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High-Temperature Adhesive Bonding of 4H-SiC Substrates

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Abstract. This study explores the application of Polycarbosilane (PCS) as an intermediate adhesive bonding technique for 4H-SiC substrates aiming to overcome the challenges of producing high-quality and cost-effective substrates for high-power electronics. Thin layers of PCS mixed with m-xylene and AIBN (azobisisobutyronitrile) were deposited onto 4H-SiC substrates via a spin coating. For demonstration purposes, these coated 4H-SiC substrates were then bonded with another 4H-SiC substrate. A defect-free, high-temperature stable bond is facilitated by annealing at high temperatures. Effusion measurements were conducted to characterize the PCS thin films and examine the organic-inorganic transitions and the resulting outgassing at high temperatures. SEM analysis confirmed the uniformity of the bonded layer. These results demonstrate PCS's potential in high-temperature applications and will stimulate further research exploring doped SiC bonding layers and their electrical properties.

Introduction

As power electronics technology advances towards higher efficiency, increased power density, and enhanced integration density on system level, traditional silicon-based solutions, which have long been dominant, are increasingly unable to meet the evolving requirements. Recent advancements in wide bandgap (WBG) semiconductor materials, however, particularly silicon carbide (SiC), are driving significant progress in the power electronics industry. SiC, as a WBG material, exhibits superior characteristics, including a significantly higher breakdown electric field and enhanced thermal conductivity than silicon, as summarized in Figure 1, positioning it as a strong candidate for high-voltage, low-loss power devices. However, despite notable advancements in SiC technology, delivering high-quality, cost-effective substrates remains a crucial challenge. [1, 2]

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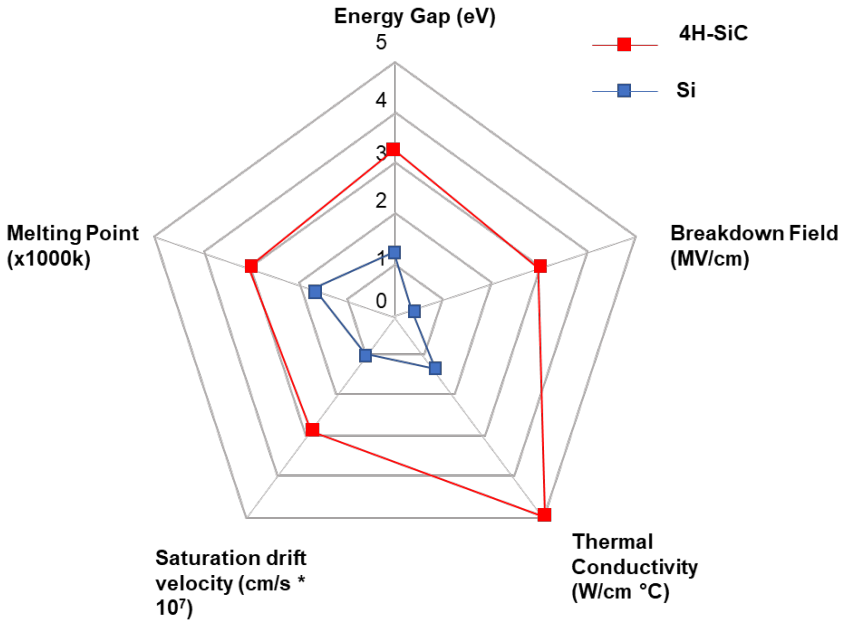

Fig. 1. Comparison of material properties of Si and SiC as summarized in [2].

Engineered substrates present a promising technological approach for achieving this challenging goal. Key techniques for producing such substrates include Smartcut™ [3], controlled spalling [4, 5], and photoelectrochemical etching [6, 7, 8]. The principle behind the Smartcut™ process includes the transfer of a high-quality device layer onto a low-resistivity handle wafer. Controlled spalling facilitates layer exfoliation via a mechanical approach, while photoelectrochemical etching utilizes tailored porosification to remove material layers from the mother wafer. The performance and the production yield of these structures significantly depend on the bonding quality of these unconventional heterostructures, which underscores the importance of developing robust, low-cost bonding techniques [9].

Intermediate bonding is a cost-effective and user-friendly alternative to conventional techniques like fusion bonding, offering several key advantages. These include uniform load distribution, tolerance to surface roughness, effective sealing, and resistance to stress and vibrations [10]. Among the various adhesive materials, including polymers, glass, metals, and ceramics, vinyl group-containing polycarbosilanes (PCS) polymers are particularly notable. PCS polymers exhibit strong adhesion to a wide range of substrates, exceptional chemical resistance, UV stability, and thermal durability, making them highly suitable for advanced bonding applications.

In this work, we have utilized the polymer from Starfire Systems Inc., commercially known as SMP-10. SMP -10 is a single-component liquid precursor that forms high-purity ceramic SiC, with a 72-78% amorphous SiC at 850-1200 °C and nanocrystalline β-SiC at 1250-1700 °C, thus offering the possibility to realize an electrically conductive bonding interface [11]. SMP-10 was deposited onto monocrystalline 4H-SiC substrates to investigate the organic-inorganic transition and to study the outgassing behavior due to thermal loading. Additionally, we investigated the potential of SMP-10 as an intermediate adhesive bonding agent, focusing on its ability to create thermally stable bonds between monocrystalline 4H-SiC substrates at high temperatures.

Experimental Details

For this study, 1 cm x 1 cm square samples were prepared from a single crystalline n-doped 4H-SiC wafer. Sample cleaning was performed using the RCA (Radio Corporation of America) cleaning protocols. The adhesive solution was prepared by mixing 10 g of SMP-10 with 30 g of m-xylene and 0.29 g of azobisisobutyronitrile (AIBN). AIBN is a radical photo-initiator that allows the curing of

the ceramic-forming polymer precursor at a substantially lower temperature. The nitrogen-containing photo-initiator can eventually also increase nitrogen in the formed ceramic layer to improve electrical conductivity.

The solution was spin-coated onto the 4H-SiC samples at 3000 rpm for 40 seconds. For demonstration purposes, a second monocrystalline 4H-SiC substrate was placed on top of the coated sample, and the assembly was pre-bonded with an EVG bonder, applying a constant force of 350 N. The samples were heated at 400 °C for one hour to facilitate bonding. Subsequently, the structure was annealed at 1550 °C at 20 mbar back chamber pressure and in an Ar atmosphere in a custom-built high-temperature furnace from HTM Reetz, resulting in a bonded sample. The bonded cross-sectional interface was then analyzed with a Hitachi SU8030 scanning electron microscope (SEM) using an acceleration voltage of 5 kV.

The solution was spin-coated onto the 4H-SiC substrate and analyzed using a HIDEN HALO RGA spectrometer to investigate the organic-inorganic transition and the associated outgassing. The samples were maintained at a vacuum pressure of approximately 10^{-5} Pa and heated to 1000 °C with a resistive heater element with a ramp rate of 10 °C/min. A schematic of the experimental setup for effusion measurement is depicted in Figure 2. During the heating process, the gaseous species released from the sample were analyzed with a quadrupole mass spectrometer and a Faraday cup from HIDEN Analytical for residual gas analysis, enabling, as a function of time, the measurement of the partial pressure of individual elements in the chamber, but close to the sample surface.

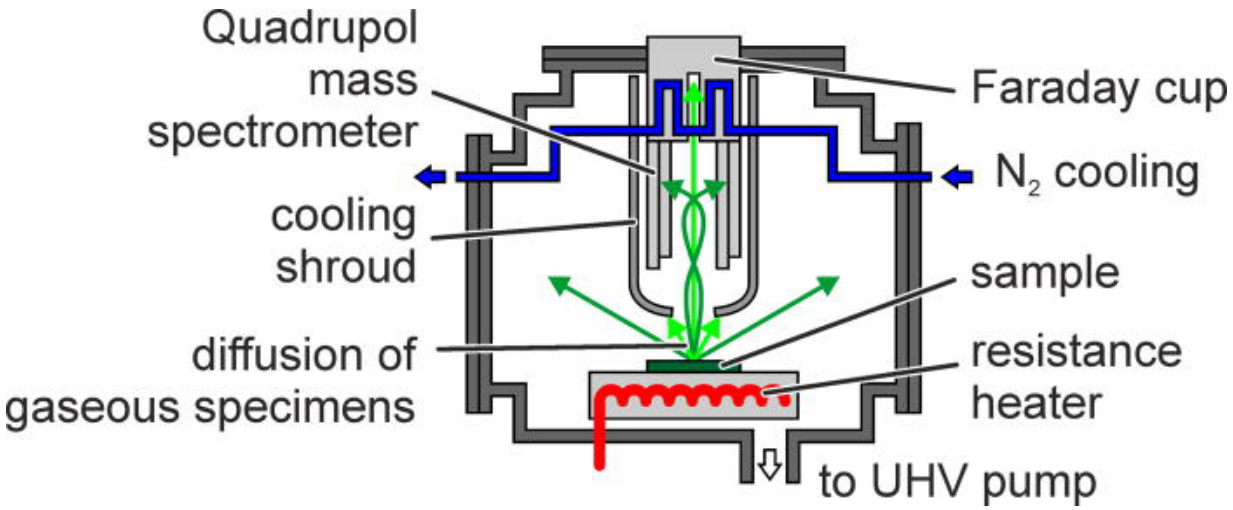

Fig. 2. Cross-sectional schematic of the effusion setup [15].

Polymer-to-ceramic conversion

The conversion of precursors into pre-ceramics and ceramic materials involves a sequence of reactions which results in changes of their compositions and properties. A cross-linking step is highly required to reduce the volatilization and depolymerization of the precursors and to increase the mass yield during the polymer-to-ceramic transformation. This cross-linking process involves reactions such as hydrosilylation and dehydrocoupling [12], as shown in Figure 3, which creates a three-dimensional polymeric network. This network renders the precursor more thermally stable, unmeltable, and insoluble. [13]

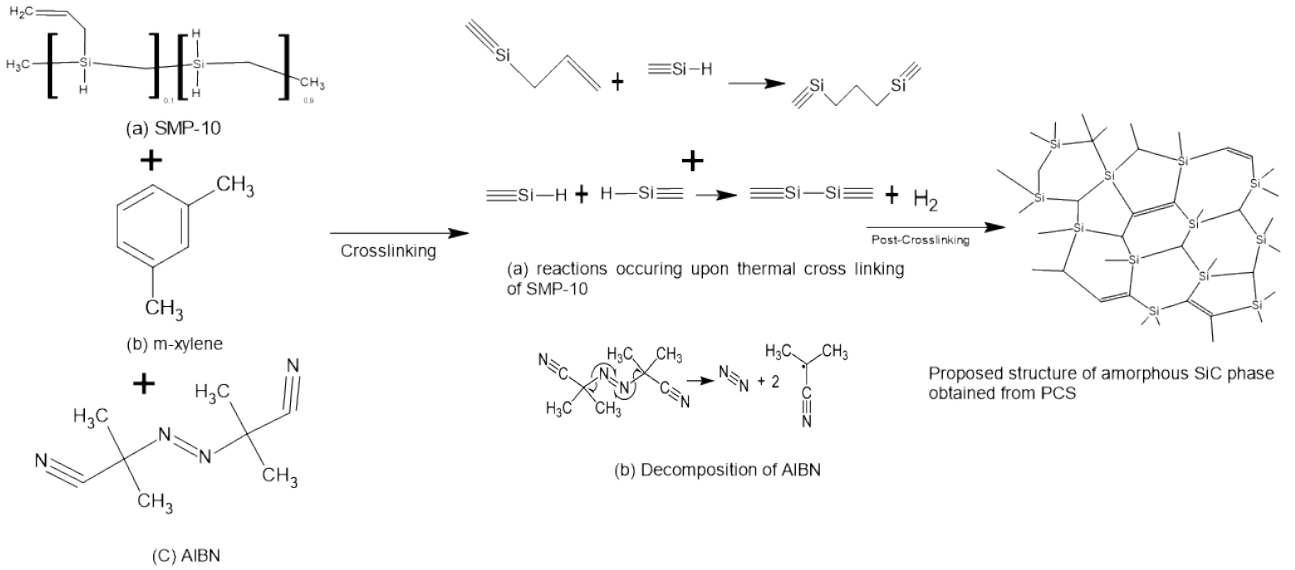

Fig. 3. Chemical reactions during the pyrolysis of SMP-10 [12, 14].

Further heat treatment transforms a thermoset polymer into an amorphous ceramic. Heating up to 400 °C removes low molecular weight polycarbosilanes (PCS). Within the temperature range of 400 °C to 550 °C, the formation of Si-CH_2-Si linkages increases the molecular weight of PCS. From 550 °C to 850 °C, the material undergoes a transformation into an inorganic structure, eliminating Si-H, Si-CH_3, and Si-CH_2-Si moieties, accompanied by the release of H_2 and CH_4.

At 700 °C, the formation of C=C bonds occurs, marking the initiation of the free carbon phase and the creation of dangling carbon bonds. At 800 °C, the material transitions into an amorphous state. The temperature range between 850 °C and 1000 °C represents a transitional phase with minimal weight loss. Crystallization begins to emerge between 1000 °C and 1200 °C, while temperatures above 1200 °C correspond to the stage of crystal growth. [14]

Results and Discussions

From the effusion measurements shown in Figure 4, we observe ionized gas molecules H^+ (amu 1) and CH_4^+ (amu 16) outgassing from the adhesive bonding layer occurring predominantly up to an annealing temperature of 900 °C. As the measurements were conducted under high vacuum conditions, we observed a minimal presence of H_2O (amu 18), CO (amu 28), and CO_2 (amu 44).

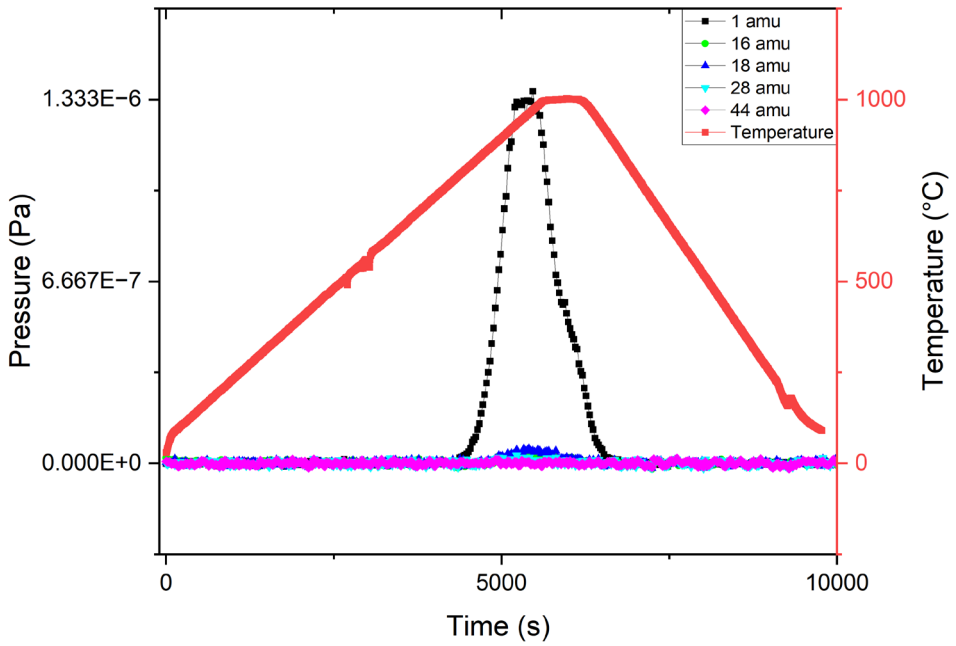

Fig. 4. Effusion measurement which illustrates the outgassing of H^+ (amu 1) and CH_4^+ (amu 16), H_2O (amu 18), CO (amu 28), and CO_2 (amu 44) with a temperature curve in red with a temperature ramp of 10 °C/min.

Additionally, we have analyzed the effect of the temperature ramp on the outgassing behavior, using ramps of 10 °C/min and 7 °C/min, as shown in Figure 5 and Figure 6. It was observed that a lower temperature ramp results in increased outgassing, as indicated by higher partial pressures. This phenomenon can be attributed to the slower heating rates, which allow the material to stay at intermediate temperatures for an extended time, allowing more time for the gaseous species to diffuse, and resulting in a higher energy input, both effects contributing to the higher pressure observed.

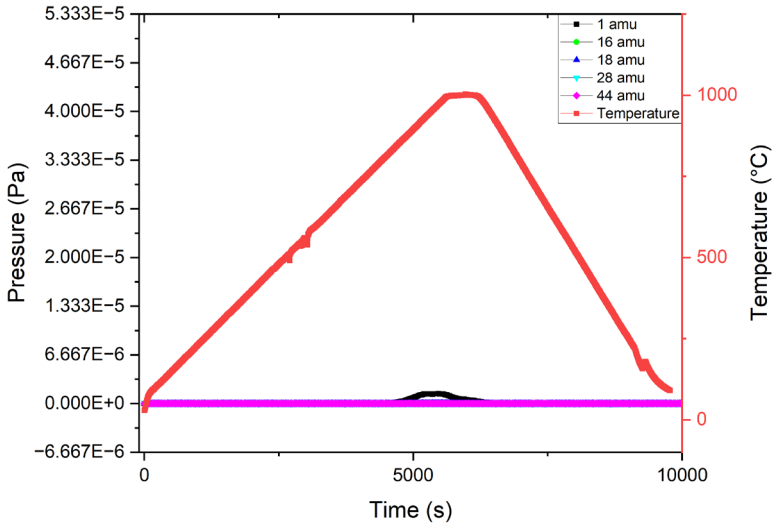

Fig. 5. Effusion measurement with 10°C/min.

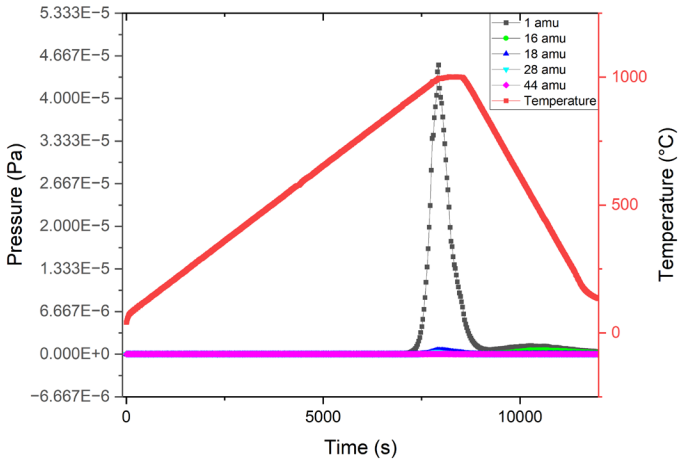

Fig. 6. Effusion measurement with 7°C/min.

Bonded Substrates

A similar effect of the lower temperature ramp was observed during the bonding of 4H-SiC substrates. At a ramp rate of 10°C/min, numerous defects, including voids and disbonded areas, appeared, as shown in Figure 7, which were attributed to the re-organization of the material during the annealing process. To minimize these defects, the procedure was repeated with a slower ramp rate of 5°C/min, and an additional step at 550 °C for 1 hour to enhance mass loss, allowing for more controlled outgassing and material organization. As shown in Figure 8, this approach resulted in void-free bonded 4H-SiC substrates, demonstrating the effectiveness of PCS as a bonding interface for SiC at high temperatures.

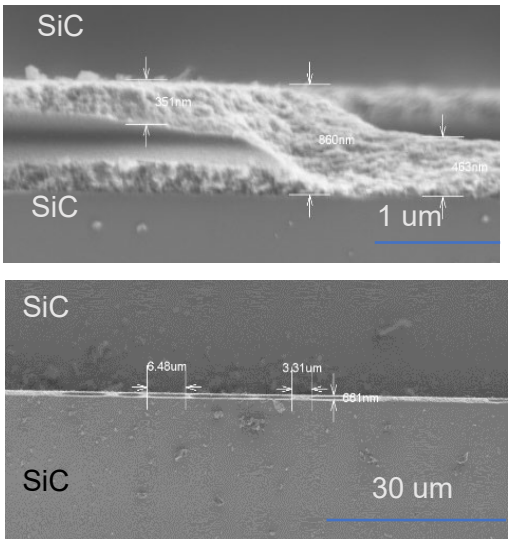

Fig. 7. The cross-sectional SEM images of the bonded monocrystalline 4H-SiC substrates at 10 °C/min temperature ramp with the presence of defects like voids and disbands.

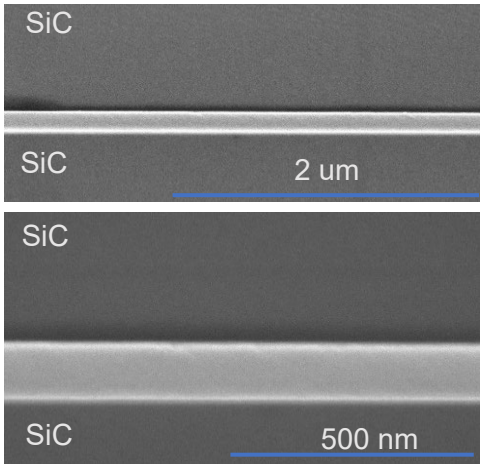

Fig. 8. The cross-sectional SEM images of the bonded monocrystalline 4H-SiC substrates at 5 °C/min temperature ramp with a uniform bonded area with no defects.

Crystallization of the SMP-10 layer was also studied by spin coating this material onto a monocrystalline 4H-SiC substrate, followed by annealing at 1550 °C for one hour with a one-hour dwell time at 550 °C in a REETZ furnace under an Ar atmosphere. However, XRD results indicated an amorphous phase, potentially due to rapid cooling, which may have inhibited crystal formation. Further investigations are needed to explore this in detail, along with the impact of *in-situ* nitrogen doping on improving electrical film conductivity.

Summary

This study demonstrates the potential of Polycarbosilane (PCS) as an effective adhesive bonding material for 4H-SiC substrates, particularly in high-temperature applications. Using PCS, combined with optimized annealing conditions, enables the formation of defect-free, high-temperature stable bonds. Effusion measurements indicated that lower temperature ramp rates play a critical role in minimizing defect formation, as material re-organization becomes the dominant factor, as even at higher outgassing rates, a defect-free interface was obtained. These findings highlight the importance of controlled outgassing and temperature management in bonding processes. PCS shows promise as a bonding interface for SiC substrates, paving the way for further research into doped SiC layers and their electrical properties for advanced power electronics applications.

Acknowledgements

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