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Biaxial characterisation of poly(ether-ether-ketone) for thermoforming: A comparison between bulge and in – plane biaxial testing

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Abstract: The biaxial response of extruded PEEK films at conditions relevant to thermoforming has been investigated extensively using a combination of load controlled (bulge test) and displacement controlled (biaxial stretcher) experiments. Results from bulge testing yielded average and maximum strain rate ranges of 2.5 – 5 s⁻¹ and 5 – 18 s⁻¹ respectively, across the forming temperature range. In-plane biaxial characterisation highlighted the anisotropic non-linear viscoelastic behaviour of the films with strong dependence on the yield and strain hardening behaviour on the temperature and strain history at conditions equivalent to the forming process. The combined approach to material characterisation highlights the pros and cons of each test method, the complementary nature of the data generated and the need to use both methods to have a complete data set for developing accurate material models and validated numerical simulations of thermoforming.

Keywords: PEEK; Biaxial testing; Bulge testing; Anisotropy; Thermoforming

1. Introduction

Thermoforming is commonplace for the large-scale manufacture of thin-walled, polymeric products due to the repeatability of the process for complex parts, with the advantage of relatively cheap production costs. Although the thermoforming process can differ greatly in scale and complexity according to the eventual application, parts are intrinsically fabricated through the radiative heating of the thermoplastic sheet above its glass transition temperature (T_g), and then forcing it to rapidly deform and take the shape of a pre-defined mould, either through air pressure, mechanical contact or a combination of both. The material is then allowed to cool and then released from the mould, with the final part created.

During the forming of the product, whilst the polymer is within its easily formable state, it is subject to a complex and dynamic multi-axial deformation. Within industry, thermoforming is currently treated as somewhat of a 'black box' process – with trial and error governing the potential relationships drawn between external process variables and product quality. This is often an unnecessarily expensive and time consuming approach, and therefore a more analytical strategy for understanding such processes is desired, leading to studies involving the characterisation of materials whilst subject to loading in two directions – thus mimicking the deformation of that experienced during the forming process. Such a systematic approach allows identification of material deformation behaviour as a function of strain rate, temperature and mode of deformation [1–4] which can then be subsequently modelled and incorporated into a process simulation.

Poly(ether-ether-ketone) is a semi-crystalline polymer belonging to the family of poly-aryl-ether-ketones (PAEKs), all of which boast excellent mechanical properties along with high thermal stability and chemical resistance. The anticipated suitability of PEEK for the fabrication of thermoformed products is as a result of its large stiffness to weight ratio, a quality deemed particularly beneficial for

high-end applications. These superior mechanical properties exhibited by PEEK have led to its implementation into a wide spectrum of diverse applications in bulk form and as a matrix material for Short Fibre Reinforced Composites (SFRC). A number of studies have examined the uniaxial tensile properties over a wide range of strain rates and temperatures. Initial studies by Cebe et al. [5] investigated the temperature dependency of mechanical properties of PEEK samples of differing thermal histories, at temperatures of 125°C, 25 °C and -100 °C. This work was further complemented by work by Alberola et al. [6], and more recently by El-Qoubaa and Othman [7], examining the effect of strain rate on similarly treated samples. Rae et al. [8], although extensively focussing on the compressive properties of PEEK, investigated the initial uniaxial tensile behaviour approaching the T_g of PEEK specimens. Conclusions drawn by all of the aforementioned studies appear to be consistent, with PEEK exhibiting a profound dependence on both temperature and strain rate – the yield stress of specimens seen to decrease with increasing temperature, and the opposite being true for increasing strain rate. Ductility was also seen to have a positive correlation with increasing sample temperature [8].

Although the above-mentioned literature has provided a comprehensive understanding of the fundamental mechanical behaviour of PEEK in uniaxial tension, to the best of the author's knowledge no studies exist concerning the simultaneous or sequential tensile deformation behaviour in more than one axis. As outlined previously, this mechanical characterisation is of particular concern in order to fully understand the material behaviour during thermoforming processes. Upon knowledge of the biaxial response of PEEK film specimens, there lies potential for a mathematical model to be fitted representative of the observed deformation behaviour, along with possible implementation into a forming simulation capable of the accurate prediction of the final shaped part. A similar modelling approach has been outlined by previous authors through either load-controlled [9–12] or displacement controlled testing [1,13–15] for a wide array of material types.

The aforementioned limitations of unidirectional testing for the characterisation of materials for thermoforming / blow moulding applications has led to a number of institutions building specialised biaxial test rigs capable of deforming polymeric films under biaxial conditions [1,16]; with research at Queen's University Belfast (QUB) focussed on capturing the deformation behaviour of polymers specifically for the development of thermoforming and stretch blow moulding simulations [3,4,17]. Buckley et al. [1] used displacement controlled biaxial testing in the development of a constitutive model representative of the stress-strain behaviour of poly-(ethylene terephthalate) (PET) films. Use of a Flexible Biaxial Film Tester allowed full control of specimen temperature, strain rate and mode of deformation equivalent to those experienced during the industrial forming of bottles and biaxially orientated films, with the resultant stress-strain behaviour used to calibrate a three-dimensional glass-rubber constitutive model. Gerlach et al. [13] then furthered this work by implementing the fitted material model into a finite element (FE) simulation. The authors also explicitly stated the necessity to calibrate material models based on process-relevant conditions. O'Connor et al. [14] used a similar approach to fit a modified version of a constitutive model [18] to the observed biaxial stress-strain behaviour of polypropylene for plug assisted thermoforming applications. The model was also incorporated into FE simulations to predict wall thicknesses of final parts subject to varying forming conditions. Whilst indispensable in understanding material behaviour and dependencies, little consideration for the actual forming history is shown – often limiting the calibration of these models to idealised equal-biaxial (EB) and constant width (CW) deformation modes. This is obviously not the case during the manufacture of complex parts using industrial forming practices where strain rate and mode of deformation will vary arbitrarily depending on the process conditions.

As outlined previously, biaxial deformation is the dominant mode experienced during thermoforming, when subject to air pressure. A complementary material characterisation method analogous to the forming process, such as the load-controlled bulge test, is vital in outlining appropriate strain rates and mode of deformations to be investigated along with validating any potential material models of the deformation behaviour. Bulge testing, sometimes referred to bubble or inflation testing, is a specialised mechanical characterisation technique for the load-controlled study of biaxial deformation of thin films. The test consists of a thin film clamped at its edges over an

orifice, in which an inflating medium of pressurised air or liquid is introduced and subsequent 'bulging' of the thin film occurs. The load and potential temperature control capabilities consider this test particularly applicable for investigation into the induced states of stress during the thermoforming process due to their analogous nature. By subjecting the thin polymer film to forming parameters equivalent to those experienced during forming, this experimental analysis can provide a detailed insight into the deformation behaviour and possible influences of external variables on the forming of the bulge.

Literature has recently emerged concerning the use of bulge testing for the characterisation of polymers for thermoforming processes [19,20]. These studies have identified the analogy between the bulge test and thermoforming although the data produced is yet to be used to characterise and model polymers for FE simulation applications. Other work has directed efforts to using the bulge test to impose a biaxial state of stress onto specimens and then attempt to model this response using constitutive equations. Sasso and Amodio [9] conducted bulge testing on rubber specimens with the experimental data produced used to fit hyperelastic material models within FE simulations of the bulge test. Tonge et al. [12] used the bulge test to characterise the stress-strain response of human skin tissues. Once more, material models of the observed behaviour were validated through FE simulations. Although not directly applicable to this work, these studies demonstrate the potential for biaxial data to be produced using with subsequent material models calibrated to describe the resultant deformation behaviour of this characterisation technique. One limitation of this approach, however, is the lack of knowledge over specific parameters (e.g. temperature and strain rate) in understanding their individual influence on the resultant material behaviour. The combination of several factors acting together creates difficulty in distinguishing the separate effects of these parameters on the stress – strain response, as tests at different temperatures will experience different speeds of deformation. It is here that displacement-controlled testing, with defined uniform strain rate at varying temperatures, has the capability to complement the findings in a load-controlled analysis.

A potential use of both load-controlled bulge testing and displacement-controlled planar biaxial tests was explored by Çakmak and Major [21] by investigating the temperature and loading rate dependency of elastomers. The material behaviour observed during bulge testing was then calibrated to the Mooney-Rivlin hyperelastic material model with stretch rate dependent strain energy density master curves plotted. The authors noted the limitations of the bulge test to sole pressure controlled experiments highlighting the favourability of both load and displacement-controlled testing for the complete biaxial characterisation of materials. While this study was extensive, no direct comparison observed in material deformation behaviour between biaxial testing methods is presented.

Although not within the field of thermoforming, Yan et al. [22] investigated the potential use of a free stretch blow (FSB) test to be used in combination with in-plane biaxial testing to characterise the material response of PET for injection stretch blow moulding applications. 3D Digital image Correlation (DIC) techniques tracked the evolution of the full-field strain history of preforms during forming whilst studying the influence of temperature and air mass flow rate. PET films were then investigated by planar biaxial tests using the probed strain histories. The authors noted the significance of load-controlled analysis for the accurate characterisation of PET and identification of conditions applicable to those experienced during the forming process.

In the present work, we propose two methods of applying a biaxial state of stress on PEEK samples: a load controlled bulge test and a displacement controlled in-plane biaxial test. The ultimate aim of this work is to use both characterisation methods to determine the constitutive response of PEEK films subject to multiaxial stress. Firstly, the bulge test is adopted to generate stress – strain data observed at the pole of the deformed specimen, along with identifying typical strain rates observed during the thermoforming process. In-plane biaxial testing is subsequently used to characterise the stress – strain response of PEEK films as a function of strain rate and temperature, with replication of the loading paths observed in the bulge test used to compare and conclusively validate the observed material behaviour.

2. Materials and Methods

2.1 Material

PEEK samples were cut from an extruded roll of 12 μm thickness, each measuring 76 x 76mm. Gel Permeation Chromatography (GPC) experiments revealed samples of having a number average molecular weight, M_n , of 45,000 g/mol whilst Differential Scanning Calorimetry (DSC), conducted in a previous study [23], calculated the percentage crystallinity of samples at $12 \pm 2\%$. Also noted in the previous work [23] was the identification of a processing window between 130 – 160 $^{\circ}\text{C}$ using Dynamic Mechanical Analysis (DMA), in which it was deemed that between these temperatures a drop in storage modulus by several decades indicated the onset of a ‘formable regime’ before cold crystallisation was observed to occur. Thus in this work, characterisation was conducted in the temperature range of $130 \leq T < 160$ $^{\circ}\text{C}$. A T_g of 150 $^{\circ}\text{C}$ was also determined by the peak in the tan delta value from the DMA.

2.2 Bulge Testing Apparatus and Analysis

The custom bulge test rig is shown in Figure 1(a) designed to test samples of a maximum thickness of 2mm up to a pressure of 8 bar, with a bulge diameter of 50mm chosen to accommodate the size of PEEK samples. The specimen is placed between the upper and lower die, securely tightened by threaded bolts around the circumference. A thermally resistant fluoroelastomeric O-ring is used to create a tight seal to prevent any air pressure leaks from occurring. Temperature control of the specimen was governed by placing the rig in a high temperature ambient environment, continually monitored by a thermocouple placed within the oven. In all tests care was taken to ensure the machine direction (MD) and transverse direction (TD) of all PEEK specimens were aligned with the x and y directions respectively. An arbitrary value of flow rate of 1 bar/s was chosen such that the deformation of the film specimen was able to be sufficiently captured, with testing terminated upon bursting of the PEEK specimen.

Previous literature details the advantages of evaluating full-field, transient, out-of-plane displacements of specimens through DIC techniques [10,11,24]. Two high speed cameras (Photron Fastcam SA1.1) were placed in plane with the custom rig to track the bulging displacement of specimens synchronously, dictated by a random speckle pattern applied manually using a paint marker. Vic-3D 7 software was used for the DIC analysis, with the options adopted in the correlation step shown in Table 1. Due to the transparent nature of the specimens, the background of the lower die was spray painted white in order to create sufficient contrast of the pattern to be recognised by the cameras. A targeted light source was also employed to improve the recorded images. One phenomenon particularly apparent however whilst using targeted lighting on smoothly curved surfaces is that of a highly concentrated area of reflection – known as specular reflection. These polar reflections appear as a spot of highly saturated pixels, and due to the ever-evolving nature of the bulge, can be seen to travel throughout the analysis from the perimeter to the pole. This can be particularly troublesome when conducting DIC analysis as the travelling reflection misleads the analysis into thinking the specimen is deforming locally during incremental correlation, even causing the path of the reflection to be erased during conventional correlation. In order to mitigate this, the technique of cross polarization [25] was adopted using a series of perpendicular linear polarizers placed between the light source and the camera lens. This method obstructs the orthogonally polarised specular reflection from reaching the camera aperture allowing only diffuse lighting to enter. One slight drawback of cross polarization however is loss of light intensity whilst passing through the polarizers resulting in the grayscale images of the specimen under investigation being too dark for post-processing tools to analyse. This was overcome through use of an intensely targeted light source directed towards the aperture of the bulge test rig, sufficiently illuminating the specimen. The adoption of polarisers are shown in Figure 1(b), along with the entire set up allowing control over experimental parameters.

Figure 1. (a) Annotated diagram of custom bulge test rig and (b) Schematic of set up used for bulge testing of specimens.

Table 1. Specifications of bulge test experiments.

Interpolation	Optimized 8-tap
Subset size	31*31 pixel
Step size	3
Filter size	15
Strain Tensor	Engineering

Typically stress values in at the pole of thin walled membranes created by internal pressure are calculated by Eq. (1), assuming the radii of curvatures on machine and transverse directions are equal:

$$\sigma = \frac{PR}{2t} \quad (1)$$

where σ is the biaxial stress in both directions, R is the radius of curvature, and t the actual thickness at the pole of the bulge. Recent work by Min et al. [26] had accounted for potential anisotropic deformation in bulging specimens, in the calculation of principal stresses at the specimen pole (Eq. (2):

$$\sigma_1 = \frac{p \cdot R_2(R_1 - t)(R_2 - t)}{t \left(R_2 - \frac{t}{2}\right)(R_1 + R_2)} \quad (2a)$$

$$\sigma_2 = \frac{p \cdot R_1(R_1 - t)(R_2 - t)}{t \left(R_1 - \frac{t}{2}\right)(R_1 + R_2)} \quad (2b)$$

where R_1 and R_2 are the radii of curvatures in the x and y directions respectively, calculated by polynomial curve fitting the co-ordinates of the deformed surface along the principal directions, as described by authors [26]. All results were calculated as the mean average of three separate tests.

2.3 In – Plane Biaxial Testing

The Queen's Biaxial Stretcher (QBS) is a purpose built biaxial stretching machine capable of replicating temperature, complex deformation modes, and rapid deformation speeds similar to those seen in thermoforming and blow moulding processes. The square specimens, of maximum allowable thickness of 2mm, are held in place by 24 nitrogen-driven pneumatic clamps as shown in Figure 2(a). Once secured, the sample is heated to the desired temperature by two convection heaters, one above and below the sample. Temperature control is provided by thermocouples placed in close proximity to the sample by the pneumatic grips on the left-hand side. Specimens are heated to the target temperature for 5 minutes to allow uniform heating through the thickness. Once the specified temperature is reached, the grips are then driven apart by two servomotors at a user-specified speed, stretch ratio and mode of deformation. The force to stretch the polymer is recorded against displacement on the x and y axis by two force transducers mounted centrally on the grips of each axis. By assuming incompressibility the force data in each direction is subsequently converted to true stress (σ_{true}) by Eq. (3) validated by O'Connor [27]:

$$\sigma_{true} = \frac{7F}{A_0 / (1 + \epsilon_{eng})} \quad (3)$$

where F is the force as measured by the force transducer, A_0 is the original cross-section area and ϵ_{eng} is the engineering strain in the specimen. Figure 2(b) shows the uniformity of deformation experienced during EB stretching of the 12 μ m thick PEEK films, with the grid initially applied to the surface of the unstretched specimen seen to deform equally across the entire area. For all biaxial stretching experiments three tests were conducted per respective temperature and strain rate, with true stress-nominal strain curves plotted as the mean average.

Figure 2(a) Annotated photo showing PEEK film mounted in the Queen's Biaxial Stretcher and **(b)** Equal Biaxial deformation of PEEK specimens (i) unstretched and (ii) biaxially stretched to a ratio of 2 by 2.

3. Results and Discussion

3.1 Bulge Testing

3.1.1 Pole deflection against Time

For Eq. (2) to be valid only deflection near the pole of the bulge is considered. Using the custom bulge test rig described in Section 0, initial testing was conducted to investigate the influence of specimen temperature on the out-of-plane pole deflection behaviour of the induced bulge over time. 3(a) shows a typical image of a mounted PEEK specimen and the contour plots of the uniform out-of-plane deflection evolving when subject to the forming pressure, at a temperature of 130 °C. The investigated temperature dependency of the deflection at the pole of the bulge is shown in Figure 3(b), with samples tested until failure between temperatures of 130 – 155 °C. It is clear from the plot that increasing temperature causes a decrease in overall time to failure, with the highest temperature test terminating twice as quickly as the lowest temperature – with failure displacement equal to 29.33 ± 1.43 mm. Also apparent are the increasing gradients of deflection against time associated with increasing temperature indicating a softer material response. Another point of note in the plot is a 'shoulder' between times of 0.07 – 0.1 s for temperatures below T_g . This can be correlated to the yielding and concurrent relaxation behaviour associated with necking at these temperatures – a phenomenon that is also seen to disappear at temperatures of 150 and 155 °C in the EB stretching experiments.

Figure 3(a) Digital Image Correlation sequence of bulge test images at 130 °C and **(b)** Out-of-plane pole deflection against time for temperatures of 130 – 155 °C until failure of specimens.

3.1.2 Strain against Time

From the Vic-3D 7 software it was possible to determine the incremental nominal strain per image frame in the principal directions at the pole of the formed bulge. This strain measurement in both axes is displayed in Figure 4 at a temperature of 130 °C, until a nominal strain of 1 – with minimal observed deviation between samples showing repeatability of the characterisation technique. A clear disparity in measured strains between principal directions can be seen, a manifestation of anisotropy present within the PEEK samples. Both directions are seen to experience similar deformation until a strain of 0.025 after which, whilst experiencing the same loading conditions, the change in strain in the TD is much greater than that in the MD with time.

Figure 4. Average nominal strain against time in x (MD) and y (TD) directions at 130 °C.

Figure 5 shows the variation of strains against time with specimen temperature in the x (MD) and y (TD) directions respectively. 1 Similar behavioural relationships can be noticed in both directions until a nominal strain of 1, after which noise is introduced into the results indicating local failure. For times less than 0.1s, the change in strain is small indicating the linear elastic deformation of samples. With increasing temperature this elastic region is seen to decrease inducing an earlier onset of the pronounced increase of strain with time – indicative of decreasing yield stress. The time taken for local nominal strains to reach a value of 1 is also seen to decrease with increasing temperature; an expected result associated with the increased formability and soft mechanical behaviour of polymeric materials at elevated temperatures.

Figure 5. Nominal strain evolution in x direction (MD) and y direction (TD) experienced at bulge pole for PEEK specimens between temperatures of 130 – 155 °C.

The evolution of strain rate was probed for each temperature and is displayed in Table 2. It is apparent that increasing specimen temperature induces an increase in average and maximum deformation rate observed in both directions, again due to the increasingly softer material behaviour.

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Table 2. Average and maximum strain rates observed during bulge testing in MD and TD.

Temperature	Machine Direction (x)		Transverse Direction (y)	
	Maximum	Average	Maximum	Average
	Nominal Strain Rate	Nominal Strain Rate	Nominal Strain Rate	Nominal Strain Rate
130	5.02	2.49	8.47	3.23
135	5.23	2.61	8.92	3.33
140	7.11	3.31	11.59	4.05
145	9.36	3.46	12.69	4.27
150	10.40	3.51	14.58	4.42
155	16.31	4.75	18.61	5.31

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3.1.3 Calculation of Stress Curves

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Eq. (2) was employed to calculate the principal stress in orthogonal directions at each time increment observed at the pole of the bulge with the mean calculated hoop (MD) and axial stresses (TD) plotted against their respective strains in Figure 6. It can be seen that increasing specimen temperature leads to an associated decrease in general stress values. Inconsistencies with this suggestion however are discernible when comparing stress-strain behaviour between temperatures of 150 and 155 °C – where post yield stress values are seen to overlap. The reasoning behind such behaviour is hypothesised to be as a result of an increased strain rate effect outweighing the perceived temperature effect. Whilst the specimen at 155 °C is softer, this also causes the material to deform at a higher rate to that compared to the material tested at 150 °C; maximum strain rates of 14 and 18 s⁻¹ observed in comparison to rates of 10 and 16 s⁻¹ seen in 150 °C tests, in hoop and axial directions respectively.

Figure 6. Calculated hoop (MD) and axial stresses (TD) against nominal strain for temperatures between 130 - 155 °C at pole of bulge.

3.2 In – Plane Biaxial Testing

3.2.1 Influence of Temperature

The influence of specimen temperature on true stress-nominal strain behaviour during EB stretching of PEEK film samples for both MD and TD is shown in Figure 7. Specimens are stretched at a strain rate of 4 s^{-1} , corresponding to the average strain rate observed during bulge tests, with the average of at least 3 tests recorded in the MD and TD to failure. For all temperatures a highly non-linear stress-strain response can be seen, exhibiting four distinct behavioural stages. Firstly a linear elastic response, unchanged by specimen temperature, until yielding at a strain of approximately 0.03, then a relaxation in stress precedes cold flow and subsequent strain hardening until failure. Similarity in this deformation behaviour is apparent with that observed in calculated bulge test stress curves shown in Figure 6.

With increasing specimen temperature the yield stress in both directions is observed to decrease, similar to that observed in uniaxial tensile experiments [8]. A prominent increase in the strain at failure of samples above T_g (150°C) is also evident, where samples are stretched from maximum strains of 0.7 to those greater than 1 post- T_g . The gradients of strain hardening regions, in the MD, appear to be unaffected by specimen temperature although for temperatures greater than 150°C there is a slight relaxation thus indicating potential entanglement slippage as a result of increased mobility of polymer chains – similar to that observed for temperatures above T_g for PET [3]. This behaviour manifests itself as a delay in these strain hardening regimes for temperatures greater than T_g . In the TD, strain hardening behaviour is only observable above temperatures of 150°C where below this, decreased ductility only allows for mostly flowing behaviour of specimens to be exhibited before failure.

Figure 7. True stress-nominal strain data for equal-biaxial stretching of PEEK specimens in machine direction (MD) and transverse direction (TD) at a strain rate of 4 s^{-1} .

3.2.2 Anisotropy

A plot of the true stress against nominal strain, in both MD and TD, is shown for the two extreme temperatures of 130 °C and 155 °C in Figure 8, along with associated error bars. Although initial stiffness and yield stresses are equal a disparity in final stress levels between MD and TD is observable. This difference in strain hardening gradients is an indication of inherent anisotropy within specimens, in agreement with the inherent anisotropy observed in Figure 4, as a result of the extrusion process inducing a preferential direction of polymeric chains in the MD. This inherent material characteristic was also demonstrated by a decrease in post-yield stress levels in principal directions, shown in the calculated stress curves from bulge tests in Figure 6. In addition, the true

stress-nominal strain behaviour produced by planar biaxial tests are seen to be highly repeatable with minimal variation evident in both directions, as shown in Figure 8.

Figure 8. Plot of true stress against nominal strain in MD and TD for temperatures of 130 and 155 °C.

3.2.3 Influence of Strain Rate

Figure 9 highlights the influence of increasing strain rate on EB stretching of PEEK samples at 130 – 150 °C. Strain rates in the range of 1 – 16 s⁻¹ were examined based on those discovered in Section 3.1.2, also in agreement with previous authors investigating material characterisation for thermoforming [4]. Similar to increasing temperature, the Young's Modulus and strain hardening gradients of samples remain unchanged across all levels. Overall stress levels however appear to increase appreciably as a result of increasing speed of stretching, likened to uniaxial tensile characterisation of PEEK [6–8]. At a temperature of 150 °C it is also clear that increased speed of stretching results in pronounced reptation of polymer chains – with this most apparent at a strain rate of 16 s⁻¹, between strains of 0.2 – 0.9 with a minimal increase in overall stress levels before strain hardening. One potential hypothesis could be due self-heating effects within the material at elevated strain rates, as observed in previous literature concerning PET [3], although further investigation would be necessary to support this. Without the privilege of planar biaxial testing, these inherent material characteristics would be unknown and thus a combined approach of load and displacement-controlled characterisation is necessary to fully characterise a material in order to understand and model the forming process.

Figure 9. Influence of strain rate on the true stress-nominal strain EB behaviour of PEEK samples at 130 °C, 140 °C and 150 °C in the MD.

3.3 Verification of Stress Curves using QBS

Validation of the calculated stress-strain behaviour observed during load-controlled bulge testing, using Eq. (2 [26]), was achieved using the strain histories from the bulge tests shown in Figure 5. Using the QBS, corresponding biaxial tests were conducted by inputting the respective strain histories in the x and y directions at each specimen temperature, with the average taken of three tests. An overview of the comparison methodology is given in the supplemental documentation for a clearer visual understanding, with the comparison plots of the resultant true stress-nominal strain between biaxial and DIC measurements for temperatures of 130, 145 and 155 °C presented in Figure 10. As is apparent in the figures, reproduction of the initial stiffness, yield stresses and subsequent strain hardening behaviour is captured until early failure of PEEK specimens in the displacement-controlled tests. Anisotropic effects captured in the variation of bulging radii appear to be accurately captured by DIC measurements with their consequent influence on the stress-strain behaviour well modelled by the stress calculation equations.

Figure 10. Comparison between calculated stress curves from bulge test and stretching experiments by QBS at 130 °C, 145 °C and 155 °C.

362 This reproducibility and coherence of both material characterisation techniques allow for the
363 complete understanding of biaxial deformation behaviour of PEEK subject to comparable loading
364 conditions. The load-controlled bulge test is invaluable in understanding the influence of process
365 conditions on the resultant deformation of the material as it provides valuable data on typical strain
366 rates experienced by the material along with its response when subjected to nonlinear loading paths.
367 The test also enables a good experimental idealisation of the thermoforming process and thus
368 provides valuable data that can be used for validating forming simulations. However, the apparent
369 weakness of this characterisation technique is the ambiguity concerning the individual contributions
370 of strain rate and temperature on the material deformation behaviour, as highlighted between
371 temperatures of 150 °C and 155 °C in Figure 6.

372 The displacement controlled biaxial stretching experiments however clearly separate the
373 effects of temperature and strain rate, as shown in Figure 7 – Figure 9. This displacement controlled
374 data is essential for material model development, where the effects of strain rate and temperature
375 need to be accurately quantified and captured.

5. Conclusions

In this work, the biaxial deformation behaviour of commercially sourced PEEK was quantified at temperatures and strain rates relevant to the thermoforming process. DIC measurements focussing on the bulge pole reported an initial disparity between strains achieved in principal directions due to anisotropy produced during fabrication of the material. Temperature dependency of samples were exhibited through increasing gradients of nominal strain against time, along with increasing specimen temperature inducing greater strain rates experienced during the analysis. Average strain rate ranges of $2.5 - 4.75 \text{ s}^{-1}$ and $3 - 5 \text{ s}^{-1}$, and maximum values of $5 - 16 \text{ s}^{-1}$ and $8.5 - 18.5 \text{ s}^{-1}$ were noted in machine and transverse directions respectively over the forming temperature range of $130 - 155 \text{ }^{\circ}\text{C}$. Calculation of true stress curves reported the highly nonlinear, temperature dependent biaxial deformation behaviour of PEEK. Increasing specimen temperature was generally associated with overall decreasing stress levels, until post yield stress levels at a temperature of $155 \text{ }^{\circ}\text{C}$ are seen to approximately equal those observed at $150 \text{ }^{\circ}\text{C}$.

Equal-biaxial stretching experiments, using the QBS, confirmed the resultant stress-strain behaviour was nonlinear viscoelastic, exhibiting a strong dependence on specimen temperature and deformation strain rate. Tests conducted between temperatures of $130 - 155 \text{ }^{\circ}\text{C}$, at a representative strain rate of 4 s^{-1} to the bulge test, presented an associated decrease in yield and post-yield stress levels with increasing specimen temperature. Stretching experiments between deformation rates of $1 - 16 \text{ s}^{-1}$ highlight an appreciable increase in overall true stress values with increasing strain rate, when plotted against nominal strain. It is this increased strain rate effect that is hypothesised to be the cause of overlap in stress values for post- T_g temperatures during bulge testing, due to the increased strain rates correlated with softened material behaviour at $155 \text{ }^{\circ}\text{C}$. Anisotropy was also proved through divergence in post-yield stress levels in principal directions. Imitation of the strain histories observed at the pole of the deformed specimen during bulge testing on the biaxial stretcher produced replicable stress-strain data to that observed during the load-controlled analysis; with initial stiffness, yield stresses and anisotropic strain hardening behaviour well reproduced.

Both load-controlled and biaxial-controlled tests are vital in understanding and characterising materials for forming practices. The load-controlled tests are analogous of the real-life process, providing representative strain rates and modes of deformation, whilst the displacement controlled biaxial tests isolate the individual effects of strain rate and material temperature on the resultant deformation behaviour.

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Supplemental Documentation:

Figure A. Overview of validation procedure between calculated stresses from bulge testing and in-plane biaxial experiments.

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