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# Investigating the application of semiconductor manufacturing technology to sealing stainless steel plates in high temperature reforming devices

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**Abstract**

The creation of high-performing and durable seals which can join stainless steel parts together is critical for applications such as reforming processes. While ferritic stainless steel is an exceptionally good choice of material for high temperature processes due to its manufacturability, cost effectiveness, and continued resistance to corrosion at high temperatures, it is also difficult to create seals which act as electrical insulators. Many insulators and dielectric materials with suitable high-temperature characteristics, such as glass, are unable to form a performant and durable seal due to the large difference in coefficients of thermal expansion (CTE). The discrepancy in expansion and contraction during heating and cooling cycles can lead to delamination from the stainless-steel substrate or fractures within the seal itself, either of which would render the equipment containing said seal inoperable and potentially causing further damage.

This paper explores methods for sealing stainless steel plates by leveraging technology from the semiconductor industry. Development of a multi-layered composite sealing and bonding method will be accomplished using equipment designed for handling and processing silicon wafers. By layering several coatings to form a seal, properties such as resistivity, adhesion, overall CTE, and self-healing properties will be able to be finely tuned to suit specific applications. Beginning with an assessment of spin-on glass, the use of fabrication methods originally developed for the semiconductor industry is investigated for the production of these composite high-temperature seals.

Reforming, High-Temperature Sealing, Semiconductor Fabrication, Wafer Bonding, Bonding, Composite Seals, Manufacturing

## 1. Introduction

In high temperature processes such as reforming operations, ferritic stainless steel is an exceptionally good choice of material for processing equipment due to its manufacturability, cost effectiveness, and resistance to corrosion at high temperatures. However, creating seals which can bond ferritic stainless-steel parts and act as electrical insulators, crucial for processes where generating a voltage differential is necessary, is difficult due to the extreme conditions this equipment is exposed to. Many existing insulators and dielectric materials with suitable high temperature characteristics are unable to function effectively as seals due large differences in coefficients of thermal expansion between the seal and the stainless-steel substrate. To overcome these issues, development of a composite seal which can self-heal during thermal cycling and is able to be tuned for physical and electrical properties is necessary. The development of this composite seal leverages fabrication techniques originating in the semiconductor industry, where uniform material deposition to form thin films is commonplace.

Previous work on the development of sealing methods for stainless-steel interfaces has focused on characterizing the mechanical and chemical interfacial mechanics between substrate and homogeneous sealing glass[1,2]. Meanwhile, work on thin layer deposition to stainless-steel substrates using semiconductor fabrication techniques have concentrated on thin layer characterization and interfacial characteristics[3–6] rather than practical application in the fabrication of high-temperature seals.

The research described in this paper utilizes these semiconductor fabrication techniques in conjunction with interfacial analysis and testing within both mechanical and electrical regimes.

## 2. Methodology

To develop a composite sealing method, methods originating in the semiconductor manufacturing industry for thin layer deposition will be independently assessed for suitability and compatibility with stainless-steel substrates. Evaluation is carried out using test samples fabricated from AISI 430 stainless-steel, chosen as a representative ferritic stainless-steel due to cost effectiveness and availability. Results from electrical and mechanical testing of individual thin layer deposition methods will be leveraged to determine material and fabrication method choices for a composite seal.

### *2.1. Sample Preparation*

Test samples were fabricated from AISI 430 stainless-steel sheet 0.5mm thick purchased from McMaster-Carr (P/N: 3803T18). As the material was received in a roll form, it needed to be flattened before individual test samples could be cut from it. This was accomplished by cutting the sheet metal into rectangular sections approximately 50mm wide and 150mm long. These sections were then flattened by heating them between two alumina slabs approximately 200mm square and

25mm thick which were ground to a flatness of less than 5µm. A thermocouple was also placed between the alumina slabs adjacent to the stainless-steel sheet to monitor the temperature at the centre of the "sandwich".

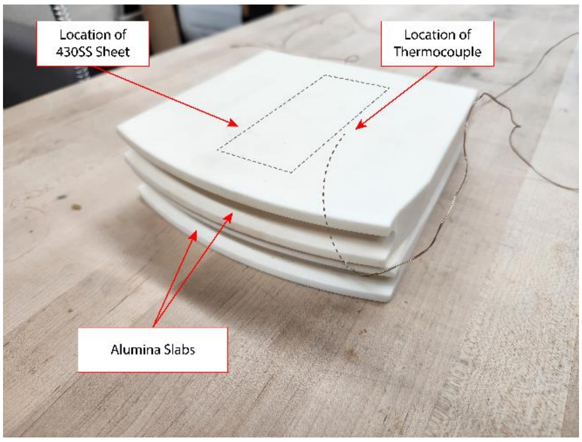


**Figure 1.** Alumina Slab "Sandwich" with Thermocouple

The flattening procedure was based on guidelines provided for annealing 430 stainless-steel by one of its major manufacturers[7]. The furnace used for the flattening process, a Vulcan A-550 Box Furnace (Fig. 2) was preheated to 760°C before the alumina slab "sandwich" was inserted into the furnace. After the core of the "sandwich" reached 760°C, this temperature was held for 20 minutes before the furnace was switched off and its contents were allowed to cool to room temperature.

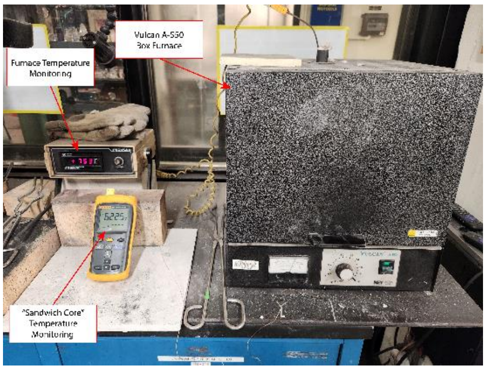


**Figure 2.** Vulcan A-550 Box Furnace Used for Sample Preparation

After the flattening process was completed, individual test samples in the form of 20mm diameter discs with two tabs for handling (Fig. 3) were cut from the sheet using a fibre laser. These samples were then polished on a Struers RotoPol grinding machine to a mirror finish, corresponding to an Ra of approximately 5µm.

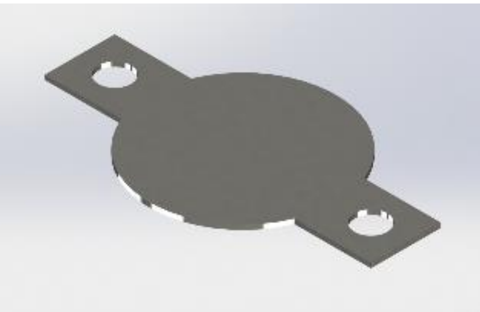

**Figure 3.** Computer Model of 430 Stainless-Steel Test Sample

### *2.2. Fabrication*

Spin-on glass (SOG) has been selected as the initial material for consideration. Applied as a liquid to a substrate on a spinner table, SOG offers good planarization and insulation characteristics; the thickness of the layer deposited is determined by the rotational speed of the spinner table (Fig. 4). Following this deposition in liquid form, the SOG is baked to form a material with similar properties to $SiO_2$. Density of the cured SOG is determined by the temperature of the bake, allowing another degree of tunability.[8]

The SOG compound used is Desert Silicon NDG-7000R, a non-doped formulation specified to provide a cured film thickness of 7000 Å when applied with a spin speed of 4000 RPM.[9]

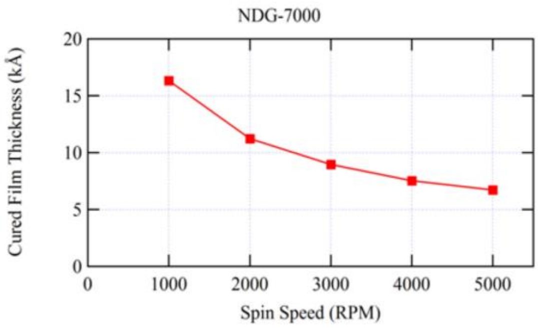


**Figure 4.** Cured Film Thickness vs. Spin Speed for NDG-7000R[9]

The spin-on glass is applied and processed using procedures common in the semiconductor industry with the ultimate goal of bonding two test samples together for mechanical and electrical characterization (Fig.5).

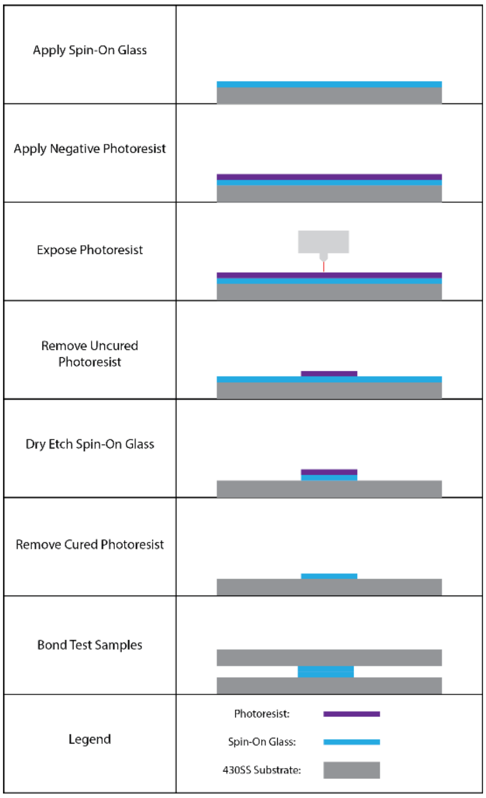


**Figure 5.** Spin-On Glass Processing

#### *2.2.1 SOG Application*

After each sample was cleaned with acetone, they were individually placed on a vacuum chuck spinner and approximately 0.25mL of Desert Silicon NDG-7000R was deposited on the centre of the sample using a micropipette. After ensuring that the sample was correctly centred and fully constrained by the vacuum chuck, the spinner was run at a speed of 1000 RPM for 120 seconds to yield a film thickness of approximately 1.5µm. The test sample was then immediately transferred to a hotplate and baked at 225°C for 240 seconds to cure the spin-on glass.

#### *2.2.2 Photoresist Processing*

After the test samples cooled, they were transferred to another spinner for photoresist application. Similarly to the previous processing step, each test sample was individually placed on the spinner and retained using a vacuum chuck. Approximately 0.25mL of AZ nLOF 2020, a negative photoresist[10], was deposited on the sample. The spinner was then run at a speed of 3000 RPM for 120 seconds to yield a thickness of 2µm. After completion of the spinning, each sample was transferred to a 110°C hotplate for a 120 second pre-exposure bake.

The next step in the photoresist processing was to expose the photoresist. As negative photoresist was used, the section which needed to remain behind after development was the region of the wafer which was exposed. This was accomplished using a Heidelberg Instruments MLA 150 Advanced Maskless aligner. A 5mm diameter circular region at the centre of each test sample was exposed using the recommended parameters for nLOF 2020. Following the exposure, the samples were transferred to a 110°C hotplate for a 120 second post-exposure bake.

Following the exposure and post-exposure bake, the photoresist was developed. Each sample was immersed in AZ 726 MIF developer [11] for 60 seconds while being agitated, after which they were removed and rinsed in four consecutive baths of deionized water.

#### *2.2.3 Etching*

Following photoresist processing, the samples were dry etched to remove the spin-on glass surrounding the central area where photoresist remained. Multiple test samples were etched simultaneously for 10 minutes with $CH_4$ plasma using a Samco RIE-230iP Plasma Etching System. This etching cycle would be expected to etch $SiO_2$ on an Si substrate at a rate of 130nm/min for a total material removal of 1.3µm.

### *2.3. Testing*

To verify the concept of using spin-on glass as an insulating layer, the resistivity of the 2µm layer of Desert Silicon NDG-7000R was measured using a Fluke 117 True RMS Multimeter. With one probe contacting the bare steel underside of a test sample and the other probe contacting the side where spin-on glass was deposited, the multimeter read open loop, indicating that the glass layer was successfully acting as an insulator.

## 3. Results

Deposition of the Desert Silicon NDG-7000R spin-on glass was successful, with an even layer applied across the entire surface of the stainless-steel test sample. While the surface of the glass remained planar, inspection under an optical microscope revealed that the spin-on glass had fractured into micron-scale pieces (Fig. 6).

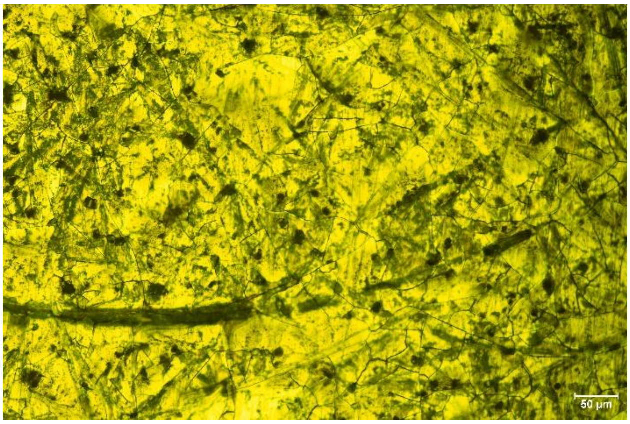


**Figure 6.** Test Sample Surface After Spin-On Glass Deposition

Apparent adhesion of the spin-on glass to the stainless-steel substrate was good, with the surface undamaged after measurement with an optical profilometer. This profilometer testing yielded an Ra of approximately 5µm, similar to that of the mirror-polished stainless-steel substrate.

Photoresist processing of the samples was also successful. After the exposure and development steps, a 5mm diameter region of cured photoresist remained at the centre of the test sample (Fig. 7)

**Figure 7.** Test Sample After Photoresist Processing

From imaging performed using an optical microscope, it was observed that the edge of the cured photoresist region had good definition with vertical sides that were not affected by any fractures in the spin-on glass surface below it (Fig. 8)

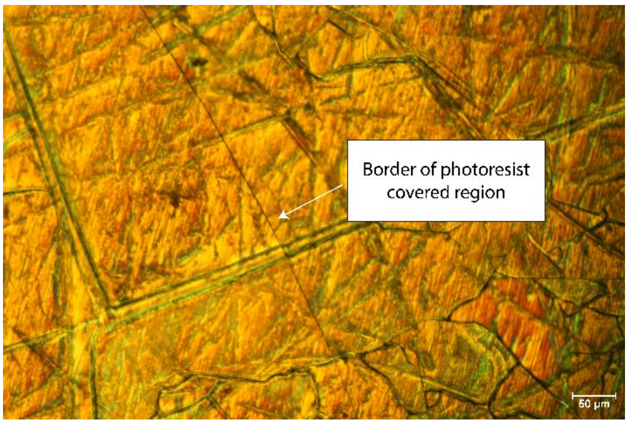


**Figure 8.** Test Sample Surface After Photoresist Processing

Following the dry etching process, the samples were again imaged using an optical microscope. Spin-on glass remained in the circular region which had been covered by the cured photoresist and the surrounding area had the majority of the spin-on glass previously present removed (Fig. 9). The same fracture pattern which had appeared when the spin-on glass was originally deposited remained, and the edge profile of the glass closely followed the shape of the photoresist pattern.

**Figure 9.** Test Sample Surface After Dry Etching

The next step taken was measurement characterization of the spin-on glass region using a stylus profilometer. It became apparent at this point that the dry etching process had caused the spin-on glass to delaminate from the stainless-steel substrate, as the stylus dislodged individual segments of glass and made measuring the profile of the spin-on glass impossible. Additionally, further review of the images previously taken revealed that the dry etching process had removed significantly more material than anticipated, with the entire cured photoresist layer removed. This indicates that the etching rate achieved was much higher than that expected for the same process carried out on a silicon substrate.

## 4. Summary & Conclusions

So far, this investigation of using semiconductor manufacturing methods for fabrication of high-temperature seals has determined that spin-on glass has promise as a material option due to its high electrical resistivity, ease of application, and compatibility with ferritic stainless-steel. However, the difficulties encountered during the dry etching process indicates that some semiconductor manufacturing processes must be adapted for use on a ferritic stainless-steel substrate and cannot simply be used as they are with silicon wafers. Once necessary adaptations are made though, these manufacturing methods which are already widely used and well understood in the semiconductor industry have the potential to be used in the fabrication of high-performance composite seals.

## 5. Future Work

For continuing development of a composite sealing method, tuning of processing methods such as dry etching for use on ferritic stainless-steel substrates will be necessary. Through this tuning process, an understanding of why the substrate material has such a significant effect on processing results will also be established.

In parallel with this tuning process, spin-on glass will continue to be testing, specifically as a bonding layer. As part of this process, test samples will be bonded together, and the bond will be mechanically characterized using a tensile testing machine.

For continuing development of a composite sealing method, assessment of additional thin layer materials and bonding techniques will be necessary. Physical vapour deposition (PVD) and chemical vapour deposition (CVD) will be investigated for suitability in fabrication of high-temperature seals, with Silicon nitride and metals such as aluminium and titanium being potential options using these methods.

To more closely simulate the conditions seals would be exposed to within a reforming stack or other high temperature processing equipment, mechanical and electrical testing at high temperatures, as well as temperature and load cycling. This high temperature testing will also be useful for determining whether the fracturing behaviour encountered in spin-on glass.

Following the development of a composite seal using these methods, testing in scaled systems which emulate reforming stacks will likely be the next step taken.

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