

Indenter

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Group 4

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1 Introduction

In this experiment, the mechanical properties of materials were investigated by a material testing machine. The properties of the elastomer polydimethylsiloxane (PDMS) were observed in tensile and indentation tests. Viscoelasticity was tested for the materials Silly Putty® and Memory Foam in a stress-relaxation and a creep test, respectively.

1.1 Motivation

The determination of mechanical properties of a material plays an important role in biomedical engineering. On the one hand, regarding the mechanical stability of materials, this becomes crucial when constructing implants that are inserted into the human body [1]. On the other hand, knowledge about the mechanical properties of specific tissues, and especially the change of these properties in pathological tissue, may provide useful information about the health condition of a human [2]. This can help developing biomarkers that can be used as diagnostic tools for a patient [2].

1.2 Theoretical Background

Materials can be uniquely characterized by their mechanical behaviour and properties, that can be determined by observing the deformation of a material when applying an external load [2]. In experiments, the behaviour under load of the respective material is commonly investigated by either applying a tensile stress, compression or shear force [2].

1.2.1 Tensile Test

When applying a tensile stress to one end of a bar while the other end is fixed, the tensile stress σ is defined as the ratio of the applied force F that is assumed to be evenly distributed over the cross-sectional area A of the bar [3]:

$$\sigma = \frac{F}{A} \quad (1)$$

The tensile strain ε is defined as the ratio between the relative length displacement of the sample ΔL to the initial length of the sample L_0 [3].

$$\varepsilon = \frac{\Delta L}{L_0} \quad (2)$$

The relationship between tensile stress and the resulting strain can be seen in Fig. 1 that shows the so-called stress-strain curve of a ductile polymer [3]. This curve is characteristic for a certain material [3].

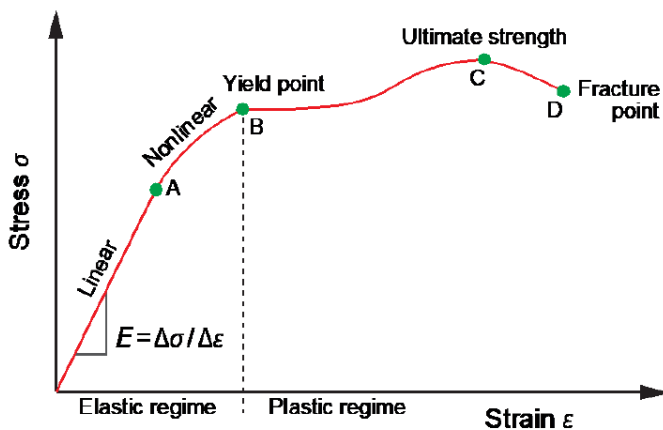


Figure 1: Stress-strain curve of a ductile polymer [2]

When only applying a low force, the stress of the material is directly proportional to the strain (linear part of the curve up to point A in Fig. 1). In this part of the curve, the material behaves fully elastic [3]. Therefore, the deformation of the material is reversible after unloading [3]. Its deformation follows Hooke's law [3], which is defined as:

$$\sigma = E \cdot \varepsilon \quad (3)$$

The proportionality factor E is called elastic modulus or Young's modulus [3].

If the applied force is increased, the curve becomes non-linear, but the material is still fully elastic up to the so-called yield point (Fig. 1 point B) [3]. Beyond this point, the deformation of the material becomes irreversible, because the material is entering the plastic regime (Fig. 1 point C) [3]. Here, the material creeps at constant stress in case of perfect plasticity [3]. If the stress further increases with increasing strain, the material hardens, what is called strain-hardening [3]. After reaching the ultimate strength, the cross-sectional area of the bar shrinks, what is known as necking, and thus, the stress is slightly decreasing until the material fails, and the bar finally breaks apart (Fig. 1 part D) [3].

Regarding polymers, the characteristic "shape of the stress-strain curve depends on the structural and chemical composition of the material" [2], with the structure depending on "the degree of polymerization and on the temperature" [2]. If a polymer is below its glass transition temperature, its stress-strain curve looks like the curve of a brittle material [2]. As it can be seen in Fig. 2, the characteristics of a brittle material are that there is no plastic deformation, and thus, no necking [3]. They directly fail at the end of the elastic regime [3]. Furthermore, the expansion of brittle materials is limited [3]. The fracture occurs with almost no deformation [3]. Semi-crystalline polymers show the characteristics of ductile materials [2]. During strain hardening phase, the polymer chains become parallel oriented [2]. A polymer above its glass transition temperature behaves like an elastomer [2]. Thus, these polymers can be extended elastically for high strains until they fracture [2].

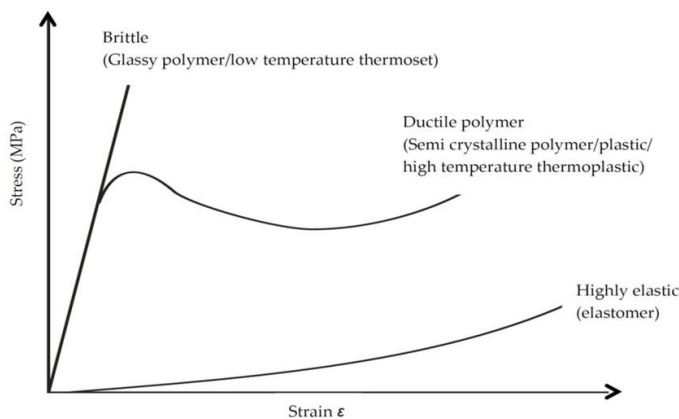


Figure 2: Stress-strain curve of brittle polymers, ductile polymers and elastomers [4]

1.2.2 Indentation Test

Another method to determine elastic-plastic (or deformation) mechanical properties of a material is the indentation test [2]. An indenter with a defined tip geometry is first approached to the sample as close as possible, and then, penetrates the sample with a predefined force or until a certain indentation depth [2]. There are several contact models describing the relation between force and the resulting indentation of the sample, depending on the tip geometry of the indenter [2]. These relationships can be seen in Tab. 1. The original contact

model referring to two spheres was developed by Hertz [2]. Sneddon extended the Hertz model to conical and cylindrical indenter geometries [2].

Table 1: Relationship between force and indentation for different indenter tip geometries

Tip Geometry	Force-Indentation Relationship
Spherical	$F(\delta) = \frac{4}{3} \frac{E}{1-\nu^2} \sqrt{R} \delta^{3/2}$ (4)
Conical	$F(\delta) = \frac{2}{\pi} \frac{E}{1-\nu^2} \tan \alpha \delta^2$ (5)
Cylindrical	$F(\delta) = 2 \frac{E}{1-\nu^2} R \delta$ (6)

R is the radius of the indenter, δ describes the indentation depth, and E is the elastic modulus of the sample [2]. In case of a conical indenter, the half opening angle α of the tip has to be known [2]. The relation of transverse to axial strain is described by Poisson's ratio ν , a dimensionless constant [2]. The Poisson's ratio can be assumed as $\nu = 0.5$ for incompressible materials [2].

Other assumptions that are made when applying these formulas are that the radius of the indenter is larger than the indentation depths, and that the elastic modulus of the indenter is much higher than the elastic modulus of the sample [2].

The indenter is fixed on a force transducer that is attached on a displacement device. When assuming the force transducer as Hookean spring, the force at the tip of the indenter can be calculated by [2]:

$$F = k \cdot \Delta d \quad (7)$$

with k as spring constant of the force transducer and Δd as relative deflection. Out of this, the indentation δ of the sample can be determined [2]:

$$\delta = \Delta z - \Delta d \quad (8)$$

with Δz as relative z-displacement of the indenter.

Out of the observed relationship between force and displacement during measurement, Young's modulus can be calculated [2]. Therefore, eventually the equations 4 and 5 for spherical and conical force-indentation relationships have to be linearized, as it will be done in this experiment (see section 3.3).

1.2.3 Creep- and Stress-Relaxation Test

Elasticity describes the property of a material to recover its original shape [3]. Thus, if an applied force is withdrawn from an elastic material, the deformation of the material is reversible [3]. The strain energy caused by the applied force is stored in the material as potential energy, what leads to a matching behaviour of the stress-strain curve while loading and unloading the material (see Fig. 3A) [2]. For non-linear elasticity the behaviour of the material while loading and unloading is also the same, but non-linear [2], see Fig. 3B. If a material has elastic-plastic properties, the stress-strain curves while loading and unloading are not similar anymore because only the elastic part is able to recover (Fig. 3C) [2]. The plastically deformed part of the material remains deformed after unloading [2]. Viscoelasticity describes the stress-strain behaviour of a material in case the elasticity is time, frequency, and/or temperature dependent [2], see Fig. 3D. The material properties are now determined by

elastic and viscous properties [2]. In general, “viscosity describes the resistance of a fluid to flow” [2]. The stress-strain curve of a viscoelastic material shows hysteresis between the loading and unloading path due to energy dissipation [2]. For higher strain rates, the area between the curves for loading and unloading, and thus, the energy dissipation is lower than for lower strain rates [2].

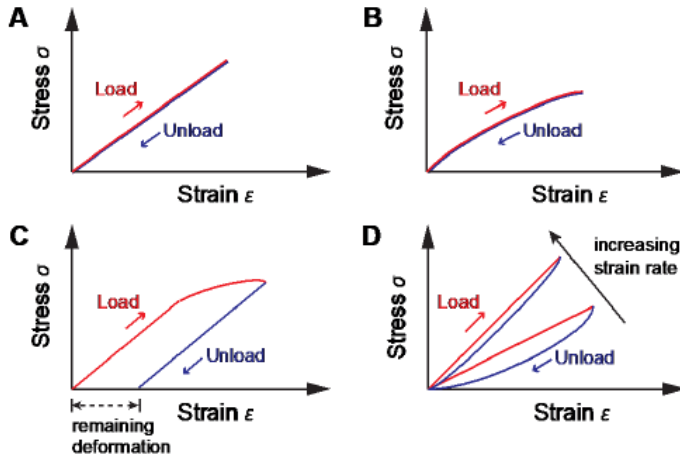


Figure 3: Stress-strain behaviour of linear elastic, non-linear elastic, elastic-plastic and viscoelastic materials while loading/unloading a material [2]

Combining a spring and a dashpot either in series or parallel enables to represent viscoelastic behaviour of a material [2]. The spring is taken as elastic component, whereas the dashpot is used for damping [2]. If the two components are connected in series, it is called Maxwell model [2]. If spring and dashpot are connected in parallel, it is called Kelvin-Voigt model [2]. There also exist more complex models that include various springs and dashpots [2]. The Kelvin-Voigt model represents viscoelastic materials, that completely recover their original shape when an applied force is withdrawn. This is the result of spring and dashpot being connected in parallel. The spring completely pulls the dashpot back when there is no force applied anymore. In contrast, the Maxwell model represents viscoelastic materials that do not completely recover their original shape. Since spring and dashpot are connected in series, the dashpot cannot be drawn back by the spring, what results in a permanent deformation.

To investigate viscoelastic materials experimentally there are two common tests [2]. The first one is the creep test [2]. Here, a constant stress is applied on a material [2]. It can be observed that the resulting strain is increasing over time (Fig. 4, left) [2]. The other test is the stress-relaxation test, where a constant strain is applied to the material [2]. As a result, the stress relaxes exponentially over time, as it can be seen in Fig. 4 on the right side [2].

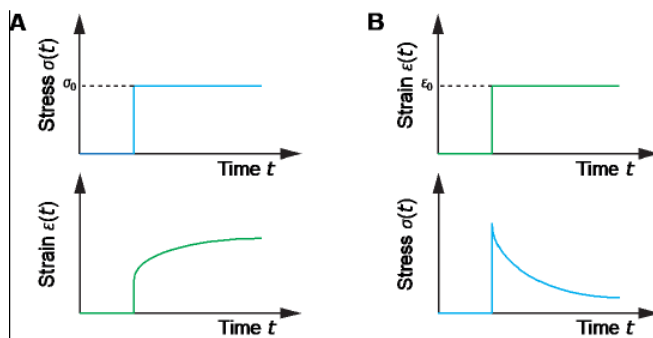


Figure 4: Behaviour of viscoelastic materials in case of a creep test (left) or a stress-relaxation test (right) [2]

2 Materials and Methods

2.1 Preparation of Elastomer Samples

First, the elastomer samples were prepared for subsequent investigation in the tensile test. Therefore, the elastomer PDMS was produced mixing varying ratios of base to curing agent. These mixtures were filled in dog-bone-like shaped forms as they are commonly used in tensile tests. The used forms varied in their width of the middle part. The height of all forms, and therefore, the obtained thickness, was approximately 1 mm. The exact composition and geometry of each sample is listed in Tab. 2.

Table 2: Composition and geometry of elastomer samples

Sample	Ratio Base/ Curing Agent	Width ideal [mm]	Base [g]	Curing Agent [g]
1	10:1	1	5	0.45
2	10:1	2	5	0.45
3	10:1	4	5	0.45
4	15:1	4	1	0.06
5	20:1	4	1	0.05

The mixture with the ratio of 10:1 was additionally used to fill a form of cylindrical shape of about 14 mm in height and 10 mm in diameter. This cylindrical PDMS sample was later used in the indentation test.

All mixed samples were then degassed for 20 min in the desiccator to remove air bubbles. Afterwards, they were placed in the oven for 60 min by 80 °C for hardening.

2.2 Determination of the Indenter's Accuracy

Calibrated weights (50 g, 100 g, 200 g, 500 g, 1000 g, 2000 g) were used for determining the indenter's accuracy. By using a wire, the weights were hung on the force transducer. The measured gravity force was then read off from the software. In general, the force was set to 0 N before every measurement, including all following tasks.

2.3 Tensile Test

After hardening of the prepared elastomer samples, tensile tests were performed. Therefore, overhangs of the dog-bone-like shaped samples were cut off. Afterwards, the obtained width as well as the thickness of the samples were measured. Then, the first sample was fixed into the grip holder of the machine and the tensile test was performed. Therefore, a standard way of 50 mm was set as expansion maximum, for which it was assumed that the sample would fracture. This was also done for the other four samples. Force and displacement were recorded by the software until the samples fractured. The standard way for sample 1 to 3 was set at 50 mm, for sample 4 at 80 mm and for sample 5 at 120 mm. For the samples 3 and 4 the initially set standard way was not sufficient to fracture the sample. So, a second standard way of 10 mm for sample 3 and of 30 mm for sample 4 was added in the software. This means, that sample 3 and 4 were first extended by a few cm, then held at this level of extension for a few seconds, and then, were further extended until the material failed.

2.4 Indentation Test

A cylindrical piece of PDMS of about 14 mm in thickness was used for the indentation test. Force-distance curves were recorded for two spherical, three cylindrical and one conical indenter that varied in their radii. The different geometries of the indenters are summarized

in Tab. 3. The tips of the indenters were approached to the surface of the sample as close as possible. Then, the sample was penetrated by the tip of the different indenters at exactly the same position by 2 mm each time, one after another. Curves for force in dependence of the time were recorded by the software during the measurement.

Table 3: Different geometries of indenters

Indenter	Shape	Diameter [mm]	Half Angle [°]
1	Spherical	10	
2	Spherical	2	
3	Cylindrical	6	
4	Cylindrical	3	
5	Cylindrical	2	
6	Conical	10	45

2.5 Viscoelasticity Test

The viscoelastic properties of Silly Putty® and Memory Foam were investigated next. It was determined which of the two materials behaves according to the Maxwell model and which one according to the Kelvin-Voigt model. Therefore, the materials were slightly deformed by hand, and it was observed how/whether the material deforms back.

2.5.1 Stress-Relaxation Test

The behaviour of Silly Putty® was observed in a stress-relaxation test. Therefore, a cylindrical indenter of 10 mm in diameter was approached to a flat position above the sample. Then, the material was penetrated by 3 mm with an intrusion velocity of 30 mm/s of the indenter. So, the measurement was position-controlled. A displacement vs. time curve was recorded for 120 s.

2.5.2 Creep Test

Last, the behaviour of Memory Foam was observed in a creep test. Therefore, a cylindrical indenter of 10 mm in diameter was approached to a flat position above the sample. The material was penetrated by 1 N with an intrusion velocity of 30 mm/s of the indenter. So, the measurement was force-controlled. A force vs. time curve was recorded for 120 s.

3 Results and Discussion

3.1 Determination of the Indenter's Accuracy

The accuracy of the indenter is analysed in the following. Tab. 4 shows the values measured as gravity force by the software for the different weights, and the expected values for the gravity force calculated by the following equation:

$$F = m \cdot g \quad (9)$$

with m as the mass of the weights in kg and $g = 9.81 \frac{m}{s^2}$ as gravitational acceleration.

Table 4: Measured and expected gravity force for different weights

Mass [kg]	Force Measured [N]	Force Expected [N]
0.05	0.489	0.491
0.10	0.979	0.981
0.20	1.960	1.962
0.50	4.898	4.905
1.00	9.796	9.810
2.00	19.592	19.620

The diagram in Fig. 5 shows the measured gravity force in dependence of the mass by the orange dots, and the expected force in dependence of the mass by the blue line. The related linear fitting functions are given in blue and orange script color. Both functions were calculated by Excel using the least square fit method. The slope of the blue line represents the gravitational acceleration. From this diagram as well as from Tab. 4 it can be seen that the measured and the expected values of the gravity force are almost identical, what leads to the deduction that the accuracy of the force measurement of the indenter is near to the optimum.

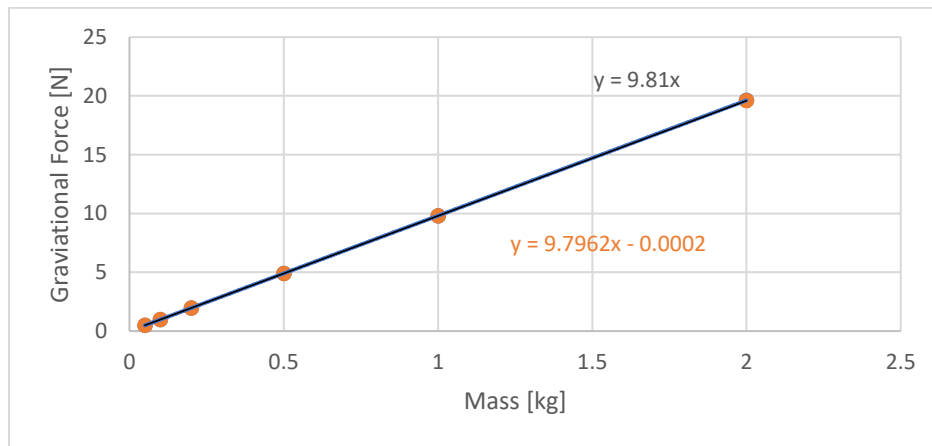


Figure 5: Measured (orange) and expected (blue) gravitational force in dependence of the mass

The absolute deviation of the slope of the measured values from the slope of the calculated values is:

$$Dev_{abs} = |Slope_{exp} - Slope_{meas}| = 9.8100 \frac{m}{s^2} - 9.7962 \frac{m}{s^2} = 0.0138 \frac{m}{s^2} \quad (10)$$

The relative deviation of the two values is:

$$Dev_{rel} = 1 - \frac{Slope_{meas}}{Slope_{exp}} = 0.0014 = 0.14 \% \quad (11)$$

This small absolute and relative deviation of the measured from the expected values can be explained by small inaccuracies either in calibration of the weights or of the indentation machine. However, these vanishing deviations can be neglected. Thus, this excellent accuracy of the indenter enables a precise force measurement for subsequent tests conducted in this practical.

3.2 Tensile Test

The dog-bone-like shaped elastomer samples to investigate in the tensile test were measured for their real width and thickness after their preparation. The measured values are shown in Tab. 5. The cross-sectional area A was calculated by the following equation (here exemplary for sample 1):

$$A = Width_{meas} \cdot Thickness_{meas} = 1 \text{ mm} \cdot 0.98 \text{ mm} = 0.98 \text{ mm}^2 \quad (12)$$

Table 5: Measured width and thickness of elastomer samples and calculated cross-sectional area A

Sample	Width Measured [mm]	Thickness Measured [mm]	A [mm ²]
1	1	0.98	0.98
2	2	1	2
3	4	1	4
4	4	1	4
5	4	0.99	3.96

Fig. 6 to 10 are showing the recorded stress-strain curves of the different elastomer samples. The values for stress (σ) and strain (ϵ) were calculated from the values of force (F) and displacement (ΔL), measured by the software during the tensile test. For calculating stress and strain, equations 1 and 2 were used. The initial length of the samples L_0 was always assumed as 14.3 mm [2]. The values for A can be seen in Tab. 5.

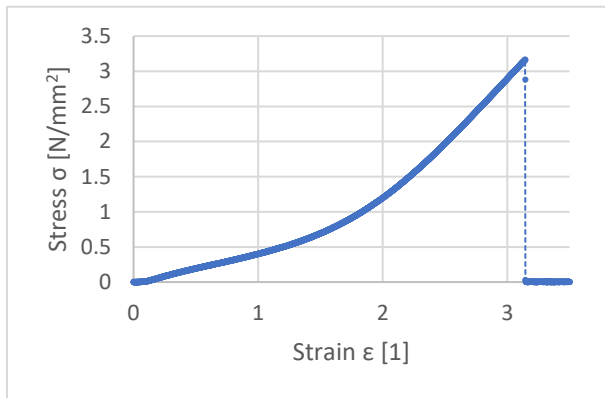


Figure 6: Stress-strain curve of sample 1

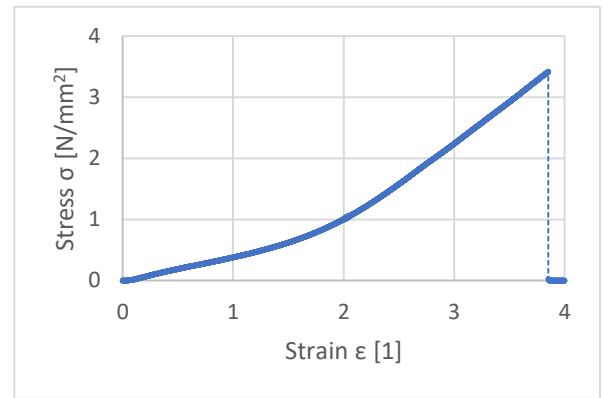


Figure 7: Stress-strain curve of sample 2

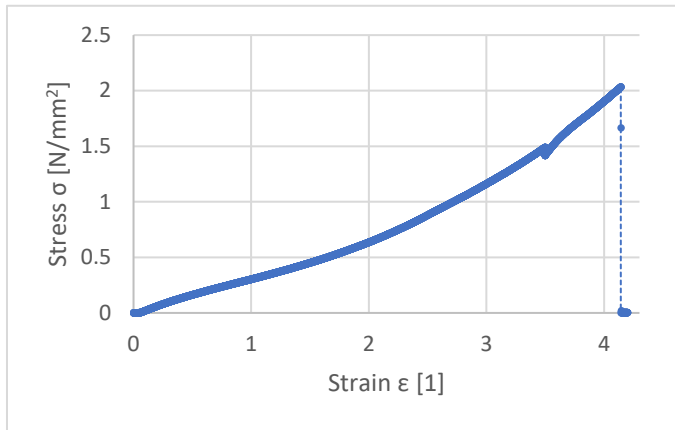


Figure 8: Stress-strain curve of sample 3

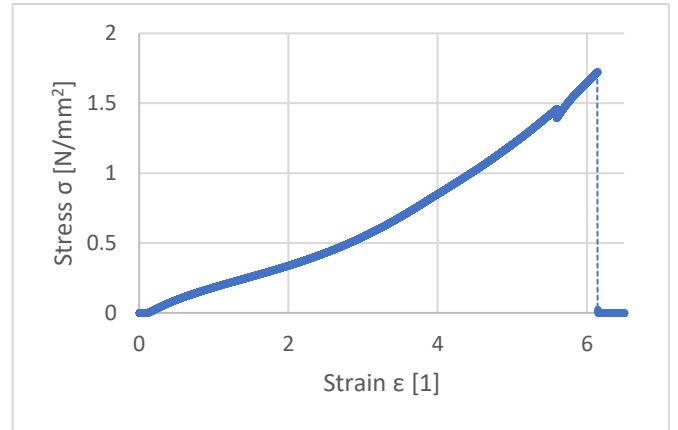


Figure 9: Stress-strain curve of sample 4

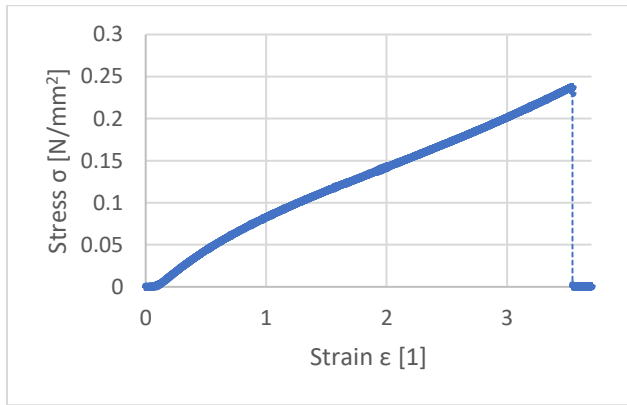


Figure 10: Stress-strain curve of sample 5

The stress-strain curves of all five samples show an increasing strain with increasing stress up to a certain point where the stress abruptly drops back to $0 \frac{N}{mm^2}$. Here, the samples fractured. Therefore, these curves represent the typical behaviour of elastomers in a tensile test. For elastomers, the deformation is always elastic, and thus, reversible. There is no plastic regime as it can be found for ductile polymers. So, there is also no yield point. The slope of the stress-strain curves is almost linear up to the point where the material fails. For low ϵ ($\sim 0 - 0.2$) the curves recorded in this experiment behave non-linear. This is a result of the samples not being under tensile force when starting the measurement, they are loose. So, the stress has to build up first in the material. Furthermore, when applying a force on the elastomer sample, its molecules have to be parallelly oriented before stretching, as they are often arranged in loops without stress.

The small kinks in the curves of the samples 3 (Fig. 8) and 4 (Fig. 9) at $\epsilon = 3.5$ and $\epsilon = 5.6$ respectively, occurred due to the fact that the initially configured standard way of the measurement was not sufficient to fracture the sample. Thus, the standard way was extended in a second step. The two curves were then stringed together.

The ultimate strength stress of the five samples can be read out of the maximum point of the respective stress-strain curves. This point coincidentally represents the upper limit of the elastic regime because of the elastomer-like behaviour of the samples. Young's modulus was calculated with equation 3 in the interval between $\epsilon = 2$ and $\epsilon = 3$ for all samples, with the least square fit function in Excel. The obtained values for the ultimate strength stress and for Young's modulus are represented in Tab. 6.

Table 6: Values for Young's modulus and the ultimate strength stress for the samples

Sample	Young's Modulus [MPa]	Ultimate Strength Stress [N/mm ²]	Value for ϵ at Ultimate Strength Stress [1]	A [mm ²]	Ratio Base/ Curing Agent [1]
1	1.72	3.17	3.14	0.98	10:1
2	1.26	3.42	3.85	2	10:1
3	0.53	2.04	4.14	4	10:1
4	0.21	1.73	6.14	4	15:1
5	0.06	0.24	3.54	3.96	20:1

Tab. 6 shows that for the samples 1 to 3, that have the same ratio of base to curing agent, but different cross-sectional areas, Young's modulus decreases with increasing cross-sectional area. This may be due to a lower ratio of crystalline and a higher ratio of amorphous parts in the affected sample cross section when preparing a sample with higher cross-sectional area. The ultimate strength stress is in approximately the same range for these three samples. Theoretically, it can be expected that samples having the same ratio of base to curing agent will fracture at the same stress. This should be due to the fact, that the stress is calculated out of the ratio force vs. cross-sectional area. Since the force required to fracture the sample increases with increasing cross-sectional area, the ratio remains the same. Although, the samples 1 to 3 have an ultimate strength stress in the same range, the values differ from each other. There are several reasons that might explain these deviations between the values. First, when calculating the stress, the cross-sectional area, to which the force is related to, is the initial cross-sectional area. It has to be considered, that the real cross-sectional area, the force acts on during the tensile test, decreases with increasing time due to elongation of the sample. Thus, the real stress becomes higher if the cross-sectional area shrinks. Furthermore, if the initial cross-sectional area of the material is larger, there is a higher possibility for a defect in the material. Therefore, the cross-linking becomes more inhomogeneous for a larger cross-sectional area, what leads to a decreasing ultimate strength stress. Moreover, the samples 1 to 3 show that the value of the strain at the ultimate strength stress increases with an increasing cross-sectional area. This is logical because the sample becomes more stable due to a higher primary linkage of the molecules that has to be broken up before the sample fractures.

When observing the influence of a changing ratio of base to curing agent, Tab. 6 shows for the samples 3 to 5 a decreasing Young's modulus with increasing ratio of base to curing agent, what makes sense because of the samples becoming softer. Thus, there is less primary linkage between the molecules. It can be seen, that in our experiment, the ultimate strength stress of the samples decreased with an increasing proportion of the base. This might be explained by the fact that the higher the ratio of the base in a sample, the less cross-linking is present. However, it has to be considered that the tensile test for the samples 3 and 4 was performed in two steps, because the initial value that was set for the standard way was not high enough to fracture the sample, what potentially influenced the ultimate strength stress. Furthermore, in sample 5 an air bubble could be detected at the point where the sample fractured. At that point the sample was significantly more instable than at the rest, what led to a much lower ultimate strength stress of $0.238 \frac{N}{mm^2}$, compared to the other samples. In Tab. 6 it can be seen that the ϵ -values at the point for the ultimate strength-stress increase with an increasing ratio of base to curing agent for the samples 3 and 4. This shows, that for a higher ratio of base to

curing agent, the elastomer sample become more extensible. The reason that the measured ϵ -value for sample 5 does not fit to this assumption is, that the sample fractured too early due to the air bubble in the middle of the polymer. In total, it can be concluded that less cross-linking in an elastomer sample leads to a higher elasticity, although, the ultimate strength stress is decreasing.

Another aspect that has to be considered in general when evaluating these results is that the two screws, where the dog-bone-like shaped elastomer samples were fixed in between, are twisted by a few degrees to each other. This might result not only in a tensile force, but also in a torsional force. However, this should not have a significant influence on the force to displacement measurement. Another issue that potentially disturbs the measurement is that the different samples cannot be fixed identically between the screws of the testing machine each time, what leads to slightly varying starting conditions that might influence the fracture point. A different strength of the clamping might play a role when thinking about the samples potentially sliding along a low distance through.

3.3 Indentation Test

For determining Young's modulus of the PDMS sample investigated in the indentation test, force vs. time curves were recorded. As it can be seen in Fig. 11 on the left side each, these curves are not linear. Therefore, the equations describing the force-indentation relationship for the different tip geometries (mentioned in 1.2.2) had to be linearized. The linearized force was then plotted against the displacement, what can be seen for the different indenters in Fig. 11, on the right side each. Young's modulus was determined by applying the least square fit function in Excel on the slope of the linearized curves, in the range between 10 s and 20 s after starting the indentation measurement. The calculated values for the slope and Young's modulus can be seen in Tab. 7.

Spherical:

$$F(\delta) = \frac{4}{3} \frac{E}{1-\nu^2} \sqrt{R} \delta^{3/2}$$

linearize:

$$F(\delta)^{2/3} = \left(\frac{4}{3} \frac{E}{1-\nu^2} \sqrt{R} \right)^{2/3} \delta$$

with a as slope:

$$F(\delta)^{2/3} = a \cdot \delta$$

$$a = \left(\frac{4}{3} \frac{E}{1-\nu^2} \sqrt{R} \right)^{2/3}$$

Young's modulus:

$$E = a^{3/2} \cdot \frac{3}{4} (1 - \nu^2) \cdot \frac{1}{\sqrt{R}}$$

Conical:

$$F(\delta) = \frac{2}{\pi} \frac{E}{1-\nu^2} \tan \alpha \delta^2$$

linearize:

$$F(\delta)^{1/2} = \left(\frac{2}{\pi} \frac{E}{1-\nu^2} \tan \alpha \right)^{1/2} \delta$$

with a as slope:

$$F(\delta)^{1/2} = a \cdot \delta$$

$$a = \left(\frac{2}{\pi} \frac{E}{1-\nu^2} \tan \alpha \right)^{1/2}$$

Young's modulus:

$$E = a^2 \cdot \frac{\pi}{2} (1 - \nu^2) \cdot \frac{1}{\tan \alpha}$$

Cylindrical:

$$F(\delta) = 2 \frac{E}{1-\nu^2} R \delta$$

linearize: is already linear

with a as slope:

$$F(\delta) = a \cdot \delta$$

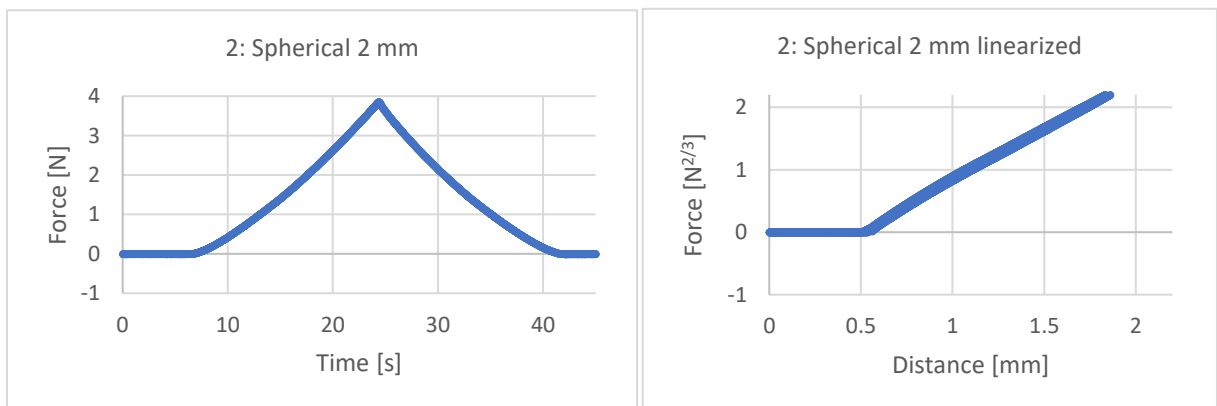
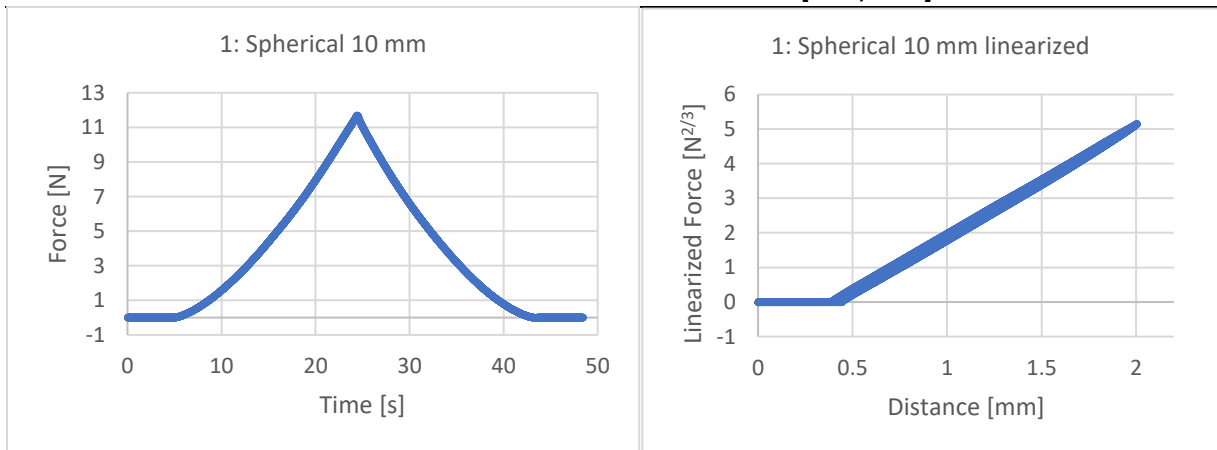
$$a = 2 \frac{E}{1-\nu^2} R$$

Young's modulus:

$$E = a \cdot \frac{1}{2} (1 - \nu^2) \cdot \frac{1}{R}$$

Table 7: Calculated values for slope and Young's modulus for different indenters

Indenter	Shape	Diameter [mm]	Angle [°]	Slope a	Young's Modulus E [MPa]
1	Spherical	10		3.15 [N ^{2/3} /mm]	1.40
2	Spherical	2		1.59 [N ^{2/3} /mm]	1.03
3	Cylindrical	6		11.37 [N/mm]	1.42
4	Cylindrical	3		5.04 [N/mm]	1.26
5	Cylindrical	2		3.56 [N/mm]	1.34
6	Conical	10	45	1.07 [N ^{1/2} /mm]	1.34



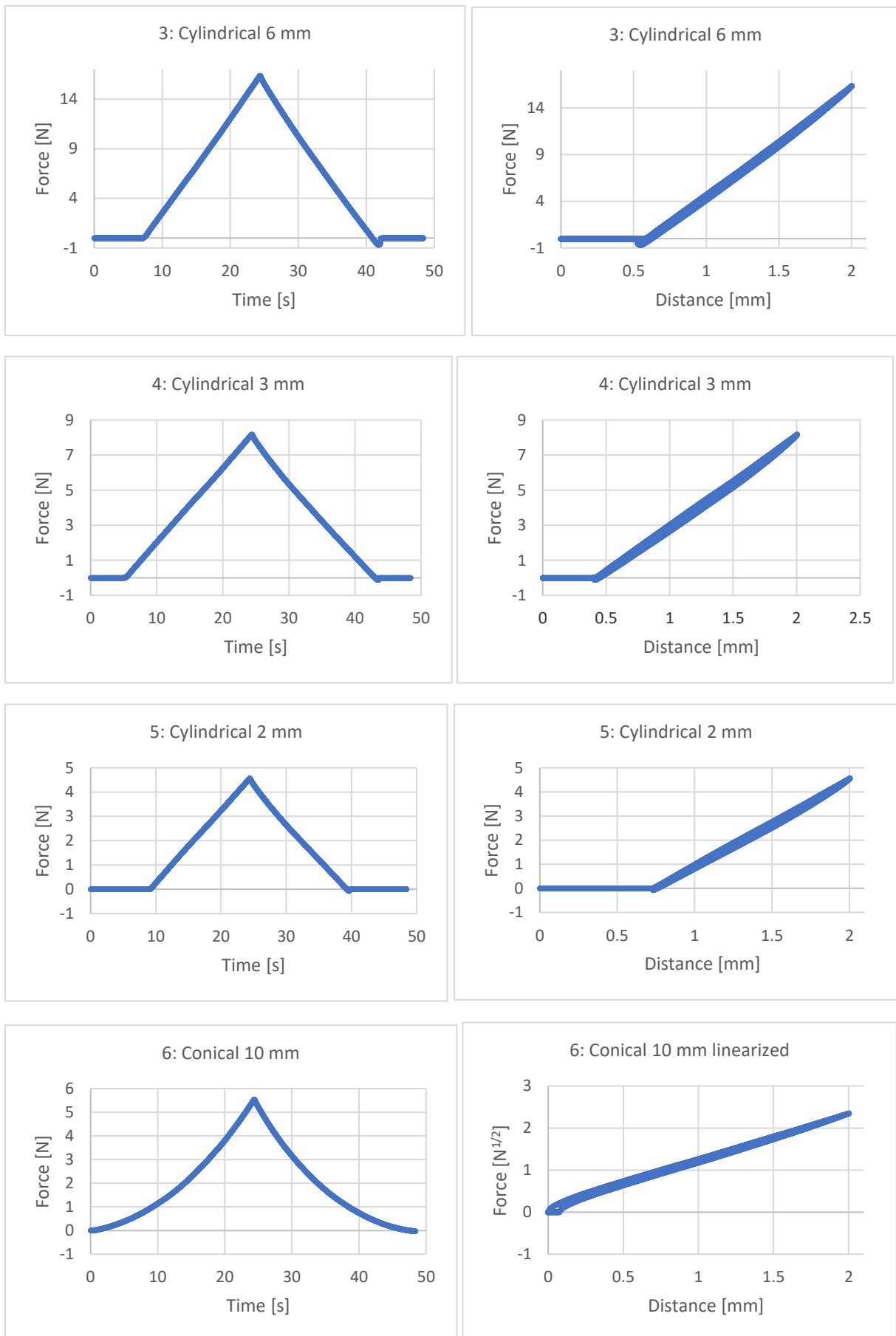


Figure 11: Measured and linearized curves of the indentation test

As it can be seen in Fig. 11, on the right side each, the measured curves that show the force dependent on the time, for spherical and conical indenters, were not linear. However, after linearizing the equations and plotting new curves of the force vs. linearized distance, the paths for loading and unloading the sample become linear. Thus, the linearization was successful. Most of the curves have a symmetrical path for loading and unloading. The force vs. distance curve of the conical indenter has slightly differing paths for loading and unloading. This might be due to either a slightly viscoelastic behavior of the material, or most likely, a minor puncture of the indenter in the material, or due to adhesion of the indenter.

As it can be seen in Tab. 7 the calculated values for Young's modulus for the different indenters vary between 1.03 and 1.42 MPa. The values also vary for the same shape of the indenters but with different geometric dimensions. The larger the effective cross section of the tip that penetrates the sample, the higher becomes Young's modulus. A potential explanation might be that if more material is loaded, the more cross links in the molecular structure are loaded. Thus, these results are more representative than just loading a smaller cross section of the sample. Furthermore, when loading a higher cross section, polymer molecules are not able to dodge the tip of the indenter. It can be observed that Young's modulus is similar when looking at the spherical, cylindrical and conical indenters with the largest diameter each. The fact that the tips not always had the same distance to the sample before starting the measurement should not have an influence, because only the linear parts of the curves were considered in these calculations.

As it could be seen in this practical, Young's modulus of a material can either be determined by conducting a tensile test, or by conducting an indentation test. Each of these two tests has its own characteristics, and therefore, influences the results of the measurement. When applying a tensile test, it has to be ensured that the set standard way is chosen sufficiently long to fracture the sample, and that it has not to be extended, as it happened in this practical. Another point that has to be considered for the tensile test is that the resulting stress is related to the cross-sectional area, that changes when a tensile force is applied and the material samples becomes elongated reducing its effective cross section. Additionally, included air bubbles can lead to an early fracture of the sample. Furthermore, the sample is fixed between the screws slightly different each time. When conducting an indentation test, Young's modulus is dependent on the geometry of the testing element as it can be seen in Tab. 7. The appropriate geometry of the testing element might also be dependent on the type of the sample. During the tensile test, the same kind of measurement is conducted every time, there are no parameters, e.g. like the tip geometry, to be chosen. In general, the tensile test seems to be a technically easier and more direct approach to determine Young's modulus. The value can just be determined by calculating the slope of the stress-strain curve. When using the indentation test, the curves recorded for conical and spherical tip geometries have to be linearized first.

When comparing the results obtained for Young's modulus via tensile and indentation test for a PDMS sample of the ratio 10:1, it can be seen that the values are approximately in the same range. As mentioned above, the exact values depend on the cross-sectional area, when applying a tensile test, and on the different geometries of the indenter, when applying an indentation test.

Furthermore, the indentation test requires some assumptions of contact models that have to be fulfilled when applying this test. Otherwise, the results of the measurement might be incorrect. As mentioned in 1.2.2 these assumptions include that the penetration depth of the

indenter has to be smaller than the sphere's radius [2]. As the penetration depth is only 2 mm in this experiment, this assumption is fulfilled. Another requirement, that the E modulus of the indenter is much higher than the E modulus of the sample is also given here, since the E modulus for steel is far higher than for polymers [2]. Furthermore, the assumption of Poisson's ratio being 0.5 for incompressible materials is fulfilled in this experiment [2].

3.4 Viscoelasticity Test

The materials investigated for viscoelasticity were Silly Putty® and Memory Foam. It was observed that Silly Putty® behaves as a Maxwell material. When deforming Silly Putty® manually by a few millimeters, it can be seen that the material does not take back its original shape, it is deformed irreversibly. Since the deformation of Memory Foam is completely reversible when waiting a few seconds, it can be deduced that this material behaves according to the Kelvin-Voigt model.

When observing the force vs. time behaviour in a stress-relaxation test for the Maxwell material, and the indentation vs. time behaviour in a creep test for the Kelvin-Voigt material, the required perfect step in strain or stress can practically not be applied. However, the response of the material still looks approximately like expected. For the perfect step, an infinite velocity when indenting the material would be required. Thus, if applying a high-speed velocity (here 30 mm/s) for a short distance (here: Maxwell model – 3 mm indentation, Kelvin-Voigt model – 4 mm indentation), the response is approximately similar as when applying an infinite velocity.

3.5 Maxwell Material

Fig. 12 and 13 show the measurement results of displacement over time and force over time for Silly Putty® when conducting a stress-relaxation test. In Fig. 12, it can be seen that the displacement from 0 to 3 mm approximately behaves like a perfect step, because the change of the displacement happens in the fraction of a second. Since the indentation depth δ_0 of the material is hold constant at 3 mm the displacement does not change over time. However, the resulting force applied to the material drops exponentially with time after having reached the desired indentation depth, as it was expected. Both diagrams only show displacement and force for the first few seconds of the measurement and not for the complete 120 s, because after a few seconds, there were no remarkable changes in the curves anymore.

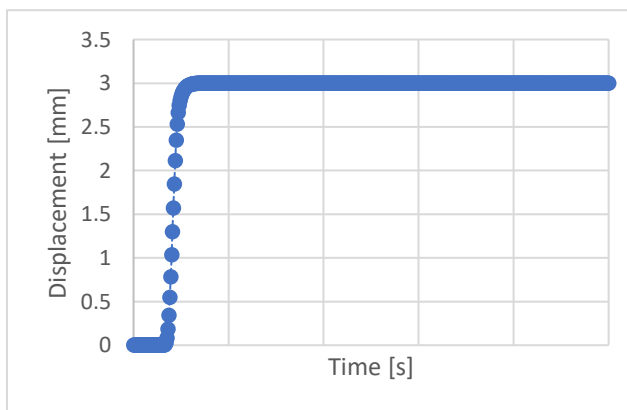


Figure 12: Stress-relaxation test, displacement vs. time

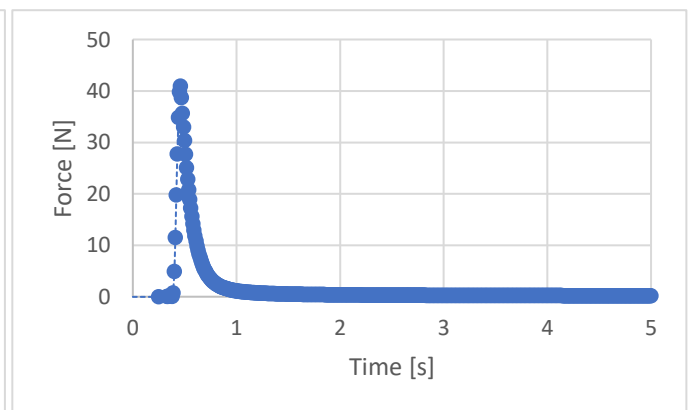


Figure 13: Stress-relaxation test, force vs. time

The relevant time period for determining Young's modulus E_0 , the relaxation time τ and the dynamic viscosity η of Silly Putty®, is between the point of maximum force F_{max} until approximately one second after this point. This part of the curve can be seen in Fig. 14. The

original curve from Fig. 13 was shifted along the time axis, so that the maximum force was set at $t = 0$ s.

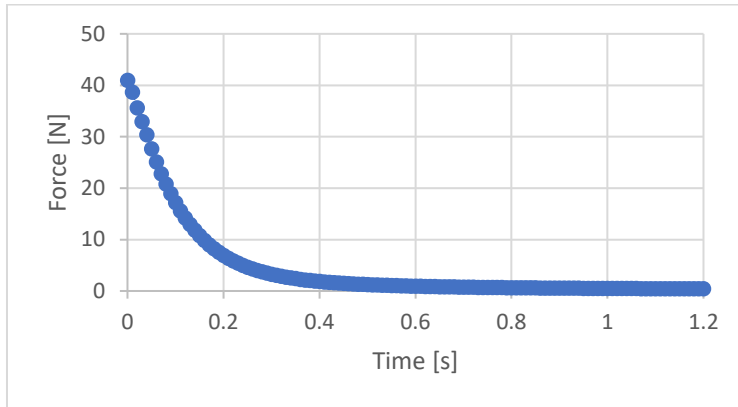


Figure 14: Part of force vs. time diagram relevant for determination of E_0 , τ and η

The force vs. time behaviour in a stress-relaxation test for a cylindrical indenter with radius R and step indentation δ_0 is given by [2]:

$$F(t) = \frac{8}{3} R \delta_0 \cdot E_0 e^{-t/\tau} \quad (13)$$

For $t = 0$ s the maximum force can be read out of the diagram. Thus, $F_{\max}$ was determined as 40.94 N. Out of this, and by knowing the radius of the indenter to be $R = 5$ mm, and the step indentation δ_0 to be 3 mm, E_0 can be calculated:

$$E_0 = \frac{3}{8} \cdot \frac{F_{\max}}{R \delta_0} = \frac{3}{8} \cdot \frac{40.94 \text{ N}}{5 \text{ mm} \cdot 3 \text{ mm}} = 1.03 \text{ MPa}$$

The relaxation rate τ is calculated from the measurement point where $\frac{t}{\tau} = 1$. This results in:

$$F(t) = \frac{8}{3} \cdot 5 \text{ mm} \cdot 3 \text{ mm} \cdot 1.03 \text{ MPa} e^{-1} = 15.16 \text{ N}$$

The respective time t for this calculated force is taken from Fig. 14 as $t = 0.11$ s. Due to the assumption that $\frac{t}{\tau} = 1$, τ results as, $\tau = 0.11$ s.

The dynamic viscosity η can be calculated by [2]:

$$\eta = \tau \cdot E_0 = 0.11 \text{ s} \cdot 1.03 \text{ MPa} = 113.3 \text{ kPa} \cdot \text{s} \quad (14)$$

The determined value of Young's modulus of approximately 1 MPa seems to be logical, as Silly Putty® is a quite soft material. It is by far softer than e.g. metals (100 GPa) or wood (10 GPa) [5]. The calculated value for the relaxation time of 0.11 s and Young's modulus of 1.03 MPa also seem logical. However, both Young's modulus and the relaxation time could be influenced during the stress-relaxation test by the geometry of the indenter and its indentation velocity.

As the viscosity is calculated out of the relaxation rate and Young's modulus, the determined value for viscosity is influenced by all parameters that influence the relaxation rate and Young's modulus. The calculated viscosity of 113.3 kPa·s for Silly Putty® shows that Silly Putty® is a highly viscous material. The viscosity was just determined as parameter in this experiment. However, a Maxwell material shows a non-Newtonian behaviour. Therefore, the viscosity is dependent on the indentation velocity.

3.6 Kelvin-Voigt Material

Fig. 15 and 16 show the results of force plotted against time, and displacement plotted against time for Memory Foam when conducting a creep test. In Fig. 15, it can be seen that the set value for the force of 1 N is reached in steady state after about 5 s. There is an almost perfect step for the force between 0 N and around 1 N. However, the feedback system needs 5 s because measuring with a constant force is significantly more difficult than measuring with a constant displacement. After having reached the desired force of 1 N after about 5 s, it can be seen in Fig. 16 that the curve for displacement shows the typical behaviour for a creep test. The displacement grows asymptotically towards a threshold value, that is 5.84 mm. Both diagrams show displacement and force for the complete measuring time of 120 s, and therefore, the maximum displacement value can be read off.

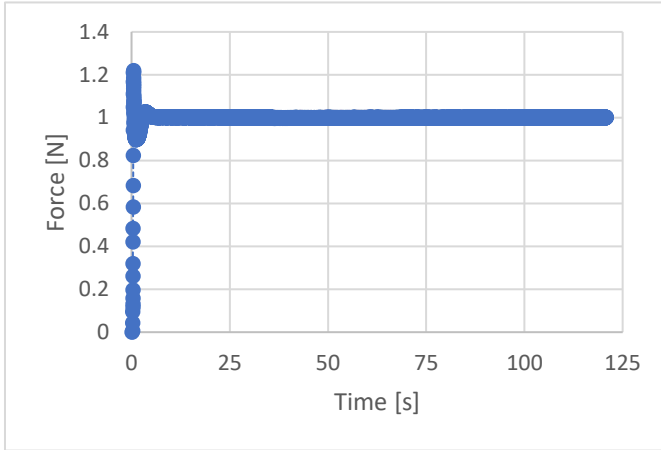


Figure 15: Creep test, force vs. time

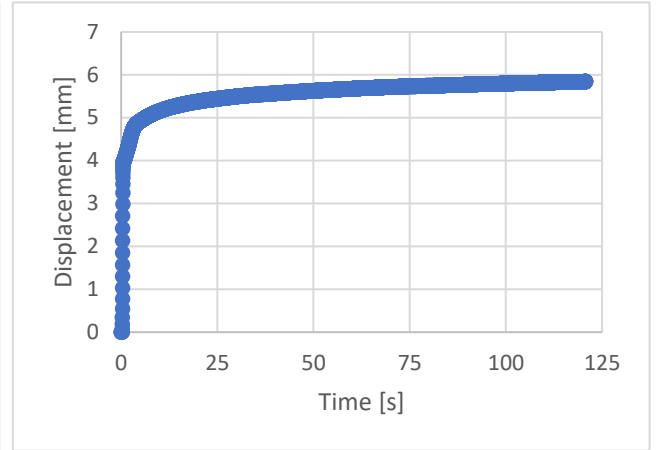


Figure 16: Creep test, displacement vs. time

For determining Young's modulus E_∞ , the relaxation rate τ and the dynamic viscosity η for the Memory Foam, the time period of interest is in between the approximately first 10 s after starting the measurement. Another point of interest is the limiting value the displacement vs. time curve is approaching to.

The indentation vs. time behaviour in a creep test for a cylindrical indenter with radius R and step force F_0 is given by [2]:

$$\delta(t) = \frac{3 F_0}{8 R} \cdot \frac{1}{E_\infty} (1 - e^{-t/\tau}) \quad (15)$$

The maximum displacement for $t \rightarrow \infty$ of 5.84 mm can be read out of Fig. 16. The force is constantly held at $F_0 = 1$ N. Out of this, and by knowing the radius of the indenter to be $R = 5$ mm, Young' modulus E_∞ can be determined:

$$E_\infty = \frac{3 F_0}{8 R \delta_{max}} = \frac{3}{8} \frac{1 \text{ N}}{5 \text{ mm} \cdot 5.84 \text{ mm}} = 12.84 \text{ kPa}$$

The relaxation rate τ can then be calculated by assuming $\frac{t}{\tau} = 1$. This results in:

$$\delta(t) = \frac{3}{8} \frac{1 \text{ N}}{5 \text{ mm}} \cdot \frac{1}{0.01284 \text{ MPa}} (1 - e^{-1}) = 3.69 \text{ mm}$$

The respective time t for this calculated indentation is $t = 0.46$ s. Due to the assumption that $\frac{t}{\tau} = 1$, τ results as $\tau = 0.46$ s.

The effective dynamic viscosity η can be calculated by [2]:

$$\eta = \tau \cdot E_{\infty} = 0.46 \text{ s} \cdot 12.8 \text{ Pa} = 5.9 \text{ kPa} \cdot \text{s}$$

Since the system needs around 5 s until a constant force is effectively applied on the Memory Foam, it has to be considered that the correct values for displacement also occur after 5 s. However, when calculating the indentation depth by equation 15, this cannot be considered. Therefore, especially the value for the relaxation time must be seen with caution. The determined value for Young's modulus of 12.84 kPa seems to make sense, as Memory Foam is a soft material. It should be noted that the Memory Foam was uneven. So, the applied force was not evenly distributed by the indenter.

For the reason that the dynamic viscosity is calculated out of the relaxation rate and Young's modulus, that are calculated out of the measured values, the calculated value for viscosity is influenced by all parameters that influence the relaxation rate and Young's modulus. These factors are the same as mentioned above for the measurement of the Maxwell material. The calculated viscosity of 5.9 kPa·s for Memory Foam is much higher than the viscosities of water (10^{-3} Pa·s) and honey (10 Pa·s) [5], but lower than the previously calculated viscosity for Memory Foam (section 3.5). Therefore, it can be assumed that the calculated viscosity for Memory Foam is approximately in the right range. However, the value will be slightly different each time when determining it in the way it was done in this experiment.

4 Conclusion

Knowledge about the mechanical behaviour of different tissues and materials is crucial in biomedical engineering, since material properties are important when e.g. building implants, or when investigating whether a tissue is pathological.

In this experiment, the mechanical properties of PDMS were investigated by standard testing methods as the tensile and the indentation test. Young's modulus was determined and compared for different ratios of base and curing agent of the PDMS samples, as well as for varying cross-sectional areas of the samples in the tensile test. It could be seen that samples with a higher cross-sectional area and a higher amount of base could be more extended compared to samples with lower cross-sectional area and a lower amount of base. However, the ultimate strength stress and Young's modulus decreased. The stress-strain relationship of PDMS showed the typical linear behaviour of an elastomer under an applied tensile load.

The influence of different tip geometries on Young's modulus was observed in an indentation test. Here, an observation was that the determined Young's modulus was higher for indenters having a larger diameter. It could be seen that the determined value for Young's modulus in the tensile test is similar to Young's modulus determined in the indentation test. However, in general, the tensile test seemed to be a better physically founded approach than the indentation test to determine Young's modulus.

The materials Silly Putty® and Memory Foam were examined for their viscoelasticity properties in a stress-relaxation and a creep test, respectively. When indenting these materials, it could be seen that Silly Putty® behaves like a Maxwell material and that Memory Foam behaves as Kelvin-Voigt material.

5 Literature

1. Prasad, K., et al., *Metallic Biomaterials: Current Challenges and Opportunities*. Materials, 2017. **10**: p. 884.
2. *Indenter Lab Instruction Manual*. University of Tübingen, Winter Term 2021/22.
3. Dietmann, H., *Einführung in die Elastizitäts- und Festigkeitslehre*. 1988, Stuttgart: Alfred Kröner Verlag.
4. Haque, S., et al., *Application and Suitability of Polymeric Materials as Insulators in Electrical Equipment*. Energies, 2021. **14**: p. 2758.
5. *Lecture Nanoanalytics and Interfaces*. University of Tübingen, Winter Term 2021/22.