Calculation of Interfacial Strain Energy Release Rate Through Indentation and 4-Point Bending

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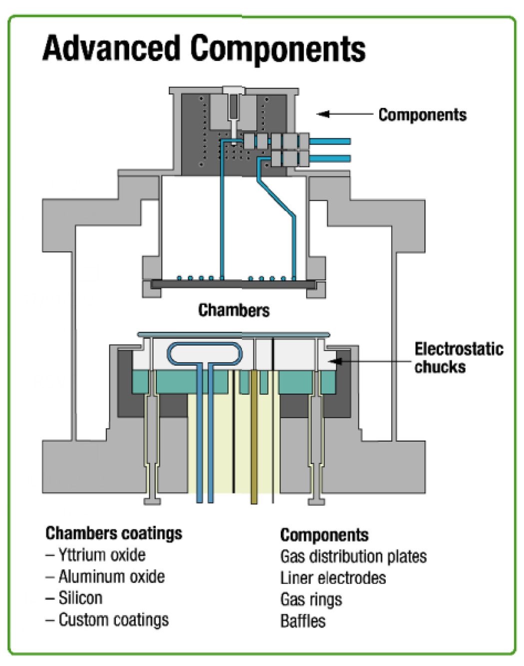
Abstract: FM Industries utilizes multiple coating machines to output their supply of semiconductors. These machines require a layer of ceramic coating to protect the interior. In this project, various coating and substrate combinations were tested to see which overall had the highest interfacial strain energy release rate, which would corroborate with a longer lasting coating for the internals of the coating machines. After testing, the best combination was deemed the Lanthanum Zirconium Oxide coating with the sealed type III anodized aluminum.

This work is sponsored by FM Industries, Fremont, California

FM INDUSTRIES, INC.
NGK group

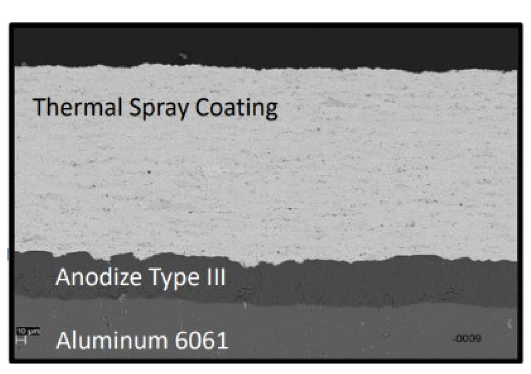
Background

Problem
Plasma etch chambers for semiconductor processing must be thermally spray coated to prevent etching the chamber by the process gasses. Due to the thermal cycling and normal wear and tear, the coating can flake off and destroy thousands of dollars' worth of microchips.



As seen by the diagram on the left, the main coatings used by FMI for the chamber's interior consist of Yttrium Oxide, Aluminum Oxide, and custom coatings. In this experiment three coatings, yttria, alumina, and lanthanum zirconate, were applied to three different substrates, 6061 aluminum, unsealed type III anodized 6061 aluminum, and sealed type III anodized aluminum. [1]

Project Goals:
Characterize the strain energy release rate of each coating on each substrate to determine the strongest adhering coating and make a recommendation to FMI.



SEM image of a thermal spray coating on Type III Anodized 6061 Aluminum [1]

Experimental Matrix

For this project the team tested a suite of different combinations of coating substrate systems. The coatings used were Yttrium Oxide, Aluminum Oxide, and Lanthanum Zirconium Oxide. Each of these coatings were applied to all three substrates. The three substrates are bare, Type III Anodized, and Type III Sealed Anodized, all of which are 6061 Aluminum.

From the three coatings and three substrates, nine coating substrate systems were created for testing.

All 9 different indentation samples photographed from above with the alumina samples highest the yttria samples in the middle and the lanthanum zirconate samples at the bottom

In addition to the samples for indentation, the team received a different set of samples for the four-point bend tests. These samples focused on a different aspect of the plasma spray coating process of whether the surfaces that are sprayed are just machined before spraying or if they are grit blasted to roughen the surface first. The team received three five inch by one inch by 0.0162-inch bend bars for each condition. The conditions were grit blasted and machined bare aluminum surfaces. The goal with these samples as well was to obtain a strain energy release rate for each condition. Understanding the mechanics of coating behavior on each preparation if substrate will help FMI make informed manufacturing decisions.

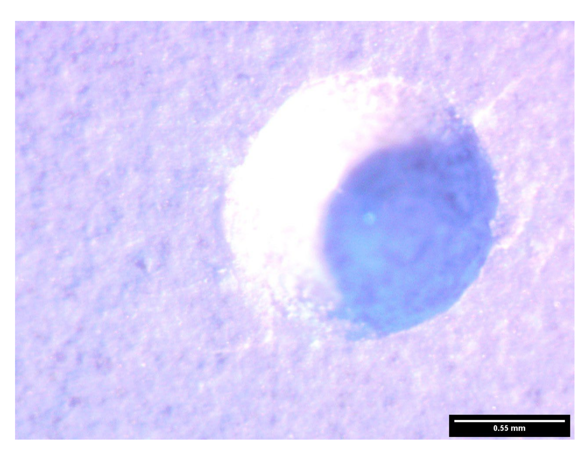
Uncoated machined aluminum 4-point bend sample used for test setup validation

Indentation Testing

Indentation tests were performed on the nine coupon samples that were received from FM Industries. Each coupon was tested three times by using the Rockwell Hardness Tester with a conical 120-degree diamond tip to apply a 150-kg load. This test was performed to identify the amount of delamination and deformation that occurred at a set load among the samples to solve for a bi-material's critical energy release rate.

$$G_c \approx \frac{dU_f}{dA} = \frac{dU_f}{dc}\frac{dc}{dA} = \frac{(\kappa \frac{dP}{dc} + \lambda)t}{(c - a\frac{da}{dP}\frac{dP}{dc})} \quad [2]$$

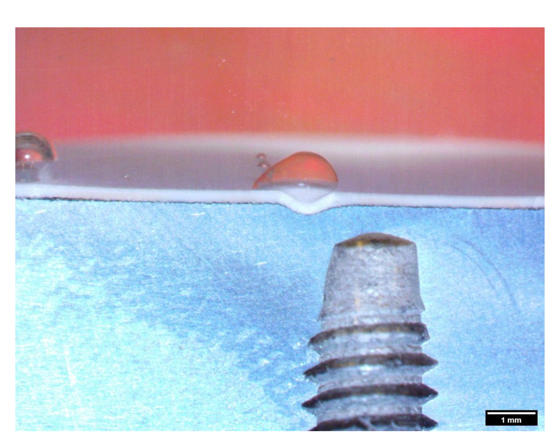
The ceramic coatings' strengths allowed for the majority of the coupons to withstand the load applied at the surface. This caused the need for cross-sectioning with a diamond saw and polishing down to the center of the indent. By doing this the team was able to measure the crack size that had occurred.

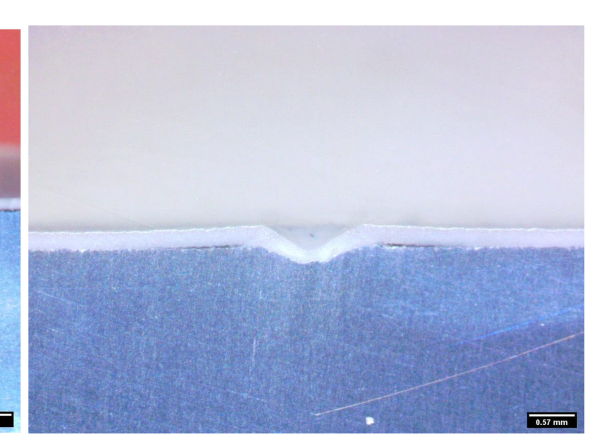


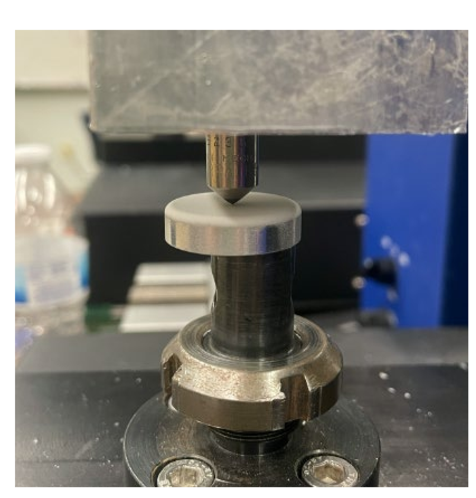
(Above left) Top view of an indent showing that the surface does not reveal crack length.

(Below left) Lanthanum Zirconate on Type III Sealed Anodized crack cross section

(Below Right) Alumina on Bare Aluminum showing the crack cross section







Variable force indentation jig

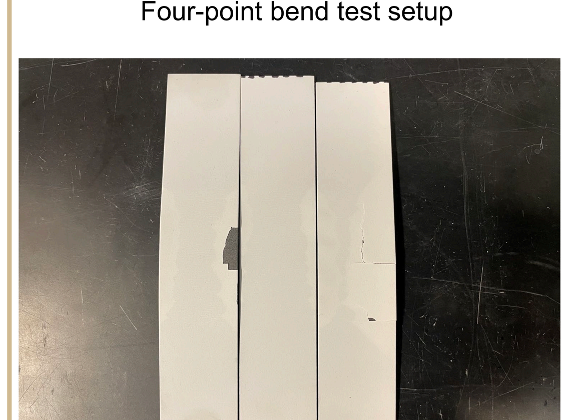
Using a custom designed and machined aluminum jig for connecting the Rockwell indenter tip to an ADMET MTEST QUATTRO load frame, the team was able to achieve variable loading forces. With these additional forces and crack lengths, the dP/dc curve can be more thorough and therefore yield a more accurate resultant strain energy release rate

Four-Point Bend Testing

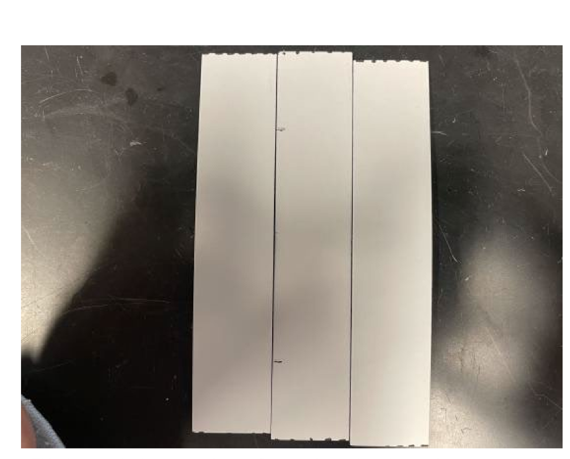
In addition to utilizing indentation testing to examine the strength of chamber coatings, four-point bend testing was utilized to simulate the thermal expansion and contraction that high-temperature chambers at FMI would experience. Through this volume change, coatings would wear and delaminate and spall over time. A test was conducted utilizing grit-blasted and machined aluminum bars as a substrate, with a thin coating of alumina on top.

Four-point bend test setup

Through following the MIL-STD 1942A and ASTM_c1161, the layout of which is shown on the left, each sample's coating was subjected to a tensile stress, due to the orientation of the sample at testing [3]. Below are the variations of both grit and machined sampled.



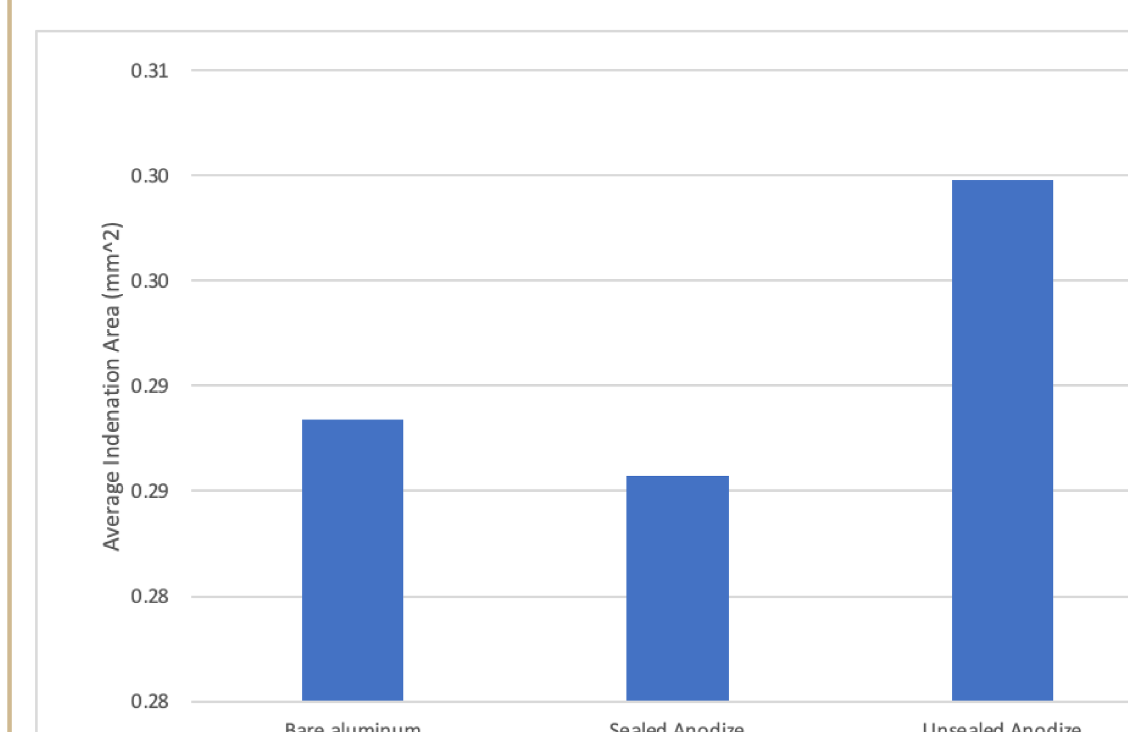
Left: Machined surface 4-point bend samples



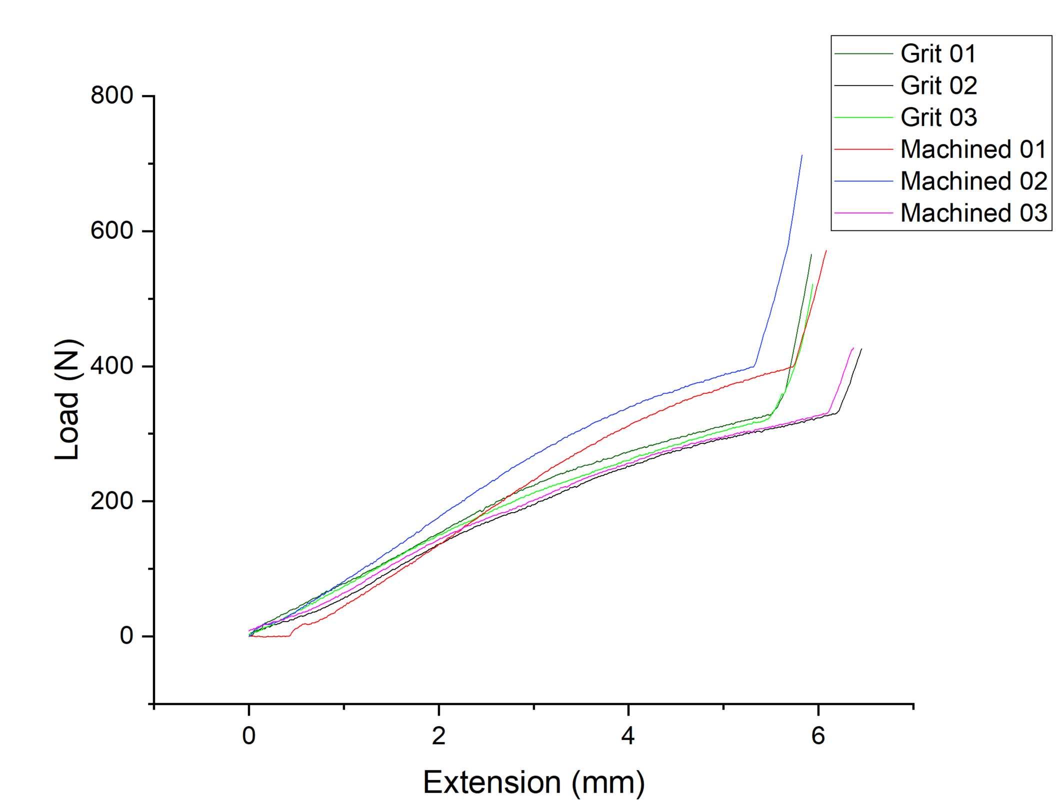
Right: grit-blasted surface 4-point bend samples

Results & Conclusions

The indentation tests yielded crack length data for each loading which could then be used to calculate the strain energy release rate of the coating on the substrate. With this the team can make a recommendation to FM Industries of the best coating of the tested coatings. The best coating substrate system the team tested was Lanthanum Zirconium Oxide coating with the sealed type III anodized aluminum. This is because it has the highest strain energy release rate the therefore the strongest adhesion to the substrate. Below the average indentation area for each coating across all substrates is shown, this relates to the strength of the interface and the higher the area is, the lower the strength is. This corroborates with our result that the sealed anodized coating is the strongest.



Average indentation area for each substrate across all coating types



Load versus extension graph for all the four-point bend samples

Interestingly, through analysis of the 4-point bend samples, visually they are more contradictory than they are graphically. The graphs show that the substrate-coating interface had minimal overall impact on the plastic region of the sample. However, visually, the grit blasted coatings show next to no delamination or spalling compared to the machined coatings. This would follow with what FMI currently utilizes for their smaller scale coatings, with the anodized and "rougher" surface allowing for more surface area for the coating to adhere to, therefore raising the energy required dramatically as opposed to the smoother more machined surface. For larger scale applications, a grit blasted surface should be able to withstand more expansion and contraction than a machined surface will.

References

[1] C. Petorak PhD, M Naim PhD, D. Hammerich “FMI Coating System for Semiconductor Etch Tools,” 9/30/2022

[2] J.-J. Kim, J.-H. Jeong, K.-R. Lee and D. Kwon, "A New Indentation Cracking Method for Evaluating Interfacial Adhesion Energy of Hard Films," Thin Solid Films, vol. 22, no. 1-2, pp. 172-179, 2003.

[3] American Society for Testing and Materials, C1611 - Standard Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature, 2018.