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Analysis of dynamic mechanical response in torsion

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Abstract

We investigate the dynamic response of industrial rubbers (styrene-butadiene random copolymers, SBR) in torsion and compare against common small amplitude oscillatory shear measurements by using a torsion rectangular fixture, a modified torsion cylindrical fixture, and a conventional parallel plate fixture, respectively, in two different rheometers (ARES 2kFRTN1 from TA Instruments, USA and MCR 702 from Anton Paar-Physica, Austria). The effects of specimen geometry (length-to-width aspect ratio) on storage modulus and level of clamping are investigated. For cylindrical specimens undergoing torsional deformation, we find that geometry and clamping barely affect the shear moduli, and the measurements essentially coincide with those using parallel plates. In contrast, a clear dependence of the storage modulus on the aspect ratio is detected for specimens having rectangular cross section. The empirical correction used routinely in this test is based on geometrical factors and can account for clamping effects, but works only for aspect ratios above a threshold value of 1.4. By employing a finite element analysis, we perform a parametric study of the effects of the aspect ratio in the cross-sectional stress distribution and the linear viscoelastic torsional response. We propose a new, improved empirical equation for obtaining accurate moduli values in torsion at different aspect ratios, whose general validity is demonstrated in both rheometers. These results should provide a guideline for measurements with different elastomers, for which comparison with dynamic oscillatory tests may not be possible due to wall slip issues. © 2016 The Society of Rheology. [<http://dx.doi.org/10.1122/1.4941603>]

I. INTRODUCTION

Dynamic measurements in torsion [i.e., dynamic deformation of a slender cylinder or of a rectangular bar, Figs. 1 and 2(b)] have been performed extensively on rotational rheometers and are often considered as an industrial standard [1]. They provide characterization of the dynamic mechanical response of a wide variety of materials including glasses [2–4], filled and vulcanized systems [5–13], as well as soft gels [14,15]. They are particularly important in

situations where the more common small amplitude oscillatory shear tests (performed with parallel plates or cone-plate geometries) are problematic or not possible at all (e.g., due to slip, transducer compliance or torque limitations). In the past, oscillatory torsional tests were performed by applying relatively large deformations on cylindrical specimens and recording both torque and normal force response as a function of time. Indeed, according to Rivlin [5,16], a state of simple torsion in a cylindrical specimen of an incompressible highly elastic material (which is isotropic in its undeformed state) with fixed ends (i.e., its length remains constant) may be produced by the application of both torque and normal compressive forces in order to maintain a pure torsional deformation. Following the same principle,

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FIG. 1. A rectangular bar (left) when twisted (center) under the action of torque. As a result, rectangular cross-sectional material layers tend to warp (right) around the rotational axis. Here, w is the width, t is the thickness, and l_{act} is the length of the specimen. We can define three relevant aspect ratios, w/t , l_{act}/w , and l_{act}/t .

Kovacs *et al.* allowed a slight vertical flexibility while maintaining torsion rigidity of a clamped prism-shaped specimen to control the tension in a rigid sample near its glass transition temperature (due to isothermal volume contractions) [17]. In a different approach, small torsional deformations were superimposed to large extensional deformations in order to probe crosslinking extension and swelling properties [12]. Different types of specimen geometries can be used in torsional tests. In the rheological literature, the most commonly used has been a cylindrical rod with different aspect ratios [2,3,5,8,10,15,18–20]. On the other hand, these dynamic mechanical tests can also be routinely performed with commercial rheometers such as a strain-controlled rheometer. Commercially available fixtures used for torsional strain-controlled tests have been designed primarily for rectangular cross section of the specimens [6,7,12,17].

Despite these significant developments, an unambiguous set of criteria for assessing dynamic torsional measurements and, more specifically, finding out and correcting possible experimental artifacts, is still missing. Two relevant examples are the effect of the specimen dimensions and the way it is mounted on the fixture (i.e., clamping) on the torsional measurements. Indeed, no systematic studies comparing the viscoelastic response of the same material by using different specimen geometries or different tool geometries are available. The only exceptions appear to be the work of Hsich who performed dynamic oscillatory tests on carbon black natural rubber compounds using both torsion rectangular and

parallel plates geometries [6] and, more recently, the work of Polacco *et al.* [21] on polymer-modified asphalt undergoing dynamic oscillatory shear (parallel plate and cone-plate) and torsional deformation. Regarding the former work, despite the fact that the two tool geometries were used in different temperature ranges (i.e., the torsional rectangular testing mode at low temperatures whereas the parallel plate testing mode at high temperatures), a good agreement between the two sets of data was reported, with the parallel plate data constituting a continuation of the torsion rectangular data in the mechanical frequency spectrum. On the other hand, no overlap region of the two data sets was obtained, apparently due to the different measurement temperature range. The latter authors, Polacco *et al.* [21], found good agreement for high length-to-width aspect ratio used in torsion, when compared to the parallel plate and cone-plate cases. Yet, often the small amounts of samples (for example, when dealing with well-characterized model systems) necessitate the use of small aspect ratios, rendering a deeper analysis of the problem necessary. Furthermore, we note that long before the work of Hsich [6], Szabo speculated the occurrence of extensional and compressional stresses in torsion with rectangular specimens, and in particular, in the region in contact with the clamps [22]. He also proposed an empirical formula in order to quantitatively describe the dynamic-mechanical response in torsion (see discussion below).

The above literature review points to the need for a general protocol for assessing torsional measurements based on appropriate choices of experimental and material geometries. In this work, we address this challenge and attempt at elucidating the role of specimen length-to-width aspect ratio and clamping on torsional measurements and the extracted viscoelastic moduli of a (not vulcanized) rubber. Dynamic frequency sweep (DFS) tests in a parallel plate geometry were also performed and used as benchmark, helping to optimize the experimental conditions of torsional tests. Indeed, the use of a rubber polymer allowed using both measurements on a comparative basis, hence assessing the torsional response. In addition, finite element method (FEM) simulations on a viscoelastic solid (which represents the experimental system reasonably closely) were carried out in order to validate the experimental measurements and analyze the stress fields inside the specimen. The combination of careful experimentation and FEM

FIG. 2. (a) A disk-shaped sample subjected to angular shear deformation, (b) a cylindrical-shaped sample under simple torsion. These two deformations are identical only for circular shapes.

analysis represents a powerful toolbox for the parametric analysis of dynamic response in torsion. In this regard, it is worth mentioning that the same approach was successfully used by Hassager and co-workers [23] for the parametric analysis of extensional measurements of macroscopic polymeric films via the Sentmanat extensional rheometer (SER) [24,25]. Yet, often the small amounts of samples (for example, when dealing with well-characterized model systems) necessitate the use of small aspect ratios, further justifying the presented analysis.

The manuscript is organized as follows: After this introduction, the sample geometries are discussed in Sec. II. Then, Sec. III analyzes the response of a rectangular solid in torsion, whereas the comparison with experiments using frequency sweeps (benchmark) and rectangular specimens or cylindrical specimens is presented in Secs. IV and V, respectively. The final comparison with FEM simulations is reported in Sec. VI. The conclusions are summarized in Sec. VII.

II. SPECIMEN GEOMETRIES

The torsional test consists of twisting a specimen under the action of torque applied at one end while keeping the other one fixed. This situation is depicted for a specimen with a rectangular cross section in Fig. 1.

In the limiting case, we can consider a disk-shaped sample between parallel plates [see Fig. 2(a)], typically used to perform shear measurements in a rotational rheometer. We denote by R and h the radius and the thickness of the sample, respectively, with $h \ll R$. In order to relate the material parameters (strain γ and stress σ) to the rheometer response (rotation angle θ and torque M), two geometry constants, the strain constant K_γ and stress constant K_σ , are used. These constants depend on the geometry sample and can be written as [26]

$$K_\gamma = \frac{\gamma}{\theta} = \frac{R}{h}, \quad (1)$$

$$K_\sigma = \frac{\sigma}{M} = \frac{2}{\pi R^3} = \frac{2R}{\pi R^4} = \frac{R}{J_t}, \quad (2)$$

with $J_t = \pi R^4/2$ is the polar moment of inertia. Both constants depend on the radial position r , i.e., the strain and the shear stress are zero in the center and have a maximum value at the edge of the disk. Due to symmetry, stress and strain only depend on the radius of the plate. This holds only if the specimen has a constant circular cross section.

For higher aspect ratio (i.e., the thickness h becomes larger than the radius R), we refer to the disk and to the applied angular shear deformation as a cylindrical specimen and simple torsion deformation, respectively [see Fig. 2(b)]. In the case of angular shear deformations, the angular displacement θ is used to describe the deformation. On the other hand, in the case of simple torsion deformation, the deformation is described by the angular displacement θ related to the cylinder length l_{act} and is called the twist ξ

$$\xi = \theta/l_{act}. \quad (3)$$

When a cylindrical test specimen is subjected to torsional deformation in the linear regime, disk-shaped layers of solid

material rotate past each other and only shear stress components exist in the sample, i.e., no stresses in the direction normal to the cross section are present. In the case of a nonaxisymmetric test specimen, such as a rectangular bar, the analysis of dynamic-mechanical properties becomes more complex. Such analysis was done first by de Saint-Venant using the theory of elasticity [27]. He developed a general theory for specimen bars with arbitrary cross section, which describes its stress distribution and predicts the maximum stress and yielding for isotropic and homogeneous systems by means of a series expansion approximation. The main assumptions of this theory are: (i) The side surface is free from any load and all of the external load in terms of torque M is applied on the cross section ends only, i.e., the definition of a pure (or free) torsion, (ii) each cross section of the bar does not change its profile in the course of deformation, i.e., a rotation of a rigid body, and (iii) sections perpendicular to the rotational axis of the bar are free to warp, i.e., there is no normal traction upon them and out of plane displacement occurs described by the so called *warping function* ψ .

III. TORSION OF A RECTANGULAR SPECIMEN

As already mentioned, for a specimen with constant circular cross section, the stress is the same at a fixed radial position r and maximum at the cylindrical surface [Fig. 3(a)]. Conversely, in a rectangular shaped specimen, the stress is no longer linear with the distance from the axis and depends on the angular position along the specimen length [Fig. 3(b)]. For a homogenous and sufficiently long rectangular cross-sectional sample, the maximum stress is obtained on the mid-points of the long sides, whereas it is zero at the four corners of the cross section, as the theory of elasticity states and it is shown in Fig. 3(b). As a result of the nonlinear stress distribution, the cross sections of the specimen are not flat anymore but rather warped (Fig. 1).

In order to perform dynamic oscillatory tests in simple torsion, a rectangular test fixture was used in two popular rheometers, a strain-controlled rheometer, ARES 2k FRTN1 from TA Instruments, USA, and a stress-controlled rheometer (operating on strain-control mode), MCR 702 from Anton Paar-Physica, Austria. In this geometry, the deformation at the top and bottom edges of the sample is hindered by the two clamps. When the sample is twisted in torsion, stresses develop at the outer edges of the rectangular sample at contact with the clamps whereas compressional stresses develop in the center. The projections of these stresses into the cross-sectional plane of the rectangular bar alter the torque

FIG. 3. Stress profile under simple torsion deformations in (a) a circular cross section (linear distribution) and (b) a rectangular cross section (nonlinear distribution).

associated with the shear deformation. Extensional and compressional stresses occur near the clamps because they resist to tendency of the test specimen to warp. This leads to an increase of the torsional stiffness, defined as GJ_t (with G being the shear modulus and J_t being the polar moment of inertia for a rectangular cross-sectional bar), i.e., a higher shear modulus is measured. Therefore, the measured value in torsion needs to be corrected.

When measuring torque and angular displacement for a rectangular geometry of the specimen, the following strain and stress constants to calculate the stress and strain are used [28]:

$$K_\gamma = \frac{\gamma}{\theta} = \frac{t}{l_{act}} \left[1 - 0.378 \cdot \left(\frac{t}{w} \right)^2 \right], \quad (4)$$

$$K_\sigma = \frac{\tau}{M} = \frac{3 + 1.8 \cdot \left(\frac{t}{w} \right)}{wt^2} \cdot 1000 \cdot g, \quad (5)$$

where (see also Fig. 1) t is the thickness (mm), w is the width (mm), l_{act} is the actual sample length (mm) (i.e., the measured length of the specimen loaded on the rheometer between the two fixtures), and g is the gravity constant (dm/s^2).

The geometry constant K_g used to convert torque and angular displacement to shear modulus is in fact the ratio between the stress and the strain constant and can be reduced to

$$G = \frac{\sigma}{\gamma} = \frac{K_\sigma M}{K_\gamma \theta} = K_g \frac{M}{\theta} = \frac{K_g M l_{act}}{l_{act} \theta} = \frac{M l_{act}}{J_t \theta} = \frac{M}{J_t \xi}. \quad (6)$$

According to de Saint-Venant's theory [27], J_t of a rectangular sample is given by

$$J_t = \frac{t^3 w}{3} - \frac{64 t^4}{\pi^5} \sum_{n=0}^{\infty} \frac{1}{2(n+1)^5} \tanh \frac{(2n+1)\pi w}{2n} \quad \text{for } w \geq t \quad (7)$$

then, the modulus in torsion can be written as

$$G = \frac{M}{J_t \xi} = \frac{M}{\theta} \frac{3 l_{act}}{wt^3 g_{SV}(u)} \quad \text{with} \quad g_{SV}(u) \cong 1 - \frac{192}{\pi^5} \frac{1}{u} \left[\tanh \frac{\pi}{2} u + 0.004524 \right]. \quad (8)$$

Since this series converges fast, higher terms can be neglected and the expression for the modulus G can be approximated as

$$G = \frac{M}{J_t \xi} = \frac{M}{\theta} \frac{3 l_{act} \cdot 1000 \cdot g}{wt^3 g(u)} \quad \text{with } g(u) = \frac{1 - 0.378 u^{-2}}{1 + 0.6 u^{-1}}, \quad (9)$$

where $u = w/t$ is the aspect ratio.

Wotzel [29] compared the $g_{SV}(u)$ function in Eq. (8) with the approximated function $g(u)$ in Eq. (9). The value of the ratio $g(u)/g_{SV}(u)$ is found to be 1 for all the aspect ratios $u > 1.5$, hence validating the above approximation of the de Saint-Venant prediction.

In a previous work, Szabo [22] observed that dynamic moduli in torsion were affected by tension and compression stresses occurring at contact with the clamps. He suggested to replace the actual sample length l_{act} in Eqs. (8) and (9) with an apparent corrected length l_{corr} in order to correct the effects of the specimen clamping for a given aspect ratio u and Poisson ratio ν

$$l_{corr} = l_{act} - K(u, \nu), \quad (10)$$

where

$$K(u, \nu) = \frac{(1 + \nu)(1 + z^2)}{250} \left[25 \left(4 \frac{1 - z^2}{1 + z^2} - 1 \right) k - 5(1 - \nu) \right. \\ \left. \times (1 + z^6) k^3 - 2(1 + \nu) z^2 k^5 \right], \quad (11)$$

with $k^2 = -(3/2)(1 + u^2) + \sqrt{[(3/4)(1 + u^2)]^2 + (5/(2(1 + \nu))) [4((u^2 - 1)/(1 + z^2)) - u^2]}$ and $z = 1/u$ and ν being the Poisson ratio.

The length correction factor K increases with the aspect ratio u , and when $u > 12$ it becomes independent of u approaching a constant value K_s that depends on the Poisson ratio ν of the material. For example, when $\nu = 0.33$, $K_s = 0.64$ and when $\nu = 0.48$ (typically, the case of rubbers), $K_s = 0.716$.

Hence, for a given aspect ratio u , the aspect ratio l_{act}/w can be chosen for convenience, i.e., in order to overcome stiffening issues it is convenient to maximize the ratio l_{act}/w . Wotzel [29] evaluated the shear moduli for different aspect ratios u and different lengths using the traditional torsion fixture on the strain-controlled RDA2 rheometer (Rheometric Scientific, USA). He found that, for constant aspect ratio w/t , the length correction according to Eq. (10) did not give the correct shear modulus when $10 < l_{act} < 30$ mm, whereas it worked for $l_{act} > 30$ mm. To overcome this limitation, he modified Eq. (10) by coupling the length correction to the ratio of sample width and actual length

$$l_{corr} = l_{act} \left[1 - K(u, \nu) \frac{w}{l_{act}} + 0.12886 \left(\frac{w}{l_{act}} \right)^2 \right] \\ \text{for } 10 < l_{act} < 30 \text{ mm}. \quad (12)$$

This modified equation along with the correction from Szabo [22], Eq. (10), provide the correct shear modulus for a specimen length in the range of 10–80 mm. We use a modification of Eq. (12), in dimensionless form with respect to the specimen width, in order to provide a more general limiting operation range in the present analysis

$$\frac{l_{corr}}{w} = \frac{l_{act}}{w} \left[1 - K(u, \nu) \frac{w}{l_{act}} + 0.12886 \left(\frac{w}{l_{act}} \right)^2 \right]. \quad (13)$$

IV. EXPERIMENTAL RESULTS FOR TORSION RECTANGULAR TEST

Experiments were performed with an industrial rubber polymer. This is a random copolymer of styrene and

butadiene (SBR), synthesized via anionic polymerization and provided by Versalis S.p.A (Ravenna, Italy). The specific system used was a blend of linear and star SBR polymer [weight average molar mass $M_w = 146\,000$ g/mol, $PI = M_w/M_n \approx 1.1$, weight fraction of styrene $w_{\text{Styrene}} = 21.7\%$, vinyl content (i.e., amount of butadiene isomers enchaines as 1,2 addition) $w_{\text{vinyl}} = 67.8\%$, glass transition temperature $T_g = -21^\circ\text{C}$]. We note that the specific SBR used here was not vulcanized, in order to facilitate comparison with conventional DFS measurements (which can be problematic with vulcanized rubbers due to large modulus and associated wall slip issues, which actually render torsion measurements necessary). The (zero-shear) viscosity of this polymer at room temperature is very high, and the sample can be considered as a viscoelastic solid. The torsion rectangular test fixtures of the ARES and MCR rheometers are employed. In order to fit the fixtures into the ovens of both rheometers and control the temperature, the maximum sample length was limited to 45 mm. Rectangular specimens with a constant aspect ratio $u \sim 4$ ($w = 12.2$ mm; $t = 3$ mm) and an actual length ranging from 4.4 to 22.3 mm were molded at 90°C for 20 min under an industrial hot-press heated by electrical platens and cooled down to room temperature for 10 min by means of a water cooling system. The same protocol was used for preparing cylindrical and discotic specimens for the other experiments discussed below. All DFS measurements in torsion were performed at a constant temperature of 30°C . The frequency range was 10^2 – 10^{-1} rad/s and the linear strain amplitude 1%. In the following, we present data from an ARES rheometer unless stated otherwise.

To account for compliance effects, we assessed instrument and sample compliance by checking first the operating range of the instrument, i.e., the region bounded by the maximum and minimum complex modulus G^* that can be measured by each transducer type using a specific geometry (length-to-width aspect ratio). The operating range of an ARES rheometer equipped with a Force Rebalance Transducer 2k FRTN1

and a high torque motor (HT) is depicted in Fig. 4(a) for three length-to-width aspect ratios of rectangular specimens in terms of specimen thickness vs G^* range, from G^* minimum (open symbols) to G^* maximum (solid symbols). The operating range is shown for the thickness range accommodated by the rectangular torsion fixture, from 0.75 to 6.65 mm. The working range for a fixed thickness of 3 mm used here is represented by the full symbols for all of the tests performed. We note that, whereas this assessment represents a guideline for obtaining data virtually free of compliance effects, slight issues at the highest frequencies (from which our DFS measurements start) still remain. The torsional compliance is $1/K = (1/G) \cdot K_g$ with G being its modulus, K_g being the ratio between K_σ and K_γ (constant stress and strain, respectively) as previously described for different testing geometries. As discussed below, for stainless steel (with a very high modulus of 73 GPa) plates of small diameter and large sample thickness, the instrument compliance can be kept to a minimum [30]. However we also note that, whereas large compliance effects occur in stiff, typically glassy materials with values of modulus of the order of GPa, non-negligible effects have been reported in the rubbery plateau region, typically in the range of MPa [31]. We can quantitatively assess this for the present industrial rubber by plotting in Fig. 4(b) the relative strain (i.e., ratio of actual strain over command strain) for rectangular torsion (empty symbols) and angular shear (full symbols) DFS measurements within the applied frequency window. The difference reflects instrument compliance (the transducer cannot measure the actual command strain as it is not sufficiently compliant). We observe that, as mentioned, at higher frequencies (where the material is in fact stiffer) the compliance problem is larger and we confirm the occurrence of the problem even for rubbers [31]. Based on this information, the largest strain deviation (also the respective deviation of the modulus) occurs at the highest frequency and is 29%. Nevertheless, the range of frequencies belonging to the rubbery plateau region at the testing temperature (1–10 rad/s) is

FIG. 4. (a) The operating range of the ARES rheometer equipped with a Force Rebalance Transducers 2K FRTN1 and a HT motor for three length-to-width aspect ratios of rectangular specimens (see text). For a fixed thickness of 3 mm, the sample operating range is represented with full symbols for all of the tests performed (7×10^5 to 1.5×10^6 Pa). (b) Relative strain as a function of frequency for rectangular torsion and angular shear measurements (empty and full symbols, respectively). Inset: Measured and sample torsion compliance (symbols and dashed line, respectively) compared to the machine torsion compliance (1.6×10^{-6} rad/g cm).

FIG. 5. (a) The storage modulus G' as a function of frequency for a SBR sample. Comparison between parallel plate (solid line) and rectangular torsion (symbols) data is shown for rectangular specimens with four different aspect ratios l_{act}/w . (b) G' as a function of the actual specimen length at four different frequencies. A nearly linear decrease with the actual specimen length is observed over the entire frequency range.

not affected by compliance at all. Moreover, the comparison between the measured and sample compliance for all geometries used and the nominal compliance of the ARES transducer 2k FRTN1 is depicted in the inset of Fig. 4(b). Considering the summation of the torsional compliance of the instrument and the sample [30],

$$\frac{1}{K_{meas}^*} = \frac{1}{K_s^*} + \frac{1}{K_t}, \quad (14)$$

where K_{meas}^* is the measured complex torsional stiffness, K_s^* is the actual complex sample torsional stiffness, and K_t is the nominal machine torsional (elastic) stiffness equal to 1.6×10^{-6} rad/g*cm, it is apparent that the measured modulus corresponds to that of the sample for all the measurements. Therefore, we can affirm that the overall results discussed below are not influenced by compliance issues.

In Fig. 5(a), the DFS data of the frequency-dependent storage modulus G' corresponding to the rubbery plateau region are shown. They were obtained at the same experimental conditions using stainless-steel parallel plates (8 mm diameter, 1 mm sample thickness). The gap between plates was appropriately zeroed by means of monitoring the plate parallelism and normal force evolution when they touch each other. DFS measurements were also performed with the torsion rectangular fixture using four different actual lengths while keeping the width and the aspect ratio u values constant to 12 mm and 4, respectively (i.e., varying the aspect ratio l_{act}/w only). We note that the torsional stiffness of the tested material is $K_{sample} = G(\pi/2)(R^4/h)$ with G being its modulus, R its radius, and h its thickness. Hence, as already mentioned, for stainless steel plates of small diameter and large sample thickness, the instrument compliance is minimum [30]. We also note however that, whereas large compliance effects occur in stiff, typically glassy materials with values of modulus are of the order of GPa, non-negligible effects have been reported in the rubbery plateau, typically in the range of MPa [31]. We can quantitatively assess this for the present industrial rubber

by plotting in Fig. 5(a) the imposed (command) strain amplitude in the DFS and the actual amplitude measured. The difference reflects instrument compliance (the transducer cannot measure the actual command strain as it is not sufficiently compliant).

When the ratio l_{act}/w decreases, the storage modulus G' tends to increase due to the rather strong friction effect of the two clamps over the whole specimen during the dynamic test (see also discussion below in conjunction with Fig. 7). All G' curves are vertically shifted [Fig. 5(a)], reflecting the linear decrease of G' with the actual specimen length for all frequencies tests [Fig. 5(b)]. An increment from $\approx 30\%$ to $\approx 160\%$ of G' values in torsion from parallel plate data was observed. We note that tests performed with specimens having different aspect ratio u revealed no significant change of the storage modulus G' within the experimental frequency window observed (data not shown). This led us to conclude that it is the specimen length that plays the major role in torsional viscoelastic response.

Figure 6 depicts the correction of G' using Eq. (13) for two different length-to-width aspect ratios. It is evident that the applied correction works well for larger aspect ratios l_{act}/w , whereas it becomes less effective for values of l_{act}/w below about 1.4. An overlap of torsional and parallel plate data (within an error of 3%–5%) for the SBR sample (for which parallel plate data can be obtained) is considered as a validation of the torsional experiment and the conditions used. A residual error of 15%–18% is observed for all the correction applied to smaller specimen length-to-width ratios. More detailed results are listed in Table I.

A major consideration in torsional test is clamping, which is known to have an impact on the measured material properties (e.g., the plateau modulus). Although a preliminary analysis was conducted to explore its influence, a thorough quantitative assessment of the role of imposed clamping is a difficult multiparameter problem and a detailed and comprehensive analysis will be the subject of future investigations. In the present work, we qualitatively analyzed the effects

FIG. 6. Comparison of frequency sweep data of SBR using parallel plate geometry (open symbols) and torsional data using rectangular geometry (dashed line) and corrected in Eq. (12) (solid line) in terms of G' . Data are shown for two length-to-width aspect ratios, 1.46 (a) and 1.2 (b).

of different levels of clamping on the same SBR sample (Fig. 7). In particular, after loading the rectangular specimen into the torsional fixture, clamping was performed by tightening the screws of the two clamps. That was achieved by applying the same number of turns on each screw in the upper and lower clamp. This yielded a normal force on the fixture which could be measured by the rheometer transducer. Hence, a quantitative estimation of the level of clamping (tight/soft) was obtained. We call *soft* clamping that leading to a normal force of 1 N and *tight* clamping to 3 N. We observe that a tighter clamping affects shorter samples more than longer ones: The storage modulus G' increases of about 11% in the largest aspect ratio l_{act}/w (≈ 1.85), and of about 51% in the smallest one (≈ 0.9). Furthermore, in Fig. 8, we illustrate how a tight/soft clamping can have a role on the effectiveness of the torsional G' correction. It is evident that, whereas the degree of correction (needed to account for the effect of l_{act}/w as discussed above) increases as the specimen length decreases ($\approx 46\%$ correction for $l_{act}/w \approx 0.9$ and $\approx 27\%$ correction for $l_{act}/w \approx 1.85$), its dependence on the clamping level is rather complex: For both the largest ($l_{act}/w \approx 1.85$) and the smallest ($l_{act}/w \approx 0.9$) length-to-width aspect ratio, the correction by using Eq. (13) is more effective for tight than soft clamping ($\approx 1.1\%$ error with respect to parallel plate data for tight clamping and $\approx 8\%$ error for soft clamping), for $l_{act}/w \approx 1.85$ ($\approx 31\%$

error for soft clamping and $\approx 18\%$ error for tight clamping for $l_{act}/w \approx 0.9$). We addressed such a correction discrepancy due to slip issues at the sample edges in contact with the torsion rectangular fixture by probing the effect on dynamic mechanical properties from two types of sample loading. We first clamped normally the rectangular specimen at the edges applying both tight and soft clamping, and in a subsequent experiment, we carefully glued the specimen ends to the testing fixture and no clamping was applied. The loading protocol ensured that the specimen ends were reasonably flat and perpendicular to the fixtures in both cases. No compression was applied when loading. The results are depicted in Fig. 8. The enhancement of the storage modulus G' is affected in virtually the same way in glued and tight clamping cases for both specimen lengths (Fig. 9). This suggests lack of slip in tight clamping and, moreover, that G' is mainly affected by the rectangular cross-sectional geometry of the specimen used. However, the good agreement of torsional G' correction with respect to parallel plate data holds for $l_{act}/w \approx 1.85$ only, whereas it needs to be improved for $l_{act}/w \approx 0.9$.

Based on the above, we propose two new empirical formulas inspired by and extending Eq. (13), in order to obtain correct values of dynamic moduli when the aspect ratio l_{act}/w approaches a value of 1 or is below 1. Figure 10 depicts the correction in terms of the storage modulus for $l_{act}/w \approx 1.2$ (a) by using Eq. (14) and for $l_{act}/w \approx 0.35$

TABLE I. Fractional difference between measured (G'_{act}) and corrected (G'_{corr}) torsional data and benchmark parallel plate (G'_{PP}) data for different length-to-width aspect ratios, comparing the initial correction given in Eq. (13) and marked as OLD, with the improved correction given in Eqs. (15) and (16) and marked as NEW.

$\frac{l_{act}}{w}$	$\frac{l_{corr}}{w} _{OLD}$	$\frac{l_{corr}}{w} _{NEW}$	$K(u, \nu)$	$\frac{G'_{act} - G'_{PP}}{G'_{PP}} (\%)$	$\frac{G'_{corr} _{OLD} - G'_{PP}}{G'_{PP}} (\%)$	$\frac{G'_{corr} _{NEW} - G'_{PP}}{G'_{PP}} (\%)$
1.85	1.48	—	0.55	31.2	3.4	—
1.46	0.9	—	0.55	64	2.5	—
1.2	0.64	0.6	0.55	111.3	15.2	3.7
0.9	0.48	0.38	0.55	120	18.2	5
0.35	0.16	0.13	0.55	163.5	17	2.8

FIG. 7. Torsional measurements performed at different levels of clamping. Data are presented as G' against angular frequency: Tight and soft clamping in torsion (triangles and circles, respectively) are compared against benchmark parallel plate data (solid line). Two data sets using two aspect ratios l_{act}/w are reported: (a) 1.85 and (b) 0.9. The storage modulus G' enhancement is almost five times larger when the length-to-width ratio is reduced by 50%.

(b) by using Eq. (15). Experimental torsional data obtained with the MCR 702 rheometer and the same empirical correction formulas are shown as in Fig. 11. A summary of the empirical correction formulas for torsional data is presented in Table I

$$\frac{l_{corr}}{w} = \frac{l_{act}}{w} \left[1 - K(u, \nu) \frac{w}{l_{act}} + 0.12886 \left(\frac{w}{l_{act}} \right)^2 - 0.07 \left(\frac{w}{l_{act}} \right)^3 \right] \quad \text{for } 0.83 < \frac{l_{act}}{w} < 1.25, \quad (15)$$

$$\frac{l_{corr}}{w} = \frac{l_{act}}{w} \left[1 - K(u, \nu) \frac{w}{l_{act}} + 0.12886 \left(\frac{w}{l_{act}} \right)^2 - 0.07 \left(\frac{w}{l_{act}} \right)^3 + 0.00857 \left(\frac{w}{l_{act}} \right)^5 \right] \quad \text{for } 0.33 < \frac{l_{act}}{w} < 0.83. \quad (16)$$

V. EXPERIMENTAL RESULTS FOR TORSION CYLINDRICAL TEST

Given the nonuniform stress distribution in the rectangular geometry [Fig. 3(b)], we now focus on cylindrical specimens having the same diameter ($R \approx 1.5$ mm) undergoing torsion. Cylindrical specimens were obtained following the same molding protocol described in Sec. IV for rectangular specimens. They were mounted on appropriately modified cylindrical torsional fixtures. Similarly to the experiments with rectangular specimens, DFS tests were also performed at the same conditions and confirm, irrespectively of the chosen aspect ratio l_{act}/R , the homogeneous stress distribution throughout the specimen's cross section, hence the reliability of the obtained data. Unlike the rectangular geometry case, the variation of the aspect ratio l_{act}/R or the level of clamping (i.e., from soft to tight or ultratight clamping by adding one turn more to both clamp screws each time) does not affect the torsion storage modulus G' , as shown by the data in Fig. 12.

FIG. 8. The effect of G' correction using Eq. (13) (line) on the torsional experimental data (closed symbols) for two aspect ratios l_{act}/w , 1.85 (a) and 0.9 (b). The empirical correction is applied to two clamping levels, tight and soft (circular and triangular full symbols for experimental torsion data, solid and dashed line for correction with Eq. (13), respectively). Comparison with data obtained using parallel plates benchmark (open symbols) is also shown.

FIG. 9. Storage modulus G' as a function of frequency for two aspect ratios l_{act}/w , 1.85 (a) and 0.9 (b) in torsion (symbols and dashed line), compared against respective values obtained with parallel plates (solid line). Comparison among three types of loading for rectangular cross-sectional specimens is also depicted in order to assess slip issues (full symbols for tight clamping and dashed line for soft clamping, empty symbols for glue loading).

VI. FEM SIMULATION RESULTS FOR TORSIONAL TESTS

The above comparative study provides confidence in performing torsional measurements but also renders an independent quantitative analysis necessary, in order to assess the influence of geometric parameters in the stress distribution profile, especially when designing new fixtures and defining new protocols for specific applications. To this end, we have carried out FEM simulations in order to predict the dynamic-mechanical response of a viscoelastic solid material in torsion. The results are compared to the experimental data discussed above. More specifically, we simulated torsional deformations for cylindrical and rectangular specimens using a linear viscoelastic model. No clamping effects are accounted for in the simulations. We solved the standard equations governing the deformation of a solid

$$\rho \frac{\partial^2 \mathbf{u}}{\partial t^2} = \nabla \cdot \boldsymbol{\sigma}, \quad (17)$$

$$\boldsymbol{\sigma} = \mathbf{C} : \boldsymbol{\varepsilon} + \boldsymbol{\sigma}_v, \quad (18)$$

where $\mathbf{u}$ is the displacement, ρ is the solid density, $\boldsymbol{\sigma}$ is the stress tensor, $\mathbf{C}$ is the fourth-order stiffness tensor, $\boldsymbol{\varepsilon}$ is the strain tensor, and $\boldsymbol{\sigma}_v$ is the viscoelastic contribution to the stress. Concerning the boundary conditions: (i) A prescribed sinusoidal angular displacement $\theta(t)$ is applied on the upper section of the specimen, (ii) the lower section is kept fixed, and (iii) the lateral surface of the solid domain is free to deform, i.e., no load is applied. The generalized (multimode) Maxwell model is used in general as constitutive equation for viscoelastic material response. This model is chosen for its simplicity and used only for fitting the shear modulus $G(t)$. To this end, it does not matter for the final result whether $G(t)$ represents the response of a viscoelastic liquid or viscoelastic solid. It approximates the viscoelastic material function $G(t)$ by means of a discrete relaxation-time spectrum using a sum of exponentials

FIG. 10. Storage modulus G' against angular frequency in torsion for three length-to-width aspect ratios $l_{act}/w \geq 1$ (a) or $l_{act}/w \leq 1$ (b). Experimental torsional data (full symbols) from the ARES rheometer were corrected using both Eq. (13) (dashed line) and Eq. (15) (solid line) for (a) 1.2, and Eq. (16) (solid line) for (b) 0.35, respectively. The residual error from parallel plate data (empty symbols) is less than 5% with both modified empirical formulas.

FIG. 11. Storage modulus G' against angular frequency in torsion for two length-to-width aspect ratios l_{act}/w : (a) 0.9 and (b) 0.35. Experimental torsional data (full symbols) from the MCR 702 rheometer corrected with both Eq. (13) (dashed line) and Eq. (15) (solid line) for (a) 0.9, and Eq. (16) (solid line) for (b). The parallel plate data (empty symbols) are shown for comparison.

$$G(t) \cong G_{\infty} + \sum_{i=1}^{N_M} G_i \exp\left(-\frac{t}{\tau_i}\right), \quad (19)$$

where the set of fitting empirical parameters $\{\tau_i, G_i\}$ are named relaxation times and relaxation strengths, respectively, G_{∞} is the long term modulus (i.e., after relaxation), and N_M is the number of modes. Only in the limit of large N_M , Eq. (19) should, in principle, approach the underlying material function $G(t)$. For sinusoidal oscillatory shear flow deformation of angular frequency ω , the following expression of the storage modulus $G'(\omega)$ is derived from Eq. (19):

$$G'(\omega) = G_{\infty} + \sum_{i=1}^{N_M} G_i \frac{(\tau_i \omega)^2}{1 + (\tau_i \omega)^2}. \quad (20)$$

The number of modes as well as the constitutive parameters are estimated by fitting the G' measurements obtained in the parallel plate 8 mm. The number of modes are progressively increased until the predicted G' matches the measurements

within a sufficient accuracy [32,33]. Three modes are required to fairly describe the experimental data. Fitting of the storage modulus G' data in the parallel plate case are depicted in Fig. 13. Finally, hexahedral elements are employed to discretize the specimen geometry. For all simulations, mesh convergence is achieved by using 8000 elements.

We carried out torsion simulations on cylindrical and prismatic specimens of different aspect ratios l_{act}/w , imposing an angular displacement $\theta(t)$ that gives a maximum strain inplane component

$$\varepsilon_p = \sqrt{\varepsilon_{xz}^2 + \varepsilon_{yz}^2} \quad (21)$$

equals to 0.01 when averaged on the midplane at $z = l_{act}/2$. We choose such a plane in order to reduce as much as possible the effect of the boundary conditions.

In Fig. 14, we report the distribution of the stress inplane component

$$\sigma_p = \sqrt{\sigma_{xz}^2 + \sigma_{yz}^2} \quad (22)$$

FIG. 12. Storage modulus G' as a function of frequency for cylindrical specimens. A good agreement between torsion (symbols) and parallel plate (solid line) data is found when changing the aspect ratio l_{act}/R (a) or applying a different clamping level (b).

FIG. 13. Storage modulus G' as a function of frequency obtained from parallel plate 8 mm and fitting results from the generalized Maxwell model with three modes (open symbols and solid line, respectively) are shown.

calculated by FEM simulations in the midplane at $z = l_{act}/2$ of a rectangular bar (aspect ratio $u = 4$) undergoing simple torsion. Three cases, corresponding to $l_{act}/w = 1.85$ (a), $l_{act}/w = 0.9$ (b), and $l_{act}/w = 0.35$ (c), are shown. The distributions are taken at a time corresponding to maximum mean stress computed on the midplane, and at a frequency of $\omega = 100$ rad/s. It is well known that the stress distribution in a cylindrical specimen is radial and independent of the aspect ratio l_{act}/R . In contrast, in a rectangular specimen, the stress distribution depends on the axial position. In addition, the stress distribution is affected by the aspect ratio l_{act}/w , as clearly visible in Fig. 14. For the largest aspect ratio l_{act}/w considered in this work [Fig. 14(a)], the maximum stress is obtained at the midpoint of the long edges, in agreement

FIG. 14. Distribution of the stress inplane component calculated by FEM simulations in the cross-sectional midplane ($z = l_{act}/2$) of a rectangular specimen with width-to-thickness aspect ratio $u = 4$, undergoing simple torsion. Results are depicted for three different values of the length-to-width aspect ratio $l_{act}/w = 1.85$ (a), $l_{act}/w = 0.9$ (b), and $l_{act}/w = 0.35$ (c). The distributions are taken at a time corresponding to maximum mean stress and at $\omega = 100$ rad/s. The bottom horizontal bar is the color code stress values (Pa units).

FIG. 15. Elastic modulus G' as a function of angular frequency, computed by FEM simulations (empty symbols) for a cylindrical specimen of aspect ratio $l_{act}/R = 7.2$ (diamonds), and three rectangular specimens of aspect ratio $l_{act}/w = 1.85$ (squares), $l_{act}/w = 0.9$ (up triangles), and $l_{act}/w = 0.35$ (circles), respectively. The experimental data for the parallel plate (solid line) are also reported for comparison. No clamping was taken into account in the simulations.

with the 2D Saint-Venant theory. On the other end, when l_{act}/w decreases, the stress along the longest edge decreases as well, while the stress on the shortest edge increases. Hence, for sufficiently short samples, the maximum stress is located at the midpoint of the shortest edges.

Despite the significantly different stress profiles in the rectangular specimens of various length-to-width aspect ratios, the computed elastic modulus G' is always the same, as shown in Fig. 15. In the same figure, we also report the elastic modulus calculated for a cylindrical specimen ($l_{act}/R = 7.2$) and the experimental data for the parallel plate. All data collapse on the same curve, suggesting that the shape and aspect ratio of the specimen do not affect the linear viscoelastic response of the material.

In conclusion, the deviations of the experimental data for the rectangular specimens from the parallel plate measurements reported in Fig. 4(a) might be related to the presence of the clamps.

VII. CONCLUSIONS

This work aims at providing simple guidelines for obtaining reliable data when performing dynamic mechanical measurements in torsion. To this end, we have utilized a combination of careful experiments performed on two different commercial rheometers, strain-controlled ARES 2k FRTN1 and stress-controlled (operating in strain-control mode) MCR 702, quantitative semiempirical analysis and FEM simulations. The chosen experimental system, an industrial nonvulcanized rubber SBR, was suitable for both angular shear and torsional measurements. This study was motivated by the observed non-negligible variance of the storage modulus G' depending on the rectangular specimen aspect ratio (length-to-width aspect ratio), during a dynamic mechanical measurement in

torsion. We have investigated specimens of different aspect ratios and have assessed the role of the level of clamping used in torsional tests. Based on our results, we have proposed a new, more successful empirical equation for obtaining the correct values of moduli in torsion. Concomitantly, FEM simulations results indicated the same linear viscoelastic response of the material from different nonlinear stress distribution profiles depending on the length-to-width aspect ratio of rectangular specimens, suggesting the importance of clamping in the experimental specimen loading protocol. More reliable torsional data can be obtained when using cylindrical specimens because of the more uniform cross-sectional stress distribution. On the other hand, the proposed methodology allows employing rectangular specimens successfully. Hence, in summary, we have extended the validity of torsional rectangular measurements in commercial rheometers to length-to-width aspect ratios as low as 0.35 by proposing new correction Eqs. (15) and (16). It is now possible to measure reliably, also by accounting for compliance issues and clamping, the latter however still in a semiquantitative way. Indeed, an outstanding challenge for future work is the implementation of clamping effects into FEM simulations in order to give further general guidelines to quantitatively correct the related observed experimental discrepancies between torsion and angular shear protocols.

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References

- [1] ASTM D5279, *Standard Test Method for Plastics: Dynamic Mechanical Properties: In Torsion* (ASTM International, West Conshohocken, PA, 2013), pp. 2–5.
- [2] O’Connell, P. A., and G. B. McKenna, “Large deformation response of polycarbonate: Time-temperature, time-aging time, and time-strain superposition,” *Polym. Eng. Sci.* **37**, 1485–1495 (1997).
- [3] O’Connell, P. A., and G. B. McKenna, “The non-linear viscoelastic response of polycarbonate in torsion: An investigation of time-temperature and time-strain superposition,” *Mech. Time-Dependent Mater.* **6**, 207–229 (2002).
- [4] Santore, M. M., R. S. Duran, and G. B. McKenna, “Volume recovery in epoxy glasses subjected to torsional deformations: The question of rejuvenation,” *Polymer (Guildf)* **32**, 2377–2381 (1991).
- [5] Rivlin, R. S., and D. W. Saunders, “Large elastic deformations of isotropic materials. VII. Experiments on the deformation rubber,” *Philos. Trans. R. Soc. A Math. Phys. Eng. Sci.* **243**, 251–288 (1951).
- [6] Hsich, H. S. Y., “Composite rheology- I. Elastomer-filler interaction and its effect on viscosity,” *J. Mater. Sci.* **17**, 438–446 (1982).
- [7] Klüppel, M., “Evaluation of viscoelastic master curves of filled elastomers and applications to fracture mechanics,” *J. Phys. Condens. Matter* **21**, 1–10 (2009).
- [8] McKenna, G., and L. Zapas, “On the small strain behavior of peroxide crosslinked natural rubber,” *Rubber Chem. Technol.* **59**, 130–137 (1986).
- [9] Penn, R. W., and E. A. Kearsley, “The scaling law for finite torsion of elastic cylinders,” *Trans. Soc. Rheol.* **20**, 227–238 (1976).
- [10] Ferry, J. D., R. G. Mancke, E. Maekawa, Y. Oyanagi, and R. A. Dickie, “Dynamic mechanical properties of cross-linked rubbers. I. Effects of cross-link spacing in natural rubber,” *J. Phys. Chem.* **2781**, 3414–3418 (1964).
- [11] R. A. Stratton, and J. D. Ferry, “Dynamic mechanical properties of natural rubber vulcanizates cross-linked by various agents,” *J. Phys. Chem.* **67**, 2781–2785 (1963).
- [12] Gent, A. N., and T. H. Kuan, “Thermoelasticity of stretched elastomers under small torsions: Effects of crosslinking, extension, and swelling,” *J. Polym. Sci. Part A-2 Polym. Phys.* **11**, 1723–1733 (1973).
- [13] McKenna, G. B., and L. J. Zapas, “Experiments on the small-strain behaviour of crosslinked natural rubber: 2. Extension and compression,” *Polymer* **24**, 1502–1506 (1983).
- [14] Janmey, P. A., “A torsion pendulum for measurement of the viscoelasticity of biopolymers and its application to actin networks,” *J. Biochem. Biophys. Methods* **22**, 41–53 (1991).
- [15] Plazek, D. J., M. N. Vrancken, and J. W. Berge, “A torsion pendulum for dynamic and creep measurements of soft viscoelastic materials,” *Trans. Soc. Rheol.* **2**, 39–51 (1958).
- [16] Rivlin, R. S., “Torsion of a rubber cylinder,” *J. Appl. Phys.* **18**, 444–449 (1947).
- [17] Kovacs, A. J., R. A. Stratton, and J. D. Ferry, “Dynamic mechanical properties of polyvinyl acetate in shear in the glass transition temperature range,” *J. Phys. Chem.* **67**, 152–161 (1963).
- [18] Duran, R. S., and G. B. McKenna, “A torsional dilatometer for volume change measurements on deformed glasses: Instrument description and measurements on equilibrated glasses,” *J. Rheol.* **34**, 813–839 (1990).
- [19] Plazek, D. J., W. Dannhauser, and J. D. Ferry, “Viscoelastic dispersion of polydimethyl siloxane in the rubberlike plateau zone,” *J. Colloid Sci.* **16**, 101–126 (1961).
- [20] Flory, A. L., and G. B. McKenna, “Chemical structure - Normal force relationships in polymer glasses,” *Polymer* **46**, 5211–5217 (2005).
- [21] Polacco, G., O. J. Vacin, D. Biondi, J. Stastna, and L. Zanzotto, “Dynamic master curves of polymer modified asphalt from three different geometries,” *Appl. Rheol.* **13**, 118–124 (2003).
- [22] Szabo, I., *Hohere Technische Mechanik* (Springer-Verlag New York, Inc., Berlin, 1956).
- [23] Hassager, O., J. Manuel, R. Marin, K. Yu, and H. Koblitz, “Polymeric liquids in extension: Fluid mechanics or rheometry?,” *Rheol. Acta* **49**, 543–554 (2010).
- [24] Sentmanat, M. L., “Miniature universal testing platform: From extensional melt rheology to solid-state deformation behavior,” *Rheol. Acta* **43**, 657–669 (2004).
- [25] Sentmanat, M. L., B. N. Wang, and G. H. McKinley, “Measuring the transient extensional rheology of polyethylene melts using the SER universal testing platform,” *J. Rheol.* **49**, 585–606 (2005).
- [26] Ferry, J. D., *Viscoelastic Properties of Polymers*, 3rd ed. (John Wiley & Sons, Inc., New York, 1980).
- [27] Saint-Venant, A. J. C. B., “Mémoire sur la torsion des prismes,” *Mémoires présentés par Divers savants à l’Académie des Sci.* **14**, 233–560 (1856).
- [28] Roark, R. J., and W. C. Young, *Formulas for Stress and Strain*, 1st ed. (McGraw Hill, New York, 1975).
- [29] Wotzel, D., “Der einfluss der probengeometrie und der viskoelastischen eigenschaften auf die korrektur dynamischmechanischer meßwerte an verschiedenen geräten (RDA, RFS, DSR),” Ph.D thesis, MartinLuther Universität Halle, Germany, 1994.

- [30] Schröter, K., S. A. Hutcheson, X. Shi, A. Mandanici, and G. B. McKenna, "Dynamic shear modulus of glycerol: Corrections due to instrument compliance.," *J. Chem. Phys.* **125**, 214507 (2006).
- [31] Hutcheson, S. A., and G. B. McKenna, "The measurement of mechanical properties of glycerol, m-toluidine, and sucrose benzoate under consideration of corrected rheometer compliance: An in-depth study and review.," *J. Chem. Phys.* **129**, 074502 (2008).
- [32] Snijkers, F., G. D'Avino, P. L. Maffettone, F. Greco, M. Hulsen, and J. Vermant, "Rotation of a sphere in a viscoelastic liquid subjected to shear flow. Part II. Experimental results," *J. Rheol.* **53**, 459–480 (2009).
- [33] Snijkers, F., G. D'Avino, P. L. Maffettone, F. Greco, M. A. Hulsen, and J. Vermant, "Effect of viscoelasticity on the rotation of a sphere in shear flow," *J. Non-Newtonian Fluid Mech.* **166**, 363–372 (2011).