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Characterization of Thermochemical Properties and Combustion Behavior In AP/HTPB Polymer Composite Solid Propellant for Enhanced Performance

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Abstract

The thermal decomposition of solid propellants containing hydroxyl-terminated polybutadiene (HTPB) binder is a complex process, driven by multiple interacting chemical and physical factors. Solid propellant combustion characteristics are profoundly influenced by the composition of the propellant, the conditions of pressure, the starting temperature, and a range of aero-thermochemical parameters. The paper explores the influence of polymeric structure in the HTPB binder on thermal decomposition characteristics of the AP/HTPB composite propellants and correlates material properties to superior performance results in composite formulations of relevance to aerospace applications. In this study, the thermal decomposition of AP/HTPB based propellant samples are analyzed through thermo-gravimetric analysis and differential thermal analysis traces were obtained on a simultaneous thermal analyzer. Thermogravimetric analysis facilitates the investigation of decomposition kinetics in solid rocket propellants. Thermogravimetric analysis is executed to assess the thermal decomposition behaviors of propellants samples across a temperature range of 27℃ to 927 ℃ at different heating rates. Propellant samples weighing 2.5 mg to 3 mg have been used for the analysis. The rate at which a solid composite propellant burns is a crucial ballistic characteristic, closely linked to the highest decomposition temperature seen in particular formulations of the propellant. The activation energy, inferred from the slope of the Kissinger plot, was found to be 230.64 kJ/mol.

Keywords: Rocket propellants, hydroxyl-terminated polybutadiene binder, Thermal decomposition, thermo-gravimetric analysis and differential thermal analysis

INTRODUCTION

Propellants consist of materials that ignite to create significant quantities of gases at high thermal levels. They can maintain their combustion process autonomously, without relying on any surrounding oxidizing agents. The amount of solid particles in the combustion gases should be kept as low as possible [7, 16, 23]. Additionally, propellants should be designed to burn at lower flame temperatures. Such propellants are suitable for rocket propulsion, particularly when the energy content is not the primary concern, but rather the reduced signature of the exhaust gases is prioritized. Few researchers explored how the rate of heating influences the thermal breakdown characteristics of two solid propellant binders: a) the widely used Hydroxyl-Terminated Polybutadiene Binder (HTPB) alongside the advanced energetic binder Glycidyl Azide Polymer (GAP), and b) polyurethanes derived from HTPB with three

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distinct curatives [14, 34]. In a separate study, (Park et al., 2020) aimed to enhance the viscosity of the propellant mixture and adjust the combustion rate by varying the particle sizes of ammonium perchlorate (AP) to 200 µm and 6 µm, respectively [24]. It is noted that increasing the particle size increased the viscosity by 70 %, while reducing the particle size resulted in increasing the burning rate. By increasing the mass fraction of HTPB in AP by 70 %, the surface temperature of the pyrotechnic composition increased by approximately 90°C. This also increased the burning rate to 2.7 cm/s [30,31]. Extensive research work on solid propellants is carried out around the globe. This study makes important contributions to polymer science by investigating the structure-properties relationship in AP/HTPB and an understanding of molecular structure in terms of thermal stability and decomposition under various conditions.

Lim et al., (2023) analysed the effect of metal composition of Electrically Controlled Solid Propellants (ECSP). They investigated the ECSP burning rate containing 5 %, 10 %, and 15 % by volume of lithium perchlorate. They observed that a higher composition of lithium perchlorate resulted in approximately 38 % faster burning and 9 % lower flame temperature. This phenomenon is attributed to increased metal oxides and particles ejected through combustion products [18].

Stone, (1960) pioneered the implication of differential thermal analysis in determining the oxidation phenomenon of various compositions. The test procedure involved controlling variables like temperature, pressure and gas composition. During the study, it was found that at atmospheric pressure ammonium perchlorate became unstable when the temperature reached 405 °C .(Y. Sun & Li, 2008)investigate the thermal decomposition mechanism of isocyanate-laden Glycidyl Azide Polymer (GAP). Through their study, they found that varying the composition of nitro glycerin (NG) and Butanetriol trinitrate(BTTN) in GAP reduced the decomposition temperature by 20°C to 40 °C . It was noted that the lower composition of NG and BTTN was beneficial in enhancing the heat of cured pyrotechnic composition that controlled the rate of decomposition [29].

Pisharath & Ang, (2007); You et al., (2012) while employing HTPB noted changes in phenomenological data, specifically, decomposition temperature intervals, mass loss and enthalpy with change in heating ate are established [25, 37]. Similar studies revealed a strong dependence on heating rate implying a more complicated decomposition mechanism on HTPB-based propellants [11, 21, 22, 28, 38].

Dong et al., (2022) In their simulation, they uncovered how the fine cross-linked configuration of the HTPB matrix impacts AP/HTPB propellants. They assessed the rate of stretching during uniaxial extension and examined how the interface between the filler and matrix influences the mechanical properties [10]. The introduction of defects on the surface of the AP filler demonstrated that this contact area is significant in influencing the strain characteristics of the matrix molecules [20].

Liu et al., (2022) demonstrated that the DSC technique, which depends on a direct relationship between peak temperature and heating rate, effectively determines kinetic parameters related to thermal breakdown, such as activation energy and rate constants [19]. One investigation found that the Ozawa method ranks among the most frequently utilized methods for assessing activation energies based on linear heating rates. This phenomenon was hinted to the emergence of iso-conversational methods [8].

Bernigaud et al., (2023); Vellaisamy & Biswas, (2020) demonstrated that the endothermic reaction during ETPE propellant decomposition progressed uniformly between 150 and 250 °C. The activation energy was consistently measured at 170 kJ/mol for α values below 0.2 [6, 33]. Furthermore, kinetic analysis revealed that liquid RDX in the pyrotechnic composition effectively prevented the evaporation of GAP.

Alvin Hong, (2022) explored the behavior of a combination of GAP and the nitramine oxidizer known as 4,10-dinitro-2,6,8,12-tetraoxa-4,10-diazaisowurtzitane (TEX) during its decomposition. Findings showed that the temperature of decomposition for TEX remained unaffected by the presence of GAP in their mixture. The investigation identified a notable exothermic peak around 232°C, attributed to the liberation of N2 [5].

Optimizing both mechanical and thermal characteristics of composite propellants depends critically on the interaction of the polymer matrix, HTPB, and the oxidizer, AP. Being a polymeric binder, HTPB impacts the combustion process but improves thermal stability and mechanical resilience by giving structural integrity at high strain. Furthermore, by interfacing HTPB, decomposition pathways can be tweaked even more for maximum general performance and safety under different conditions [2-4].

RESEARCH GAP AND OBJECTIVE OF THE STUDY

The literature gap highlighted in this study underscores several critical aspects about solid propellants and their thermal decomposition behavior. Typically, optimizing combustion efficiency is a challenging task. It is essential to minimize the presence of solid particles in combustion gases while maintaining low flame temperatures [9]. This is crucial for applications requiring minimal exhaust gas temperature. While the influence of heating rate on HTPB-based propellants has been explored, further investigation into the complex decomposition mechanisms under varying heating conditions is essential. It is also important to know that different heating rates impact decomposition temperature intervals, mass loss, and enthalpy changes. This process is vital for enhancing propellant performance during combustion and ensuring safety [12,13].

Extensive research work is needed to focus on the understanding of propellant decomposition, chemical kinetics and combustion mechanisms. Methods for finding activation energy estimation for kinetic parameter determination are crucial for advancing accuracy in thermal analysis. Moreover, the study of specific propellant formulations, including AP-HTPB remains pivotal. Further investigations are needed to clarify these components' influence over thermal decomposition behavior. This phenomenon subsequently affects the propellant performance characteristics. Thorough kinetic analyses are essential for uncovering the fundamental mechanisms that control propellant decomposition pathways and reaction kinetics across diverse environmental conditions. Addressing these gaps will significantly enhance the understanding and applications of solid propellants, thereby improving their efficiency, performance, and safety in aerospace propulsion systems. This research study seeks to fill the gaps by carrying out an in-depth analysis of the thermal decomposition behavior of the AP/HTPB composite solid propellants under controlled conditions using thermogravimetric and differential scanning calorimetry analyses. Thermogravimetric and differential scanning calorimetry analyses are conducted to identify the structure-property relationships of the AP/HTPB composite. The stability of the polymer matrix and its interaction with AP as a reinforcing filler are determined by these analyses. For the study, solid rocket propellants containing HTPB binder are used.

EXPERIMENTAL WORK

The experimental setup included a NETZSCH STA 449 C simultaneous thermal analyzer, which is used to perform both TGA and DSC analyses. The setup allowed for the evaluation of thermal decomposition behaviors and kinetic parameters of the AP/HTPB composite solid propellants under controlled conditions.

The thermal decomposition of solid rocket propellants utilizing an HTPB binder is a multifaceted process that can encompass a range of chemical and physical interactions. The combustion behavior of these solid propellants is significantly influenced by factors such as the composition of the propellant, pressure, initial temperature, and several factors related to the aero-thermo-chemical dynamics within rocket motors [12, 17, 26]. Thermo-Gravimetric Analysis (TGA) is employed to analyze the thermal decomposition data of propellant samples at different heating rates, spanning temperatures from 27 °C to 927 °C [35,36]. In order to analyze the thermal degradation behavior of AP-HTPB solid propellants, thermo-gravimetric analysis was performed at three different heating rates: 5, 10, and 20 K/min, within a flowing argon atmosphere (60 ml/min). The samples, each weighing about 2.5 mg, were heated in aluminum crucibles. Figure 1 presents the NETZSCH STA 449 C simultaneous thermal analyzer [27, 32].

Figure 1. Simultaneous thermal analyzer by NETZSCH, Model STA 449 C.

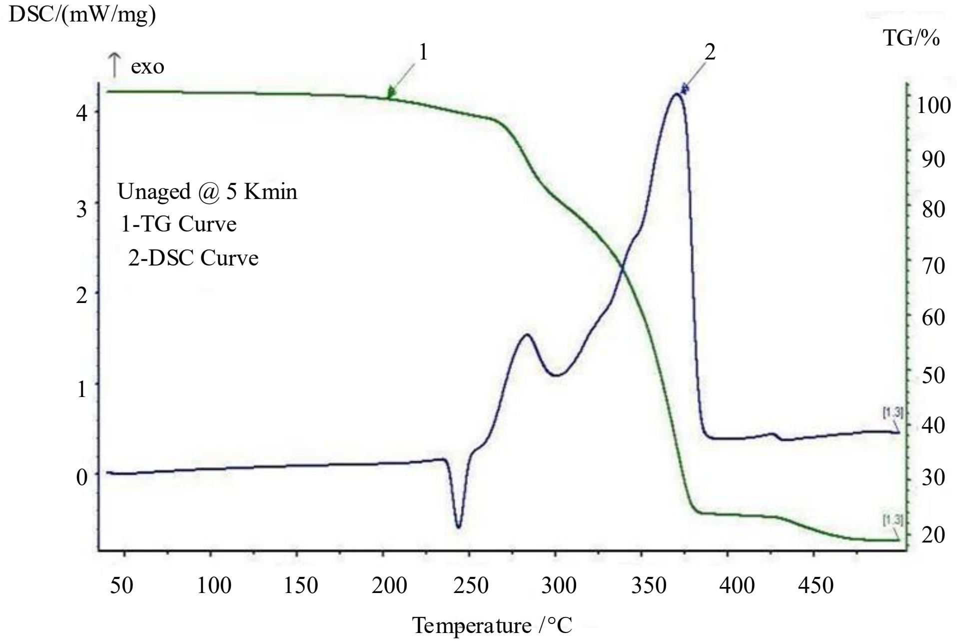

Figure 2. TGA, DSC traces for degradation of AP-HTPB (unaged) composite solid propellant at 5 K/min heating rate.

RESULTS AND DISCUSSION

Investigating the kinetic characteristics of the decomposition process may yield valuable insights into the complex nature of thermochemistry. The thermal decomposition behavior of the HTPB polymer matrix within the composite brings into prominence polymer-filler interactions and contributes toward a better understanding of polymer physics and the influence of molecular structure on composite performance. The HTPB matrix decomposes in stages; the primary initial degradation is from the low-molecular-weight compounds within the binder, whereas more complex breakdowns associated with crosslinked polymer chains occur in later stages. It is important to recognize that these stages are very important for the material's stability since the polymer matrix is a structure that decelerates the decomposition rate and provides controlled release of energy—very important for applications in high-performance propellants. To identify these parameters for specific compounds, isothermal thermogravimetric analysis (TGA) curves are generated. The thermal decomposition data of the propellant samples are analyzed through TGA/DSC traces obtained on a simultaneous thermal analyzer, in the temperature range of 25 °C to 200 °C. The DSC technique utilizes heat to evaluate the progression of chemical or physical changes that occur over a spectrum of temperatures. This calorimetry approach consists of measuring the energy needed to achieve a zero temperature difference between a substance and its reference, as each specimen undergoes the same temperature protocol. During a DSC experiment, Pin-holed type crucibles made of Alumina have been used. Propellant samples weighing 3 mg to 5 mg have been used for the analysis. Eventually, the material initiates a chemical or physical change that either emits or takes in heat. As the temperature climbs, this transformation continues until it reaches completion. The ordinate value at any specific time or temperature indicates the disparity in heat flow between a reference sample and the unknown material, which is connected to the kinetics of the process. Calculating the area under the heat flow curve allows for the determination of the enthalpy change related to the thermal event of interest.

A thermogravimetric thermal analysis was executed at a heating rate of 5 K/min to investigate how the AP-HTPB composite solid propellant degrades thermally. The TGA/DSC data were collected using a NETZSCH STA (model 449 C) simultaneous thermal analyzer, within a temperature range of 323 K to 773 K, maintaining the same heating rate of 5 K/min under a flowing argon atmosphere at 60 ml/min. Each sample weighed around 2.3 mg and was analyzed in aluminum crucibles [15]. Figure 2 presents the TGA/DSC traces that illustrate the degradation process of unaged AP-HTPB composite solid propellant at this specified heating rate. The AP/HTPB composite shows the ability to engineer thermal and structural stability through the choice of polymer and composite material. The effectiveness of the polymer in encapsulating and binding with the AP particles is determined by its molecular structure that affects the decomposition rate as well as stability. This directly falls into current advancements in polymer engineering for aerospace applications.

The TGA curves indicate that that the thermal degradation process unfolds in two significant stages, regardless of the plasticizer dioctyl adipate (DOA), the HTPB binder, or the decomposition of total ammonium perchlorate [1]. The decomposition patterns in the AP/HTPB composite are such that the structure-property relationships within the polymer influence the overall thermal behavior of the composite. In HTPB, both linear and cross-linked structures support the tailored degradation rate. It is consistent with evidence showing how the molecular architecture of polymer matrices controls the decomposition stages, thereby affecting combustion rates and energy output. Hence, such findings underscore the relevance of structural features in polymer-bound propellants, wherein the choice of binder and composite interaction design can be optimized for performance in energy release and thermal stability. This two-step degradation pattern is validated through the onset/endset functionality in NETZSCH software. The first phase, occurring from 246.4 K to 272.3 K, shows a mass reduction of 7.03%, which can be attributed to the degradation of the DOA plasticizer. The second phase, illustrated in Figure 3, spans from 333.0 K to 351.9 K and is associated with the partial decomposition of the HTPB/TDI binder, resulting in a cumulative mass loss of 31.42%.

The final mass loss observed between 431.6 K and 438.1 K is associated with the residual HTPB/TDI binder, accounting for 0.48% of the initial mass of the sample. This finding aligns with thermal decomposition studies reported in existing literature. The residual mass after decomposition is measured at 26.25%. To determine the kinetic parameters of thermal decomposition, such as the pre-exponential factor and activation energy, the Differential Scanning Calorimetry (DSC) method is applied, capitalizing on the direct relationship between peak temperature and heating rate. Using the NETZSCH STA 449 C analyzer, DSC curves were captured over a temperature span from 298 K to 773 K, with sample masses around 2.0 mg, all housed in hermetically sealed aluminum pans. Three separate heating rates—5, 10, and 20 K/min—were utilized, and the resulting DSC curves for the composite samples are shown in Figure 3. The exothermic peak temperatures (Tp) obtained from these curves facilitate the calculation of kinetic parameters.

$$k = Ae^{\left(\frac{-E}{RT_p}\right)} \quad (1)$$

where ‘k’ is the rate constant and ‘R’ is the universal gas constant. ‘E’ is activation energy, and ‘A’ is the pre-exponential factor.

There exists a clear linear correlation between the heating rate “log β” and the inverse of the absolute temperature “Tp^{-1}”, allowing us to derive the following linear equation:

$$log\beta = a\left(T_p^{-1}\right) + b \quad (2)$$

where a and b signify the parameters of the linear equation.

Assuming the rate constant follows the Arrhenius principle and that the exothermic reaction occurs in a single step, the conversion at the peak rate remains unaffected by the linear heating rate. Given these conditions, equation (2) can be utilized to determine the maximum temperature of the exothermic peak at various heating rates [30]. By conducting several experiments with different heating rates, a plot of log β versus 1/Tp can be generated, which enables the direct calculation of activation energy from the slope of the curve.

By incorporating the exothermic peak temperatures and heating rate information into the NETZSCH software, researchers can extract the Arrhenius kinetic parameters (E, A) linked to the thermal breakdown of composite materials. These parameters can subsequently be utilized in equation (1) to calculate the overall rate constant. Using DSC curves obtained at three different heating rates—5, 10, and 20 K/min—the activation energy and kinetic parameters for the decomposition of propellant samples are investigated. Figure 3 demonstrates that the first stage of this process is characterized by endothermic behavior, whereas the second stage exhibits exothermic characteristics.

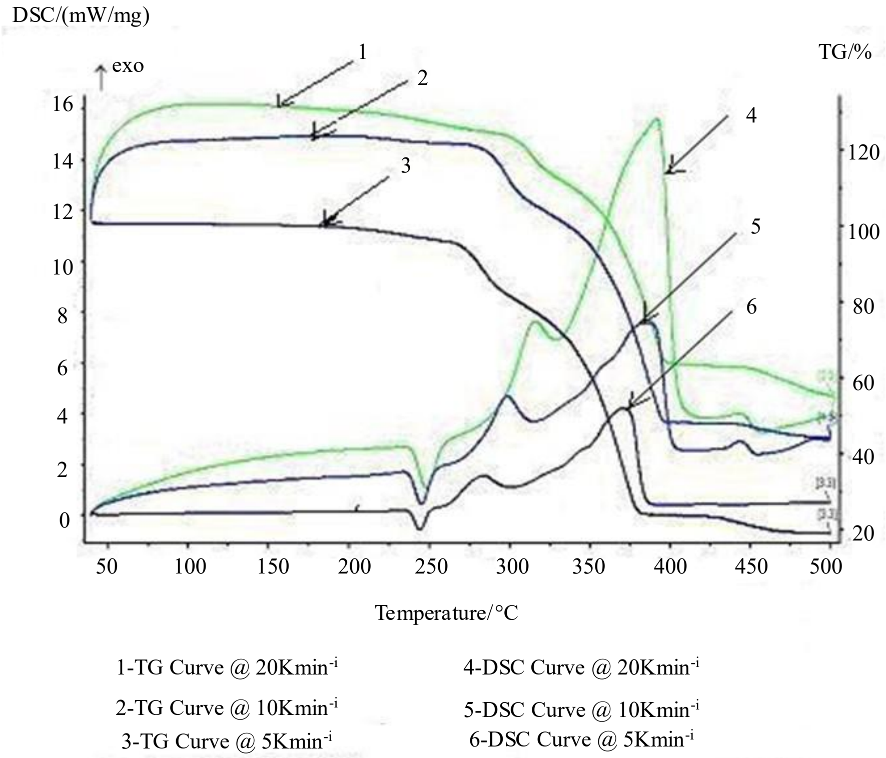

Figure 3. TGA, DSC traces for degradation of AP-HTPB composite solid propellant at 20, 10 and 5 K/min heating rates.

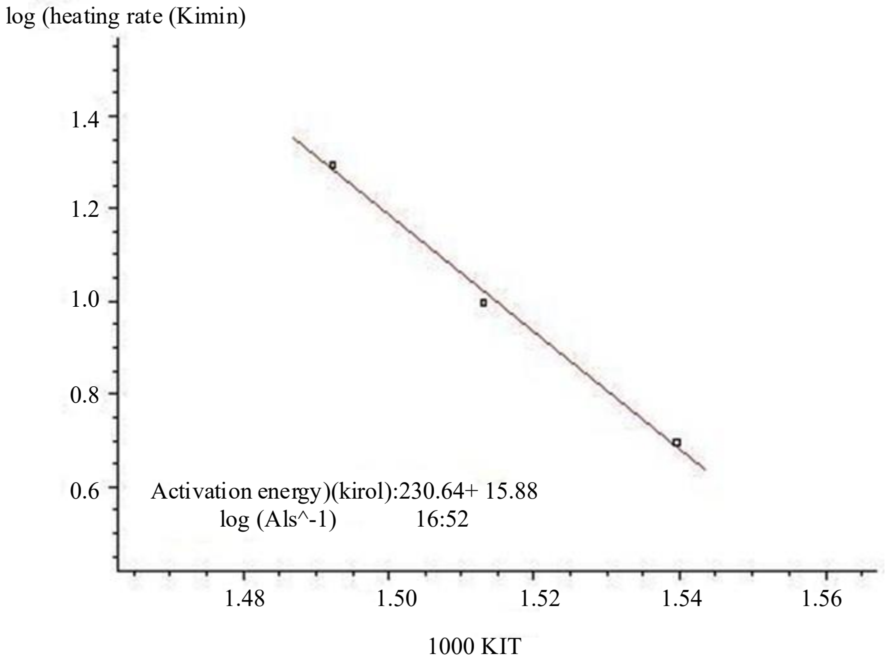

Figure 4. Kissinger plot for AP-HTPB propellant.

The endothermic event exhibits a consistent behavior across the various heating rates tested, with all conditions showing the same peak temperature occurring around 250 K. In contrast, the exothermic events display varying maximum temperatures of 624.9 K, 631.7 K, and 638.9 K for heating rates of 5, 10, and 20 K/min, respectively. In Figure 4, the correlation between log β and the reciprocal of the absolute temperature is displayed for each peak in the exothermic phase [8]. Studies on the decomposition kinetics of the AP-HTPB propellant through thermogravimetric analysis at heating rates of 5 to 30 K/min reveal calculated activation energies spanning from 100 to 230 kJ/mol. The slope of the Kissinger plot (ln(β) versus 1/Tp) in Figure 4 yields an estimated activation energy of 230.64 kJ/mol, which is in agreement with earlier literature results [6, 15, 38].

CONCLUSIONS

The study explored the thermal decomposition characteristics of AP-HTPB composite solid propellants through the application of thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). The results revealed a multi-stage decomposition process characterized by distinct mass loss steps corresponding to the degradation of plasticizer and binder components. The first stage involved the degradation of the plasticizer DOA raw material at temperatures ranging from 246.4 to 272.3 K, contributing to a 7.03% mass loss, while the second stage, occurring between 333.0 and 351.9 K, corresponded to the partial decomposition of the HTPB/TDI binder, resulting in a total mass loss of 31.42%. Key kinetic parameters of these decomposition processes, such as rate constant and activation energy, were determined using DSC and TGA data at different heating rates.

A residual mass corresponding to 26.25% of the initial sample mass remained after decomposition. Calculated from DSC curves at heating rates of 5, 10, and 20 K/min, the activation energy for thermal decomposition is determined to be 230.64 kJ/mol, consistent with literature reports [6, 15, 38, 39, 40]. Overall, the findings underscore the importance of understanding the kinetics and thermal behavior of solid propellants, which are critical for optimizing their performance and safety in rocketry applications. This work reveals the thermochemical properties which underscore the critical role HTPB plays as a polymeric binder in composite propellants. In fact, HTPB's influence on thermal decomposition behavior and stabilization of combustion characteristics underscores the importance of polymer selection in achieving the targeted propellant performance outcomes. The behavior of the AP/HTPB system matches known polymer physics principles, where structure-property relationships guide the stability of the material and controlled energy release, making it appropriate for aerospace applications. The thermal decomposition behaviour of the AP/HTPB composite propellant is a source of insight into its combustion characteristics. Even though the present work is not extensive in detailing all specific combustion measurements, it addresses thermal stability and decomposition behavior that are important factors instrumental in influencing combustion properties. Future research could explore additional factors influencing decomposition kinetics, such as pressure and propellant formulation variations, to further refine predictive models for propellant performance and reliability in aerospace applications.

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