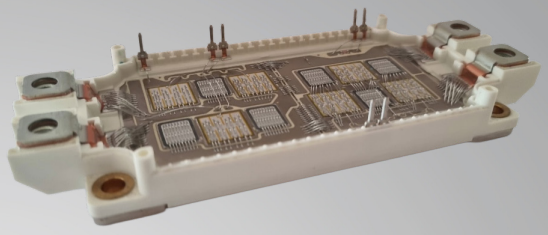
# Characterization of Thermally Aged Silicone Gels for Power Semiconductor Modules



## Abstract

This paper presents a comprehensive evaluation of silicone gels used in power semiconductor module packaging. Silicone gels are widely used as encapsulation materials, serving critical functions within the package such as preventing partial discharges and shielding inner module components from environmental impact. Five commercial gels were subjected to thermal aging at extreme low and high temperatures: -50 °C and 200 °C respectively. Characterization techniques including FTIR, DSC, TGA, and hardness measurements were employed to assess changes in material composition and properties under thermal stress. The results reveal varying degrees of degradation, providing valuable insights into the selection of suitable silicone gels based on specific application and operating conditions.

## 1. Introduction

Power semiconductor modules are part of a rapidly growing market, providing solutions for electric vehicles (EVs), renewable energy, and industrial applications [1]. As a key to the next generation of products, innovative and robust power module packages must meet the requirements for higher power density, greater reliability, and lower costs [2]. Moreover, the adoption of wide band gap (WBG) semiconductor technologies such as silicon carbide (SiC) in place of established Si-based semiconductor devices offers the potential for higher efficiency [3]. Simultaneously, this shift drives the adaptation of packaging technologies and materials to withstand the increased demand for operational conditions such as high temperatures and harsh environments. Silicone gels are considered as key packaging and insulating materials for power semiconductor modules due to their dielectric properties, excellent sealing adhesion, and elasticity, especially in the presence of wire bonds [4, 5]. During operation, these silicone gels undergo thermal fluctuations, which, over prolonged use in harsh environments, lead to structural and property changes.

In recent years, several studies have explored the fundamental physical and chemical properties of silicone gels [6, 7, 8] focusing on aspects such as viscosity, adhesion [9, 10], and degradation behavior under high humidity conditions [11, 12]. Recent publications have examined the dielectric properties of silicone gels [5, 13], particularly the influence of repetitive partial discharges [14], the impact of high temperature on dielectric properties, and internal structure of the gels [15, 17]. For the qualification of next-generation power module packages, understanding the influence of thermal aging on material properties is crucial. However, there remains a lack of comprehensive studies addressing both low– and high-temperature aging, conditions commonly encountered in automotive and rail applications, where large temperature fluctuations are typical.

This study presents the characterization results of commercial silicone gels subjected to thermal aging at both high– and low temperatures, providing insights into their suitability for demanding power semiconductor applications.

## 2. Experimental Plan

This section outlines the methodology used to prepare and evaluate five commercial silicone gels under thermal aging conditions. It includes details on gel selection, sample preparation, and curing protocols, followed by exposure to extreme temperatures (–50 °C and 200 °C) for up to 2000 hours.

### 2.1. Gel Selection and Sample Preparation

Five commercially available gels were selected, which were labeled as gels A, B, C, D, and E, considering their operating temperature range, dielectric properties, volume resistivity, and curing conditions. Gels are either two-part agents or only one part which means no mixing is required. To protect business confidentiality, detailed specifications of the gels are not disclosed in this paper. Table 1 outlines the preparation methods used for each gel. Gels were cured according to the manufacturers' datasheet instructions.

Table 1. Gel preparation details

| Preparation Requirement | Gel A | Gel B | Gel C | Gel D | Gel E |
|---|---|---|---|---|---|
| Vacuum Time (minutes) | 20 | 20 | 20 | 20 | 20 |
| Vacuum Pressure (mbar) | 25 | 25 | 25 | 25 | 25 |
| Curing Temperature (°C) | 135 | 100 | 100 | 125 | 110 |
| Curing Time (minutes) | 90 | 60 | 10 | 15 | 30 |

For gels that required mixing, they were mixed uniformly for two minutes. The gels were then poured into labeled glass containers and kept under vacuum for 20 minutes to remove the trapped air. Finally, the gels were placed in an air circulation oven for curing according to the information given in Table 1.

### 2.2. Thermal Aging

The cured gel samples were then divided into two groups. The first group of samples was placed in a temperature-controlled oven at 200 °C and the second group in a freezer at -50 °C. The samples were randomly taken out after 500 hours, 1000 hours, and 2000 hours for further analysis.

### 2.3. Characterization

This section presents the characterization methods that were performed in this study, including thermal, physio-chemical, mechanical, and morphological analysis to assess material degradation and performance.

#### 2.3.1. Thermal Analysis

Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) are the most widely-used characterization methods for analysis of the thermal decomposition of materials such as silicone gels. The thermal stability of the gels was evaluated at various stages of the aging test using the equipment and parameters listed in Table 2.

Table 2. Details of TGA and DSC system and parameters

| Thermal Analysis | System | Temperature Range (°C) | Rate (°C/minute) |
|---|---|---|---|
| Vacuum Time (minutes) | 20 | 20 | 20 |
| Vacuum Pressure (mbar) | 25 | 25 | 25 |
| Curing Temperature (°C) | 135 | 100 | 100 |
| Curing Time (minutes) | 90 | 60 | 10 |

### 2.3.2. Physio-Chemical Analysis

FTIR analysis is considered a fast acquisition method for spectroscopic information from polymer-based materials such as silicone gels. The change in the characteristic absorption of the key functional groups such as Si-O-Si, C-H, Si-CH3, and Si-(CH3)2 bonds was monitored as-cured and through thermal aging. The system used was a Nicolet™ iS50 FTIR Spectrometer in transmission mode, with a resolution of 4 cm-1 in a scanning range of 400-4000 cm-1 with 30 scans. Figure 1 shows the FTIR spectra of the five selected silicone gels in the as-cured condition.

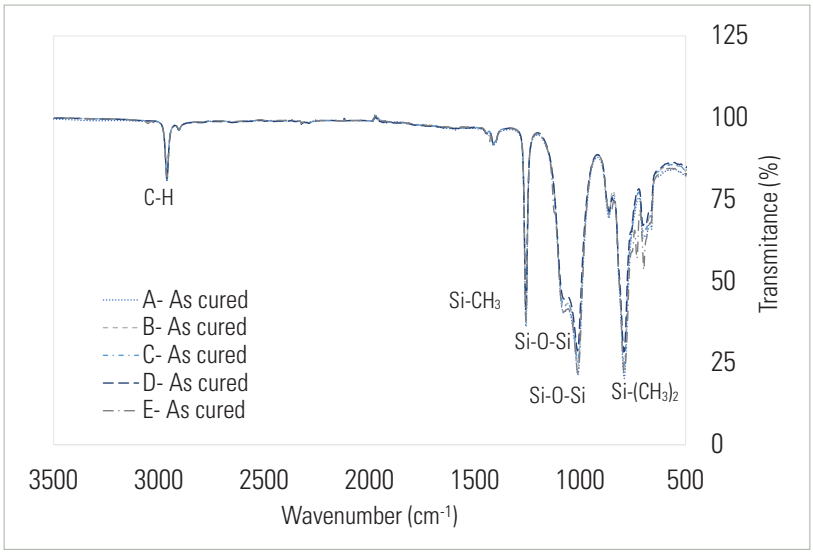



Figure 1. FTIR spectra of the five selected gels, as-cured condition

### 2.3.3. Mechanical Analysis

Hardness measurements were conducted on the silicone gels using texture analyzer in compression mode. The mechanical resistance of the samples to stress was measured using a Stable Micro Systems TA.XTplus texture analyzer, based on industry standard for bloom strength, using the parameters shown in Table 3. Figure 2 shows the texture analyzer with its typical graph results for gel A in the as-cured condition.

Table 3. Texture analyzer test settings

| Test Parameters | Values |
|---|---|
| Probe diameter | 10 mm |
| Test speed | 0.5 mm/s |
| Trigger force | 4 g |
| Depth of measurement | 10 mm |


Figure 2a. Texture analyzer

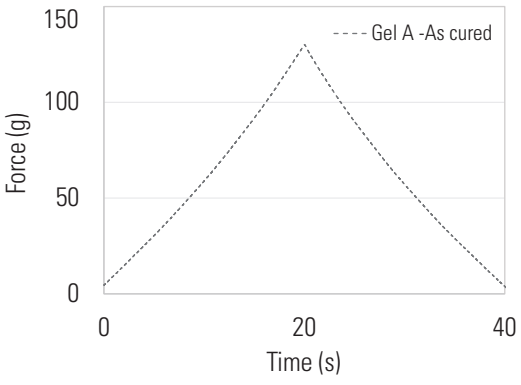



Figure 2b. Typical graph results – Gel A as-cured

### 2.3.4. Morphological Observation

The samples were closely observed during the aging tests to monitor changes in surface morphologies, crack initiation, and delamination. A Keyence microscope VHX-6000 was used for visual observation and imaging. The details of the test characterization plan are presented in Table 4.

Table 4. Details of test characterization plan

| Method | As Cured | 1000 hours | | 2000 hours | |
|---|---|---|---|---|---|
| | | -50°C | +200°C | -50°C | +200°C |
| FTIR | ✓ | ✓ | ✓ | ✓ | ✓ |
| TGA | ✓ | ✓ | ✓ | ✓ | ✓ |
| DSC | ✓ | – | – | – | – |
| Hardness | ✓ | ✓ | ✓ | ✓ | ✓ |

## 3. Results

In this section, the results of the analytical evaluation based on the characterization methods highlighted in the previous section are discussed.

### 3.1. Thermal Analysis Results

This subsection presents the thermal stability and degradation behaviour of the silicone gels using thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). It highlights onset decomposition temperatures, mass loss rates, and thermal events observed across different aging conditions to assess gel performance under extreme temperatures.

#### 3.1. 1. TGA Results

The TGA thermal curves were plotted for each single gel at different exposed temperatures, as shown in Figure 3. The key evaluation criteria were based on:

- thermal stability of the gels at operation temperature up to 250 °C
- mass loss rate between 250 °C and 650 °C, and
- mass loss at 650 °C and beyond

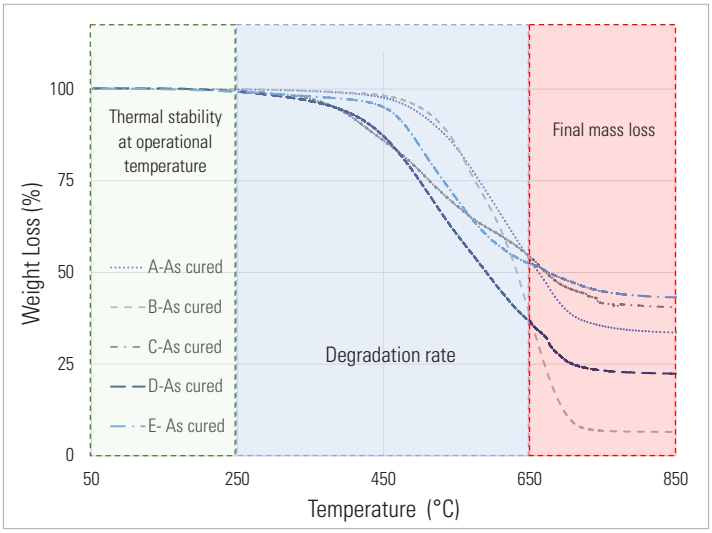



Figure 3. TGA curve – the key characterization criteria – all the Si Gels in as-cured condition

As shown in Figure 4, all the gels remained stable as-cured and aged up to 130 °C. The first thermal event occurred for gel E in the as-cured condition at 135 °C, followed by gel D in the as-cured condition at 185 °C. It appears that these two gels have the highest content of low temperature volatile materials. The remaining gels started degrading at -50 °C, when aged for 1000 hours.

The thermal decomposition of the remaining gels started at approximately 250°C. As seen in Figure 4, the thermal decomposition of most gels, including gel E, improved after high-temperature aging. This might be an indication of possible cross-linking between molecular chains which results in a slower degradation rate [5, 7].

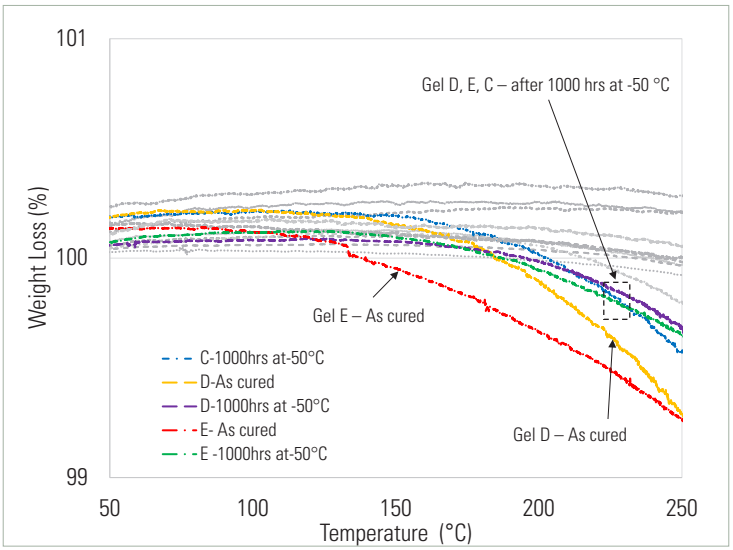



Figure 4. Close-up TGA curves of gels – start of thermal decomposition temperature of some gels highlighted as-cured and after 1000 hours at various aging temperatures

The onset temperature for each gel was extrapolated in accordance with ISO 11358-1. The extrapolated values were plotted for both the the as-cured state and after 1000 hours at various aging temperatures, as dispalyed in Figure 5.

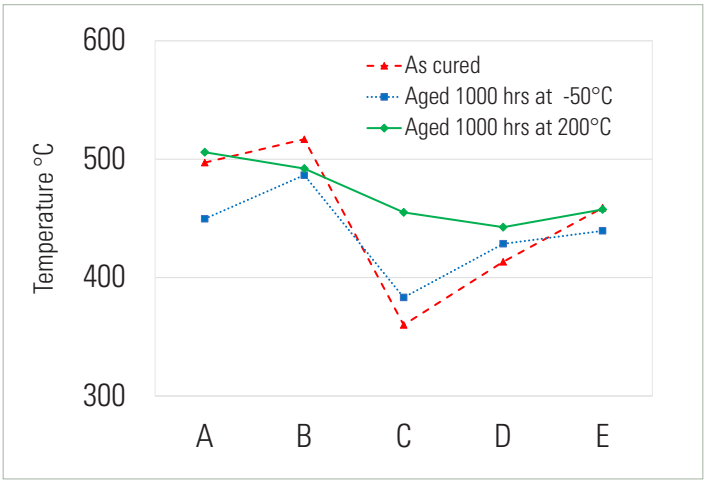



Figure 5. TGA onset degradation temperatures, as-cured and after 1000 hours at various aging temperatures

Similar trends were observed in the onset temperature results from the thermal endurance characterization. Most gels aged at high temperature exhibited higher onset temperatures compared to their as-cured condition.

The average rate of degradation was calculated as the percentage ratio of degraded mass at 5% mass loss and 60% mass loss to degradation time. Figure 6 presents the results of the average degradation rate, showing that gel E consistently exhibited the slowest degradation rates across all tested conditions.

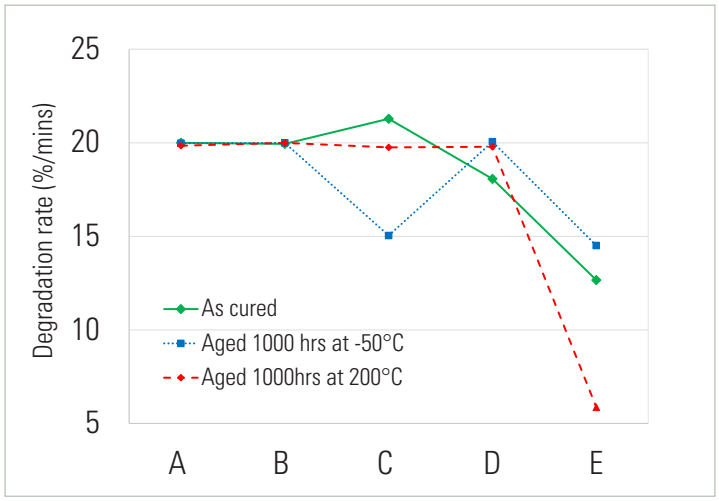



Figure 6. TGA Average degradation range at 5% mass loss and 60% mass loss, as-cured and after 1000 hours at various aging temperatures

Considering the overall mass loss at 650 °C and beyond, as can be seen in Figure 7, gel E shows the minimum mass loss followed by gel C.

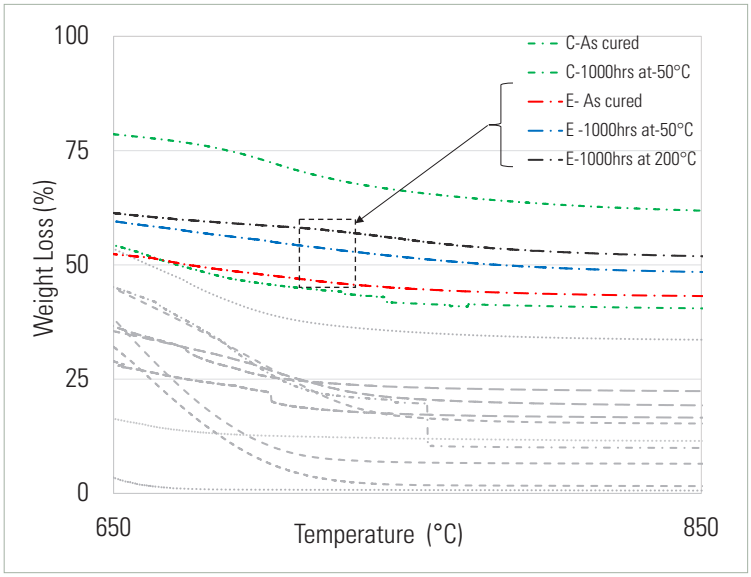



Figure 7. Close-up TGA curves of all the selected gels (only those discussed highlighted), as-cured and after 1000 hours at various aging temperature, total mass loss at 650 °C and beyond

### 3.1. 2. DSC Results

The thermal behavior of the selected gels in the as-cured condition was investigated using DSC. The thermograms of the selected gels were analyzed during cooling from -20 °C to -150 °C at a rate of 10 °C/min. Details of the observed thermal events are provided in Table 5 and the results of two selected gels (C and D) are shown an exemplary in Figure 8.

Table 5. Details of thermal events observed for selected gels in as-cured condition based on DSC thermogram

| Gels | Glass State $T_g$ onset (°C), (mW) | Thermal Event 1 $T_{on}$ to $T_{end}$ (°C), (mW) | Thermal event 2 Ton to Tend (°C), (mW) |
|---|---|---|---|
| A | -127, -1.5 | -85 to -104, -1.7 | -32 to -52, -3.2 |
| B | –* | -33 to -52, -3.16 | – |
| C | -125, -1.2 | -75 to -95, -1.5 | -42.7 to -62, -2.5 |
| D | -124, -0.22 | – | – |
| E | -115, -0.53 | – | – |

* Glass transition temperature not detected within the tested temperature

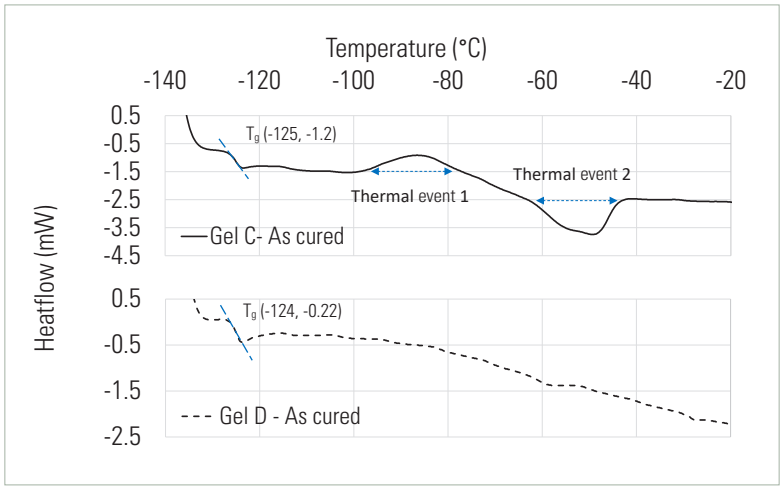



Figure 8. DSC results of the gel C & D in as-cured condition

As listed in Table 5, gels A and C exhibited the most thermal events below zero temperature. For gels A, B, and C, the thermal events began within the operational temperature range (typically down to -50 °C) of the majority of semiconductor modules. However, gels D and E demonstrated the highest thermal stability, as no thermal events were detected within the studied temperature range.

### 3.2. FTIR Results

The results of the change in characteristic transmittance/absorption intensity of the key functional groups of the silicone gels are reported in this section. Si-O-Si infrared bands were monitored between 1000-1010 and 1070-1090 wavenumber (cm-1). C-H stretching was studied between 2955-2965 wavenumbers cm-1, between 1255-1260 wavenumbers cm-1 for Si-CH3, and between 785-795 wavenumbers cm-1 for Si-(CH3)2, in the as-cured condition and after exposure to various aging temperatures.

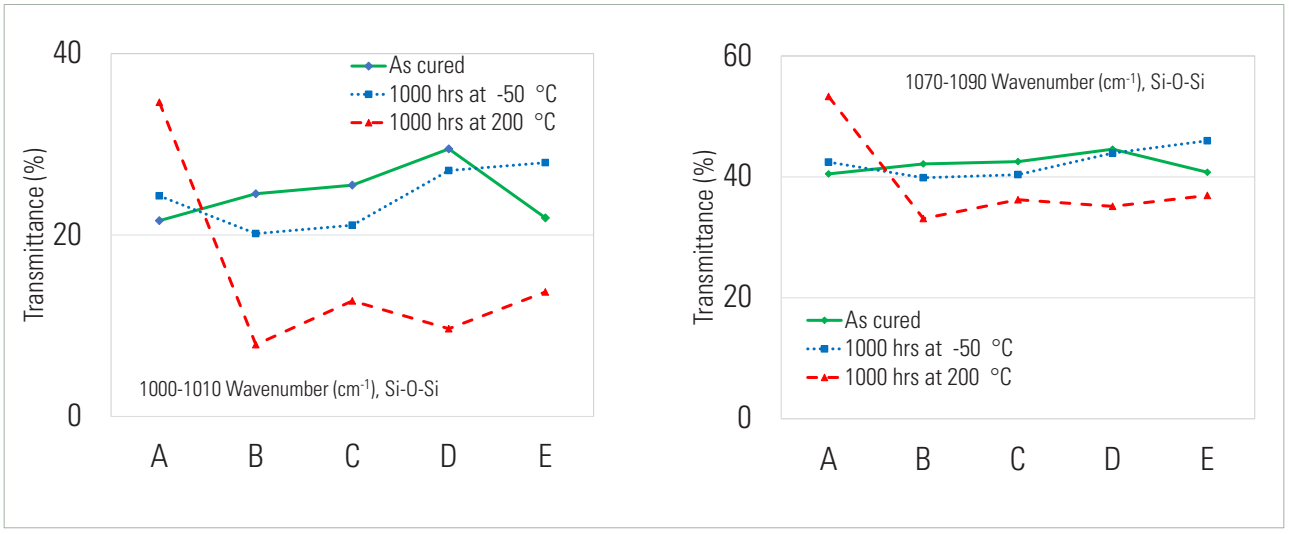



Figure 9. Change in transmittance of the Si-O-Si bonds in as-cured and various thermal aging conditions

The transmittance peaks of the bonds were obtained from the FTIR data in as- cured conditions and at various aging temperatures. The values were plotted in Figure 9 and Figure 10. The gels A and E show less transmittance/higher absorption density compared to gels B, C, and D. This potentially indicates that the cross-linking degree of these A and E gels is higher after curing. Based on this observation, gels A and E behave more like an elastomer; therefore, less adhesion properties are expected [5, 7]. As shown in Figure 9 and Figure 10, there are no apparent changes recorded in the transmittance intensity of the investigated functional groups after 1000 hours at -50 °C, suggesting all gels have good stability at freezing temperatures.

The transmittance intensity of gel A stored at 200 °C for 1000 hours has shown a significant increase in comparison to the other gels. The lower absorption in both functional groups indicates both chemical bonds were vulnerable and broken under the influence of high temperature. For the other gels, the absorption intensity increased at a comparable rate, suggesting that heat promoted an increase in the presence of the Si-O-Si and Si-CH3 bonds. Overall, the difference in absorbance intensity of gels C and D in the as-cured state and after thermal aging was low, which indicates good thermal stability within the investigated temperature range.

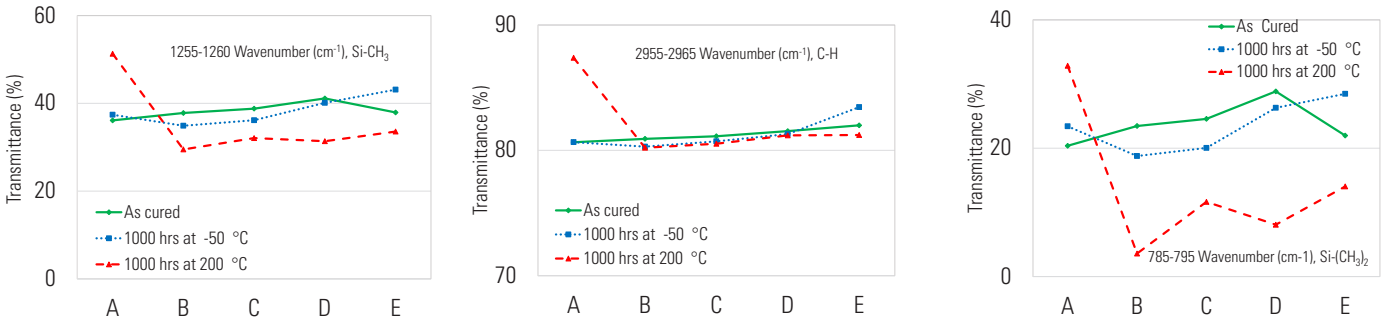



Figure 10. Change in the transmittance of the C-H, Si-CH3, and Si-(CH3)2 bonds in as-cured and various thermal aging conditions

### 3.3. Hardness Measurement Results

The results of the hardness testing of the gels are presented in this section. The gel samples that were aged at -50 °C were removed from the freezer and allowed to thaw at room temperature, after which the gels were kept in a controlled lab environment before texture analysis. As can be seen in Figure 11, almost all the gels were stable in terms of hardness value compared to the as-cured value, after 1000 hours and 2000 hours, which indicates that the chemical degradation of the gels at low temperature is minimal [11].

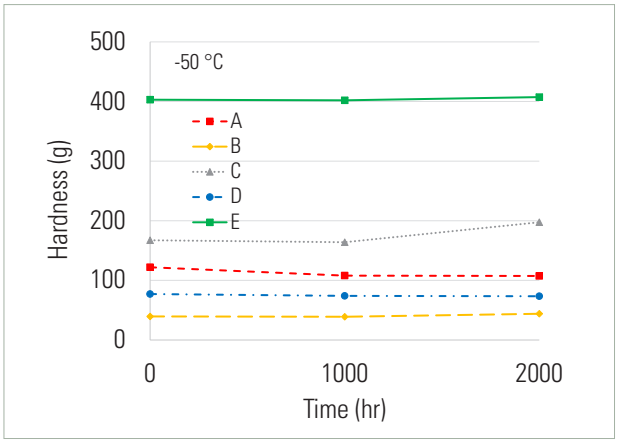



Figure 11. Hardness graph of the selected gels exposed to -50°C

Figure 12 presents the hardness values for the gels exposed to the 200 °C temperature. All the gels show an enhanced degradation in physical properties over time. As discussed in the thermal analysis section, the new cross-links are formed in molecular chains of the gels resulting in an increase in the hardness of all the gels. As seen in Figure 12, two measurements are missing after 2000 hours: gel B that became very hard and overloaded the tester using a 5 kg load, and gel D that became very brittle and formed several cracks and broke through during measurement. Based on the measurement values, gel C exhibited the least change in hardness during the observed time period of aging at 200 °C. However, overall, all gels experienced a loss of key mechanical properties such as tackiness, flexibility, and adhesion that are essential for optimal gel performance. These physical changes are likely to result in poor performance of power modules, particularly in applications requiring higher operational temperatures.

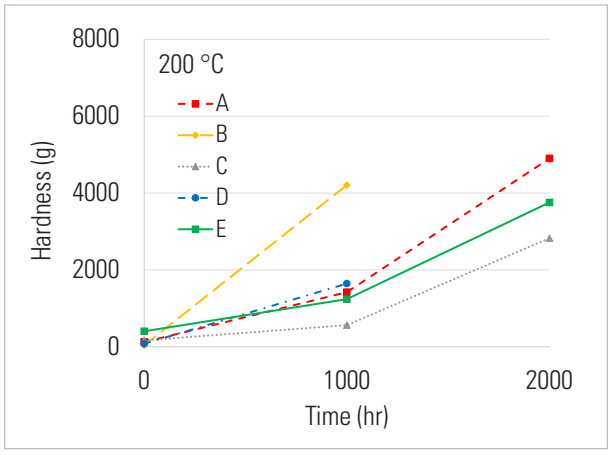



Figure 12. Hardness graph of the selected gels exposed to 200°C

### 3.4. Observation Results

During the study, gels in petri dishes and glass beakers were visually inspected every 500 hours. Imaging was used to determine whether any visible material changes had occurred. After 500 hours of exposure to freezing temperatures (-50 °C), gels A and B began to exhibit both surface and internal cracking. Figure 13 illustrates the surface condition of gel A after 500 hours.

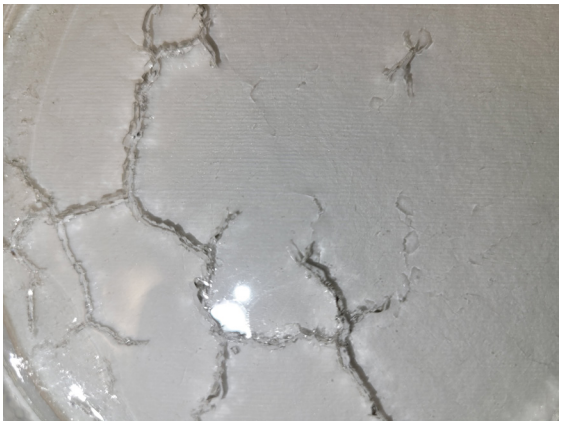


Figure 13a. Gel A surface condition, after 500 hours at -50°C

Figure13b. Close-up image of surface cracking

Based on visual observations and microscopic imaging, gels A and B, which were removed from the oven at 200 °C, began to show surface cracking after 500 hours. In contrast, no surface cracking was observed in gels C, D, and E. Additionally, all gels except gel E developed discoloration, transitioning from a colorless or pale yellow to a darker yellow or brown. Table 6 summarizes the discoloration findings.

Table 6. Close- up image of gels discoloration post high tem-perature aging

| Gels | As-cured | Post-1000 hours | Post-2000 hours |
|---|---|---|---|
| A | | | |
| B | | | |
| C | | | |
| D | | | |
| E | | | |

## 4. Summary of the Results

This section summarizes the investigation results of thermally aged silicone gels. As previously mentioned, gels E and D began to show slight weight loss within the operational conditions of most available power semiconductor packages. Additionally, their onset thermal decomposition temperatures were among the lowest observed. However, the amount of weight loss was less than 1%, meaning all gels, both in their as-cured and aged conditions, demonstrated stability within the 30 °C–250 °C range. Beyond 250°C, gel E degraded at a significant slower rate compared to the other gels and maintained this trend under both aging conditions. Based on the degradation rate analysis, gel E was identified as the most stable gel among the tested gels.

Based on DSC results, Gels D and E showed no significant thermal events at freezing temperatures in their as-cured state. In contrast, the other gels exhibited thermal activity within the -50 °C to -200 °C temperature range, which is within the operational conditions of most semiconductor modules. FTIR analysis revealed a broad overlap in infrared absorption bands among the selected silicone gels, indicating similar chemical compositions (see Figure 1). Regarding adhesion capability of the gels in the as-cured condition, gels B, C, and D showed superior properties compared to the other gels. The FTIR results of the exposed gels under thermal aging indicate that gels D and C experienced minimal variation in absorbance intensity, suggesting greater thermal stability relative to the other gels examined in this study.

In terms of gel strength measurements, all gels maintained their strength after exposure to -50 °C. Post-exposure data at 200 °C revealed that gel C degraded more slowly than the others. However, it is important to note that none of the gels, including those marketed as high-temperature resistant, were able to retain their mechanical and physical properties under prolonged high-temperature aging. This raises serious concerns about the suitability of these gels for power semiconductor packages that require exposure or testing at elevated temperatures such as 200 °C.

In terms of visual observations under extreme low and high temperatures, gels A and B demonstrated the weakest performance. Signs of micro surface cracking and inner cracking were evident during the initial exposure period. In packaging applications, such cracks have the potential to lead to the degradation of isolation properties. Figure 14 summarizes the performance ranking of the five selected gels, rated from 1 (worst) to 5 (best), based on the findings of this study.

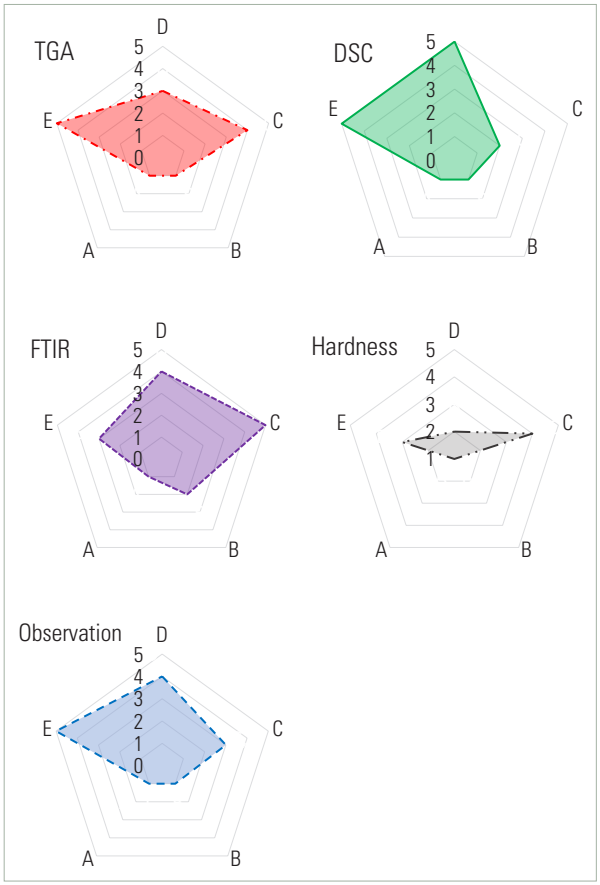



Figure 14. Overall performance results of the five selected gels based on the analytical results

## 5. Conclusion

This paper presents the results of an investigation into various commercial silicone gels subjected to thermal aging at -50 °C and 200 °C. Characterization techniques including FTIR, TGA, DSC, and hardness measurements were employed to assess property changes due to temperature exposure. The comprehensive data obtained from the study enabled a classification of each gel's suitability for power semiconductor packages, depending on specific application requirements.

Further research is needed to evaluate the interaction between these gels and other components within semiconductor packages. Additionally, exploring the impact of thermal aging on the dielectric properties of the gels would provide valuable insights.

## 6. Acknowledgement

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