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Quantification of RTV Intumescence using X-ray Imaging

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Abstract

The thermal degradation and macro-scale response of room-temperature vulcanizing silicone (RTV) to high temperatures and high heating rates is investigated using synchrotron X-ray imaging combined with *in situ* heating under a controlled atmosphere. RTV silicone is generally used for high-temperature sealing, bonding and thermal insulation in extreme environments. On planetary exploration probes, it is used as a gap-filler between ablative Thermal Protection System (TPS) tiles. Intumescence, or swelling due to heat exposure, of RTV has been observed in past experiments simulating atmospheric entry, and was identified as a critical phenomenon to predict when designing a heat shield. In this study, fundamental experiments at low-heating rates (< 64 °C/min) were performed using micro-computed tomography where the full three-dimensional structure of RTV samples was captured at various temperatures. High heating rates relevant to atmospheric entry conditions (>100 °C/min, $\lesssim 5000$ °C/min) were resolved using X-ray radiography to image the bulk morphological changes of the material. A suite of experiments performed in different test gases enabled the quantification of RTV intumescence at different heating rates and environments. The effects of gas environments, degassing, adhesion and porous wicking on the RTV intumescence response were also studied. *In situ* data was correlated with thermogravimetric analysis and mass-spectrometry to understand the underlying chemical changes that cause RTV to intumesce. Intumescence obtained from the *in situ* setup showed good agreement with previous high-enthalpy tests of RTV simulating planetary entry.

Keywords: RTV560, intumescence, synchrotron X-ray tomography, deep learning image segmentation, thermal protection systems, atmospheric entry

1 Introduction

Room temperature vulcanizing silicones (RTV) are widely used as sealants, gaskets, and adhesives due to their favorable mechanical bonding properties, good high-temperature performance and resistance to aging [1–9]. RTV is also used as an adhesive for high temperature applications such as fire protection and in spacecraft’s thermal protection systems (TPS). In the case of TPS, RTV is commonly used as a bonding agent to adhere heat shield tiles to the spacecraft substructure [10]. The RTV560 variant is a high-temperature space grade compound that has been successfully used on NASA missions such as Mars Science Laboratory and Mars2020 as a gap-filler between heat shield Phenolic Impregnated Carbon Ablator (PICA) tiles [11–13]. RTV560 is also used on SpaceX’s Dragon capsule [14], and will be used for NASA’s upcoming mission Dragonfly that will enter the atmosphere of Saturn’s moon – Titan [15]. Recently, Varda Space Industries also adopted a tiled heat shield design with PICA as its TPS solution and RTV as gap-filler [16–18]. The currently ongoing Artemis missions also use a tiled heat shield composed of AVCOAT with gaps filled with a composite mixture of RTV mixed with phenolic microballoons [19].

As the temperature on the surface of atmospheric entry capsules can exceed 2,000 K, differential chemical degradation and ablation occur at the TPS materials-gap filler interface [20]. While the degradation and ablation of PICA are well-characterized [21, 22], they remain poorly understood for RTV [20, 23]. High-temperature experiments performed on RTV have shown significant intumescence, or swelling due to exposure to heat, along with pyrolysis, pore formation, shrinkage and melting [11, 24, 25]. During atmospheric entry, intumescence causes the gap-filled RTV to swell into the freestream flow, acting as a protrusion into the boundary layer which can cause transition of the flow into turbulence and augment heating on the heat shield surface. The protruded “fence” also acts as a localized region exposed to increased heat flux, causing further swelling and melting that deposits over the pristine TPS surface, thereby altering the TPS material response. Hence, characterizing the change in morphology of RTV has been of active interest for its applications in TPS, fire protection, and high-temperature resistance of hypersonic leading-edge materials [20, 26, 27].

Past experimental investigations simulating atmospheric entry heating conditions have shown extensive RTV intumescence causing fencing between tiles and melting [11, 28–30] and have suggested transition to turbulence based on fence heights [31, 32]. Recent experiments on an axi-symmetric sample of RTV friction-fit into a holder of PICA were performed in nitrogen plasma at the Plasmatron X inductively coupled plasma facility [33]. The experiments showed significant intumescence upon heating, causing RTV to swell approximately 120% of its initial height [24]. Fig. 1 shows the PICA-RTV sample during sample insertion and at 52 seconds after plasma exposure. A pronounced protrusion of the charring silicone sample past the sample surface is observed, altering the flowfield by pushing the boundary layer edge upstream. This experiment motivated the need to understand the chemical and micro-scale evolution under high heating rate conditions and their effect on the macro-scale response.

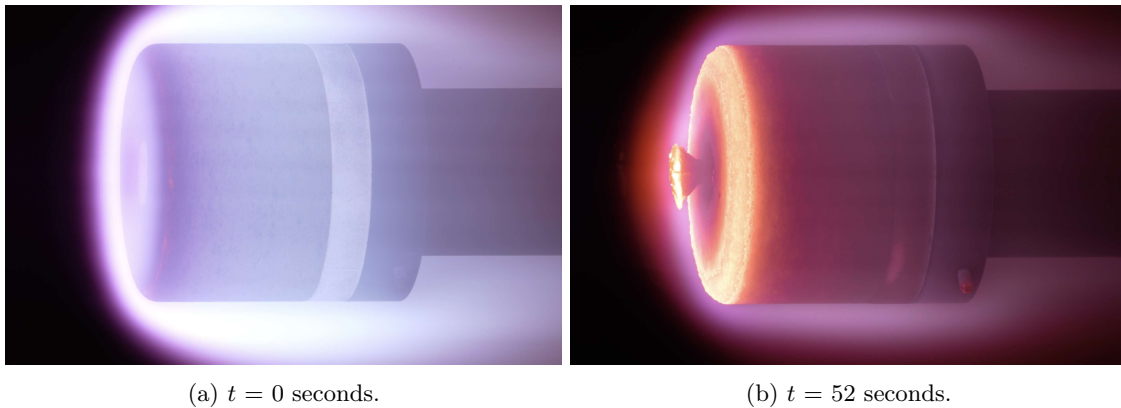


Fig. 1: PICA-RTV sample exposed to nitrogen plasma at the (a) beginning and maximum extent of swelling at 52 seconds, when tested at a cold-wall heat flux of 125 W/cm^2 and a stagnation pressure of 2100 Pa [33].

For evaluating the morphological evolution of RTV with temperature, previous studies employed non-destructive imaging using X-ray micro-computed tomography (μ -CT) studies [25, 34]. In these studies, RTV was heated in an environmental chamber using infrared (IR) lamps while flowing Argon gas, and μ -CT scans were taken *in situ* as the RTV samples were being heated. From the *in situ* μ -CT scans, total volume, porosity, average X-ray attenuation were quantified as a function of temperature, for three different heating rates. The results suggested a tight correlation between volume change and heating rates. However, these experiments were performed at relatively low-heating rates (< 60 °C/min), preventing a full understanding of RTV intumescence, extent of intumescence as a function of heating rate, and correlation to flight-relevant conditions. The present study, therefore, builds on these previous efforts and addresses these phenomena.

The effect of integration of RTV into a porous assembly is also studied. To integrate PICA tiles into a spacecraft TPS, as for example with the forebody heat shield of Mars 2020 and MSL, the liquid precursor of RTV560 is mixed with a curing agent and used to bond the tiles back-face onto the spacecraft substructure. The liquid-RTV is also poured into ≈ 3 mm wide gaps between tiles to serve as a gap-filler. The highly porous structure of PICA promotes the fluid RTV to progressively wick into the tile pores, while it cures and adheres to the PICA surface. Wicking and adhesion therefore alter the properties of both RTV and PICA at the interfaces. The effect of wicking and adhesion on intumescence of RTV into PICA and its carbon-fiber substrate, FiberForm [35, 36], is examined.

The manuscript is organized as follows: RTV pyrolysis decomposition using thermogravimetric analysis and mass spectrometry is presented. Results from low-heating rate *in situ* μ -CT of constrained RTV are presented and correlated to RTV decomposition, and the effect of constraining RTV is discussed. This is followed by high-heating rate *in situ* radiography and comparison with intumescence estimates from the Plasmatron X experiment shown in Fig. 1 [33]. The final section discusses the effect of wicking of RTV into porous media and how porous relief from wicking affects intumescence.

2 Results

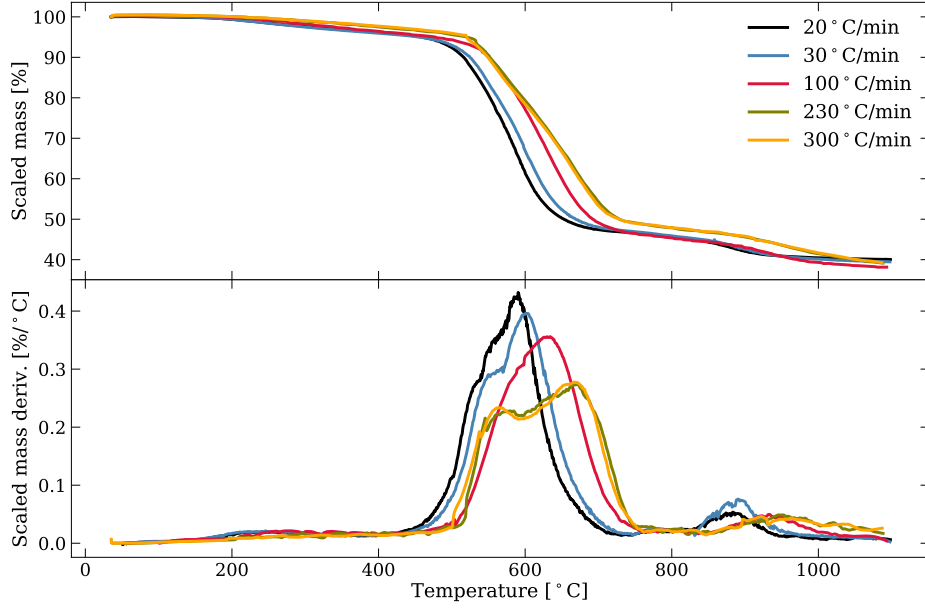
2.1 Pyrolysis decomposition and heating rate dependence

Results from TGA measurements are shown in Fig. 2 for heating rates from 20 to 300 °C/min. The scaled mass derivative at the lowest heating rate of 20 °C/min shows 5 distinct pyrolysis peaks, with the most prominent decomposition occurring at 550 °C (second peak) and 595 °C (third peak). An $\approx 40\%$ char yield is measured and is unaffected by the heating rate. A rightward shift of the decomposition curves towards high temperature is observed at increasing heating rates, showing delay in the onset and progression of the decomposition steps. At higher heating rates of 230 and 300 °C/min, the main pyrolysis decomposition peak splits into two distinct peaks which can be attributed to either a change in reaction order, or diffusion-limited transport of pyrolysis products due to the low material permeability. This is further investigated using MS and intumescence quantification experiments. The heating rate-dependent mass loss suggests a significant effect of pyrolysis delay on the morphological evolution of RTV, and is explored in the following sections.

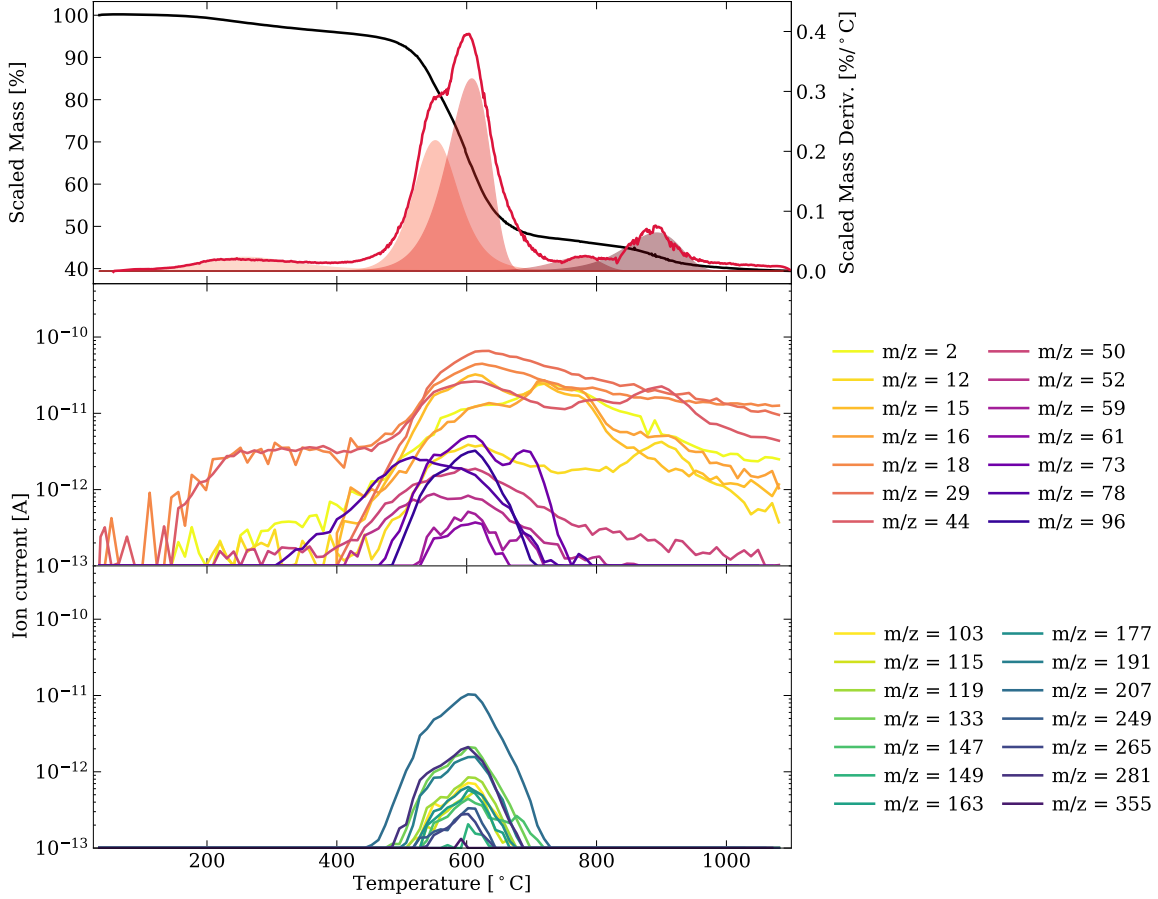
2.1.1 TGA-MS

Mass spectra collected at increasing heating rates are presented in Fig. 2b as a function of temperature for a heating rate of 30 °C/min. TGA-MS results for other heating rates are include in the Supplementary Information. In Fig. 2b, the second and third panels show fourteen predominant pyrolysis species in the $m/z = [0 - 100]$ and $m/z = [100 - 355]$ amu ranges, respectively. Mass spectra reveals the following mechanism:

1. During the first decomposition step (scaled mass $\gtrsim 94\%$), production of H_2 , water, CO_2 and benzene ($m/z = 2, 18, 44, 78$) is measured. Water production is observed from very low temperatures and continues throughout the experiment, indicating adsorbed water on the surface of RTV that is thermally desorbed. At higher temperatures, H_2 , CO_2 and benzene production is observed, which is attributed to the devolatilization of the poly-dimethyl siloxane (PDMS) resin in RTV. Benzene production indicates the presence of poly-diphenyl siloxanes (PDPS) which are commonly added for thermal stability and controllability of polymerization to PDMS-based compounds [37–39]. Additionally, as RTV production using DBT catalyst can produce H_2 during curing [9, 40–42], H_2 could be present in nano-scale closed-pores. As porosity increases with



(a) TGA curves at heating rates ranging from 20 to 300 °C/min.



(b) Mass spectra of key species from TGA-MS at 30 °C/min.

Fig. 2: TGA-MS of RTV.

pyrolysis advancement (as seen previously using *in situ* scanning electron microscopy [24]), H_2 and water initially contained in closed pores, are exposed and outgassed. While water desorption occurs from the beginning of the experiment, H_2 , CO_2 and benzene production is seen to occur with the onset of pyrolysis, thereby occurring at higher temperatures with increasing heating

rates. Note that H_2 detection from mass spectrometry is a very common and well-known occurrence due to the ionization of hydrogen containing molecules (H_2O , C_xH_y , etc.) prior to arriving at the detector [43]. The H_2 production observed therefore could originate from many sources in the sample and cannot solely be attributed to the release of trapped H_2 in the pores.

2. The second decomposition step ($94\% \gtrsim$ scaled mass $\gtrsim 80\%$) leads to the production of H_2 , water, C, CO, CO_2 , benzene along with breakage of the PDMS backbone, leading to formation of large, volatile, cyclic PDMS compounds ($96 \leq m/z \leq 355$). Production of methyl-functional group and methane ($m/z = 15, 16$ amu) from the PDMS and PDPS is also observed, which could occur as the volatile gases convect through the transfer line from the TGA to the MS. The cyclic compounds are labeled based on mass-spectra reported by Smith *et al.* [2, 44] and are tabulated in Table 1 of the Supplementary Information.
3. The third decomposition step ($80\% \gtrsim$ scaled mass $\gtrsim 50\%$) causes further outgassing of the cyclic compounds. Furthermore, production of a variant of phthalates ($m/z = 149$) is observed during the third reaction. As phthalates are common plasticizers used in manufacturing rubber and plastic compounds [2, 44], outgassing of the plasticizer could indicate rigidization of RTV, consistent with past observations that indicated hardening of RTV60 after 700°C [24]. Additionally, carbothermal reduction of hematite (Fe_2O_3) to magnetite (Fe_3O_4) occurs around 650°C causing CO_2 production [24, 45].
4. With the onset of the fourth decomposition step ($50\% \gtrsim$ scaled mass $\gtrsim 46\%$), a prominent spike in production of m/z of 59, 73 and 149 amu is observed indicating further decomposition of the polysiloxane resin and further outgassing of the plasticizer. Chacon *et al.* also note a possible polymer-to-ceramic transition in this regime, causing the RTV to significantly harden, forming a rigid char [45].
5. The final decomposition step (scaled mass $\lesssim 46\%$) produces C and CO_2 . Chacon *et al.* attribute this to the production of CO_2 from the flux-calcined soda-ash in RTV above 800°C and reaction of CO_2 with the carbonaceous char to produce CO [45].

2.2 In situ Micro-CT at low heating rates

Fig. 3 shows cross-sections of RTV heated at $30^\circ\text{C}/\text{min}$ in an Argon environment. At room temperature, RTV does not show any microstructural features, other than closed porosity from the initial mixing/curing process. As the sample is heated to $\approx 500^\circ\text{C}$, a bulging of the sample is observed, with no observable change in microstructure. At 535°C , the RTV intumesces further, developing cracks and pores followed by shrinkage due to pyrolysis. Further heating leads to significant foaming of the material due to pyrolysis. Therefore, starting from an initially non-porous, impermeable material, RTV is seen to intumesce, foam, shrink and form a highly porous, permeable char.

The evolution of volume, porosity and height of the constrained sample as a function of temperature are shown in Fig. 3, along with those for an unconstrained sample heated at the same heating rate from previous experiments [25]. The TGA derivative at $30^\circ\text{C}/\text{min}$ is also co-plotted for reference. Firstly, Fig. 3b shows that both unconstrained and constrained RTV samples first swell at a nearly linear rate, primarily attributed to thermal expansion. This is followed by pressure buildup within the closed pores leading to nonlinear increase in volume. Past this phase the material starts shrinking significantly due to advancement of pyrolysis, causing porosity development and mass loss. Volume of the unconstrained RTV is seen to linearly rise only until the end of the first pyrolysis reaction, followed by shrinkage, suggesting that the swelling in RTV is due to thermal expansion and closed porosity (trapped H_2 , water and air bubbles). Once the first pyrolysis reaction is completed, macro-pores are developed within RTV causing permeable pathways to form within the material, relieving the closed-pore gas pressure and causing the RTV to shrink. In the case of the constrained sample, the volume increases until the onset of the second pyrolysis reaction and a delay in the onset of shrinkage is observed due to a delay in formation of permeable pathways to crack and outgas.

Porosity and height of RTV as a function of temperature (shown in Figs. 3c and 3d, respectively) also exhibit a delay for the constrained sample, aligning with the onset of the second pyrolysis reaction. In summary, augmented swelling and delay in shrinkage are observed in the case of constrained samples compared to unconstrained samples.

2.2.1 Heating Rate Dependence

Unconstrained and constrained RTV samples were tested at 13, 30 and $64^\circ\text{C}/\text{min}$. Volume, porosity and height of the samples for the three heating rate experiments are shown in Fig. 4. Volume and

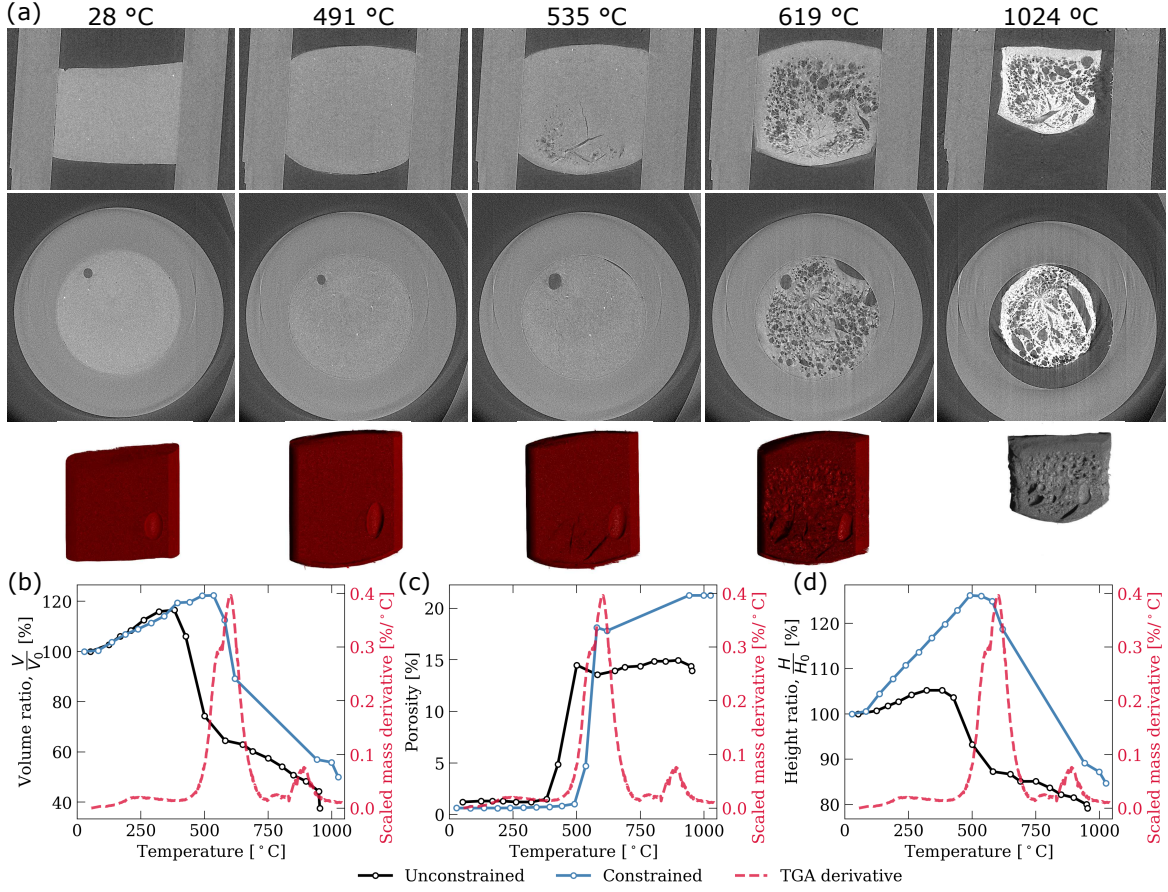


Fig. 3: (a) Vertical and horizontal cross-sections along with 3D slices of *in situ* μ -CT of RTV at 30 °C/min, along with (b) total volume, (c) porosity and (d) maximum height of RTV as a function of temperature, compared with the TGA derivative for unconstrained (black) and constrained samples (blue).

height ratios show a clear heating-rate dependence, with the curves shifting to higher temperatures with increasing heating rates due to a delay in pyrolysis at higher heating rates. The maximum volume and height ratios also increase with heating rate, caused by an increase in closed-pore pressure at higher temperatures due to the delayed pyrolysis. Increased heating rate causes a delayed pyrolysis onset, which in turn may lead the closed and open-pore gases to heat to higher temperatures. The increase in temperature then augments pore-pressure before pyrolysis can occur, thereby causing the RTV to swell to a higher extent. Porosity as a function of temperature (in Fig. 4b) shows that RTV is initially non-porous, but porosity drastically increases at temperatures around 300 °C with the onset of pyrolysis. Additionally, the final porosity is seen to be a function of the heating rate. Height of the RTV samples as a function of temperature also shows a similar heating rate-dependent behavior as seen in Fig. 4c)

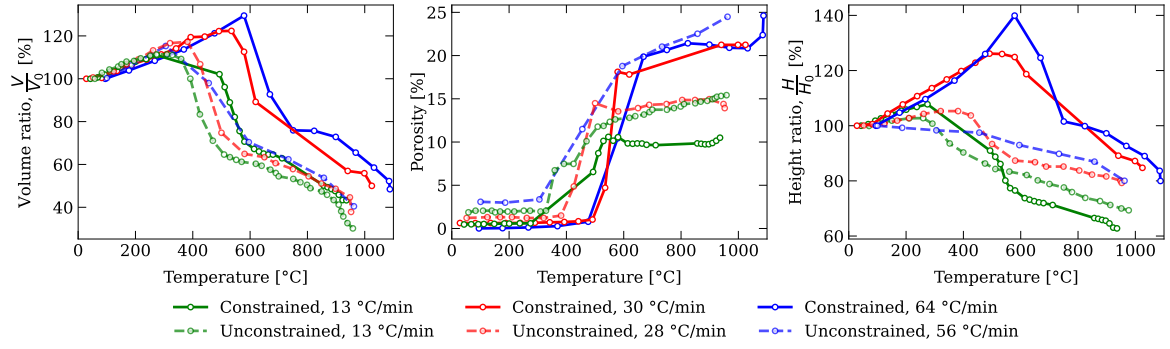
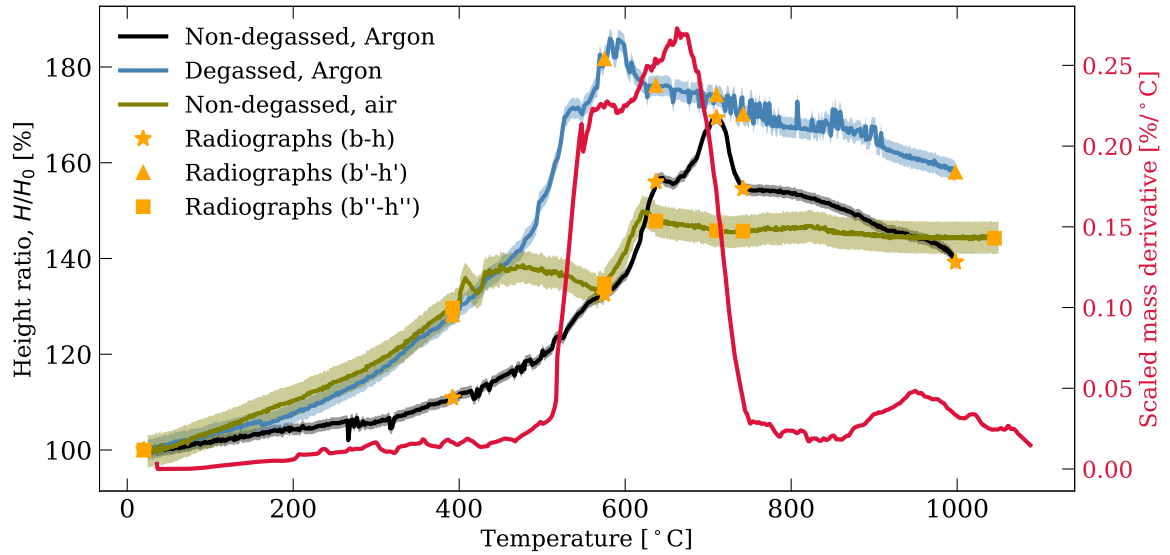


Fig. 4: Material quantities vs temperature, compared with TGA.

2.3 *In situ* Radiography at high heating rates

2.3.1 Intumescence at 228 °C/min

Fig. 5a shows the percent swelling, expressed as height ratio ($= \text{current}/\text{initial height} \times 100$), as a function of temperature for RTV samples heated at 228 °C/min co-plotted with the TGA derivative at 230 °C/min. Fig. 5(b-g) show the radiographs of the two samples at key temperatures. The behavior of non-degassed and degassed samples in argon and air are separately discussed below.



(a) Height ratio of RTV heated at 228 °C/min along with the derivative from TGA collected at 230 °C/min (in red). Error bands indicate uncertainty due to initial tilt of the sample.

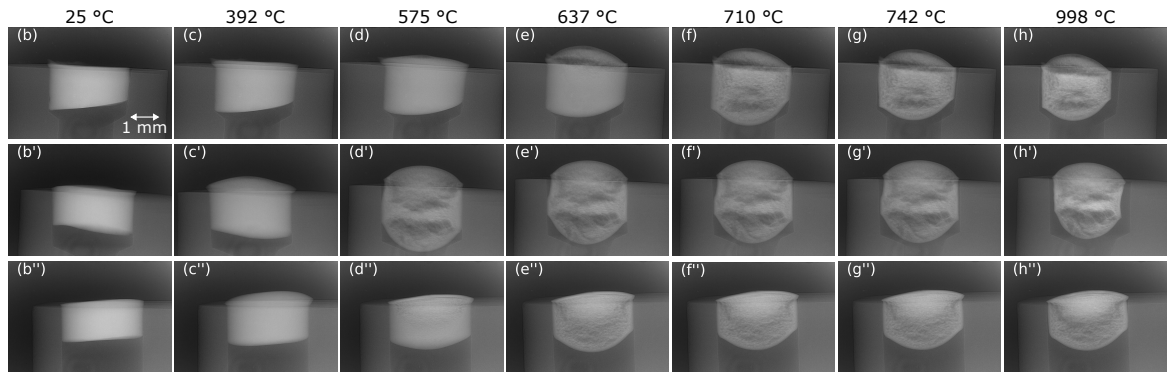


Fig. 5: Height ratio and radiographs of (b-g) non-degassed and (b'-g') degassed RTV in Argon along with (b''-g'') non-degassed RTV in air at key temperatures when heated at 228 °C/min.

Non-degassed RTV in Argon

The RTV sample first expands due to thermal expansion and buildup of gas pressure within closed pores at $T \leq 575$ °C (Fig. 5(b-c)). The height ratio curve up to 575 °C is similar to those from the *in situ* μ -CT data showing $\approx 20\%$ height increase. At 575 °C, a steep rate of swelling is observed, which corresponds to the onset of the second pyrolysis reaction (Fig. 5(d-f)). Contrastingly, in the 30 °C/min μ -CT experiments, shrinkage of the RTV was observed with the onset of the second pyrolysis step. This discrepancy is attributed to the large temperature gradient within the RTV in the top-heating configuration, causing it to bulge and crack layer-by-layer in order to outgas. After the third pyrolysis reaction, the RTV relieves its gas pressure causing it to shrink (Fig. 5(f-g)). Finally, the fourth and fifth pyrolysis reaction causes further shrinkage of the RTV (Fig. 5(g-h)).

Degassed RTV in Argon

For degassed RTV, the height ratio shows an initially linear expansion with a slope (i.e., coefficient of thermal expansion) greater than that of the non-degassed sample. With the onset of the first pyrolysis reaction, the sample swells significantly as seen in Fig. 5(d'), due to presence of only closed porosity and absence of permeable, nano-scale pathways due to the degassing process [24, 46]. Furthermore, pore pressure buildup is seen to occur throughout the RTV sample instead of occurring layer-by-layer originating from the top of the sample (Fig. 5(d'-g')). After expansion, the degassed RTV is seen to undergo pyrolysis shrinkage and reduce in height by $\approx 20\%$. Overall, the extent of intumescence is higher for the degassed RTV than that of the non-degassed RTV.

Non-degassed RTV in Air

The height ratio curve in air is seen to initially follow that of the degassed sample in Argon and to depart to a slower rate of swelling compared to the degassed sample in Argon at $T > 500$ °C. Radiographs above 575 °C show the formation of a dense surface scale which persists throughout the test (Fig. 5(d''-h'')). This is a glassy layer formed due to the interaction of RTV with atmospheric oxygen. The glassy layer is expected to significantly rigidize the top surface of the sample, inhibiting foaming, intumescence and shrinkage. Post-test scanning electron microscopy and energy dispersive X-ray spectroscopy (SEM/EDS) confirmed the formation of a silica scale on the surface of the sample. SEM/EDS results are presented in Supplementary Information.

2.3.2 Effect of heating rate

Height ratios for different heating rates are shown in Fig. 6 for (a) non-degassed and (b) degassed samples tested in Argon, along with (c) non-degassed samples tested in air, respectively. Height ratios curves for the non-degassed samples show a rightward shift similar to that observed in the *in situ* μ -CT (Fig. 4(c)) due to delayed pyrolysis onset. A pronounced height augmentation with heating rate is also observed with increasing heating rate, causing RTV to swell up to 220% of its initial height (120% growth in height). After maximum swelling, the RTV samples shrink by $\approx 20\%$ due to gas pressure relief and pyrolysis shrinkage. A similar rightward and upward shift in height ratio curves can be observed for the degassed samples and the non-degassed air test samples, as seen in Fig. 6b and Fig. 6c, respectively.

For the air tests, however, the height ratio curves show that the samples exhibit swelling with increase in temperature but not shrinkage after pyrolysis. Post-test tomographies revealed the air samples to form a glassy layer coating of a silicate or silicon-oxycarbide around the sample, causing it to trap pyrolysis gases. Additionally, post-test observations showed a rigid, white coating formed around the sample and in the sample enclosure, indicating that the RTV reacted with the ambient air, forming a glassy layer on and around the RTV surface.

Fig. 6d presents the maximum height ratio ($\frac{H_{max}}{H_0}$) of non-degassed and degassed RTV tested in Argon and non-degassed RTV tested in air, along with those from the *in situ* μ -CT campaign in Argon. For non-degassed RTV in Argon, $\frac{H_{max}}{H_0}$ from *in situ* μ -CT and *in situ* radiography are seen to closely correspond. With increase in heating rate, an initially linear increase in $\frac{H_{max}}{H_0}$ is observed (13 °C/min < rate < 228 °C/min) followed by a plateau up to 1000 °C/min and a rise beyond 1000 °C/min. The degassed samples in Argon show higher $\frac{H_{max}}{H_0}$ than those of the non-degassed, indicating the adverse effect of degassing the RTV samples. In air, the non-degassed $\frac{H_{max}}{H_0}$ follows a similar trend. In all three cases, $\frac{H_{max}}{H_0}$ reaches $\approx 220\%$ (i.e., 120% growth) as the highest heating rates are approached.

The final height ratios ($\frac{H_{final}}{H_0}$) at ≈ 1050 °C (i.e., post-shrinkage) are shown in Fig. 6e for the three cases. While $\frac{H_{final}}{H_0}$ of non-degassed and degassed samples in Argon are lower than $\frac{H_{max}}{H_0}$,

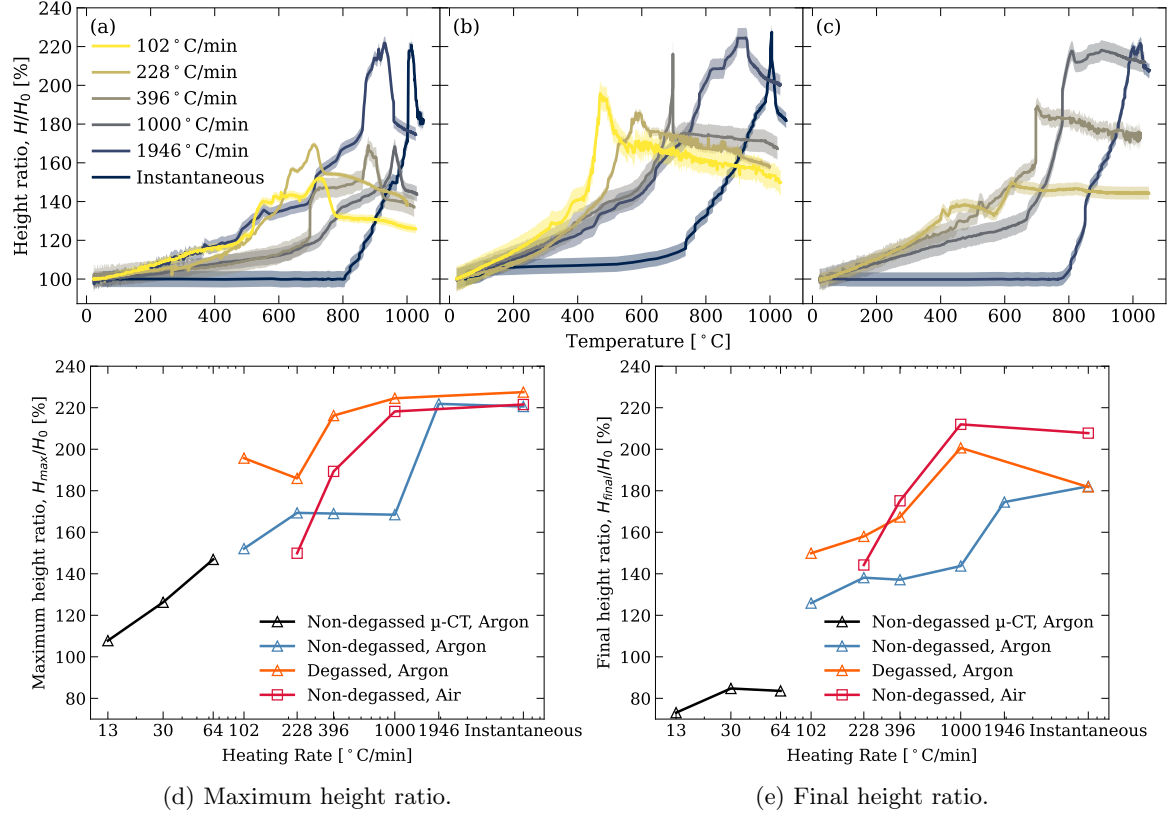


Fig. 6: Height ratio for (a) non-degassed and (b) degassed RTV as a function of temperature for different heating rates when tested in Argon and (c) air. (d) Maximum and (e) final height ratio of samples as a function of temperature for various heating rates.

$\frac{H_{final}}{H_0}$ for air are approximately similar to $\frac{H_{max}}{H_0}$. Therefore, using *in situ* X-ray imaging, the extent of intumescence of three different configurations of RTV was quantified as a function of heating rate.

2.3.3 Correlation to Plasmatron X tests

The height ratio of RTV friction-fit into a PICA holder exposed to nitrogen plasma in the Plasmatron X facility at 125 W/cm² cold-wall heat flux and 2100 Pa stagnation pressure (shown in Fig. 1) along with the height ratio curves from the *in situ* radiography experiments are shown in Fig. 7. The height ratio curve from Plasmatron X follows a similar trend to that from the *in situ* radiography experiments, exhibiting initially linear expansion followed by a steep rate of swelling due to pressure buildup. The extent of intumescence in the Plasmatron X facility is 120% of its initial height, similar to that noted at the highest heating rates in the *in situ* radiography tests. Therefore, good qualitative agreement of the height ratio curves between the two facilities suggests that RTV intumescence in high-enthalpy environments is driven by the high-heating rates of the plasmas, causing the RTV to drastically intumescence due to thermal expansion and pressure buildup.

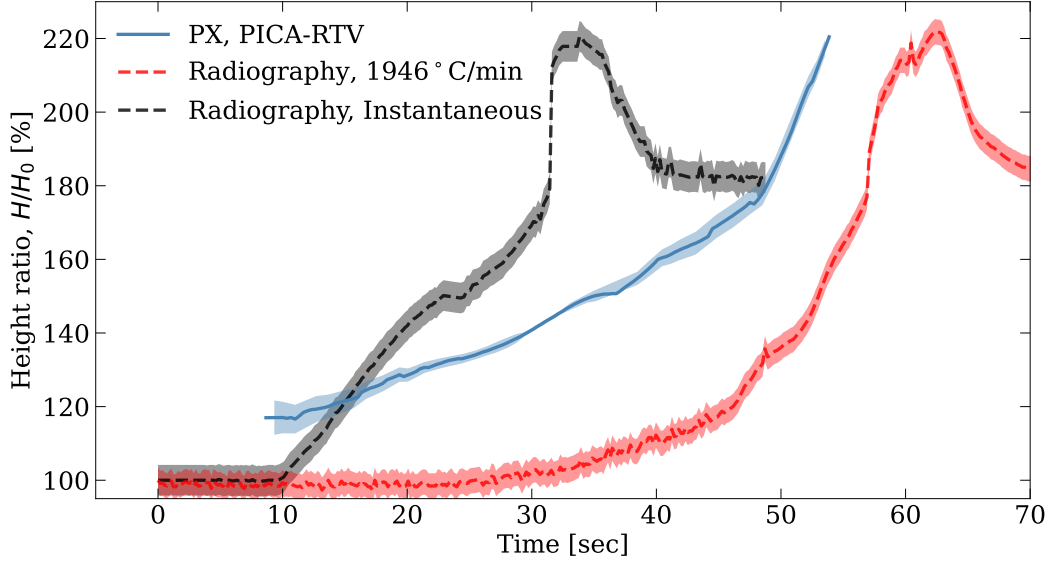


Fig. 7: Height ratio of RTV intumescence in the Plasmatron X high-enthalpy test, along with that from the *in situ* radiography tests.

2.4 Wicking of RTV into porous TPS

The next set of experiments mimics the actual configuration of RTV as a gap filler between porous tiles. The swelling behavior is analyzed for RTV within both FF and PICA sample holders.

Fig. 8 shows cross-sections of the room temperature sample of RTV wicked into (a-a') FF and (b-b') PICA. The extent of wicking was approximately 500 μm and 150 μm in FF and PICA in all directions, respectively, for a sample diameter of 3 mm, i.e., approximately 33% and 10% of wicking. Higher extent of wicking is observed in the case of FF due to its higher porosity and permeability compared to that of PICA. In the case of FF, the seepage of RTV near the edges of the sample into FF causes porosity development within the RTV as seen in Fig. 9(a). Formation of such open porosity causes the permeability of virgin RTV to increase, aiding with gas pressure relief upon heating. Similar porosity development on the periphery of the RTV sample cured into the PICA holder can be observed. Fig. 8(b') also shows a gradient in grayscale value and porosity in the RTV due to wicking. The higher grayscale value at the interface indicates densification of the RTV at the interface which is attributed to co-polymerization of the liquid RTV with the PICA. Its detailed chemistry needs to be further investigated. Additionally, within the PICA holder, the resin phase becomes more visible due to higher X-ray attenuation from the wicked RTV.

Radiographs from instantaneous heating of friction-fit graphite-RTV along with wicked samples of FF-RTV and PICA-RTV at various key temperatures are shown in Fig. 9a. Unlike the graphite-RTV case that exhibited significant bulging, cracking and outgassing, the FF-RTV and PICA-RTV samples show that the RTV bulging is localized to the surface due to adhesion of the RTV on its periphery to the holder, due to porous relief from wicking. A clear pyrolysis front, indicated by a reduction in the grayscale value due to porosity formation, is observed in the wicked samples. In the case of FF-RTV, the pyrolysis front propagates from the periphery towards the center of the sample, causing the RTV to swell locally. Cracking of the RTV sample is observed at 1010 $^{\circ}\text{C}$ indicating further gas-pressure relief within the sample. Similarly, the PICA-RTV sample shows a pyrolysis-front propagating from the top of the sample, causing the RTV to expand locally. Adhesion of the RTV to PICA causes it to crack which relieves gas-pressure and reduces intumescence.

Fig. 9b shows the temperature profiles from the calibration experiments for FF-RTV and PICA-RTV when heated instantaneously, compared with profiles from the friction-fit graphite-RTV calibrations. Firstly, the FF-RTV TC1 and TC2 temperatures are higher when compared to graphite-RTV temperatures due to the lower thermal conductivity of FF causing more localized heating on the top of the sample. On the other hand, for the PICA-RTV sample, both TC1 and TC2 temperatures are much lower than the graphite-RTV and FF-RTV temperatures, expected due to the high pyrolysis blowing of PICA cooling the sample and contaminating the sample enclosure, thereby blocking the IR radiation from reaching the sample surface.

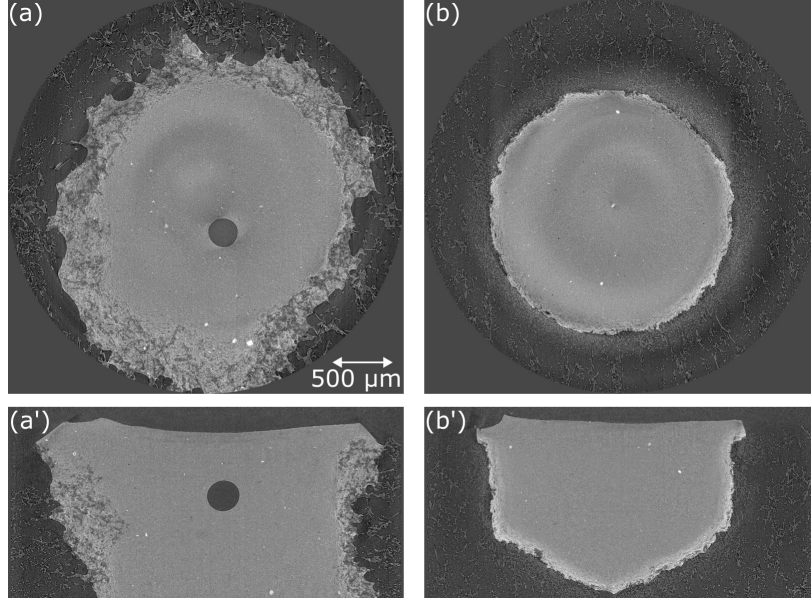


Fig. 8: Cross-sections of RTV wicked into a (a-a') FF holder and (b-b') PICA holder.

Fig. 9c shows the height ratios as a function of TC1 temperature. It is evident that due to wicking of RTV into the porous TPS, the extent of intumescence is significantly reduced in the FF-RTV and PICA-RTV cases, with only $\approx 25\%$ and $\approx 17\%$ growth in sample height, respectively. Due to adhesion and formation of permeable pathways within RTV, gas-pressure buildup from heating is alleviated, causing the RTV to intumescence to significantly reduce. Additionally, as the PICA-RTV sample experienced a lower heating rate compared to the FF-RTV and Graphite-RTV samples, the height ratio curve is seen to shift leftward compared to the other two cases.

3 Materials and Methods

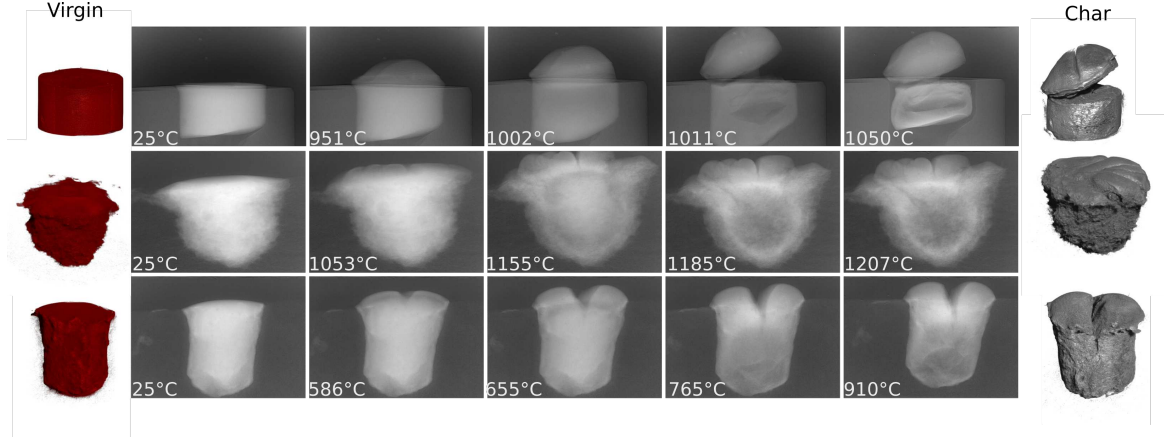
3.1 RTV560

Room temperature vulcanizing silicone (RTV560) was used for these experiments [46]. RTV was cured following the standard curing procedure recommended by Momentive Inc. [46]. The liquid silicone compound RTV was poured into a container and mixed with $\approx 0.3\%$ -weight dibutyltin dilaurate (DBT) as a standard curing agent. The mixture was allowed to cure at room-temperature for 24 hours, resulting in a solid rubber. Air entrapment during mixing of the base compound with the curing agent caused the formation of trapped air bubbles. Both visual observation of sectioned specimens and microscopy images showed these features in the form of closed pores [24]. Furthermore, hydrogen gas is produced as a byproduct of the DBT reacting with the RTV [9] which could also be present as nano-scale closed porosity in the RTV. As degassing while curing is recommended by Momentive Inc. [46], an additional batch of RTV was mixed following the above approach and placed in a degassing chamber (≈ 26 mm of Hg) for 24 hours while it cured. The degassing step caused the RTV to first foam and collapse, removing macro-scale porosities from trapped air bubbles. The nano-scale pores however remained [24].

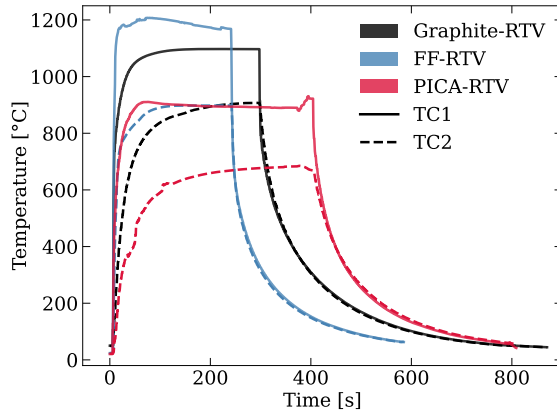
The experiments conducted and analyses presented in this manuscript were performed on samples of ≈ 3 mm characteristic size. To study the effect of wicking and adhesion of RTV, liquid RTV mixed with the DBT catalyst was poured into holders of PICA and its carbon-fiber preform, FiberForm (FF). Cured samples of RTV in both FF and PICA were imaged at higher resolutions to observe microstructural changes in RTV due to wicking.

3.2 Thermogravimetric Analysis with Mass Spectrometry

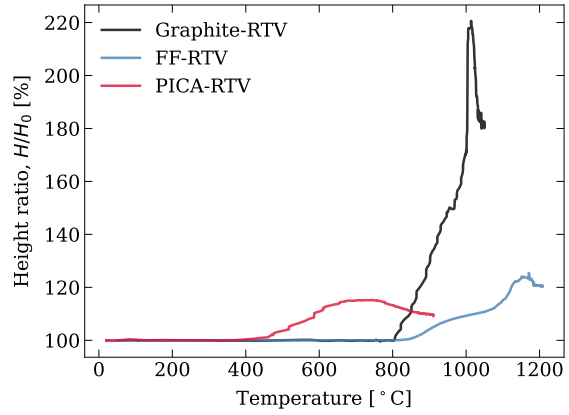
TGA coupled with mass spectrometry (MS) was performed to analyze the pyrolysis gas production due to thermal decomposition, at increasing heating rates. The measurements were performed using a Netzsch thermogravimetric analyzer (TG309 TG Libra Supreme, Netzsch Analysis & Testing, Germany) connected to a mass spectrometer (QMS403, Netzsch Analysis & Testing, Germany), equipped



(a) Radiographs at various temperatures from instantaneous heating of RTV in friction-fit graphite, FF and PICA, respectively.



(b) Temperature response of RTV in different holders.



(c) Height ratio vs temperature.

Fig. 9: (a) Radiographs at key temperatures, (b) temperature response from thermocouples, and (c) height ratio as a function of temperature, when friction-fit or cured into a holder and heated instantaneously.

with a secondary electron multiplier that amplifies the signal of incoming ions, enabling identification up to mass/charge (m/z) ratios of 512 amu. The ionization energy for the mass spectrometer was 70 eV. Nitrogen was used as both the ambient and carrier gas, and corresponding spectral signals at 14 and 28 amu were omitted from the analysis.

Sample masses of 11 ± 1 mg were selected based on the resolution of the TGA scale and the accuracy of the results, and used consistently across the experiments. Before each experiment, the TGA furnace was maintained at 35 °C and nitrogen was flowed for 10 minutes to remove ambient environmental gases. After the 10 minute purge, TGA-MS was performed at heating rates of 20, 30, 100, 230 and 300 °C/min, up to a maximum temperature of 1100 °C. A “fast” bar-scan mode was used with an exposure time of 20 ms per amu. During the experiments, temperature was controlled based on the signal of a thermocouple in contact with the alumina crucible that housed the RTV sample.

3.3 Environmental chamber and *in situ* heating

The environmental chamber and operating procedure in this study were the same as those described in previous experiments [25, 34, 47], shown in Fig. 10a. To enable controlled pyrolysis, the environmental chamber was tested for leak tightness, purged, and fed with a constant Argon flow of at 0.091 g/s. Argon purges were then performed to flush the chamber from the pyrolysis products. The chamber was assembled using a quartz tube enclosure, housing a graphite stub that served as crucible for the RTV sample and mounted atop an alumina tube. A type-K thermocouple was routed through a

custom vacuum feedthrough into the chamber and interference-fit into the center hole of the graphite crucible. The thermocouple was maintained outside the X-rays field of view, in order to minimize artifacts from the highly-attenuating metal wire.

The sample enclosure was surrounded by confocal arrangements of either 3 or 6 infrared lamps (L6409-G, International Light Technologies, Massachusetts USA) (Fig. 10b) with 56 mm focal radius. This configuration allowed to heat up cylindrical RTV samples up to temperatures of 1100 °C (Fig. 10c), similar to previous *in situ* experiments [48–52]. Heating rates ranging from 13 °C/min to 1946 °C/min were achieved by controlling the current delivered by two power supplies (30A/24V each), each feeding three lamps.

For low heating rate experiments of 13, 30 and 64 °C/min, side heating with 6 IR lamps was used. For higher heating rate experiments (100 to 1946 °C/min), top heating with 3 IR lamps was used. Both setups are explained in detail in § 4.

3.4 X-ray Imaging

In situ tomography and radiography experiments were performed at Beamline 8.3.2 of the Advanced Light Source (ALS) at Lawrence Berkeley National Laboratory [49, 50, 53, 54]. The environmental chamber was mounted on the 0° to 180° rotation stage of the μ -CT setup and placed in-line with the X-ray beam generated by the synchrotron source. For the *in situ* experiments the beam was operated in white light mode (no dispersion from the monochromator) with a critical energy of 11.5 keV, such that tomographies could be taken in rapid succession at ≈ 1 tomography/min. The X-ray flux attenuated by the sample was converted to visible light by means of a 0.05 mm LuAG scintillator (Crytur, Turnov, Czech Republic) and collected by a 2560×2160 pixels pco.edge 5.5 sCMOS camera (PCO, Kelheim, Germany). To adequately resolve the microstructure of the samples, the *in situ* tests were captured with a 2× lens (Mitutoyo, Kawasaki Japan), providing 3.25 μ m/pixel resolution.

For tomography experiments, the exposure time was set to 20 ms per frame and a total of 1313 radiographs were acquired over the 180° rotation, along with 10 bright fields and 10 dark fields. The sample to scintillator distance was kept at 115 mm, which was the minimum amount feasible given the *in situ* setup, to minimize phase-contrast and preserve resolution. Successive tomographies were collected while ramping the temperature of the chamber, providing time-resolved 3D behavior of the samples.

For radiography experiments, the exposure time was set to 12 ms per frame and images were collected continuously with a 5 ms interval. Radiographs were collected while heating and cooling the samples along with pre/post-heating and post-cooling tomographies which were used for computing initial and final volumes.

4 Experimental Protocol

Two experimental campaigns were performed; low-heating rate *in situ* μ -CT (13 to 64 °C/min), and high-heating rate *in situ* radiography (100 to 1946 °C/min). The protocol for both campaigns are detailed as follows.

4.1 Micro-CT at low-heating rates

RTV was heated using the 6-lamp setup shown in Fig. 10b at heating rates of 13, 30 and 64 °C/min, and tomographies were acquired at ≈ 60 second intervals. For these experiments, RTV was constrained in a quartz tube in order to approximate one-dimensional swelling and to mimic RTV in a heat shield gap-filler configuration. Behavior of constrained RTV from this campaign is compared with our past experiments on unconstrained RTV [25, 34] at similar heating rates. Temperature of the sample was measured throughout the test using a type-K thermocouple located at the bottom of the quartz tube which was housed in a graphite stub. A hot-zone calibration was performed for the 6-lamp setup with the type-K thermocouple, and the temperature profile is shown in Fig. 10c. A uniform-heating spot of ≈ 5 mm with a temperature of ≈ 1040 °C is observed. Samples of 3 mm diameter were used such that they were fully contained in the uniform heating region. Temperature histories for the three heating rates are shown in Fig. 10d, with each marker indicating the start of a tomography. The time windows that lack data for the 13 °C/min (1000 - 1800 sec, 3000- 3600 sec) and 30 °C/min (900-1700 sec) were due to data acquisition issues.

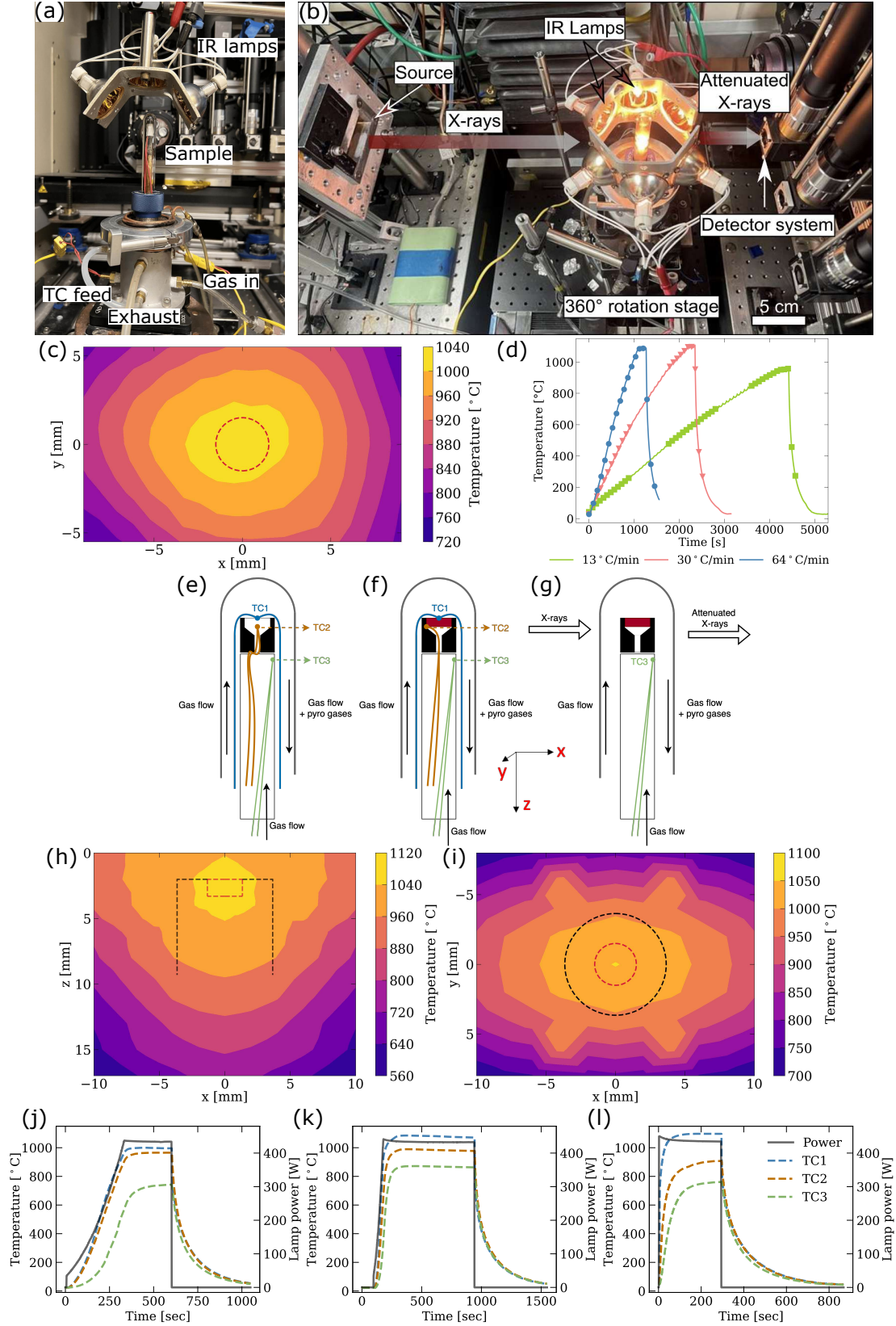


Fig. 10: (a) Environmental chamber for sample with 3 IR lamps in top-heating configuration, (b) side-heating configuration with 6 IR lamps, (c) hot zone map for side-heating, (d) temperature profiles for *in situ* μ -CT. Schematics of configurations used for (e) obtaining heat map, (f) *ex situ* calibrations and (g) *in situ* radiography testing, along with heat maps in the (h) x-z plane at y = 0 mm, and (i) x-y at z = 5 mm. Input lamp power and temperature profiles from *ex situ* calibration tests producing heating rates of (j) 228 °C/min, (k) 1000 °C/min and (l) instantaneous heating.

4.2 Radiography at high-heating rates

High-heating rate experiments were imaged in radiography mode using three IR lamps with the sample enclosure in the hot-spot (Fig. 10a). A top-heating approach was chosen in this case such that the intumesced RTV would remain in the hot-zone of the lamps, as well as uni-directional heating similar to that seen during atmospheric entry could be simulated. Voltage and current were varied linearly to the lamps to vary the heating rate of the experiments, providing heating rates up to 1946 °C/min.

With the top heating setup, calibrations were first performed to identify the hot-zone of the 3 lamps with a single thermocouple at the surface of a graphite holder, and the sample was placed in the hot-zone for the *in situ* tests. A heat map of the temperature in the three-lamp setup in Fig. 10h and 10i show the focal spot of the 3 lamps to be about 5 mm, similar to that of the side-heating setup.

Using this setup, non-degassed and degassed RTV samples constrained in graphite were tested in Argon and air at numerous power ramp-rates. For the tests, RTV was friction-fit into a graphite holder with the holder having a hole in the bottom to allow gas to flow through. Graphite was used instead of quartz in order to promote uniform heating from the top and sides of the sample. Additionally, a sample of ≈ 1.5 mm height and 3 mm diameter was used for all the tests, based on field-of-view constraints. Samples of non-degassed RTV poured into FiberForm and PICA holders were also tested in an Argon environment, where wicking of RTV into the porous holders affected intumescence.

4.2.1 *Ex situ* sample temperature calibration for high-heating rates:

Thermal calibration of the setup was performed to obtain correlations of lamp-power ramp rate versus sample temperature. Calibrations were performed using two 36 AWG thermocouples (denoted TC1 and TC2 in Fig. 10e) placed at the top and bottom of the RTV sample. A third 24 AWG thermocouple (denoted TC3) was also used that measured chamber temperature. During radiography collection, the configuration shown in Fig. 10g was used with only the 24 AWG TC3 thermocouple, and enabled verification of measurements of the calibration test and the *in situ* radiography test.

For the calibrations, non-degassed RTV samples were friction-fit into a graphite holder with the junction of TC2 touching the side walls of the graphite and RTV. The junction of TC1 was centered on the RTV sample and the insulated thermocouple wires were taped around the graphite holder with fiberglass tape to prevent movement of the thermocouple junction. The instrumented sample assemblies were subjected to the same lamp power ramp-rates as the *in situ* tests with radiography, and the thermal response was measured in Argon flow.

Temperature response of RTV heated in Argon was measured using the configuration shown in Fig. 10f. Fig. 10j shows the input power along with temperature profiles for conditions producing heating rates of 228 °C/min, 1000 °C/min along with a condition of instantaneous heating.

The temperature response of RTV in the first two cases is seen to be approximately linear, whereas the instantaneous heating shows a non-linear temperature response, with an initial rate of ≈ 5000 °C/min. A higher variation between top and bottom sample temperatures (TC1 and TC2) is seen as the power ramp-rate is increased, due to thermal inertia of the material. With increasing ramp-rate, the steady-state TC2 temperatures are significantly lower than those from TC1 due to intumescence of RTV, causing the conduction path length to increase and thereby producing a lower TC2 temperature [27, 55]. Therefore, as heating rate is increased, a reduction of heat transfer is observed between the top and bottom of the sample due to intumescence of the RTV. In the calibration experiments, as the RTV was seen to swell upwards and envelop the TC1 junction, temperature from TC1 was treated as the representative sample temperature, and was used for correlation with radiographs.

5 Deep Learning Segmentation

5.1 Micro-CT

Deep Learning for Scientific Image Analysis (DLSIA) was used for segmenting the μ -CT datasets [56]. Within the DLSIA toolbox, a new class of convolutional neural networks (CNNs) called 3D sparse mixed-scale networks (SMSNets) were used, due to their low number of tunable parameters compared to conventional U-Nets and their pre-pruned network giving them an advantage over mixed-scale dense networks (MSDs). To generate SMSNets, an ensemble of stochastically configured MSDs is

created by varying key hyperparameters. In this study, the 3D variant of SMSNets, called SMS3D, was used.

To train the SMS3D model ensemble, images of virgin and charred RTV were manually labeled and split into training and test data. The number of sparse networks was varied from 2 to 8, with negligible improvement beyond 7 networks. In order to ensure that training data encompassed all the possible artifacts (such as motion artifacts, grayscale value change due to densification, porosity development), cross-sections from the highest heating rate (64 °C/min) were used for annotations. Cross-sections along the length and height of the sample were selected from tomographies showing the virgin, partially charred and fully charred state of the sample. The tomography slices were normalized by subtracting the mean and dividing by the standard deviation of that particular tomography. The data was then selectively downsampled by a factor of 2 in all directions using a customized DLSIA downsample function [57], and 7 NNs were trained using the training data. Due to better performance of neural networks in two-class classification and due to similar attenuation of the RTV sample and the quartz holder, both the RTV and the quartz holder were segmented as one “solid” class along with a “void” class for the pore space. The quartz holder was later cropped from the segmented images in post-processing.

For inference, all the tomography datasets were normalized and downsampled in a similar manner, after which the segmented data was upsampled. Both the training and inference tomography datasets were partitioned into “chunks” using the qlty package [58]. Segmentation of the 3D datasets was performed on the NERSC supercomputer on four A100 GPUs (40 GB RAM), AMD EPYC 64-core processors with 200 GB available RAM. Additionally, grayscale-value based edge-detection was performed to remove the quartz tube and a connected-components “dusting” operation was performed to remove floating artifacts, producing segmented binary data differentiating solid and void space within a tomography [59]. From the segmented and cropped data, quantities such as solid volume, pore volume, width and height of the samples were computed.

5.2 Radiography

A 2D variant of SMSNets within DLSIA was used for segmenting radiography images. An ensemble of two networks was trained using manually annotated data. Three-class segmentation was performed to obtain the RTV, void and the graphite holder from the radiographs. The RTV sample height (defined as the distance from top to bottom at the center of the RTV) was computed and correlated with temperature.

6 Conclusion

The intumescence of room temperature vulcanizing silicone (RTV560) while subjected to heating was studied using non-intrusive *in situ* X-ray imaging at the Advanced Light Source, Beamline 8.3.2. *In situ* heating from infrared lamps with a combination of μ -CT and radiography were performed to capture a broad range of dynamic morphological changes of RTV.

At lower heating rates, *in situ* μ -CT was used to obtain 3D tomographies of RTV samples and assess change in morphology. Application of a novel 3D SMSNets allowed for segmentation of the *in situ* tomographies and quantification of intumescence, shrinkage and porosity variations, which correlated with the pyrolysis decomposition of RTV measured from thermogravimetric analysis. As RTV was heated, it was seen to intumesce due to thermal expansion and gas-pressure buildup with temperature. Intumescence was followed by cracking and pore formation with advancement of pyrolysis and shrinkage due to porous relief and outgassing. The *in situ* μ -CT campaign also showed augmented intumescence due to constraining the sample swelling to one direction. Finally, quantitative estimates revealed heating-rate dependence of intumescence due to delayed pyrolysis with increasing heating rate.

For higher heating rate studies, *in situ* radiography was performed for non-degassed and degassed RTV in Argon and air environments. Similar to the *in situ* μ -CT findings, this study also revealed highly heating rate-dependent intumescence which correlated well with pyrolysis decomposition from TGA. Degassed sample intumescence was much higher than that of non-degassed samples due to a lower extent of open-porosity within the degassed samples, causing gas-pressure buildup to be higher when heated. Tests performed in air revealed similar extents of intumescence with minimal shrinkage due to formation of a rigid, silica surface, revealing the effect of reactive environments on

RTV intumescence. Furthermore, comparing the *in situ* radiography and high-enthalpy experiments of non-degassed RTV showed good agreement for the height of intumescence as a function of time.

Finally, the effect of porous relief due to wicking of RTV into a porous TPS material was considered. High-resolution static scans revealed wicking of RTV into FiberForm and PICA and formation of open-porosity within RTV due to wicking. *In situ* experiments of the wicked RTV showed significantly lower intumescence than that when RTV was friction-fit into a holder, indicating that adhesion and formation of permeable pathways from wicking alleviate gas-pressure as RTV is heated. Therefore, from this effort, a large database of intumescence experiments for RTV was collected, and shall be used for assessing fencing heights on heat shields.

Future work shall focus on obtaining nano-scale porosity change of RTV with temperature using nano-CT and gas-adsorption techniques, and further correlation of RTV intumescence from *in situ* experiments at the ALS to those from high-enthalpy testing. Furthermore, material response models for intumescence shall be implemented into NASA’s state-of-the-art material response solver, Porous Materials Analysis Toolbox based on OpenFOAM (PATO) [60], to capture the underlying physics of intumescence accurately, and shall be validated with ALS and Plasmatron X experimental data.

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Data Availability

The data that support the findings of this study will be available on Materials Data Facility. The data is also available from the corresponding author on reasonable request.

Author contributions

S.O.: Writing – review & editing, Writing – original draft, Visualization, Validation, Project administration, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **C.W.F.:** Writing – review & editing, Project administration, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **H.S.B.:** Writing – review & editing, Investigation, Data curation, Methodology. **P.H.Z.:** Writing – review & editing, Validation, Investigation, Methodology, Formal analysis, Conceptualization. **R.A.R.:** Writing – review & editing, Validation, Investigation, Methodology, Data curation, Formal analysis, Conceptualization. **D.Y.P.:** Writing – review & editing, Supervision, Project administration, Methodology, Investigation, Data curation, Formal analysis, Conceptualization. **S.F.I.:** Writing – review & editing, Supervision, Methodology, Investigation, Conceptualization. **N.N.M.:** Writing – review & editing, Supervision, Methodology, Investigation, Conceptualization. **F.P.:** Writing – review & editing, Writing – original draft, Supervision, Methodology, Investigation, Funding acquisition, Conceptualization.

Competing interests

The authors declare no competing interests.

References

- [1] Jerschow, P.: Silicone Elastomers. Smart Publications, Shrewsbury (2001)

- [2] Winefordner, J.D.: The Analytical Chemistry of Silicones. John Wiley & Sons, New York (1991)
- [3] General Electric: RTV Silicone Rubber Keeps the Pressure in the X-15. Advertisement. Advertisement describing the use of GE RTV silicone rubber in the X-15 hypersonic research aircraft (1962)
- [4] Cherney, E., Gorur, R.: RTV silicone rubber coatings for outdoor insulators. *IEEE Transactions on Dielectrics and Electrical Insulation* **6**(5), 605–611 (1999)
- [5] Huzaimah Kamal Hamadi, S., Isa, M., Nizam Md Arshad Hashim, S., Othman, M.: Review on RTV silicone rubber coatings insulator for transmission lines. In: *IOP Conference Series: Materials Science and Engineering*, vol. 864, p. 012188 (2020). IOP Publishing
- [6] Cherney, E.: RTV silicone-a high tech solution for a dirty insulator problem. *IEEE Electrical Insulation Magazine* **11**(6), 8–14 (2002)
- [7] Cherney, E., Gorur, R.: RTV silicone rubber coatings for outdoor insulators. *IEEE Transactions on Dielectrics and Electrical Insulation* **6**(5), 605–611 (2020)
- [8] Mower, T.M.: Thermomechanical behavior of aerospace-grade RTV (silicone adhesive). *International Journal of Adhesion and Adhesives* **87**, 64–72 (2018)
- [9] Torres, X.M., Stockdale, J.R., Pacheco, A., Legett, S.A., Bezek, L.B., Benedikt, B., Labouriau, A., Adhikari, S.: Relationship between filler type, thermomechanical properties, and aging of RTV silicone foams. *Polymers* **17**(14), 1998 (2025)
- [10] Curry, D.M.: Space shuttle orbiter thermal protection system design and flight experience. NASA Technical Report No. 104773, NASA Lyndon B. Johnson Space Center (1993)
- [11] Beck, R.A., Driver, D.M., Wright, M.J., Hwang, H.H., Edquist, K.T., Sepka, S.A.: Development of the mars science laboratory heatshield thermal protection system. *Journal of Spacecraft and Rockets* **51**(4), 1139–1150 (2014)
- [12] Edquist, K., Dyakonov, A., Wright, M., Tang, C.: Aerothermodynamic design of the Mars Science Laboratory heatshield. In: *41st AIAA Thermophysics Conference*, p. 4075 (2009)
- [13] Hwang, H., Bose, D., Wright, H., White, T.R., Schoenenberger, M., Santos, J., Karlgaard, C.D., Kuhl, C., Oishi, T., Trombetta, D.: Mars 2020 entry, descent, and landing instrumentation (MEDLI2). In: *46th AIAA Thermophysics Conference*, p. 3536 (2016)
- [14] NASA + SpaceX Work Together — APPEL Knowledge Services. <https://appel.nasa.gov/2010/10/17/40s-space-x-html/>. (Accessed on 10/17/2022) (2010)
- [15] Lorenz, R.D., Turtle, E.P., Barnes, J.W., Trainer, M.G., Adams, D.S., Hibbard, K.E., Sheldon, C.Z., Zacny, K., Peplowski, P.N., Lawrence, D.J., *et al.*: Dragonfly: A rotorcraft lander concept for scientific exploration at titan. *Johns Hopkins APL Technical Digest* **34**(3), 14 (2018)
- [16] Borner, A., Venkatapathy, E., Gasch, M., Peterson, K., Monk, J., Stackpoole, M.: Recent developments of thermal protection materials to enable lower cost space missions. In: *21st International Planetary Probe Workshop (IPPW)* (2024)
- [17] Rao, A.P., Crespo, J.D., Valentini, P., Murray, V.J., Vaughan, E.I., Davis, Z.S., Alviani, R., Kulakhmetov, M.: Return of OSPREE: In situ spectroscopic measurements during atmospheric reentry. *Journal of Spacecraft and Rockets*, 1–14 (2025)
- [18] Leistikow, K.J., Nawar, N., Borrero-Negrón, J.I., Kulakhmetov, M., Valentini, P., Rao, A.P.: Quantification of spacecraft heatshield contaminants seen in reentry shock layer emissions using calibration-free libs. *Journal of Analytical Atomic Spectrometry* (2026)
- [19] Venkatapathy, E.: Ablators-From Apollo to future missions to Moon, Mars and beyond. In: *International Astronautical Congress* (2019)

- [20] Mansour, N.N., Panerai, F., Lachaud, J., Magin, T.: Flow mechanics in ablative thermal protection systems. *Annual Review of Fluid Mechanics* **56**(1), 549–575 (2024)
- [21] Milos, F.S., Chen, Y.-K.: Ablation and thermal response property model validation for phenolic impregnated carbon ablator. *Journal of Spacecraft and Rockets* **47**(5), 786–805 (2010)
- [22] Oruganti, S., Capponi, L., Ringel, B., Oldham, T., Panesi, M., Elliott, G., Izquierdo, S.F., Mansour, N., Panerai, F.: Low-density ablator response to induction plasmatron aerothermal environments (2026)
- [23] Milos, F.S., Scott, C.D., Del Papa, S.V.: Arcjet testing and thermal model development for multilayer felt reusable surface insulation. *Journal of Spacecraft and Rockets* **51**(2), 397–411 (2014)
- [24] Oruganti, S., Mansour, N.N., Panesi, M., Panerai, F.: Microstructure of pyrolyzing RTV silicone. *Polymer Degradation and Stability* **207**, 110237 (2023)
- [25] Foster, C.W., Oruganti, S., Panerai, F.: Real-time quantitative imaging of RTV silicone pyrolysis. *Polymer Degradation and Stability* **214**, 110403 (2023)
- [26] Alongi, J., Han, Z., Bourbigot, S.: Intumescence: Tradition versus novelty. A comprehensive review. *Progress in Polymer Science* **51**, 28–73 (2015)
- [27] Russell, G.W., Strobel, F.: Modeling approach for intumescent charring heatshield materials. *Journal of spacecraft and rockets* **43**(4), 739–749 (2006)
- [28] Mahzari, M., Beck, R., Hwang, H., Monk, J., Morgan, J., Williams, J., Edquist, K.T.: Development and sizing of the mars2020 thermal protection system. In: *AIAA AVIATION 2022 Forum*, p. 3951 (2022)
- [29] Alunni, A., Gokcen, T.: Assessment of fencing on the orion heatshield. In: *46th AIAA Thermophysics Conference*, p. 3534 (2016)
- [30] Driver, D.M., Carballo, J.E., Beck, R., Prabhu, D., Santos, J.A., Cassell, A., Skokova, K., Tang, C., Hwang, H.H., Slimko, E., *et al.*: Arcjet testing in shear environment for mars science laboratory thermal protection system. *Journal of Spacecraft and Rockets* **51**(4), 1151–1166 (2014)
- [31] White, T.R., Mahzari, M., Bose, D., Santos, J.A.: Post-flight analysis of mars science laboratory’s entry aerothermal environment and thermal protection system response. In: *44th AIAA Thermophysics Conference*, p. 2779 (2013)
- [32] Tang, C., Edquist, K., Wright, M., Sepka, S., Cassell, A.: Numerical simulations of protruding gapfillers on the mars science laboratory heatshield. In: *41st AIAA Thermophysics Conference*, p. 4077 (2009)
- [33] Oruganti, S., Capponi, L., Ringel, B.M., Oldham, T., Panesi, M., Elliott, G., Panerai, F., Mansour, N.N.: High-Enthalpy Testing of Thermal Protection Materials in the Plasmatron X for Titan Atmospheric Entry. In: *AIAA Scitech 2024 Forum*, p. 0863 (2024)
- [34] Foster, C., Oruganti, S., Panerai, F.: In-situ imaging of RTV silicone pyrolysis. In: *AIAA AVIATION 2023 Forum*, p. 3616 (2023)
- [35] Ringel, B.M., Semeraro, F., Ferguson, J.C., Barnard, H.S., Dias, B., Schlepütz, C.M., Barnard, E.S., Schickler, S., Levy, K., Shacterman, S., *et al.*: Carbon fiber oxidation in 4d. *Advanced Materials* **37**(42), 2502007 (2025)
- [36] Panerai, F., White, J.D., Cochell, T.J., Schroeder, O.M., Mansour, N.N., Wright, M.J., Martin, A.: Experimental measurements of the permeability of fibrous carbon at high-temperature. *International Journal of Heat and Mass Transfer* **101**, 267–273 (2016)

- [37] Grassie, N., Francey, K., Macfarlane, I.: The thermal degradation of polysiloxanes—part 4: Poly (dimethyl/diphenyl siloxane). *Polymer Degradation and Stability* **2**(1), 67–83 (1980)
- [38] Deshpande, G., Rezac, M.E.: The effect of phenyl content on the degradation of poly (dimethyl diphenyl) siloxane copolymers. *Polymer Degradation and Stability* **74**(2), 363–370 (2001)
- [39] Deshpande, G., Rezac, M.E.: Kinetic aspects of the thermal degradation of poly (dimethyl siloxane) and poly (dimethyl diphenyl siloxane). *Polymer Degradation and Stability* **76**(1), 17–24 (2002)
- [40] Der Weij, F.W.: The action of tin compounds in condensation-type RTV silicone rubbers. *Die Makromolekulare Chemie: Macromolecular Chemistry and Physics* **181**(12), 2541–2548 (1980)
- [41] Cervantes, J., Zárraga, R., Salazar-Hernández, C.: Organotin catalysts in organosilicon chemistry. *Applied Organometallic Chemistry* **26**(4), 157–163 (2012)
- [42] Myers, L.: Hydrogen gas from cured RTV silicones. Technical report, Mason and Hanger-Silas Mason Co., Inc., Amarillo, TX (United States) (1977)
- [43] Brenner, D.W.: Empirical potential for hydrocarbons for use in simulating the chemical vapor deposition of diamond films. *Physical review B* **42**(15), 9458 (1990)
- [44] Smith, A.L., Parker, R.: Trace analysis involving silicones. *The analytical chemistry of silicones*, Wiley, New York, 71–95 (1991)
- [45] Chacon, L.A., Deaton, B., Bessire, B.K., Poovathingal, S.J.: Chemical and structural degradation of room temperature vulcanizing (rtv) silicone at high temperatures. *Polymer Degradation and Stability* **220**, 110661 (2024)
- [46] RTV511, RTV560 & RTV577: Silicone elastomers: Momentive. <https://www.momentive.com/en-us/categories/adhesives-and-sealants/rtv511-rtv560-and-rtv577>
- [47] Foster, C.W., Oruganti, S., Panerai, F.: Super-resolved microstructure of pyrolyzing superlight ablators. *npj Materials Degradation* **9**(1), 9 (2025)
- [48] Maire, E., Withers, P.J.: Quantitative X-ray tomography. *International Materials Reviews* **59**(1), 1–43 (2014) <https://doi.org/10.1179/1743280413Y.0000000023>
- [49] Barnard, H.S., Macdowell, A.A., Parkinson, D.Y., Larson, N.M., Peterson, J.C., Panerai, F., Mansour, N.N., Gao, Y.: Synchrotron x-ray micro tomography at the advanced light source: In-situ sample environments for advanced aerospace materials. *Microscopy and Microanalysis* **24**(S2), 444–445 (2018) <https://doi.org/10.1017/S1431927618014472>
- [50] Bale, H.A., Haboub, A., MacDowell, A.A., Nasiatka, J.R., Parkinson, D.Y., Cox, B.N., Marshall, D.B., Ritchie, R.O.: Real-time quantitative imaging of failure events in materials under load at temperatures above 1,600 °C. *Nature materials* **12**(1), 40–46 (2013) <https://doi.org/10.1038/nmat3497>
- [51] MacDowell, A.A., Barnard, H., Parkinson, D.Y., Haboub, A., Larson, N., Zok, F., Panerai, F., Mansour, N.N., Bale, H., Gludovatz, B., Acevedo, C., Liu, D., Ritchie, R.O.: High temperature x-ray micro-tomography, New York, NY USA, p. 050005 (2016). <https://doi.org/10.1063/1.4952925>
- [52] Haboub, A., Bale, H.A., Nasiatka, J.R., Cox, B.N., Marshall, D.B., Ritchie, R.O., MacDowell, A.A.: Tensile testing of materials at high temperatures above 1700 °C with in situ synchrotron X-ray micro-tomography. *Review of Scientific Instruments* **85**(8), 083702 (2014) <https://doi.org/10.1063/1.4892437>
- [53] Panerai, F., Bessire, B., Haskins, J., Foster, C., Barnard, H., Stern, E., Feldman, J.: Morphological evolution of ordinary chondrite microstructure during heating: implications for atmospheric

- entry. *The Planetary Science Journal* **2**(5), 179 (2021)
- [54] Foster, C., Phillippe, C., Villafañe Roca, L., Panerai, F.: In situ micro-computed tomography of re-entry fabrics under tensile loading. In: TMS Annual Meeting & Exhibition, pp. 1681–1692 (2024). Springer
 - [55] Anderson JR, C.E., Dziuk JR, J., Mallow, W.A., Buckmaster, J.: Intumescent reaction mechanisms. *Journal of Fire Sciences* **3**(3), 161–194 (1985)
 - [56] Roberts, E.J., Chavez, T., Hexemer, A., Zwart, P.H.: DLSIA: Deep learning for scientific image analysis. *Applied Crystallography* **57**(2), 392–402 (2024)
 - [57] Zwart, P.H.: `downsample.py`. <https://github.com/phzwart/dlsia/blob/main/dlsia/core/utils/downsample.py>. GitHub repository (n.d.)
 - [58] Zwart, P.H.: `qlty`: Handling large tensors in scientific imaging deep-learning workflows. *Software impacts* **21**, 100696 (2024)
 - [59] Gostick, J.T., Khan, Z.A., Tranter, T.G., Kok, M.D., Agnaou, M., Sadeghi, M., Jervis, R.: Porespy: A python toolkit for quantitative analysis of porous media images. *Journal of Open Source Software* **4**(37), 1296 (2019)
 - [60] Lachaud, J., Mansour, N.N.: Porous-material analysis toolbox based on openfoam and applications. *Journal of Thermophysics and Heat Transfer* **28**(2), 191–202 (2014)

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