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Tensile testing of ceramic fibres by video extensometry

D. COIMBRA*, R. GREENWOOD, K. KENDALL
Birchall Centre for Inorganic Chemistry and Materials Science, School of Chemistry and Physics, Keele University, Keele, Staffs, ST5 5BG, UK
E-mail: chd73@keele.ac.uk

A computer-assisted video extensometer was used to measure the Young's modulus and tensile strength of commercially available alumina fibres (11.5 μm in diameter). The results showed excellent agreement with manufacturers reported values (384 ± 12 GPa and 3132 ± 296 MPa for Young's modulus and tensile strength, respectively). The machine was initially tested with 100 μm diameter SiC monofilaments to identify optimum experimental conditions. The mechanical properties of these fibres were independent of the crosshead speed and fibre length. This non-contacting extension measuring system allowed testing of fragile materials and a submicron resolution could be achieved with a high-precision CCD camera. The results in term of precision and resolution therefore meet the requirements for strain measurements in mechanical ceramic materials testing.

1. Introduction

The accurate determination of the mechanical properties of ceramic materials, e.g. Young's modulus and tensile strength, is essential in many applications, such as the reinforcement of ceramics with strong fibres to make composites [1, 2].

Stress and strain are the two main parameters from which the mechanical properties are calculated during uniaxial testing [3]. For rigid materials, the strain is usually measured by using mechanical extensometers or by bonding foil gauges to the specimen [4]. Non-contacting extension measuring devices are however more suitable for delicate specimens such as ceramic fibres. Elastic constant determination using dynamic vibrational and ultrasonic methods have been used in the past [5], yet it is still difficult to achieve good accuracy in determination of strain using non-contacting and static methods.

Several non-contacting extension measuring systems, in particular laser and video extensometers, have been developed over the last few years to record the plastic behaviour of polymers and metals [6–9]. Computer-assisted video-controlled extensometry provided accurate true-strain measurements of ductile materials over large plastic strains. However, the technique has never been exploited to record very low strain responses, typical of elastic behaviour of ceramic materials at room temperature. The aim of this paper is to show how the mechanical properties of ceramic fibres can be determined by video-extensometry at very low strain levels.

First, the components of the experimental apparatus and the basic principles of the method will be presented. The capabilities of the technique will be illustrated by testing silicon carbide monofilaments and single strand alumina fibres. The influence of different factors such as the specimen length or the crosshead speed will be discussed. The results will ultimately illustrate the limitations and potentials of the video extensometer to record very low strains and to measure in particular elastic constants of fragile materials.

2. Experimental apparatus

2.1. System components

The video extensometer (ME-46 Full Image Videoextensometer, Messphysik Laborgeräte GES.m.b.H., Austria) used in this study is based on the same principles of the system used by G'Sell *et al.* [8] to record the intrinsic plastic behaviour of ductile materials. The system (Fig. 1) is composed of:

– a fast-processor PC (Pentium based main board), which allows real-time acquisition and analysis of the data,
– a universal testing machine (LRX, Lloyds Instruments Ltd, UK) fitted with a calibrated 50N load cell,
– a 16 Bit analogue/digital interface for connecting the extensometer to the testing machine, allowing the load signal to be saved simultaneously with strain,
– a video camera, fitted with a high precision CCD (Charge Coupled Device) chip,
– a high precision variable focal length (or "zoom") lens, which covers a wide range of specimen sizes,
– a digitising interface card fitted into the PC and connected to the camera. This card converts the PAL

* Author to whom all correspondence should be addressed.

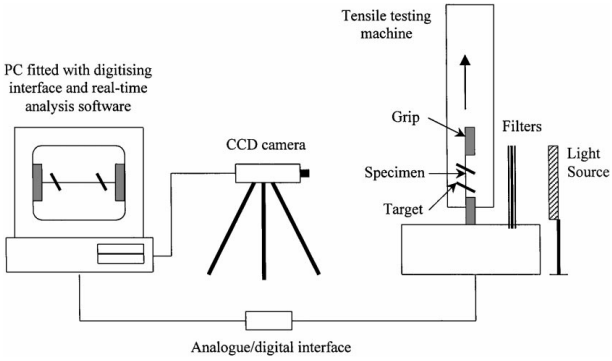

Figure 1 General diagram of the video-extensometer connected to the tensile testing system.

video signal into an 8 Bit digit format whilst simultaneously generating a 640×480 pixel image on the PC monitor. The brightness of each pixel is analysed on a 256-level grey scale which results in a minimum theoretical displacement resolution better than 17 Bit (1 : 131073) of the camera field of view,

– an operating software,

– a post-test application software, which allows processing of the data captured during testing.

2.2. Principles of operation

The video extensometer determines the change in distance ($\Delta\ell$) between marked targets caused by mechanical strain to the specimen. The strain (ε) is then calculated as a percentage of the original length between the targets (ℓ_0) measured before testing, i.e.:

$$\varepsilon = \frac{\Delta\ell}{\ell_0} \tag{1}$$

The camera is focused on the specimen to which contrasting marks (targets) have been attached. The camera image is digitised (Fig. 2) and the resulting grey scale values (0 to 255) of each pixel stored in a "frame buffer". It is then possible to produce a grey scale (contrast) diagram for every horizontal scan line and for every vertical column of pixels. The operating software uses the frame buffer data to automatically detect the targets and follow them during testing. The targets produce rapid contrast in grey scale and this data is differentiated to produce distinct peaks. A zone (or "measuring area") is defined around these values in the frame buffer so that only data related to this area is further processed. Several scan lines can be selected to obtain a mean value, resulting in better resolution and accuracy. The resulting length is finally transmitted to the PC.

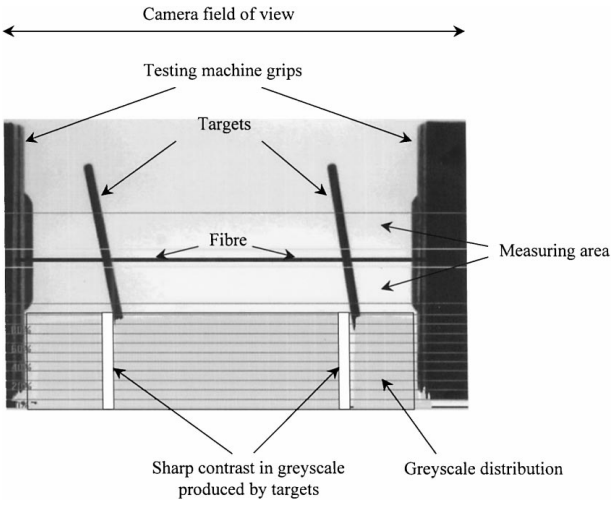

Figure 2 Digitised camera image during testing (camera field of view = 80 mm).

After all the selected measurements processes have taken place, the next frame is scanned, the frame buffer data refreshed and the measurement cycle repeated. If the scan speed is greater than that of the PAL system (25 Hz), the same data is re-analysed and the average values recorded.

The testing of ceramic fibres required very particular settings. The distance between the camera (kept absolutely horizontal) and the specimen had to remain constant during testing, as any movement resulted in a modified image size that was interpreted by the operating software as a change in strain. Therefore the specimen had to be perfectly aligned and rigidly fixed to the testing machine in order to achieve accurate and reliable results.

The samples were lit from the back and care was taken to ensure a constant intensity of illumination and provide an even light distribution in the background. A light source made of two halogen tubes was used for this purpose (Fig. 1). A more consistent light distribution was achieved by placing a series of filters made from frosted glass and tracing paper between the light source and the specimen.

Thin strips of paper (less than 1 mm wide) were used as targets. They were glued onto the specimen with a minimum amount of cyanoacrylate adhesive or "superglue" (RS, UK) to avoid any influence on the mechanical properties of the specimen. The targets projected on both sides of the specimens and the measuring area was split into two separate halves around the fibre. Targets were slightly tilted (approx. 5°) for better accuracy, as shown in Fig. 2. This set-up procedure ensured a maximum grey contrast between the targets (black on the digitised image) and the background, which was kept very bright by correctly setting the camera lens diaphragm.

Optimum results were found for a camera field of view on the digitised image of approximately 80 mm. The minimum theoretical resolution that can be obtained is then 0.6 μm. This resolution was greatly improved by increasing the number of scan lines, i.e. enlarging the measuring area. Due to the absolute method of measurement utilised, the initial distance between the targets did not need to be precisely set for each specimen. However, for stiff materials having very low strain rates such as ceramics, the ratio between the initial distance between the targets (ℓ_0) and the camera field of view should be kept as large as possible (greater than 1 : 2). Hence for a camera field of view of 80 mm, ℓ_0 was therefore set larger than 40 mm.

The testing machine was fitted with a 50 N calibrated load cell. A 16 Bit analogue/digital interface allowed

the operating software to record the load simultaneously with strain. The card was configured to operate in a bipolar mode over a ± 5 V maximum range. The load signal was calibrated by setting the voltages at zero load and full-scale load.

Application software (Messphysik Laborgeräte GES.m.b.H., Austria) was used to process the data captured by the PC during testing. After the test and specimen parameters had been entered, the selected results, e.g. Young's modulus and tensile strength, were automatically calculated from the raw experimental data (strain and load).

3. Experimental procedure

Silicon carbide monofilaments (Sigma Verbundwerkstoffe, Germany), 100 μm in diameter, were tested first to calibrate the system described in the previous section. The manufacturer reported values of 400 GPa and 3500 MPa, for Young's modulus and tensile strength respectively. However, the fibres used in this study were uncoated and very sensitive to handling damage. Specimens were carefully manipulated with tweezers but any handling produced surface microcracking, which lowered the tensile strength. This problem has been overcome in the last few years by applying carbon coatings to the fibres [10]. This operation makes the fibres handleable and preserves the initial strength. This discrepancy in strength due to handling is very important, and values lower than 2000 MPa have been reported by Shatwell. Because of the sensitivity of uncoated Sigma fibres to experimental conditions, measured tensile strengths were not compared with any reference value.

Both extremities of the fibres (approx. 1 cm of the fibre length) were glued onto the accurately aligned grips of the testing machine by using cyanoacrylate adhesive. Care was taken to ensure a good alignment of the specimens. A cyanoacrylate activator (spray) was used to increase the speed of bonding between the ceramic specimen and the metallic grips. A very strong bond was maintained in seconds. This method presented several advantages:

– cyanoacrylate was strong enough to avoid any slipping of the fibres and keep them rigidly fixed throughout testing,
– positioning the specimens could be accurately reproduced,
– no sample preparation was required,
– the mounting of fibres was fast and therefore the duration of a test was reasonably short (less than 10 minutes).

Three batches of at least 10 filaments corresponding to different initial lengths were prepared: 80, 120 and 160 mm. The effect of the crosshead speed on the results was also studied by fixing the specimen length (120 mm) and varying the speed from 0.1 to 0.6 mm/min. Commercially available polycrystalline alumina fibres (Nextel^{TM} 610, 3M, USA), 11.5 μm in diameter, were also tested following the experimental procedure previously described. The results could be directly compared to values found in the literature [11]. 25 fibres were tested and their length was approximately 120 mm. The crosshead speed was 0.2 mm/min. In all cases, the targets were placed on the specimen with an initial separation distance greater than 50 mm for better accuracy. All the measurements were performed at room temperature.

4. Results and discussion

A typical experimental stress-strain curve is shown in Fig. 3. All the ceramic fibres tested in this study were characterised by a linear elastic deformation followed by rupture. The Young's modulus was automatically determined by regression and the best straight line fit was calculated using the least mean squares method. The upper value was taken at 90% of the value at which the slope of the regressional line reduced by 10% from its calculated maximum. The lower limit was taken at 20% of the upper value. The tensile strength was calculated after determination of the maximum load before rupture and by entering the specimen dimensions in the post-processing software.

4.1. Silicon carbide monofilaments

4.1.1. Influence of the crosshead speed

The minimum programmable crosshead speed of the testing machine was 0.1 mm/min. Two other values (0.2 mm/min and 0.6 mm/min) have been used to test the sensitivity of the video extensometer. The values of Young's modulus and tensile strength obtained from the different test speeds are given in Figs 4 and 5.

The results obtained for Young's modulus and tensile strength were very consistent, with little variation on changing the crosshead speed from 0.1 to 0.6 mm/min. Very good agreement was found between the Young's moduli experimental data and the expected values. Tensile strength values (2100 to 2600 MPa) were lower than 3500 MPa, as expected from surface microcracking caused by handling [10].

Hence the crosshead speed had no effect on the mechanical properties of SiC monofilaments, between 0.1

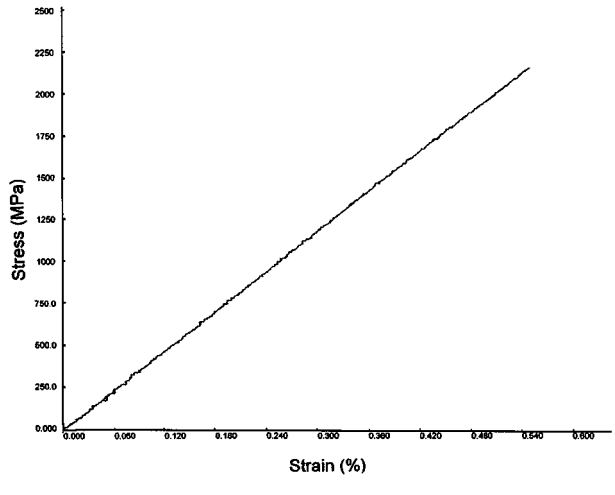

Figure 3 Typical experimental stress-strain curve (SiC monofilament).

TABLE I Mechanical properties of Nextel^{TM} 610 alumina fibres

Young's modulus (GPa)			Tensile strength (MPa)		
Experimental mean value	Standard deviation	Expected mean value	Experimental mean value	Standard deviation	Expected mean value
384.1	12.3	380	3132	296	3200

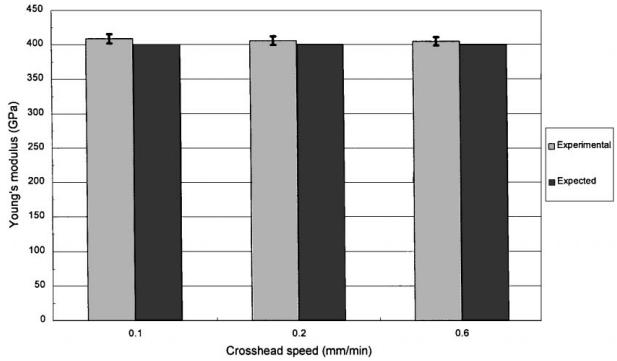

Figure 4 Effect of crosshead speed on Young's modulus of Sigma SiC monofilaments.

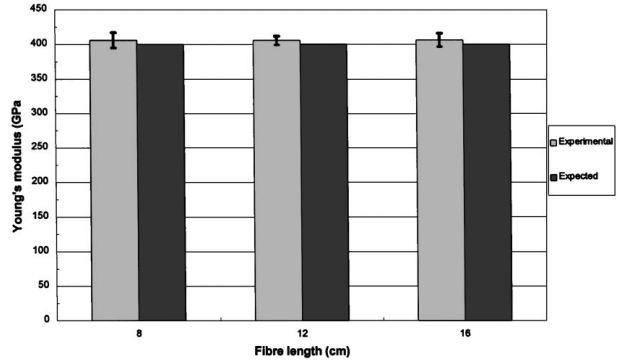

Figure 6 Effect of fibre length on Young's modulus of Sigma SiC monofilaments.

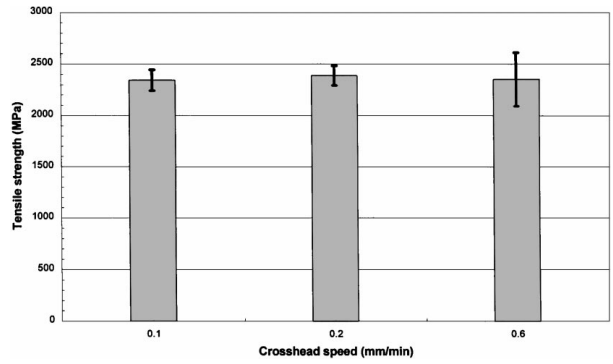

Figure 5 Effect of crosshead speed on tensile strength of Sigma SiC monofilaments.

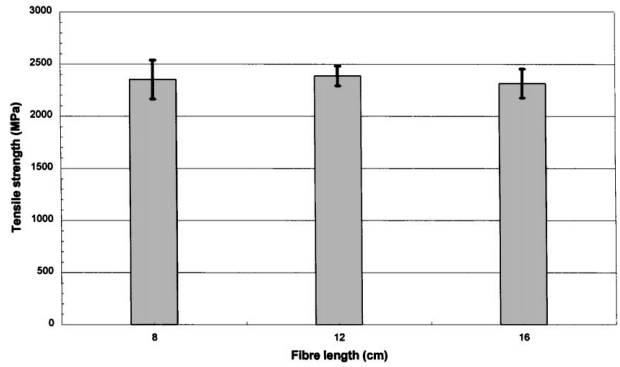

Figure 7 Effect of fibre length on tensile strength of Sigma SiC monofilaments.

and 0.6 mm/min. 0.2 mm/min will be used in future experiments.

4.1.2. Influence of the fibre length

Three different fibre lengths were now studied: 80, 120 and 160 mm. The crosshead speed was maintained at 0.2 mm/min and the initial distance between the targets was kept between 40 mm and 60 mm in all cases.

The results are shown in Figs 6 and 7. The mechanical properties were similar to the previous results, and no influence of the specimen length could be noticed. Therefore, testing fibres with the video extensometer did not require specific specimen lengths to get reproducible results. However, due to the configuration of the testing machine, fibres could not be shorter than 80 mm.

4.2. Polycrystalline alumina fibres

The results, listed in Table I, showed very good agreement between the experimental mean values and the expected data (less than 2%). The variation coefficients of the experimental Young's modulus and tensile strength data are 3.20% and 9.45%, respectively. The second is somewhat high, but in excellent agreement with results discussed by Wilson [11].

5. Conclusions

The mechanical properties of 100 μm diameter SiC fibres were found to be independent of the crosshead speed and length of the specimen. However, the optimum experimental conditions were defined for a crosshead speed of 0.2 mm/min and a minimum fibre length of 80 mm. Results obtained under these optimum conditions on commercial polycrystalline alumina fibres (Nextel 610^{TM} from 3 M) were satisfactory.

This study showed how video extensometry could be successfully used to record elastic deformations and brittle failure of ceramic fibres, using a simple specimen preparation method. This non-contacting technique was characterised by a direct and accurate method of measurement of the load and strain parameters during tensile testing. Its reliability and practicability have been demonstrated by testing SiC monofilaments and alumina fibres, and comparing the results for Young's modulus and tensile strength with known values. Few

limitations have been found and the potentialities of the system may be exploited further.

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