



ELECTROMECHANICAL TESTING OF CARBON FIBERS

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Abstract

An electromechanical method for determining the strains in non-embedded carbon fibers is developed. The method is extended to carbon fibers in polymer based composites and verified through Raman spectroscopic measurements.

In the electromechanical method a correlation between the strain and change in resistance in the fiber is found through tensile test of single non-embedded fibers. This correlation is used for estimating the quality of the interface between a carbon fiber and a polymer matrix.

Through a number of experiments it has been found that the developed method is reliable, cheap and easy to use. The results obtained from the electromechanical measurements are shown to correlate well with results obtained from Raman measurements.

1 Introduction

E-glass fiber is the most widely used fiber material in the production of wind turbine rotor blades, which is mainly due to the low cost of these fibers. As the size and performance requirements for the wind turbines are increased, carbon fiber reinforced composites have gained increased interest for these blades, due to their high specific modulus and strength.

However, a number of problems have to be addressed when changing from E-glass fibers to carbon fibers. One of the problems is to determine the quality of the interface between the reinforcing fibers and the matrix material to obtain the best possible utilization of the carbon fibers.

A precise tool for determining the interfacial properties of carbon fiber composites is by determining the strain in the fibers under loading using Raman spectroscopy. The method has been widely used to investigate the interfacial behavior

and the effect of fiber sizing in carbon fiber composites [1-3]. However, the use of Raman spectroscopy may not be a feasible method for companies which may have to test a large number of material systems to find an appropriate material system for their products. In order to perform the large number of tests it is necessary to have a method where the tests can be performed in a limited amount of time. Furthermore, the method has to be cheap and easy to use.

An alternative non-destructive method to determine the strain in fibers during loading is to utilize that the electrical resistance of carbon fibers changes during loading. However, relatively few studies of the electromechanical properties of a single carbon fiber have been performed. This is mainly because an appropriate method to connect the single fibers to the electrical measurement setup is required. It has been shown that the electrical properties of carbon fibers depend on their macroscopic properties such as the elastic modulus and their microstructure. Furthermore, it has been shown that the change in electrical resistance of the carbon fibers is mainly due to dimensional changes rather than changes in the resistivity [4-8].

Due to the difficulties involved in handling single fibers, electrical resistance measurements has been focused on multiple fibers to monitor load transfer, damage and failure properties. This includes non-embedded fiber bundles [9,10], laminated composites consisting of both carbon fibers and a mixture of carbon and glass fibers [11-17].

A limited amount of work have been performed on electromechanical studies of single fibers embedded in an epoxy matrix to study the load transfer and damage properties of the individual carbon fibers [18-22].

The main idea of the present work is to use the carbon fibers as stress/strain sensors and to compare the strain of embedded fibers obtained from the

electromechanical measurements to fiber strains determined by use of Raman spectroscopic measurements. A method based on electromechanical measurements would be a useful method to investigate the interfacial properties between fiber and matrix as well as the damage of the embedded fibers. Furthermore, the electromechanical measurements could provide a cheap method to determine the interfacial properties in a non-destructive way and would therefore be industrial feasible.

2 Experimental method

A single carbon fiber was used for each test specimen and the fiber used was a PAN based TohoTenax HM 35 fiber. The TohoTenax fiber has been heat treated at 600 °C with an isotherm of 4 hours in an inert gas atmosphere. The matrix material used for the samples was the epoxy resin LM E20 with hardener LM H20 supplied by LM Glasfiber. The epoxy is mixed in the ration 100:30 and the recommended curing temperature is 90 °C for 4 hours.

2.1 Preparation of test specimen

Two different types of samples were prepared for the electromechanical measurements. The first type of sample was a single non-embedded carbon fiber used to determine the relation between the straining of the fiber and change in resistance. The test specimen is based on a rectangular cardboard frame containing a rectangular hole as shown in Fig. 1.

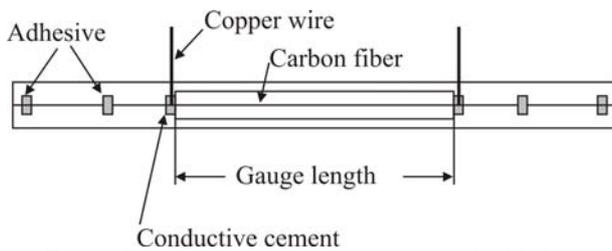


Fig. 1. Schematic illustration of non-embedded test specimen

The single fiber is mounted in such a way that it extends along the long axis of the cardboard frame crossing the rectangular hole as shown in Fig. 1. The fiber was attached to the cardboard with adhesive at four points and two copper wires were connected to the fiber using conductive carbon cement. With the cardboard frame it is possible to position the non-embedded fiber in the tensile test rig. The two ends of the frame are fixed in the rig and the two

legs of the frame are cut. Thereby it is possible to apply the load to the non-embedded fiber in the testing rig.

The second type of specimen used in the electromechanical measurements was a single carbon fiber embedded in an epoxy matrix. The specimen has a thickness of 2 mm and is shaped as a dog-bone. The test specimen is shown in Fig. 2 and the gauge length of these samples is 60mm.

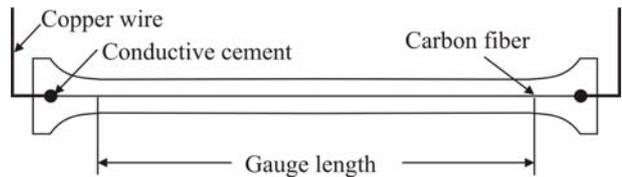


Fig. 2. Schematic illustration of dog bone shaped specimen

A silicon mold was used for the manufacturing of the dog-bone shaped specimens. A single carbon fiber was carefully aligned and fixated in the silicon mold to ensure it was positioned in the middle of the dog-bone specimen. Furthermore, a pair of copper wires was fixed in the silicon mold and electrical contact between the fiber and the copper wire was established using conductive carbon cement as used for the non-embedded fibers. After fixation of the fiber and copper wire the epoxy was poured in the silicon mold using a syringe. Afterwards the silicon mold was placed in a furnace and cured at 90 °C for 4 hours as described previously.

2.2 Experimental procedure

To determine the change in resistance of the carbon fiber a Wheatstone bridge is used and the test samples are coupled in a balanced half bridge. With the use of a Wheatstone bridge the relatively small changes in resistance can be measured with large accuracy. Furthermore, temperature compensation can be obtained with the Wheatstone bridge which in some situations will be advantageous. The bridge excitation voltage used in the current setup is 10 V. With the used bridge voltage no notable heat increase of the specimen was observed. The electromechanical testing of the carbon fiber specimens were performed by recording the bridge output voltage.

The output voltage from the Wheatstone bridge is directly related to the resistance during straining through the relation given in Eq. 1.

$$\Delta R = \frac{4V_0 R_0}{V_s} \quad (1)$$

Where V_0 is the bridge output voltage, R_0 is the resistance of the test specimen before straining and V_s is the bridge excitation voltage.

Before performing any tests on the samples with non-embedded fibers the resistance in the individual samples is measured using a multimeter. From the tensile tests of the non-embedded fibers a relationship is established between the applied strain and the change in resistance in the sample. In the same way the resistance of the dog-bone samples is measured before and after curing. In that way the change in resistance caused by the curing process can be determined. When knowing this change and the relation between applied strain and change in resistance an estimate of the residual strains in the embedded fiber can be obtained

The Raman equipment used for the Raman spectroscopic measurements is the Renishaw inVia Raman microscope equipped with a Renishaw RL633 HeNe laser providing an excitation wavelength of 632.8 nm. A modified Olympus microscope with a $\times 50$ objective lens giving a spot size of the laser of approximately 2-10 μm was used. The Raman spectrum of an unloaded HM35 fiber is shown in Fig. 3.

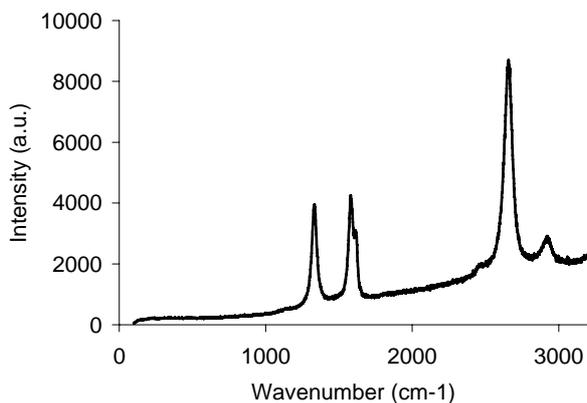


Fig.3. Raman spectrum of the HM 35 fiber.

The Raman spectrum consists of three major peaks and the position of these peaks are all strain sensitive. In this work the position of the peak at approximately 2661 cm^{-1} was used to determine the fiber strain. The tensile strain sensitivity of this peak was found to be $20 \text{ cm}^{-1}/\%$ which is consistent with values reported [1] and [23].

The straining rig used was a purpose build rig fitting under the Raman microscope and it is

constructed as a conventional tensile testing rig, Fig. 4.

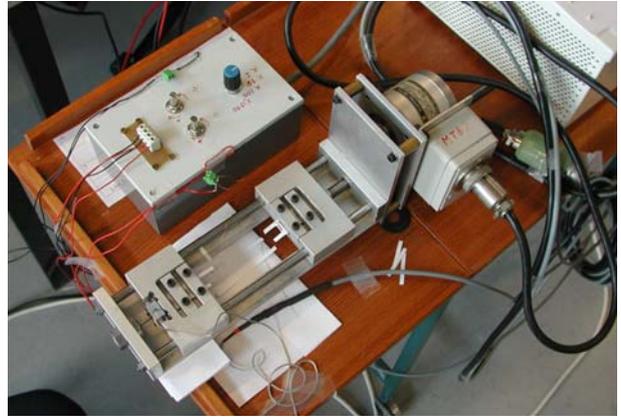


Fig 4. The straining rig and the control box for the Wheatstone bridge used for the electromechanical measurements.

The tensile test of the non-embedded fibers was performed using a constant cross head speed of 0.001 mm/min and the strain in the fiber was determined from the total cross head displacement. In these tests the low cross head speed was used to ensure that the fiber did not fail instantly and the bridge output voltage and the cross head displacement was recorded once per second. For the non-embedded fiber the applied force is not measured since the applied force is too small to sample with the load cells at hand.

For the dog-bone shaped specimens a constant cross head speed of 0.025 mm/min was used and the applied load on the sample was measured using a 2kN load cell. Once per second the cross head displacement, the bridge output voltage and the applied load was recorded. Raman measurements were performed on the dog-bone shaped specimens at specified loads of 0, 50, 75, 100, 125, 150, 175 and 200 N. At these loads the tensile test was paused and the Raman measurements were performed on the embedded fiber. The measurements were performed over a length of 140 μm in steps of 20 μm giving a total of eight measuring points for each load step.

3 Results and discussion

To determine the relation between the straining of the fiber and the change in resistance determined from the bridge output voltage, tensile tests were performed on non-embedded fibers as described above. Tensile tests have been performed using

different gauge lengths to examine whether or not the determined strain sensitivity factor is influenced by the gauge length used. During this examination it was found that the gauge length had no influence on the strain sensitivity factor. However, when shortening the gauge length the noise in the system became more and more pronounced in the obtained strain sensitivity factors. On the other hand, the longer the gauge length became the more difficult it was to handle the samples. Therefore, a gauge length of 60 mm has been chosen and the results presented are all obtained with this gauge length. The initial resistance in the samples was measured to be in the range 14-19 k Ω . The experimentally determined relation between the change in relative resistance determined from the bridge output voltage and the applied strain for the HM 35 fiber is shown in Fig. 5.

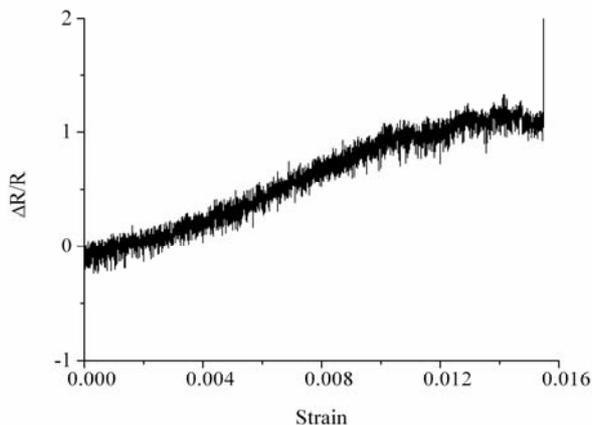


Fig. 5 Change in resistance versus applied fiber strain.

In many of the tested specimens no change in resistance is observed in the beginning of the test due to the fact that the fiber is not fully stretched when the test is initiated. At the end of the test, just before failure, the slope of the curve also changes probably due to slippage of the fiber in the carbon cement contact points. This change does not influence the applicability of the electromechanical testing method since the strains measured in carbon fibers in carbon fiber/epoxy composites typically are below 0.012.

Two different strain sensitivity factors are determined from the experimental data. The average relative strain sensitivity factor is determined as the slope of the linear part of the strain/relative resistance curve shown in Fig. 5. The average of the strain sensitivity factor is calculated for a series of tests and is found to be 108.6 with a standard deviation on 11.

The ultimate failure strain for the HM 35 fiber is reported by TohoTenax not to exceed 0.009. From the electromechanical tests of the non-embedded fiber it was generally observed that the fiber failed at an applied strain of approximately 0.005 which is significantly less than the value given by TohoTenax. In all tests the fiber failed at the point where the fiber is connected to the copper wire. Therefore, the reason for the large discrepancy in ultimate fiber strain is most probably introduction of stress concentrations at the connection points.

It is important to note that the determined strain sensitivity factor is only valid for the examined HM 35 fiber. The reason is that the strain sensitivity factor depends on both the micro structural composition and the diameter of the fiber. Fibers of different grade and diameter may therefore result in different strain sensitivity factors. The same observations may be done if testing fibers of low quality where large variations in the micro structural composition may be observed.

From the strain sensitivity factors it is possible to calculate the strain in the fibers of a carbon fiber composite.

The dog-bone shaped specimens had a gauge length of 75 mm and the initial resistance in the fibers before curing of the samples was in the range 18-21 k Ω . After curing, the resistance in the specimens increased to 21-24 k Ω due to compressive residual strains caused by thermal coefficient mismatch between the fiber and the matrix material.

A typical stress/strain curve for the dog-bone measurements is shown in Fig. 6.

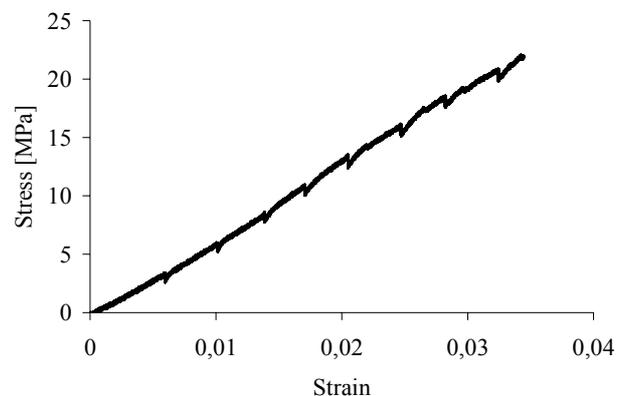


Fig. 6. Stress/strain curve for dog-bone sample

In the stress/strain curve shown in Fig. 6 eight small drops in the stress are observed. Each of these drops corresponds to an applied strain at which the tensile test is paused and Raman measurements are

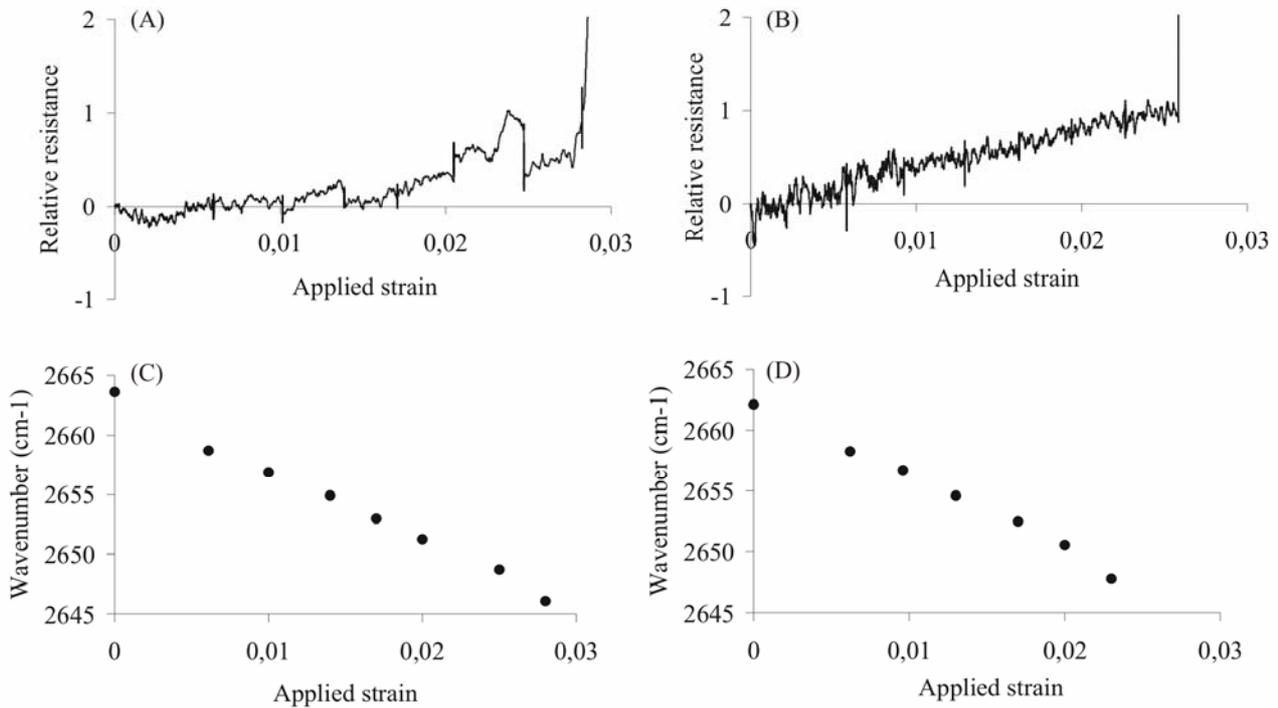


Fig. 7. The relative resistance (A, B) and the peak position (C, D) as a function of the applied strain

performed. Each decrease in the stress is due to relaxation of the matrix material but the size of the decreases is minimal and therefore not estimated to influence the obtained results.

In Fig.7 the relative resistance and the position of the 2661 cm-1 peak is plotted versus the applied strain for two different samples. A linear relation between both the relative resistance and the peak position with applied strain is observed. In Fig. 7(A) a sudden increase and decrease is observed for an applied strain between 0.02 and 0.025. This increase and decrease is most probably due to slippage in the

contact points between the carbon fiber and the copper wire. This behavior is not observed in any of the other samples. The singularities observed in Fig. 7(A) and (B) at applied strains of 0.029 and 0.026, respectively, correspond to fiber failure. The applied strain equals the strain applied to the dog-bone sample and not necessarily the strain level in the embedded fiber. A difference in strain levels could be a result of an interface between the carbon fiber and the matrix material of a poor quality and thereby a poor load transfer.

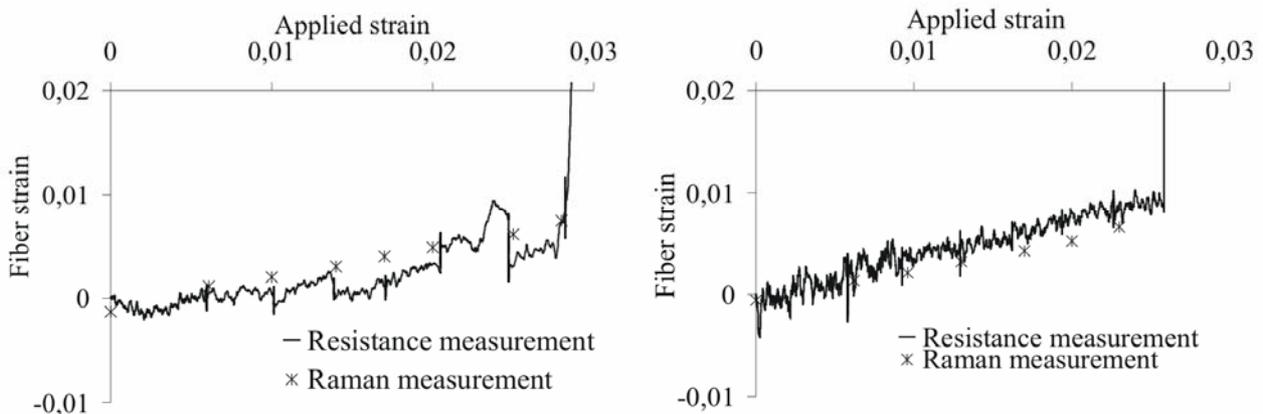


Fig. 8. Strain in the fiber obtained through resistance measurements and Raman measurements as a function of the applied strain.

In Fig. 7(C) and (D) each point corresponds to the average value of eight Raman measurements performed at different positions of the fiber as described previously.

From the results presented in Fig. 7 the strain in the fiber can be calculated in two different ways: from the change in relative resistance and the change of the peak position. In Fig. 8 the fiber strain calculated using both methods is shown as a function of the applied strain. For all samples a good correlation between the fiber strains determined through the two different methods are found. Through a least square fitting of all the experiments performed, the proportionality factor between the fiber strain and the applied strain is determined. The proportionality factor between the applied strain and the fiber strains based on the electromechanical measurements and the Raman measurements is determined to 0.36 and 0.30, respectively. These values also show a very good correlation between the two different methods.

From Fig. 8 it is seen that the strain level in the fiber is much smaller than the strain applied to dog-bone sample. As mentioned previously this is probably due a poor quality interface between the two material constituents.

4 Conclusions

An electromechanical method for determining the strains in non-embedded carbon fibers has been developed. A correlation between the strain and change in resistance in the fiber is found through tensile test of single non-embedded fibers. This correlation is then used for extending the electromechanical method to carbon fibers embedded in a polymer. A series of carbon fiber/epoxy sample has been examined and the results obtained from the electromechanical measurements have been verified through Raman spectroscopic measurements.

Through a number of experiments it was shown that the developed method is reliable, cheap and easy to use. The results obtained from the electromechanical measurements correlated well with results obtained from Raman measurements.

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