Study of mechanical property of shear thickening fluid (STF) for soft body-armor

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Abstract
STFs have been widely used for soft body-armor because of STF reversible and repeatable thickening characteristics. However, shear thickening mechanism characterized by rheological properties cannot well explain thickening mechanism of STF against the penetrations and impacts. In this study, a cycle experiment of quasi-static squeeze and pull-out of STF by metal rod were carried out on the INSTRON. Effects of velocity, interface condition and boundary et al were discussed. Typical displacement-load curves show that STF thicken in the normal load when the rate reach critical value. Taking a pull-out experiment as example, force without thickening of STF is a constant value, about 1.5 N. The maximum force with thickening of STF is about 150 N, which increases 100 times due to the thickening of STF. A simple simulation of clustering behavior of nano-particles in STFs in the squeeze process is depicted. The experiment and simulation studies have proved that SiO2 nano-particles in STF will rearranged to cause a jammed zone in the normal load.

1. Introduction

In general, a material both high strength and flexibility has rarely been realized because of these two contrary properties. STF/high performance fabric composite materials have successfully conquered this difficulty [1, 2]. Shear thickening fluid (STF) is dense colloidal suspensions of micro or nano-sized particles. Liquid-harden conversion of STF when driven strongly has been observed. Moreover, the STF reverses to liquid-like state when perturbation removed. The conversion is repeatable and reversible. Rheological tests have shown that thickening of STF arises under shear stress [3–5]. However, thickening performance characterized by mechanical properties of STF under normal loading was few reported.

Over the years, a number of studies of STF mainly focuses on the preparation, rheological properties, shear thickening mechanism and application of STF. Studies demonstrated that improvement of ballistic, impact, stab and puncture resistant properties of STF treated textile materials are available compared with untreated fabrics [6–8]. Recently, interests have been shown in the preparation of high performance STF and its rheological properties, so as to realize tuning shear thickening behavior of STF and further extend the application of STF. J. H. Ge [9] added silicon carbide nanowires to silica based STF, and the rheological properties showed that the initial and maximum viscosity are significantly improved due to addition of SiC nanowires, at the same time, the viscosity of STF is enhanced with increasing of the content of SiC nanowires. Sahoo et al [10] reported that the ability to tune the shear thickening behavior of silica through synthesis of an organically modified silica. The shear-thickening fluids (STFs) prepared using organically modified and non-functionalized silica were further applied on Kevlar fabric in order to study yarn pull-out and impact properties of STF impregnated fabric. Liu et al [11] investigated that the influences of rheological property on the impact performance of Kevlar fabrics impregnated with SiO2/PEG shear thickening fluid. The results that the critical shear rate is 0.6–3.2 s−1 for a suspension system composed of 650 nm silica particles, whereas the critical shear rate is 169–627 s−1 for that of
100 nm particles. Impact test results of STF–Kevlar and neat Kevlar fabrics show that the STF made of 100 nm SiO2 improved the anti-impact performance of the Kevlar fabric and increased the energy absorption up to 56.6%, whereas it decreased in the treatment by STFs made of 650 nm SiO2. The different trends of the two suspension systems are attributed to the specific value of shear rate with critical shear rate. J. B. Qin et al [12] prepared a novel shear thickening fluid (STF) composed of silica microsphere and ionic liquids (ILs). Rheological results indicate that this STF presents a unique double continuous shear thickening behavior and good conductivity.

There are three main working modes of STF in the personal protective materials: shear, squeeze and tension flow in which squeeze and tension flow are typical normal deformation [13]. The traditional rheological test of STF can only characterize shear thickening property of STF. Now, tensile and extrusion rheometer have been developed to study the rheological properties of STF under normal loading. White et al [14] reported relationship between tensile rate and tensile viscosity by tensile rheometer. Test results were similar compared with traditional shear rheometer. STF begins to harden from Newton behavior with increase of tensile rate. When viscosity reaches the maximum, STF appear brittle fracture. Chen Qian [15] studied the rheological properties of STF in squeeze mode, and investigated viscosities of STF with different parameters (squeeze speed, concentration of dispersive phase and roughness of contact surface). Moreover, some scholars have also done many attempts to study the mechanical properties of STF under normal loading. An adult jumps into the corn-starch pool quickly without sinking. For this phenomenon, Waitukaitis et al [16] captured the dynamic process of metal rod impacting cornstarch–water suspension using high-speed photography, embedded force sensor and X-ray imaging technology. They developed a model for the impact-generated solidification originated from initially transforms compressible particles into a growing jammed region. Waitukaitis et al confirmed that impact behavior is few related to steady-state shear, but to transient mechanical response. Lim et al [17] investigated high-velocity impact resistant property of STF by using the split Hopkinson bar. Experimental results show that liquid-solid transition of STF is very obvious in the impact process. In addition to the steady-state and low-frequency dynamic shear effects, STF also shows thickening phenomena when subjected to transient normal stress effects. The excellent impact resistance of STF is also one of reasons for its long-term attention. Fu et al examines the rheological, impact resistance and energy absorption characteristics of an STF with concentrated polymer submicron particles under various temperatures. The study shows that the impact resistance of the STF increases with the decrease in temperature. In particular, the impact resistance of STF shows an early response time when the impact resistance slowly picks.

Although different methods and theories have been presented regarding the thickening property and mechanism of STF with applied stress. There is still no clear whether the dominant mechanism of energy absorption of STF/fabric is thickening response or friction enhancement. In this paper, both quasi-static squeeze and pull-out cycle experiments of STF with metal rod are tested in order to explore thickening properties of STFs under normal stress and to determine effect of velocity, interface condition and boundary of STF. Morphologies of STF in the squeeze process of the rod are also depicted by means of the Jamming phenomenon in condensed-state physics. Moreover, it is postulated that SiO2 nano-particles in STF will rearranged to cause a jammed zone. This paper, probably for the first time, attempts to use a new approach to methodically investigate the thickening of STF under normal loading.

2. Experimental section

2.1. Materials

To prepare STFs, spherical silica particles with 100 ~ 650 nm (Shenzhen nano Port Co., Ltd.) and 1:2 mixed solution of PEG 200 and PEG 400 (Sinopharm Chemical Reagent Co., Ltd.) were used. The STFs was prepared by dispersion 75 wt% silica particles in PEG. Micro-morphology of STFs was observed by SEM. Different sizes of spherical SiO2 particles were uniformly dispersed in PEG solution, shown in figure 1. Steady-state Rheological property of STFs was performed using Anton-paar MCR301 rheometer with the shear rate range 0.1–1000 s⁻¹. The shear rate and viscosity curve of STFs was obtained as shown in figure 2. It can be seen that viscosities of the STFs undergo three stages under shear stress. Slight shear-thinning behavior of STFs first occurs, and then increases with an increase of shear rate. In particular, as the shear rate reaches a critical value, the viscosity of the STF abruptly increases which marks shear thickening phenomenon. However, viscosity of STFs is a sharply decrease in again after a period of shear thickening. The threshold value for STFs is about 1 s⁻¹. Protective materials with STFs applied in the range of shear thickening of STFs [18].

2.2. Experiments

Quasi-static squeeze and pull-out tests of STFs which can describe altering of force is performed on self-reformed INSTRON mechanical testing instrument and self-designed accessories, as shown in figure 3. Down
gripper was replaced by work platform mounted on pedestal of INSTRON by screws which can place large container with special adhesive tape. Figure 3(b) is schematics of stainless-steel rod which was mounted in the upper grip of INSTRON in the experiment. Two sets of experiments were performed for STFs. Firstly, STFs were poured into a big container and stand still for several hours. The INSTRON was set into compression. The rod was then pushed into the STF at a set speed to a total displacement of 50 mm. The point of zero displacement is defined as less than 1 mm gap of rod and surface of STF. The compression and the stretching test in this experiment is a complete cycle with the same velocity. When compression test end, STFs remained motionless for several hours until STF recovered to stable equilibrium. The instrument was reset to stretching mode, and rod was pulled out from STFs until the rod is completely out of the liquid level of STF. Load versus displacement data were recorded using a 1 KN load cell, as shown in figure (4).

Influence of contact area was investigated by altering the cross section of the rod (diameter of 20 mm, 40 mm and 60 mm). Moreover, the influence of surface roughness of the rod was considered. Plastic sandpapers of different mesh were pasted around and bottom of rod, as shown in figure 3(c). To understand the role of
boundaries, we changed the suspension depth (H). STF is a rate-dependent material, so squeeze and pull-out speed was a key parameter. Velocity range was widely set. The minimum speed is 10 mm min$^{-1}$ and the maximum speed is 500 mm min$^{-1}$. The full parameters and variation of experiments is given in Table 1.

### Table 1. Experimental parameters and variation.

| Parameters                  | Variation |
|-----------------------------|-----------|
| Velocities (mm min$^{-1}$)   | 10  20  60 100  150  300  500 |
| Contact area (D mm$^{-1}$)   | 20 40 60 |
| Suspension depth (H)         | 35 55 75 |
| Contact surface roughness (mesh) | No 1500 4000 |

3. Results and discussion

3.1. Typical Load-displacement curves

The typical force-displacement curves for STFs are shown in figure 5. Assuming hydrostatic pressure is excluded, for the extrusion process of STFs [figure 5 (a)], The shape of the plot was divided into three distinct zones namely, free diffusion phase (zone I), viscous flow phase (zone II), thickening phase (zone III). Force of zone I was found to be almost zero and the reason is that SiO$_2$ particles in the STFs surrounded by liquid film of dispersing medium freely diffuse when the bar was slowly disturbed downward. There are no contact between the nano-particles. So, the force of STF is very small. It is also noted from the figure that the zone II is various as compared to the zone I. The zone II is characterized by no-linear growth value of force, which is most probably caused by increased contact frequency of SiO$_2$ particles under the rod with space between the rod and the container reduced due to the drop of the rod. The zone III is characterized by a sharp rise in force which means thickening of STF due to SiO$_2$ particles are condensed into blocks.

Figure 5(b) is load-displacement curve measured during the quasi-static pull-out process of the rod from STFs. The plot also has three distinct zones. The first zone was found to be almost linear and there is substantial gain in force. It is assumed that SiO$_2$ particles contact each other to form the blocks due to STF suddenly stressed from stable state, when the rod penetrates. It means thickening of STF. the pull-out force is large and the slope of this zone is quite steep, which can be defined as the pull-out modulus of STF. The characteristic of the second
zone is similar to zone II of figure 4(a). This zone is also called as viscous flow phase. SiO₂ particles frequently contact around the bar as the bar move upward. Force keeps on increasing and the peak force generation occurs invariably in this zone. Zone III is characterized by a drop in force which means the thickening of STF gradually disappears with free diffusion of particles, and STF adhering to the rod also gradually fall off.

It is important to note that STF diffuse around the rod during the test, including lateral and longitudinal diffusion. When STF flows upward along the sides of the rod, lateral space is increased which can result in the decrease of the contact force of the SiO₂ particles. On the contrary, the longitudinal space under the rod becomes smaller and smaller, and the solid content of SiO₂ particles in STF becomes larger and larger. The particles can frequently contact with each other. So, the longitudinal diffusion of STF plays a dominant role in the extrusion process. Further, two typical graphs revealed that thickening of STF occurs in the quasi-static extrusion and pulling-out process at a critical rate and a in the figure 4 is the starting point of thickening of STF. There is a little difference of stress of STF at point a in the two test cycles due to the stability of STF before the pull-out test.

3.2. Velocity effect
An investigation of the variation of the mechanical properties of STF as a function of velocity was performed on the INSTRON. A narrower zone I with the velocity is observed as shown in figure 5. The result means that STF appears thickening earlier with the velocity. Using INSTRON to test the extrusion flow behavior of non-Newtonian fluid, which cannot accurately obtain the velocity distribution in the dispersion system. STF is a rate sensitive material where the extrusion rate (τ) can be defined that is the ratio of moving speed of the rod (v) to the distance of the rod and the bottom of the container (s) [15]. It can be expressed as:

\[ \tau = \frac{v}{s} \] (1)

From figure 6 and equation (1), when the rod has higher moving velocity and the distance between rod and bottom of container becomes smaller and smaller as the rod moves downward. Extrusion rate is increasing. Furthermore, the solid content in the local region of STF under the rod is also increasing with rod moving downward. The particles are close to each other and contact frequently. So sharp increasing value of extrusion force is observed, which means earlier thickening of STF under normal loading.

3.3. Boundary effect
For a fixed volume container, the bottom boundary is caused by the suspension depth. Figure 7 (a) presents the force generated against displacement of the rod in STF at 55 mm and 75 mm respectively when the moving velocity of the rod is 150 mm min⁻¹. It is noted from the figure that for the smallest H, thickening of STF is earlier formed, resulting from a higher extrusion rate. To verify this, we simultaneously measured the force generated in STF at 35 mm, 55 mm and 75 mm respectively when the moving velocity of the rod is 300 mm min⁻¹ as shown in figure 7 (b). It is also evident that the result is similar to that of 150 mm min⁻¹ which suggests the importance of suspension depth on the mechanical property of STF under normal loading. This has some important implications: first, when lowering the suspension depth, nano-particles of STF under the rod have sharp motion due to higher extrusion rate. Second, for lower depth, solid content of STF under the rod increase. This means that particles contact each other frequently in smaller space and particle agglomerate.
3.4. Effect of interface condition

To verify the importance of interface condition, we first investigate the effect of contact area on the mechanical property of STF (Figure 8(a)). It is evident that the larger cross-section of the rod shows a faster rise in force (narrower zone I) during the penetration process of the rod, which means earlier thickening of STF. Especially when the cross section of the rod is increased to 60 mm, it is observed that this rise is sudden (narrower Zone I fall to 1 mm) and later force shows increase linearly which has a steep slope. These results can be attributed to thickening of STF, which mainly occurs in the distinct under the rod. So, larger contact area will lead to thickening zone of STF increase.

The effect of interface condition was also examined with the surface roughness of the rod. Figure 8(b) presents a significant increase of the resistance to penetration by a smooth rod and a narrower zone I. This result can be attributed to the wet friction between nano-particles and rod. Chen et al. [19] presents that the slipping of chain structure of nano-particles is mainly near the rod, while the rough surface of the rod modified by sandpaper restricts the movement of nano-particles near the rod which hinders the formation of particle chain.

3.5. Mechanical properties of STFs in the pull-out mode

The global mechanical properties of STF were investigated by squeeze and pull-out test. It is seen from 3.1 that the thickening of STF mainly occurs in the first zone and the peak force generation occurs invariably in Zone II. For better understanding, pull-out modulus and peak force characterize mechanical properties of STF in the pull-out process. Figure 8 displays the variation of the applied force as a function of rod displacement for STF at

![Figure 6](image6.png)

**Figure 6.** Velocity dependent behaviors of the STFs.

![Figure 7](image7.png)

(a) 150 mm/min  (b) 300 mm/min

**Figure 7.** Force versus displacement curves for STFs due to the role of boundary by changing the suspension depth.
different pull-out velocities. It is noted from figure 9 (a)-(c) that STF does no produce thickening and there is almost no force generated, when pull-out velocities are 6 mm min$^{-1}$, 20 mm min$^{-1}$ and 60 mm min$^{-1}$ separately. However, when velocity reach 100 mm min$^{-1}$, force of zone I in figure 9(d) keeps sharply increasing and there is steep slope. A comparison between graphs at 300 mm min$^{-1}$ and 500 mm min$^{-1}$ revealed that pull-out modulus of STF had slightly increasing and the peak force almost no changed at 500 mm min$^{-1}$ as compared to that of 300 mm min$^{-1}$, which means that STF had been completely thicken at 300 mm min$^{-1}$. Figure 10 presents the peak force of STF in the pull-out process with different velocities. The peak pull-out force of STF without thickening is a constant value, about 1.5 N when pull-out velocity does not reach the critical rate of STF. However, the maximum drawing force of STF is about 140 N after complete thickening of STF. The drawing force increases nearly 100 times. It is pertinent to mention here that for a definite dispersion system of STF, the thickening of STF in the pulling-out process has the following characteristics: the starting point of thickening of STF namely the critical rate and the maximum thickening degree namely the peak force, which has a good guide role for our practical application.

In this work, mechanical properties of STFs are investigated via quasi-static squeeze and pull-out circle test. To provide further insights into the thickening characteristic of STF under normal load, photos and simulation of thickening behavior of STF in the extrusion process are presented, as clearly shown in figure 11. It is noted that
**Figure 10.** The peak force of STF in the pulling-out with different velocities.

**Figure 11.** Photos and simulation of thickening behavior of STF in the extrusion process.
the rod pushes into STF, causing a growing depression, which simply represented as depressed depth (h) and radius of liquid level depression (r). h, r also increases at a certain speed. The liquid-solid transformation of STF is not caused by chemical reaction, but is physical process of particles clustering in STF under external load. This phase transition is similar to the jamming phenomenon in the condensed-state physics [16]. In order to understand the clustering behavior of circle particles in STFs, a simple simulation of the rearrangement of SiO₂ nano-particles in STF was presented according to contact force model. The particles are in the lubricated state in the lower rate, and each particle is separated by the dispersion medium film [20]. Particles freely move without friction and contact. As rate increase, SiO₂ particles move sharply and the probability of contact between particles increases. Then, the liquid film is destroyed. The adhesive force and friction force of particles promote them to condense, and jamming phenomenon is formed [21, 22], also note that the consistency of STF can change locally while the rod continues to descend. It is easier to condense between particles, so, a developing solid due to jamming phenomenon is extending forward, where the solid growth driven by the rod’s motion is established [23, 24]. However, A more systematic study on quantification and multi-scale in liquid-solid transition of global system of STF under normal loading is ongoing.

4. Conclusions

The present study proves thickening behavior characterized by mechanical properties of STF under normal loading. To universally explore thickening property of STF, a series of quasi-static extrusion and pull-out tests, including effect of velocity, boundary (Suspension depth) and interface (Contact area and surface roughness) et al were performed. The results demonstrate that thickening phenomenon of STF happen under normal extrusion and pull-out load when rate reach a critical value. Moreover, it is found that thickening of STF occurs earlier, when descent speed of the rod is higher, which is observed by narrower zone I in force-displacement curve. The suspension depth was selected as boundary condition. For lower suspension depth, thickening behavior of STF is earlier and more obvious. Thickening characteristics of STF were observed at different interface conditions, which revealed that mechanical property of STF is nearly linear with larger cross area of the rod; and the roughness of the rod surface has a slight effect on the thickening behavior of STF. Extrusion and pulling-out is a complete cyclic process. The thickening of STF during pulling-out is mainly in the first stage. Taking pull-out velocity as example, the force without thickening of STF is a constant value, about 1.5 N. The maximum force with thickening of STF is about 150 N, which increases 100 times due to the thickening of STF. This leads to a very important conclusion that mechanical property of STF is greatly improved due to the thickening of STF. Finally, a simple simulation of clustering behavior of nano-particles in STFs in the squeeze process is depicted by means of the Jamming phenomenon in condensed-state physics. Moreover, the experimental and simulation studies have proved that SiO₂ nano-particles in STF will rearranged to cause a jammed zone. These results also show that STF thicken in the normal load when the rate reach critical value, and thickening mechanics of STF can be interpreted by a growing jamming. More research works will be required to confirm the extent and role of thickening of STF during these loading events in future in order to apply in the field of flexible mechanical protective materials.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

Declaration of Competing Interest

None.

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