Material Properties of a Structural Silicone for Linear Adhesive Glass-Metal Connections

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During recent years, adhesives are more frequently used for glass applications in buildings, and according knowledge on adhesive glass-metal connections increased exponentially. However, further research regarding the performance of such connections remains indispensable to further optimise existing concepts or to develop and implement new technologies. To analyse the mechanical behaviour of adhesive glass-metal connections, computer simulations based on the finite element method can be performed. To do so, material properties of all components have to be introduced in the form of stress-strain curves, which can be obtained through experimental tests on adhesive bulk material. Here, the focus is on adhesives suitable to transfer significant loads between a structural glass panel and a thin-walled steel frame. In this contribution, the material properties of a structural silicone, Sikasil® SG-500, were determined through several experiments. Tensile tests on dumbbells, compression tests on cylindrical samples and thick adherends shear tests (TAST) were performed according to the available standards. Different displacement rates were considered to study the visco-hyperelastic behaviour of the structural silicone. Subsequently, possible theoretical material laws were assessed for their ability to approximate the experimental obtained stress-strain relationships. This paper describes the test procedures, reports about the fabrication process of the test samples, presents the obtained experimental data and proposes possible material laws for the silicone. From the tests, no significant differences in tensile strength and shear strength were obtained for the displacement rates used in this research. However, the stiffness in tension, compression and shear did depend on the test speed.

Keywords: structural silicone, material properties, tensile tests, compression tests, TAST, material law

1. Introduction
The use of glass in structural elements encounters the challenge of designing proper connections between glass components mutually and between glass components and other parts of a structure. Typically, locally installed metal connectors ensure a connection between such elements. However, structural adhesive bonds in glass systems have known a significant rise in recent years, due to several advantages compared to the mechanical connections. Mechanical connections using bolts, clamps or friction-grip fixings introduce peak stresses in the glass and glass treatments, such as drilling and tempering are often required. In case of adhesive connections, the latter is omitted and a redistribution of high local stress concentrations, depending on the stiffness of the adhesive, becomes possible. Other advantages are an avoidance of condensation, and thermal and acoustic bridges, automation of prefab structural glass components and a reduction of the weight of the construction (Dispersyn et al. 2013). The structural behaviour of such bonded connections is determined by the geometry, the stiffness and the load-carrying capacity of the adhesive joint. Hence, detailed knowledge of the mechanical values and the durability of adhesives is necessary. Discontinuities in the boundary areas are of principal importance too and require close examination (Feldmann and Kasper 2014). By performing finite element analysis, reliable and accurate results on the mechanical behaviour of adhesive glass-metal connections can be obtained. For that purpose, material properties of the adhesive under tension, compression, shear, etc. in the form of stress-strain curves have to be implemented in the finite element software. To obtain these relationships between loads and displacements, experimental tests on adhesive bulk material have to be performed.

The focus of this research lies on adhesives suitable to transfer significant loads between a structural glass panel and a thin-walled steel frame and to manage differential thermal expansions. Structural silicones appear to be ideal as considerable strength is obtained due to a large bonding area (linear adhesive connections) and as the flexibility allows for absorption of differential thermal expansions. Moreover, these types of sealants are already widely used in structural sealant glazing systems. Therefore, the aim of this research was to determine the material properties of Sikasil® SG-500 through uniaxial tensile tests, compression tests and thick adherend shear tests (TAST). To study the visco-hyperelastic material behaviour of this structural silicone, different displacement rates during the tests were considered. From the experimental results, material laws to implement in finite element software can be derived and assessed for their applicability. This paper describes the test procedures that can be used to determine tensile, compression and shear behaviour of structural adhesives and presents the obtained experimental results in case of the structural silicone Sikasil® SG-500.
2. Experimental program
A selection tool for adhesives in structural applications with glass and metal was developed at Ghent University in cooperation with Delft University of Technology (Belis et al. 2011). An extensive experimental research program on several adhesives from several adhesive families, i.e. silicones, MS-polymers, polyurethanes, laminates, acrylates and epoxies, was performed to allow designers to select potential adhesives based on loading conditions and environmental conditions. Using the adhesive selection tool developed during this research project, the structural silicone Sikasil® SG-500 was designated as potential candidate for hybrid cold-formed steel-glass panels. This silicone is a fast-curing two-component adhesive. By mixing a base compound (Component A) and a catalyst (Component B), polycondensation reactions are triggered and curing occurs. The volume mixing ratio of Component A to Component B is 10:1. This adhesive is already used for structural glazing applications and bonding of solar modules (Sika 2015).

2.1. Tensile tests
The mechanical behaviour in tension of the structural silicone can be implemented in finite element software when the tensile characteristics are determined from uniaxial tensile tests on bulk material (Nhamoinesu and Overend, 2012). These tests were performed according to NBN EN ISO 527-1 (NBN 2012a) and NBN EN ISO 527-2 (NBN 2012b), standards which are generally applicable on adhesives and films. According to these standards, a so-called dog bone or dumbbell specimen is loaded in tension along its longitudinal axis at a constant speed until failure occurs. The dog bones were fabricated by injecting silicone material by means of an automated dispensing gun into a mould made from PTFE, a non-sticking material which allows easy demoulding. The geometry of these specimens of type 1B according to NBN EN ISO 527-2 (NBN 2012b) is depicted in Fig. 1 and the corresponding dimensions are summarised in Table 1.

![Fig. 1 Dimensions of a dog bone sample (Type 1B) according to NBN EN ISO 527-2 (NBN 2012b).](image)

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Signification</th>
<th>Dimensions [mm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>( l_1 )</td>
<td>Length of narrow parallel-sided portion</td>
<td>60±0.5</td>
</tr>
<tr>
<td>( l_2 )</td>
<td>Distance between broad parallel sided portions</td>
<td>108±1.6</td>
</tr>
<tr>
<td>( l_3 )</td>
<td>Overall length</td>
<td>180</td>
</tr>
<tr>
<td>( r )</td>
<td>Radius</td>
<td>60±0.5</td>
</tr>
<tr>
<td>( b_1 )</td>
<td>Width of narrow portion</td>
<td>10±0.2</td>
</tr>
<tr>
<td>( b_2 )</td>
<td>Width at ends</td>
<td>20±0.2</td>
</tr>
<tr>
<td>( h )</td>
<td>Thickness</td>
<td>4±0.2</td>
</tr>
</tbody>
</table>

After 24 hours of curing, the samples were demoulded and stored for another six days in a climatic chamber with a temperature of 20±1°C and a relative humidity of 60±3%. The specimens were then tested using a universal electromechanic test machine Instron 5982 dual column floor frame with a load cell of 10 kN. According to the aforementioned standard, displacement-controlled tests can be carried out with speeds varying from 0.125 mm/min to 500 mm/min. During this research, five specimens were tested at a speed of 1 mm/min to simulate quasi-static loading. To study the effect of the test speed, another five samples were tested at 5 mm/min and another five specimens at a speed of 20 mm/min. During testing, the load and the displacement of the cross-head were continuously measured and registered until failure of the sample occurred. Fig. 2a) illustrates a test specimen at the beginning of the tensile test and Fig. 2b) illustrates the same specimen just before failure.
2.2. Compression tests

To determine the behaviour of the structural silicone Sikasil® SG-500 in compression, uniaxial compressive tests on bulk material were performed according to NBN EN ISO 604 (NBN 2003). During such a test, a solid cylindrical specimen is compressed at a constant speed along its longitudinal axis between two compressive plates. The cylindrical specimens with a diameter of 30 mm and a height of 15 mm, as illustrated in Fig. 3a) and Fig. 3b), were produced using a similar process and under similar environmental conditions as in case of the dog bones. Again, the samples were demoulded after 24 hours and stored in a climatic chamber for another six days. Next, the samples were tested on a universal testing machine with a load cell of 100 kN, as depicted in Fig. 3c). For compressive tests, test speeds may vary between 1 mm/min and 20 mm/min according to NBN EN ISO 604. In this experimental program, five specimens were tested at a speed of 1 mm/min, another five specimens at a speed of 5 mm/min and another five at a speed of 20 mm/min. The load and the displacement of the cross-head were continuously measured and registered until 95 kN was reached. Depending on the surface texture, the top and bottom side of the cylinder can shift differently with respect to the compression plates. This can lead to significant variations in the deformations which might influence the material characteristics. According to NBN EN ISO 604, this effect is more distinct when the stiffness of the material is low, which is the case for Sikasil® SG-500. Therefore, PTFE was sprayed on the compression plates to reduce friction (Dispersyn et al. 2013; Dias et al. 2013).
2.3. Thick Adherends Shear Test (TAST)

Due to the flexibility of the considered structural silicone, its behaviour in shear can easily be determined by performing a Thick Adherends Shear Test (TAST) according to NBN EN 14869-2 (NBN 2011). This test is mainly used to determine the shear modulus and shear strength of low-stiffness adhesives. A TAST-specimen consists of two step-shaped thick adherends which overlap over a short distance. This configuration minimises rotation of the lap joint, resulting in approximately uniform stresses in the adhesive (Vogt 2009; NBN 2011). The specimens used during this research are depicted in Fig. 4a) and Fig. 4b). An adhesive layer of 2 mm, which is larger than the minimum required thickness of 0.05 mm according to the standard, was applied between two aluminium blocks under similar conditions as already mentioned. After a total of seven days, the samples were tested in tension using a universal testing machine with a load cell of 10 kN, as illustrated in Fig. 4c). Five specimens were tested at a speed of 0.5 mm/min, which is prescribed by EN 14869-2 (NBN 2011). To assess the effect of the test speed on the shear characteristics of the adhesive, another five specimens were tested at a rate of 1 mm/min.

3. Results and discussion

3.1. Tensile tests

The relationship between the tensile stress $\sigma$ and the tensile strain $\varepsilon$ of each specimen is obtained by dividing the applied force by the loaded area and by taking the ratio of the obtained displacement to the initial testing length. The specimens failed at a maximum strain $\varepsilon_{\text{fail}}$ to which the maximum stress $\sigma_{\text{fail}}$ corresponded. Fig. 5a) depicts the average curves of each sample corresponding to the different displacement rates used in this research. Fig. 5b) illustrates the initial stress-strain relationship up to a strain of 0.2.
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Fig. 5a) Average tensile stress-strain curve for different displacement rates and b) Initial tensile stress-strain relationship up to a strain of 0.2.

Several parameters are derived from the relationship between stress and strain for each specimen. The average values of these parameters and the according standard deviation for each displacement rate are presented in Table 2.

To obtain the Young’s modulus $E_t$ in tension for the structural silicone, ETAG-002 (EOTA 2001) refers to NBN EN ISO 527-1 (NBN 2012a), which postulates that this parameter can be calculated according to Eq. 1.

$$E_t = \frac{\sigma_2 - \sigma_1}{\varepsilon_1 - \varepsilon_2}$$

(1)

where $E_t$ = Young’s modulus;
$\sigma_1$ = the stress in MPa as measured on the deformation value of $\varepsilon_1 = 0.0005$;
$\sigma_2$ = the stress in MPa as measured on the deformation value of $\varepsilon_2 = 0.0025$.

From Fig. 5a) a quasi-bilinear behaviour of the structural silicone in tension can be detected. Table 2 contains, therefore, two additional moduli corresponding with the slope of the linear least-squares regression line from the linear region for strains smaller than 0.015 ($E_{\text{linear},1}$) and from the linear region in case of strains larger than 0.2 ($E_{\text{linear},2}$).

Table 2: Characteristics of Sikasil® SG-500 in tension ($\varepsilon_{\text{fail}}$ = average strain at failure, $\sigma_{\text{fail}}$ = average stress at failure, $E_t$ = Young’s modulus, $E_{\text{linear},1}$ = modulus for the linear approximation of the first linear region of the graph, $E_{\text{linear},2}$ = modulus for the linear approximation of the second linear region of the graph).

<table>
<thead>
<tr>
<th>Test speed</th>
<th>$\varepsilon_{\text{fail}}$</th>
<th>$\sigma_{\text{fail}}$ [MPa]</th>
<th>$E_t$ [MPa]</th>
<th>$E_{\text{linear},1}$ [MPa]</th>
<th>$E_{\text{linear},2}$ [MPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 mm/min</td>
<td>1.57 ± 0.150</td>
<td>1.54 ± 0.072</td>
<td>3.90 ± 0.277</td>
<td>4.45 ± 0.398</td>
<td>0.91 ± 0.023</td>
</tr>
<tr>
<td>5 mm/min</td>
<td>1.23 ± 0.184</td>
<td>1.48 ± 0.110</td>
<td>3.88 ± 0.556</td>
<td>4.44 ± 0.757</td>
<td>1.08 ± 0.060</td>
</tr>
<tr>
<td>20 mm/min</td>
<td>1.15 ± 0.267</td>
<td>1.43 ± 0.206</td>
<td>4.09 ± 0.440</td>
<td>4.52 ± 0.648</td>
<td>1.04 ± 0.028</td>
</tr>
</tbody>
</table>

The differences in stress at failure $\sigma_{\text{fail}}$ between the different displacement rates are considered to be not statistically significant ($\alpha = 5\%$), although a small decrease of 7.1% with an increase in test speed from 1 mm/min to 20 mm/min is observed. The Young’s modulus $E_t$ of the structural adhesive increases by 4.9% with an increase of the displacement rate from 1 mm/min to 20 mm/min. However, no significant ($\alpha = 5\%$) difference between the values is obtained. For the slope of the linear regression line from the linear region for strains smaller than 0.15 ($E_{\text{linear},1}$) too, there is no statistically significant difference ($\alpha = 5\%$). However, for larger displacements ($\varepsilon \geq 0.2$), the modulus $E_{\text{linear},2}$ significantly ($\alpha = 5\%$) differs when comparing a displacement rate of 1 mm/min to a test speed of 5 mm/min. Between displacement rates of 5 mm/min and 20 mm/min, no statistically significant ($\alpha = 5\%$) difference is observed. At lower displacement rates, relaxation is more prominent, which results in lower stresses for the same deformations, hence in a lower stiffness. This could explain the lower value for $E_{\text{linear},2}$ for a displacement rate of 1 mm/min compared to 5 mm/min or 20 mm/min. The insignificant difference in stiffness $E_{\text{linear},2}$ for test speeds of 5 mm/min and 20 mm/min may indicate that relaxation phenomena are similar for both displacement rates. Additional tests with higher displacement rates – NBN EN ISO 527-1 (NBN 2012a) allows test speeds up to 500 mm/min – makes further investigation of these phenomena possible. Alternative tests on H-specimens as described in ETAG-002 (EOTA 2001) could be performed as well. Nardini and Doebbel (2015) performed such tests to determine dynamic tensile strengths of Sikasil® SG-500 and Sikasil® SG-550.
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From the experimental tensile stress-strain relationship, a material law for the considered structural silicone can be derived. Fig. 6 illustrates the experimental data for the tensile tests with a displacement rate of 1 mm/min with the corresponding material laws of Neo-Hooke, Mooney-Rivlin and Arruda-Boyce. Although, these laws underestimate the stresses for strains up to 0.25, they are all numerically stable and can therefore be used. The material law of Neo-Hooke fits the experimental data the best. The parameter $C_{10}$, which characterises this material law, is equated to 0.33502415 based on the experimental data from this research. Additional material laws can possibly be evaluated or only small-strain behaviour, for example, can be considered to obtain an even better fit of the experimental results by a theoretical relationship between tensile stress and tensile strain.

![Fig. 6 Experimental data and material laws for Sikasil® SG-500 under tension.](image)

3.2. Compressive tests

The compressive stress $\sigma$ can be calculated by dividing the applied load by the loaded area, whilst the compressive strain $\varepsilon$ is defined as the ratio of the displacement to the initial height of the samples. The mean stress-strain curves, up to a strain of -0.75, for the different displacement rates are illustrated in Fig. 7a). Fig. 7b) depicts the stress-strain relationships up to a strain of -0.4. Table 3 summarises the mean Young’s moduli, as calculated from Eq. 1, for each displacement rate.

![Fig. 7a) Average compressive stress-strain curves for different displacement rates and b) Initial compressive stress-strain relationship up to a compressive strain of -0.4.](image)

<table>
<thead>
<tr>
<th>Test speed</th>
<th>$E_t$ [MPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 mm/min</td>
<td>1.97 ± 0.080</td>
</tr>
<tr>
<td>5 mm/min</td>
<td>2.20 ± 0.131</td>
</tr>
<tr>
<td>20 mm/min</td>
<td>2.22 ± 0.110</td>
</tr>
</tbody>
</table>

Table 3: Characteristics of Sikasil® SG-500 in compression ($E_t$ = Young’s modulus).
In compression, the Young’s modulus of the Sikasil® SG-500 increased by 11.7% with an increase of the displacement rate from 1 mm/min to 5 mm/min, which is statistically significant ($\alpha = 5\%$). A further increase of the test speed from 5 mm/min to 20 mm/min did not change the stiffness significantly ($\alpha = 5\%$). Hence, as in tension, the behaviour of the structural silicone in compression alters if the displacement rate is increased from 1 mm/min to 5 mm/min. A further increase to 20 mm/min has no significant effect on the compression behaviour of the silicone.

NBN EN ISO 604 (NBN 2003) does not prescribe lower test speeds than 1 mm/min or higher test speeds than 20 mm/min, hence the largest difference in stiffness measurable according to that standard is included in Table 3.

A material law for the structural silicone can be derived from the experimental results in a similar way as for tension. Fig. 8 depicts the experimental data for the compression tests with a displacement rate of 1 mm/min up to a strain of 0.2 with the corresponding material laws of Neo-Hooke, Mooney-Rivlin and Arruda-Boyce. The material laws of Neo-Hooke and Arruda-Boyce do not approximate the experimental data and cannot be used. The material law of Mooney-Rivlin approximates the compressive behaviour only for small strains ($\varepsilon > -0.05$). The parameters $C_{10}$ and $C_{01}$, which characterise this material law, are in this case equal to -1.04300517 and 1.18469401 respectively. Alternative material laws and/or adapting the compression strain region to expected values for a considered application will most likely reduce the present discrepancy between the experimental data and the theoretical relationship between compressive stress and strain.

3.3. Thick Adherends Shear Test (TAST)

A plot of the shear stress $\tau$ against the shear strain $\gamma$ can be used to derive certain parameters needed for finite element analysis. The shear stress $\tau$ is calculated as the ratio between the applied force and the cross-sectional area of the adhesive layer. The shear strain $\gamma$ can be obtained by dividing the measured deformation by the thickness of the structural silicone layer. With the maximum shear strain $\gamma_{\text{fail}}$, at which the specimens failed, corresponds the maximum shear stress $\tau_{\text{fail}}$. Fig. 9a) depicts the mean shear stress-strain curves for a displacement rate of 0.5 mm/min and 1 mm/min. Fig. 9b) illustrates the shear behaviour up to a shear strain of 0.5.
NBN EN 14869-2 (NBN 2011) defines the shear modulus as the gradient of the linear, low-strain region of the plot of shear stress against shear strain. This parameter can be calculated, according to the same standard, with Eq. 2.

\[
G = \frac{\tau}{\gamma}
\]  

(2)

here \(\tau\) is the shear stress in MPa measured on the deformation value \(\gamma = 0.5\), which corresponds to a point in the linear region of the curve.

Table 4 summarises the mean values of the stress and strain at failure and the shear modulus for a displacement rate of 0.5 mm/min and 1 mm/min.

Table 4: Characteristics of Sikasil® SG-500 in shear (\(\gamma_{\text{fail}} = \) average shear strain at failure, \(\tau_{\text{fail}} = \) average shear stress at failure, \(G = \) shear modulus).

<table>
<thead>
<tr>
<th>Test speed</th>
<th>(\gamma_{\text{fail}}) [(%)]</th>
<th>(\tau_{\text{fail}}) [MPa]</th>
<th>(G) [MPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.5 mm/min</td>
<td>2.03 ± 0.104</td>
<td>1.15 ± 0.022</td>
<td>0.49 ± 0.023</td>
</tr>
<tr>
<td>1 mm/min</td>
<td>1.95 ± 0.037</td>
<td>1.16 ± 0.092</td>
<td>0.54 ± 0.014</td>
</tr>
</tbody>
</table>

No statistically significant difference (\(\alpha = 5\%\)) in shear stress at failure \(\tau_{\text{fail}}\) or shear strain at failure \(\gamma_{\text{fail}}\) was observed for displacement rates of 0.5 mm/min and 1 mm/min. However, the structural silicone did behave significantly (\(\alpha = 5\%\)) stiffer in case of the higher test speed as the shear modulus \(G\) increased by 10.2 %. Although EN 14869-2 (NBN 2011) only stipulates a deformation rate of 0.5 mm/min, it appears that the shear stiffness does depend on the displacement rate. Alternative tests on H-specimens, such as described by ETAG-002 (EOTA 2001), could also be used to determine the shear stiffness of the structural silicone. Nardini and Doebbel (2015) performed such tests on Sikasil® SG-500 with a displacement rate of 5 mm/min. The secant modulus between \(\gamma = 0\) and \(\gamma = 0.515\) was found to be 0.49 MPa, which is the value which was also found in this research for shear tests on TAST-specimens with a displacement rate of 0.5 mm/min.

Fig. 10 represents the experimental data of the shear tests with a displacement rate of 0.5 mm/min and the corresponding material laws of Neo-Hooke, Arruda-Boyce and Yeoh. The material laws of Neo-Hooke and Arruda-Boyce overestimate the shear stress up to a shear strain of 0.5, after which both laws (severely) underestimate the shear stress-strain behaviour. The material law of Yeoh fits the experimental data the best. The characterising parameters of this material law are \(C_{10}\), \(C_{20}\) and \(C_{30}\) of which the values, based on the experimental data, are represented in Table 5. To enhance the correlation between the experimental data and the material laws, additional theoretical stress-strain relationships can be investigated or the experimental data can be limited to the expected interval which depends on the application.

![Fig. 10 Experimental data and material laws for Sikasil® SG-500 under shear.](image)

Table 5: Parameters for the material model of Yeoh for the structural silicone under shear.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>(C_{10})</th>
<th>(C_{20})</th>
<th>(C_{30})</th>
</tr>
</thead>
<tbody>
<tr>
<td>Value</td>
<td>0.081171595</td>
<td>0.01713645</td>
<td>-0.00085658</td>
</tr>
</tbody>
</table>
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4. Conclusion
In this research, tensile tests, compression tests and shear tests according to available standards were performed on the two-component structural silicone Sikasil® SG-500. To investigate the visco-hyperelastic behaviour of the adhesive, test speeds were varied from 1 mm/min over 5 mm/min to 20 mm/min. The tensile strength and shear strength did not change significantly with variable displacement rates. The stiffness of the structural silicone under tension and compression on the other hand did alter significantly with an increase of the test speed from 1 mm/min to 5 mm/min. An additional increase to 20 mm/min had no significant effect on the stiffness in tension and compression. Therefore, additional tensile tests with higher displacement rates (up to 500 mm/min) could clarify whether the adhesives may exhibit an even stiffer behaviour or not. Also in shear, an increase of the test speed from 0.5 mm/min to 1 mm/min resulted in a significant increase of the shear modulus. Material laws for the behaviour of the structural silicone under tension, compression and shear were derived. In tension, the stress-strain relationship was approximated best by the Neo-Hooke material law, whilst in shear the material law of Yeoh demonstrated a good fit. The material law of Mooney-Rivlin approximated the compressive stress-strain behaviour of the structural silicomes for small displacements ($\varepsilon < 0.05$).

5. Future work
To investigate the material behaviour of the structural silicone Sikasil® SG-500 more thoroughly, additional tests can be performed to reduce the scatter on the results. Intermediate or higher displacement rates than used during this research may increase insight in the visco-hyperelastic behaviour of the material. Furthermore, the behaviour at different temperatures could be studied as well as the alteration of the properties with time. The determination of the stress-strain curves in tension, compression and shear of the structural adhesive as presented in this paper is the first step to investigate adhesive connections between glass and metals using finite element software. As already partially done in this paper, material laws for the considered adhesive can be derived from these relationships. Investigating additional material laws and adapting the strain-interval to values which are expected in practice, hence which depend on the considered application, will contribute to an improved theoretical description of the real in-service behaviour of the structural silicone. To validate these derived relationships between stress and strain, additional tests will be performed and modelled in a finite element program using the aforementioned material laws.

Acknowledgements
Scheledebov nv is gratefully acknowledged for the production of the test specimens used during this research.

References