Scalable, Infiltration-Free Ceramic Matrix Composite (SIF-CMC) manufacturing for Concentrated Solar Power: Investigation of particle-loaded preceramic resin

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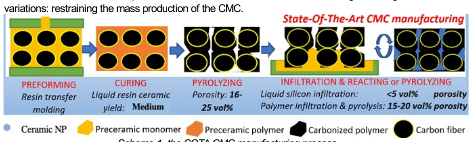
Abstract

Due to their unique combination of high temperature, corrosion resistance, and toughness, Ceramic Matrix Composites (CMC) are the optimal materials to be used in the next generation of Concentrated Solar Power (CSP), which will operate at working temperatures up to 800 °C. However, today, CMC is too costly to be used in CSP applications due to its complex manufacturing process based on repeated infiltration-pyrolysis cycles. To solve this issue, PARC is developing a scalable, infiltration-free method (SIF) to manufacture CMC, which can reduce the production cost, increase batch consistency, and leverage all of the intrinsic advantages of current CMCs. The key to the SIF method is using a flowable preceramic resin with high loading of functional components. The formulation impregnates the fiber during curing and leads to high-density, low-shrinkage parts upon pyrolysis. In this paper, we demonstrate a proof-of-concept of the SIF process and report the relationships between key process parameters in the manufacturing process: functional material loading in the resin vs. resin's flowability and morphology changes upon pyrolysis.

SIF-CMC Manufacturing

Although CMC has been invented for decades, it is still only used in price-insensitive applications, like turbine engines, nuclear power plants, or space shuttles. This is because the manufacturing process of the CMC is tedious, labor-intensive, and unreliable, preventing the CMC from mass production. In the state-of-the-art (SOTA) CMC manufacturing process, Scheme 1, the preceramic resin is firstly impregnated into the fiber tow and then cured and pyrolyzed. Because of the medium char yield of the resin, the pyrolyzed parts are full of pores, which is unsuitable for structural and load-bearing applications. To densify these porous parts, liquid silicon or preceramic resin is infiltrated into pores. This densification process normally takes more than 1 run and a long period of time. Besides its challenges in quality control, the infiltration process introduced dissimilarity within the matrix, negatively impacting the CMC's load-bearing and corrosion resistance. The infiltration process is labor-intensive, and time-consuming, causing batch-to-batch variations: restraining the mass production of the CMC.

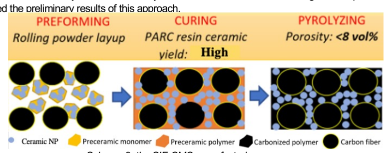


Scheme 1. the SOTA CMC manufacturing process

Scheme 2 illustrates the SIF-CMC manufacturing processes. Unlike the SOTA CMC, the SIF-CMC does not require the infiltration process. This process change relies on the extremely high char yield of the preceramic resin under development by this project.

The char yield of the preceramic resin used in the SOTA CMC manufacturing process is normally ~50 wt%. Even after decades of development, the best flowable preceramic resin has a char yield of ~65 wt%. For SIF-CMC manufacturing, resin with at least 80 wt% char yield is required which cannot be realized even by plain preceramic resins. Previous research has found that adding ceramic particles can increase the char yield of the preceramic resins but sacrifice the resin's flowability, which is essential for fiber impregnation. The dilemma of high char yield and high flowability of the particle-filed preceramic resin challenges SIF-CMC manufacturing.

In this project, PARC is developing a scalable particle functionalization and dispersion process to formulate resin with suitable char yield and flowability to realize SIF-CMC manufacturing. In this presentation, we discussed the preliminary results of this approach.



Scheme 2. the SIF-CMC manufacturing process

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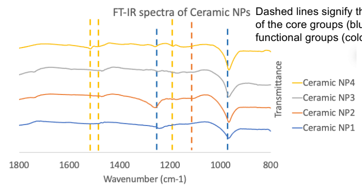
Particle functionalization

To increase the dispersibility of particles in a resin and to maintain the flowability of the mixture, we functionalized the particles through several established treatments to attach the ceramic NP with different functional groups. The as-received ceramic NPs and the ceramic NPs with different functionalization were named: Ceramic NP1, NP2, NP3, and NP4



Dashed lines signify the fingerprint of the core groups (blue) and functional groups (colored)

Figure 1. The FT-IR spectra of Ceramic NPs

FT-IR spectra and XPS element scans prove that Ceramic NPs were successfully functionalized with different functional groups through different treatments.

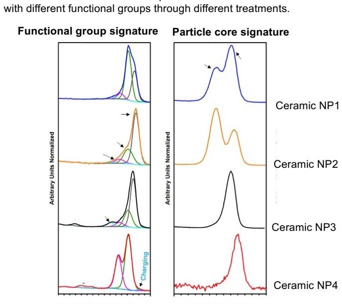


Figure 2. The XPS element scans of Ceramic NPs

To further analyze the performance of the Ceramic NPs, their char yield and dispersibility in two different solvents were characterized and summarized in Table 1.

There is no weight loss for both Ceramic NP1 and Ceramic NP2 when heated to 900 ºC in nitrogen. There is a 12 wt% weight loss for Ceramic NP3 and an 18 wt% weight loss for Ceramic NP4 due to the decomposition of the functional groups on both particles. Both Ceramic NP3 and NP4 were proved to be heavily functionalized.

The particle size measured by DLS indicates the particle's dispersibility in the solvent, against the aggregation. The significant size dropping of the Ceramic NP 3 proves its high dispersibility in both solvents. The large aggregation of the Ceramic NP4 is due to the poor solubility of its functional groups in the solvent 1.

Table 1. The char yield and dispersed particle size of SiC NPs with different treatments

Characterizations		Ceramic NP1	Ceramic NP2	Ceramic NP3	Ceramic NP4
Char yield (%)		100	100	88	82
Particle size (nm)	Solvent 1	430	308	200	396 & 1613
	Solvent 2	367	356	258	414

Particle-filled Formulation: Flowability

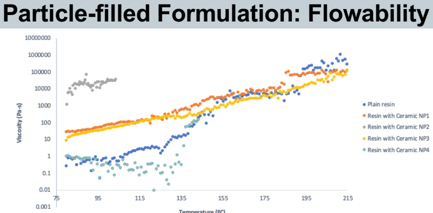


Figure 3. Viscosity changing during heating of resin with different Ceramic NPs

Maintaining the flowability of resin while loading more particles is the key to the reach the resin requirement of SIF-CMC manufacturing. To realize this concept, the particle is expected to be fully covered with a functional group to keep it highly dispersible in the resin.

Figure 3 presents the viscosity changes when adding Ceramic NPs: Ceramic NP1 and NP2 do not interact with the resin; Ceramic NP3 interacts strongly or even bonded with the resin molecule; Ceramic NP4 is hydrodynamically dispersed in the resin. The resin with Ceramic NP4 shows the targeting rheological behavior that is required for SIF-CMC manufacturing.

Particle-filled Formulation: Char yield

Table 2. The char yield of the resin with a certain loading of different Ceramic NPs

Samples		Plain resin	Resin with Ceramic NP1	Resin with Ceramic NP2	Resin with Ceramic NP3	Resin with Ceramic NP4
Char yield (%)	Measured	**65**	**65**	**70**	**70**	**66**
	Calculated	58-65	71	71	69	68

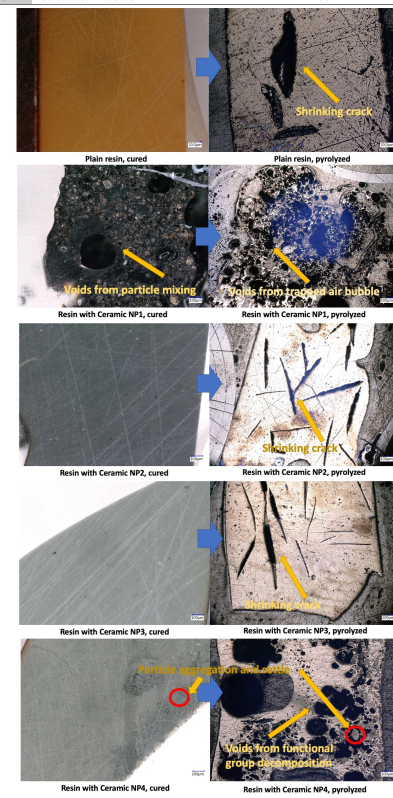


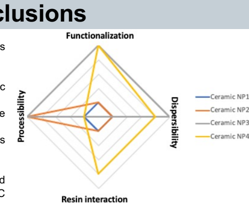
Figure 4. The microscope image of resins with different Ceramic NPs

Conclusions

In this presentation, we have proved that it is feasible:

- To heavily functionalize ceramic particles;
- To tune the dispersibility of ceramic particles by functionalization;
- To alter the particle interaction with the resin by changing the functional groups;
- To control the behavior during the process by optimizing the functional groups.

Overall, it is feasible to prepare flowable and high char yield resin to realize SIF-CMC manufacturing.



PARC is developing a novel functionalization procedure to realize SIFCMC manufacturing by checking all the boxes. Meanwhile, PARC is excited to extend this flowable, high char yield, and minor shrinkage resin in other applications:

- Ceramic additive manufacturing
- Ultra-high temperature applications
- Complex environment applications
- Bonding of similar or dissimilar materials

We are looking forward to talking with you.