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Ultrahigh Temperature Materials for Hypersonic Systems Readiness

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NPS NRP Executive Summary

Ultrahigh Temperature Materials for Hypersonic Systems Readiness

Period of Performance: 10/25/2021 – 09/30/2022

Report Date: 07/29/2022 | Project Number: NPS-22-N263-B

Naval Postgraduate School, Graduate School of Engineering and Applied Sciences (GSEAS)



NAVAL RESEARCH PROGRAM
NAVAL POSTGRADUATE SCHOOL
MONTEREY, CALIFORNIA

ULTRAHIGH TEMPERATURE MATERIALS FOR HYPERSONIC SYSTEMS READINESS

EXECUTIVE SUMMARY

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Project Summary

To achieve the strategic and operational goals required for the survivability of aircraft traveling at hypersonic speeds, it is imperative to study which materials could serve as thermal protection, capable of withstanding extreme heat and oxidative and ablative conditions. The study conducted aimed to support the hypersonic RDT&E efforts by validating the use of diverse fabrication routes as an operational alternative to generate the ultrahigh temperature ceramics (UHTC) zirconium diboride (ZrB_2) and silicon carbide (SiC). The work included the integration of the UHTC as the surface layer of composites containing graphitic fibers and carbonaceous matrices. A technical assessment was performed to determine if the newly achieved composite materials had the chemical makeup and microstructural characteristics desired employing scanning electron microscopy and X-ray diffraction techniques. The study evaluated the potential of the composite to withstand oxidative and ablative conditions encountered by systems used in hypersonic flight, simulated by the use of an oxygen acetylene torch.

The carbothermal reduction of zirconium oxide employing boron and carbon black as precursors, at temperatures in excess of 1450 °C, rendered high-purity ZrB_2 . The use of a one-component liquid precursor (SMP-10) successfully produced diverse polymorphs of silicon carbide (SiC). ZrB_2 and its mixture with graphite did not present significant morphological changes after exposure to ultrahigh temperatures; however, ZrO_2 peaks of reduced intensity were detected in their XRD patterns, with those containing graphite being smaller than the bare ZrB_2 . SiC from SMP-10 produced a glassy sintered byproduct, while commercial SiC did not suffer any changes. Zirconium diboride exhibited the potential to withstand extreme environments, as initially hypothesized. Future efforts should include the evaluation of mechanical properties of the sintered composites.

Keywords: *ultrahigh temperature ceramics, UHTC, hypersonic, transition metal borides*

Background

When hypersonic speeds (3000 mph and above) are reached, the friction between air and the high-speed object generates extremely high temperature regions (>2000°C), high mechanical stresses and a decrease in the materials' tolerance to damage. Those severe conditions call for the use of new, highly engineered materials that can withstand not only high temperatures, but also environments that tend to oxidize the components of the system passing through at those speeds, which reduces their strength and causes erosion and material degradation. Ceramic tiles have traditionally been used for aircraft thermal protection; however, their brittleness, low damage resistance and high cost have limited their application. Metallic thermal protection structures, super alloys, and intermetallic compounds have also been considered; however, their melting temperatures and susceptibility to oxidation are drawbacks. Therefore, the search for ultrahigh temperature resistant materials is still a fertile area of research. Recent advances in the field suggest that ceramic materials, such as transition metal borides integrated with carbonaceous matrices and silicon carbides, have the potential to withstand ultrahigh temperatures and conditions such as those encountered during hypersonic flight.

Findings and Conclusions

The carbothermal reduction of zirconium oxide employing boron and carbon black as precursors, at temperatures in excess of 1450 °C, rendered a higher-purity ZrB_2 than the specimens generated by atmospheric plasma fabrication approaches, and based on the data collected by this study, is the



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most indicated method to produce zirconium boride. The use of a one-component liquid precursor (SMP-10) successfully produced diverse polymorphs of silicon carbide (SiC). The precursor decomposition takes place in three stages and can be readily mixed with ZrB_2 without promoting its oxidation once the SMP-10 samples have suffered the initial decomposition step at 160°C.

ZrB_2 and its mixture with graphite did not present significant morphological changes after exposure to ultrahigh temperatures; however, ZrO_2 peaks of reduced intensity were detected in their XRD patterns, with those containing graphite being smaller than the bare ZrB_2 . Zirconium diboride, thus, has the potential to withstand extreme environments, as initially hypothesized.

SiC from SMP-10 produced a glassy sintered byproduct, while commercial SiC did not suffer any changes. Carbon nanotube composite mixtures presented the largest levels of oxidation due to the presence of the iron employed as catalyst during their production, which turned into FeO and Fe_2O_3 , depending on the nanotube location in the mixture, internal or superficial, respectively.

Recommendations for Further Research

Since there are no standard methods to test ultrahigh temperature ceramics, if the use of an oxygen acetylene torch is to be continued, an in-situ temperature monitoring system or probe should be employed to accurately determine treatment conditions and their effects. High power lasers should be also considered.

Further research should include mechanical properties assessment of sintered monoliths of the diverse zirconium boride, silicon carbide, and carbonaceous combinations.

Acronyms

UHTC ultrahigh temperature ceramics

