DETERMINATION OF ENVIRONMENTAL DEGRADATION OF MATRIX AND FIBRE MATERIALS WITH A NOVEL, STATISTICALLY RELIABLE MICRO-ROBOTIC APPROACH

Essi Sarlin[1], Mathias von Essen[2], Sarianna Palola[3], Mari Lingren[4], Pasi Kallio[5], Jyrki Vuorinen[6]

[1]Department of Materials Science, Tampere University of Technology, P.O. Box 589, 33101 Tampere, Finland
Email: essi.sarlin@tut.fi, Web Page: http://www.tut.fi/en
[2]Department of Automation Science and Engineering, Tampere University of Technology, P.O. Box 692, 33101 Tampere, Finland
Email: mathias.vonessen@tut.fi, Web Page: http://www.tut.fi/fibrobotics
[3]Department of Materials Science, Tampere University of Technology, P.O. Box 589, 33101 Tampere, Finland
Email: sarianna.palola@tut.fi, Web Page: http://www.tut.fi/en
[4]Outotec Research Center, Kuparitie 10, 28330 Pori, Finland
Email: mari.lindgren@outotec.com, Web Page: http://www.outotec.com/en
[5]Department of Automation Science and Engineering, Tampere University of Technology, P.O. Box 692, 33101 Tampere, Finland
Email: pasi.kallio@tut.fi, Web Page: http://www.tut.fi/en
[6]Department of Materials Science, Tampere University of Technology, P.O. Box 589, 33101 Tampere, Finland
Email: jyrki.vuorinen@tut.fi, Web Page: http://www.tut.fi/en

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Abstract
In this study, environmental degradation of typical composite matrix and reinforcement materials were studied with a new micro-robotic approach. Polyamide (PA), ECR glass and carbon fibres were exposed to different environments, namely hot water (PA), acidic water solution (glass) and hot oxidative environment (carbon). Subsequently, the degradation of the materials was evaluated based on the micro-robotic tensile test results as well as on the microscopic characterization. The tensile properties were compared with the results obtained by a traditional fibre tensile method (ISO 5079). The results of the micro-robotic and the traditional test method were similar. It was found that the micro-robotic specimen manipulation allows testing of a wider range of materials and thinner fibres than is currently feasible with traditional methods. It also reduces the effect of measurement inaccuracies.

1. Introduction

In different fields of industry, such as aerospace or rail and road traffic industries, polymer matrix composites are used in applications in which the components are exposed to aggressive environments, but which also require high reliability and a long lifetime. Thus, unexpected strength loss of a composite structure within the lifetime of the component is unacceptable. The conventional experimental methods to study the mechanical properties of polymer composites do not always produce sufficiently high throughput to detect small, but statistically valid changes in material properties induced by ageing. Therefore, more reliable and effective test methods are welcome to the variety of test methods to study the long-term behaviour of composites in aggressive environments.

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Recent developments in the automation and robotic engineering have opened new possibilities for materials science to study the mechanical properties of fibrous materials in a statistically relevant manner. Automated tensile test equipment, such as FAVIMAT+ (Textechno Herbert Stein GmbH & Co. KG) and MTT680 (Dia-Stron Ltd.), are already commercially available. Both of the aforementioned equipment resemble traditional tensile test equipment, but are specialized for measuring tensile properties of fibrous materials.

Micro- and nanorobotics is an emerging trend in characterization, manipulation and assembly of microscopic specimen. Use of micro-robotics has been reported in applications such as (i) manipulation of nanowires [1], (ii) characterization of collagen scaffolds [2], (iii) processing of graphene [3], (iv) microinjection and characterization of living cells [4, 5], (v) assembly of microscopic objects [6] and (vi) characterization of pulp fibres [7, 8]. One of the major benefits of the robotic approach is the possibility to automate tasks that otherwise require tedious and time consuming manual work [9, 10]. This paper presents the FIBRobot, which is a micro-robotic concept and platform for characterization of fibrous materials. The objective of the FIBRobot is to develop automated testing methods to study different mechanical and interfacial properties of polymer composites at single fibre level.

In this study, we report the applicability of the FIBRobot module for tensile testing. The major advantage of the introduced system is its capability to manipulate and characterise individual fibres with a significantly higher throughput than what is in the reach of conventional methods. The applicability of the approach is demonstrated by determining the environmental degradation of both matrix and fibre materials. Polyamide (PA), glass and carbon fibres were aged in different environments: in water or in an acidic water solution at elevated temperature. Afterwards the tensile properties of the fibres were determined.

2. Micro-robotic approach

FIBRobot fibre characterization platform is designed bearing modularity in mind. The core of the platform includes an imaging set-up for visual inspection of the samples and a microhandling set-up for picking and placing the studied specimen. The imaging set-up consists of a camera (Point Grey Research Inc.) and motorized microscope optics (Navitar Ltd.). The samples are picked up and placed into the FIBRobot tensile test module with two 3-DOF micro-manipulators (SmarAct GmbH) that are equipped with micro-grippers.

Different characterization tasks are implemented as modules that can be integrated into the FIBRobot platform. The main components of the FIBRobot tensile test module are (i) a load cell (FUTEK Advanced Sensor Technology Inc.), (ii) a single-axis micro-translation stage (Physik Instrumente GmbH & Co. KG.) and (iii) an in-house built fibre clamping mechanism. The tensile test module is attached to the top of a two-axis translation stage (SmarAct GmbH) which is used for positioning of the sample under the imaging set-up's field of view. The FIBRobot platform with the tensile test module is presented in Figure 1.

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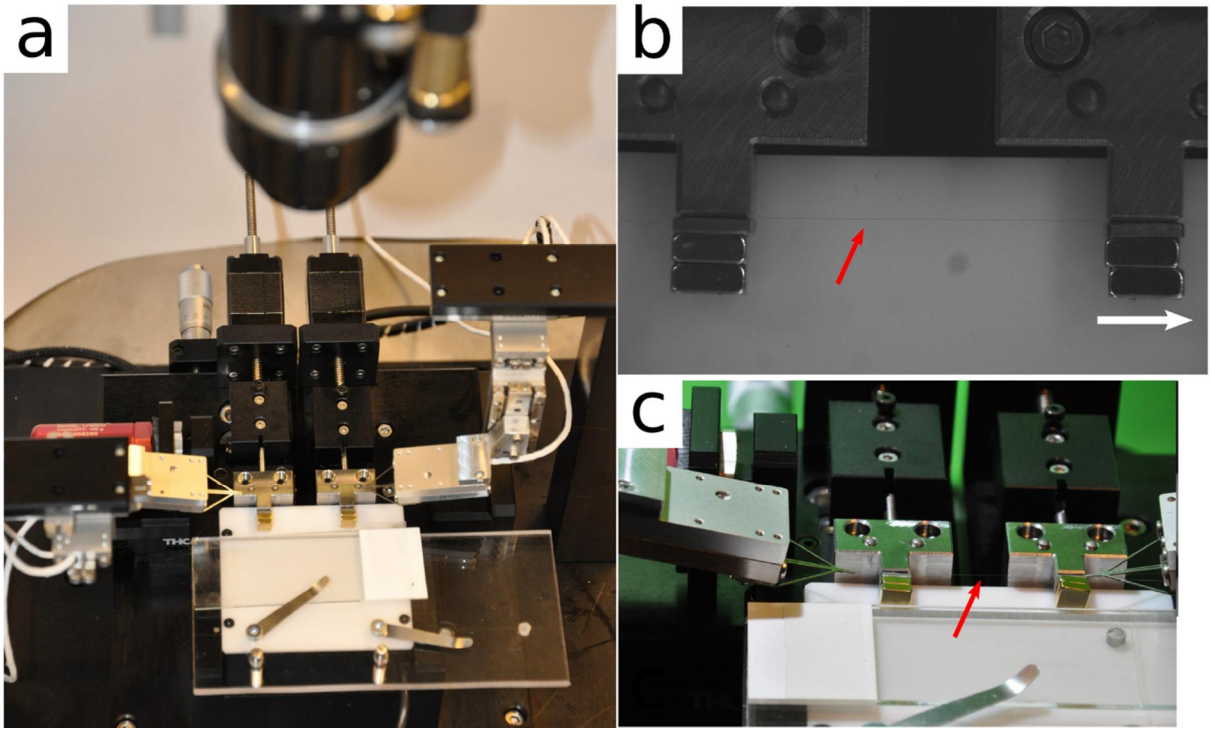

Figure 1: a) Overview of the FIBRobot characterization platform, b) polyamide fibre attached between the clamps during tensile test (red arrows indicate fibre location and white arrow indicates the clamp that is moved with micro-translation stage) and c) glass fibre positioned with micro-gripper prior to tensile test.

The tensile test procedure is as follows. Single fibres are manually positioned on a standard 75x26 mm microscope slide and the microscope slide is placed on a back light illuminated (Falcon Illumination Sdn. Bhd.) sample holder (Figure 1a). The sample holder is positioned so that the samples are in the imaging set-up's field of view. Next, the operator selects the sample to be tested by using a graphical user-interface. The microhandling set-up grabs the sample from its both ends and positions the sample between the clamps of the tensile test module (Figure 1c). Finally, the tensile test is performed by moving the micro-translation stage until the sample breaks (Figure 1b). The required force is measured with the load cell and displacement data is obtained from the internal encoder of the micro-translation stage. Image data is acquired and recorded from the entire measurement procedure.

3. Materials and Methods

Within thermoplastic polymer composite matrices, polyamide (PA) is a commonly used material, whereas glass and carbon are the most typical reinforcements in composites. The tensile properties of a PA yarn, an ECR-glass fibre and a carbon fibre were studied before and after ageing. Since PA is susceptible to hot water [11], it was aged in deionised water at 95 ºC for 12 hours. After ageing, a part of the fibres were left in the deionized water at room temperature and tested in a wet stage whereas the other part was dried and tested in a dry stage. The glass and carbon fibres were studied in the dry stage. The tensile strength of carbon fibres decreases due to oxidation which makes them vulnerable to high temperatures [12]. Thus, the carbon fibres were aged at 400 ºC in air for 5 hours. The glass fibres were aged in an acidic solution of water and sulfuric acid (pH 2). The materials and the ageing environments are summarized in Table 1.

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Table 1: The studied materials and ageing environments.

Material	Diameter	Sample	Ageing
Polyamide fibre (in-house produced)	25 μm	PA_ref	-
		PA_aged_dry	At 95 ºC in deionised water for 12 h, tested in dry stage
		PA_aged_wet	At 95 ºC in deionised water for 12 h, tested in wet stage
ECR-glass fibre (P-D Glasseiden GmbH, Germany)	12 μm	G_ref	-
		G_aged	At 95 ºC in acidic water solution (pH 2) for 4 weeks
Carbon fibre (Toray Industries, Inc, Japan)	5 μm	C_ref	-
		C_aged	At 400 ºC in air for 5 h

The tensile strength of the fibres was studied before and after ageing by the FIBRobot, the micromechanical testing method described in the previous Chapter. To compare the FIBRobot test results to a conventional test method, the PA_ref fibres were also tested with a Favigraph (Textechno Herbert Stein, Germany) according to the ISO 5079:1995. The tests were done in a controlled environment (20 ºC ± 2 ºC, 65 %RH ± 4 %RH according to the ISO 139:2005) excluding the carbon fibres which were tested in a ambient laboratory environment (23 ºC, 30 %RH). The rate of extension was for PA fibres 20 mm/min (in both test methods) and 6 mm/min for carbon and glass fibres. The initial fibre length was 22.5 mm in the FIBRobot tests and 20 mm in the Favigraph tests. The number of samples per test was 50 for the PA_ref samples for both test methods and 10 in other FIBRobot tests.

To study the effect of the ageing on the fibres appearance, scanning electron microscopy (SEM) Zeiss ULTRAplus (Zeiss, Germany) was employed. The samples were coated with a thin carbon layer before analysis to ensure conductivity. The SEM analysis was also used to measure the fibre diameter (results shown in Table 1).

4. Results and Discussion

The tensile test results of PA_ref samples obtained by the two test methods are shown in Figure 2. Both methods yielded very similar force-elongation curves and the breaking forces coincided within the standard deviation values (237.8 mN ± 29.9 mN with the FIBRobot and 255.3 mN ± 26.7 mN with the Favigraph). The FIBrobot results were slightly lower in average when compared with the Favigraph values. In the Favigraph results (Figure 2.b), the pretension shifted the initial starting point of the curves from the graph origin. The elongation at break was 30% in average by the FIBRobot and 33% by the Favigraph which are at the range of typical values for PA fibres [13].

To study the distribution of the tensile test results of PA_ref sample, a normal distribution was fitted to the breaking force values obtained by both methods as shown in Figure 3. The origin of the variation in the breaking force values is most probably the uneven porosity of the in-house prepared PA fibre. The linear density values measured by the Favigraph support this assumption: although the PA fibre seemed to have a uniform thickness according to the SEM studies, the linear density had a relatively high variety (6.00 ± 0.75 dtex). The skewness of the FIBRobot and the Favigraph results was 0.756 and 0.945, respectively, which indicates that the PA_ref breaking force distribution was moderately skewed ($0.5 < |\text{skewness}| > 1.0$).

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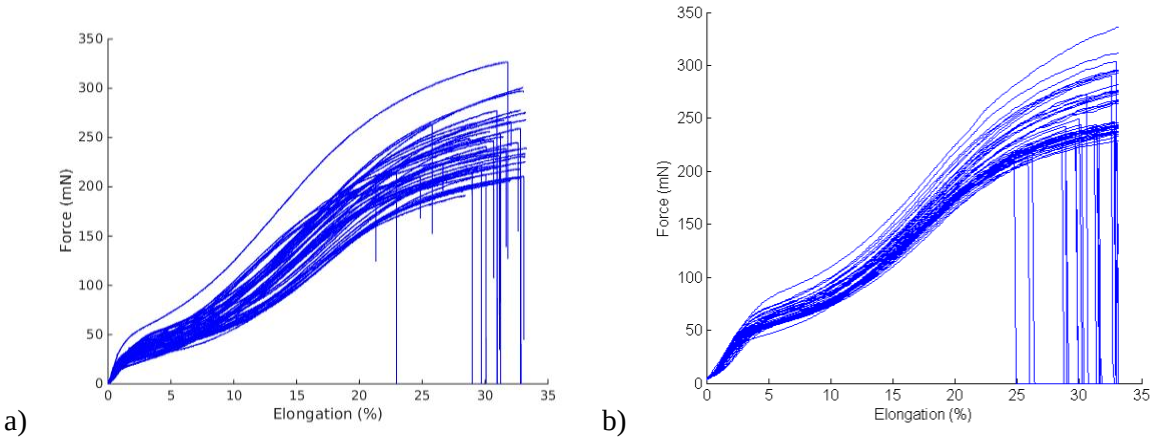

Figure 2: The tensile test results obtained by a) the FIBRobot and b) the Favigraph.

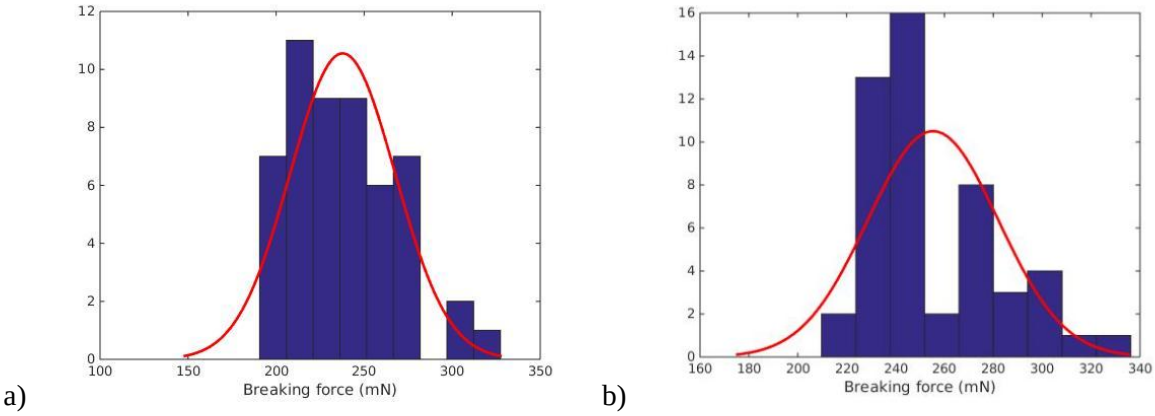

Figure 3: The breaking force values obtained by a) the FIBRobot and b) the Favigraph and the fitted normal distributions.

The effect of ageing on the tensile strength is shown in Figure 4. The aged PA fibres lost roughly 60% of the tensile strength and the tensile properties were similar in dry and wet stages, although in general the tensile properties of polyamide decrease in wet stage [13]. These indicate that the ageing in hot water has caused hydrolysis in PA and not only water uptake [14]. The decrease in the breaking force of the glass fibre was 82% due to the ageing in sulphuric acid solution. Although having better resistance to acid when compared to E-glass, the studied ECR glass was strongly affected by the ageing. Since the diameter of the aged glass fibres was difficult to define for the individual tested fibres (see Figure 5), the results are shown in the form of breaking force instead of tensile strength. Thus, these results describe the changes in the behaviour of fibres, not the changes in the material properties. The oxidation of carbon fibres occurs quickly when the temperature of air rises above 500 ºC which is seen as a decrease in the tensile strength [15]. Consequently, the observed strength loss (29%) was in the expected range. The strong scattering of the breaking force values after ageing can be explained by the different environments experienced by the individual fibres in the aged fibre bundle. The fibre bundle was originally tight but was clearly loosened during ageing.

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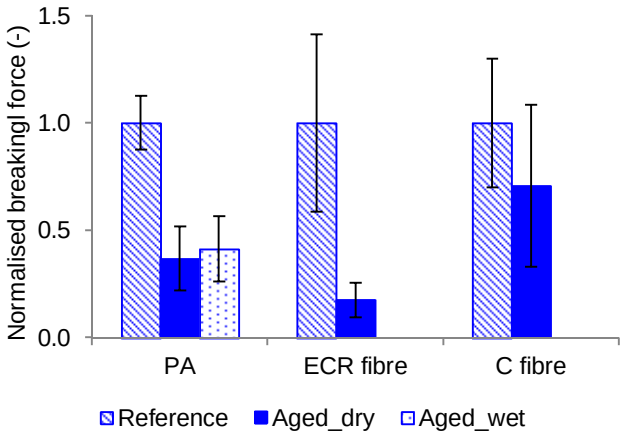

	Breaking force (mN)	
	Reference	Aged
PA	237.8±29.9	100.2±35.6 (dry)
		108.6±36.5 (wet)
G	162.0±67.1	28.4±13.1
C	51.9±15.6	36.7±19.6

Figure 4. The normalised and absolute breaking force and standard deviation vales before and after ageing for PA, glass and carbon fibres.

The FIBRobot concept provides significant added value to testing of single fibres due to the sample manipulation assisted by micro-robotic handling and microscope optics. The micro-robotic specimen manipulation also reduces the effect of measurement inaccuracies, e.g., due to the damage induced during sample preparation and manipulation when compared with traditional methods in which the specimen is manipulated manually. Also, the FIBRobot is suitable to analyse fragile (glass) or high modulus (carbon) fibres that can cause problems in clamping the specimen or in measuring the linear density with the Favigraph.

When studied by SEM (Figure 5), PA fibres did not exhibit clearly visible changes in their appearance after ageing. The aged glass fibres showed severe cracking and exfoliation of the outer layers of the fibre. This kind of behaviour has also been reported for E-glass fibres in acidic environments [16, 17]. The surface roughness of the carbon fibres increased indicating material oxidation and subsequent loss at microscopic scale [15] which also resulted in decreased tensile strength. The most severe degradation was seen in the glass fibres which was consistent with the tensile test results.

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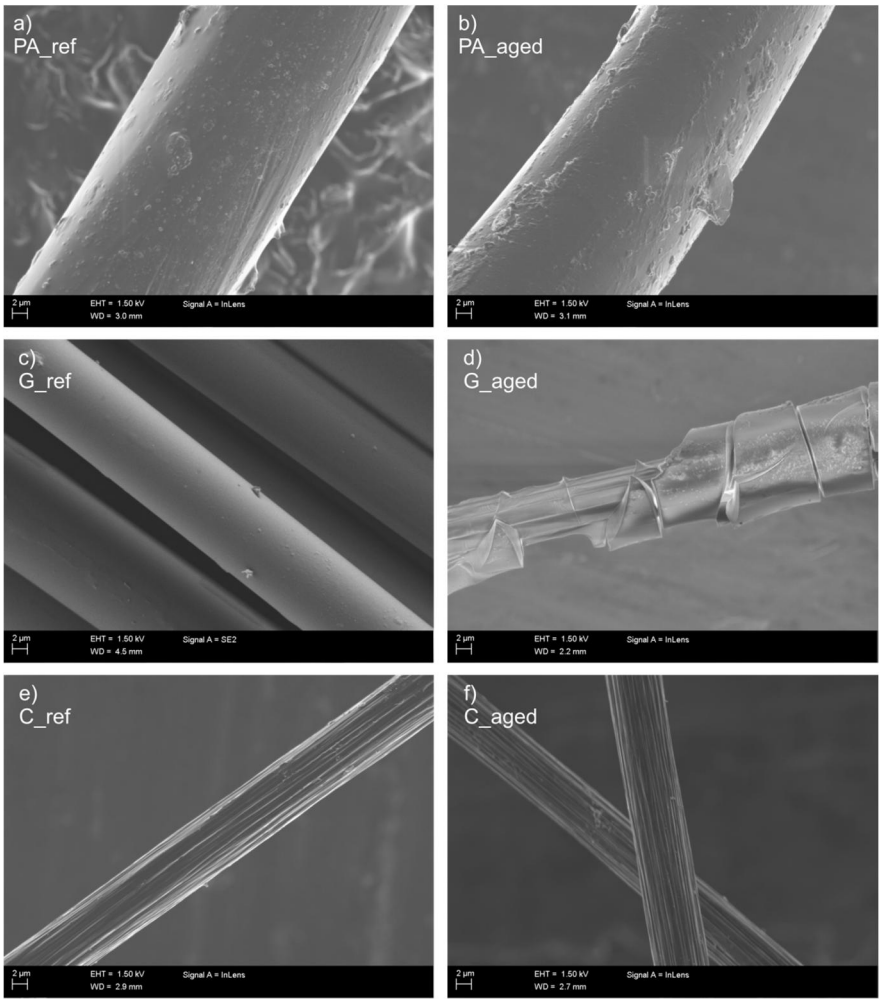

Figure 5: Unaged and aged PA, glass and carbon fibres.

5. Conclusions

In this study, we demonstrate the feasibility of a novel micro-robotic tensile test module by applying it to an investigation of the environmental degradation of polyamide (PA), ECR glass and carbon fibres. The obtained tensile test results were compared against the values of a traditional fibre tensile method. The micro-robotic approach was proven to be suitable to study the micro-scale properties of common matrix and fibre materials and the results were similar with both methods.

The micro-robotic concept provides significant added value to testing of single fibres. The micro-robotic specimen manipulation allows to test a wider range of materials and thinner fibres than is currently feasible with traditional methods and it also reduces the effect of measurement inaccuracies. Further, the equipment has potential to be modified for testing of numerous material properties as well as to be integrated in other characterization methods, such as in scanning electron microscopes.

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