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Evaluation of the creep behaviour of the carbon fibre in an unidirectional pultruded reinforced composite using nano-indentation technique

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Abstract

The interfacial strength (IFS) between carbon fibres and polymeric matrices has important implications for the mechanical properties of composite materials, which can be evaluated by a push-out method using an instrumented nano-indentation machine to assess the fibre's movement under increasing load and constant load (creep). In this paper, the nano-creep behaviour and time-dependent properties of carbon fibres in a reinforced composite were investigated in the first time. The indentation displacement and indentation creep rate of the fibre in the composite were measured at different testing conditions in terms of loading rate, peak load, holding time, elevated temperature (room temperature, 50 and 100°C). Berkovich and cone-shaped diamond indenters were used in the creep tests. The cone-shaped indenter had some advantages over the Berkovich indenter in terms of an extended range of displacement before interfering with the surrounding resin. A single fibre in a thin slice required a critical vertical load to be pushed out under the continuous loading mode, which was strongly linked to the thickness of the composites sample, the location of the fibre in the resin and other environmental factors such as temperature. Both the displacement and creep rate reduced with the increase of temperature due to the resistance caused by the expansion of the fibre and the resin. The nanoindentation creep test results were analysed by an instrumental logarithmic software, and it was found that the creep strain rate sensitivity parameter increased with the load and the holding time but decreased with the increment of the test temperature.

Key words: Creep; elevated temperature, unidirectional composite; carbon fibre; nano-indentation

1. Introduction

The properties of interface between fibre and resin often influence significantly the composite performance in all types of composites, such as failure mode and the fracture toughness of composites. Interfacial debonding is an important mechanism of energy absorption during the failure of a composite. Intensive work has been done to understand the influence of the interface on the mechanical behaviour of fibre reinforced composites [1]. Nanoindentation enables local probing of spatial variations in mechanical properties, time-dependent behaviour and strain rate dependence, and information on interfacial fracture tendency and mechanisms may be obtained depending on the location of which the nanoindentation tests were made [2]. Multicycle grid nanoindentation tests can be applied to study the induced damage mechanisms by analysing the elastic deformation as indentation energy absorbing mechanism right after pop-in [3]. Similar strategy has been used to investigate the viscoelastic behaviour of different area of flax fibres [4] and load and displacement of the PEEK and CFs reinforced PEEK [5]. Recently, a push-out method has been developed by the authors based on nanoindentation to assess the debonding mechanism of carbon fibres in an epoxy matrix and investigate the viscoelastic response of carbon fibre [6]. Generally, the tests are carried out by continuously increasing load until debonding or failure is detected, however, the creep behaviour of the fibre under a load lower than the critical load is also very important to evaluate the mechanical strength between the fibre and the resin. The dwell time of loading (and also unloading) is usually not considered in the evaluation of elastic properties. However, time-dependent material properties can influence the loading diagram from which elastic properties are extracted [7]. Chudoba and Richter reported that the creep effect sometimes led to a net increase in indenter depth with decreasing load during the early stages of unloading [8]. Lu et al. [9] validated the techniques of measuring linear creep compliance in the glassy state using nanoindentation with the Berkovich and spherical indenter tips and proposed methods to measure the local surface creep compliance of time-dependent materials. Yang and Zhang [10] suggested a simple Kelvin model to describe the indentation elastic-viscoelastic-viscous behaviour of polymers by using a flat-ended punch indenter and Berkovich tips. Wang found that the adhesive polymers showed higher reduced elastic modulus (Er) and hardness (H) at elevated temperatures as compared to room temperature [11]. Coronado et.al found that interlaminar crack initiation and propagation under mode-I with static and fatigue loading of a carbon-fibre epoxy composite had the highest stiffness values at 9 °C, and less fracture energy was required to initiate the delamination when the temperature decreases[12]. The viscoelastic behaviour of the

T700GC/M21 composite material depended on the temperature and strain rate, and the shear modulus increased with the decrease of the temperature [13].

The creep behaviour of the bulk composites materials at different temperatures, orientations of carbon fibre and humidity have been reported by different groups [14], [15], [16], [17], and there is a lot of reports on the interfacial strength between the carbon fibre and resin like pull-out [18], push-in/push-out [19], microbond etc.[20]. However, there is no report on the creep behaviour of individual fibre in a composite at a micro-level. In this research, a methodology based on using the instrumented nano-indentation machine to investigate the creep behaviour and time-dependent properties of the individual carbon fibre in a carbon fibre reinforced composite has been set up. A loading-dwell-unloading protocol was designed to evaluate the creep behaviour. Different shaped-diamond indenters were used in the creep test and the effect of different testing conditions in terms of loading rate, peak load, holding time on the creep behaviour of the fibres at room and elevated temperature were studied.

The logarithmic method and the Boltzmann integral method were used by Chen to analyse and fit the creep data collected by NanoTest platform to study the time-dependent behaviour of a PP at different temperatures[21]. The Boltzmann integral method required more variables, more time and a careful selection of the initial value which could degrade the accuracy of the solution[22]. With the logarithmic method, although only measures of extent and rate were used to describe the visco-deformation behaviour, in practice the creep curves normally showed a quasi-logarithmic form, therefore it could predict the creep response over a relatively long time, thus producing an excellent fit to the raw data. In the past, this method has been used for a range of polymer systems to identify changes in creep behaviour in our group, therefore it is used in the current research to analyse the creep data.

2. Material and Methods

2.1 Material and sample preparation

A commercial carbon fibres reinforced unidirectional composite rod (Toray Rebar S12) containing T700s fibres of approximately 7 μm in diameter. It has an elastic modulus between 140-150 GPa, and the onset glass transition temperature (Tg) is 84.03 °C and the midpoint is 104.1°C as tested earlier [6]. The thin disc piece (<1 mm) was cut and attached to an aluminium stud and ground/polished on both sides progressively to 28-46 μm thick. The piece was then stuck to a home-made holder (Figure 1a/b) with grooves etched by Electrical discharge machining (EDM) with a width <100 μm and depth ~3μm (Figure 1c) and narrower grooves etched by a Lasea L5 laser micromachining centre using nanosecond/ femtosecond lasers (Figure 1 d/e). The designed grooves had a width of 25 μm and a depth up to 12 μm (Figure 1e). Three samples with thickness of 28-30, 32-34, 42-46μm were used in the test.

2.2. Nano-indentation creep test

Creep refers to the time-dependent deformation of material at a constant stress level lower than the yield stress [23]. In an indentation creep test, a constant load is applied to the indenter and the change in indentation depth (displacement) is monitored as a function of time. Indentation creep rate is determined according to Equation 1 which is a ratio as follows [24]:

$$creep\ rate = \frac{d_1 - d_0}{d_0} \qquad (1)$$

Where d_0 is the indentation depth (in nm) at time of reaching the peak load (which is kept constant) and d_1 is the indentation depth (in nm) at the end of dwell time of holding the constant test, and the creep displacement is the difference of displacement (d_1 -d_0)**.**

A Micro Materials NanoTest system was used for the nanoindentation creep testing, and two diamond indenters were used: a Berkovich diamond indenter and a specially made cone-shaped indenter. Firstly, a suitable area (i.e. between the two solid lines in Figure 1c&d) was identified under optical microscopy, followed by an individual fibre selected for the specified test. Indentations on selected individual fibre were load-controlled to different peak loads (10-50 mN) at various loading rates (1 and 5 mN/S) for different durations (5s, 60s, 120s and 300s) to investigate the creep and time-dependent properties. All the tests were loaded from an initial load of 10 μN to a peak load at a specified loading rate and recorded at an interval of 0.05 second. The test was normally carried out at least three times under same conditions at room temperature, 50°C and 100 °C, and an average creep displacement was used in the statistics. The effect of the thickness of the disc (28 μm to 46 μm) was also investigated. The parameters involved and the denotation of the code sequence in the experiments are listed in Table 1. For example, sample C100D35-1-60-1 is the first creep test carried out using a cone indenter (C) at a temperature of 100 °C (100D) under a load of 35 mN with a loading rate of 1 mN/s and hold for 60 seconds (35-1-60). Sample code like B30-1-60_2 without specified temperature is a default room temperature test.

The fibres in the composite after creep tests were observed via the onsite microscopy or by a JEOL 7000 SEM after unloaded from the holder.

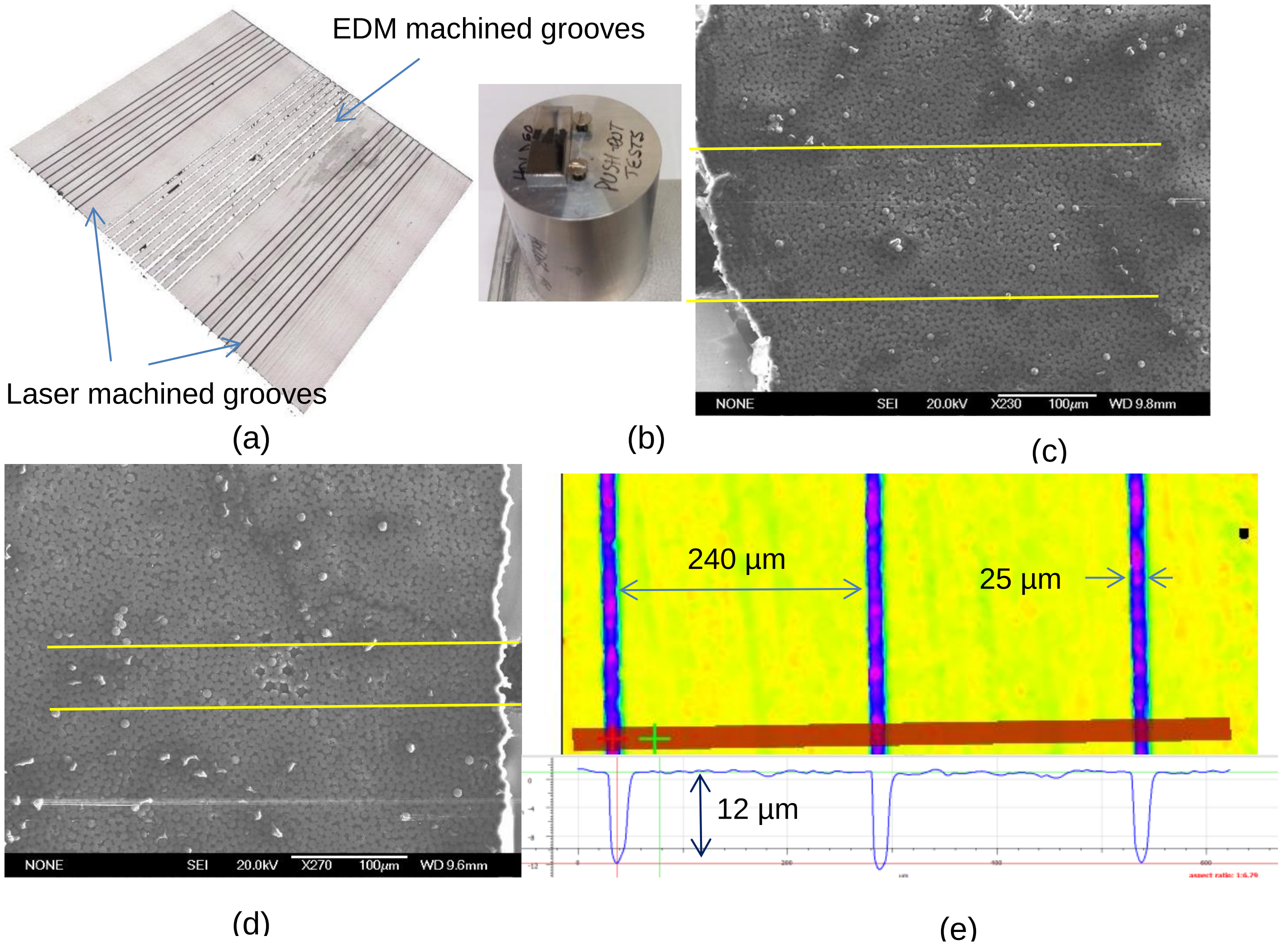


Figure 1. The composite discs and the holder (a) sketch of the surface of the holder, (b)the disc on the holder (c) disc on the EDM machined groove, (d)disc on the laser machined groove after creep test and (e) the geometry of the laser machined grooves.

Table 1. Parameters and denotation of the sample code for the creep experiments.

Creep Experiment	Indenter
Indenter	B for a Berkovich indenter and C for a cone indenter
Temperature	RT (default):23°C, 50D: 50°C and 100D:100°C
Load (mN)	10, 15, 20, 25, 30, 40, 45, 50.
Loading rate (mN/s)	1 and 5
Holding time (Second)	5, 60, 120, 300

3. Experimental results

3.1 Typical load-displacement curve of the push-out test

In the push-out test, the load was applied on a single fibre using the diamond indenter and a counter plate was used to support the thin disc sample. The shape of the force-displacement curve obtained is illustrated in Figure 2 which has a similar curve as reported in [25]. There was an initial non-linear stage (OA) until a conformal contact was reached between the indenter, the specimen and the supporting plate especially, followed by a linear elastic region (Figure 2, AB). As the load increased, cracks initiated and propagated along the interface, producing the non-linear section around point B. Debonding of the fibre was marked by a constant load (Point B to C) with increased displacement, and this load corresponded to a critical load (P). The average interfacial shear strength (IFSS) at the fibre/matrix interface is given by:

$$IFSS = \frac{P}{2\pi re} \quad (2)$$

where P is the applied load, r is the fibre radius, and e the sheet thickness. Between points C and D, the contact between the indenter and the surrounding matrix resulted in a further increase in load until a peak load was reached. Further creep of fibre can be seen from D to E. For the creep test, a load lower than the critical load (P) was normally chosen as the peak load. Therefore, the displacement from B to D was not shown in a creep result. The remaining displacement OF was related to the single fibre movement. The elastic deformation returned after the load was removed (EF).

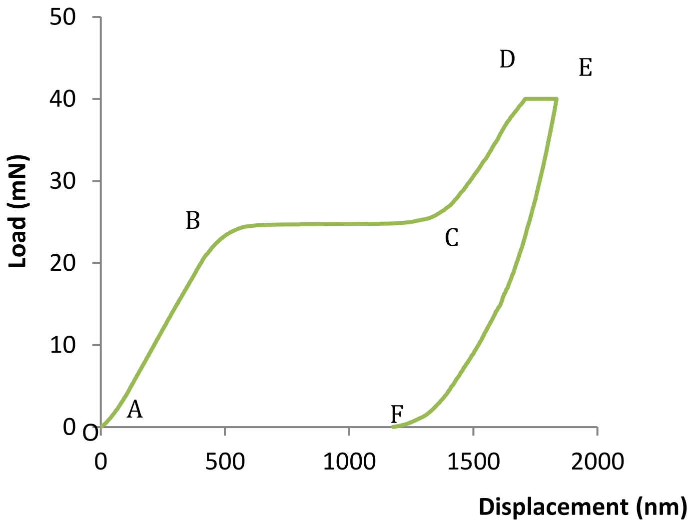
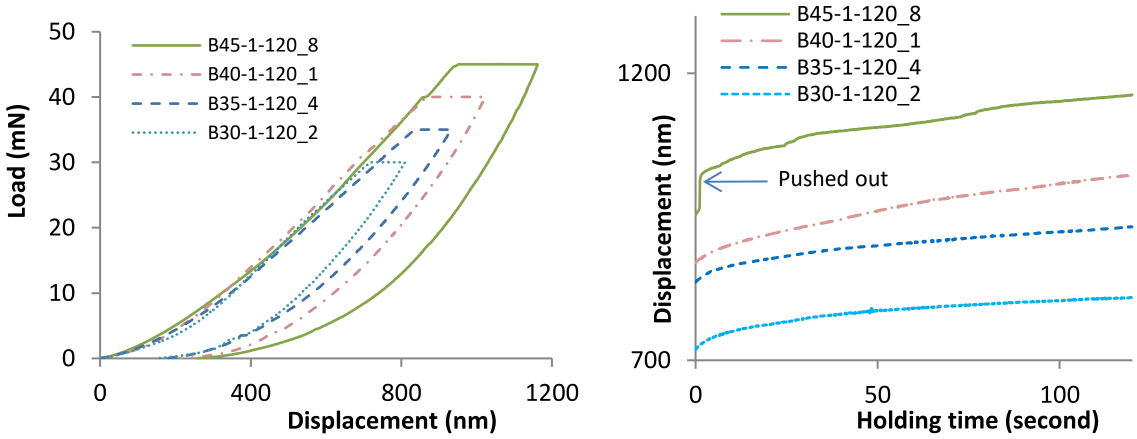


Figure 2. A typical load against displacement curve on a single fibre in carbon fibre composite disc (28 µm thick) using a Cone indenter (C40-1-60)

3.2 Creep behavior of the individual fibre in the composite under different conditions

The evolution of load against displacement curves of a single fibre under different peak loads using Berkovich indenters is shown in Figure 3a. The Berkovich indenter has sharp and well-defined tip geometry, so the initial displacement has a non-linear increase region up to 200 nm which is correlated with the elastic and plastic deformation of the fibre until a conformal contact with the indenter. With the increase of the applied load, a linear increase region of displacement against the load can be seen. The linear growth remains until the peak load (30/35/40/45 mN) is reached. The change of the displacement at different peak loads is shown in Figure 3b, and it can be seen that the higher the peak loads, the larger the displacement at holding time. This is in agreement with Battisti's work and most of the displacement was elastic behaviour especially at lower load and onset of cracking between fibre and resin could be found at higher load which led to increase of elastic displacement and crack propagation and slipping of the fibre [26]. At 45 mN, a jump in displacement occurred during the holding period as shown in Figure 3b, suggesting the failure of the interface and the fibre was pushed out at this load (B45-1-120-8). The shear strength was calculated at about 24 MPa for this 42 µm thick disc. The displacement of fibres increased further by extending the holding time. The average critical load was about 46 mN for this thickness.

Further tests on a thinner disc (34 µm) for a shorter holding time (60 seconds) had the same trend as seen in Figure 4. The creep rate was calculated based on equation 1 and the creep distance at peak load both increased with the load, despite being more scattered at a higher load. The large range of the data at the load of 40 mN was due to the fibre being pushed out before 40 mN for some tests, as well as perhaps the uneven thickness of the slice. The critical load for this 34 µm thick disc was around 40 mN. Clearly, by extending the holding time, both the creep rate and creep distance increased.



(a) (b)

Figure 3. (a) Typical load against displacement curve of a single fibre in a 42 μm thick disc with different peak loads in a loading rate of 1 mN/s and hold for 120 seconds at peak load, (b) the displacement of the indenter at peak load against the increment of time at dwelling period.

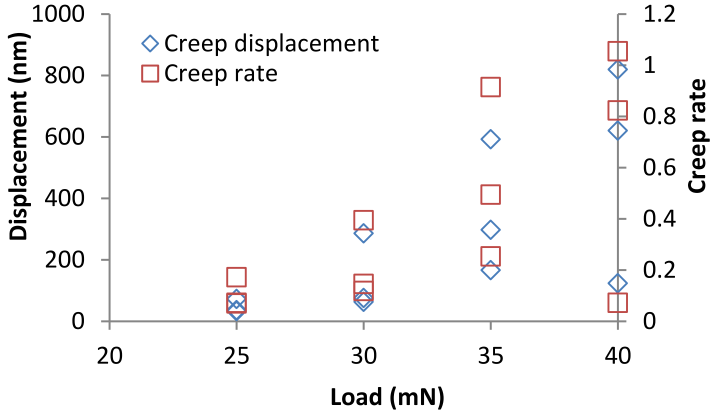
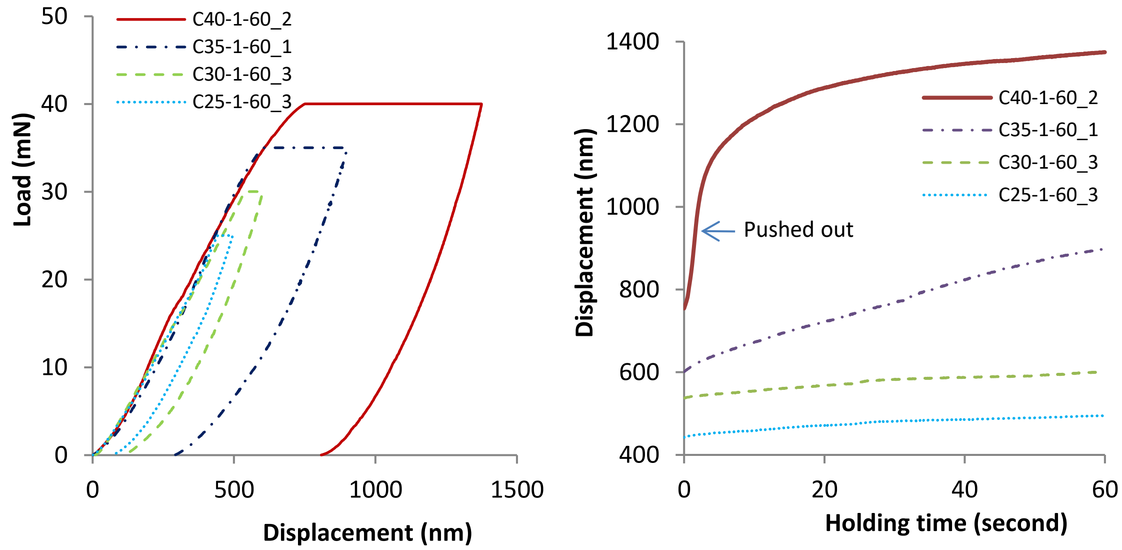


Figure 4. Displacements (left) and creep rate (right) of the single fibre in a 34 μm thick disc applied different peak loads in a loading rate of 1 mN/s for 60 s using a Berkvoic indenter.

As displayed in Figure 5a, when a cone-shaped indenter was used, the non-linear increase region was reduced significantly to under 20 nm, which indicated a rapid conformal contact with the fibre. With the increase of the applied load, a linear increase region can be seen until the peak load reached. Once the peak load was reached and kept, a plateau of displacement was produced. As seen in Figure 5a&b, the higher the peak loads, the further the displacement. At the load of 40 mN, a jump in displacement occurred during the dwelling period as shown in Figure 5b, suggesting that the fibre was pushed out at the holding time (C40-1-60-2). The critical load using a cone-shaped indenter was just above 40 mN for a 32 μm thick disc.



(a) (b)

Figure 5. (a) Typical load against displacement curve of a single fibre in a 32 µm thick disc with different peak loads in a loading rate of 1 mN/s and hold for 60 seconds at peak load, (b) the displacement of the indenter at peak load against the increment of time at dwelling period.

The creep rate was calculated based on equation 1, and the initial displacement at the beginning of peak load was recorded and compared in Figure 6 for two different indenters. The creep tests were carried out on single fibres in a 32 µm thick disc with a loading rate of 1 mN/s and hold for 60 seconds at peak load of 30 mN. It was found that the creep rate was generally higher and the initial displacement (D(0)) at peak load was lower when using a cone indenter, while the creep rate was generally lower and the initial displacement was higher when using a Berkovich indenter as shown in Figure 6.

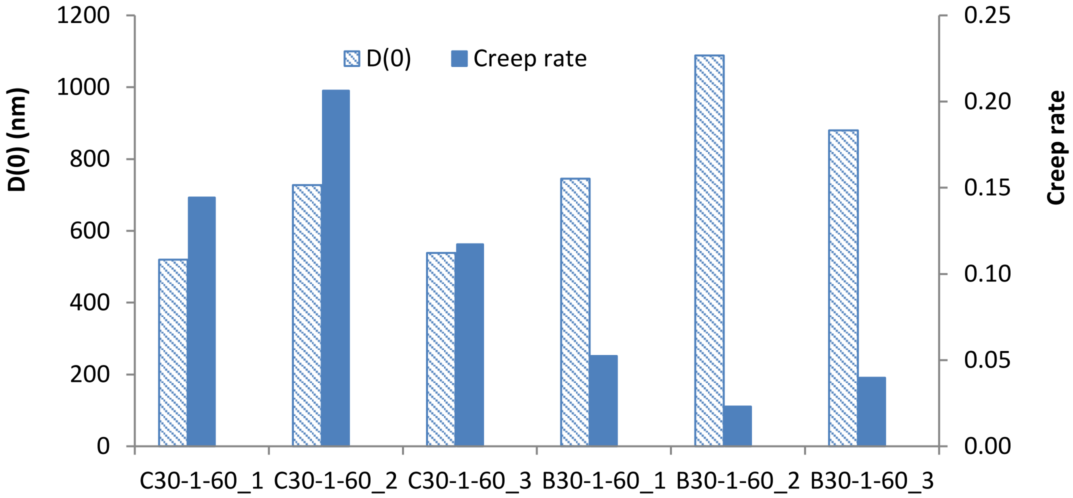


Figure 6. The creep rate and initial displacement of a single fibre in a 32 µm thick disc with a loading rate of 1 mN/s and hold for 60 seconds at peak load of 30 mN for two different indenters

Two loading rates of 1 and 5 mN/s were used to compare their creep rate at two different peak loads of 30 and 35 mN with a holding time of 120 seconds as shown in Figure 7. The impact of the loading rate was limited, and the creep rate increased slightly at lower load (30 mN), but it didn't change much at 35 mN. Unsurprisingly, a fibre in a thin composite disc (34µm) had a higher displacement, and therefore a higher creep rate than that of a thick disc (46 µm) in the same loading conditions as seen in Figure 8.

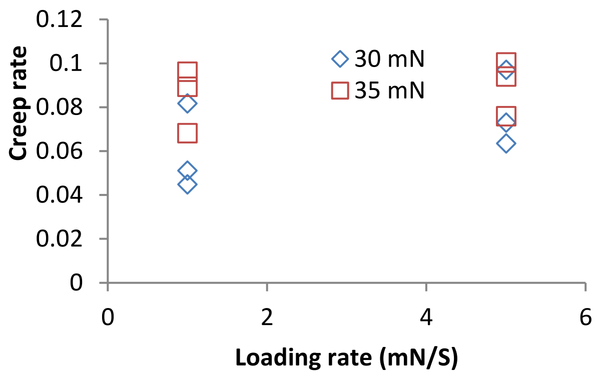


Figure 7. Creep rate of the single fibre in a 34 μm thick disc at different peak loads (30/35 mN) in different loading rate (1/5 mN/s) with a holding time of 120 seconds.

The effect of temperature on the creep behaviour was investigated with a peak load of 35 mN and holding for 60 seconds by the cone-shaped indenter. The thin slice was about 32 μm thick and the loading rate was 1 mN/s. It was found that with the increment of temperature, the initial displacement was higher, but both the displacement at peak load and creep rate of the fibre was reduced (Figure 9a&b). At 100°C, the load-displacement curve became less smooth. This might be due to the expansion of the fibre and resin at the elevated temperature, which increased the friction and resistance between the fibre and resin during the test.

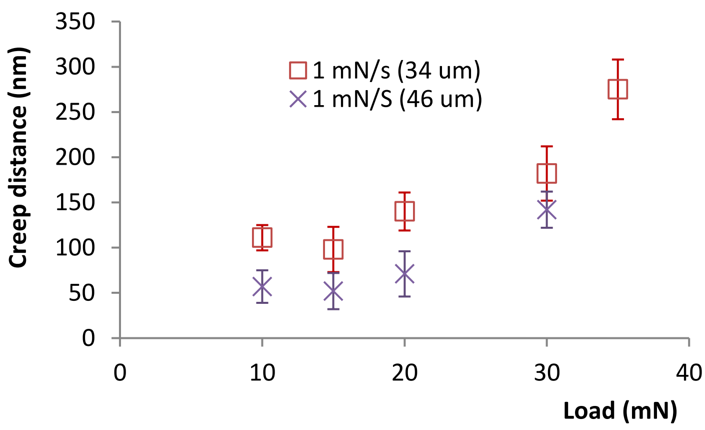


Figure 8. Displacements of the single fibre in a disc with different thickness applied different peak loads in a loading rate of 1 mN/s for 120 second using a Berkovic indenter.

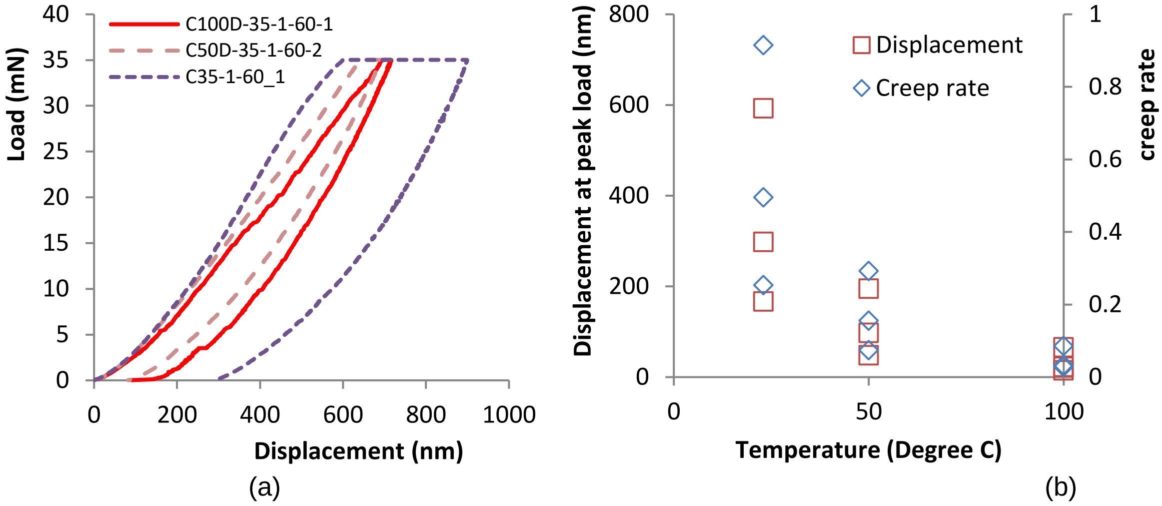


Figure 9. (a) load against displacement curves and (b) Displacements and creep rate with a peak load of 35 mN using a cone-shaped indenter at elevated temperatures (loading rate: 1mN/s and dwell time: 60 seconds, 32-35μm thick disc)

4. Discussion

Interface is a key area which determine the properties of a heterogeneous system like carbon fibre reinforced composite. At micro-level, load transfer through interface has been characterised by: bond strength, interfacial shear stress, critical energy release rate etc.[1], however, creep is still a relative untouched area. In this investigation, the displacement (creep), the creep rate, the initial indentation depth at the time of reaching the peak load have been studied with two different shaped diamond indenters at different loading conditions i.e. peak load, holding time, loading rate and elevated temperatures. A cylindrical flat-end punch is favourable for such a push-out test [27], but a smaller diameter <6 μm required to perform the experiment makes the punch very fragile and difficult to machine. Therefore, in this experiment, the Berkovich diamond indenter and a specially made cone-shaped diamond indenter were used to investigate the creep behaviour of the single fibre in the composite thin disc.

The Berkovich indenter has a well-defined three-sided pyramid tip (Figure 10a/c), with a total included angle of 142.3 degrees and a half angle of 65.27 degrees, measured from the axis to one of the pyramid flats. The diameter of the fibres in the Toray Rebar S12 is about 7 μm, to avoid a Berkovich indenter intercepting with the resin matrix or adjacent fibres, the effective penetration depth is about 1.2 μm (Figure 10a&b), therefore a value over this is invalidated. During the loading process, the sharp tip first contacted and penetrated into the fibre and it gradually pressed the fibre with the increasing contact area, and there was an initial non-linear stage until a conformal contact was reached between the indenter, the specimen and the supporting plate as seen at point A in Figure 11 (B30-1-120_2). The displacement then increased linearly against the load until peak load reached due to the stiffness of the interface or the stable crack growth between carbon fibre and the polymeric matrix [28], and it continued to increase at the dwell time until the unloading process. The value change of the displacement at peak load was closely related to the bonding of the fibre and composite, therefore it can be used to analyse the creep behaviour. The elastic deformation returned after the load was removed, and the retaining displacement was majorly attributed to the dislocation of fibre. The typical impression mark was visible on the fibre after the removal of the load as seen in Figure 10b. Mueller measured the imprint and push-in of the glass fibre by AFM after push-out test and the depth was about 200 nm [29]. A load-displacement curve like B30-1-120_x was frequently found when test piece was placed on the EDM machined wide grooves, and this was majorly due to a span of 100μm led to larger deformation of the thin slice under load resulted in an elastic bending of the thin sample between the supports which was also reported earlier [19]. Although displacement of fibre could be identified, the samples had such behaviour were omitted from the statistics. When the test piece was placed on the narrower laser machined grooves (25 μm), these irregularities can be eliminated in most cases.

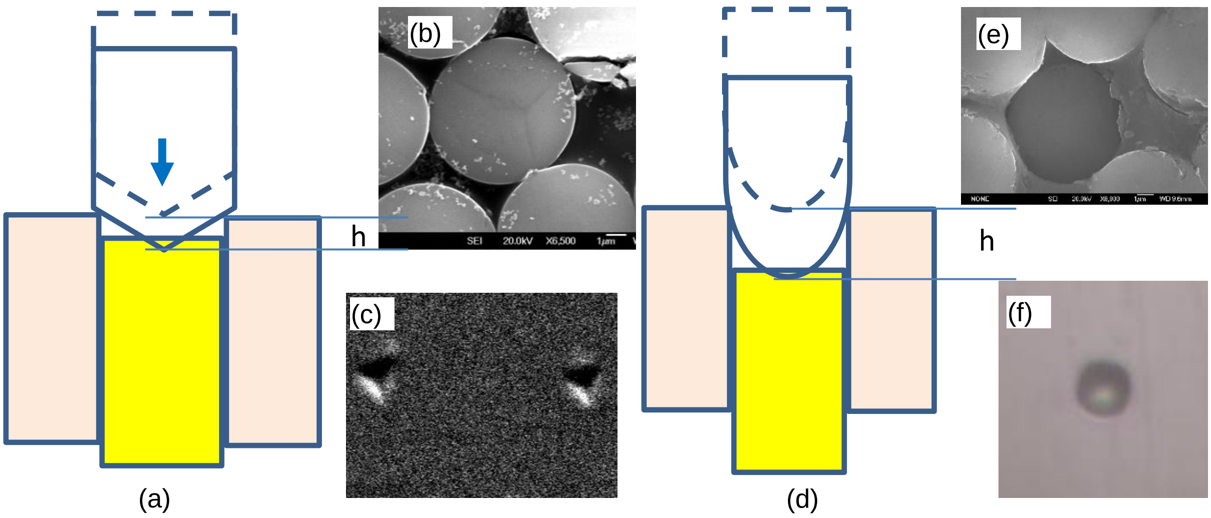


Figure 10. Push-out test using a Berkovich (a/b/c)and a specially made cone indenter (d/e/f) and their indentation on fibre (b/e) and solid surface (c/f)

A specially made cone-shaped indenter with a spherical end surface (Φ5 μm) was also used in this experiment (Figure 10d/f) and the effective depth to avoid it intercepting with the resin matrix and other fibres was extended to about 2.2 μm. In the meantime, the spherical end surface has a large contact area with the fibre which can spread the load evenly and accelerated the conformation of the tip with the fibre (Figure 10d/f). The initial non-linear stage can be minimised even eliminated to point B as shown in Figure 11 (C30-1-60-3) which is similar to using a flat punch [30]. The displacement increased linearly against the load until peak load reached, and it continued to increase at the dwell time until the unloading. There was no permanent impression visible on the fibre after the removal of the load as seen in Figure 10e as examined by SEM.

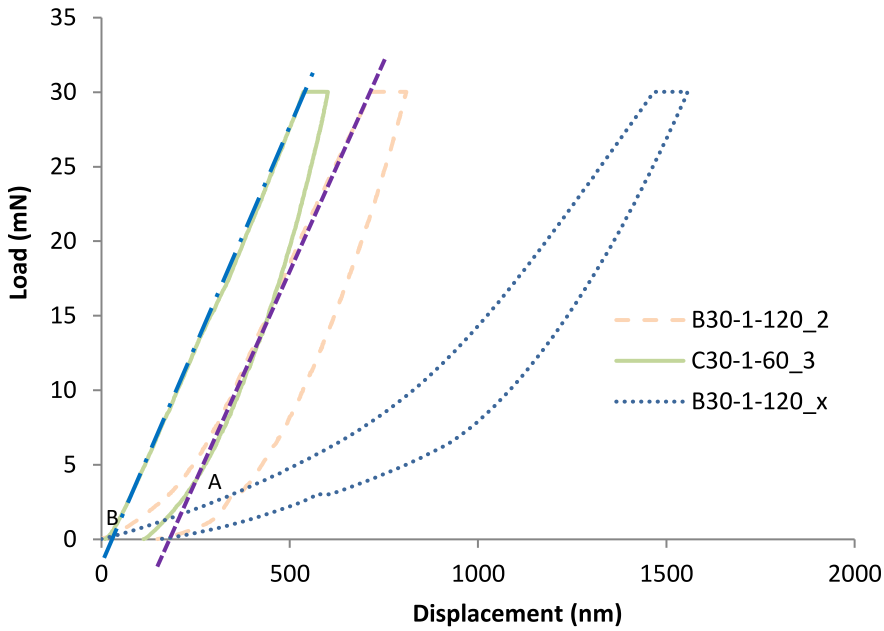


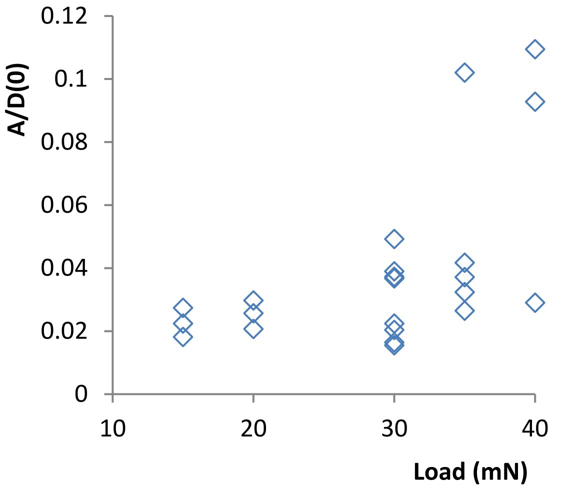
Figure 11. Typical load against displacement curve of a single fibre in a 42 µm thick disc with different indenters (Loading rate: 1 mN/s and hold time at peak load: 60 and 120 seconds).

Several methods can be used to analyse the nanoindentation creep test, including constant depth, constant rate of loading, and constant indentation strain rate methods [12]. The logarithmic method can be used not only for linear viscoelastic materials, but also non-linear viscoelastic materials, and with the quality of the fit, it is possible to predict the creep response over a relatively long time [8]. The indentation creep behaviour was analysed in the instrument software using the logarithmic equation [21]:

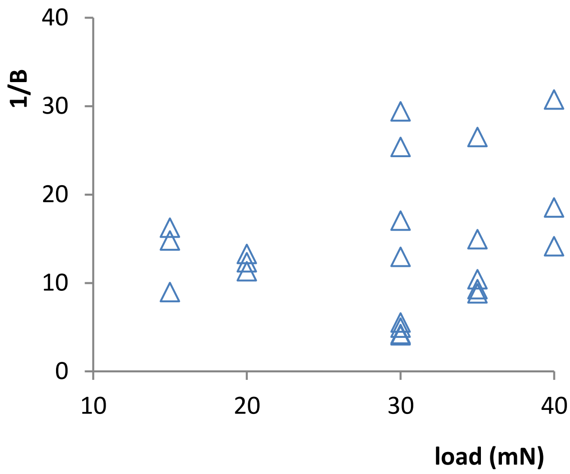
$$\Delta d / d(0) = [A / d(0)] \cdot \ln(Bt + 1) \qquad (3)$$

where A, creep extent, and B, the time constant, d(0) is the initial depth in the hold period, and Δd (creep) is the increase in depth during the hold period. Δd/d(0) is the dimensionless indentation creep strain or creep rate. This equation has proven successful in interpreting loading rate effects and in the determination of the creep strain rate sensitivity A/d(0). The creep strain rate sensitivity increased gradually against peak load and it became more scattered at higher load as shown in Figure 12a. However, the change of the time constant 1/B against load (Figure 12b) is not significant and again, it became more scattered at higher load.

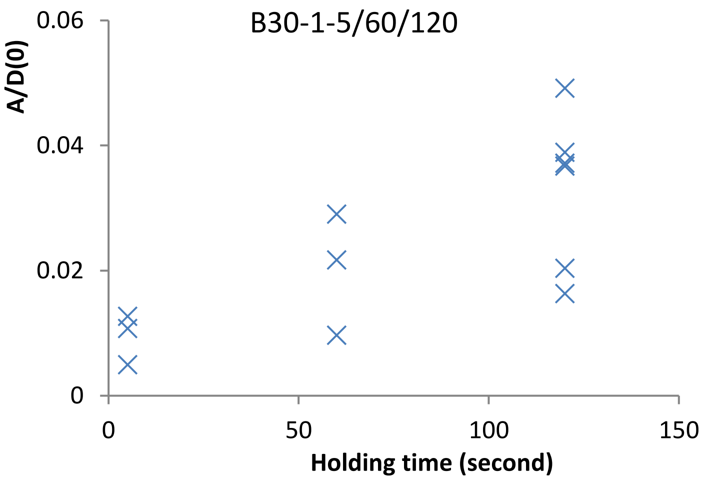
Extending the holding time, the strain rate sensitivity increased (Figure 12c); this had a similar trend to displacement at peak load.



(a)



(b)



(c)

Figure 12. (a) Strain rate sensitivity and (b) time constant against different peak loads using a Berkovich indenter at room temperature (loading rate: 1mN/s and dwell time: 120 seconds, 34-38µm thick disc) (c) Strain rate sensitivity changes against holding time at 30 mN on a 38µm thick disc

As the onset temperature for the glass transition is about 84°C and the midpoint is 104.1 °, the creep tests were carried out at 50°C and 100°C under a peak load of 25/35 mN using a cone-shaped indenter (loading rate: 1mN/s and dwell time: 60 seconds). The strain rate sensitivity decreased with the elevated temperature at both loads of 25 and 35 mN as shown in Figure 13. In the meantime, as seen from the load-displacement curve (Figure 9), the initial displacement was higher at a higher temperature, however, the creep rate and the strain rate sensitivity reduced slightly. This may have been due to the expansion of the fibre and the resin at an elevated temperature or the removal of the moisture in the composite [11]. Tehrani suggested material inhomogeneity and the ongoing curing of sample while being tested at elevated temperatures may also contribute to the decrease of strain rate sensitivity [31].

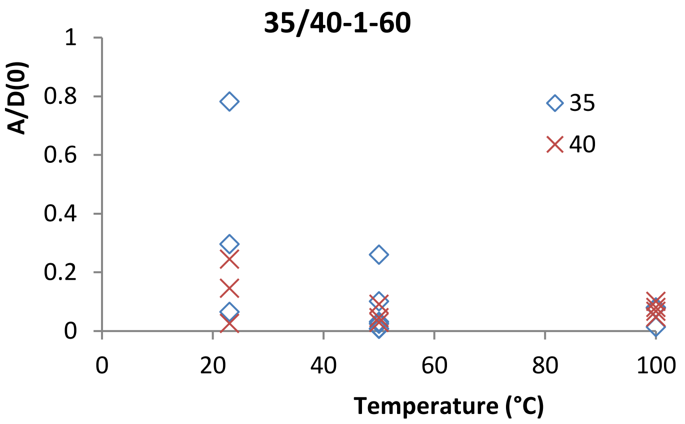


Figure 13. Strain rate sensitivity using a cone-shaped indenter at elevated temperatures (peak load of 35/40 mN, loading rate: 1mN/s and dwell time: 60 seconds, 34 μm thick disc)

By adopting the methodology developed in this work, the mechanical properties of the fibre and polymer matrix in the varied surface modified carbon fibre reinforced composite can be assessed under increasing or constant load at different temperatures and humidity. By investigating the deviation in the load-displacement curve, the associated debonding and the interfacial shear strength (IFSS) and stiffness can be calculated at different loading conditions, and therefore propose the new strategies of strengthening the composite materials.

5. Conclusions

A method to evaluate the interfacial creep of carbon fibre in a polymeric matrix by a push-out method under constant load via an instrumented nanoindentation is set up in this work. It has been used to determine the creep of the unidirectional T700 carbon fibres in a commercial carbon fibres reinforced composite (Toray Rebar S12) at different testing conditions. Initial displacement at peak load, creep (displacement at peak load), creep rate and strain rate sensitivity have been used to evaluate the creep behaviour of the fibre in the composite and the following conclusions can be drawn:

- A thin composite disc piece (<50 μm thick) should be carefully prepared and stuck closely to a holder with grooves. The width of the grooves should be larger than the diameter of the fiber (>7 μm) and accommodate a few fibers (<30 μm for best result) with a depth >3μm to allow the movement of the fiber. Nanoindentation instrument needs an accurate location control to identify the individual fibre before and after creep test.

- Both the Berkovich and Cone indenter were used for the creep test and the latter had some advantage due to its extended range of displacement to 2.2 µm from 1.2 µm before interfering with the surrounding polymer matrix.
- Critical load/stress, when a single fibre was pushed out under a continuous increasing load mode, was used to calculate the interfacial strength between the fiber and the resin. Creep of the fibre at a stress/load lower than the critical load during the dwell time of the push-out test and the displacement/creep rate were closely relating to the loading conditions. Generally, the displacement distance increased with the increment of the peak load and the holding time at the peak load. Fibre in a thin composite disc had a large displacement under the same load conditions.
- The fibre became difficult to creep at elevated temperature and the displacement and creep rate were both reduced with the increment of the temperature (50 and 100°C) due to the resistance caused by the expansion of the fibre and polymer matrix under a cone indenter.
- The nanoindentation creep test results were analysed by an instrumental logarithmic software, and it was found that the creep strain rate sensitivity parameter increased with the load and the holding time, but decreased with the increment of the test temperature.

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References

[1] S. Zhandarov, E. Mäder, Characterization of fiber/matrix interface strength: Applicability of different tests, approaches and parameters, Composites Science and Technology 65(1) (2005) 149-160.
[2] A. Ureña, J. Rams, M.D. Escalera, M. Sánchez, Characterization of interfacial mechanical properties in carbon fiber/aluminium matrix composites by the nanoindentation technique, Composites Science and Technology 65(13) (2005) 2025-2038.
[3] P. Kavouras, D.A. Dragatogiannis, D.I. Batsouli, C.A. Charitidis, Effect of local microstructure on the indentation induced damage of a fiber reinforced composite, Polymer Testing 61 (2017) 197-204.
[4] V. Keryvin, M. Lan, A. Bourmaud, T. Parenteau, L. Charleux, C. Baley, Analysis of flax fibres viscoelastic behaviour at micro and nano scales, Composites Part A: Applied Science and Manufacturing 68 (2015) 219-225.

[5] S. Ji, C. Sun, J. Zhao, F. Liang, Comparison and Analysis on Mechanical Property and Machinability about Polyetheretherketone and Carbon-Fibers Reinforced Polyetheretherketone, Materials (Basel, Switzerland) 8(7) (2015) 4118-4130.

[6] S. Corujeira Gallo, X. Li, Z. Zhang, C. Charitidis, H. Dong, Viscoelastic response of carbon fibre reinforced polymer during push-out tests, Composites Part A: Applied Science and Manufacturing 112 (2018) 178-185.

[7] J. Němeček, Nanoindentation applied to materials with an inner structure, Key Engineering Materials, 586 (2014) 55-58.

[8] T. Chudoba, F. Richter, Investigation of creep behaviour under load during indentation experiments and its influence on hardness and modulus results, Surface and Coatings Technology 148(2–3) (2001) 191-198.

[9] H. Lu, B. Wang, J. Ma, G. Huang, H. Viswanathan, Measurement of Creep Compliance of Solid Polymers by Nanoindentation, Mechanics of Time-Dependent Materials 7(3) (2003) 189-207.

[10] S. Yang, Y.-W. Zhang, Analysis of nanoindentation creep for polymeric materials, Journal of Applied Physics 95(7) (2004) 10.1063/1.1651341.

[11] X. Wang, Y. Li, S. Wang, Y. Deng, D. Xing, S. He, Investigating the nanomechanical behavior of thermosetting polymers using high-temperature nanoindentation, European Polymer Journal 70 (2015) 360-370.

[12] P. Coronado, A. Argüelles, J. Viña, V. Mollón, I. Viña, Influence of temperature on a carbon–fibre epoxy composite subjected to static and fatigue loading under mode-I delamination, International Journal of Solids and Structures 49(21) (2012) 2934-2940.

[13] J. Berthe, M. Brieu, E. Deletombe, G. Portemont, Temperature effects on the time dependent viscoelastic behaviour of carbon/epoxy composite materials: Application to T700GC/M21, Materials & Design (1980-2015) 62(Supplement C) (2014) 241-246.

[14] Y.L. Li, W.J. Chen, M.Y. Shen, C.L. Chiang, M.C. Yip, Study on the Mechanical Properties and Creep Behaviour of Carbon Fiber Nano-Composites, Advanced Materials Research 284-286 (2011) 557-564.

[15] W.K. Goertzen, M.R. Kessler, Creep behavior of carbon fiber/epoxy matrix composites, Materials Science and Engineering: A 421(1) (2006) 217-225.

[16] N.P. Lorandi, M.O.H. Cioffi, C. Shigue, H.L. Ornaghi Jr., On the creep behavior of carbon/epoxy non-crimp fabric composites, Materials Research 21(3) (2018).

[17] A. Chafidz, F.H. Latief, U.A. Samad, A. Ajbar, W. Al-Masry, Nanoindentation Creep, Nano-Impact, and Thermal Properties of Multiwall Carbon Nanotubes–Polypropylene Nanocomposites Prepared via Melt Blending, Polymer-Plastics Technology and Engineering 55(13) (2016) 1373-1385.

[18] J. Zhou, Y. Li, N. Li, X. Hao, C. Liu, Interfacial shear strength of microwave processed carbon fiber/epoxy composites characterized by an improved fiber-bundle pull-out test, Composites Science and Technology 133 (2016) 173-183.

[19] M.C. Medina, J.M. Molina-Aldareguı´a, C. Gonza´lez, M.F. Melendrez, P. Flores, J. LLorca, Comparison of push-in and push-out tests for measuring interfacial shear strength in nano-reinforced composite materials, Journal of Composite Materials 50 (12) (2016) 1651-1659.

[20] W. Tan, F. Naya, L. Yang, T. Chang, B.G. Falzon, L. Zhan, J.M. Molina-Aldareguía, C. González, J. Llorca, The role of interfacial properties on the intralaminar and interlaminar damage behaviour of unidirectional composite laminates: Experimental characterization and multiscale modelling, Composites Part B: Engineering 138 (2018) 206-221.
[21] J. Chen, G.A. Bell, H. Dong, J.F. Smith, B.D. Beake, A study of low temperature mechanical properties and creep behaviour of polypropylene using a new sub-ambient temperature nanoindentation test platform, Journal of Physics D: Applied Physics 43(42) (2010) 9.
[22] M.L. Oyen, Sensitivity of polymer nanoindentation creep measurements to experimental variables, Acta Materialia 55 (2007) 3633-3639.
[23] E.P. Koumoulos, P. Jagdale, I.A. Kartsonakis, M. Giorcelli, A. Tagliaferro, C.A. Charitidis, Carbon nanotube/polymer nanocomposites: A study on mechanical integrity through nanoindentation, Polymer Composites 36(8) (2014) 1432-1446.
[24] S. Mani, C. Palanisamy, M. Murugesan, R. Balasubramanian, Estimation of distinctive mechanical properties of spark plasma sintered titanium–titanium boride composites through nano-indentation technique, International Journal of Materials Research 106(11) (2015) 1182-1188.
[25] G. Lin, P.H. Geubelle, N.R. Sottos, Simulation of fiber debonding with friction in a model composite pushout test, International Journal of Solids and Structures 38(46-47) (2001) 8547-8562.
[26] A. Battisti, D. Esqué-de los Ojos, R. Ghisleni, A.J. Brunner, Single fiber push-out characterization of interfacial properties of hierarchical CNT-carbon fiber composites prepared by electrophoretic deposition, Composites Science and Technology 95 (2014) 121-127.
[27] W. Mueller, J. Moosburger-Will, M. Sause, S. Horn, Microscopic analysis of single-fiber push-out tests on ceramic matrix composites performed with Berkovich and flat-end indenter and evaluation of interfacial fracture toughness, Journal of the European Ceramic Society 33 (2013) 441-451.
[28] J.J. Sha, J.X. Dai, J. Li, Z.Q. Wei, J.M. Hausherr, W. Krenkel, Measurement and analysis of fiber-matrix interface strength of carbon fiber-reinforced phenolic resin matrix composites, Journal of Composite Materials 48(11) (2014) 1303-1311.
[29] W.M. Mueller, J. Moosburger-Will, M.G.R. Sause, M. Greisel, S. Horn, Quantification of crack area in ceramic matrix composites at single-fiber push-out testing and influence of pyrocarbon fiber coating thickness on interfacial fracture toughness, Journal of the European Ceramic Society 35(11) (2015) 2981-2989.
[30] J. Tezcan, S. Ozcan, B. Gurung, P. Filip, Measurement and Analytical Validation of Interfacial Bond Strength of PAN-Fiber-Reinforced Carbon Matrix Composites, Journal of Materials Science 43(5) (2008) 1612-1618.
[31] M. Tehrani, M. Safdari, M.S. Al-Haik, Nanocharacterization of creep behavior of multiwall carbon nanotubes/epoxy nanocomposite, International Journal of Plasticity 27(6) (2011) 887-901.