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# NOVEL HIGH-RESOLUTION INTERFEROMETRIC MATERIALS TESTING DEVICE FOR THE DETERMINATION OF THE VISCOELASTIC BEHAVIOUR OF HIGH-TECH PLASTICS

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**Abstract** – The use of high-tech plastics in the field of mechanical engineering is increasing dramatically. The time-dependent change of strain ε(t) during constant load (viscoelasticity, creeping) is a fundamental property of plastics and exact knowledge of these properties is required for the design of plastic parts. A novel high-resolution materials testing device is presented in this paper. It offers reaction-free interferometric measurement of the viscoelastic behaviour of plastics at a constant bending load. The resolution of the deflection is 1 nm.

Therefore, unlike the three point bending test described in the ISO 899-2:2003 standard [1], the device is suitable for the measurement of viscoelasticity ε(t) at very small strain values ($\varepsilon_{max} < 0{,}1$ %) and after a very short loading time (t < 1 d).

A detailed description of the device and the parameters is shown as well as measurements of the time-dependent strain ε(t) of a high-tech plastic. Very good reproducibility of the measurements was achieved, which makes the device very suitable for measuring viscoelastic behaviour.

**Keywords:** plastic, viscoelasticity, creep

## 1. INTRODUCTION

The state of the art for the determination of the viscoelastic behaviour of plastics is described in the ISO 899-2:2003 standard. Testing set-ups designed according to this standard essentially consist of two supports carrying the specimen and one indenter to introduce a constant load symmetrically on the specimen. Often, the time-dependent bending of the specimen is measured by devices that cause an additional measurement force on the specimen and thus a measurement error. These systems are normally set up for large forces, deflections and strains. Hence, the resolution for the determination of viscoelastic behaviour at small loads and strains is very poor. Furthermore, stick-slip can appear between the supports and the specimen, which also leads to an error. Generally, devices described in the standard require plenty of room and are quite expensive.

The materials testing device presented here, is designed for the measurement of small rectangular plastic specimens of the size 35 x 25 x 3 mm³. One side of the specimen is clamped in the fixture. The load is applied to the free side of the specimen via a cantilever. A microchemical actuator is used for the automatic alternation of the load. The spontaneous and the time-dependent deflections of the specimen are measured with a metrologically traceable MJI-series miniature retroreflector interferometer made by SIOS Meßtechnik GmbH in Ilmenau, Germany. Temperature and relative humidity are kept constant within $\Delta T < \pm 0.05$ K and $\Delta\varphi < \pm 0.05$%, respectively, during the entire measurement. The complete size of the device and its hood is 500 x 350 x 300 mm³.

## 2. MATERIALS TESTING DEVICE

### *2.1. Principle of measurement*

The plastic specimen (red) is clamped in a fixture on one side (A). On the other side (B) a cantilever is clamped to the specimen in a similar way. The cantilever is used to apply the load as shown in Fig. 1.

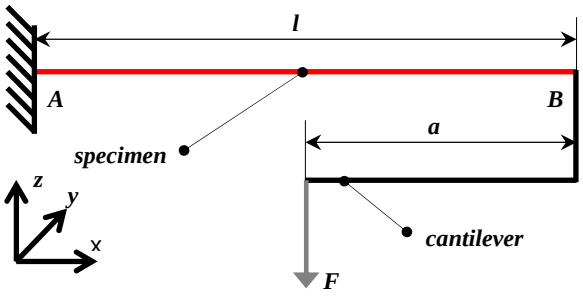


Fig. 1. Principle of measurement

The length a is chosen to be a = l/2, which allows an s-shaped deformation to be induced while the load is applied. Point B shifts parallel to the z-axis. The gradient of the bending line at location B equals f´ = 0. Thus, it is easy to measure the displacement f of point B with a retroreflector interferometer:

$$f = -\frac{F\ l^3}{E_0\ w\ h^3} \tag{1}$$

Therefore, the maximum strain of the specimen equals:

$$\varepsilon_{max} = -\frac{3\ F\ l}{E_0\ w\ h^2} \tag{2}$$

Whereas w is the width and h the height of the specimen.

### *2.2. Measurement set-up*

The set-up consists of the following components (see Fig. 2): fixture (1), cantilever (2), weight with a connecting rod (3), load changer (4), interferometer with retroreflector and 90° tilting mirror (5), specimen (6). Additionally, the set-up is covered with a hood.

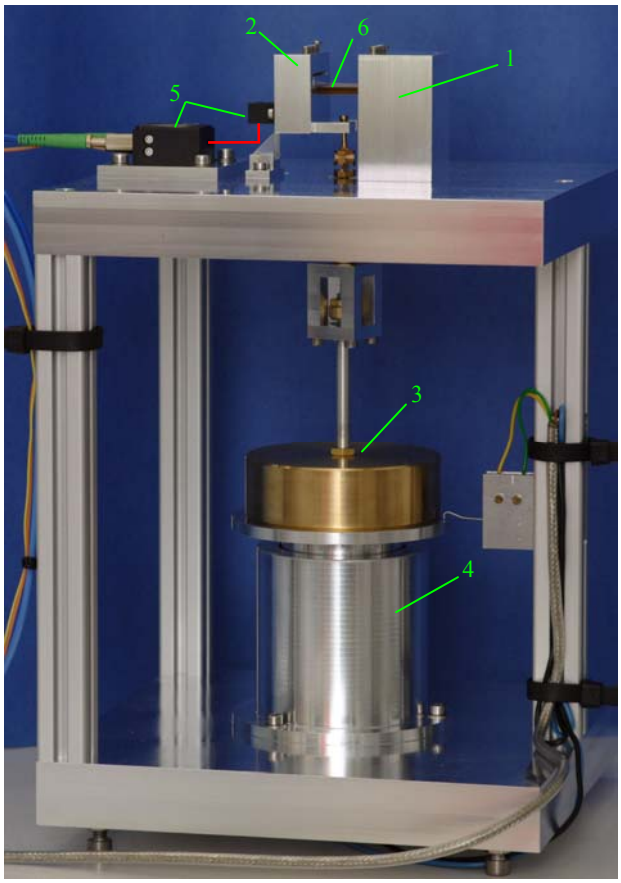


Fig. 2. Materials testing device

The fixture consists of a parallel-guided clamp based on the principle of a bench vice. The clamping force is generated by three screws, which are tightened with a defined torque $M_S$. The cantilever is clamped to the specimen in the same way. The weight (m = 1kg) is deposited on the load changer. It is then applied to the cantilever and consequently to the specimen by lowering the level of the load changer. The generated bending of the specimen is measured with the retroreflector interferometer.

### *2.3. Controlling temperature and relative humidity*

A Sensirion SHT 11 sensor is used to control the temperature and relative humidity. It offers a temperature resolution of 0.01 °C and a relative humidity resolution of 0.03%. The set-up is also covered by a hood and to generate a stable relative humidity level, a jar containing silica gel is placed under the hood as well.

### *2.4. Load changer*

The mechanical principle of a cylinder stroke is employed for the load changer, which is driven by an electrochemical actuator (ECA) from the company Silberkraft.

The actuator extends when connected to a voltage, and discharging makes it contract again. A very smooth motion of the cylinder can be achieved, yielding a shock-free loading of the weight on the specimen. A speed of up to 0.1 mm/s is possible.

### *2.4. Miniature interferometer*

In the miniature retroreflector interferometer a He-Ne laser with $\lambda = 633$ nm and a frequency stability of $3 \cdot 10^{-7}$ is used as light source. The maximum retroreflector translation rate is 600 mm/s. The measurement range is 150 mm with a resolution of 1 nm.

### *2.5. Plastic specimens*

The plastic specimens used for the investigations are of the dimensions l x w x h = 35 x 25 x 3 mm³ (see Fig. 3). The specimens are clamped at a position 8 mm from each edge, resulting in a free bending length of $l_f$=19 mm.

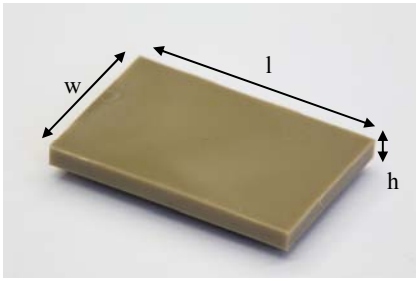


Fig. 3. Plastics specimens

From equation (1) a deflection of f = 25 µm results from a load of m = 1 kg, assuming a specimen flexural modulus of elasticity $E_0$ = 4000 N/mm² (average value of the high-tech plastics investigated). In this case the maximum strain is $\varepsilon_{max} = 6.2 \cdot 10^{-4}$ (cf. equation 2). With these values the breadth of applications for this device becomes apparent because. The viscoelastic behaviour of the plastic specimen can now be measured using very small deflection and strain values.

### *2.6 Suitability of the device for $E_0$ - determination*

The suitability of the presented device also for the determination of the flexural modulus of elasticity $E_0$ should be discussed here. By knowing the dimensions of the specimen, the deposited weight m and the measured value of

deflection f, the flexural modulus of elasticity $E_0$ can be determined. Equation (1) can be used to express $E_0$:

$$E_0 = -\frac{F\ l_f^{\,3}}{f\ w\ h^3} \tag{3}$$

The dimensions $l_f$, w, and h, the measured deflection f, the weight m and thus the force F are afflicted with uncertainties. These uncertainties lead to a combined uncertainty of the $E_0$-determination as follows:

$$u_c(E_0) = E_0 \cdot \sqrt{\left(\frac{u_h}{E_0}\right)^2 + \left(\frac{u_w}{E_0}\right)^2 + \left(\frac{u_{l_f}}{E_0}\right)^2 + \left(\frac{u_F}{E_0}\right)^2 + \left(\frac{u_f}{E_0}\right)^2} \tag{4}$$

The values $u_x$ are the uncertainty contributions to the combined uncertainty $u_c(E_0)$. By normalizing with $E_0$ the relative uncertainty contributions $u_x/E_0$ were calculated based on equation (3):

$$\frac{u_h}{E_0} = 3\frac{u(h)}{h}; \qquad \frac{u_w}{E_0} = \frac{u(w)}{w}; \qquad \frac{u_{lf}}{E_0} = -3\frac{u(l_f)}{l_f};$$

$$\frac{u_F}{E_0} = -\frac{u(F)}{F}; \qquad \frac{u_f}{E_0} = \frac{u(f)}{f};$$

| **h in mm** | **w in mm** | **$l_f$ in mm** | **F in N** | **f in mm** |
|---|---|---|---|---|
| 3 | 25 | 19 | 9.81 | 0.025 |
| **u(h) in mm** | **u(w)in mm** | **$u(l_f)$ in mm** | **u(F) in N** | **u(f) in mm** |
| 0.1 | 0.1 | 0.05 | 1e-5 | 1e-5 |
| **$u_h/E_0$** | **$u_w/E_0$** | **$u_{lf}/E_0$** | **$u_F/E_0$** | **$u_f/E_0$** |
| 1e-1 | 4e-3 | -8e-3 | -1e-5 | 4e-4 |

Tab. 1 Values for the uncertainty evaluation

Table (1) shows the values that were used for the uncertainty calculation, assuming a specimen flexural modulus of elasticity $E_0 = 4000$ N/mm². The values u(h) and u(b) are caused by the fabrication tolerances of the inject-moulded specimen. The uncertainty $u(l_f)$ results from the installation of the specimen to the measurement setup. The uncertainty of the load u(F) follows from the uncertainty of the mass determination using a precision balance. Furthermore the uncertainty of the measurement of the deflection u(f) is assumed as 10 nm. Based on these values the combined uncertainty of the determination of the flexural modulus of elasticity $E_0$ was calculated:

$$u(E_0 = 4000\,N/mm^2) \approx 400\,N/mm^2$$

The main contribution to the uncertainty of $E_0$ is caused by the height h of the specimen. The influences of u(F) and u(f) are negligible. With that kind of specimen $E_0$ can be determined with an uncertainty of about 10%.

Furthermore the influence of the tightening torque $M_S$ affects the determination of $E_0$. The main reason for this effect is additional stress which is induced to the specimen by the clamping forces (see Fig.4). Clamping forces cause a higher stiffness of the specimen and thus a higher $E_0$ will be determined.

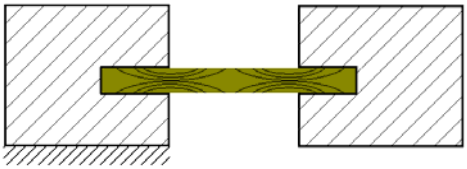

Fig. 4. Stress in the specimen

These considerations show, that the specimen and the device are not well suited for the determination of the flexural modulus of elasticity $E_0$.

### *2.7 Determination of the viscoelastic behaviour*

In the following the viscoelastic behaviour is discussed based on the time-dependence of the strain ε(t) after changing the load. For a better comparability of different investigated plastic specimen it is useful to use relative values. The relative strain $\varepsilon_{rel}(t)$ can be calculated from the measured deflections of the specimen:

$$\varepsilon_{rel}(t) = \frac{\varepsilon(t \geq 0)}{\varepsilon(t=0)} - 1 = \frac{f(t \geq 0)}{f(t=0)} - 1 \tag{5}$$

Whereas t = 0 is the time when the load change is completed. The load change is considered to be completed when the load is applied completely after loading or taken off completely after unloading. From equation (5) it is obvious that the determination of $\varepsilon_{rel}(t)$ is, unlike the determination of $E_0$, independent from the dimensions (l, w, h) of the specimen and of the flexural modulus of elasticity $E_0$.

## 3. RESULTS

### *3.1. Stability of temperature and relative humidity*

Initially, the set-up was not covered during experimentation. Over a period of 7 h, the variations of temperature und relative humidity in the laboratory averaged $\Delta T = \pm 0.3$ K and $\Delta\varphi = \pm 3\%$. In particular, the humidity variations could significantly affect the measurement result ε(t), especially considering the partly high moisture absorption of some plastics.

The set-up was then covered by a hood in order to improve its temperature and humidity stability. Additionally, a jar containing silica gel was placed under the hood. Using these steps the variations of temperature and humidity were reduced to $\Delta T = \pm 0.05$ K and $\Delta\varphi = \pm 0.05\%$, respectively (see Fig. 5). ISO 899-2:2003 only requires a temperature variation of $\Delta T < \pm 2$ K.

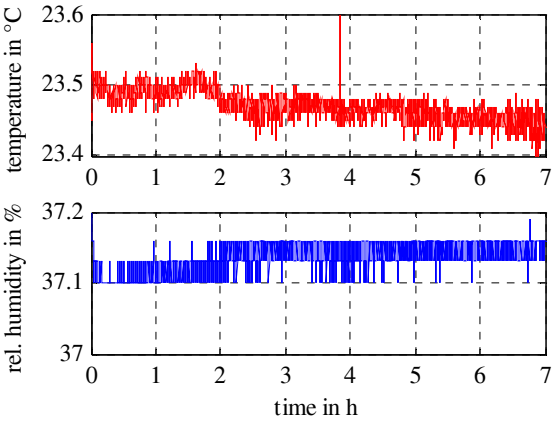


Fig. 5. Stability of temperature and relative humidity

### *3.2. Load cycle*

A specimen deflection measurement ($E_0$ = 2800 N/mm²) during one load cycle is shown in Fig. 6. Here, a smooth and absolutely shock-free alternation of load is obvious and no overshoot is identifiable.

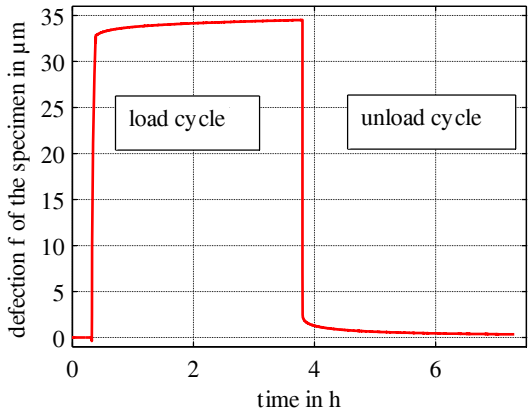


Fig. 6. Smooth alternation of load

Thus, the electrochemical actuator functionality conforms to the ISO 899-2:2003 standard and is suitable as a drive for the load changer. The requirement of a constant load is also achieved because a weight is used to generate the load.

### *3.3. Reproducibility of the measurement of $\varepsilon_{rel}(t)$*

The measurements of $\varepsilon_{rel}(t)$ of one specimen were repeated several times to check the reproducibility. After every measurement the specimen was removed and reclamped. Defined tightening torques $M_S$ where generated by a torque wrench ($M_S$ = 0.75 N.m). The time between the measurements was chosen as 48 h.

The relative strains are shown in Fig. 7, both for the load cycle and the unload cycle. The sign of $\varepsilon_{rel}(t)$ during unloading has been reversed.

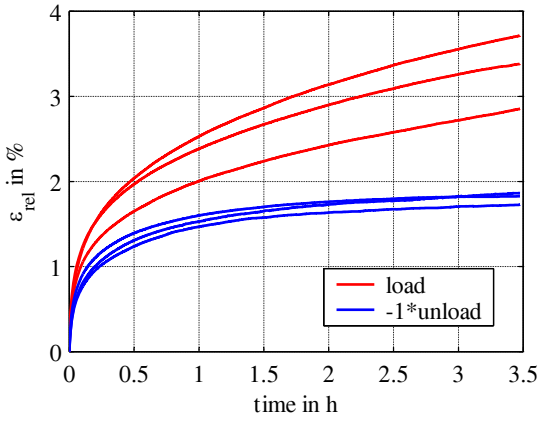


Fig. 7. Reproducibility of the $\varepsilon_{rel}(t)$ curves, $M_S$ = 0.75 N.m

The results show very good reproducibility for the $\varepsilon_{rel}(t)$ unload curves (see Fig. 7, blue). In contrast, the reproducibility of the $\varepsilon_{rel}(t)$ load curves is a little lower (see Fig. 7, red). Two main reasons were detected for this effect:

On the one hand, the poor reproducibility of the $\varepsilon_{rel}(t)$ load curves is caused by the torque affecting the fixture while under load ($M = F \cdot l/2$). Unlike the load cycle, the unload cycle is not affected by any additional torque.

On the other hand, it was determined, that in contrast to the unloading speed the loading speed and thus the loading time shows a lower reproducibility. The length of the time interval for loading or unloading affects the $\varepsilon_{rel}(t)$ curves, because the load is already applied partly in this time interval. It is obvious, that the viscoelastic effects occur if any load is applied. In contrast to that, the starting point $t = 0$ of the $\varepsilon_{rel}(t)$ curves is defined as the point of time where the load change is completed. That implies that different loading or unloading speeds effect different $\varepsilon_{rel}(t)$ curves. Hence, for a better reproducibility of the $\varepsilon_{rel}(t)$ curves the loading speed should be kept constant.

Additionally to these two influences the reproducibility of both, the load and the unload curves, is affected by the uncertainty of finding the starting point ($t = 0$) where the load change is completed. This uncertainty is due to the low measuring frequency (0.25 Hz) and the non ideal step function of loading or unloading (loading and unloading time $t_{load\ change} > 0$).

Furthermore, the influence of the tightening torque $M_S$ of the clamping screws on the $\varepsilon_{rel}(t)$ curves was investigated by measuring the $\varepsilon_{rel}(t)$ curves at different tightening torques. A slight effect of the tightening torque $M_S$ on the $\varepsilon_{rel}(t)$ curves was determined. The main reason for this effect is additional stress which is induced to the specimen by the clamping forces (see Fig.4). These stresses are partly relieved during the load cycles and thus affect the $\varepsilon_{rel}(t)$ curves slightly. Unlike for the measurement of $E_0$, the effect of $M_S$ is not crucial for the measurement of $\varepsilon_{rel}(t)$.

## OUTLOOK

Eventually, an improvement of the reproducibility and a reduction of the influence of the tightening torque $M_s$ can be achieved by the modifying the specimen geometry. The specimen should be thinner (softer) in the bending zone than in the clamping zone (Fig. 8).

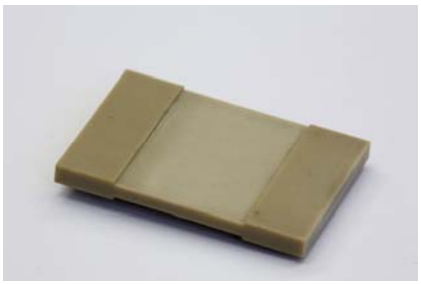

Fig. 8. New specimen geometry

A thinner design of the specimen in the bending zone means that the stress caused by the clamping does not expand into the area of bending (see Fig. 9). Future measurements will be taken to demonstrate the benefit and to investigate the influence of the clamping forces.

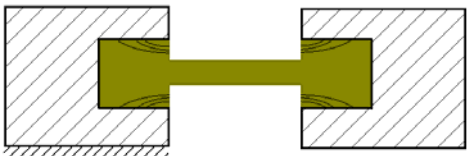

Fig. 9. Stress in the specimen (new geometry)

## 5. CONCLUSIONS

A materials testing device was developed for the non destructive identification of the viscoelastic properties $\varepsilon_{rel}(t)$ of plastics at small strains. Due to the small size of the device, temperature and relative humidity were kept constant during the measurement ($\Delta T < \pm 0.05$ K; $\Delta\varphi < \pm 0.05\%$). To investigate the creep caused by temperature changes, the set-up can be operated in a climate chamber. Due to the measurement principle (bending load) and the use of a miniature interferometer, the relative strain $\varepsilon_{rel}(t)$ of the plastic can be determined with a very high resolution.

It was shown that the device is very well-suited for measuring $\varepsilon_{rel}(t)$ at small absolute strains ($\varepsilon_{max} < 0.1\%$). Thus, the device enables a non destructive determination of the viscoelastic behaviour of high-tech plastics. Very good reproducibility was achieved for the unload curves. The measurement results of a high-tech plastic were presented and the influence of the tightening torque $M_S$ was discussed. Suggestions for improvement of reproducibility were given as well as for reducing the influence of the tightening torque. These improvements will be investigated in the future.

## ACKNOWLEDGMENTS

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