

Abstract: Archimedes density testing is used to measure the density of post-processed ceramic matrix composite (CMC) seal segments in aircraft turbines. Presently, ethanol is used as the immersion solvent but poses health safety risks to technicians. This study seeks to evaluate alternative solvents that pose a decreased safety risk while maintaining accuracy and not adversely affecting the bulk component or its coating. Microstructural and elemental analysis was carried out to evaluate the effect of each solvent on the CMC components.

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Cypress, CA
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Background & Objectives

Rolls-Royce (RR) utilizes Archimedes density method to determine the density of ceramic matrix composite (CMC) components used in seal segments in aircraft turbines.

Archimedes method is a density measurement technique based on Archimedes' Principle of Buoyancy. The process involves:

1. Measuring a sample's mass in air
2. Immersing the sample in a fluid and recording the immersed mass

Next, the difference in dry versus immersed mass is calculated (Eq. 1), followed by calculating the sample's volume (Eq. 2), and finally calculating the sample's Archimedes density (Eq. 3).

$$m_{displaced} = m_{air} - m_{fluid} \text{ (Eq. 1)}$$

$$V_{sample} = \frac{m_{displaced}}{\rho_{fluid}} \text{ (Eq. 2)}$$

$$\rho_{sample} = \frac{m_{air}}{V_{sample}} \text{ (Eq. 3)}$$

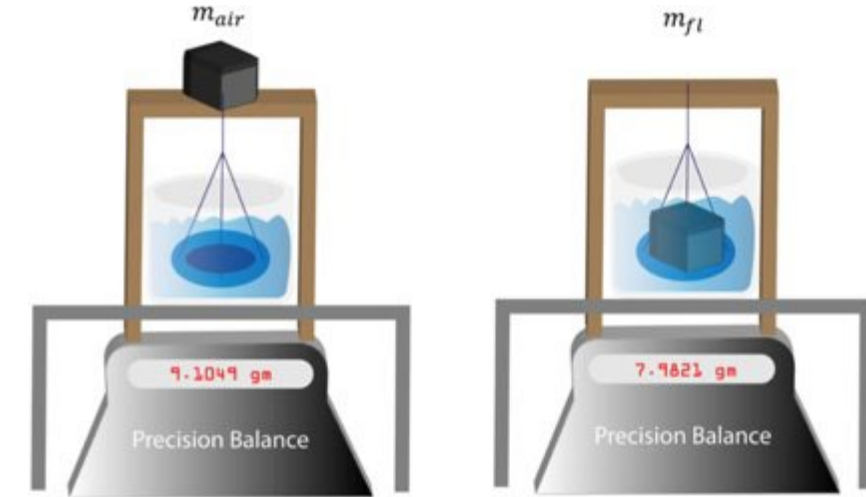


Fig. 1: Archimedes setup [1]

Currently, RR uses ethanol as the immersion fluid. Ethanol poses health and safety risks, including increased flammability risk and eye irritation. The objective of this work is to identify a safer solvent. This solvent should

- Minimize health and safety risks
- Retain accuracy of density measurements
- Not adversely affect the CMCs microstructure

Archimedes density testing, combined with subsequent microstructural and elemental analysis are used to determine the optimal solvent to potentially replace ethanol.

Solvent Research

The focus of this study is to determine a suitable alternative solvent to ethanol. Solvent candidates were screened on health hazards, flashpoint, evaporation rate, and cost.

Ethanol (control solvent)

| Advantages | Disadvantages |
|---|---------------------|
| Suitable evaporation rate | Highly flammable |
| No further processing steps needed (e.g., cleaning) | Eye irritation risk |

Flammability and Toxicity

Liquids are considered flammable if they have a flashpoint below 93°C. Flammability risk is broken down into four categories (Fig. 2).

| Category | Flashpoint | Boiling point |
|----------|------------|---------------|
| 1 | Below 23°C | Below 35°C |
| 2 | Below 23°C | Above 35°C |
| 3 | Above 23°C | Below 60°C |
| 4 | Above 60°C | N/A |

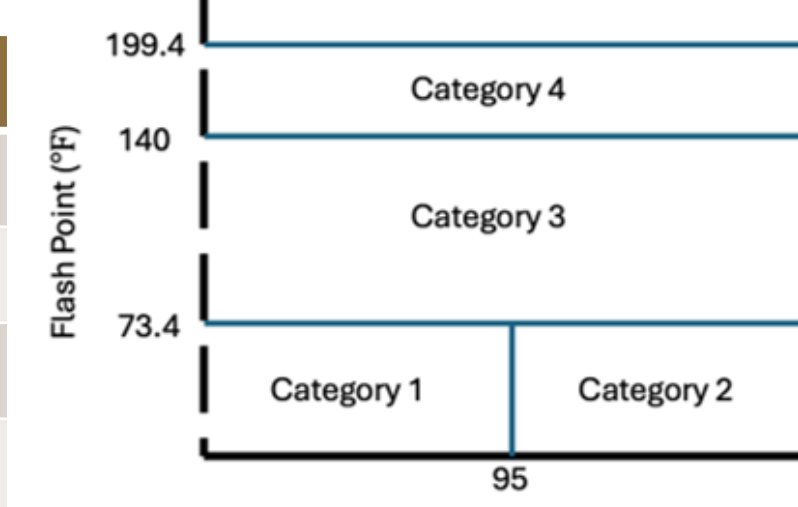


Fig. 2: Flammability chart [2]

Toxicity risks include eye irritation, organ toxicity, and skin irritation.

Final Candidate Solvents

| Solvent | Hazards | Flashpoint | Evaporation Rate | Cost |
|-------------|---------------------------|------------|------------------|--------------|
| Ethanol | Flammable, eye irritation | 13°C | 3.3 | ~\$180/Liter |
| Isopropanol | Flammable, eye irritation | 12°C | 2.9 | ~\$87/Liter |
| TRIM C-390 | None | >105°C | -- | ~\$14/Liter |
| NOVEC 7100 | None | -- | -- | ~\$300/Liter |
| DI Water | None | None | 0.3 | ~\$35/Liter |

Isopropanol (IPA): Common household chemical used in sanitizers and rubbing alcohol. Like ethanol, IPA is flammable, which is a major constraint.

TRIM C-390: Synthetic fluid used by Rolls-Royce as a coolant for machining CMCs. This is mixed as 90:10 DI water to TRIM C-390. Low flammability risk but can cause eye and skin irritation.

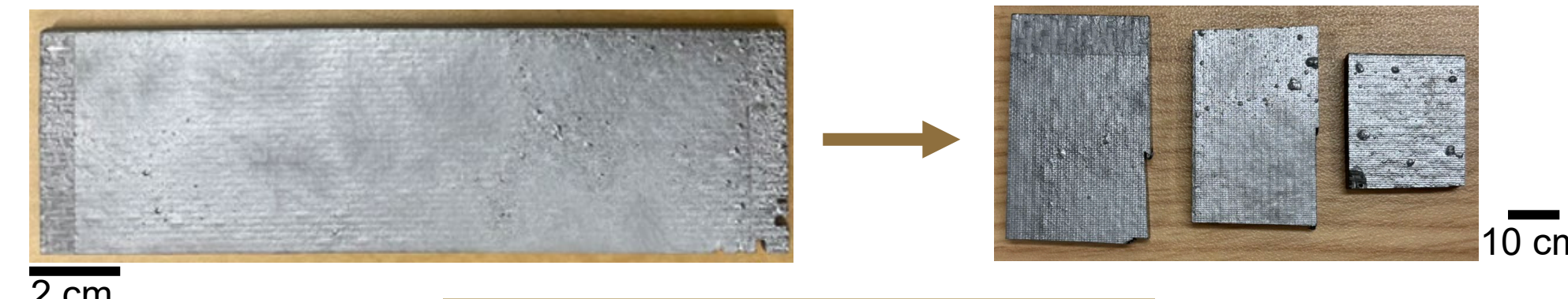
NOVEC 7100: Fluorinated solvent that is non-flammable and has low toxicity.

*Note: Evaporation rate is relative to n-Butyl Acetate (=1)

Archimedes Density Testing

Sample Preparation

2 sample plates were received from RR. A total of 16 pieces were sectioned from the plates using a diamond saw.



Archimedes Testing

The goals to be achieved include:

- Testing three candidate solvents using Archimedes method
 - Analyze effect of DI water on the CMC's coating
 - Conduct Gage Repeatability & Reproducibility statistical analysis
- We developed an Archimedes setup to be used based on Rolls-Royce's currently employed setup and ISO-18754. Our setup (Fig. 3), is shown below.

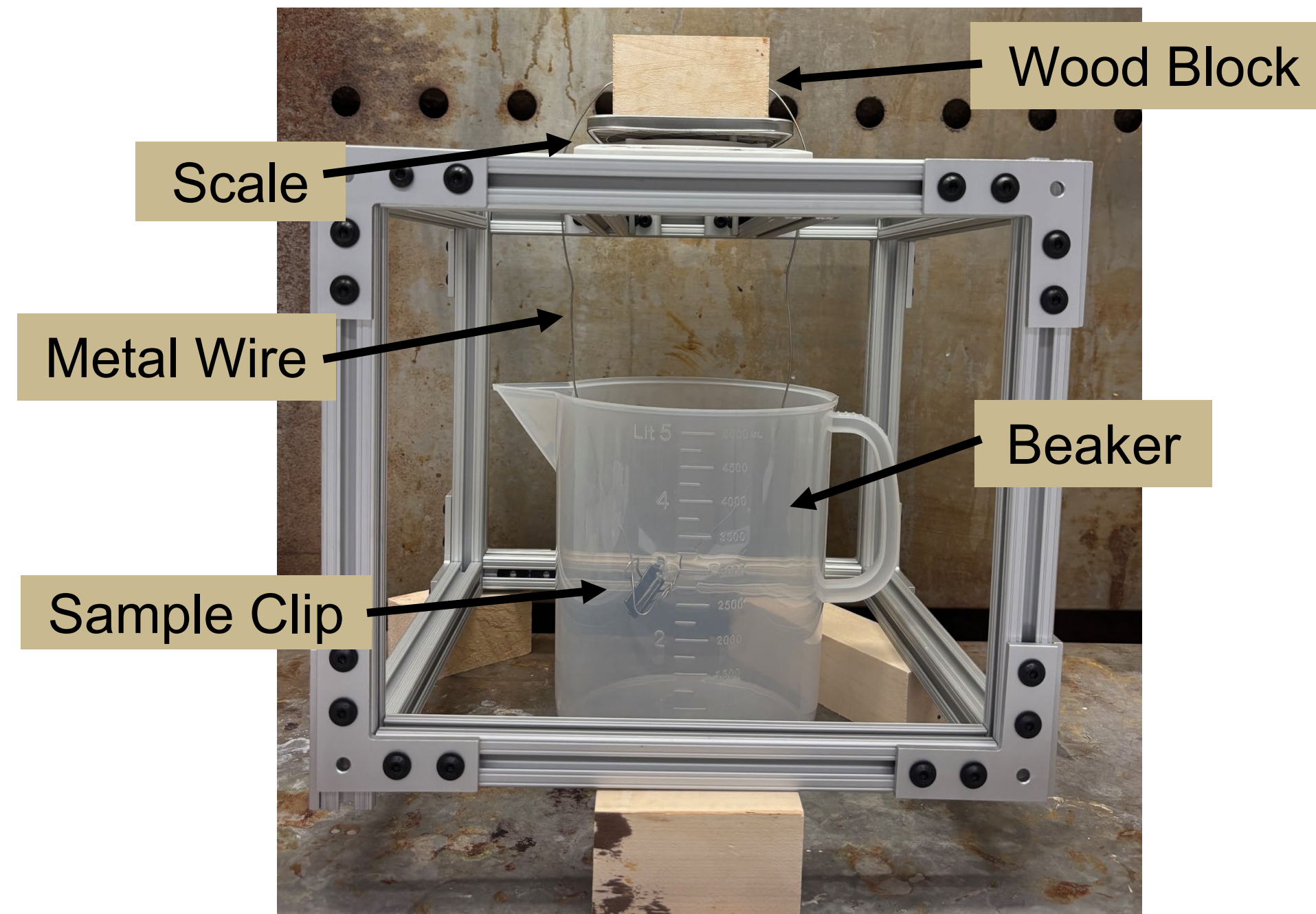


Fig. 3: Archimedes density testing setup

Archimedes Procedure

Pre-testing

- Samples placed in furnace for 2 hours at 125 °C to remove residual moisture

Archimedes measurements

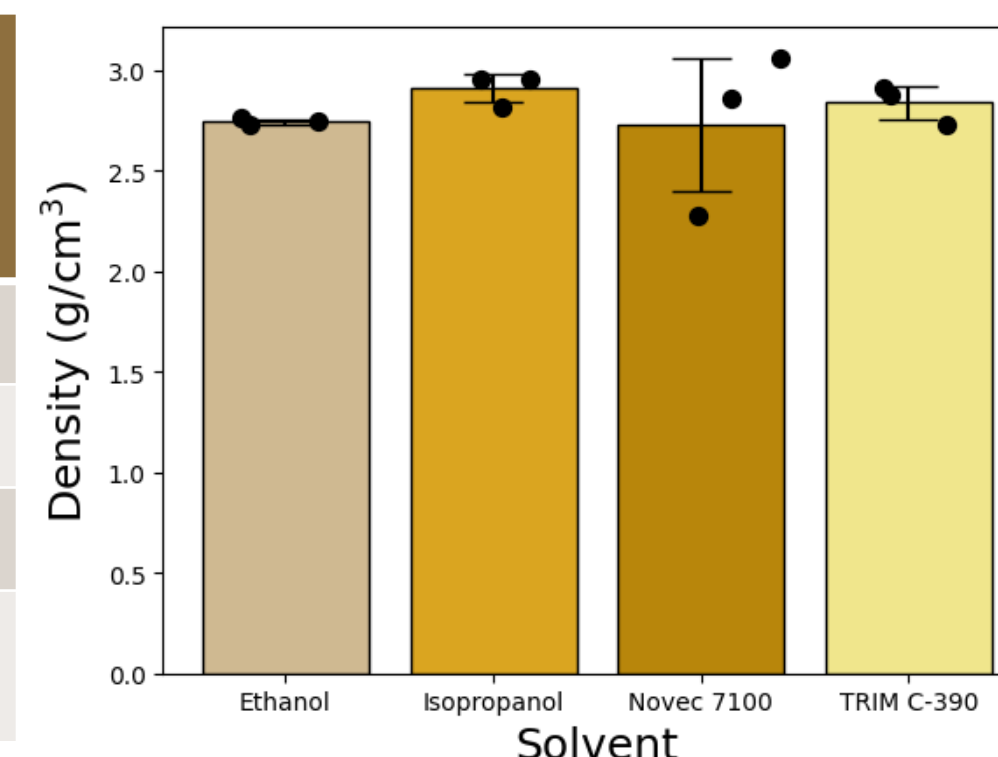
- Dry mass of samples recorded before immersion
- Three roles assigned for testing to meet Gage R&R requirement
 - Operator A: Attach sample to clip and remove sample
 - Operator B: Lift block from scale to assist in attaching and removing sample
 - Operator C: Track immersion time of 30 seconds
- Immersed mass recorded and sample removed after 30 seconds
- Samples removed then patted dry and stored for SEM/EDS analysis

Archimedes Density Results

Round 1 Archimedes Testing

- 3 samples were tested per solvent, with a soak time of 30 seconds
- Temperature of each solvent was measured as fluid density is a function of temperature

| Solvent | Average Density (g/cm ³) | Temp. (°F) |
|-------------|--------------------------------------|------------|
| Ethanol | 2.74 | 21.4 |
| Isopropanol | 2.91 | 22.6 |
| Novec 7100 | 2.99 | 22.6 |
| TRIM C-390 | 2.84 | 22.9 |



ANOVA statistical testing revealed that the difference between measured means is not statistically significant

Round 2 Archimedes Testing

- 3 samples were tested in every solvent, with parameters otherwise maintained from Round 1 (Ethanol, Novec 7100, IPA, TRIM C-390)
- Samples were dried in between testing in each solvent for 2 hours at 125°C

| Solvent | Average Density (g/cm ³) | Temp. (°F) |
|-------------|--------------------------------------|------------|
| Ethanol | 2.71 | 72.6 |
| Isopropanol | 2.66 | 71.4 |
| Novec 7100 | 2.64 | 72.3 |
| TRIM C-390 | 2.74 | 74.6 |

As before, ANOVA statistical testing revealed that the difference between measured means is not statistically significant

All solvents produce viable Archimedes density measurements

Microstructural & Chemical Analyses

Scanning electron microscopy (SEM) was used to conduct microstructural analysis of samples before and after Archimedes testing. Quantitative and qualitative analysis was carried out, with an emphasis placed on changes in fiber diameter, solvent residue, or structural defects such as pores.

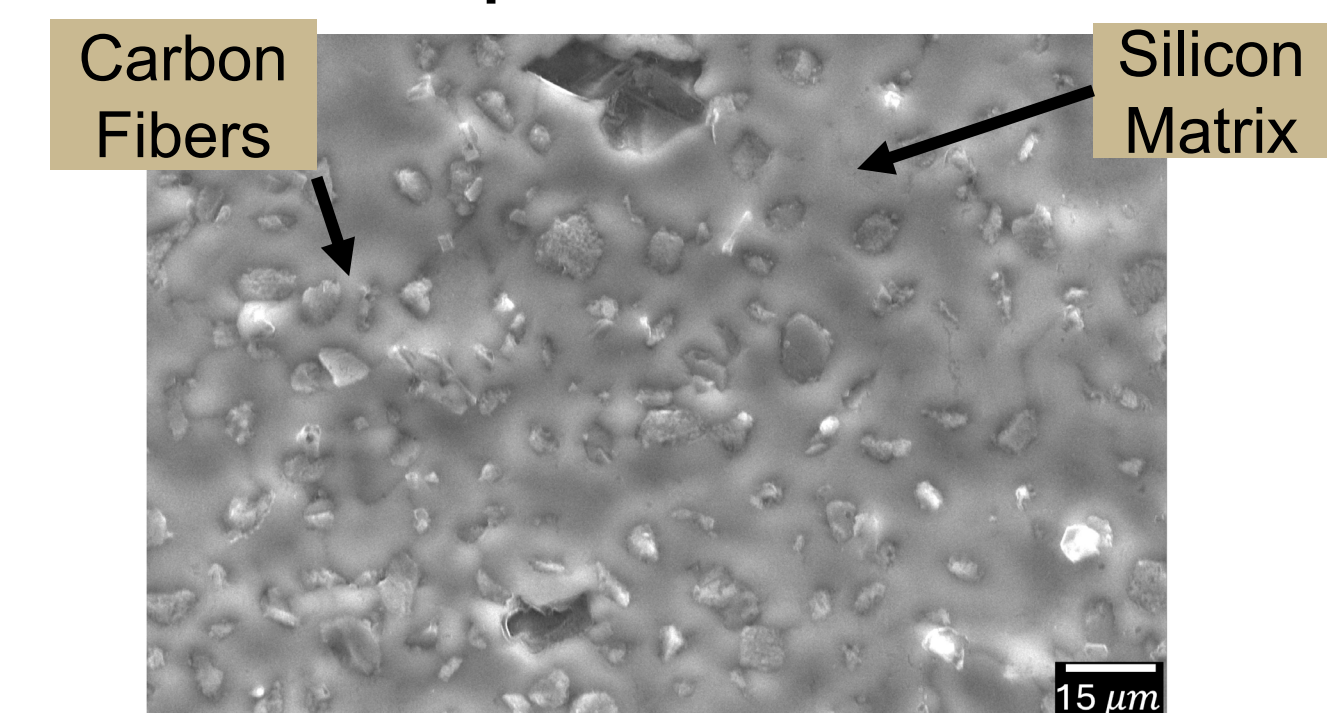


Fig. 4: Representative SEM image of CMC microstructure

Chemical Analysis

Energy Dispersive Spectroscopy (EDS) was used concurrent to SEM imaging to verify and measure sample chemistry and confirm the chemical integrity of tested samples before and after solvent exposure.

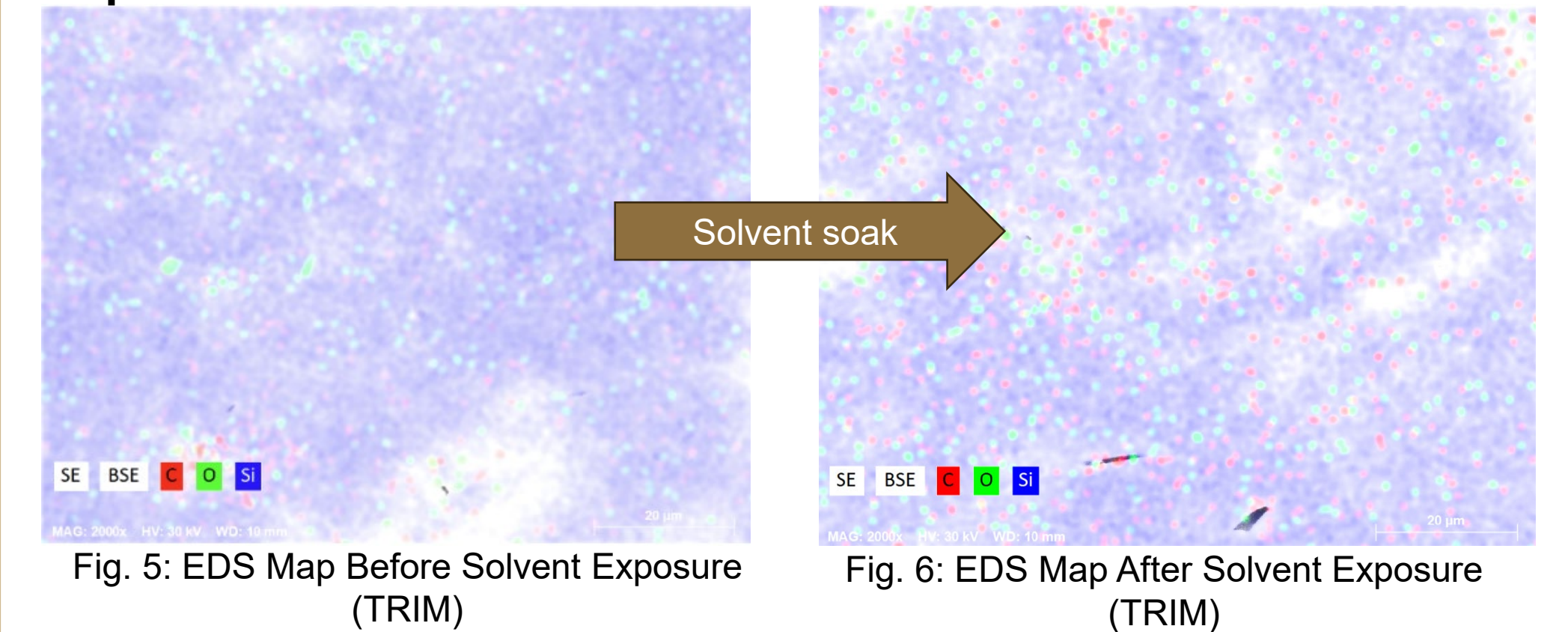


Fig. 5: EDS Map Before Solvent Exposure (TRIM)

Fig. 6: EDS Map After Solvent Exposure (TRIM)

Element ranges before (Norm. mass concentration):

| Sample | Si | C | O |
|-------------|--------------|-------------|-------------|
| Ethanol | 86.8 – 87.3 | 12.0 – 12.6 | 0.60 – 0.75 |
| Isopropanol | 71.5 – 72.8 | 15.3 – 15.9 | 0.85 – 0.98 |
| Novec 7100 | 86.9 – 87.4 | 11.8 – 12.3 | 0.70 – 0.85 |
| TRIM C-390 | 72.44 – 85.1 | 14.9 – 27.6 | 0.90 – 1.10 |

Element ranges after (Norm. mass concentration):

| Sample | Si | C | O |
|-------------|-------------|-------------|-------------|
| Ethanol | 86.8 – 87.3 | 12.0 – 12.6 | 0.60 – 0.75 |
| Isopropanol | 71.5 – 72.3 | 15.6 – 26.6 | 0.85 – 0.98 |
| Novec 7100 | 71.5 – 87.1 | 12.1 – 27.2 | 0.79 – 1.27 |
| TRIM C-390 | 76.4 – 90.0 | 8.93 – 24.1 | 0.95 – 1.06 |

Composition Summary

- SEM images similar for all samples
 - Slight variation in fiber distribution and amount
 - Fiber distribution and count varies by location
- EDS composition within range for all tested samples
 - Solvent exposure had no impact on detected elements
 - Point scans of fibers contained higher carbon content, and matrix contained higher silicon content

Conclusion & Recommendations

The solvent TRIM C-390 is the best alternative to ethanol for Archimedes density testing.

| Category | Advantages of TRIM C-390 |
|---------------|---|
| Cost | \$14/Liter |
| Flash Point | >105°C |
| Availability | Already used in the CMC machining process at RR |
| Precision | Not statistically different from ethanol |
| Effect on CMC | No detectable effect on the microstructure or elemental composition |

References & Acknowledgments

Acknowledgements

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References

- [1] Balbaa, M., Mekhiel, S., Elbestawi, M., & Mclsaac, J. (2020). On selective laser melting of Inconel 718: Densification, surface roughness, and residual stresses. *Materials & Design*, 193, 108818. <https://doi.org/10.1016/j.matdes.2020.108818>
- [2] OSHA. (2016). *Hazard Classification Guidance for Manufacturers, Importers, and Employers*. Hazard Communication. <https://www.osha.gov/sites/default/files/publications/O SHA3844.pdf>