Self-healing Composites for Aggressive Environments

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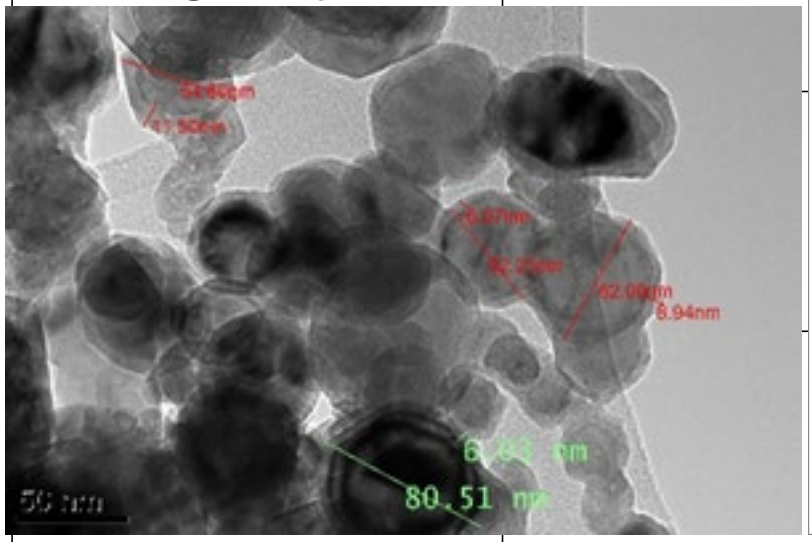
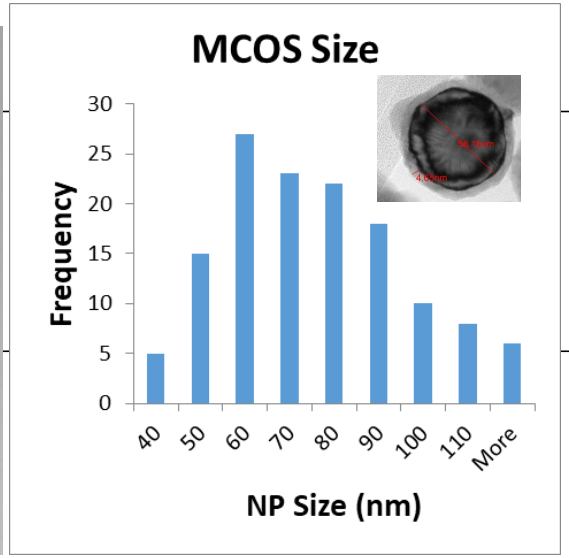
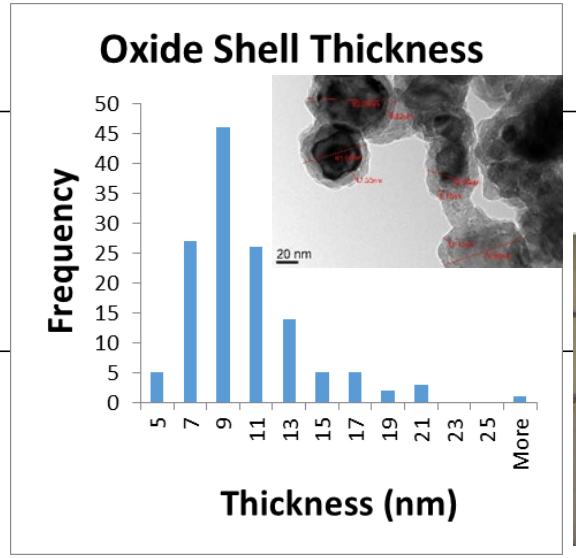
- Grant #: IGEM25-005
- Reporting Period: 7/1/24 –12/31/24

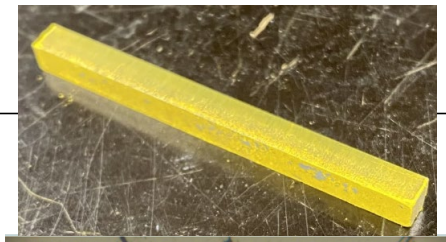
Summary of progress towards proposed milestones

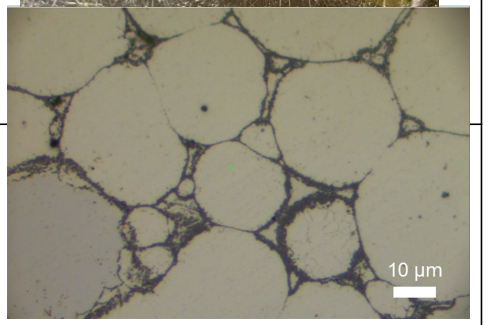
Milestone 1: Preparation of FeCr and Fe-B alloy powder	•The FeCr and Fe-B alloy powders were prepared using mechanical alloying by ball milling. In addition, NiCrMo alloy powder representing Inconel 625 was also prepared. •The alloy powders are used as the matrix for the composite preparation. Therefore, there is no specific metric. •This milestone is complete without any issues.
Milestone 2: Preparation of Fe-Fe_xO_y core-shell nanocapsules with desired dimensions	•Iron nanoparticles (diameter 20 – 100 nm) were oxidized at different temperatures (120 – 300 °C)and time intervals (0 – 60 minutes) in a controlled atmosphere. Transmission electron microscopy was carried out to analyze the thickness of the oxide shell and metal core. •The metal core and oxide shell (MCOS) nanocapsules preparation step was optimized by oxidizing the iron nanoparticles at 145 °C for 20 minutes. This process can produce large-scale MCOS particles (500 grams per batch). • MCOS particles of Sn-SnO_2, Zn-ZnO, Ni-NiO, and Bi-Bi_2O_3 are being prepared for ceramic-based composite materials that could exhibit self-healing behaviors. •This milestone is also complete without any major issues.
Preparation of Self-Healing Composites	•The composite samples were prepared by mixing 88 - 90 wt% of Fe alloy (matrix), 5 – 10% MCOS, and 2 – 5 wt% of cellulose. The mixture was compacted using a die set and hydraulic press with a maximum load of 10 ton to 50 mm X 4 mm X 4 mm square prism samples. •The IN 625-MCOS samples were sintered at 900 °C for 1 h. Bi_2O_3 – MCOS (Bi core-oxide shell) composite was sintered at 450 °C. •To demonstrate self healing behavior, 3-point bend testing will be performed at high temperatures. •3-point bend test fixtures are designed. Manufacturing of these fixtures is in progress.











Summary of expenditures and budget performance

- ***Key Insights***
- Spending is ON TRACK with the proposal.
- Major expenditures included:
  - Support of one M.S. student, one Ph.D. student, and one undergraduate intern.
  - Installing a hydraulic press for powder compaction
  - Purchase of expensive metal nano and micron-sized powders.
  - Characterization of MCOS
- Challenges and Changes
- Currently, CAES facilities are not available for material characterization and sample preparation.
- In-house designed loading fixtures will be fabricated and used for mechanical testing.
- In addition to metal – MCOS composites, ceramic-MCOS composites will be prepared and tested for self-healing behavior.

	Budgeted, $	Spent, $	Balance, $
Salary	115,410.40	46317.71	69,092.69 (60%)
Fringe	8,875.00	4,163.16	4,711.84 (53%)
UG Student intern support	35,190.60	5700	0
Travel, Materials, supplies and analysis		12925.08	16565.52 (47%)
Tuition	38,124.00	12,056.00	26,068.00 (68.4%)
Total	197,600.00	81161.95	116,438.05 (58.9%)

Projection of work in next reporting period

- Preparation of 3-point bend samples of other metal matrix-MCOS composites, as well as ceramic-MCOS composites, using compaction and sintering methods. The sintering temperature will be optimized to achieve 100% theoretical densities.
- Completion of manufacturing in-house designed loading fixtures to carry out the 3-point bend testing at high temperatures.
- 3-point bend testing of the samples at different temperatures to demonstrate self-healing of the metal-MCOS and ceramic-MCOS composite materials
- Microstructural characterization of the MCOS composite materials before and after 3-point bend testing using scanning electron, transmission electron, and Raman microscopy.
- The students will present the results at the American Nuclear Society (ANS) Student Conference 2025, which will be held in Albuquerque, NM, from April 3 to 5, 2025.
- Two manuscripts will be prepared and submitted for publication in peer-reviewed journals.