# **RHEOLOGY OF BALLISTIC CLAY: THE EFFECT OF TEMPERATURE AND** SHEAR HISTORY

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# Abstract

Ballistic clay is used as a backing material for standards-based ballistic resistance tests for the purposes of providing a measure of the energy transferred to the body when a threat is defeated. However, this material exhibits complex thermomechanical behavior under actual usage conditions. In this work, we characterize rheological properties of the standard backing clay material, Roma Plastilina No. 1, used for body armor testing, using a rubber process analyzer. Test methods employed include oscillatory strain sweep, frequency sweep, and oscillatory strain ramp. The results show that the material is highly nonlinear, thermorheologically complex, and thixotropic. The modulus decreases under dynamic deformation and partially recovers when the deformation is discontinued. Experimental protocols developed in this study can be applied for the characterization of other synthetic clay systems.

#### Introduction

An oil-based modeling clay, Roma Plastilina No.  $1^{1}$ (RP1), was chosen by both the National Institute of Justice (NIJ) and the Department of Defense (DoD) as the standard backing material for body armor testing since the 1970s [1-3]. Body armor is tested for both penetration and deformation effects (the backface signature) by placing the armor against a backing material, also known as a ballistic witness material (BWM). Therefore, the mechanical and rheological properties of the backing material play a critical role in certification and testing of body armor because most standards allow a maximum indentation depth of 44 mm behind the armor after testing. Unfortunately, over the decades since RP1 was adopted as the standard, changes have been made to the RP1 formulation by the clay manufacturer. Newer versions of RP1 are stiffer at room temperature than the original RP1 selected by the NIJ and the DoD. Ballistics practitioners and researchers must now thermally treat the material prior to use in order to meet clay validation specifications

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originally developed in the 1970s. Therefore, the ballistic testing community has begun to look for an alternative ballistic witness material to replace RP1 [4,5].

Along with the additional thermal treatment step that makes the verification process of RP1 for ballistic evaluation more cumbersome, another important reason that requires an alternative backing material is the complex rheological behavior of RP1. Specifically, because of its multiphase formulation, the rheological properties of clay are known to be nonlinear in nature, and to depend on work and thermal history, temperature, and time [6-8]. For example, an intuitive observation is that the clay becomes softer after it is worked by hand and becomes harder over time during storage or while it is not used. Such time- and shear-history-dependent complexity is termed as thixotropy. Characterization of a thixotropic material is nontrivial because of the influence of different factors, i.e., time, temperature, and shear history, are coupled in the resulting rheological response. In spite of this, very limited rheological studies have been conducted on RP1 and other clay-like materials. Therefore, the challenges of using BWM are to 1) develop a comprehensive understanding of the rheological properties of the backing material and 2) identify characteristic responses which can be related to backface deformation during the armor ballistic testing.

In this work, we perform rheological studies on the RP1 ballistic clay using a rubber process analyzer (RPA). The RPA is essentially a strain-controlled rotational shear rheometer for rubber testing. The first advantage of the RPA is that is offers a higher torque range for solid materials like clav than that of a commercial rheometer. so that information under larger deformation can be assessed. Second, it provides a consistent sample loading procedure, as ensured by the pneumatic pressure system together with the automatic gap closure, such that the loading effects from sample to sample are minimized.

## **Materials**

RP1 clav was used as received from the manufacturer. The clay bricks from the manufacturer were cut into 70 mm-thick square blocks using a stiff blade. Each sample weighs approximately 5 g to ensure consistency. The sample was placed between two sheets of polyester films and loaded onto the lower die of the RPA for measurements.

<sup>1</sup> The full description of the procedures used in this paper requires the identification of certain commercial products and their suppliers. The inclusion of such information should in no way be construed as indicating that such products or suppliers are endorsed by NIST or are recommended by NIST or that they are necessarily the best materials, instruments, software or suppliers for the purposes described.

## Experimental

Rheological experiments were performed using a rubber process analyzer (RPA). The RPA is equipped with radial serrated bi-cone shape platens with a fixed gap of 0.48 mm. Dynamic strain sweep experiments were performed at 1 Hz from  $0.005^{\circ}$  strain (equivalent to  $0.07^{\circ}$ % strain) to 50° strain (equivalent to 697 % strain) at four temperatures of 25 °C, 30 °C, 40 °C, and 50 °C. A fresh sample was used for each test and was loaded at the test temperature. The sample was held for 30 min to allow thermal equilibrium before each run.

Frequency sweep experiments were performed at  $0.01^{\circ}$  strain (equivalent to 0.14 % strain) and 0.5° strain (equivalent to 7 % strain) from 0.05 Hz to 50 Hz at four temperatures of 25 °C, 30 °C, 40 °C, and 50 °C. To ensure that no additional loading effects or thermal history influence the measurement, the same loading procedure was applied, which is using a fresh sample for each run, loading the sample at the test temperature, and allowing the sample for thermally equilibrate for 30 min prior to measurement.

The breakdown and recovery experiments were performed at 25 °C and 1 Hz using two shear histories. The first consists of increasing the oscillatory shear strain stepwise from 0.01° (equivalent to 0.14 % strain) to 10° (equivalent to 14 % strain) and then decreasing the strain from 10° to 0.01°. The second experiment is to use two shear strain amplitudes of 0.01° and 1° alternatively.

#### Results

The dynamic shear storage (G') and loss (G") moduli as a function of strain amplitude at various temperatures for RP1 are shown on a double logarithmic plot in Figure 1. At small strains, the moduli show seemingly linear behavior as the storage modulus barely exhibits a plateau. An examination of the relative intensity of the third harmonic magnitude  $I_3/I_1$  from Fourier analysis of the oscillatory stress waveform (results not shown) show that the strain corresponding to  $I_3/I_1 < 3$  % (linear range) is within 0.3 % for all temperatures, which is consistent with the linear range reported by Seppala et al. [7] using a rheometer. As strain increases, both G' and G" decrease. The modulus values are lower at higher temperature, and the curves show similar shape at different temperatures.

The complex shear modulus  $G^*$ , obtained as  $|G^*| = \sqrt{G'^2 + G''^2}$ , is normalized by the complex modulus magnitude at 0.007 % strain for each temperature and plotted as a function of strain amplitude in Figure 2. The modulus curves at different temperature nominally collapse into a single curve for the temperatures examined, indicating similar strain dependence. This



Figure 1. Dynamic shear storage modulus (G', left panel) and loss modulus (G'', right panel) as a function of strain amplitude for RP1 at various temperatures ranging from 25 °C to 50 °C. Standard uncertainty in modulus values associated with this technique is approximately 10 %.



Figure 2. Reduced  $|G^*|$  as a function of strain amplitude for RP1 at various temperatures ranging from 25 °C to 50 °C. Standard uncertainty in modulus values associated with this technique is approximately 10 %.

result also suggests that in the measured temperature range, there is no significant thermal transition or structural change that affects the strain dependence of the mechanical behavior.

The results of frequency sweep experiments measured at 0.14 % strain and 7 % strain are shown in Figure 3 and Figure 4, respectively. As expected, the



Figure 3. Dynamic shear storage modulus (G', left panel) and loss modulus (G'', right panel) as a function of frequency for RP1 measured at a strain of 0.14 % for various temperatures ranging from 25 °C to 50 °C. Standard uncertainty in modulus values associated with this technique is approximately 10 %.



Figure 4. Dynamic shear storage modulus (G', left panel) and loss modulus (G'', right panel) as a function of frequency for RP1 measured at a strain of 7 % for various temperatures ranging from 25 °C to 50 °C. Standard uncertainty in modulus values associated with this technique is approximately 10 %.

modulus decreases as temperature increases, and the responses show similar trends at different temperatures. When small deformation is applied (Figure 3), as frequency decreases, G' decreases with a rapid drop first followed by a more moderate decrease, while G'' shows a reduction followed by a plateau. When a large strain of 7 % is applied, the storage and loss moduli share a similar



Figure 5. Reduced  $|\eta^*|$  as a function of frequency for RP1 measured at strain amplitudes of 0.14 % and 7 % for various temperatures ranging from 25 °C to 50 °C. The arrows show data measured at the corresponding strain. Standard uncertainty in viscosity values associated with this technique is approximately 10 %.



Figure 6. van-Gurp Palmen plot [9] for RP1. The phase angle ( $\delta$ ) in degree is plotted as a function of complex modulus ( $G^*$ ). Standard uncertainty in modulus values associated with this technique is approximately 10 %.

trend, i.e., decrease followed by a plateau, as frequency decreases. To better compare the frequency dependence, the normalized complex viscosity  $\eta^*$ , which is defined as  $|\eta^*| = \sqrt{(G'/\omega)^2 + (G''/\omega)^2}$ , is plotted in Figure 5 for data measured at those two strain amplitudes. As shown in the figure, the complex viscosity decreases as

frequency increases. At a fixed strain, the frequency dependence is the same for all temperatures; however, the complex viscosity shows a greater frequency dependence at higher strain amplitude. This finding suggests that a thorough investigation of the clay behavior requires information over a broad range of both strain amplitude and frequency, because the effects of both factors are involved in the direct impact on the BWM during the verification process of the BWM and ballistic testing.

way to evaluate if a material One is thermorheologically simple or complex is by examining the van Gurp-Palmen plot [9], which is plotting the phase angle ( $\delta$ ) against the corresponding complex shear modulus  $G^*$ . For a thermorheologically simple material, within the linear viscoelastic range, the time-temperature superposition (TTS) technique can be used to describe the viscoelastic behavior of the material over a wide range of times or frequencies by shifting the data obtained at different temperatures to a reference temperature [10]. As shown in Figure 6, even for the data obtained at 0.14 %, which is considered to be within the linear region, the curves at different temperature cannot superpose. This result indicates that the RP1 clay is a thermorheologically complex material and TTS does not apply.

Figure 7 shows the results of breakdown and recovery experiments by applying sequential continuous oscillatory shear steps. In the upper figure of Figure 7 where a ramp-up in the shear strain followed by a rampdown in the shear strain, a breakdown in the complex modulus was observed during the first four steps  $(0.01^{\circ} \rightarrow 0.1^{\circ} \rightarrow 1^{\circ} \rightarrow 10^{\circ})$ . When a large strain followed by a small strain is applied  $(10^\circ \rightarrow 1^\circ)$ , the modulus jumps to a higher initial value; however, the modulus continues to decrease during continuous shearing in this step. Moreover, the modulus values for the two pink curves in Figure 7 do not match, indicating that the rheological properties are strongly dependent on previous shear history. Upon further decreasing the strain to 0.1° and to 0.01°, a recovery in the complex modulus is observed. However, the final modulus value dose not reach the initial value, suggesting that some structure in the material has been disrupted and did not recover to the original state within the time of the measurement. Using an alternating oscillatory shear strain of 1° and 0.01°, as shown in the lower figure of Figure 7, an alternating breakdown and recovery in the complex modulus is observed. Strong thixotropy is detected as shown by the evolving modulus during each step. Again, the modulus value is not completely recovered after the first breakdown step.



Figure 7. Complex shear modulus  $G^*$  as a function of time for the breakdown and recovery experiments. The sequence of applied oscillatory shear strain is from top to down as indicated in the figure. The color of each curve corresponds to the responses obtained from the corresponding strain amplitude is applied. Standard uncertainty in modulus values associated with this technique is approximately 10 %.

#### Conclusions

In the present work, we studied the effects of temperature and shear history on rheological properties of Roma Plastilina No. 1 clay using a rubber process analyzer. The results show that the clay is a highly nonlinear, thermorheological complex, and thixotropic material. The modulus decreases under dynamic deformation and partially recovers when the deformation is discontinued. Experimental protocols developed in this study are expected to be applied for the characterization of other synthetic clay systems. The continuing clay research at NIST is anticipated to provide fundamental information on the current standard backing material, to extract suitable material parameters that govern the material performance in actual usage conditions, and to help to guide the development of replacement materials.

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