



## **A Study on Creep Behavior of Composite Solid Propellants Using the Kelvin-Voigt Model**

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**Abstract:** A Kelvin-Voigt model consisting of a spring and a dashpot in parallel was applied for the viscoelastic characterization of solid rocket propellants. Suitable values of spring constants and damping coefficients were employed by a least squares fit of the errors to generate creep curves using a Dynamic Mechanical Analyzer (DMA) for composite solid propellants. Three different composite propellant formulations based on HTPB/AP/Al having burning rates of 5 mm/s, 15 mm/s and 20 mm/s were tested under different stress levels varying from 0.1 MPa to 3 MPa and at different temperatures varying from 35 °C to 85 °C. Creep behavior with recovery was studied and analyzed to evaluate the viscoelastic properties. The change in spring constants, representing elastic deformation, was very small compared to the damping coefficients for the propellants studied. For a typical propellant formulation, when the stress level was increased, the spring and damping coefficient both increased significantly whereas for an increase in temperature, they remained nearly constant. However, the ratio  $E/\eta$  was observed to be constant and independent of stress level. It was also observed that the variation of  $E$  and  $\eta$  varied linearly with increase in stress whereas their ratio showed a logarithmic variation. A mathematical correlation was developed to evaluate the viscoelastic properties during creep of composite propellants.

**Keywords:** mechanical properties, viscoelasticity, propellant, creep, DMA

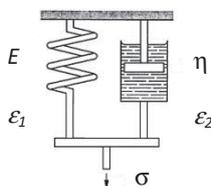
### **1 Introduction**

Solid rocket propellants are most commonly used in missiles and rockets to deliver the desired thrust. Composite solid rocket propellants are generally formulated

by dispersing a solid oxidizer such as ammonium perchlorate (AP) (65-80%) and a metallic fuel such as aluminum powder (0-18%) in a liquid binder such as hydroxyl terminated polybutadiene (HTPB) (10-15%) using certain processing aids [1]. Basically, the term 'binder' represents a multi-component system consisting of prepolymer, curing agent, cure catalyst, plasticizer, bonding agent and antioxidant. Each of the components contributes something to the final binder characteristics. Once cured, the binder makes the composite propellant flexible, which decreases the likelihood that the propellant will fracture under stress and pressure. The mechanical properties of solid rocket propellants are very important and must possess sufficient structural capabilities in order to withstand the various loads that occur during handling, processing, transportation, storage, acceleration and firing [2-5]. A solid propellant rocket motor, stored either in the vertical or horizontal condition over a period of time, experiences a constant load which may affect the dimensional stability, long term durability and reliability of the propellant grains. In other words, an HTPB-based composite propellant exhibits time-dependent mechanical behavior. Generally, in order to simulate long term storage, structural integrity analysis is performed using mechanical properties evaluated at low strain rates using a universal tensile testing machine (UTM). Composite propellants, being viscoelastic materials, have mechanical properties that are both time and temperature dependent, and often the phenomenon of nonlinear viscoelasticity is present. Since the mechanical properties of composite propellants are mostly affected by their polymer based binders, the main objective of this study was to characterize the viscoelastic behavior of composite propellants based on HTPB [6-10]. Moreover in the recent past, the mechanical behavior of composite solid propellants based on HTPB resin have been simulated using empirical relations, but the dependence of creep strain on stress and temperature (which is very important in order to understand the long term behavior of solid propellants) is seldom presented and explained [11-13]. An attempt is made in this paper to simulate the creep behavior of HTPB-based composite solid propellants. The creep behavior can be best represented by the Kelvin-Voigt model. The Kelvin-Voigt model consists of a Hookean spring and a Newtonian dashpot arranged in parallel and is the best way to represent creep behavior whereas in Maxwell models (where the spring and dashpot are arranged in series) is the best way to represent relaxation behavior [14-17]. In the present study, the creep-recovery response of three different composite propellant formulations based on HTPB resin were studied using a dynamic mechanical analyzer (DMA) for various loading conditions [18-20]. The spring constant and damping coefficient are material constants that were determined by curve fitting experimental data. In addition, the dependence of creep strain on stress was analyzed and presented.

## 2 Mathematical Formulation

Viscoelastic models generally consist of some combination of Hookean springs and Newtonian dashpots arranged in either series or parallel to illustrate the interplay between viscous and elastic responses and to develop differential equations that describe the stress-strain-time relationship. A spring represents the elastic behavior of the material and obeys Hooke's law. It is specified by a spring constant  $E$ . The stress developed in the material is assumed to be proportional to the strain (Equation 1). The dashpot represents the viscous behavior of the material and obeys Newton's law of viscosity. In this case, stress is proportional to the strain rate (Equation 2) and the element is specified by a damping coefficient  $\eta$ .



**Figure 1.** Kelvin-Voigt model with single spring and single dashpot in parallel

The simplest model consists of a spring element and a dashpot element arranged either in series or parallel depending upon the material behavior response. When both elements are arranged in series, it is called a Maxwell model and if they are in parallel, is said to be a Kelvin-Voigt Model. The Kelvin-Voigt model as shown in Figure 1 best describes creep behavior [20-23]. The mechanical behavior of the material is described by two constants, the spring constant  $E$  and the damping coefficient  $\eta$ .

$$\text{Stress, } \sigma_1 = E \times \varepsilon_1 \quad (1)$$

where  $E$  = spring constant,  $\varepsilon_1$  = strain in spring due to stress  $\sigma$

$$\text{Stress, } \sigma_1 = \eta \times \frac{d\varepsilon_2}{dt} \quad (2)$$

where:  $\eta$  = damping coefficient of dashpot,  $\frac{d\varepsilon_2}{dt}$  = strain rate

Since both the components are connected in parallel in the Kelvin-Voigt model, the strain at any time in both components of the system is the same *i.e.*

$$\text{Strain, } \varepsilon = \varepsilon_1 = \varepsilon_2 \quad (3)$$

However, the stresses developed in both the elements due the total applied stress ( $\sigma$ ) are different. The stress developed in the spring element is termed  $\sigma_1$  and that developed in the dashpot element  $\sigma_2$ .

$$\text{The total applied stress, } \sigma = \sigma_1 + \sigma_2 \quad (4)$$

The governing equation of the above Kelvin-Voigt model is represented as Equation 5.

$$\sigma = \sigma_1 + \sigma_2 = E \times \varepsilon + \eta \times d\varepsilon/dt \quad (5)$$

Equation 5 can be further re-written using the initial condition (when,  $t = 0$ ,  $E = 0$  and  $0 < t < t_0$ , stress =  $\sigma$  and strain =  $\varepsilon$ ) as Equation 6.

$$\varepsilon = \frac{\sigma}{E} \left[ 1 - e^{-\frac{E}{\eta}t} \right] \quad (6)$$

If a propellant specimen is represented by this model, then the two independent constants “ $E$ ” and “ $\eta$ ” can characterize the viscoelastic behavior of propellants completely. Creep tests are carried out at constant stress and the variation of strain with time is recorded. Differentiating Equation 6 with respect to time and eliminating the time variable, a simple linear equation (Equation 7) is obtained as

$$\frac{d\varepsilon}{dt} = \frac{\sigma}{\eta} - \frac{E}{\eta} \varepsilon \quad (7)$$

The creep behavior of any propellant specimen is given by the strain-time dependence obtained during testing. As per Equation 7, if  $\frac{d\varepsilon}{dt}$  is plotted against strain, the slope of the line represents  $\frac{E}{\eta}$  and the intercept represents the ratio,  $\frac{\sigma}{\eta}$ . The variation of viscoelastic material constants of different propellant formulations were analyzed to predict the mechanical behavior of solid propellants under constant loading conditions.

### 3 Experimental

#### 3.1 Materials

Ammonium perchlorate (AP) with an average particle size of  $300 \pm 10 \mu\text{m}$  was procured from Pandian Chemicals Ltd., Cuddalore. Average particle sizes of

50  $\mu\text{m}$  and 6  $\mu\text{m}$  were obtained by grinding 300  $\mu\text{m}$  AP. Aluminum powder, with an average particle size of  $15 \pm 3 \mu\text{m}$  was procured from The Metal Powder Company, Madurai. Copper chromite and iron oxide (used as burning rate modifiers in propellant formulations) were also procured and incorporated into the formulations after drying at 105  $^{\circ}\text{C}$  for 4 h. The antioxidant, 2-phenyl naphthylamine (NONOX-D) with an assay value of 95%, a melting point of 108  $^{\circ}\text{C}$  and a specific gravity of 1.24  $\text{g}/\text{cm}^3$ , was also procured and used as-received. HTPB, manufactured by free radical solution polymerization, with an average molecular weight of 2500, a hydroxyl value of 43 mg KOH/g, a viscosity of 5500  $\text{mPa}\cdot\text{s}$  at 30  $^{\circ}\text{C}$ , a specific gravity of 0.90  $\text{g}/\text{cm}^3$  at 30  $^{\circ}\text{C}$  and a glass transition temperature ( $T_g$ ) of  $-72^{\circ}\text{C}$  was procured from Anabond Ltd., Chennai. The plasticizer, dioctyladipate (DOA) with an acid value of 0.5 mg KOH/g and a saponification value of 303 mg KOH/g, the curing agent toluene diisocyanate (TDI) with a specific gravity of 1.22  $\text{g}/\text{cm}^3$  and an assay value of 99.0%, and a bonding agent, a mixture of 1,1,1-trimethylol propane (with a hydroxyl value of 1220 mg KOH/g and a specific gravity of 1.17  $\text{g}/\text{cm}^3$  at 30  $^{\circ}\text{C}$ ) and 1,4-butanediol (with a hydroxyl value of 1220 mg KOH/g and a specific gravity of 1.013  $\text{g}/\text{cm}^3$  at 30  $^{\circ}\text{C}$ ) were also procured and used as-received.

**Table 1.** Propellant compositions investigated

Propellant composition	Type-I [wt.%]	Type-II [wt.%]	Type-III [wt.%]
AP, 300 $\mu\text{m}$	52.5	30.12	15.0
AP, 50 $\mu\text{m}$	15.5	35.38	19.4
AP, 6 $\mu\text{m}$	-	-	29
Al	18	18	18
Copper chromite	0.00	0.3	0.35
Iron oxide	0.00	0.2	0.25
HTPB	10.08	10.08	11.2
DOA	3.0	5.0	5.78
Bonding agent	0.12	0.12	0.12
NONOX-D	0.10	0.10	0.10
TDI	0.70	0.70	0.80

Three composite propellant formulations were studied having burning rates of 5 mm/s, 15 mm/s and 20 mm/s. Details of the compositions of the propellant samples studied are given in Table 1.

### 3.2 Methods

All experimental mixing of composite propellant slurries was carried out at a 5 kg batch level in a vertical planetary mixer. The method for preparation of propellant composition is described as follows:

The prepolymer resin *i.e.* the HTPB plus plasticizer DOA plus antioxidant NONOX-D and the bonding agent (a mixture of 1,1,1-trimethylol propane and 1,4-butanediol in the ratio of 1:2) was mixed in a vertical planetary mixer. The mixer was agitated for 30 min followed by mixing under vacuum for another 30 min to drive out entrapped air. After this, burning rate modifiers (copper chromite and iron oxide) were added and mixed thoroughly for 10 min. Furthermore, Al powder was added to the mix. After addition of Al, the composition was mixed for another 20 min. After this, AP (trimodal, with three average particle sizes of 300  $\mu\text{m}$ , 50  $\mu\text{m}$  and 6  $\mu\text{m}$ ) was added and mixed for 30 min to achieve a homogeneous material. The overall mixing temperature was maintained at  $40 \pm 2$  °C. After addition of all the solid ingredients, further mixing of the composition was carried out under vacuum for 30 min. At this stage, the curing agent toluene diisocyanate (TDI) was added and mixed for another 40 min. Then the composition was cast in a mould under vacuum and cured at 50 °C for five days.

Afterwards the propellant samples were prepared from each propellant type. A dynamic mechanical analyzer (DMA Q800) was used to characterize the viscoelastic creep behavior of the propellants. After the dual cantilever clamp was calibrated for position and mass, a creep test was performed and the variation of strain with time was recorded for each propellant formulation using rectangular specimens (length  $\times$  width  $\times$  thickness =  $35 \times 13 \times 3$  mm<sup>3</sup>) in dual cantilever mode. The test was conducted for each propellant formulation at a constant temperature (35 °C), varying the stress from 0.1 MPa to 3 MPa. Constant stress was applied for 600 s followed by recovery lasting 1200 s for each propellant specimen. The permanent strain was also recorded after recovery for each propellant. To study the effect of temperature on viscoelastic constants, another creep test was performed at a constant stress (1 MPa) while varying the temperature from 35 °C to 85 °C for each propellant formulation. Creep curves were generated and analyzed in both conditions.

## 4 Results and Discussion

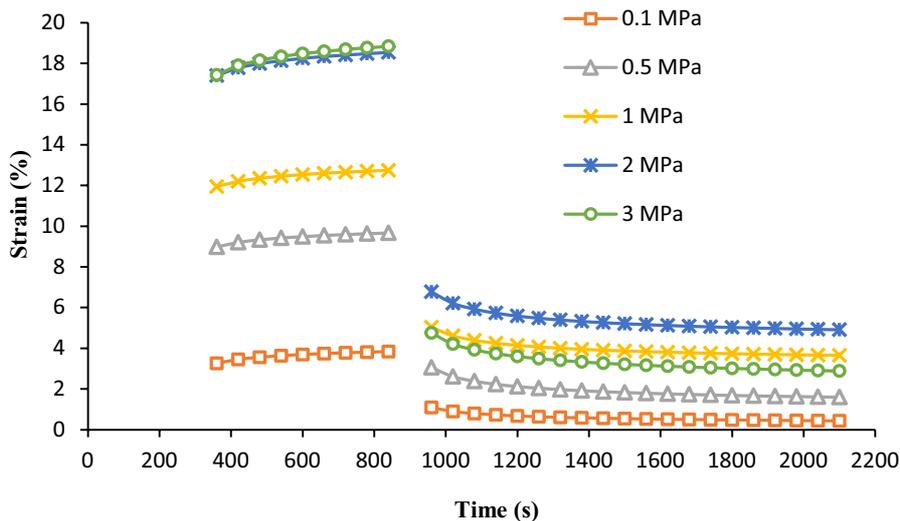
HTPB-based composite propellant formulations were used for the experiments to assess their viscoelastic behavior under constant loading conditions of stress and

temperature. Samples were prepared from three different composite propellant formulations having burning rates of 5 mm/s, 15 mm/s and 20 mm/s.

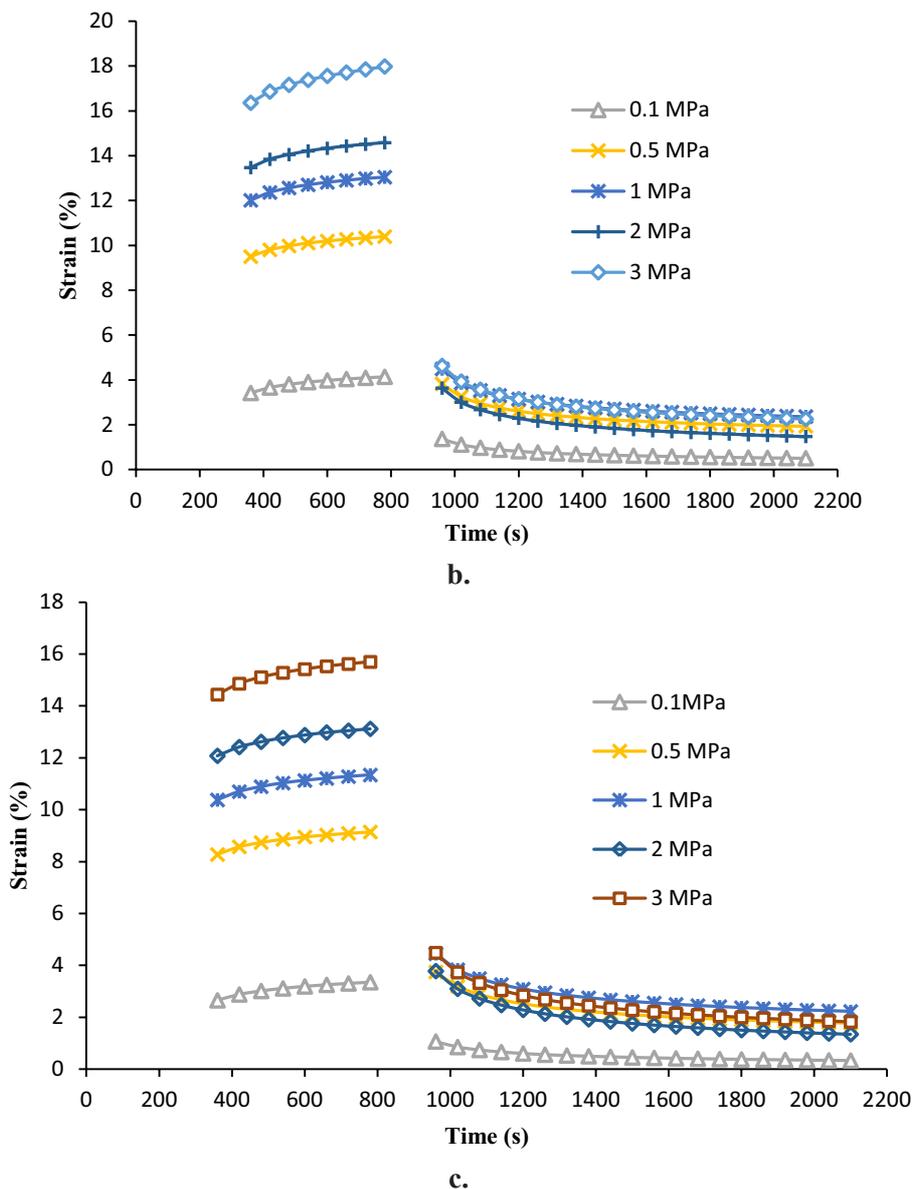
#### 4.1 Effect of applied stress on creep behavior

Each propellant sample was conditioned in a desiccated environment at a temperature of 27 °C before testing. For the three propellants studied, the variation of strain with time was plotted at each stress during loading and recovery as shown in Figures 2a, 2b and 2c. The amount of strain remaining in Type-I propellant can be seen to increase with increase in stress up to a certain stress level after which it decreases. For this propellant, the strain remaining after recovery was observed to be 0.4% at 0.1 MPa reaching a maximum of 4.9% at 2 MPa and then decreasing to 2.8% at 3 MPa. A similar trend was also seen in the other two tested solid propellants (Figures 2b and 2c). The value of the permanent deformation represents the viscous part of the viscoelastic behavior. In order to obtain the viscoelastic material constants using Equation 7, the strain rate  $d\varepsilon/dt$  was plotted against strain  $\varepsilon$ .

For each propellant, the spring constants and damping coefficients were computed using a least squares fit at different stresses. The two ratios and were first evaluated and plotted against stress to study their dependence on the independent variable. The material constants at different stresses for each propellant formulation studied are given in Table 2.



a.



**Figure 2.** Variation of creep strain with recovery vs. time for 3 types of propellant at different stresses: a) for Type-I, b) for Type-II, c) for Type-III

**Table 2.** Value of constants of Kelvin-Voigt model at different stresses for studied propellants

Stress [MPa]	Spring constant, $E$ [MPa]			Damping coefficient, $\eta$ [MPa·s]			$\frac{\sigma}{\eta}$		
	Type-I	Type-II	Type-III	Type-I	Type-II	Type-III	Type-I	Type-II	Type-III
0.1	2.5	2.4	2.8	526.3	526.3	666.67	0.0002	0.00019	0.00015
0.5	5.1	4.8	5.4	1086.9	1041.6	1162.79	0.00046	0.00048	0.00043
1	7.7	7.6	8.0	1666.7	1666.7	1666.7	0.0006	0.0006	0.0006
2	10.8	13.5	15.0	2247.2	2898.5	3448.3	0.00089	0.00069	0.00058
3	15.9	16.7	18.8	3333.3	3750.0	4166.7	0.0009	0.0008	0.00072

The elastic and viscous portion of the propellant viscoelastic behavior is governed by the percentage of binder and binder-filler interaction present in the formulation. It is clear from Table 2 that the spring constants  $E$  and damping coefficients  $\eta$  increase with increase in stress for all the three propellants tested. The spring constants ranged from 2.4 MPa to 18.8 MPa whereas the damping coefficients ranged from 526.3 MPa·s to 4166.7 MPa·s. It may also be observed that the damping coefficients are numerically higher than the spring constants. As the damping coefficients represent the viscous part of the propellant formulations, it is clear that the propellants tested are viscosity-dominant under the creep loading conditions studied. Compared to the spring constants, the damping coefficients show better sensitivity to the applied stress levels. The variations of  $E$  and  $\eta$  with stress have been plotted and best-fit curves were obtained by least squares minimization of errors. It was observed that both constants have a linear variation. The governing linear equations for determining the spring constants and damping coefficients are given in Table 3.

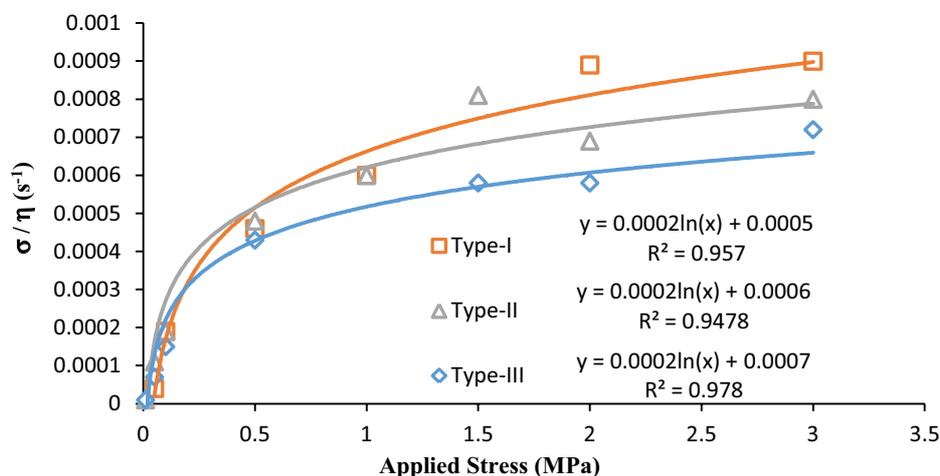
**Table 3.** Variation of  $E$  and  $\eta$  with stress for three types of propellants

Propellant	Variation of $E$ with stress	Variation of $\eta$ with stress
Type-I	$E = 4.3834\sigma + 2.6535$ $R^2 = 0.9877$	$\eta = 912.28\sigma + 567.88$ $R^2 = 0.9833$
Type-II	$E = 4.9374\sigma + 2.4277$ $R^2 = 0.9802$	$\eta = 1327.9\sigma + 379.08$ $R^2 = 0.9996$
Type-III	$y = 5.6217x + 2.7365$ $R^2 = 0.9885$	$\eta = 1261\sigma + 587.96$ $R^2 = 0.9819$

However their ratios,  $\frac{E}{\eta}$  at different stress levels are more or less constant for each type of propellant. Therefore, the ratio  $\frac{E}{\eta}$  may be considered to be independent of stress. The average value of  $\frac{E}{\eta}$  was found to be 0.0046. It was

also observed that the ratio,  $\frac{\sigma}{\eta}$  shows a logarithmic variation (see Figure 3). The governing equations of the logarithmic variation for all three propellants (with a minimum of 96% confidence level) are also shown in Figure 3. It is clear that the governing equations can be re-written as  $\frac{\sigma}{\eta} = 0.0002 \ln \sigma + C \times 10^{-4}$ ; where  $C = 5, 6$  and  $7$  for propellant type-I, II and III with  $R^2$  values of 0.957, 0.9478 and 0.978, respectively.

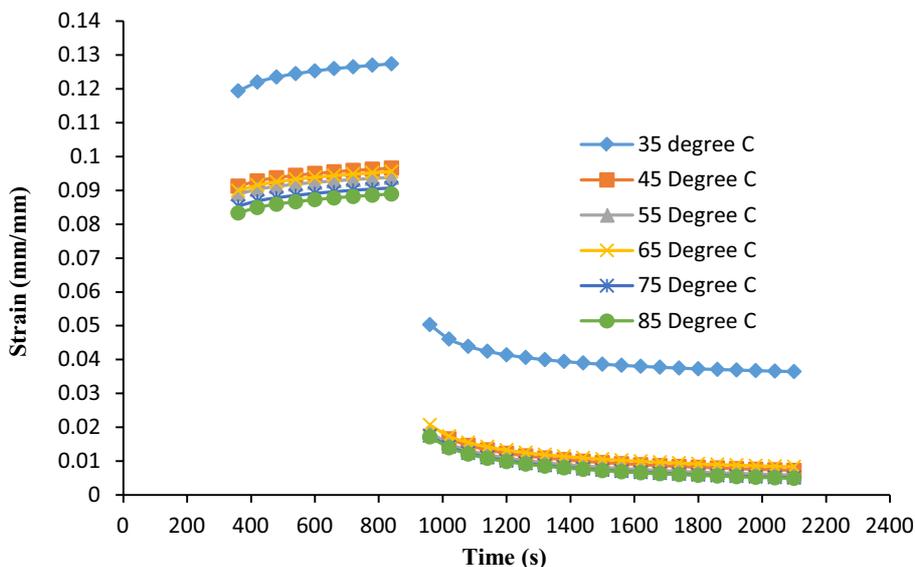
Thus mathematical expressions have been generated to obtain the material constants at any stress level for the propellant compositions studied.



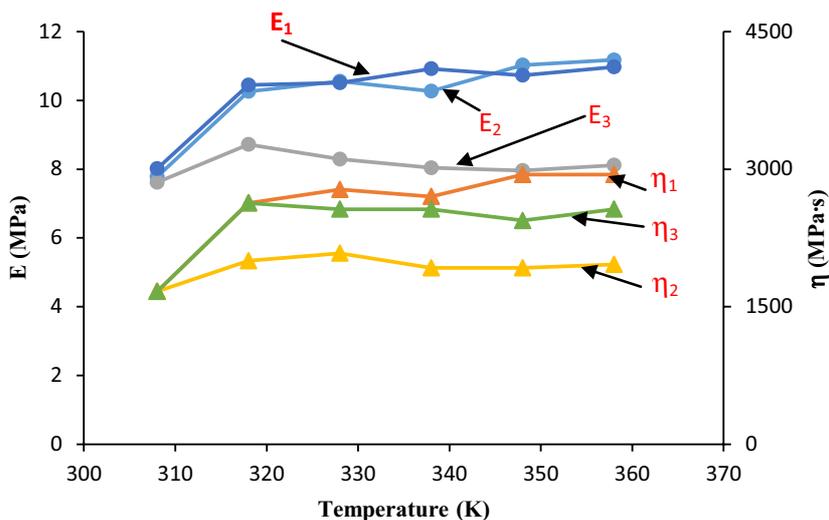
**Figure 3.** Variation of  $\sigma/\eta$  with stress

## 4.2 Effect of temperature on creep behavior

Each propellant sample was conditioned in a desiccated environment at different temperatures varying from 35 °C to 85 °C before testing. The tests were performed at a constant stress level of 1 MPa. The variation of strain with time was plotted at each temperature during loading and recovery as shown in Figure 4. It is clear from Figure 4 that the creep strain at 35 °C is significantly higher than the strains at higher temperatures. Similar trends were observed in all three propellant formulations. The strain remaining after recovery was observed to be 3% at 35 °C whereas at higher temperature it was less than 1%. The spring constants  $E$  and damping coefficients  $\eta$  were also plotted against temperature (see Figure 5).



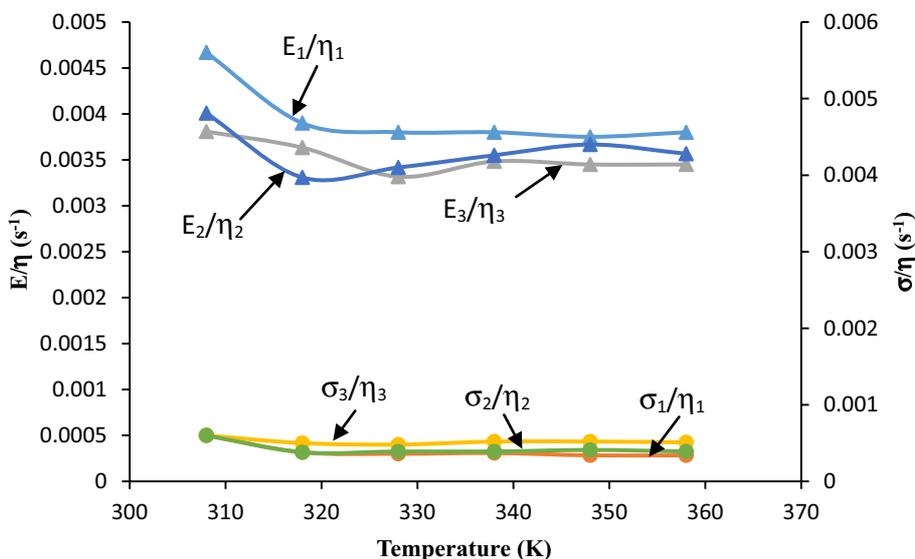
**Figure 4.** Variation of creep strain with recovery vs. time for Type-I propellant at different temperatures



**Figure 5.** Variation of  $E$  and  $\eta$  with temperature for the propellants studied

It can be seen that the spring constants and damping coefficients of all three propellants were approximately 8 MPa and 1667 MPa·s respectively at 35 °C for an applied stress of 1 MPa. There was no significant change in both

constants at temperatures greater than 45 °C. The ratios  $\frac{\sigma}{\eta}$  and  $\frac{E}{\eta}$  were computed and observed to be nearly constant with increase in temperature (see Figure 6). The mathematical formulation developed in this study can be implemented to understand the creep behavior of solid propellants for different loading conditions of stress and temperature. The viscoelastic properties of the solid propellants can be determined by the correlation developed for the HTPB-based propellant compositions.



**Figure 6.** Variation of  $E/\eta$  and  $\sigma/\eta$  for Type-I, II and III propellants with temperature

The composite propellant formulations studied showed that they are viscosity-dominated and possess a larger permanent strain after recovery when subjected to higher stress levels. This outcome can be used to simulate the structural behavior of propellants in a rocket motor subjected to a constant loading condition. The strain induced in a rocket motor propellant due to its storage conditions either in the vertical or horizontal orientation at different temperatures may be analytically calculated using the viscoelastic material constants measured. This will help to evaluate the margin of safety of rocket motors due to creep.

## 5 Conclusions

Solid rocket composite propellants based on HTPB/AP/Al have been modelled using the Kelvin-Voigt model. The propellant specimens tested at different level of stress and temperature using a dynamic mechanical analyzer can be simulated using two material property variables namely a spring constant  $E$  and a damping coefficient  $\eta$ . Creep curves with subsequent recovery were generated for three different propellants having burning rates of 5 mm/s, 15 mm/s and 20 mm/s. The strain was found to increase with increase as the stress level was varied from 0.1 MPa to 3 MPa at a given time. Permanent strain was observed after recovery for all three propellants studied. The permanent strain was more significant in the case of stress recovery as compared to temperature recovery. The spring constants of the propellants studied were evaluated and found to vary from 2.4 MPa to 18.8 MPa whereas the damping coefficients varied from 526.3 MPa·s to 4166.7 MPa·s. The ratio  $E/\eta$  for all three propellants studied was constant in the entire stress range whereas the ratios  $E/\eta$  and  $\sigma/\eta$  were nearly constant with temperature. It was also observed that up to a stress level of 1 MPa, there was no significant change in the values of the spring constants and damping coefficients for all the propellants studied. However at a higher stress level (2-3 MPa), both material constants increased significantly. The approach presented in this paper can be applied to any solid propellant in order to evaluate the material constants that govern their viscoelastic behavior. The mathematical correlation developed can be used to predict the creep behavior of solid rocket propellants at different stress levels and temperatures.

### Abbreviations and symbols

$E$	spring constant
$\eta$	damping coefficient
$\sigma$	stress
$\varepsilon$	strain
$t$	time
$d/dt$	the first derivative as a function of time

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