Reiterated tension testing of silicone elastomer

P. Meier*, M. Lang and S. Oberthür

A peristaltically actuated device, PADeMIS, composed of silicone rubber (SR) is under development for use in minimally invasive surgery. During locomotion, the device will be subject to a few thousand load changes involving varying, sometimes high, strains. The design is being optimised by finite element analysis, for which a constitutive law for the mechanical behaviour of silicone rubber is required. Uniaxial and biaxial tension tests have been performed on specimens of used silicon rubber. Synchronous fitting of uniaxial and biaxial tensile data gives significantly better results than using uniaxial or biaxial results alone to derive constitutive laws. The mechanical properties of SR were found to change from loading to loading up to few thousand cycles. Hence, simulating the deformation of SR structures is problematic. Furthermore, variability was observed between batches of SR.

Keywords: PADeMIS, Artificial worm, Uniaxial tension, Lifetime

Introduction

A Peristaltically Actuated Device for Minimal Invasive Surgery (PADeMIS) is being developed at the Technical University of Ilmenau. The device will move actively like an earthworm carrying a hollow tube inside its back. The tube and the active part of the device provide a channel in which endoscopic tools can be inserted for use in invasive surgery. The first application of PADeMIS is in minimally invasive spine surgery. The device will be inserted into the spinal canal at the os sacrum and moved cranially between the vertebral bodies and the dura mater spinalis. Therefore, PADeMIS has to be designed to fit the spinal canal: the outer diameter of PADeMIS must be adjustable from 4 to 10 mm with a minimum inner diameter of 2 mm for the endoscopic instruments. The device will be made of silicone rubber and will consist of 'worm' segments, each made of at least two layers of silicone enclosing pads. The pads of serially arranged segments will be filled periodically with fluid, thus producing a peristaltic locomotion (Fig 1). The design of a single segment, shown in Fig. 2, is being optimised by Finite Element Analysis (FEA) for which a constitutive law as input property is essential. The loading case of the deformed pads will be approximately biaxial. On the other hand, uniaxial tension tests are generally much easier and less time-consuming to perform. However, in order to gain permission for medical use, many long-term experiments have to be carried out to examine mechanical fatigue and stress-softening[1].

The aim of this paper is to study whether a constitutive law (Mooney–Rivlin law) fitted to uniaxial tension tests can be used to describe biaxial tests or, more generally, whether fitting of a constitutive law to one specific loading case leads to a reliable prediction of deformations under other loading conditions. Furthermore, a simultaneous fit of uniaxial and equibiaxial experimental data is presented. Beyond this, results of some reiterative tension tests are presented.

Technical University of Ilmenau, Faculty of Mechanical Engineering, 98684 Ilmenau, Germany

*Corresponding author, email petra.meier@tu-ilmenau.de

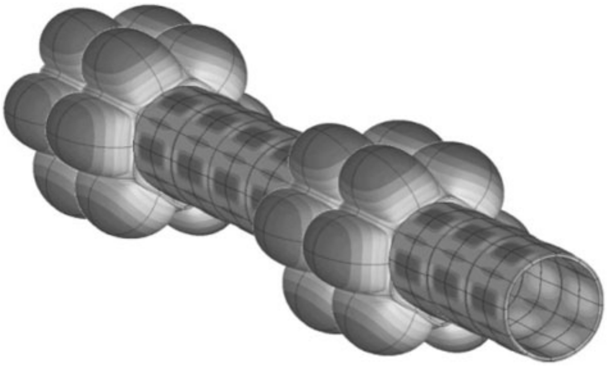
Experimental

The segments of PADeMIS are filled periodically with fluid to produce the peristaltic locomotion. This results in nearly equibiaxial stress in the silicone membrane (Fig. 2). Therefore, the constitutive law for the FEA optimization of the design should be evaluated by equibiaxial tension tests. The disadvantages of the equibiaxial tension tests are intense consumption of time and manpower. Hence, for stability tests and investigations of stress-softening[1] over a long period of time, uniaxial tension tests are more convenient.

Silicone rubber samples

The samples were produced from the liquid injection molding silicone elastomer MED-49xx from NUSIL distributed by Polytec. The xx indicates the shore hardness of the silicone rubber adjusted with silica filler by the manufacturer. In this paper, results from experiments performed with MED-4930 and MED-4950 are shown. The two components are mixed steadily in a 25% hexane solution. Then they are cast in moulds (120 mm × 120 mm or 170 mm × 170 mm) and degassed for more than 30 h. The next step is the curing of the silicone rubber. The thickness of the sheets was measured with a layer thickness measurement Dualscope made by Fischer. The used layers had a thickness of 500–900 μm and the standard deviation of a

Published by Maney on behalf of the Institute
Received 13 September 2005; accepted 12 October 2005
DOI 10.1179/174328905X59737



1 Schematic view of PADeMIS with serial arrangement of two filled and four unfilled segments

test sample was less then 10%. For details of the setup and the evaluation see Ref. 2.

Equibiaxial tension test

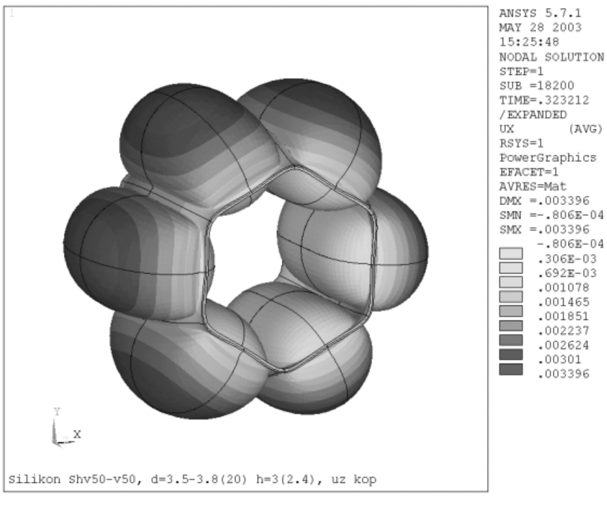
The equibiaxial stress can be measured by inflating a thin silicon sheet as described by Rivlin and Saunders.[3] The silicon test sample is fixed to an aluminium plate by an aluminium ring of diameter 60 mm and 9 points are marked. In the middle of the aluminium plate, a pressure supply and a connection for a pressure sensor are included. Then the aluminium plate with the silicone sheet is mounted at a three axes positioning unit made by ISEL. On the vertical axis, a tip is fixed. The positions of the markers can be measured during deformation due to the increasing pressure by aligning this tip with the crossings of the marker lines.

Uniaxial tension test

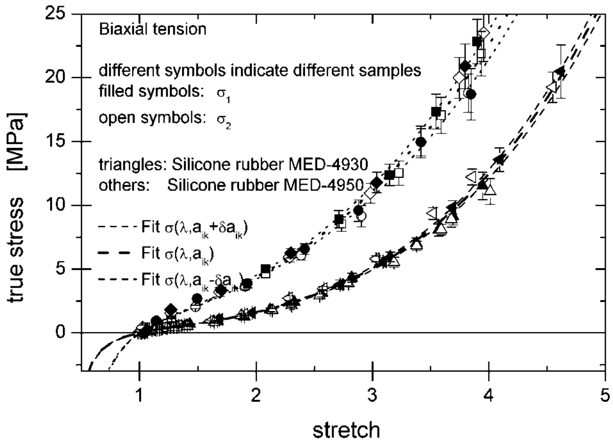
For the uniaxial tension test, the silicone rubber sheet is cut into pieces 80 mm × 5 mm. A 20 mm long region is marked around the centre The sample is fixed in clamps and attached to the test stand. Two test stands are used. The samples for the synchronous fitting are loaded with different weights and the resulting length is measured. The samples for the repeated tension tests are stretched by a stepping motor at 10 mm s^{-1} and the resulting force is measured.

Constitutive law: theory

To perform FEA simulations of the design of PADeMIS, a constitutive law of the silicone rubber is



2 FEA of the deformation of one segment of PADeMIS



3 Experimental equibiaxial tension test for silicone rubber MED-4930 and MED-4950

needed as input parameter. A well known constitutive law for rubber-like materials is the extended Mooney–Rivlin law, which is based on polynomials[4,5]

$$W=\sum_{i+k=1}^{m} a_{ik}(I_1-3)^i(I_2-3)^k+\frac{1}{2}\kappa(I_3-1) \quad (1)$$

where W = strain energy density, I_1, I_2, I_3 = invariants of the deformation tensor, κ =bulk modulus, m=order of model and a_{ik} = the Mooney–Rivlin constants describing the material.

Fitting procedure

The experimental data are fitted with a LabVIEW program. A modified version of the LabVIEW Levenberg–Marquardt χ^2 minimization subroutine from National Instruments Corporation is used. (For programming the Levenberg–Marquardt method, see Ref. 6).

The property χ^2 describing the distance between the fitted curve and the experimental value is defined as

$$\chi^2=\sum^{n}\left(\frac{\sigma_{\mathrm{fit}}(\lambda,a_{ik})-\sigma_{\mathrm{meas}}}{\delta\sigma_{\mathrm{meas}}}\right)^2 \quad (2)$$

$\sigma_{\mathrm{fit}}(\lambda,a_{ik})$ = calculated constitutive law derived from equation (1), σ_{meas} = stress from measured, $\delta\sigma_{\mathrm{meas}}$ = experimental errors and n = number of experimental results. In addition, $\sigma_{\mathrm{fit}}(\lambda,a_{ik}+\delta a_{ik})$ and $\sigma_{\mathrm{fit}}(\lambda,a_{ik}-\delta a_{ik})$ are calculated to get an impression of the errors of the fitting process.

For simultaneous fitting, the LabVIEW Levenberg–Marquardt χ^2 minimization subroutine is modified:

$$\chi^2=\chi^2_{\mathrm{uni}}+\chi^2_{\mathrm{bi}} \quad (3)$$

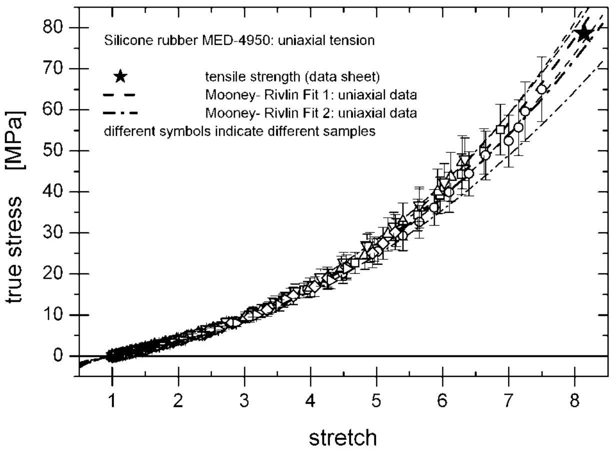
where χ^2_{uni} = χ^2 of the uniaxial fitting procedure and χ^2_{bi} = χ^2 of the biaxial fitting procedure as calculated by equation (2).

Results

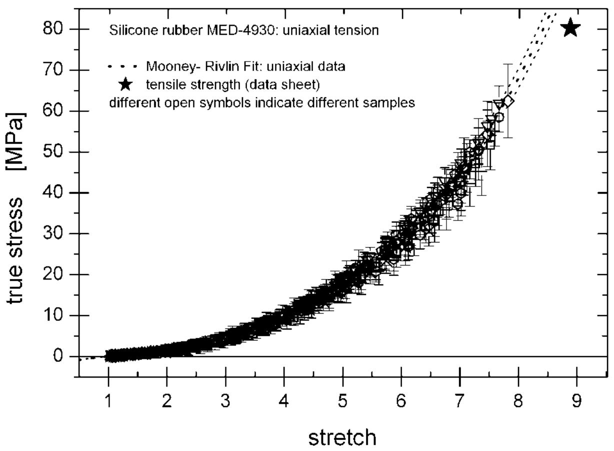
First tension test

Equibiaxial tension test

In Fig 3, the measured stress–stretch dependency for equibiaxial tension of the silicone rubbers MED-4930 and MED-4950 and the fitted stress–stretch relations are shown.



4 Experimental data and fits of the uniaxial tension tests for silicone rubber MED-4950

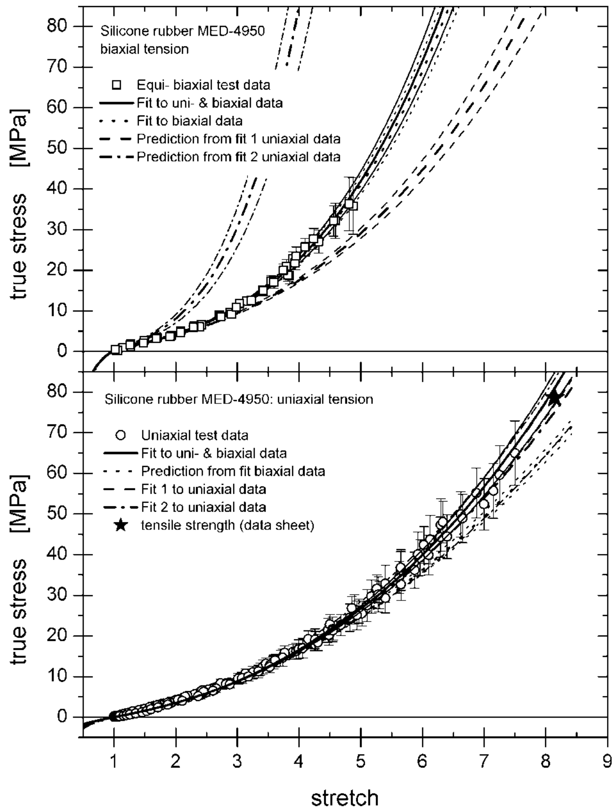


5 Experimental data and fits of the uniaxial tension tests for silicone rubber MED-4930

As can be seen, the stresses and stretches in direction 1 and 2 are identical within a 10% margin. Thus, the reproducibility of measurements of different samples is very good. The experimental results can be sufficiently approximated with fitting methods. The values of the fitted parameters can be seen in Table 1. There was no acceptable fitting result with parameters $a_{20} \neq 0$.

Uniaxial tension test

The experimental data of the uniaxial tension tests of silicone rubber MED-4950 and MED-4930 is shown in Figs. 4 and 5, respectively. For uniaxial tension of MED-4950, two curves are shown. For both materials,



6 Fitted constitutive law for equibiaxial and uniaxial tension tests of silicone rubber MED-4950

the fitted stress–stretch characteristics closely match the tensile strength specified in the data sheet of the manufacturer.

With regard to χ^2 and $\delta a_{01} / a_{01}$ and $\delta a_{20} / a_{20}$, the fit 2 of material MED-4950 is much worse than fit 1. For both materials, values of the parameters a_{01} could not be found. Thus, fitting of uniaxial tension tests result in uncertainties for the parameters a_{0k}. The values of the fitted parameters can be seen in Table 1.

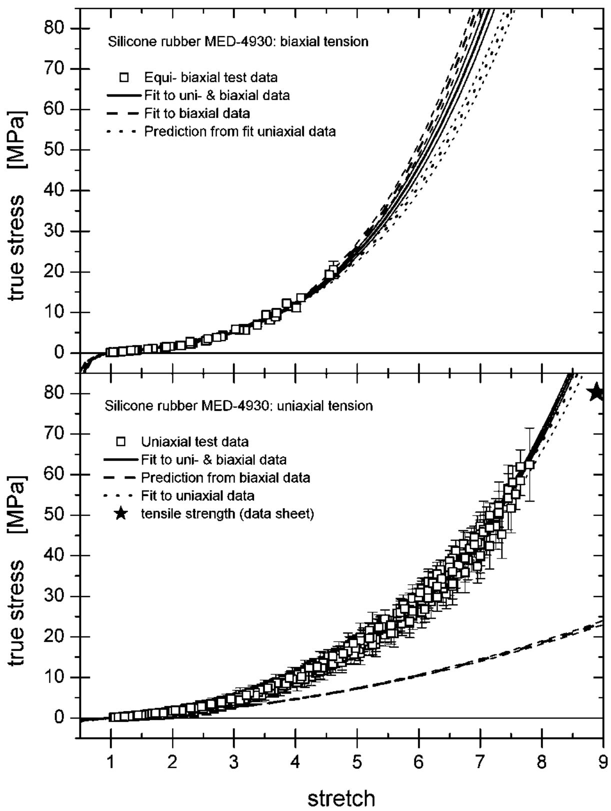
Optimisation of the constitutive law to equibiaxial and uniaxial tension tests

The fitted constitutive laws are used to calculate a stress–stretch characteristic to predict the other loading case. The calculated stress– stretch relations for biaxial and uniaxial loading for silicone rubber MED-4950 and MED-4930 are shown in Figs. 6 and 7, respectively. In the MED-4950 biaxial loading case the errors between the stress–stretch characteristics calculated from the

Table 1 Mooney–Rivlin parameters of silicone rubber MED-4930 and MED-4950

Parameters	a_{10}*	δa_{10}*	a_{01}*	δa_{01}*	a_{20}*	δa_{20}*	χ^2	ν
MED-4950								
Biaxial fit†	503	12	13	1			73	46
Uniaxial fit 1	497	6			0·9	0·2	718	217
Uniaxial fit 2	484	19	134	27	0·5	0·3	1003	216
Simultaneous bi- and uniaxial fit	493	5	10	1	0·9	0·2	800	264
MED-4930								
Biaxial fit	144	3	15	0·5			283	58
Uniaxial fit	245	6			2	0·1	87	432
Simultaneous bi- and uniaxial fit	192	2	1·3	0·4	3	0·05	544	492

*kPa
†Unlisted parameters are equal to zero. For explanation of χ^2 see section 'Silicone rubber samples' and Ref. 2; ν is the degree of freedom of the fit.



7 Fitted constitutive law for equibiaxial and uniaxial tension tests of silicone rubber MED-4930

parameters of fit 2 from the uniaxial data and the experimental data are enormous. Also, a constitutive law fitted to the biaxial loading case cannot estimate the MED-4930 uniaxial data. From Table 1, it can be seen that a constitutive law for the silicone rubbers MED-4950 and MED-4930 needs (at least) the parameters a_{10}, a_{01} and a_{20}, but fits to one loading case give no satisfying results for all three parameters.

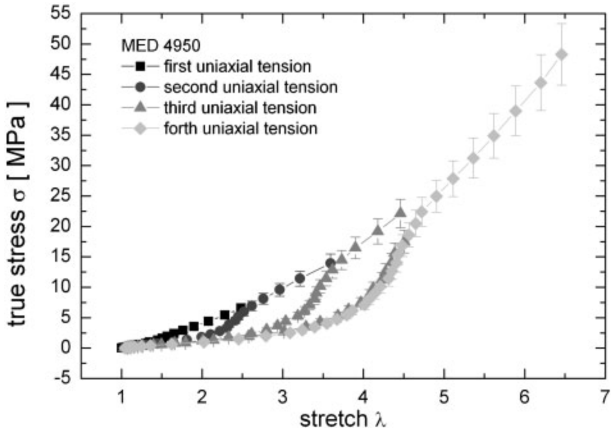
The stress–stretch relations fitted to biaxial and uniaxial simultaneously (according to equation (3)) are also shown in Figs. 6 and 7 respectively. Thus, the Mooney–Rivlin approach of the constitutive law is able to describe the biaxial and uniaxial loading case simultaneously. The parameters a_{ik} of silicone rubber MED-4930 and MED-4950 are listed in Table 1. It is possible to find the three parameters a_{10}, a_{01}, and a_{20} with small errors and acceptable χ^2 via the simultaneous fit. The validity of the resulting constitutive law for other loading cases can be estimated by calculating the stress from the constitutive law and considering the orders of stretch λ for each parameter of the constitutive law. The parameters belonging to terms with high orders of stretch λ for the simulated load case should also belong to terms with high orders of stretch λ for the experimental setup, too.

Reiterated tension tests

For filled rubber, stress-softening due to rearrangement of the filler particle is well known. PADeMIS will undergo a few thousand cycles during a surgery. Thus, long term load cycles are performed to characterise the material.

Tensions test with increasing loading

Samples are loaded till the stretch λ_1 is reached, then relaxed and subsequently loaded till stretch $\lambda_2>\lambda_1$ is



8 Successive uniaxial tension tests of MED-4950 to increasing stretches

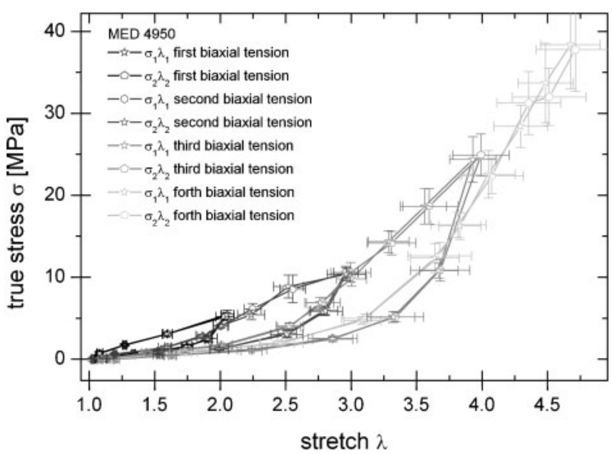
reached. Afterwards, loading till stretch $\lambda_3>\lambda_2$ is performed. During the first discharging, the samples show a large hysteresis and, during recharging, the samples follow approximately the discharging curve up to the previous maximum load. Then the stress–stretch relation switches to the relation of undamaged samples. This behaviour conforms to the stress-softening described by Mullins[1]. Stress–stretch relations of typical samples of MED-4950 are shown in Figs. 8 and 9 for uniaxial tension and equibiaxial tension tests, respectively.

Successive tension tests to same load

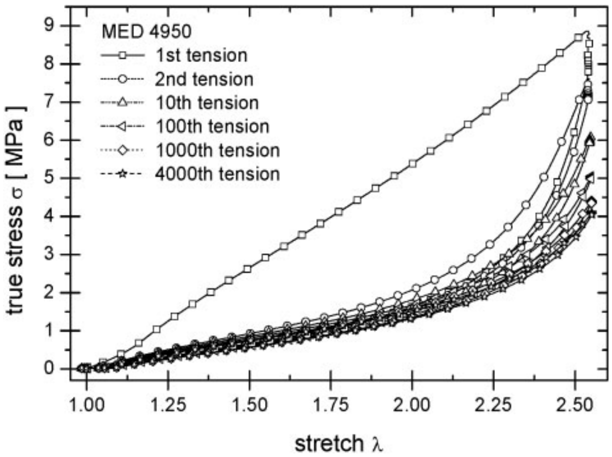
The segments of PADeMIS will be filled with volume control. Observing the resulting pressure could give information about the environment and contact area between PADeMIS and surrounding tissue, assuming a constant stress–stretch relation for the material preloaded at least to the used load. Thus, successive tension tests to the same length are performed. Unfortunately, equibiaxial tension tests are very time consuming, so only results for uniaxial tension are presented.

Decreasing maximal stress

Figure 10 shows the stress–stretch relations for a sample loaded 4000 times to a stretch $\lambda\approx2{\cdot}6$. The maximal stress decreases permanently with increasing number of load cycles. There is no final state for the stress–stretch relation. Although the decreasing of the maximum stress



9 Successive biaxial tension tests of MED-4950 to increasing stretches



10 Decreasing maximal stress during successive uniaxial tension tests of MED-4950 to same stretch

is approximately an exponential function, but the sample fails before a steady state is reached.

Plastic deformation

Usually SR is described as a hyperelastic material, but, as shown in Fig. 11, there is a significant plastic deformation of the sample. The plastic deformation can make up to 50% of the initial length of the sample for large stretches. The first load cycle causes quite a large plastic deformation, whereas, for successive load cycles, the plastic deformation increases smoothly. The amount of plastic deformation depends on the amount of maximum stretch.

Lifetime

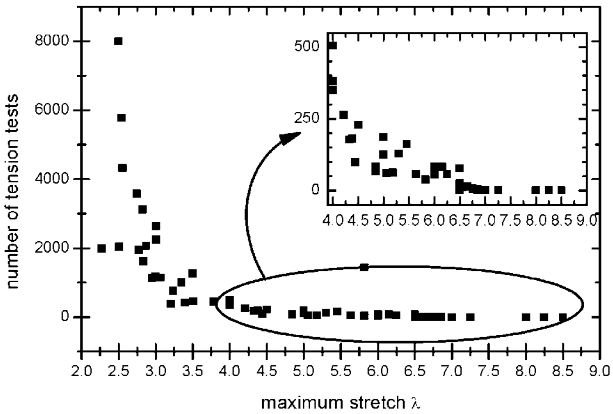
To estimate the range and endurance of the artificial worm PADeMIS successive tension tests up to the failure of the SR were performed. For MED-4950, the results are shown in Fig. 12. For large stretches, $\lambda > 7$, the life cycle is rather short and only a few load cycles can be performed. For smaller stretches, the life cycle increases up to a few thousand load cycles for samples stretched by a factor of 3 or less. Unlike Hookean materials such as steel, which can be loaded, to small strains, for a few million load cycles without failure, SR seems to accumulate failures from previous stretch cycles. This means the SR seems to remember previous stretches. So the integral of the strain energy density W over the time of total load t should be considered. Unfortunately, this is experimentally difficult. Therefore the sum of the strain energy density W over the number of tension tests N

$$\int W \mathrm{d}t \tag{4}$$

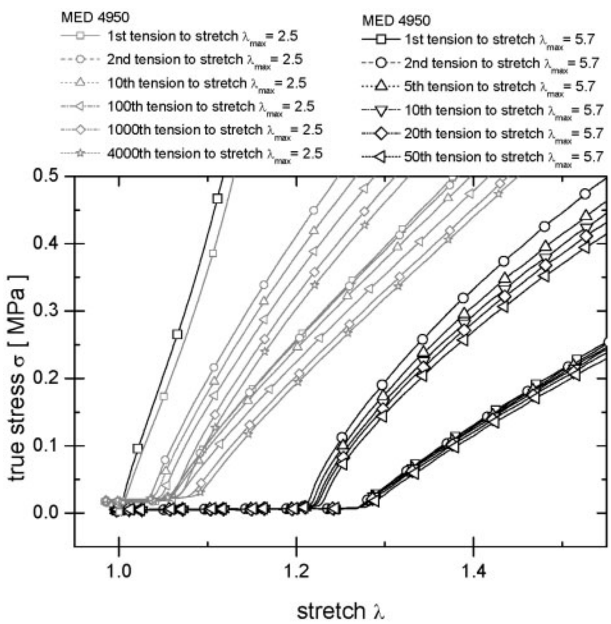
and the integral of the strain energy density W over stretch λ

$$\sum_{\mathrm{N}} W(\lambda_{\max}) \tag{5}$$

are calculated as a rough approximation (Fig. 13). As parameters for the strain energy density W values, fitted stretched samples are used. But both approximations lead to a unsatisfactory prediction of the sample life.



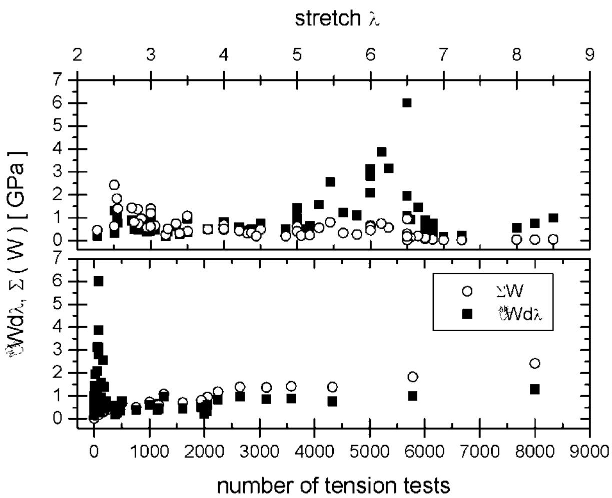
12 Number of iterations of uniaxial tension tests of MED-4950 to same stretch λ till failure of sample



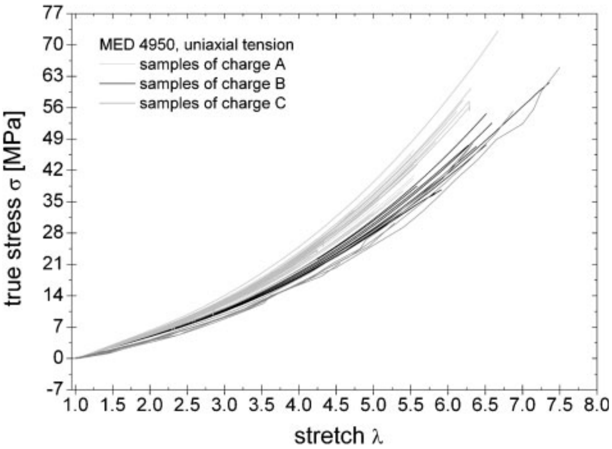
11 Progress of plastic deformation during successive uniaxial tension tests of MED-4950 to the same stretch

Varying quality of batches

Even though SR has a nonlinear complex stress–stretch relation, the manufacturer indicates only a tensile



13 Integral of strain energy density and sum over strain energy density in dependency of maximal stretch and number of iteration



14 Uniaxial tension tests of MED-4950 for samples prepared from different batches of SR

strength and a maximal elongation. On the contrary, an expiry date of a few months is guaranteed. Because of this, the stress–stretch relation of samples prepared from different batches are compared in Fig. 14 The differences between the batches are significantly higher than the variance within a batch. When obtaining SR for production of PADeMIS, the manufacturer has to be asked for the same or a similar batch.

Conclusions

The experiments presented in this paper were performed as the basis for simulations of structures made of silicon rubber. On the one hand, it is shown that fitting experimental data of one loading case can lead to wrong parameters in the Mooney–Rivlin law and therefore to wrong predictions for other loading cases or complex deformation states. On the other hand, for silicone rubber, a set of Mooney–Rivlin parameters describing uniaxial and equibiaxial experiments simultaneously were found. Unfortunately, SR shows both Mullins effects for load cycles to the same length and stress-softening for load cycles to the same length for reiterative tension tests. Therefore, a steady-state constitutive law cannot be found. Simulations of a structure, which has to perform a few thousand load cycles, is either linked with rough approximations or extensive time consumption. The relative short lifetime of SR should be considered and there is also a rather high variance for different batches of the same material delivered.

Acknowledgment

Many thanks are due to the students Samer Khader and Andreas Münch for performing thousands of tension tests and to Mike Stubenrauch for correction of the manuscript. Furthermore, we would like to thank the TKM (Ministry of Education of Thuringia) for financial support of the NachwuchsgruppePeristaltische Sonde Peristaltic Device.

References

1. L. Mullins: *Rubber Chem.Technol.*, 1969, **42**, 339–362.
2. P. Meier, S. Khader, R. Preuss, J. Dietrich, and D. Voges: in ‘Constitutive Models for Rubber III’, J. Busfield and A. Muhr (eds), p. 99–106; 2003, Lisse, The Netherlands, Swets & Zeitlinger.
3. R. S. Rivlin and D. W. Saunders: *Phil. Trans. Roy. Soc*, 1951, **A243**, 251.
4. M. Mooney: *J. Appl. Phys.*, 1940, **11**, 582–593.
5. R. S. Rivlin: ‘Forty years of non-linear continuum mechanics’, Proc. IX Int. Congress on Rheology, Mexico, 1984, 1–29.
6. W. H. Press, S. A. Teukolsky, W. T. Vetterling and B. P. Flannery: ‘Numerical recipes in C’; 2002, Cambridge, UK, Cambridge University Press.