



Studies on Ablative Performance of Silicone Low-Density Ablative Material

Sujatha Maddu^{1,2} · Tanu Srivastava^{1,3} · Naresh Kumar Katari¹ · Karuna Merugu⁴ · Rambabu Gundla¹ · S Krishna Mohan³

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Abstract

In the present studies, silicon based low-density ablative material has been studied for its ablative performance to protect flight hardware from temperature raises due to aerodynamic forces for a longer duration. The oxyacetylene flame test estimates ablative properties at 100–120 W/m² heat flux. The time duration for the flame test is taken 200 s. The material shows low mass and linear ablation rate with high ablative effectiveness. The degradation kinetics of silicon ablator is studied by Thermo Gravimetric Analysis (TGA) method. Non-isothermal TGA is performed in the temperature range of 30 °C to 900 °C at various heating rates, viz. 5, 10, 15, and 20 °C/min. Knowledge of the kinetic parameters of thermal decomposition is used to predict the degradation kinetics of the material at high heating rates, which the ablator faces during aerospace applications. During ablation, the silicone ablative material consists of a virgin zone, reaction zone, and char zone. These are explained by kinetic parameters determined with the help of free software called Kinetic Calculation.

Keywords Ablation · Thermal degradation · TGA · Kinetic parameters · Oxyacetylene flame test

1 Introduction

Ablation in ablative materials is a complex phenomenon, and much research has been done on this subject [1–6]. Ablation is an effective and reliable process primarily used in flight structures to protect the hardware from high external temperatures. Knowledge of degradation kinetics help in modeling of ablation phenomenon. In ablation, the heat is dissipated

through thermochemical degradation reactions. As a result, char is formed on the material's surface, which becomes a barrier between the virgin material and the hyperthermal atmosphere. It prevents direct plume contact with virgin and gives transpiration cooling at the same time. Elastomeric ablative materials are widely used for thermal protection in aerospace applications. Some examples of elastomeric systems are nitrile butadiene rubber, styrene-butadiene rubber, silicone rubber, EPDM, etc. The most used reinforcements are carbon, glass, ceramic fibers, and thermally stable fillers [7]. As ablative materials are critical to the safety of flight structures, it is necessary to investigate their ablation behaviors in advance. In this work, ablative properties and kinetics of degradation of a low-density ablative material based on silicone matrix and hollow glass microspheres as filler [8, 9] are studied by oxyacetylene flame test (OFT) and thermogravimetric analysis (TGA). OFT gives an insight into the ablation rate and ablative effectiveness. The oxyacetylene flame test is simple, cost-effective, and valuable in estimating ablative materials' thermal processes [10, 11]. At the same time, the TGA of materials allows us to calculate hypothetically the time required for the degradation of materials at the desired temperature, which leads to calculating kinetic parameters. These kinetic parameters help analyze

✉ Naresh Kumar Katari
nkatari@gitam.edu

✉ Karuna Merugu
kmerugu@gitam.edu

¹ Department of Chemistry, School of Science, GITAM (Deemed to Be University), Hyderabad Campus, Rudraram, Patancheru, Hyderabad, Telangana 502329, India

² Department of Chemistry, St. Anns College for Women, Mehdiapatnam, Hyderabad, Telangana 500028, India

³ Defence Research and Development Laboratory, Dr. APJ Abdul Kalam Missile Complex, DRDO, Hyderabad 500058, Telangana, India

⁴ Department of Chemistry, GITAM School of Science, GITAM Deemed to Be University, Bengaluru, Karnataka 562163, India

decomposition rates at various temperatures. The Committee of International Confederation for Thermal Analysis and Calorimetry (ICTAC) offers guidance for obtaining kinetic data for the thermal decomposition of inorganic solids and thermal and thermo-oxidative degradation of polymers and composites. These recommendations for kinetic measurement are focused on thermal analysis methods such as Thermogravimetric analysis (TGA), Differential scanning calorimetry (DSC), and Differential thermal analysis (DTA) [12]. Currently, isoconversional methods are widely used because it assumes that the rate of reaction does not change with the variation of heating rate and depends only on temperature. It involves temperature measurements for the constant degree of conversions at different heating rates [13, 14], and kinetic software is available for calculating kinetic parameters [15–17]. The estimated kinetic parameters activation energy and pre-exponential factor are utilized to generate plausible degradation mechanisms for ablative material.

2 Experimental

2.1 Raw Materials

Sylgard 184, a commercially available Vinyl terminated silicone resin from Dow Corning, is used as the matrix material. It is a two-part system. Part A is low viscous resin (viscosity: 3500–5500 cps), and Part B is a hardener / cross-linking agent. When both parts are mixed in a 10:1 ratio by weight, respectively, it polymerizes with a cured density of 1.03 g/cm³. The HGMs of K-25 grade from 3 M India Ltd is used as filler. Karstedt's catalyst (from Sigma Aldrich) is also purchased. Primer SS-4155 is purchased from Momentive India Ltd.

2.2 Sample Fabrication for Property Evaluation

Sylgard 184, part A (100 g), and part B (10 g) are mixed in a ratio of 10:1 by weight. 40 g of HGM particulates are reinforced slowly in the resin by mixing with hands. Karstedt's catalyst (0.01 g /10 g of resin) is added to the mixture [9]. The composite mixture is then sprayed on a square type of metal plate (Mild steel) of size 120 × 120 × 3.5 mm³. The ablative material is coated on the substrate with a 5 mm thickness. The application of ablative material by spraying method is given in our earlier studies [9]. Now castings can cure at room temperature for about 3–4 h. 08 samples are

prepared for analysis, of which 07 plates are kept for OFT, and from one plate, the coating is scratched out for TGA analysis.

3 Instrumentation

3.1 Oxyacetylene Flame Test (OFT)

The nozzle is typically placed on the sample's surface at a distance of 40 mm from the sample surface, and the flame impinges precisely at the center of the sample. The heat flux maintained is 100–120 W/cm² through the regulation of flow rates of oxygen and acetylene. This is kept constant throughout the experiments. The test is conducted for 200 s, and the substrate back wall temperature is measured by placing a K-type thermocouple on the back side of the substrate. The schematic diagram of the OFT setup is given below in Fig. 1.

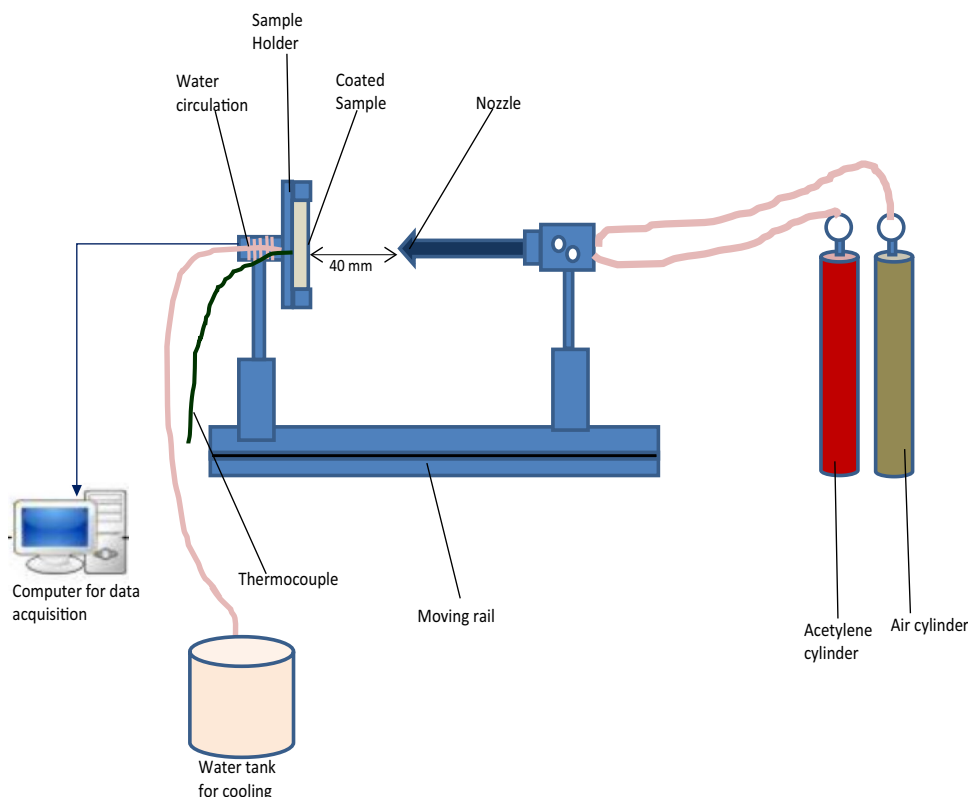
After the test, samples are visually inspected to check char layer formation and ablative layer intactness to the substrate. The charred layers are removed such that unaffected layers are observed. The thickness and mass of the sample are then taken. The ablative parameters—Linear Ablation Rate (LAR), Mass Ablation Rate (MAR), and Ablative Effectiveness (AE) of ablative materials are determined.

3.2 Thermal Properties

The non-isothermal TGA experiments at different heating rates (5, 10, 15 & 20 °C/min) are performed using a thermo gravimetric analyser (TA instrument, model TGA–55). Approximately 10 mg of finely powdered sample is placed in a small platinum crucible for each run under an air atmosphere with a 60 ml/min flow rate. The instrument collects the weight loss variation concerning the time and temperature automatically and is determined through the TA universal analysis software. The experiments are done multiple times under identical conditions to confirm the reproducibility of the results. Thermal stability in the material is studied concerning its thermal degradation behavior as a function of time and temperature.

The open-source software Kinetic Calculation [15] has been used to calculate kinetic parameters and generate kinetic equations of thermal decomposition. This is a user-friendly software that allows calculating kinetic parameters using by Vyazovkin method [18] and Flynn–Wall–Ozawa (FWO) method [19, 20]. The software has a provision for

Fig. 1 Schematic diagram of OFT setup



dividing the entire process into multistage decomposition processes and accurately calculating the mechanism's function, which is not dependent on the models. The following equations are used for kinetic parameters:

Vyazovkin method

$$\frac{d\alpha}{dT} = \frac{A}{\beta} \cdot e^{-E_a/RT} \cdot \alpha^m (1 - \alpha)^n - \ln(1 - \alpha)^p \tag{1}$$

FWO method

$$\ln(\beta) = \ln\left(\frac{AE_a}{Rg(\alpha)}\right) - 5.331 - 1.052\left(\frac{E_a}{RT}\right) \tag{2}$$

4 Results and Discussions

4.1 Thermal Stability

Thermo-oxidative degradation profiles of ablative material are recorded in the temperature range from 30 °C to 1000 °C at varying heating rates of 5, 10, 15 & 20 °C/min, and the results are given in Fig. 2. The shifting of Thermogravimetric (TG) curves of the material to higher degradation temperatures with the increase of heating rate is a general phenomenon for thermally activated processes. The degradation is a multi-step process, and variation in heating rate

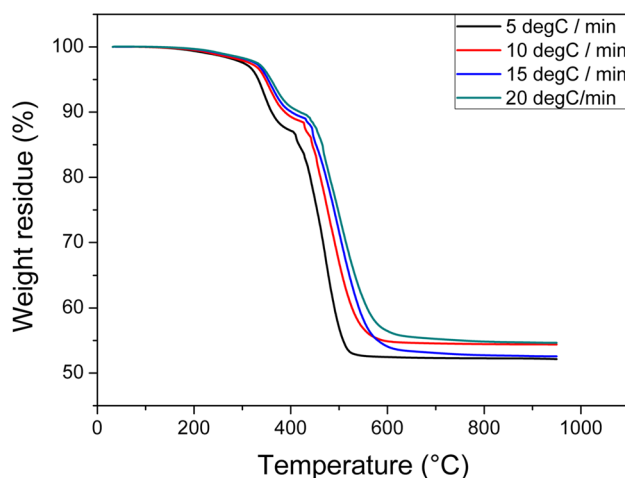


Fig. 2 Thermo-oxidative degradation profiles of ablative material at different heating rates

does not affect the pattern for degradation. In this thermo-oxidative degradation, the weight change of ablative material is minimal up to 350 °C due to the loss of volatile contents present in the material. After that temperature range of 350 °C – 440 °C shows another degradation step. A rapid weight loss in ablative material is started after 440 °C at all four heating rates. This may be considered the primary

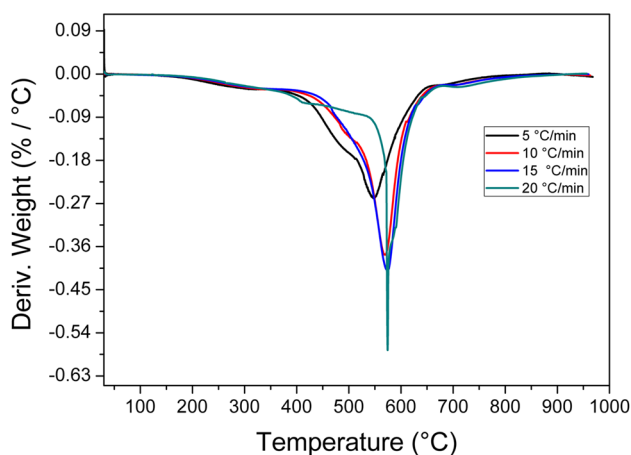


Fig. 3 Derivative TG (DTG) curves of ablative material at different heating rates

and final degradation process. After 630 °C, the weight loss started diminishing slowly, and weight residue was constant.

The char yield at 990 °C is approximately 53% to 57% and is not affected significantly by which heating rates. This residual mass is correlated well with the quantity of inorganic filler used in the ablative compound [21]. The first derivatives of the TG (DTG) curves obtained for ablative material are shown in Fig. 3 at different heating rates.

As the heating rate increases, the onset and end set degradation temperatures are also increasing. It is observed from the DTG plots that below 350 °C, the rate of thermal

degradation is low. The maximum degradation rates are narrow in the temperature region of 525 to 571 °C at all heating rates. After the maxima, the decomposition is slow again until it finishes at the final degradation temperature. The volatile/moisture evaporation region and end set of degradation are identical for all heating rates.

Nevertheless, the heating rate affects the maximum and minimum points of DTG curves which are shifted towards higher temperatures as the heating rate increases. This phenomenon can be explained based on heat transfer limitation. At a low heating rate, a large instantaneous heat is provided, and a long time may be required for the purge gas to reach equilibrium with the temperature of the furnace or the sample. In the same temperature region, there is a short reaction time at the higher heating rate, and the temperature needed for the sample to decompose becomes higher. This causes the maximum rate curve to shift to the right [22, 23]. Yang Xue et al. reported similar results of maximum degradation temperature for silica-filled silicone rubber composite [24].

4.2 Kinetic Studies

Kinetic studies are done using Kinetic Calculation software [15] which uses Vyazovkin and FWO methods. In order to verify the results given by the software, the FWO method, as per ASTM E1641, is also used. First, the software is run for the entire temperature range (35 – 986 °C).

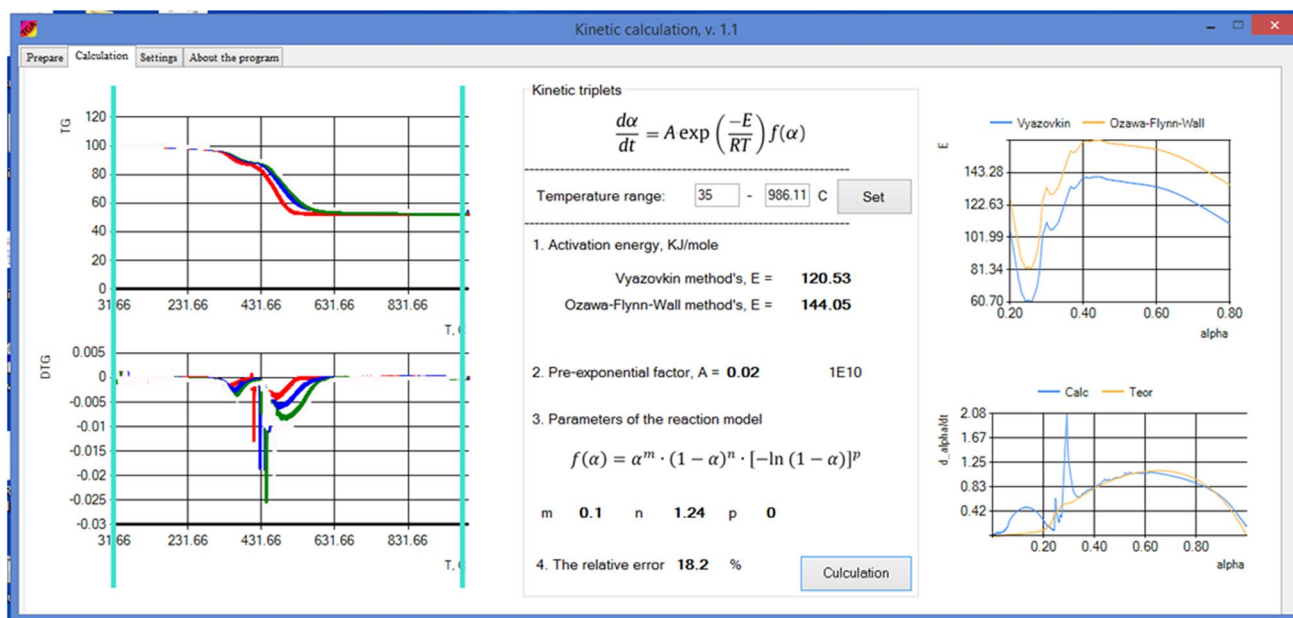


Fig. 4 Kinetic parameter for temperature range 35 – 986 °C

The screenshot of the results is given in Fig. 4. The overall activation energy (E_a) calculated by the Vyazovkin and FWO methods are 120.53 and 144.05, respectively. E_a 's dependency on the conversion degree is also shown in the top right corner. This also confirms that the thermo-oxidative degradation is not a single-step process. The bottom right corner plot shows the correlation between experimental and calculated data. The relative error

(18.2%) is greater than 10%, indicating a multi-step degradation mechanism [25].

The kinetic equation for the whole process can be written as

$$\frac{d\alpha}{dT} = \frac{2 \times 10^8}{\beta} \cdot e^{-120530/RT} \cdot \alpha^{0.1} (1 - \alpha)^{1.24} \tag{3}$$

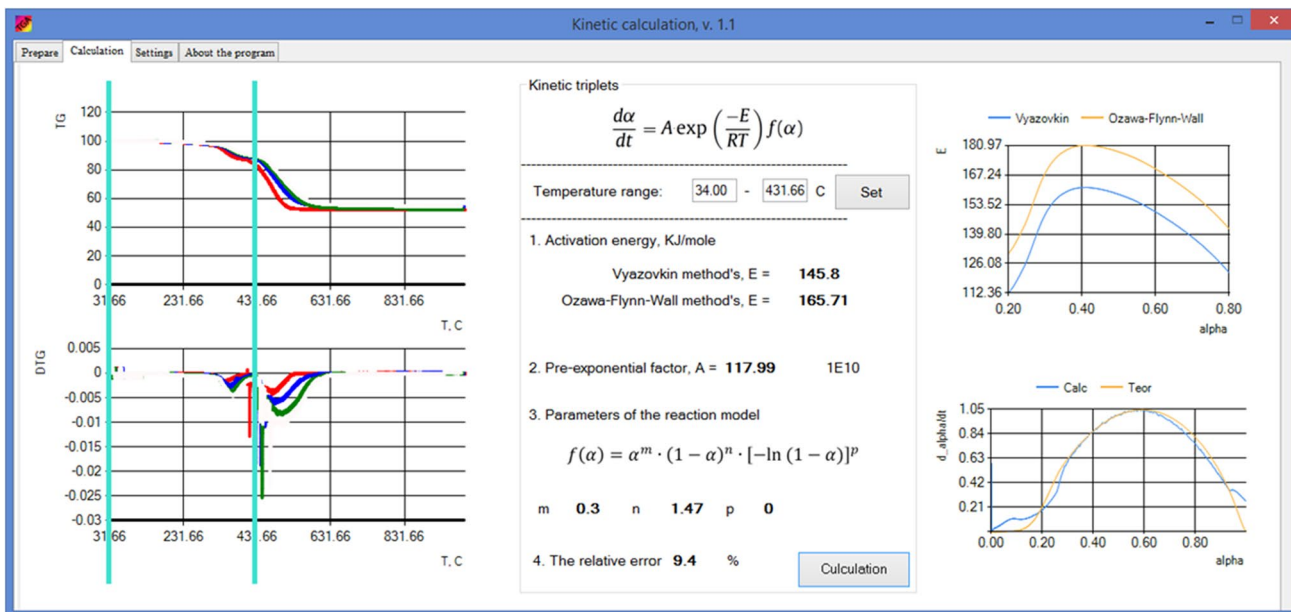


Fig. 5 Kinetic parameter for temperature range 34 – 431 °C

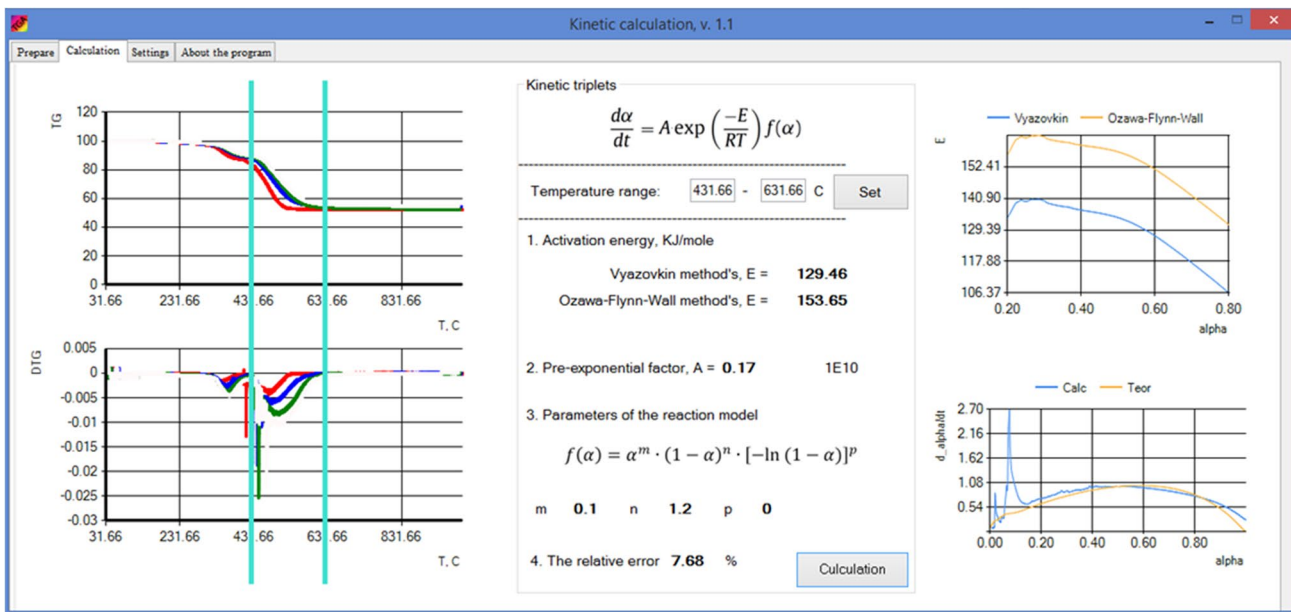


Fig. 6 Kinetic parameter for temperature range 431 – 631 °C

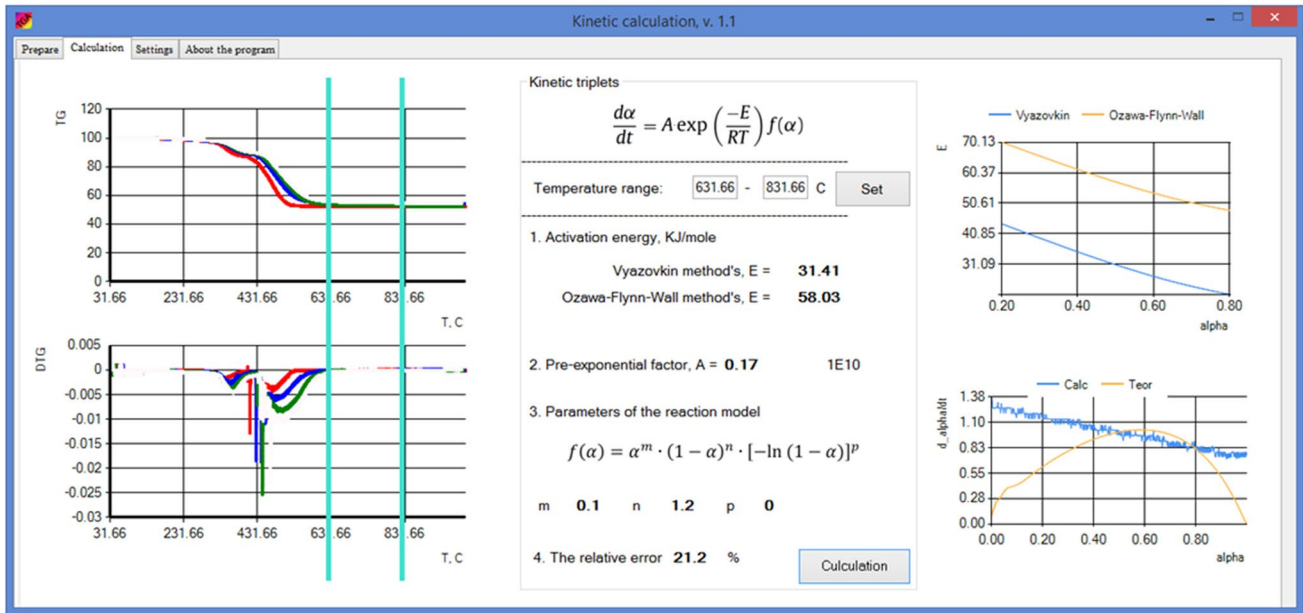


Fig. 7 Kinetic parameter for temperature range 631 – 831 °C

Now the software is run in three different temperature ranges 34–431 °C, 431–631 °C, and 631–831 °C. The results are shown in Figs. 5, 6, and 7. In Figs. 5 and 6, the relative error is less than 10%, indicating that these steps are single-step mechanisms. However, in Fig. 7, the relative error is more than 10%, and activation energy is low compared to earlier steps. It indicates the ending of the degradation process with multiple steps.

The kinetic equations for all the steps can be written as. Temperature range 34 °C—431 °C

$$\frac{d\alpha}{dT} = \frac{117.99 \times 10^{10}}{\beta} \cdot e^{-145800/RT} \cdot \alpha^{0.3} (1 - \alpha)^{1.47} \quad (4)$$

Temperature range 431 °C—631 °C

$$\frac{d\alpha}{dT} = \frac{1.7 \times 10^9}{\beta} \cdot e^{-129460/RT} \cdot \alpha^{0.1} (1 - \alpha)^{1.2} \quad (5)$$

Temperature range 631 °C – 831 °C

$$\frac{d\alpha}{dT} = \frac{1.7 \times 10^9}{\beta} \cdot e^{-31410/RT} \cdot \alpha^{0.1} (1 - \alpha)^{1.2} \quad (6)$$

The activation energy is also calculated as per ASTM E1641 (FWO method) at various conversion degrees. Here the graph has been plotted between the log of the heating rate and the inverse of temperature. The slope of the graph

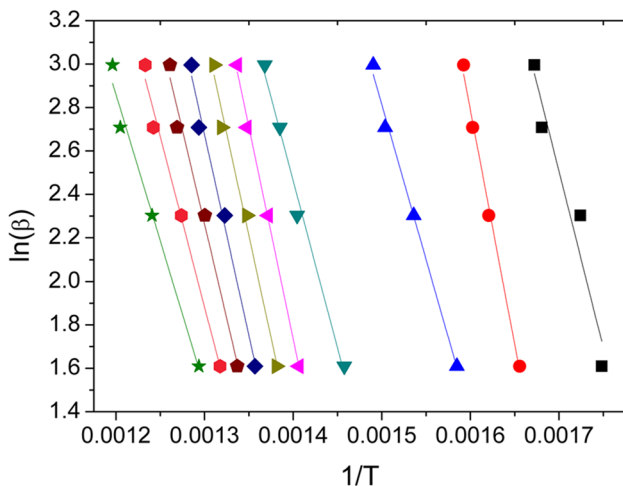


Fig. 8 Log of heating rate vs. inverse of the temperature plot

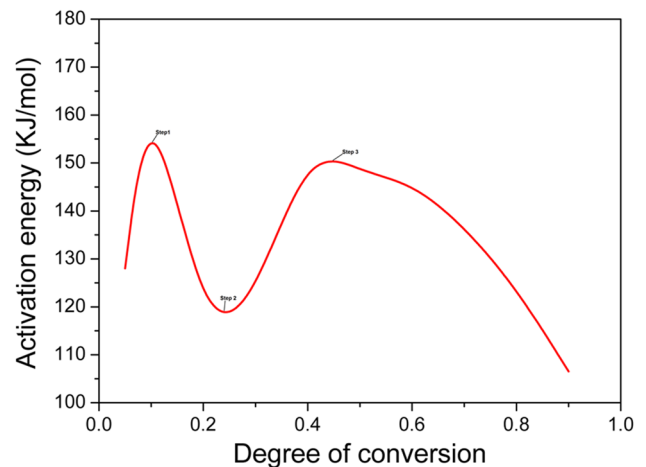


Fig. 9 Activation energy with the degree of conversion

Table 1 Oxyacetylene test results of ablative material

Properties	Value	Formula
Linear Ablation rate	0.039 mm/sec	$LAR = \frac{d_i - d_f}{t}$
Mass Ablation rate	0.050 g/sec	$MAR = \frac{m_i - m_f}{t}$
Back Wall Temperature	104 °C at the end of 200 s	
Ablative effectiveness (J/g)	9.05×10^4 J/g	$AE = \frac{H_f \times t}{\rho \times l}$

calculates the Ea for each degree of conversion. The results are shown in Figs. 8 and 9.

This also gives similar results as from software and confirms multistep thermo-oxidative degradation of ablative material.

4.3 Ablation Studies

The oxyacetylene flame test is done on three samples, and the averages of the three results are given in Table 1.

where,

d_i is the initial thickness of the sample in mm

d_f is the final thickness of the sample after the test in mm and

t is the time of the test in seconds

m_i is the initial mass of the sample in grams,

m_f is the final mass of the sample after the test in gram

H_f is the heat flux applied to the sample in W/cm^2 ($J/sec \cdot cm^2$)

ρ is the density of the sample in g/cm^3

l is ablation or char depth in cm

Fig. 10 Coated sample and SEM images of ablative material

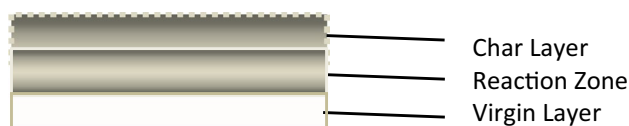
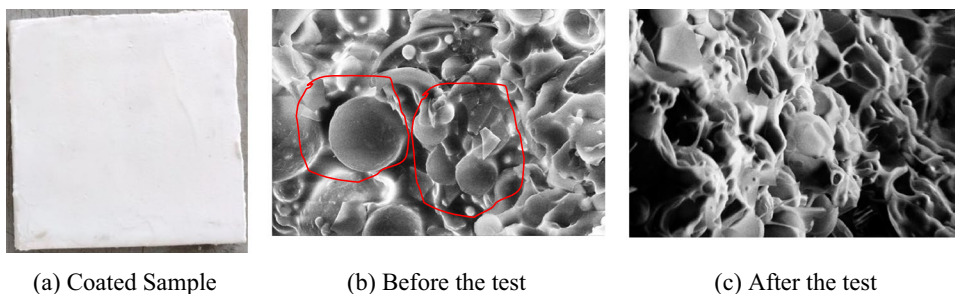


Fig. 11 Schematic diagram of ablative material after ablation

The low linear and mass ablation rate and high ablative effectiveness show that the material has good properties. Figure 10a is coated sample before the test. The SEM images were taken before and after the oxyacetylene flame test (Fig. 10b and c). The test results show before the test (Fig. 10b) that the HGMs are primarily intact and dispersed in the resin, but the HGMs are not intact. They are broken and eroded (Fig. 10c).

4.4 Thermal Decomposition Mechanism

After the ablation, the material consists of the char layer, the reaction zone, and the virgin layer, as shown in Fig. 11 below.

The char layer generates porosity, and hot gases are released after the material's decomposition. The char contains mainly silicon dioxide, which is confirmed by FTIR and EDX analysis of char. The FTIR spectra (Fig. 12) show absorption peaks of stretching at 1261 cm^{-1} and 1075 cm^{-1} , bending at 795 cm^{-1} and rocking at 590 cm^{-1} wave number of the Si–O–Si bonds in SiO_2 [26].

The EDX analysis also shows more silica content than carbon compared to virgin material. (Fig. 13 and Table 2).

The thermo-oxidative degradation occurs in the reaction zone, and its mechanism has been elaborately reported in the literature. It follows the Andrinov mechanism for releasing volatile oxidation products and forming silicon dioxide, as mentioned in Fig. 14 [27, 28].

The significant events in the thermo-oxidative degradation of vinyl silicones are the first initial wt loss due

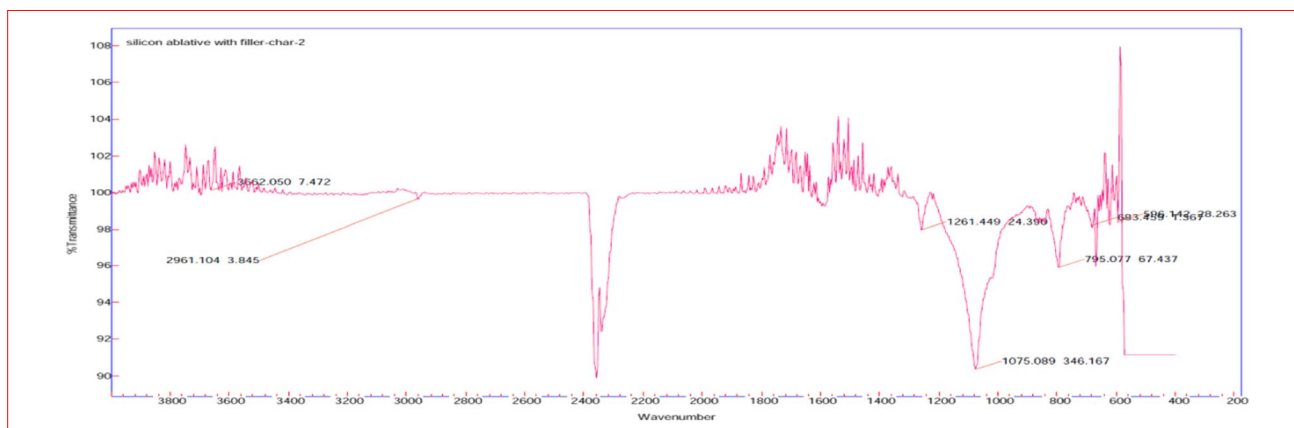


Fig. 12 FTIR spectrum of char after ablation

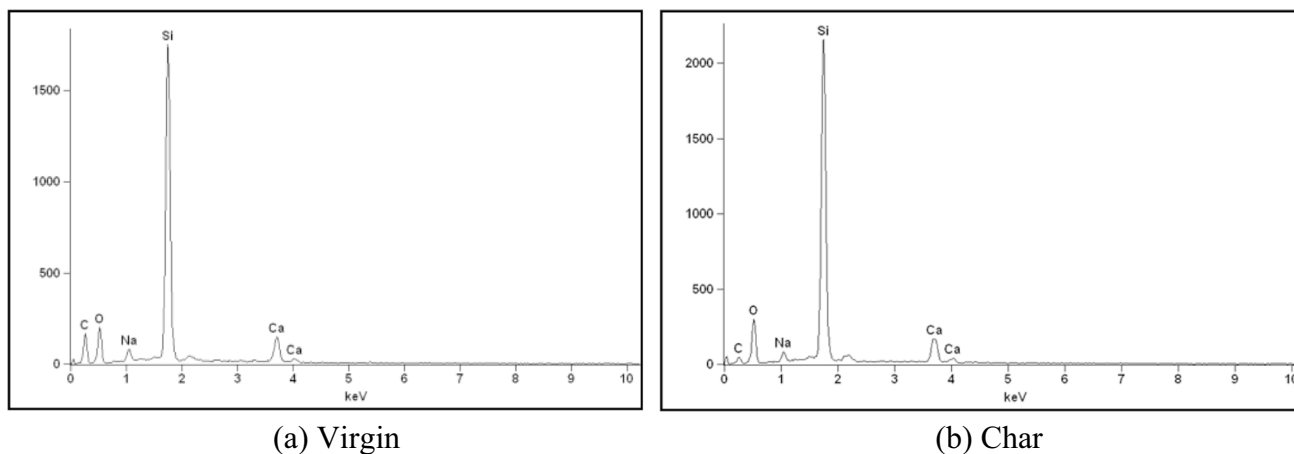


Fig. 13 EDX spectra of ablative material before and after ablation

Table 2 Elemental composition of Ablative material

Type of atoms	Elemental Composition %	
	Before Ablation	After Ablation
C K	34.69	10.03
O K	31.33	43.37
Na K	2.03	2.28
Si K	27.74	37.98
Ca K	4.21	6.34

to solvent removal. The second stage, decomposition at a temperature range of 350 to 440 °C where weight loss is approximately 12%, may be assigned to the vinyl group's breakage and side chains. The third-stage decomposition (i.e., in the temperature range of 440 to 630 °C) is due to the dimethyl siloxane unit of the silicone, where weight loss is about 30%. This decomposition happens in the reaction zone, and the weight residue is constant due to the formation of a hard silica char layer. The relatively low activation energy also indicates that side chain

Fig. 14 Thermo-oxidative degradation mechanism of polysiloxane

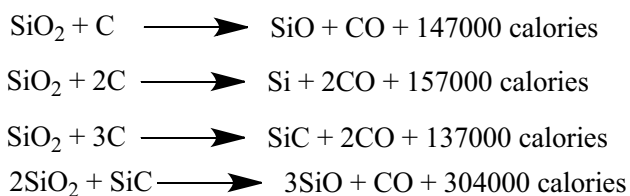
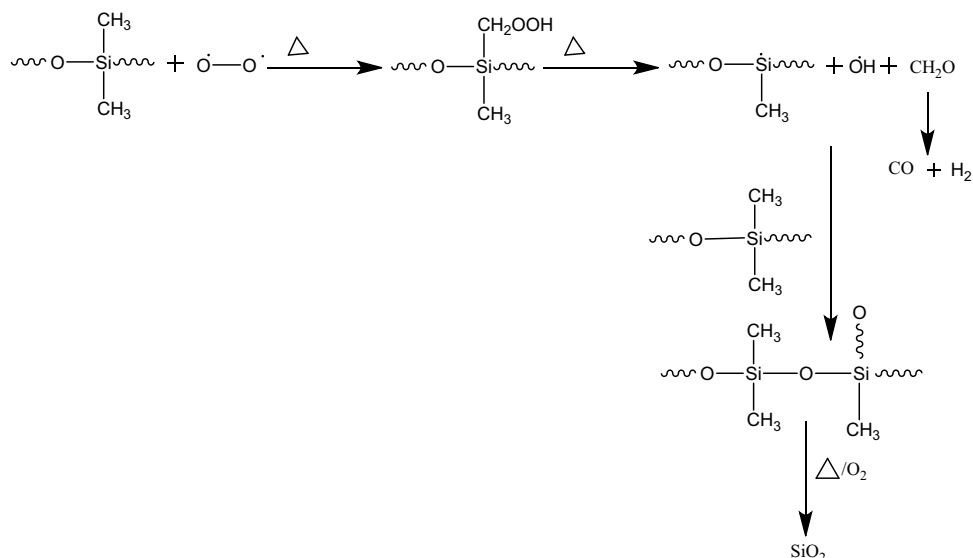


Fig. 15 Reactions between silica char and carbon residue

cleavage occurs in thermo-oxidative degradation because -Si-O- has high bond energy. The reaction between carbon residue and silica is reported at the 1250–1750 °C temperature range [29]. These endothermic reactions (Fig. 15) may occur in the char layer and contribute to transpiration cooling [30].

5 Conclusion

A simple oxyacetylene flame test studies a silicone low-density ablative material's ablative properties for a lengthy application and kinetic parameters are determined using open-source software. The ablative properties show low

linear and mass ablation rates with high ablative effectiveness. So the material is a good candidate for thermal protection of flight hardware in long-duration applications. It is tried here to predict the kinetic model for degradation by thermo gravimetric analysis. The kinetic degradation model is in good agreement with the experimental values shown by the software. The dependency of activation energy with degree conversion also shows the multistep degradation process, and a similar conclusion has been reported in the literature.

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Declarations

This article does not contain any studies with human or animal subjects.

Competing interests The authors declare no competing interests.

Research involving Human Participants and Animals This article does not contain any studies with human or animal subjects.

Statement on the Welfare of Animals This article does not contain any studies with human participants or animals performed by any authors.

Consent to Participate I, Naresh Kumar Katari, on behalf of all authors, hereby declare that we participated in the study and development of this manuscript.

Consent for Publication I have read the final version and consent for the article to be published in your esteemed journal.

Conflicts of Interest The authors declare that no known conflicts of interest are associated with this publication.

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