

Silicon and Silicon Carbide Survivability in an In-Cylinder Combustion Environment

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Abstract: Silicon Carbide is often proposed as a sensor material for use in harsh environment applications such as monitoring gas turbines and internal combustion engines. However, little SiC survivability research has been reported for these environments. In this work, data is reported for silicon and amorphous silicon carbide (a-SiC) coated die survivability in a combustion engine with an exhaust temperature of 800°C. It was found that an oil residue was deposited on the test samples, the surfaces of both sample types were roughened, and no measurable oxide was grown. Preliminary data indicate that the oil deposition rate increases with time for both samples, but more oil adheres to the silicon. There appears to be no correlation between material type and the severity of the surface roughening. These results lay the groundwork for robust in-cylinder SiC sensor design and operation.

Key words: Silicon Carbide, a-SiC, Harsh Environment Sensors, In-Cylinder Testing, Ion Beam Sputtering

1. INTRODUCTION

In-cylinder combustion monitoring is needed both to improve current combustion engine efficiency and to drive fuel flexibility technologies. Sensors in the cylinder could provide information about combustion events, allowing for engines which can alter sparkplug timing to burn a variety of fuels with minimum emissions. Furthermore, individual cylinders could be monitored to ensure that all cylinders were running stoichiometrically. A suite of SiC, or SiC encapsulated, MEMS sensors would be well suited to combustion monitoring given their ability to operate at elevated temperatures and in corrosive environments [1]. Previous work has demonstrated an in-cylinder silicon carbide pressure sensor operating in a clean alcohol flame [2].

Before implementing such sensors, the effects of the typical automotive engine environment on sensor materials must be fully characterized to design successful and robust in-cylinder MEMS. This research measured the deposition of combustion byproduct residue deposition from fuels characteristic of operating automotive engines, as well as the oxide growth rate and the surface roughening of Si and SiC in the combustion environment.

2. BACKGROUND

In combustion engine environments the greatest concerns for sensor survivability and usability are the deposition of combustion byproducts, the growth of oxides, as well as physical and corrosive wear. Most automotive components near the engine typically have some films or oils deposited during normal operation.

Furthermore, particulates in the cylinder could potentially cause physical and corrosive wear on components. Finally, high temperatures and fluctuating pressures within the cylinder environment could potentially cause oxidation and other unforeseen conditions.

For MEMS based sensors, small changes in surface conditions such as oxide growth, material wear, and film deposition can largely affect the operation of the device, especially since these phenomena may cause changes in sensor characteristics without causing sensor failure. Effects such as the loss of mass and stiffness to a membrane can change the calibration of a pressure sensor or the frequency of oscillations of a resonator. Thus, the time effects of environmental factors must be characterized to properly design sensors for this harsh environment.

Previously, ion beam sputtered amorphous SiC (a-SiC) has been proposed as a potential encapsulation material since it uses line of sight deposition [3,4]. Given the previously demonstrated resistance to corrosive media, such as hot KOH, it is believed that this material would be well suited to the engine harsh environment. However, a-SiC films have a higher oxidation rate than that of the three types of crystalline SiC due to the higher number and concentration of active sites [5]. Furthermore this material has not been tested in a typical automotive engine. The experiments described in this paper investigate the question of Si and SiC sensor survivability in a transportation combustion environment by examining Si and SiC samples inserted in a combustion engine.

3. EXPERIMENTAL SETUP AND TESTING

The testing was conducted in a Waukesha Motor Company Cooperative Fuel Research engine (CFR). A test fixture, *Figure 1*, was designed to allow the test die to be mounted with direct exposure to the combustion chamber, *Figure 2*. The test die were mounted on tungsten disks with high temperature ceramic adhesive. The engine was run at 1800 rpm, stoichiometrically combusting standard US-grade 91 octane gasoline. The exhaust temperature was maintained at 800°C and the coolant temperature was 125°C. Based on a steady state conduction model, the interior cylinder wall temperature was calculated to be approximately 170°C. Since the die surface is thermally isolated from the cylinder wall, the surface temperature is expected to be approximately 300°C. Test durations of 20, 40, and 60 minutes were conducted. Samples before and after testing are shown in *Figure 3*.

During testing, samples became coated with an oily residue. To measure the oil film thickness, a portion of the film was removed with a razor blade and an optical microscope was used to compare step heights between the oil coated and now clean surface. Three separate locations were measured on each sample and the average thickness is reported. The oil was then removed completely with isopropyl alcohol. Given the low temperatures, no oxide growth was expected; however, to verify this assumption, the surface was patterned with an array of one-micron wide lines. The step heights across the lines were measured using an atomic force microscope (AFM).

The AFM was also used to measure the surface roughness of three locations on each of two 60 minute Si and two 60-minute SiC test samples. Control samples of Si and SiC were also scanned for surface roughness to determine the before and after effects of the two materials.

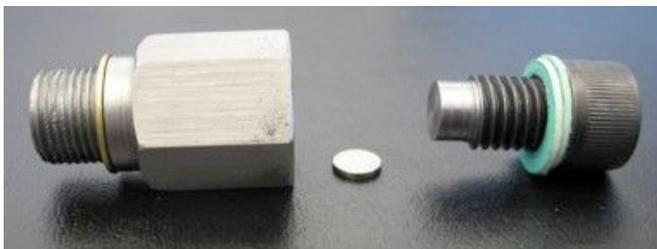


Figure 1: Test fixture for Si and SiC in-cylinder oxide testing. The Si or SiC die are mounted to the tungsten disk (center) with a high temperature ceramic adhesive (Cotronics 490LE). The tungsten disk diameter is 9.5 mm.

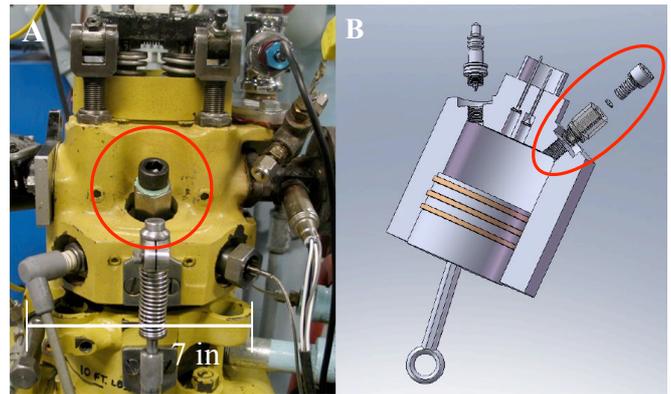


Figure 2: A) Waukesha Motor Company Cooperative Fuel Research engine with the actual test fixture in place (circled in red). B: Schematic of the cylinder showing the position of the test fixture (circled in red)

4. RESULTS

An oily residue was deposited on both the Si and SiC samples while in the combustion engine environment, and is shown in *Figure 4*. *Figure 5* shows results of the film thicknesses as a function of time and material. A greater thickness of residue adhered to the Si, ranging from 4 microns to 8 microns, than the SiC, ranging from 0.5 to 4.5 microns.

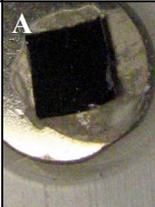
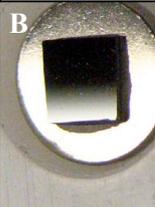
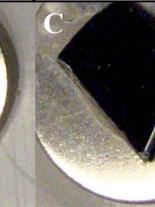
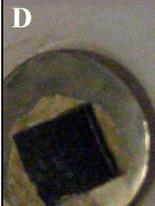
	20 min	40 min	60 min
Before Engine Testing			
After Engine Testing			

Figure 3: A-F Silicon Carbide Testing. (top row) Group 1 samples before engine testing. (bottom row) The same sample after 20, 40, 60 minute engine runs. The sample in F has been removed for processing.

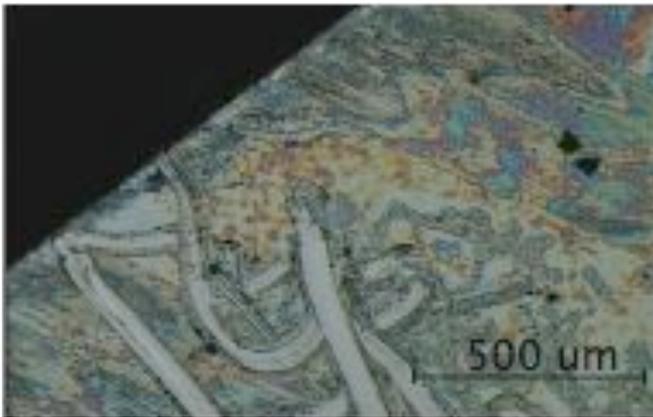


Figure 4 - Silicon die after a 20 minute test. The 3 μm thick oily residue on the die surface is visible in this 10x magnification optical microscope image.

An oxide thickness verification measurement was conducted using atomic force microscopy. An AFM window of 5 microns by 0.625 microns was used. Three locations were tested on each sample to find an average oxide thickness. Each location on each sample had two-dimensional traces taken to show the line profiles across each sweep. However, as expected, no oxide was detectable.

The surface roughness results for Si and SiC are shown in Figure 6 and Figure 7. The surface roughness values were 0.23 nm Ra and 0.42 nm Ra respectively. The control samples both had mean surface roughness values that were lower than the average roughness of their corresponding tested samples. The SiC roughened to an average of 1.1206 ± 0.6506 nm, and a representative example is shown in Figure 6. The Si roughened to an average of 0.8346 ± 0.2653 nm, and a representative example is shown in Figure 7. This difference does not appear to be statistically significant.

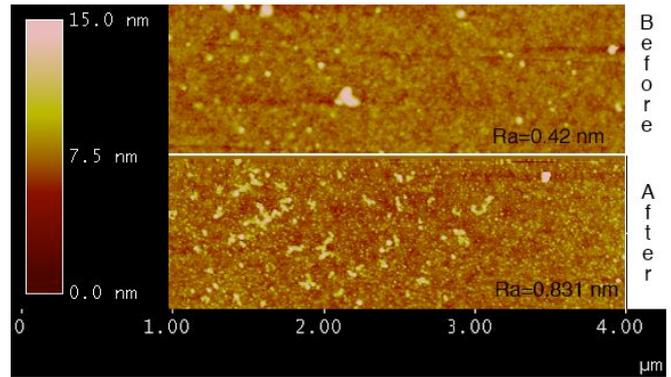


Figure 6- AFM roughness images of SiC before and after a 60 minute test in the CFR. The average roughness (Ra) for each image is shown.

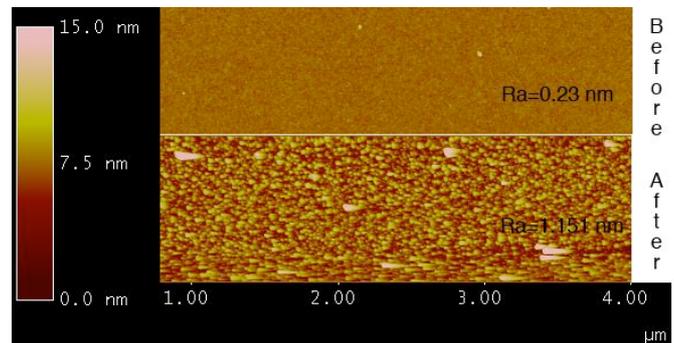


Figure 7- AFM roughness images of Si before and after a 60 minute test in the CFR. The average roughness (Ra) for each image is shown.

Oily Residue Thickness

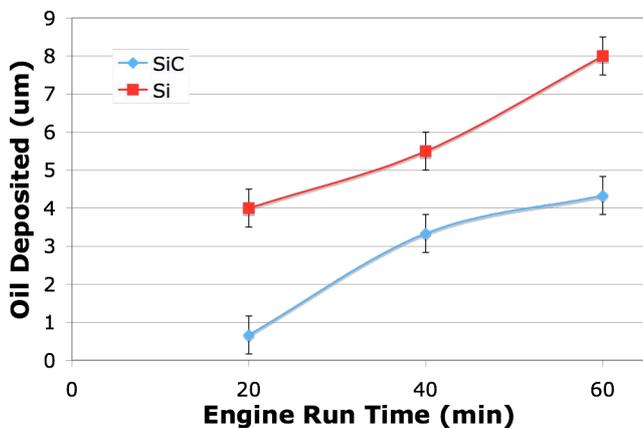


Figure 5- Oil residue on Si and SiC samples- 3 samples for each material at each time period.

5. DISCUSSION/CONCLUSIONS

The oily residue deposited on the samples was thicker than expected, possibly due to the recessed location of the test die within the fixture, which results in a surface temperature closer to that of the steady state wall, 170°C, than the exhaust, 800°C. This lower temperature can allow a greater build up of contaminants. The build up of contaminants can likely be resolved by either moving the sensor closer to the cylinder, heating the sensor or finding a different material with lower oil adhesion characteristics. If heating is determined to be the optimum solution, the power input for heating would be minimal because of the small thermal mass afforded by MEMS sensors.

The lack of oxide growth is commensurate with the relatively low temperature predicted at the sensor surface. However, the oily residue may be acting as a passivation layer. This will be investigated by testing in a higher temperature environment and for longer durations. If the trend of immeasurable SiC oxidation within the combustion environment continues at higher temperature exposures for longer periods of time, this will be extremely beneficial for sensors designed for the in-cylinder environment. Such sensors will not have

to account for added mass or stress to structures such as diaphragms or beams from oxide growth, which could lead to device drift or failure.

While the surface temperatures seen in a standard combustion engine are unlikely to cause significant diffusion deep into the sample, the surface roughness of Si and SiC samples were measured pre- and post-engine testing to correlate back to the introduction of other materials into the exposed surface. A greater roughening of the SiC than the Si would indicate the presence of enhanced surface diffusion in the amorphous film [6].

However, in this case, the surface roughening of SiC suggests that particulates from combustion could be bombarding and damaging the surface. There may also be a small native oxide growth at 300°C that is not detectable by AFM measurements. Future studies will use surface analysis techniques to further investigate. There is no clear evidence that Si or SiC roughens more than the other within the 60-minute test period given the large variation in roughening between different samples. Longer test durations are needed to definitively determine the material wear within the combustion environment.

The preliminary results indicate that future MEMS sensors for in-cylinder engine environments need to incorporate strategies which mitigate the effect of deposited oils and residues. Furthermore, these results show that for tests up to 1 hour, that physical damage and oxidation of sensor materials is negligible. However, long-term testing is needed to characterize how these materials will perform over the life of a typical automotive engine.

6. FUTURE WORK

Future experiments will focus on the characterization of the effects of spark plug proximity, oil layers on oxide growth, and different fuel types, as well as long-term operation. Rutherford Back Scattering (RBS) or other analytical techniques will be conducted to determine the elements present on the Si and SiC surfaces.

A wire feedthrough has been designed and built. Sensors will be mounted in the engine and real time in-situ data will be collected. These sensors will be tested for oil residue deposits, oxide growth and surface roughening post testing in the engine.

7. ACKNOWLEDGEMENTS

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