Universality in the Non-Newtonian Viscous Flow Behavior of $\text{As}_x\text{Se}_{1-x}$ Liquids: Results from Capillary Rheometry

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Abstract

The shear rate dependence of the viscosity of $\text{As}_x\text{Se}_{1-x}$ ($5 \leq x \leq 40$) liquids is studied using capillary rheometry. All liquids display non-Newtonian rheological behavior in the form of shear thinning. The onset of such behavior is characterized by shear rates that are nearly four orders of magnitude slower than the average rate of structural relaxation. Proper normalization of the viscosity vs. shear rate data demonstrates a remarkable universality in this behavior, irrespective of chemical composition, structure, topology and temperature. The existence of such universality suggests an underlying commonality in the fundamental atomistic mechanism of shear thinning in these chalcogenide liquids.

1. Introduction

The unique compositional flexibility of chalcogenide glasses in the form of continuous alloying enables tuning of optical, electronic, thermo-mechanical and other properties relevant to various technological applications of these materials [1-5]. Chalcogenide optical elements have various uses in diverse technologies including sensors, image projectors, displays and optoelectronic devices for telecommunications [6-9]. Complex, precision optical elements can be manufactured using numerous materials and methods. However, a great majority of conventional machining processes are unsuited for producing small features that are less than 500 microns. The fabrication of microstructured surfaces using polymers is derived from processes developed by the semiconductor industry for making integrated circuits. For example, photolithography and ion etching techniques can create micron-scale surface features but such methods are not suitable for large-scale manufacturing [8]. Molding or hot embossing on the other hand, can form micron to sub-micron sized features. Unfortunately, for many photonic applications, polymers are not ideal as they are mechanically weak, undergo environmental degradation over time and cannot withstand exposure to high temperature and humidity. Moreover, they are also not completely transparent in the infrared. All these shortcomings can potentially be circumvented by using chalcogenide glasses which, owing to their low glass-transition temperatures (T_g) compared to oxide glasses, are amenable to the conventional polymer processing/forming techniques such as injection molding, extrusion, embossing, thermo-forming, blow-molding, rotational molding and, of course, fiberization [1]. However, during such forming or processing, the parent liquids may be subjected to large strain rates [1, 10, 11].

It is well known that glass-forming liquids display a Newtonian behavior for low to moderate deformation rates [12-14] where the shear viscosity η is independent of the applied strain rate $\dot{\gamma}$. It is useful to recall here that by definition $\eta = \frac{\tau_{shear}}{\dot{\gamma}}$, where τ_{shear} is the applied shear stress. Hence, τ_{shear} varies linearly with $\dot{\gamma}$ in the Newtonian regime. However, sufficiently high stresses and strain rates typically lead to a change from the linear Newtonian to a non-Newtonian behavior where the shear viscosity decreases with increasing shear rate i.e. the liquid displays shear thinning [12-20]. Such shear thinning behavior of the parent liquid may influence the physical properties of the resulting glass and hence, a fundamental mechanistic understanding of the cause and effect of this behavior is important in optimization of the processing parameters.

Most of the previous studies of shear thinning in glass-forming liquids focused on oxide glasses [12-20] and only a few reports can be found in the literature on the non-Newtonian behavior in chalcogenide glasses and liquids [1, 10, 11, 17]. However, surprisingly little is known regarding the atomistic mechanism(s) of shear thinning in supercooled glass-forming liquids under high strain rate. In the present work, we report the results of a systematic study of the Newtonian vs. non-Newtonian behavior in binary As_xSe_{1-x} liquids with $5 \leq x \leq 40$, using capillary rheometry. It is well known that as the composition gradually changes from pure Se to stoichiometric As_2Se_3 , the structure of the As_xSe_{1-x} glasses transforms from one dominated by selenium chains to a network of corner-shared $AsSe_3$ pyramids and the average coordination number increases from 2 to 2.4. Such compositional variation of the connectivity and topology of the structural network in this glass-forming system is accompanied by a wide variation in the thermophysical and elastic properties and provides the opportunity to explore the systematics of the corresponding variation in their non-Newtonian shear thinning behavior.

2. Experimental

2.1. Glass Synthesis

As_xSe_{1-x} glass samples with x = 5, 10, 20, 30 and 40 were synthesized in 90g batches by melting a mixture of the constituent elements ($\geq 99.999\%$ purity, metals basis) in an evacuated (10^{-6} Torr) fused silica ampoule in a rocking furnace. The stoichiometric composition As₂Se₃ (x=40) was found to be prone to crystallization, when subjected to high shear rates in the non-Newtonian regime during capillary rheometry measurements. This problem was circumvented by adding 3 mol% GeSe₂ to the batch, such that the final glass composition was (GeSe₂)₃(As₂Se₃)₉₇. The mixtures were heated to up to 700°C, rocked for at least 24 hours to ensure melt homogeneity, and subsequently quenched in water. Resulting glass samples were broken into small pieces for viscosity measurement with a capillary rheometer.

2.2. Capillary Rheometry

The viscosity of As_xSe_{1-x} liquids was measured as a function of shear rate, using a twin-bore capillary rheometer (Rosand RH2000, Malvern Instruments). The basic experimental setup for capillary rheometry consists of extruding the material under investigation through a capillary die by applying pressure with a piston. The pressure at the entrance of the capillary is monitored with a transducer, as a function of time and piston speed. The shear stress τ_{shear} and the apparent shear rate $\dot{\gamma}$ are determined from the pressure and piston speed, respectively:

$$\tau_{shear} = \frac{\Delta PR}{2L} \quad (1)$$

$$\dot{\gamma} = \frac{4R_b^2 V_b}{R^3} \quad (2)$$

where ΔP is the pressure drop, R is the radius of the die, L is the bore length, R_b is the bore radius and V_b is the piston speed. The shear stress is then corrected for the die entrance and exit effects on the pressure drop (Bagley correction) and the apparent shear rate is corrected for the possible non-parabolic velocity profile effect (Rabinowitsch correction). The true viscosity can then be obtained as the ratio of the corrected shear stress and shear rate.

The circular capillary dies used in this study are made of tungsten carbide and have an entrance angle of 90° and a diameter of 0.75 mm (1 mm) with a capillary length of 15 mm (16 mm). The bores that contained the liquid under investigation are made of nitrided steel and have a diameter of 9.5 mm and a total length of 250 mm. A stainless steel piston with a phosphor bronze tip drives the liquid into the capillary die where the pressure is monitored with a pressure transducer. The temperature inside the bore is monitored and controlled through three zones along the barrel length. The bore geometry necessitated the use of approximately 80 g of glass to fill the total bore volume. Before the start of every run, the glass sample was loaded into the bore that was pre-heated to a temperature corresponding to a viscosity of approximately 1000 ± 100 Pa·s for the composition. These temperatures were obtained from a previous literature report of the temperature dependence of viscosity of $\text{As}_x\text{Se}_{1-x}$ liquids [21]. Following thermal equilibration, the piston was advanced to apply a pressure of 1 MPa onto the sample for 3 min to let the trapped air escape the melt column. For each measurement, the piston was advanced at a constant speed corresponding to the desired shear rate while simultaneously monitoring the pressure. The pressure was recorded as a function of time until attainment of equilibrium was ensured. Shear rates were varied over a range of 50 s^{-1} to up to 12000 s^{-1} . Data were collected both as a function of increasing and decreasing shear rate to ensure reversibility.

As mentioned above, the Rabinowitsch correction rectifies the difference between the apparent shear rate and the shear rate at the bore wall, due to the non-parabolic velocity profile associated with the non-Newtonian behavior. The magnitude of this correction was found to be negligible for the present experiments. On the other hand, the Bagley correction is often used to compensate for the pressure drop between the entrance and the exit of the die. This correction requires a twin-bore measurement where two pistons push the same liquid out of two bores through two dies of different lengths simultaneously at the same shear rate. Such a measurement was carried out to estimate the magnitude of the Bagley correction for the present experiments. The two dies used for this measurement were of same diameter of 1 mm and an entrance angle of 90° but with different capillary lengths of 16 mm and 0.25 mm, respectively. This correction uniformly lowered the measured viscosities by ~48 Pa·s on an average, over the entire range of shear rates used in the present study and does not have a significant effect on the shape of the viscosity vs. shear rate curve. Therefore, in this work we present the data acquired by single-bore measurement without the Bagley correction.

3. Results and Discussion

The shear rate dependence of viscosity for all glass compositions is shown on a log-log plot in Fig. 1. It is clear that for every composition, at the temperature of measurement, the viscosity remains nearly independent of shear rate $\dot{\gamma}$ at low shear rates $< 1000 \text{ s}^{-1}$. This behavior is characteristic of a Newtonian liquid. Further increase in $\dot{\gamma}$ beyond 1000 s^{-1} results in a steady decrease of viscosity indicating the onset of non-Newtonian behavior in the form of shear thinning. In the non-Newtonian region, the logarithm of viscosity decreases linearly with the logarithm of shear rate, thus, implying a power-law behavior where η decreases with $\dot{\gamma}$ as η

$\sim \dot{\gamma}^{-n}$ with $n \leq 1$ (Fig. 1). Such power-law behavior has previously been reported in the literature for shear thinning in polymer melts and solutions as well as for oxide and metallic glass-forming liquids where n typically varies between 0.75 and 1 [13, 14, 22, 23]. The compositional variation of the power-law exponent n for all $\text{As}_x\text{Se}_{1-x}$ compositions was obtained from linear least squares fitting of the non-Newtonian region in Fig. 1. The n values fall around 0.90 ± 0.04 , irrespective of glass composition. When taken together, these results indicate that both the onset and the degree of the non-Newtonian behavior are independent of chemical composition as well as of any corresponding variation in the atomic structure and topology. This behavior is fully borne out in Fig. 2 where the η vs. $\dot{\gamma}$ data in Fig. 1 are plotted as normalized quantities. The η values are normalized to the corresponding Newtonian viscosity η_0 while $\dot{\gamma}$ is normalized by multiplying with the average shear relaxation time τ , given by the Maxwell relation $\tau = \frac{\eta_0}{G}$ where η_0 is the Newtonian viscosity and G is the infinite (high) frequency shear modulus. The η_0 can be obtained from the low shear rate value of the viscosity in Fig. 1 and the G values are obtained from the literature [24]. The dimensionless product $\tau\dot{\gamma}$ is commonly known in the theory of viscoelasticity as the Weissenberg number (denoted henceforth, as W). It is clear from the η/η_0 vs. W plot in Fig. 2 that the η vs. $\dot{\gamma}$ data for all $\text{As}_x\text{Se}_{1-x}$ liquids collapse onto a single master curve that can be described well by the semi-empirical relationship [13, 25, 26]:

$$\frac{\eta}{\eta_0} = \frac{1}{1 + \left(\frac{W}{\alpha}\right)^\delta} \quad (3)$$

In this expression $\alpha = \sigma_y/G$, where σ_y is the tensile strength of the liquid and δ is a numerical constant (typically $\delta \leq 1$). A least squares fit of Eqn.3 to the data in Fig. 2 yields $\alpha = 3.5 \times 10^{-4}$ and $\delta = 1$.

This remarkable universality in the non-Newtonian behavior of $\text{As}_x\text{Se}_{1-x}$ liquids across the wide composition range is rather intriguing as it demonstrates that non-Newtonian behavior of chalcogenide liquids is independent of atomic structure, connectivity, topology and of the extent of non-exponential, non-Arrhenius or non-linear relaxation behaviors that are the hallmark of glass-forming liquids. It also provides excellent predictive power as the knowledge of η_0 and G at any temperature is sufficient to predict the viscosity of a liquid at any shear rate if the master curve for the corresponding class of liquids is known. It will be interesting in this regard to test whether the same master curve can describe the non-Newtonian behavior of chalcogenide liquids in other glass-forming systems such as Ge-P-Se and the sulfides and tellurides. An important attribute of the master curve in Fig. 3 is the Weissenberg number W for the onset of the non-Newtonian behavior which is $\sim 10^{-4}$ for the $\text{As}_x\text{Se}_{1-x}$ liquids. Considering $W = \tau\dot{\gamma}$, an onset value of $W \sim 10^{-4}$ implies that the shear rate at which the liquid becomes non-Newtonian is significantly slower than the rate of structural relaxation i.e. $\dot{\gamma} \ll [\tau]^{-1}$. A similarly low onset value of $W \sim 10^{-3}$ was also reported by Simmons and coworkers for the soda-lime-silica glass system while, for polymers $W \sim 1$ [14, 22]. Such a large difference in the onset value of W between inorganic and polymeric glass-forming liquids warrants further study but it is tempting to suggest that the atomistic origin of this difference is likely associated with the corresponding difference between the fundamental flow mechanisms between these two classes of liquids.

The viscosity of $\text{As}_{30}\text{Se}_{70}$ liquid was measured as a function of shear rate at two different temperatures of 292°C and 261°C corresponding to Newtonian viscosities of 790 and 2250 Pa·s, respectively, to examine the effect of temperature on the non-Newtonian behavior. The results are compared on a normalized η/η_0 vs. W plot in Fig. 4. Again, the viscosity vs. shear rate behavior at both temperatures can be described by one master curve, indicating that the

universality in the non-Newtonian behavior of the $\text{As}_x\text{Se}_{1-x}$ system remains unaffected by temperature. Similar independence of the non-Newtonian behavior on temperature has also been reported in the literature for phosphate glass-forming liquids, over a wide temperature range corresponding to viscosities ranging between 10^5 and 10^9 Pa.s [27].

It is important to clarify here that the shear rate in capillary rheometry is controlled through application of pressure. Since pressure alone may affect viscosity, the rheometry data reflect the combined effects of both pressure and shear rate on viscosity. Therefore, separate measurements of isostatic pressure dependence of viscosity are necessary to estimate the relative importance of these two controlling factors on viscosity. Such measurements on chalcogenide liquids, though rare in the literature, have been reported for liquid Se by Harrison [28] and for As-sulfide liquids by Brazhkin and coworkers [29,30]. These reports indicate that the effect of pressure on the viscosity of liquid Se is negligible for pressures of at least up to 1 GPa [29-31]. On the other extreme, for the As_2S_3 liquid the isothermal viscosity actually increases with pressure [29,30,32], unlike the observation made in the present study of lowering of viscosity with increasing shear rate. Although no data were reported for As_2Se_3 , the pressure dependence of viscosity for this liquid is likely to be similar to that for As_2S_3 . Therefore, considering the fact that for the capillary rheometry measurements reported here, the maximum applied pressure corresponding to the highest shear rate remains ≤ 100 MPa, it is safe to conclude that the observed non-Newtonian behavior is entirely dominated by shear rate and any effect of isostatic pressure is negligible.

Finally, it is interesting to note that in a previous study of the indentation creep behavior of Ge-Se glasses at ambient temperature by Guin et al. [17] it was suggested that the strain rate sensitivity of these glasses increases with increasing Se content. This observation was attributed

to the topological characteristics of chalcogenide glass structure. The structure of Se-rich glasses is dominated by long Se chains and it is likely that such structural elements are prone to shear induced alignment which may facilitate viscous flow and consequently shear thinning via chain sliding and by avoiding chain entanglement. A potential drawback of such indentation studies is that they were performed at the same temperature for all glasses and therefore, at different T/T_g and Newtonian viscosities. Furthermore, such studies were performed in the glassy state below T_g where the system is far from equilibrium and the separation of elastic and viscous response can be challenging. The scenario of composition and structure dependent strain rate sensitivity proposed in the literature on the basis of such indentation studies is in contradiction with the results obtained in the present study that show universal non-Newtonian behavior in As-Se liquids, irrespective of their chemical composition and atomic structure/topology. Although chain alignment under large stress has indeed been experimentally observed in selenide glasses with high Se content, such structural modifications may not in fact be mechanistically consequential in giving rise to the shear thinning in these liquids.

Conclusions

The viscosity of glass-forming liquids in the As_xSe_{1-x} system is measured as a function of shear rates ranging between 50 and 12000 s^{-1} , at temperatures corresponding to a Newtonian viscosity of $\sim 10^3$ Pa.s. A transition from Newtonian to Non-Newtonian shear thinning behavior was observed for all liquids near a Weissenberg number of $\sim 10^{-4}$. Viscosity data, when normalized to the Newtonian value and plotted against the Weissenberg number, display a universal pattern for all liquids, regardless of their chemical composition, structure and other

related properties. Such universality in the non-Newtonian behavior questions the validity of the conventional wisdom of shear thinning arising from topological modifications of the melt structure at high shear rates, in the form of alignment of structural units facilitating viscous flow.

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Figure 1.

Log-log plot of the shear rate dependence of viscosity for $\text{As}_x\text{Se}_{1-x}$ liquids, measured at temperatures corresponding to Newtonian viscosities close to 1000 Pa·s for each composition.

Figure 2.

Data from Fig. 1, plotted as normalized viscosity η/η_0 vs. Weissenberg number $W=\dot{\gamma}\tau$. Symbols and color scheme are the same as in Fig. 1. Solid line through the data points is the least squares fit to Eqn. 3. Vertical dashed line serves as a marker separating the Newtonian region to the left from the Non-Newtonian region to the right and corresponds to the strain rate where $\eta/\eta_0=0.5$ and $W = \alpha$ (see Eqn. 3 in text).

Figure 3.

Normalized viscosity vs. Weissenberg number plot for As₃₀Se₇₀ liquid at two different temperatures. The Newtonian viscosities at the two temperatures and corresponding symbols are shown in the inset.