

Temasek Defence Systems Institute

Determining Heat Treatment Methods for Additive Manufactured Silicon Nitride-BNNT Composites

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Background:

Silicon nitride (Si_3N_4) ceramics are highly valued for their exceptional mechanical and thermal properties, making them ideal for high-performance applications. However, challenges like brittleness and high-temperature sintering requirements hinder their wider use. Incorporating boron nitride nanotubes (BNNT) into Si_3N_4 via additive manufacturing and molten salt synthesis (MSS) offers potential improvements. This study explores techniques to enhance Si_3N_4 performance while enabling low-temperature sintering.

Objective:

The primary goal is to develop heat treatment methods that enable low-temperature sintering of Si₃N₄ composites while preserving the structural integrity of BNNT. The aim is to improve mechanical and thermal properties and explore the effects of different sintering parameters.

Methodology:

Fabrication. Si₃N₄ composites with 2% & 4% BNNT were additively manufactured using LCM.

<u>Post-Processing</u>. Debinding and sintering techniques were applied with cold isostatic pressing (CIP) and molten salt synthesis (MSS) using KBr salt.

Sintering Environment. Samples were sintered in both argon and air at various temperatures and dwell times. The protective role of KBr was emphasized to prevent BNNT oxidation.

<u>Characterization</u>. The structural and crystallographic properties were evaluated using scanning electron microscopy (SEM) and X-ray diffraction (XRD).

Key Findings:

BNNT Preservation. MSS successfully preserved BNNT integrity even at temperatures up to 1100°C in both argon and air atmospheres.

<u>Incomplete Sintering</u>. Despite CIP, the chosen sintering parameters were insufficient, leading to sample fragility and disintegration during salt removal.

<u>Phase Composition</u>. XRD confirmed the presence of α -Si₃N₄ and β -Si₃N₄, but BNNT detection was hindered by low concentration and peak overlap.

<u>Surface Issues</u>. KBr crystallization on the sample surface and the presence of sharp corners contributed to structural weaknesses.

Way ahead:

- 1. Optimize sintering parameters to achieve complete densification.
- 2. Implement advanced characterization techniques (e.g., Raman spectroscopy) to better identify BNNT.
- 3. Address geometric design considerations to minimize stress concentration and fragility.
- 4. Explore additional processing methods to improve the uniform dispersion of BNNT

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