

A tool for rapid quenching of elongated polymer melts

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Abstract In this article, we present a device for rapid quenching of elongated polymer melts. The tool is an accessory to the uniaxial elongational rheometer RME of Meissner and Hostettler. It is intended to be used for microscopic and other investigations of stretched polymers. The device allows us to solidify a polymer melt by pouring liquid nitrogen on it and to cut it at the nearly same instant of time. Then the sample can be easily removed from the stretching apparatus. Solving the heat diffusion equation for a polymer melt, which is cooled by liquid nitrogen, we theoretically estimate the quenching time of this method. To demonstrate that this quenching procedure indeed rapidly cools a polymer melt, the stress birefringence of elongated and subsequently quenched polystyrene melts is measured and the stress-optical coefficient C is determined. The experimental value of the stress-optical coefficient is $|C| = 4.65 \times 10^{-9} \text{ Pa}^{-1}$, which agrees well with the data in literature. Using this tool for elongation experiments with the RME, polymer melts can be solidified in between approximately 0.2 and 2.0 s, depending on the thickness of the sample.

Keywords Extensional rheology · Rapid quenching · Polystyrene · Flow birefringence

Introduction

The elongation of polymer melts and other highly viscoelastic liquids has been the focus of experimental studies for many decades (see, e.g., McKinley and Sridhar 2002; Hachmann and Meissner 2003). Deformation processes on different length scales stimulate many fundamentally oriented investigations. In addition, the transient elongational viscosity and strain-hardening as well as the elasticity and the stability of the stretched melt are highly relevant parameters in polymer processing such as film blowing, blow molding, and calendering. Several techniques were developed to stretch molten polymers and to measure the elongational viscosity. The uniaxial elongational rheometer RME applies the technique of clamps with rotating metal belts and allows one to extend viscoelastic liquids to large stretch ratios (Meissner and Hostettler 1994). A large number of other techniques are commonly applied in rheological laboratories, e.g., the extensional rheometers developed by Münstedt (1979) and Muller and Froelich (1985), the filament stretching rheometer (McKinley and Sridhar 2002; Bach et al. 2003), and rheometers based on the windup technique (Maia et al. 1999; Sentmanat 2004).

Elongated polymeric melts have an anisotropic molecular and microscopic structure. Examples for phenomena that appear in elongational flows are the stress-induced birefringence of stretched homopolymer melts (Matsumoto and Bogue 1977; Venerus et al. 1999; Kröger 2004), the extension of drops in two-phase polymer blends (Delaby et al. 1994; Gramespacher and Meissner 1997; Heindl et al. 2004; Handge and Pötschke 2004, 2005, 2005; Oosterlink et al. 2005), and the deformation of microdomains in melts

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of microphase-separated block copolymers (Lee et al. 2006). In addition, isolated carbon nanotubes in the matrix of a polymer nanocomposite can be oriented by elongational flows (Fornes et al. 2006; Handge and Pötschke 2006). To achieve a profound understanding of these deformation processes, a large interest exists to observe and to quantify them. Presently, there are only a few in situ methods that are applicable for such experiments, e.g., scattering. Therefore, quenching the extended melt and subsequent transmission electron or optical light microscopy investigations is a popular method to get information about the microstructure (Delaby et al. 1994; Heindl et al. 2004; Handge and Pötschke 2004, 2005). Because this method requires solidified objects, a strong demand exists for a fast quenching technique of extended polymeric materials.

In this work, we present a device to quench elongated polymer melts rapidly. First, we describe the mechanism of the device that was developed for elongational experiments with the RME. Then we theoretically estimate the quenching time that can be achieved with our tool. Finally, we show that quenching with this tool indeed leads to a very short quenching time by performing elongational experiments with polystyrene melts and measuring the birefringence of the solidified sample.

Description of the quenching device

The construction and the mode of operation of the uniaxial elongational rheometer RME is described in detail by Meissner and Hostettler (1994). Figure 1 shows a schematic diagram of the extension device of the RME. The polymer sample is clamped between two metal belts that rotate so that a constant Hencky strain rate is applied to the sample. A leaf spring measures

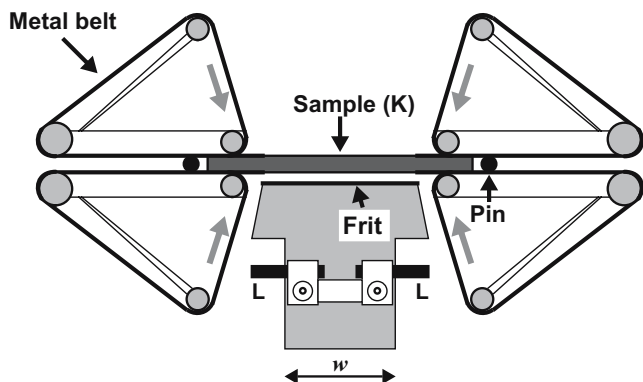


Fig. 1 Scheme of the stretching device of the RME; see Meissner and Hostettler (1994) for details. The metal belts rotate in the direction of the *arrows*. Dimensions are not to scale

the force that is caused by the deformed melt, and the transient elongational viscosity μ can be determined. Because the sample is clamped between the metal belts, a feasible quenching technique for the rapid cooling and the removal of the melt is needed.

A possible choice for a medium to cool a deformed polymeric melt is liquid nitrogen. However, it is necessary to pour liquid nitrogen on the sample in a controlled way because, otherwise, parts of the rheometer could be damaged. In addition, a device is needed for cutting the elongated sample, and, at last, the cut-off has to be removed from the RME to avoid remelting of the sample. Obviously, these steps need to be performed as quickly as possible so that the effect of relaxation processes during the quenching procedure is minimized.

In this study, we developed the tool that is depicted in Fig. 2 for rapidly quenching polymer melts. The device is an accessory to the RME. It consists of an open stainless steel container (A) (see Fig. 3a), which is attached to a grip (B). Two sharp edges (C) are fixed by screws to the side walls of the container and can slightly rotate to adjust their position for cutting. Before the experiment, the container is fully dipped into liquid nitrogen and is filled with the cooling liquid (Fig. 3b). The grip is attached via a u-shaped bar with the container and hence remains in air during the filling procedure. Initially, the container is fixed in position I by a hook (D). The hook is released if the bar (E) is pressed. Then the container is tilted such that the liquid nitrogen runs out from the opening (Fig. 3c). The tilting of the container is caused by a spring (F), which exerts a force on the container and is stopped by a thin horizontal plate (G) of phosphorus bronze and with a thickness of approximately 0.3 mm. Two steel bars (H) that facilitate the insertion of the tool are connected to the tool (see Fig. 4). The distance between these two bars equals the width w of the middle part of the table of the RME (Fig. 1).

Fig. 2 Photograph of the quenching device



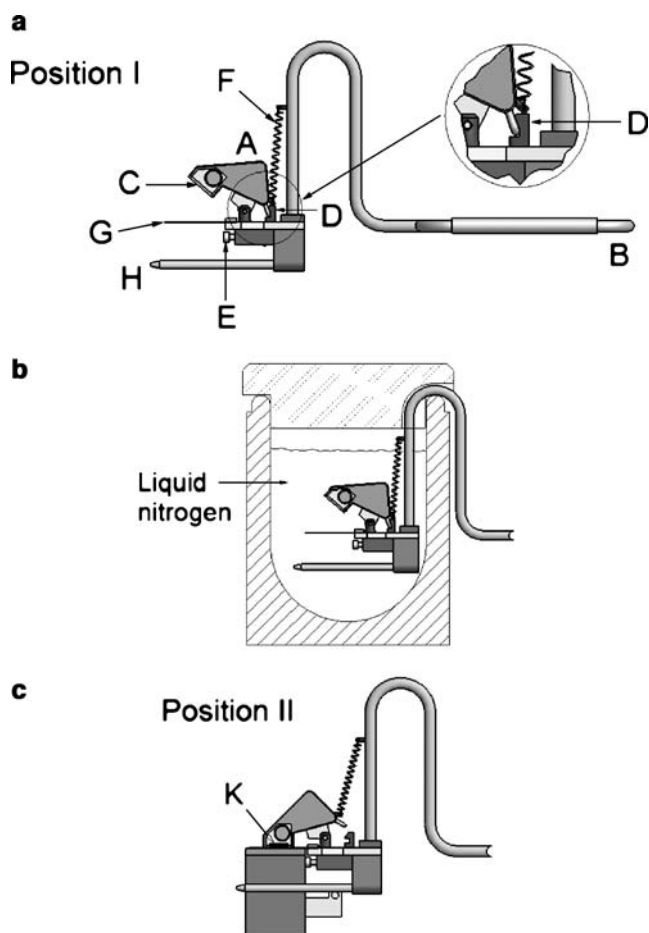


Fig. 3 A tool for quenching stretched polymeric materials. **a** The container (A) is initially put in position I where it is fixed by a hook (D). **b** Before the experiment, the container is filled with liquid nitrogen. **c** If the bar (E) releases the hook (D), the spring (F) tilts the container and the liquid nitrogen runs out. Then the container cuts the stretched polymer melt (K) with its sharp edges (C)

To cool an elongated polymer sample (K), the chamber of the RME is opened and the tool is inserted into the chamber. Two horizontal bars (L) are mounted at the table of the RME on an appropriate height (Figs. 1 and 4). The steel bars (H) of the quenching tool are placed on the horizontal bars (L) and adjusted so that the tool can be moved toward the table. The positions of these bars are fixed such that the bronze plate is placed only marginally above the frit of the RME. Optimally, the tool is inserted with a very slight nonzero angle between the bronze plate and the horizontal frit of the RME. Then the tool is quickly pushed toward the table. The thin bronze plate slides on the frit of the RME under the sample. When the release bar (E) touches the table and is further pushed, the hook (D) opens and the container tilts into position II (see Fig. 3c). At the same instant of time, the liquid nitrogen runs out on the stretched polymer melt and quenches

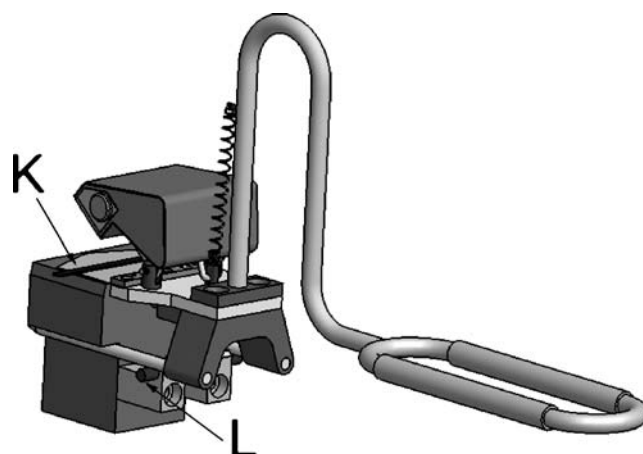


Fig. 4 Scheme of the quenching device and the table of the RME with a bar (L), which facilitates the insertion of the tool

it. The bronze plate suppresses the flow of hot gaseous nitrogen through the frit and avoids that liquid nitrogen of the container can flow in an uncontrolled way on the table of the RME. When the container opening touches the bronze plate, the sample is squeezed between the plate and the sharp edges. Because the polymer melt is soft, the sample is cut at these positions. Then the tool, still keeping the quenched sample inside the container, can be quickly removed from the RME. By putting the container back into position I, the solidified sample can be taken. The whole quenching procedure that starts with opening the chamber of the RME and ends with removing the tool from the chamber can be performed during approximately 6 s. In our experiments, we opened the chamber of the RME approximately 4 s before the maximum Hencky strain ϵ_{\max} was attained. Immediately after the extension of the sample was terminated, the tool was pushed toward the table of the RME and the container was tilted. After roughly 2 s, the tool and the sample were taken off the RME.

Analysis of the cooling process

To estimate the time for cooling a polymer melt, we theoretically consider the quenching process. Because quenching of a polymeric material with liquid nitrogen results in a complex nonstationary and spatially nonuniform temperature distribution in an open multiphase system, we present here a rough approximation of the real situation. In spite of its simplicity, our approach leads to an estimation of the relevant timescale.

Before the experiment, the tool is thermally equilibrated in liquid nitrogen. Consequently, the temperature of the cooling liquid and of the bronze plate of the tool is approximately $T_0 = -196$ °C. The temperature of the sample equals the measurement temperature

T_e , and we denote the solidification temperature (glass transition or melting temperature) of the sample by T_s . To quench the polymer melt, the tool is inserted into the RME. When the container is tilted, the liquid nitrogen is poured on the sample and quickly solidifies the polymer melt. For simplicity, we assume that the temperature of the upper and lower surface of the sample is constant during quenching and equal to $-196\text{ }^\circ\text{C}$. This approximation is valid if quenching is faster than boiling of liquid nitrogen. The temperature $T(\vec{r}, t)$ in the polymer melt as a function of its spatial coordinates $\vec{r} = (x, y, z)$ and time t follows from the heat diffusion equation:

$$\frac{\partial T}{\partial t} = \frac{k}{\rho c_p} \Delta T \tag{1}$$

where k denotes the thermal conductivity, ρ the density, c_p the specific heat at constant pressure of the sample, and $\Delta = \partial^2/\partial x^2 + \partial^2/\partial y^2 + \partial^2/\partial z^2$ the Laplace operator in three dimensions. We assume that the x , y , and z axes are parallel to the directions of thickness, width, and length measurements of the sample (see Fig. 5). Because the liquid nitrogen is poured on the whole sample, we also assume that the temperature is a function of x and t only. The initial condition for Eq. 1 is given by $T(x, t = 0) = T_e$ for $0 \leq x \leq d$, where d is the thickness of the sample at quenching time. The boundary conditions for $t \geq 0$ are $T(x = 0, t) = T(x = d, t) = T_0$. Then the solution of the heat equation is

$$T(x, t) = T_0 + 4(T_e - T_0) \sum_{m=1, m \text{ odd}}^{\infty} (m\pi)^{-1} e^{-a_m t} \sin(m\pi x/d) \tag{2}$$

with $a_m = m^2\pi^2/(d^2\tau_{th})$ and the reciprocal of the thermal diffusivity $\tau_{th} = \rho c_p/k$. To determine the quenching time, we introduce the dimensionless temperature

$$\Theta(x, t) = [T(x, t) - T_0]/(T_e - T_0). \tag{3}$$

The dimensionless temperature Θ is shown in Fig. 6 for $T_0 = -196\text{ }^\circ\text{C}$, $T_e = 170\text{ }^\circ\text{C}$, and $T_s = 105\text{ }^\circ\text{C}$ at

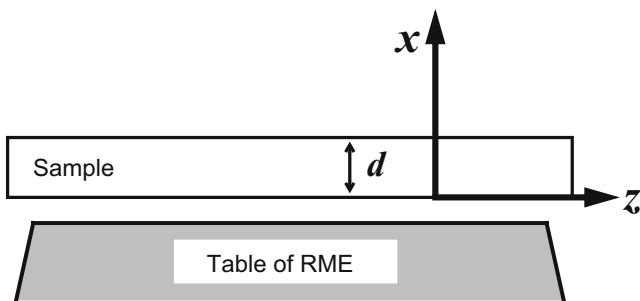


Fig. 5 Cartesian coordinates system for Eq. 1

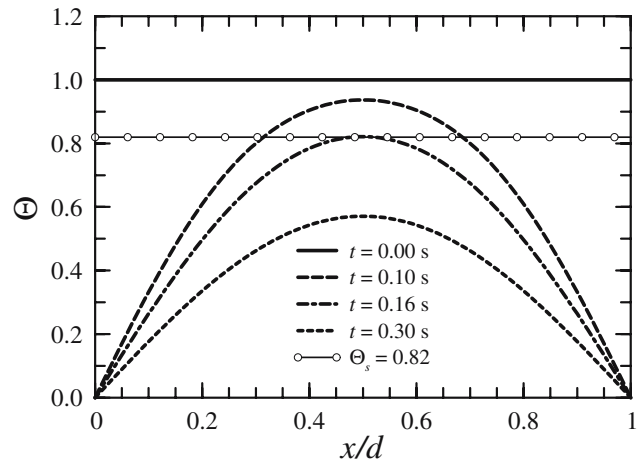


Fig. 6 Dimensionless temperature $\Theta(x, t) = [T(x, t) - T_0]/(T_e - T_0)$ in the sample as a function of x/d at various times t . The reciprocal of the thermal diffusivity is $\tau_{th} = 2 \times 10^7\text{ s m}^{-2}$, and the thickness d of the sample at quenching time is $d = 0.43 \times 10^{-3}\text{ m}$. In addition, $T_0 = -196\text{ }^\circ\text{C}$, $T_e = 170\text{ }^\circ\text{C}$, and $T_s = 105\text{ }^\circ\text{C}$ hold. The dimensionless solidification temperature $\Theta_s = (T_s - T_0)/(T_e - T_0)$ is given by $\Theta_s = 0.82$

$t = 0, 0.10, 0.16,$ and 0.30 s . In our experiments the thickness h of the sample before elongation is $h = 1.5 \times 10^{-3}\text{ m}$. The thickness d at quenching time is given by $d = h \exp(-\epsilon_{\max}/2)$, where ϵ_{\max} denotes the maximum Hencky strain. In Fig. 6, we set $\epsilon_{\max} = 2.5$ and hence $d = 0.43 \times 10^{-3}\text{ m}$. For $\rho = 10^3\text{ kg m}^{-3}$, $c_p = 2 \times 10^3\text{ J K}^{-1}\text{ kg}^{-1}$, and $k = 0.1\text{ W m}^{-1}\text{ K}^{-1}$ the reciprocal of the thermal diffusivity is $\tau_{th} = 2 \times 10^7\text{ s m}^{-2}$. At $t = 0$ the dimensionless temperature Θ is spatially constant and equal to one. At $t = 0.10\text{ s}$ the temperature field attains a roughly parabolic shape. The temperature $T(x, t)$ has dropped below T_s for $0 \leq x \leq 0.31d$ and $0.69d \leq x \leq d$, i.e., at the boundaries of the sample. At $t = 0.16\text{ s}$ we have $T(x, t) \leq T_s$ for $0 \leq x \leq d$ so that the sample has fully solidified within 0.16 s . A simple criterion for a successful quench experiment is given by $(d/\pi)^2\tau_{th} \ll \langle \tau \rangle$ (see Eq. 2), where the average relaxation time $\langle \tau \rangle$ of the macromolecules can be estimated by $\langle \tau \rangle = J_e^0 \eta_0$ with the equilibrium elastic creep compliance J_e^0 at zero shear rate.

We close the analysis with a remark on the dependence of cooling time on the thickness of the sample. Eq. 2 reveals that the relevant timescale of this relaxation process is given by $d^2\tau_{th}$. Therefore, the timescale of quenching is proportional to $h^2 \exp(-\epsilon_{\max})$. The quenching time t_{quench} is defined by the minimal time so that $T(x, t) \leq T_s$ for all x holds in Eq. 2. The quenching time t_{quench} exponentially depends on the maximum Hencky strain ϵ_{\max} and is given by

$$t_{\text{quench}} = t_0 \exp(-\epsilon_{\max}) \tag{4}$$

with $t_0 = 1.94$ s for the parameters $\Theta_s = 0.82$, $\tau_{th} = 2 \times 10^7$ s m⁻², and $h = 1.5 \times 10^{-3}$ m. Thus, t_{quench} strongly depends on the thickness of the stretched sample.

Experiments

To demonstrate the functionality of the quenching tool, we performed melt elongation experiments with polystyrene and quenched the elongated samples. Then the birefringence of the solidified polystyrene was measured and the stress-optical coefficient C was determined. The experiments were performed with a commercially available polystyrene which is provided by BASF AG (Ludwigshafen, Germany) under the trade-name PS 158K (lot no. GK 01142/102). The number and weight average M_n and M_w of the molecular weight, the density ρ , and the glass transition temperature T_g are listed in Table 1. Samples for shear and elongation experiments were prepared by compression molding PS 158K for 10 min at $T = 180$ °C under vacuum. The diameter of the cylindrical samples for shear experiments was 22.0 mm and their thickness was 2.0 mm. The dimensions of the samples for elongational experiments were $1.5 \times 7.0 \times 56.0$ mm³. We performed linear viscoelastic shear oscillations using the Universal Dynamical Shear Rheometer 200 (Paar Physica, Stuttgart, Germany) with a plate–plate geometry at temperatures $T = 140$ to 180 °C with an increment of 10 °C. Then a time–temperature shift to the reference temperatures $T = 150$, 160, and 170 °C were performed using the software LSSHIFT, and a relaxation time spectrum $H(\tau)$ was fitted to the complex modulus by applying the nonlinear regularization routine of the software NLREG (Freiburg Materials Research Center 2001). The uniaxial elongational rheometer RME was used for melt elongation experiments. The test temperatures were $T = 150$, 160, and 170 °C; the applied Hencky strain rate $\dot{\epsilon}_0 = 0.1$ s⁻¹; and the maximum Hencky strain was varied between $\epsilon_{\text{max}} = 1.6$ and $\epsilon_{\text{max}} = 2.8$. The elongation experiments were performed following the recommendations of Schweizer (2000). The chamber of the RME was opened approximately 4 s before

the maximum Hencky strain had been attained. Then the tool was inserted into the RME and at time $t_{\text{max}} = \epsilon_{\text{max}}/\dot{\epsilon}_0$ the sample was quenched.

Using a polarization microscope DM R (Leica, Wetzlar, Germany) with a tilting compensator K 2143 (Leica), we measured the retardation δ_r of the sample. The birefringence Δn is given by

$$\Delta n = \delta_r/d \quad (5)$$

with the thickness d of the stretched sample. Because the change of density ρ of polystyrene during cooling is about 7% (cf. Table 1), the corresponding decrease of d roughly is 2.2% and neglected here. The stress-optical coefficient C is the ratio of Δn and the measured tensile stress σ :

$$C = \Delta n/\sigma, \quad (6)$$

where the tensile stress σ is given by $\sigma(t) = \mu(t)\dot{\epsilon}_0$ at $t = t_{\text{max}}$ with the time-dependent elongational viscosity $\mu(t)$.

The results of our rheological experiments are presented in Fig. 7. The complex modulus $G^* = G' + iG''$ is shown in Fig. 7a. The frequency dependence of the storage and the loss modulus of PS 158K is typical for melts of polydisperse homopolymers with the entanglement plateau at large ω and the terminal zone at low ω . The transient elongational viscosity $\mu(t)$ of polystyrene PS 158K for $\dot{\epsilon}_0 = 0.1$ s⁻¹ at $T = 150$, 160, and 170 °C is presented in Fig. 7b. We also plot the linear viscoelastic prediction $\mu^o(t) = 3\eta^o(t)$, where $\eta^o(t)$ denotes the transient shear viscosity in the linear viscoelastic regime (Ferry 1980):

$$\eta^o(t) = \int_{-\infty}^{\infty} \tau H(\tau)[1 - \exp(-t/\tau)] d \ln \tau. \quad (7)$$

The time-dependent elongational viscosity increases with time t . Polystyrene behaves in a linearly viscoelastic way at small t . At larger times, we have $\mu(t) > \mu^o(t)$, which is denoted by strain-hardening.

Figure 8 presents the birefringence $|\Delta n|$ of the elongated and rapidly quenched samples as a function of tensile stress σ . The birefringence increases linearly

Table 1 Glass transition temperature T_g , number and weight average M_n and M_w of the molecular weight, and density ρ of polystyrene PS 158K (BASF AG)

	T_g (°C)	M_n (g/mol)	M_w (g/mol)	M_w/M_n	ρ at 25 °C (g/cm ³)	ρ at 150 °C (g/cm ³)	ρ at 160 °C (g/cm ³)	ρ at 170 °C (g/cm ³)
PS 158K	105	101,000	305,000	3.04	1.050	0.995	0.989	0.983

The density for $T = 170$ °C is taken from Gramespacher and Meissner (1997) and calculated for $T = 150$ and 160 °C using $d\rho/dT = -6.05 \times 10^{-4}$ g/(cm³ K) (Schrader 1999)

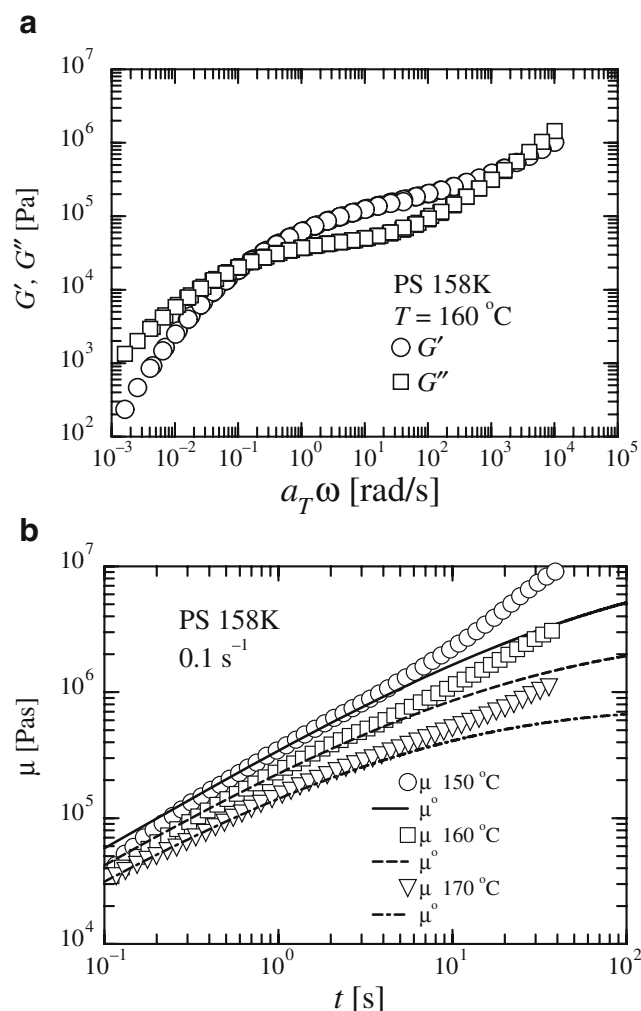


Fig. 7 Rheological properties of polystyrene PS 158K. **a** Storage and loss modulus G' , G'' as a function of frequency ω at $T = 160$ °C. The shift factor is denoted by a_T . **b** Transient elongational viscosity $\mu(t)$ at $T = 150, 160,$ and 170 °C for $\dot{\epsilon}_0 = 0.1$ s $^{-1}$ and its linear viscoelastic prediction $\mu^0(t) = 3\eta^0(t)$ (see Eq. 7)

with stress for our test parameters. The stress-optical coefficient C is the slope of the Δn vs σ curve. Applying a least-squares fit to our data, we find $|C| = (4.65 \pm 0.07) \times 10^{-9}$ Pa $^{-1}$. This C value agrees well with literature data; see, e.g., Muller and Froelich (1985) ($|C| = 4.7 \times 10^{-9}$ Pa $^{-1}$), Venerus et al. (1999) ($|C| = 4.8 \times 10^{-9}$ Pa $^{-1}$), and Luap et al. (2005) ($|C| = 4.6 \times 10^{-9}$ Pa $^{-1}$). Thus, the elongated polystyrene samples were indeed rapidly solidified. Because our data yield $\langle \tau \rangle \approx 328$ s at $T = 150$ °C, $\langle \tau \rangle \approx 145$ s at $T = 160$ °C, $\langle \tau \rangle \approx 26$ s at $T = 170$ °C, and $(d/\pi)^2 \tau_{th} \approx 0.4$ s for $d = 0.43 \times 10^{-3}$ m, the inequality $(d/\pi)^2 \tau_{th} \ll \langle \tau \rangle$ is fulfilled at these temperatures. The value of $\langle \tau \rangle$ decreases with measurement temperature, and therefore our experimental values of $|\Delta n|/\sigma$ scatter at most for $T = 170$ °C.

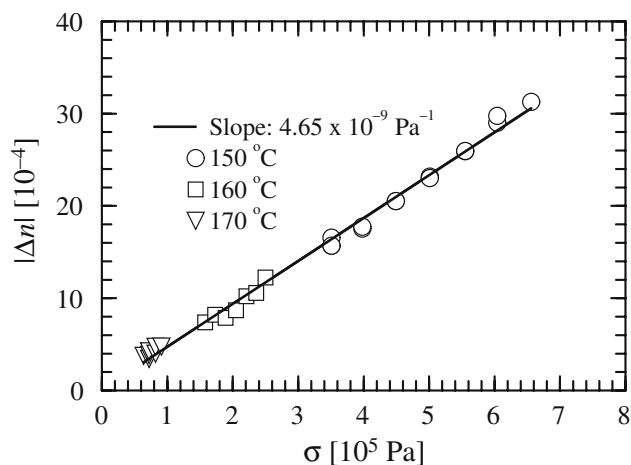


Fig. 8 Flow induced birefringence $|\Delta n|$ as a function of tensile stress σ for polystyrene PS 158K. The applied Hencky strain rate was $\dot{\epsilon}_0 = 0.1$ s $^{-1}$, and the temperature was $T = 150, T = 160,$ and $T = 170$ °C. The linear curve is the result of a least-squares fit with slope 4.65×10^{-9} Pa $^{-1}$

Conclusions

In this study, we developed a tool to rapidly solidify elongated polymer melts. The tool is an accessory to the RME and serves for the determination of the flow-induced anisotropy of polymeric melts. It allows us (1) to pour liquid nitrogen in a controlled way on the stretched polymer, (2) to cut the quenched sample, and (3) to remove it quickly from the chamber of the RME. For example, this tool can be used to freeze in the microstructure of elongated blends of immiscible polymers and tremendously improves the accuracy of the morphological analysis. We applied this newly developed quenching procedure to melt elongation experiments with polystyrene. Melts of polystyrene were elongated to different maximum strains at three temperatures and subsequently quenched. Then the birefringence was measured and the stress-optical coefficient C of polystyrene was determined. Our experimental result is $|C| = 4.65 \times 10^{-9}$ Pa $^{-1}$, which agrees well with data in literature. In conclusion, polymeric materials can be rapidly solidified using this procedure.

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