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# 1 Motivation

In biomedical technologies, material properties play an important role in different ways. For example, different pathological cells as cancer cells have different stiffness compared to healthy cells which leads to a possible selection of cells through their material properties [1]. Another example are implants. To reduce tissue degradation in people wearing implants as hip or knee prostheses, implants aim to have as similar as possible material properties in comparison to human tissue [2]. The same approach is also used in tissue engineering, where tissue is synthetically produced in the lab [3]. These relevant material properties include e.g. stiffness and surface structure. To measure especially the stiffness of materials different techniques were developed during the past decades [4].

## 2 Theoretical Background

### 2.1 Mechanics

Material properties are a useful tool to compare different materials to each other and decide which materials are suitable to replace certain other materials. One of the most common material properties is the Young's modulus. The Young's modulus describes the behavior of a material under uniaxial stress or strain and is defined as

$$E = \frac{\sigma}{\epsilon} \quad (1)$$

where  $\sigma$  is the stress applied. The stress is defined as

$$\sigma = \frac{F}{A} \quad (2)$$

with  $F$  being the force and  $A$  the cross-section area.

$\epsilon$  is the deformation of length which is called axial strain and is defined by equation 2 [5].

$$\epsilon = \frac{\Delta L}{L} \quad (3)$$

Further interesting material constants are the bulk modulus, which is a measurement of the resistance against compression of a certain material and the shear modulus which refers to the resistance against shear forces of a material. However these two moduli are not part of the labwork and also would exceed the scope of this protocol [6].

### 2.2 Tensile tests

Tensile tests are usually done with dog-bone-shaped samples and measure the force over time during elongation which is then increased until the material breaks. While doing the elongation, the force which is applied is measured. These tensile tests only measure traction force [7]. The different stress-strain curves look different for different material types as elastomers or ductile materials. By taking the slope of the stress-strain diagram in the linear

regime, the Young's modulus of the tested material can be calculated [8]. An example of a typical stress-strain curve for elastomers can be seen in figure 3.

## 2.3 Indentation

A very commonly used test to find out about material properties is indentation. Here a specifically designed form is pressed onto a material which responds to stress in a specific way. By that, one can figure out the material properties [8]. The indenter plays an important role and defines the reaction of the sample material of the shape and material properties. For different indenter shapes, different formulas can be used to calculate the Young's modulus which describe the relationship between force and indentation with the respective geometries of the indenters [8]. Table 1 shows the relation for the different indenters and the linearized formulas which are used to calculate the Young's modulus [9]. Since the experiment does not deliver the Young's modulus of the sample, formula 4 shows a way to transfer the slope parameters to the formulas given in table 1 in column 3 by linearization of the indentation  $\delta$ . These formulas are defined in the Hertzian model.

$$F^x = a * \delta \quad (4)$$

Where F is the force x the respective exponent, a is the slope of the measured force-indentation curve and  $\delta$  is the indentation.

| Tip geometry | Force-indentation relationship                                                     | Linearized formula                                                                             |
|--------------|------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------|
| Spherical    | $F(\delta) = \frac{4}{3} \cdot \frac{E}{1-v^2} \cdot \sqrt{R} \cdot \delta^{3/2}$  | $F^{2/3} = \left( \frac{4 \cdot E \cdot \sqrt{R}}{3 \cdot (1-v^2)} \right)^{2/3} \cdot \delta$ |
| Conical      | $F(\delta) = \frac{2}{\pi} \cdot \frac{E}{1-v^2} \cdot \tan \alpha \cdot \delta^2$ | $F^2 = \left( \frac{2 \cdot E \cdot \tan \alpha}{\pi \cdot (1-v^2)} \right)^2 \cdot \delta$    |
| Cylindrical  | $F(\delta) = 2 \cdot \frac{E}{1-v^2} \cdot R \cdot \delta$                         | $F = \frac{2 \cdot E \cdot R}{(1-v^2)} \cdot \delta$                                           |

Table 1: The formulas for the different indenters used in this experiment. F is the force, E the Young's modulus v the Poisson's ratio which was assumed to be 0.5, R is the radius,  $\delta$  the indentation and  $\alpha$  the half-angle of the conical indenter [9].

## 2.4 Viscoelastic materials

Viscoelastic materials show viscous and elastic characteristic properties. Two basic rheological models can be considered. The Kelvin-Voigt model can be described as an equivalent circuit with a dashpot and a spring which are parallel while the Maxwell model has these components in series. Graphically, the difference between the two models is, that the Maxwell material does not move back into its original shape while the Kelvin-Voigt material fully

recovers its original shape [10][11]. For Maxwell materials, an optional test is a stress relaxation test where a constant strain is induced and the stress is measured [10]. An option for testing a Kelvin-Voigt material is the creep test, which uses constant stress and measures the development of the strain [12][11].

### 3 Experimental Setup and materials

To start the experiment, first the samples had to be prepared. For that PDMS was mixed with PDMS cross-linker in mixture ratios of 10:1, 15:1 and 20:1. After mixing the two components the substance was poured into the forms, which were then placed into the desiccator to remove gas bubbles inside the substance and avoid irregularities in the samples. Then the samples were baked for 40 minutes at 80°C. After finishing the preparation, different tests were performed. One probe of each mixture was produced for a tensile test and put into dog-bone-shaped forms as shown in figure 1. Only for the 1:10 mixture three different samples with different widths of the bridge in the middle. A cylinder of the 10:1 mixture was produced for the indentation tests. Last, two viscoelasticity tests, the creep and relaxation test were performed for a Maxwell and a Kevin-Voigt material. The machine used was a Zwicki Line 500 testing machine.

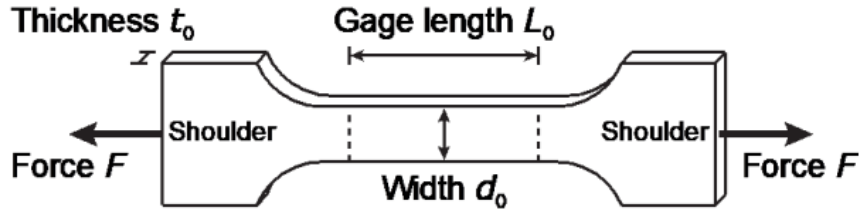


Figure 1: Schematic of the dog-bone shaped PDMS sample

## 4 Results

### 4.1 Loading cell evaluation

The first step was an evaluation of the testing machine. Therefore different normed weights were used to record the applied force by them on the machine. Figure 2 shows the recorded points as well as the linear function resulting of the measurements.

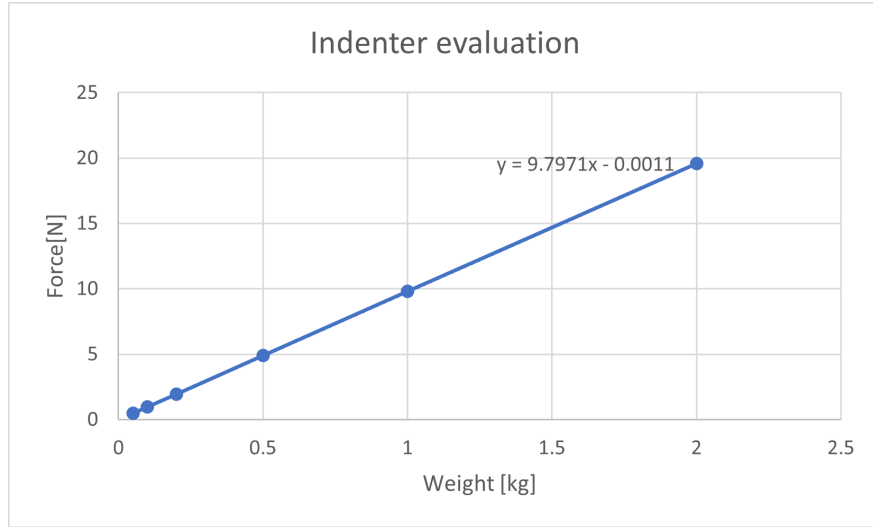


Figure 2: Force-weight curve of the normed weights to evaluate the machine

### 4.2 Tensile test

For the tensile tests of the different PDMS probes, the samples were clamped into the machine and then a stress test was performed where the elongation was increased consistently until the material broke. Prior to the testing procedure, the samples were inspected and overhangs were cut with a scalpel. No obvious material errors were found. The geometries as well as the ultimate tensile stress and the calculated Young's moduli of the five used samples based on the end of the linear regime are displayed in table 2. Since the stress-strain curves can be approximated by a linear function, the maximum of the respective function was used. The resulting diagrams of the tensile testing procedure are shown in figures 3 and 8-11.

|                | Ultimate tensile stress<br>[ $N/mm$ ] | Young's Modulus<br>[ $Pa$ ] | Width<br>[ $mm$ ] | Mixture | Thickness<br>to<br>[ $mm$ ] | Gage length<br>[ $mm$ ] |
|----------------|---------------------------------------|-----------------------------|-------------------|---------|-----------------------------|-------------------------|
| <b>Sample1</b> | 1.392.886                             | 697.998                     | 1                 | 1:10    | 1                           | 14,3                    |
| <b>Sample2</b> | 4.637.650                             | 1.166.864                   | 2                 | 1:10    | 1                           | 14,3                    |
| <b>Sample3</b> | 2.195.540                             | 736.557                     | 4                 | 1:10    | 1                           | 14,3                    |
| <b>Sample4</b> | 711.459                               | 345.747                     | 4                 | 1:15    | 1                           | 14,3                    |
| <b>Sample5</b> | 834.699                               | 462.327                     | 4                 | 1:20    | 1                           | 14,3                    |

Table 2: Different sample geometries and Young's moduli calculated by the end of the linear regime aswell as the ultimate tensile stress

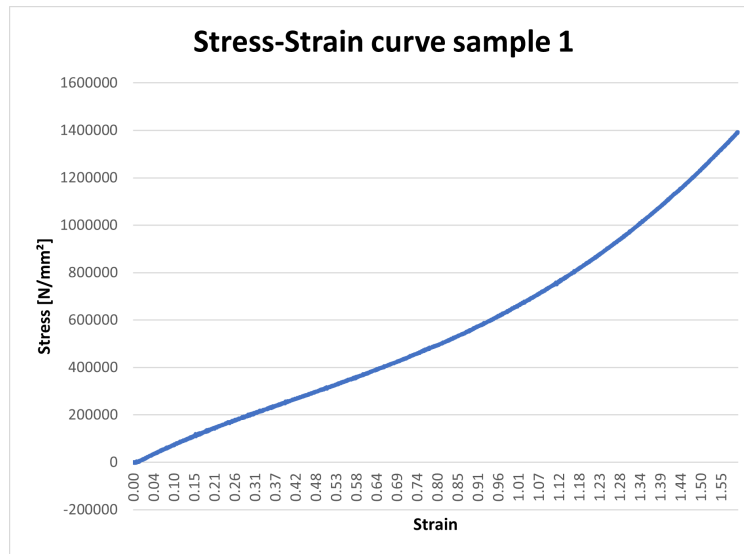


Figure 3: Sample 1 Stress strain curve

### 4.3 Indentation test

For the indentation tests, the cylindrical samples of the 10:1 PDMS mixture were used. For the indentation tests, different forms were used for indentation. One conical, three cylindrical and two spherical metallic indenters were used and force-distance curves were recorded. To calculate the Young's modulus the three formulas shown in table 2 were linearized and transformed. Table 2 shows the different geometries of the indenters and the resulting Young's moduli for the different indenters after the formulas were linearized and transformed. Figure 12 shows the distance-time curve for the indentation test while figure 4 displays the force-distance curve. Figure 5 displays the force-time curve. The linearized force against distance curve is shown in figure 15.

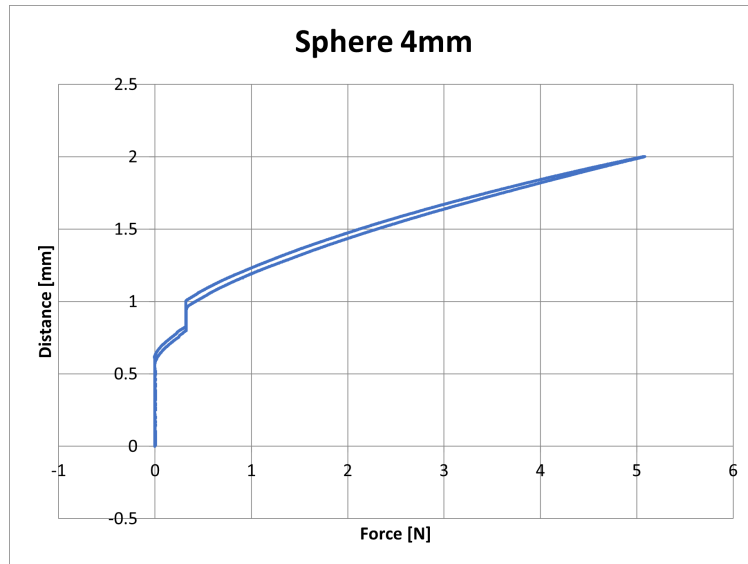


Figure 4: Force-distance curve for the indentation test of the 4mm diameter sphere

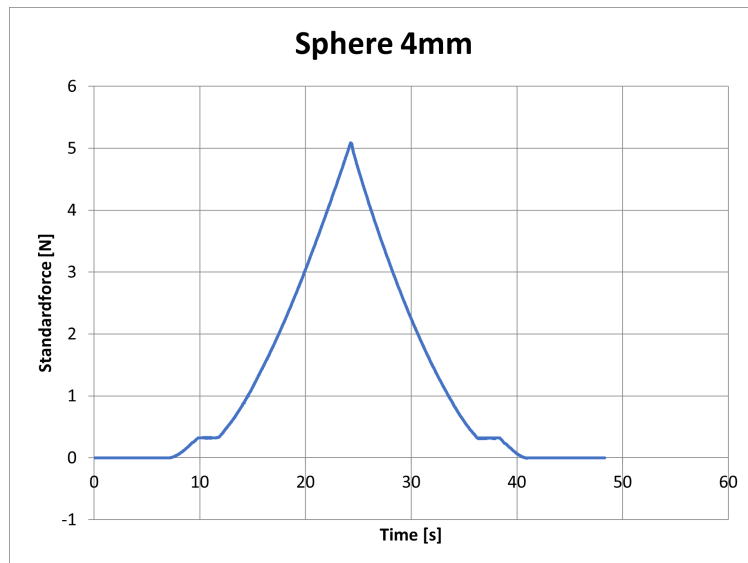


Figure 5: Force-time curve for the indentation test of the 4mm diameter sphere

| Indenter   | Indenter diameter/angle | Youngs modulus [Pa <sup>2</sup> ] |
|------------|-------------------------|-----------------------------------|
| Sphere 1   | 4mm                     | 1.494.917                         |
| Sphere 2   | 10mm                    | 1.771.689                         |
| Cylinder 1 | 10mm                    | 1.840.687                         |
| Cylinder 2 | 6mm                     | 1.739.237                         |
| Cylinder 3 | 4mm                     | 1.693.856                         |
| Conical    | 30°                     | 3.764.301                         |

Table 3: Different Young's moduli calculated by linearized formulas for the different indenters which were used in the experiment.

#### 4.4 Viscoelasticity tests

For the viscoelasticity tests, two materials were tested with different testing techniques. Memory foam which is known from bicycle saddles was chosen as a representative for the Kelvin-Voigt model and a creep test was performed. The distance-time and force-time curves are displayed in figures 6 and 16.

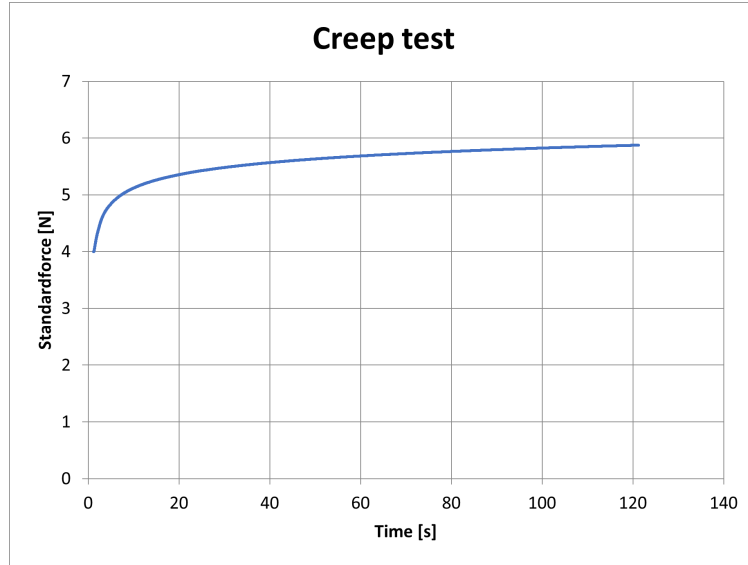


Figure 6: Distance-time curve for the creep test

The relaxation time  $\tau$  was read off figure 6 manually, as the time where 2/3 of the maximum distance that was indented, to be  $\tau=0.2s$ . The Young's modulus and the viscosity were calculated to be  $E=25.600 \text{ Pa}$  and  $\eta =5.120 \text{ N} * s/m^2$  by using formula 10 of the instruction manual.

The other material was Silly Putty, which is a silicone polymer and a representative of the Maxwell model. Here, a stress relaxation test was performed. The distance-time and force-time curves are displayed in figures 7 and 17.

The relaxation time  $\tau$  was read off figure 7, as the time where 1/3 of the maximum force was reached, to be  $\tau=0.14s$ . The Young's modulus and the viscosity were calculated to be  $E=375.000 \text{ Pa}^2$  and  $\eta =52.500 \text{ N} * s/m^2$  by using formula 9 of the instruction manual.

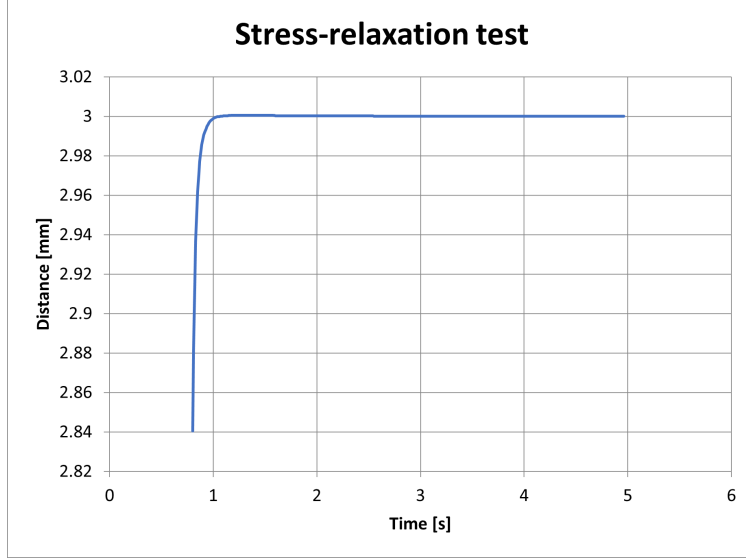


Figure 7: Distance-time curve for the stress-relaxation test

## 5 Discussion

### 5.1 Loading cell evaluation

The evaluation of the testing machine showed a slope value of  $9.8 \text{ N/m}^2$ . Since this experiment measured the gravitational acceleration, which has a theoretic value of  $9.81 \text{ N/m}^2$ , the deviation is only  $0.01 \text{ N/m}^2$  and the relative deviation  $0.1\%$ . This shows the accuracy of the testing machine. Possible deviations could still happen due to the hook which is used to hang up the weights or small deviations due to the graphical evaluation. However, overall the accuracy is very good.

### 5.2 Tensile test

The five tensile tests show typical elastomer stress-strain curves with a linear regime in the beginning and then 2 turning points in the function. Sample 4 shows an irregularity in figure 10 at the beginning which was caused by a bump against the table where the machine was localized. Table 1 displays the different samples and their characteristics as well as the ultimate tensile stress and the calculated Young's moduli. The ultimate tensile stress is higher for the samples of the mixtures 1:10 since a higher percentage of crosslinker is present in those samples. Since the ultimate tensile stress is an indication for material properties it should be the same for samples 1, 2 and 3. However this is not the case in the experiment. This could be caused by various factors. First, the bump against the table could have impacted the outcome of the measurement of sample 2. Furthermore, it was attempted to exclude air inclusions in the samples but not confirmed. Hence, the samples could have shown irregularities. Another possible influence factor is the breaking point of the sample. Since the theory only applies to the same breaking point for all samples and contrary to that in the practical all samples broke at different locations, this could also lead

to deviations. Another possible source of error is the geometry of the sample which is used in the calculations. Since the cross-section of the sample changes during the elongation and gets thinner, the geometry changes and by that makes the formulas inaccurate. The Young's modulus should also be the same for samples 1-3 and then deviate for samples 4 and 5. However, the same problems influence the outcome of this calculation. Still, samples 1-3 all show higher values than samples 4 and 5 which is according to the expected outcome.

### 5.3 Indentation test

For the different indenter geometries, the Young's modulus varies from around 1.4 MPa to 1.84 MPa while one outlier is located at 3.76 MPa. The distance-time and force-distance curves in figures 12 and 4 of the different indenters show that the testing procedure worked as expected. The bump which can be seen in the force-time and the force-distance curves in figures 4 and 5 could be caused by a small cavity between the sample and the sample table. After the force was big enough the cavity closed which then caused the bump. Another possible explanation for the results could be a loose screw in the experiment setup. The necessary force first increased slowly since the indentation in the beginning requires less force as displayed in figure 4. During the experiment, the force then increases more and more until reaching the maximum at maximum indentation depths. The origin of the highly deviating Young's modulus values for the conical indenter is unknown. One possible reason could be a small deviation of the indenter tip angle which can lead to high changes in the Young's modulus. However, deviations of the different measurements can also occur due to several other reasons. The material which was used could have been impacted by the other indentation measurements and by that cause deviation. Furthermore, some of the indenters as the 10mm sphere or cylinder were almost as big as the sample, which could have led to material compressions and by that changes of the outcome. However, most of the tests showed quite similar Young's moduli, which shows the legitimacy of the use of this method.

As it can be seen the indentation and tensile test results differ highly since the indentation test delivers almost double the values for the Young's modulus compared to the tensile test. However, these deviations are typical when comparing indentation and tensile testing and also occur for other materials [8]. Since the tensile and indentation test also included certain inaccuracies regarding breaking point, material consistency and testing procedure, this could also have caused the deviations. Furthermore the linearization step also simplified the calculations which also could lead to inaccuracies in the outcome. However the linearization step is an easy tool to approximate the Young's modulus without complicated analysis. The success of the linearization can also be seen in figure 15.

### 5.4 Viscoelasticity test

The viscoelasticity tests both showed the expected outcome and accorded to the theoretical values. The creep test for the memory foam showed the typical distance-time behavior which can be seen in figure 6. After applying a constant force the indentation first increases fast and then grows slower until it reaches a limit which is similar as a limited growth pattern. The stress relaxation test of the Silly Putty also showed the typical behavior. After indentation of the specific distance of 3 mm the necessary force to keep this indentation

depths decreased over time while the decrease was high in the beginning and then lower the longer the experiment was performed. Both tests use a perfect step of force for the creep test and distance for the stress-relaxation test in theory, which cannot be realized in practice. However the limits are reached fast enough as shown in figures 6 and 7 which is the reason why the tests also work in practice and can be used to calculate the instantaneous modulus, the infinite elastic modulus and the viscosity of certain materials.

## 6 Conclusion

In general, the different experiments showed the expected outcomes, while small deviations occurred due to inaccuracies or time reasons. With more time most of the inaccuracies could have been avoided but this would have exceeded the time frame of the practical work. The relevance of material property measurements will even rise since the research on some properties as the cell stiffness of cancer cells is still in research. Furthermore, new and faster measurements will be necessary which can measure in new environments like the human body.

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# Appendix

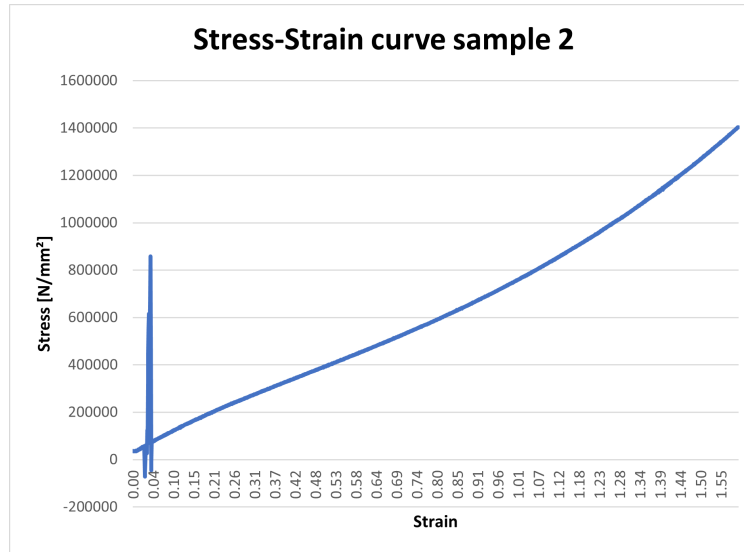


Figure 8: Sample 2 Stress strain curve

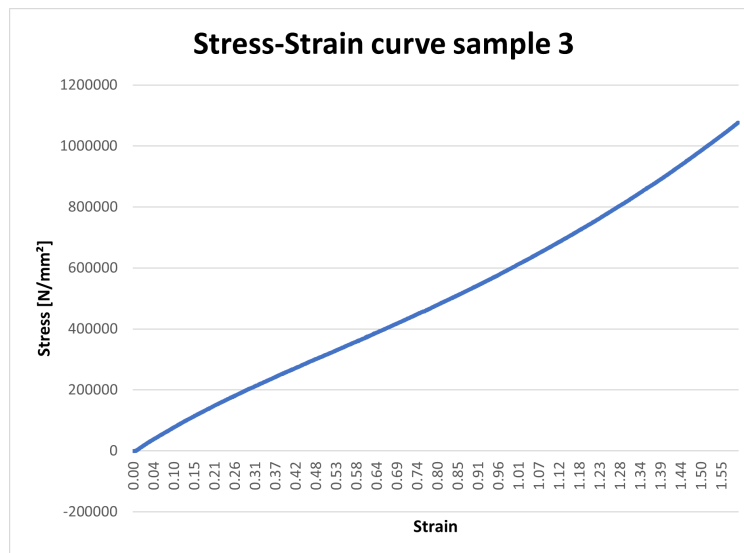


Figure 9: Sample 3 Stress strain curve

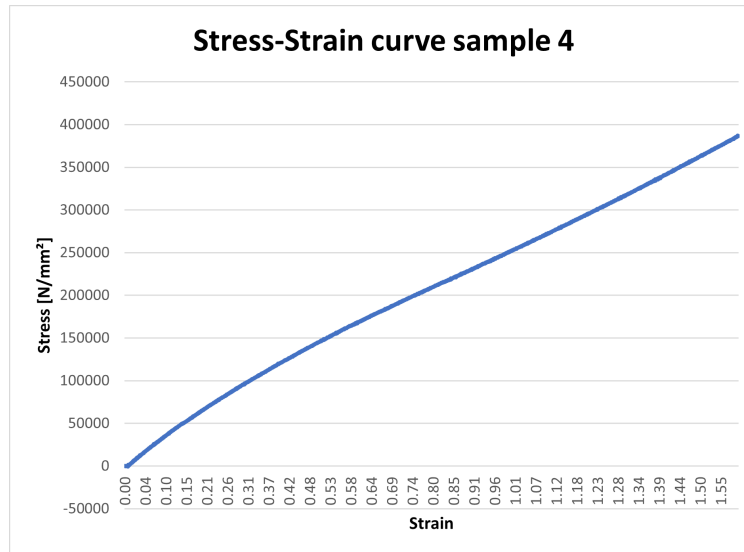


Figure 10: Sample 4 Stress strain curve

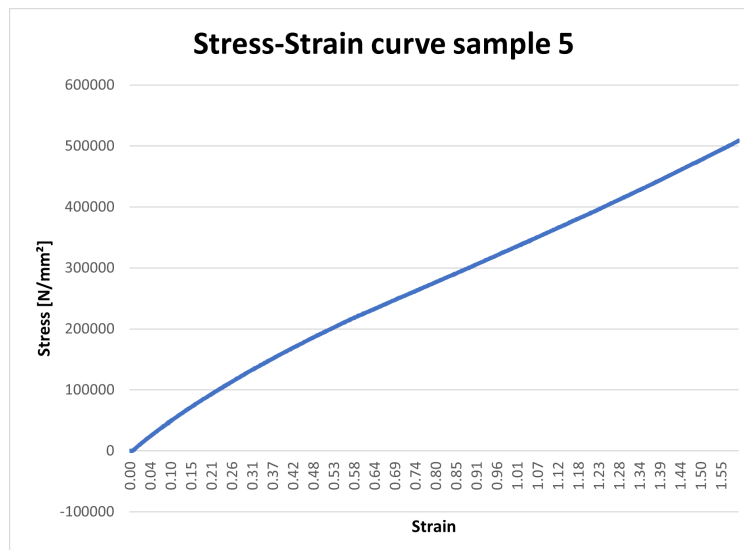


Figure 11: Sample 5 Stress strain curve

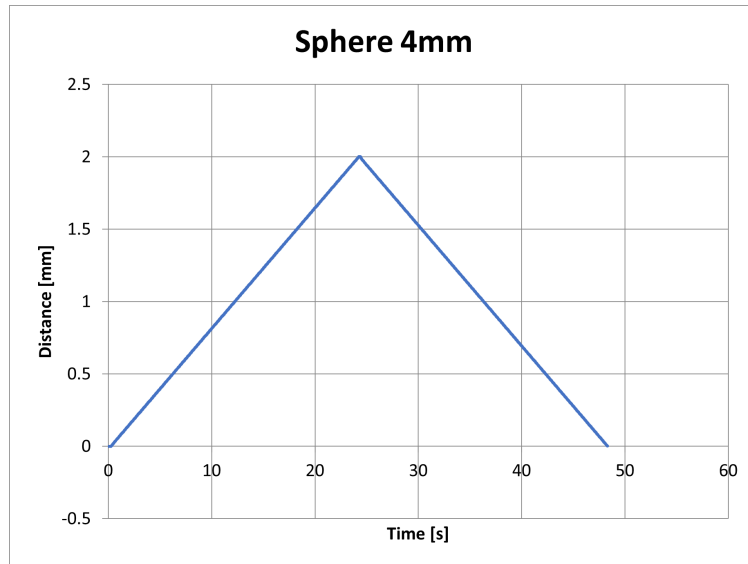


Figure 12: Distance-time curve for the indentation test of the 4mm diameter sphere

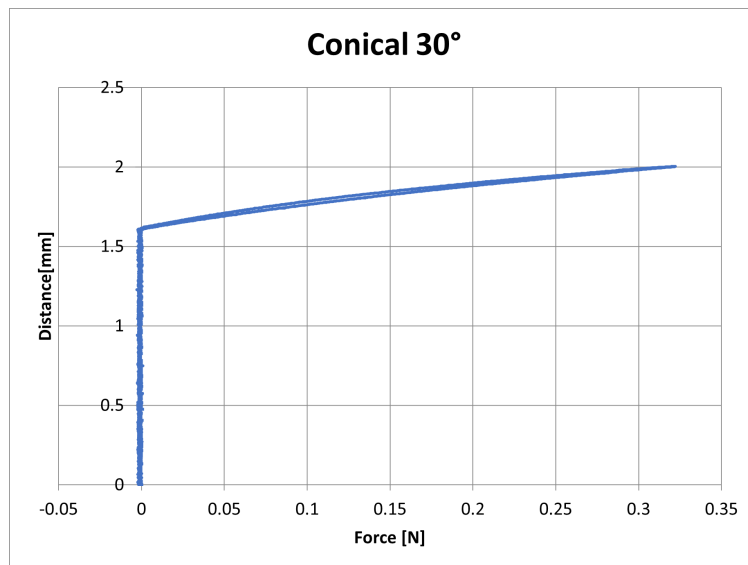


Figure 13: Force-distance curve for the indentation test of the conical indenter

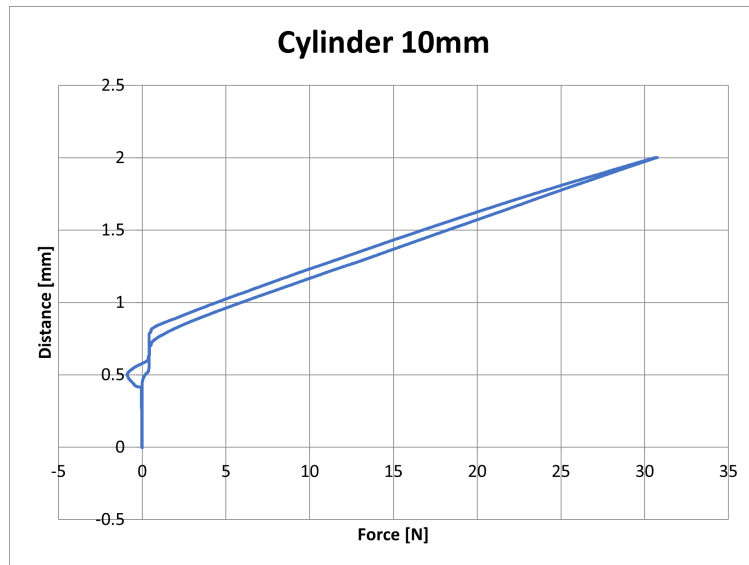


Figure 14: Force-distance curve for the indentation test of the cylinder

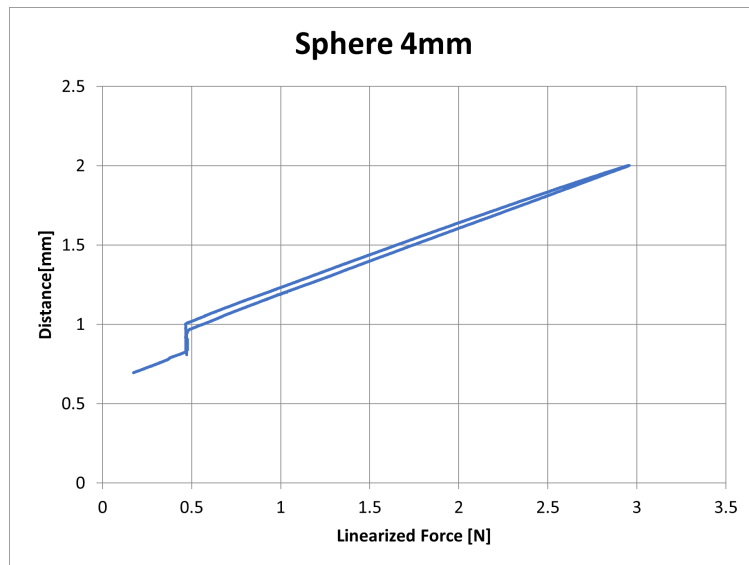


Figure 15: Force-distance curve for the indentation test of the sphere with the linearized force

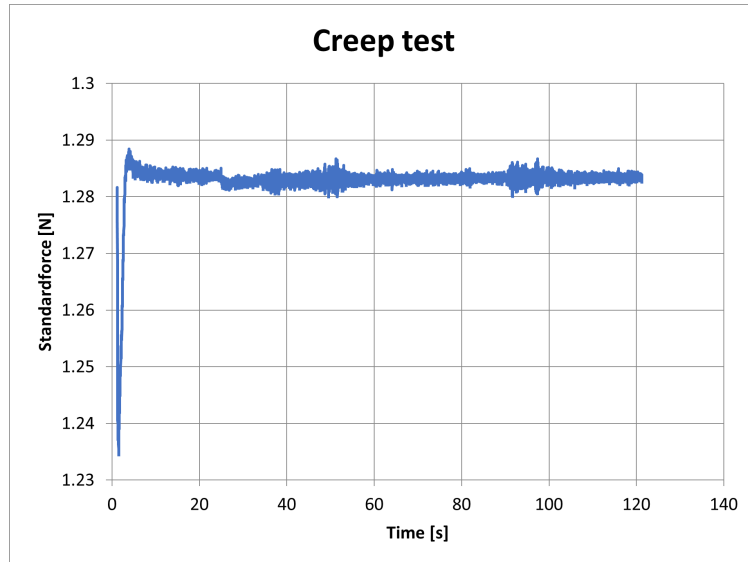


Figure 16: Force-time curve for the creep test

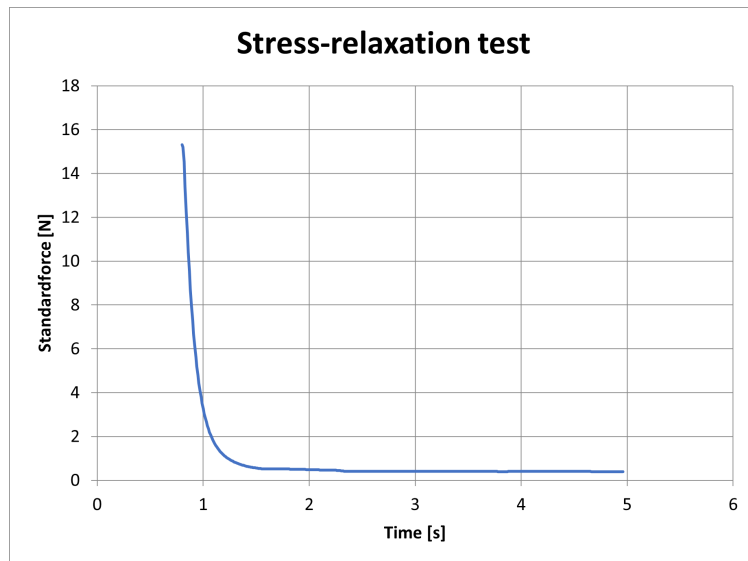


Figure 17: Force-time curve for the stress-relaxation test