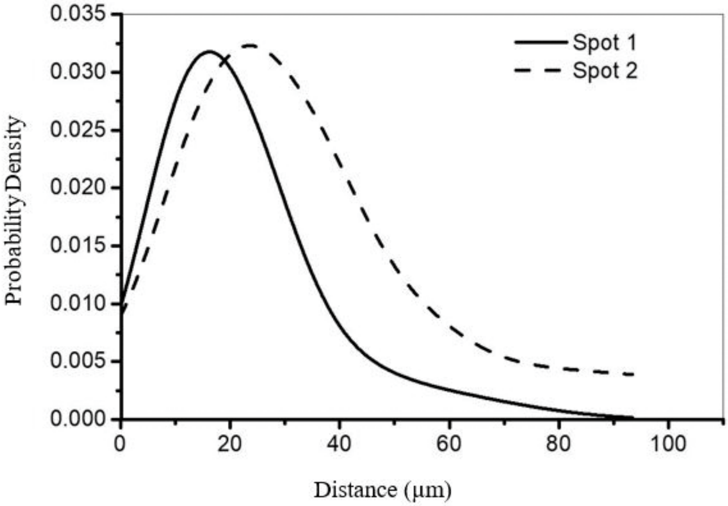


Figure 5: Probablity density versus distance of the fibres in spot 1 and 2

<u>Evaluation of the fibre orientation</u>

The orientation distribution of the carbon fibres in the images shown in Figure 2 was assessed by using stereographic projection techniques. Using these techniques, a stereogram of the orientation of the carbon fibres was obtained as shown in Figure 6. The distribution of fibre angles in Figure 6 indicates that most of the carbon fibres are within the same range of dip angle and corresponding bearing angle.

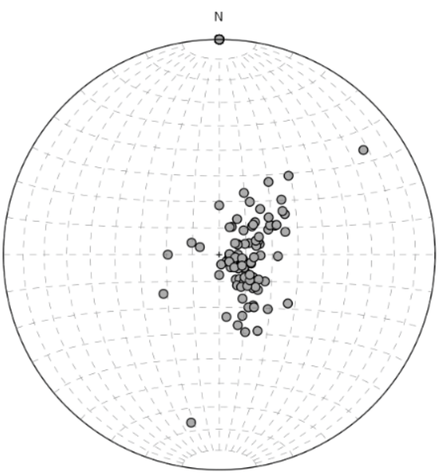


Figure 6: Stereogram of the fibre orientations

Evaluation of the fibre length

The graph in Figure 7 demonstrates the probability density of carbon fibre length in the sample containing 0.5% volume fraction of carbon fibre (Mixture #1). The initial length of the fibres before mixing process was about 6 mm. After the mixing, a length of fibres in the range of 1-1.2 mm represented the highest probability density (see Figure 7). This result matched the outcomes of the study performed by Li and Obla (1994) in which the length of the fibres having the highest probability density was about 1.3 mm [5]. Measuring fibre length using this approach was necessary, as fibres in thin section were necessarily truncated, unless perfectly parallel with the thin section surface.

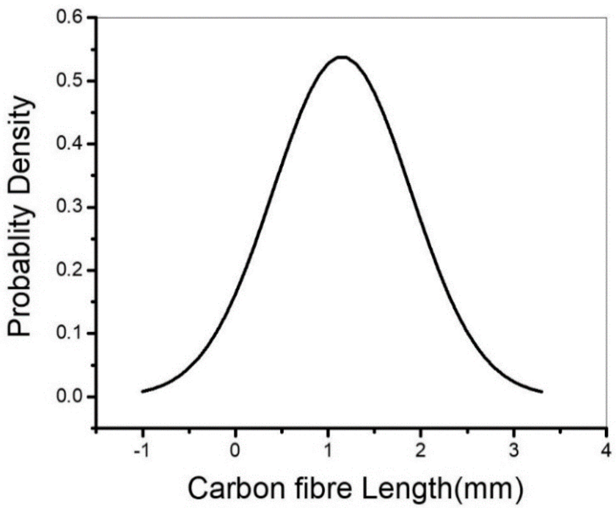


Figure 7: Probablity Density of carbon fibre length (Mixture # 1)

CONCLUSION

The dispersion quality of carbon fibre in CFRC sensors significantly changes the strain sensitivity of the sensors. The distribution, orientation and length of the fibres were evaluated using various image analysis techniques. The following conclusions can be drawn from the study:

The distribution of carbon fibres was analyzed by measuring the nearest distances from the edge of the carbon fibres. The proposed technique evaluates the distribution of the carbon fibres in a 2-D plane by obtaining images form SEM and aligning them to images from the same area but obtained from an optical microscope. This analysis allows the evaluation of the quality of fibre distribution and can be used as a tool to assess the dispersion of fibres.

The post-mixing length of the carbon fibres was quantitively measured from the fresh paste (Mixture #1). This method was much easier than the measurement of the carbon fibre length from the thin sections cut from hardened specimens.

The orientation of the carbon fibres in the 2-D thin section was studied precisely by measuring both the dip and bearing angles. This procedure was found to be useful in eliminating the errors in orientation measurements of the carbon fibres.

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