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Temperature dependent bulge test for elastomers

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ABSTRACT

The bulge test is a particularly convenient testing method for characterizing elastomers under biaxial loading. In addition, it is convenient to utilize this test for validating material models in simulation due to the heterogeneous strain field induced during inflation. During the bulge test the strain field for elastomers covers uniaxial tension at the border to pure shear and equibiaxial tension at the pole. Elastomeric materials exhibit a hyperelastic material behavior, with a dependency on temperature and loading rate. The temperature effect on the mechanical behavior during biaxial loading is considered in the present study. A bulge test setup combined with a temperature chamber is developed in order to characterize this effect, and an exemplary temperature dependent characterization of a poly(norbornene) elastomer is performed with this setup. The equibiaxial stress–strain curves measured at 60 °C, 20 °C and −20 °C are presented.

1. Introduction

Nowadays, product design and development relies on simulation tools as finite element analyses (FEA). A number of material models are available in commercial programs for predicting the material's macroscopic mechanical behavior under complex multiaxial stress states. Various models include also time, temperature and loading amplitude dependent functions (e.g., Bergström and Boyce, 1998; Lion, 1997) and can be applied for polymeric materials. The trend to utilize FEA under realistic in service loadings even for "simple" (mechanically non-crucial) products is increasing, mainly driven by OEMs in order to validate their requirements before assembling the parts. The material parameters of the models are usually determined by uniaxial tests (tension, compression, static and/or cyclic). However, the real strain and stress state is rather multiaxial. Taking uniaxial data and computing multiaxial deformation is a conventional procedure in engineering, even though other procedures can be found in the literature (e.g., Mars and Fatemi, 2004; Sasso et al., 2008). Mechanical multiaxial experiments, on the other hand, are very complex to perform. The main problem is that the strain and stress field induced during testing is heterogeneous and therefore the evaluation gets complicated. Even the use of advanced material characterization techniques to determine the full field strain (FFS) distribution such as digital image correlation (DIC) or optical coherence tomography (OCT) (Leiss-Holzinger et al., 2012) is not sufficient. Primarily due to the lack of a relationship between the measured forces and the (heterogeneous) strain fields, stresses cannot be measured at the same position as the strains. Further limitations might arise by induced inhomogeneity during clamping, specimen machining, conditioning, etc. In addition to these difficulties, reliable multiaxial experiments will become even more complicated if temperature, rate and loading amplitude parameters are also taken into account.

As summarized above, multiaxial experiments to characterize the material's behavior are not easily achievable. Nonetheless, biaxial experiments are well-known and reliable results are presented in the literature (Reuge et al., 2001; Hannon and Tiernan, 2008). Biaxial in addition to uniaxial testing gives a more accurate characterization for many applications and product development processes. For polymeric materials and metals this would cover the forming processes, such as blow molding and thermoforming. In biomechanics the characterization of human skins is essential and biaxial testing simulates a stress state that is more realistic to in vivo loading than uniaxial testing (Tonge et al., 2013a).

Different test methods have been proposed and they were summarized by Zouani et al. (1999). They divided the test methods based on the loading systems (LS): *single LS* (cantilever bending, bulge test, pantograph-type frame loading, etc.) and *two or more LS* (a thin-walled tube under e.g., tension–torsion, a round bar under torsion-bending). Biaxial characterizations were carried out for various types of biological and engineering materials, such as soft tissues (Wineman et al., 1979; Coudrillier et al., 2012), fabrics

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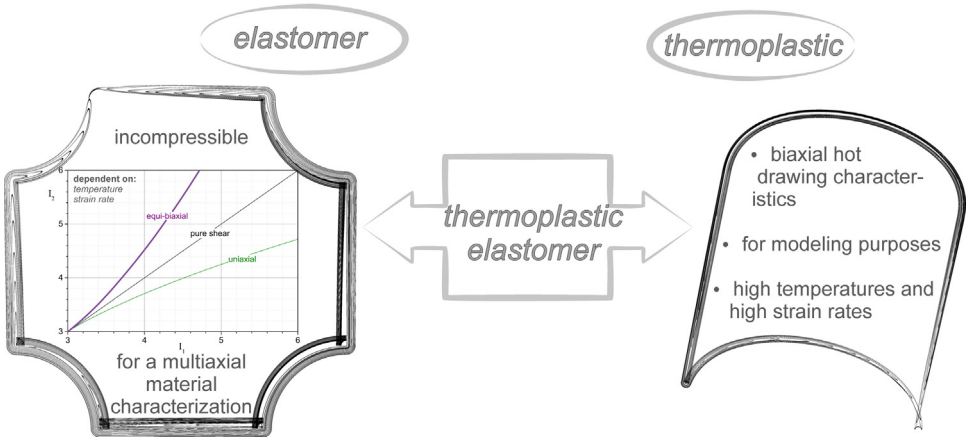

Fig. 1. Overview of the polymer class specific applicability and necessity of biaxial material characterization.

(Kawabata et al., 1973), fabric polymer composites (Buet-Gautier and Boisse, 2001) and metals (Hannon and Tiernan, 2008).

An overview of the necessity and the application purposes of biaxial testing for different polymer classes is shown in Fig. 1.

In general, for elastomeric (incompressible) materials biaxial testing is of interest as it covers along with uniaxial as well as pure shear testing the multiaxial mechanical characterization of the material's behavior (e.g., Charlton et al., 1994). To capture the viscoelastic in addition to the hyperelastic behavior, the inherent temperature and loading rate dependencies on the mechanical behavior also have to be taken into account. For thermoplastic (compressible) materials, biaxial testing is necessary in order to characterize their formability during hot drawing. Strictly speaking, the stress and strain state during forming can vary depending on the final product's geometry and process. In hot drawing processes the uniaxial/planar tension is likely to be more dominant than in biaxial hot-stretching. However, for process characterization and modeling purposes in process relevant temperature and strain rate conditions, biaxial testing of thermoplastics is important and necessary. For poly(ethylene terephthalate), extensive research was performed by Buckley and his co-authors, and they proposed constitutive models (Adams et al., 2000; Buckley et al., 1996) for forming simulations as well as a biaxial hot-drawing experimental test setup, the "Flexible Biaxial Film Tester" (e.g., Buckley and Lew, 2011). This test setup is based on an in-plane biaxial tensile testing method (*two or more LS*). To measure the biaxial viscoelastic behavior of ABS for thermoforming processes, another experiment has been proposed by Hummel and Nied (2004). It is based on a planar tensile test (*single LS*) in a heated chamber and the pure shear stress state ($\lambda_1 = \lambda$, here: $\lambda_2 \approx 1$ and $\lambda_3 \approx 1/\lambda$) was assumed and characterized at elevated temperatures. In contrast, Detrois (2001) modified the well-known membrane inflation test (*single LS*) and proposed a temperature controlled inflation test setup to determine an out-of-plane biaxial (bulge) stress state. Therefore, a specimen is fixed in a chamber filled with silicone oil for convective heating and the load is applied by a ram. During loading, the specimen with a pattern on the surface is optically recorded and using a correlation algorithm the strain and the curvature of the inflated specimen is computed. For the sake of completeness, it should be mentioned that the characterization of metal sheets and strips for forming processes will be standardized in the forthcoming ISO 16808 (2013). This standard is based on the bulge test with optical measuring systems.

The bulge test method is of particular interest, as the inflation of the material under thin-shell assumptions and neglecting bending stiffness (membrane theory) is strongly defined and equibiaxial stresses can be easily determined. This method was adopted by Treloar (1944) and investigations into the stress–strain behavior of inflated vulcanized latex sheets were presented. In addition to the equibiaxial region (pole), he also analyzed the principal strains of the whole specimen. The conclusion of this part of his work was that the border of the specimen has a rather planar tension and only a small region at the pole is equibiaxial. Recently, Machado et al. (2012) presented a methodology to compute the membrane curvature of the bulge test from 3D-DIC measurements. Essentially, they also observed the same principal strain state during bulge testing of silicone rubber. However, a very convenient calculation scheme was proposed based on the surface representation in curvilinear coordinates. From that scheme the circumferential and meridional curvatures, and also the respective stresses, can be computed. Therefore, in the literature many advances in bulge testing have been presented including cyclic testing (Zouani et al., 1999), rate dependent testing (Grolleau et al., 2008) and the above mentioned stress–strain evaluation method, among others. A comprehensive study for biaxial testing of planar anisotropic tissues has been presented by Tonge et al. (2013a) including a material parameter identification scheme for biological materials (Tonge et al., 2013b). They preferred the bulge test rather than an in-plane biaxial tension test setup as bulge testing prevents microstructural rearrangements with loading due to the clamping of the entire specimen edge (Tonge et al., 2013a).

Nevertheless, the characterization of temperature dependent elastomer behavior under (equi)biaxial loading is less found. For incompressible materials, the equivalence of equibiaxial tension and uniaxial compression has been observed (Treloar, 1973; Charlton et al., 1993; Ogden, 1972). So, uniaxial compression test is an alternative testing procedure to determine the equibiaxial behavior of an incompressible elastomer. Treloar (1973) presented stress–stretch data of a uniaxial extension and compression test. The compression data were derived from bulge testing in order to avoid the high compression forces. During compression testing the bulging of the elastomer should be minimized by lubricating the compression plates, where the loading is applied to the specimen, and by choosing proper specimen geometries (e.g., Charlton et al., 1993). Moreover, a displacement transducer or any optical measurement system is needed to determine the actual strain state in the specimen. Accurate compression tests require high experimental effort and advanced instrumentation.

From the above summarized considerations, the objective of and motivation for this paper is to present an experimental method to characterize temperature dependent elastomers under (equi)biaxial strain and stress. Therefore, the bulge test is adopted and combined with a thermal chamber. The test setup also includes a 3D-DIC measurement and evaluation technique. Based on the fact that stresses and strains can be *directly* determined at the same

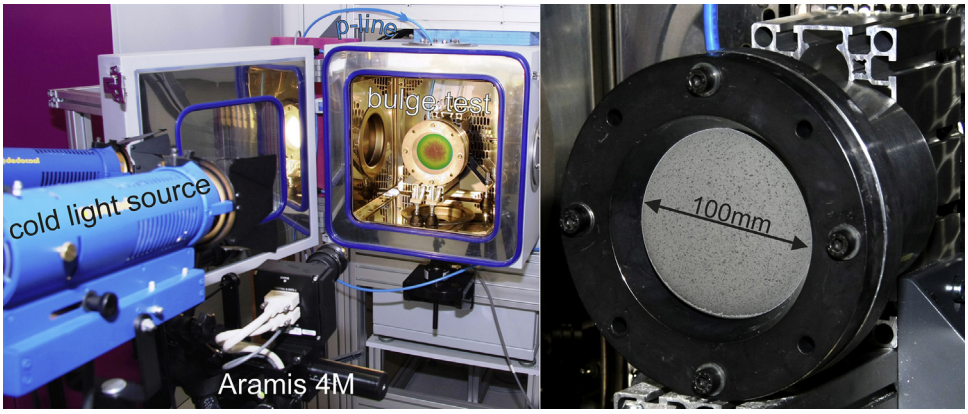

Fig. 2. Test setup of the bulge test in the temperature chamber (left) and a picture of the specimen with the speckle pattern for the DIC evaluation (right).

position on the specimen, this biaxial test method was preferred as opposed to other methods (e.g., planar biaxial tensile test). In planar biaxial tests, for instance, the strain field can be measured by an optical system; however, there is no (general) relation between the measured load and the biaxial stress state. Inverse methods could be applied (finite element updating method by Johlitz and Diebels (2011) and virtual field method by Promma et al., 2009) or a direct calculation method by Çakmak and Major (2013) (higher experimental effort, lower computational time). Moreover, the bulge test represents a convenient test setup to validate temperature dependent material models for FEA. Srivastava et al. (2010) presented a thermo-mechanically coupled large deformation theory for amorphous plastics including the material parameter estimation based on simple compression tests at various strain rates and temperatures. To validated their theory and material model, they compared the experimental observations with numerical for biaxial forming tests with good agreement (cf. Srivastava et al., 2010).

2. Experimental setup

The setup was a custom design based on the well-known bulge test pressure chamber. This pressure chamber is placed in a temperature chamber, in which an entrance on the top of the chamber is used to set the pressure line for the test equipment. In order to capture the specimen during inflation with cameras, the temperature chamber features a large window (300 mm × 300 mm) placed on the door. Two cameras are positioned and calibrated in front of the chamber to take pictures for the subsequent 3D digital image correlation analysis (DIC) procedure. Fig. 2 demonstrates the established test setup and the region of the specimen under investigation.

A more detailed illustration of the test setup is shown in Fig. 3. The gray line represents the compressed air line, and the red line shows the data acquisition as well as the control signal line. The compressed air (10 bar) is filtered and reduced to 6 bar before being conducted to a pressure sensor (Festo 0.2–2 bar). The sensor controls and regulates the air pressure at the desired loading rate and the pressure level, and is controlled by a custom made Labview program (Leonhartsberger et al., 2012).

All data is processed via the Ethernet NI CompactDAQ (National Instruments Corporation) chassis. The actual pressure in the chamber is measured and read-out twice, in the Labview-program and in the DIC data acquisition (Aramis 4 M, GOM mbH, Braunschweig, D). All images for the analyses therefore also have the actual pressure state embedded for the stress calculation. The diameter evolution of the inflated specimen is determined using the DIC evaluation software (Aramis Software). This information is needed for the stress calculation of the specimen.

2.1. Data reduction and stress calculation

The mechanical analysis of an inflating specimen is strongly defined. For elastomers the thin-shell assumptions by neglecting the bending stiffness (membrane theory) are valid if the ratio of specimen diameter to thickness is high. The equibiaxial stresses at the pole of the inflated specimen can then be determined from the actual (bubble or bulge) diameter D and thickness $t(\varepsilon)$ (e.g., Reuge et al., 2001; Machado et al., 2012; Çakmak and Major, 2013).

$$\sigma_{eq} = \frac{p \cdot D}{2 \cdot t(\varepsilon)} \tag{1}$$

The reduction of the thickness during testing can be described by Eq. (2) under the assumption of incompressible material behavior. Alternative descriptions of the thickness reduction can also be defined.

$$t(\varepsilon) = \frac{t(0)}{(1+\varepsilon_{major}) \cdot (1+\varepsilon_{\min or})} \tag{2}$$

This is a basic evaluation of the stress state during bulge testing. Machado et al. (2012) presented an evaluation scheme for the bulge test based on the determination of the surface curvature tensor and the membrane stress tensor of an inflated silicone elastomer. With this method, the circumferential as well as the meridional stress can be determined at every stage and position of the specimen. An application of this method can be found in Çakmak and Major (2013) to evaluate the stress state throughout the measured area on the specimen.

3. Material and specimen

The described and specified bulge test with temperature control was utilized to measure a poly(norbornene) elastomer (Norsorex® 49625-6, 55 Shore A, Astrotech GmbH, A). To determine the glass transition temperature T_g of the material, differential scanning calorimetry (DSC1, Mettler Toledo) was conducted. The onset value of T_g was identified at −4 °C. To get a better understanding of the material's viscoelastic behavior, dynamic mechanical analyses (DMA) were performed in tension at temperatures from −60 °C to 100 °C and frequencies from 1 Hz to 56 Hz. A sinusoidal excitation with mean level and dynamic amplitude of 1% strain was the loading condition during DMA. The diagram of Fig. 4 shows the frequency dependent DMA results measured under isothermal conditions (loss factor tanδ vs. storage modulus E'). From the plot of the dissipative (loss) mechanical properties and the storage modulus, a trend between the different isothermal measurement points is observable. It can be concluded that the loss factor tanδ is a

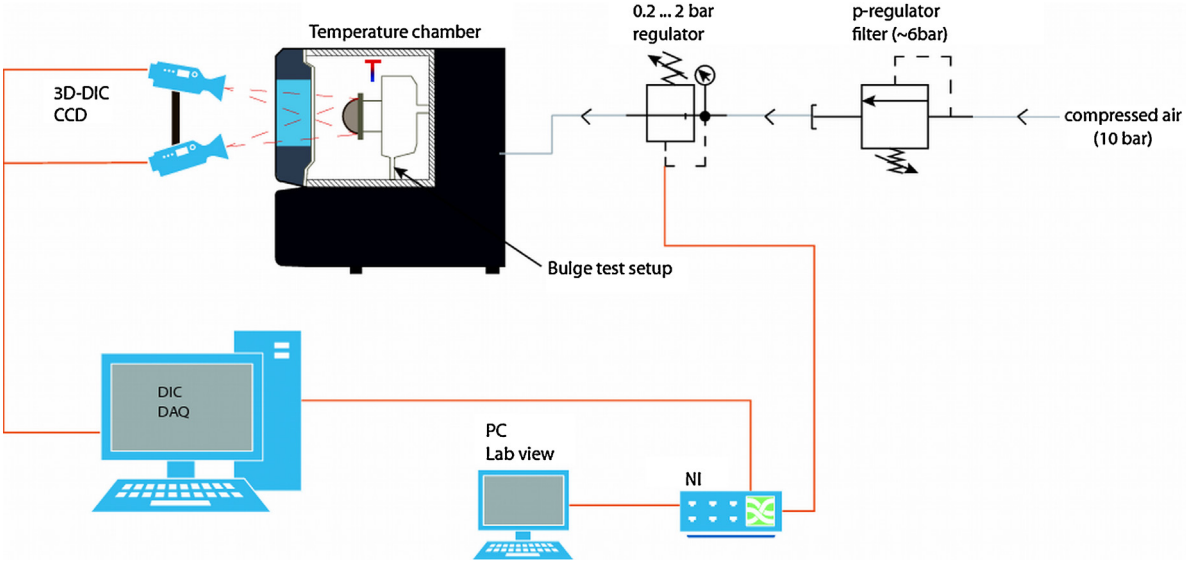

Fig. 3. Schematic illustration of the measurement and evaluation setup.

unique function of the storage modulus and the material's behavior is thermorheologically simple (Jones, 2001).

According to the comprehensive review article of Lendlein and Kelch (2002), norbornene polymers exhibit shape memory properties. To demonstrate, if the material under investigation also reveals this entropy effect, a stripe of the material was twisted and fixed at room temperature (RT), exposed to elevated temperatures (~70 °C) for 15 min, rapidly cooled below T_g with liquid nitrogen (LN) and afterwards the fixation was opened and put in atmospheric air (labor condition). The temperature of the material was measured at the surface. With increasing temperature the stripe disentangled slowly until RT was attained. The temporary (frozen) shape was achieved and even after 30 min changes could not be observed (see Fig. 4). Finally, the stripe in the temporary shape was exposed in the temperature chamber to air at ~70 °C and the permanent shape was regained. Fig. 4 illustrates a part of the process in pictures and each of them is labeled according to the previously described stages. These stages are also indicated in the diagram with the corresponding numbers and letter in the boxes in order to give the reader an idea of the actual mechanical state of the material.

The measured specimen size was 100 mm in diameter and 2 mm in thickness ($D/t = 50$). Three temperatures (60 °C, 20 °C and −20 °C) were chosen to perform the pressure controlled bulge test.

4. Results and discussion

The applied pressure over the measurement time is shown in Fig. 5. The red line is a linear fit of the pressure data and indicates an applied pressure rate of 0.05 bar/s (0.5 MPa/s). Fig. 5 also shows the measured major and minor strains within the analyzed region in the DIC evaluation software at the investigated temperatures.

At the beginning of the curves some irregularities are observable. These are mainly due to the clamping of the specimen. The specimens were clamped at room temperature and then exposed to temperature changes, causing thermal expansion and leading to misalignments. A further observation is that there is almost no

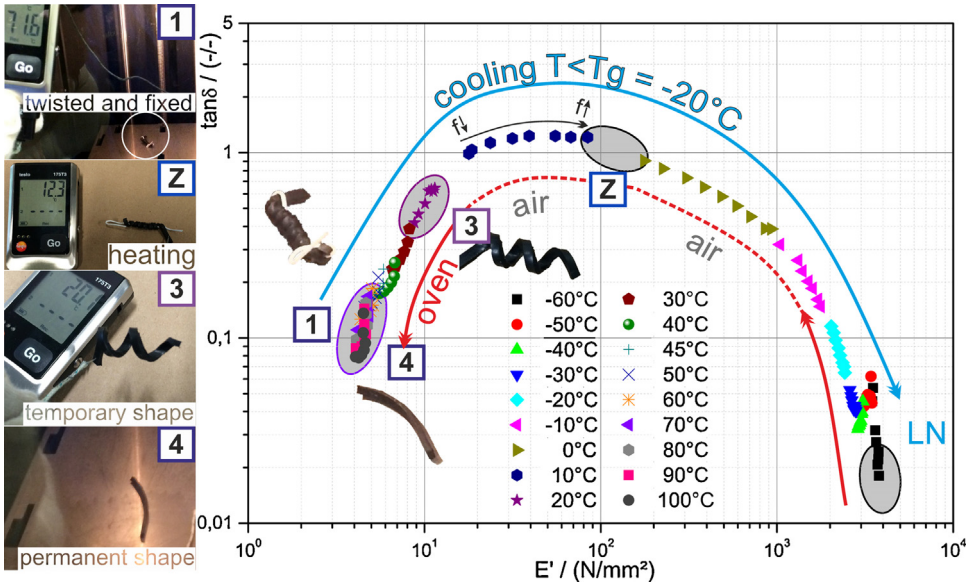

Fig. 4. Illustration of the shape memory properties of the investigated material including the loss factor-storage modulus diagram. *Pictures:* shape memory capability of poly(norbornene) elastomer; *Diagram:* temperature and frequency dependent viscoelastic behavior determined by dynamic mechanical analysis (DMA) in tension.

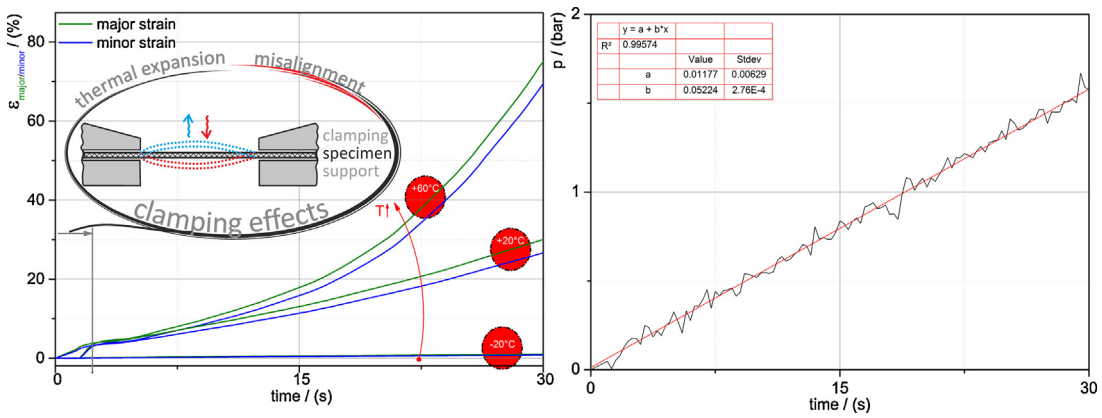

Fig. 5. Temperature dependent major and minor strain (left) while inflating the specimen under pressure control (right).

difference in major and minor strain at −20 °C, but at the other temperatures there is a difference beginning at approximately 7 s. The difference is small compared to the measured strains and as a result it has not been analyzed or considered further. In fact, this is a combinatory effect of experimental error and a non-perfect equibiaxial strain state within the analyzed region in the DIC evaluation.

Using the previously specified data reduction and stress calculation scheme, the stresses are calculated and the stress–strain curves are illustrated in Fig. 6. The smoothed data (dashed lines) is shown in green, red and magenta for 60 °C, 20 °C and −20 °C, respectively. The data was smoothed using the FFT-filter method. To compare the results, the temperature dependent uniaxial tensile behavior of the material is also shown here. Uniaxial tests were performed according to EN ISO 527-2 with the 5A type specimen at the specified equilibrium temperatures. The full lines correspond to these data and are displayed with the same colors as mentioned previously. These data are averaged out of three measurements.

As expected the material has a high temperature dependency. Measured strains are independent of temperature, but elongation does decrease when temperature is reduced. All observable irregularities at the beginning of the experiment, as described earlier, were corrected by linear regression of the data points to zero loading. The equibiaxial behavior of the material can be seen in the extension (dashed gray lines) as well as in the compression region. As mentioned earlier, equibiaxial tension is equivalent to uniaxial compression for incompressible materials. Treloar's (1973) plot was adopted here to demonstrate the trend of the uniaxial behavior of the poly(norbornene) elastomer from extension to compression. The measurements at −20 °C are below the glass transition temperature of the material and so there is almost no difference in the slope of the uni- and biaxial data. In contrast the trend of the equibiaxial data at the other temperatures differs from uniaxial data, however the initial slope is similar.

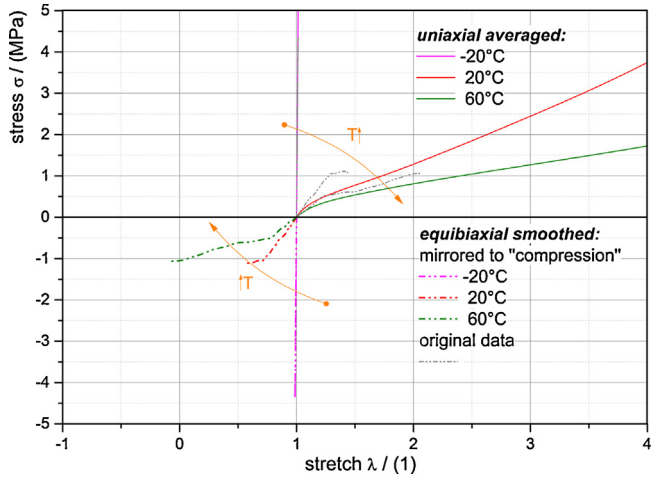

Fig. 6. Biaxial and uniaxial stress–stretch curve of the measured elastomer at 60 °C, 20 °C and −20 °C displayed in green, red and magenta, respectively. The biaxial data is also shown mirrored in the compression region due to the equibiaxial tension and uniaxial compression equivalency.

5. Conclusion

The authors believe that the bulge test is one of the preferred biaxial testing experiments as the stresses are determined at the same position as the strains. To do so, the assumptions of membrane theory have to be valid. The higher the specimen's ratio of diameter to thickness, the more accurately this assumption is fulfilled. The limitation thereby is the experimental setup, along with the maximum adjustable and applicable loading and data acquisition parameters. In this paper, a bulge test setup in a temperature chamber is presented as an extension of the current setups for elastomer testing. Temperature dependent biaxial tests of poly(norbornene) were performed with reasonable results. During testing, the specimen was recorded by cameras and the full field strain distribution was calculated using a digital image correlation algorithm. Necessary advances are needed for elastomer testing in the initial state, where the thermal expansion has to be compensated for by the clamping. Biaxial in addition to (at least) uniaxial tests are required to characterize the mechanical behavior of an elastomeric (incompressible) material (cf. Fig. 1). Fig. 6 demonstrates that under the assumption of incompressibility the uniaxial compression behavior can be derived from equibiaxial tension tests. Due to lubrication of the loading plates, specimen geometry (error in parallelism, bulging etc.) and high compression forces, the equibiaxial tension experiments are preferred rather than uniaxial compression. Moreover, the bulge test can be utilized for different biaxiality ratios (λ_1/λ_2) by using elliptical instead of ring fixations. Bulge test is more versatile as uniaxial compression test and can be further developed. For thermoplastics a hot drawing characterization test setup is desirable and some promising solutions are under examination in ongoing research projects. In summary, it can be stated that the temperature controlled bulge test can be utilized for elastomers to characterize the temperature effects on the mechanical biaxial behavior.

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