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Lead Institution

University of South Carolina

Science Based Nano-Structure Design and Synthesis of Heterogeneous
Functional Materials for Energy Systems

(HeteroFoaM)

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Final Report

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Executive Summary:

The electrochemical science and engineering that makes many energy conversion and storage technologies work rests on our knowledge and understanding of heterogeneous materials and material systems. The function and functionality of those systems share many common features across a wide range of technologies including fuel cells, batteries, capacitors, and membranes. The science that controls that functionality for these complex material systems is typically summoned in fragments to design a specific device. The HeteroFoaM Center was part of an ongoing attempt to create a codified multiphysics science-based approach to that general subject, across multiple scales in space and time, for heterogeneous functional materials, or "HeteroFoaM" as we call it. The technical scope of the Center was driven primarily by conversion of chemical energy to electricity, membranes for selective transport, and charge storage devices. The principal motivation for this approach is to establish the science that controls emergent properties in heterogeneous functional materials as a foundation for design of functional material systems with performance not bounded by constituent properties. The HeteroFoaM Center brought together scientists from Universities (Princeton, Santa Barbara, U. Utah, U. South Carolina, Clemson, U. Connecticut, RIT) and National Laboratories (Argonne, SLAC, Brookhaven) as an interdisciplinary team to define and codify HeteroFoaM as a material class, construct an integrated science that describes the equilibrium and non-equilibrium response of those materials to combined mechanical, chemical, and electrical applied fields, used that science to explain how the properties of HeteroFoaMs depend on the details of how they are made, and used that foundation to create new HeteroFoaM material systems with emergent properties that greatly exceed the capabilities of the constituents. In addition to more than 200 archival publications that advanced the science and methodology of the subject of material systems, the HeteroFoaM team achieved "world best" performance for capacitors and solid oxide fuel cells, and created a new foundation for the design of heterogeneous ceramic membranes for the processing and nuclear industry. At the general level, HeteroFoaM is a new atoms-to-systems paradigm that is intended to bring heterogeneous functional materials (for which the system is rarely simply the sum of the parts) into the modern age of computational materials to achieve the savings of time and cost associated with trial and error Edisonian design.

DOE Energy Frontiers Research Center for Heterogeneous Functional Materials; the “HeteroFoaM Center”

Synopsis of five year accomplishments:

Devices that convert and store energy are generally made from heterogeneous constituent materials that act and interact to selectively conduct, transport, and separate mass, heat, and charge. Controlling these actions and interactions enables the technical breakthroughs that have made fuel cells, batteries, and solid state membranes, for example, essential parts of our society. In the biological sense, these materials are ‘vascular’ rather than primitive ‘cellular’ materials, in which the arrangements and configurations of the constituents (including their void phases) play essential and definitive roles in their functional capabilities.

In 2009 a group of investigators, with lifetime investments of effort in the understanding of heterogeneous materials, recognized that the design of such material systems is not an optimization problem as such. Local interactions of the constituents create “emergent” properties and responses that are not part of the formal set of constituent characteristics, in much the same sense that society and culture is created by the group interactions of the people involved. **The design of emergent properties is an open question in all formal science**, but for energy materials the lack of this foundation science relegates development tasks to Edisonian trial and error, with anecdotal success and frequent costly failures. That group defined, for the first time, multi-scale heterogeneous functional materials with functional disordered and void phase regions as “HeteroFoaM,” and formed the first multidisciplinary research team to define and codify the foundation science of that material class. **The primary goal** of the HeteroFoaM Center was, and is, to create and establish the multi-scale fundamental knowledge and related methodology required for the rational and systematic multiphysics design of heterogeneous functional materials and their interfaces and surfaces for applications in energy transformation and storage. **The scope** of the HeteroFoaM center was focused on the discovery and development of the control science of key phenomena across multiple length scales **that create functionality** in heterogeneous materials and their structured interfaces, boundaries, and surfaces for applications in energy technologies.

The HeteroFoaM Center defined a critical path and established an essential foundation for progress in the field of heterogeneous functional materials. Perhaps the single most important element of progress was the establishment of the capability to design, characterize, and model heterogeneous functional materials at the conformal level, i.e., for a limited set of material systems, the HeteroFoaM team defined how to control the order / disorder at the atomic level, the surfaces, and the interfaces for selected constituent morphologies, and to use multiphysical models to explain the remarkable property variations resulting from that control science for several heterogeneous material systems. For those cases we defined “meso-structures” (at various scales) where the interactive physics of constituent phases acted to create emergent properties, e.g., strongly emergent mixed conductor behavior and ionic transport.

The general approach used by this EFRC is shown in Fig. 1. The HeteroFoaM Center created the genre of Heterogeneous Functional Materials with functional surfaces and interfaces (including void phases) called **HeteroFoaM as a science platform** to enable rational analysis and design of functional material systems by focusing on the meso-interactions that drive emergent response. The team firmly established this approach with over 180 archival

publications (see “Publications” section), 7 patent applications, and over 100 invited lectures in 15 countries on this topic, enabled by building a remarkably effective and uniquely coherent research team. Indeed, **our team was our principal strength**; this problem eluded solution earlier because such a team was not available.

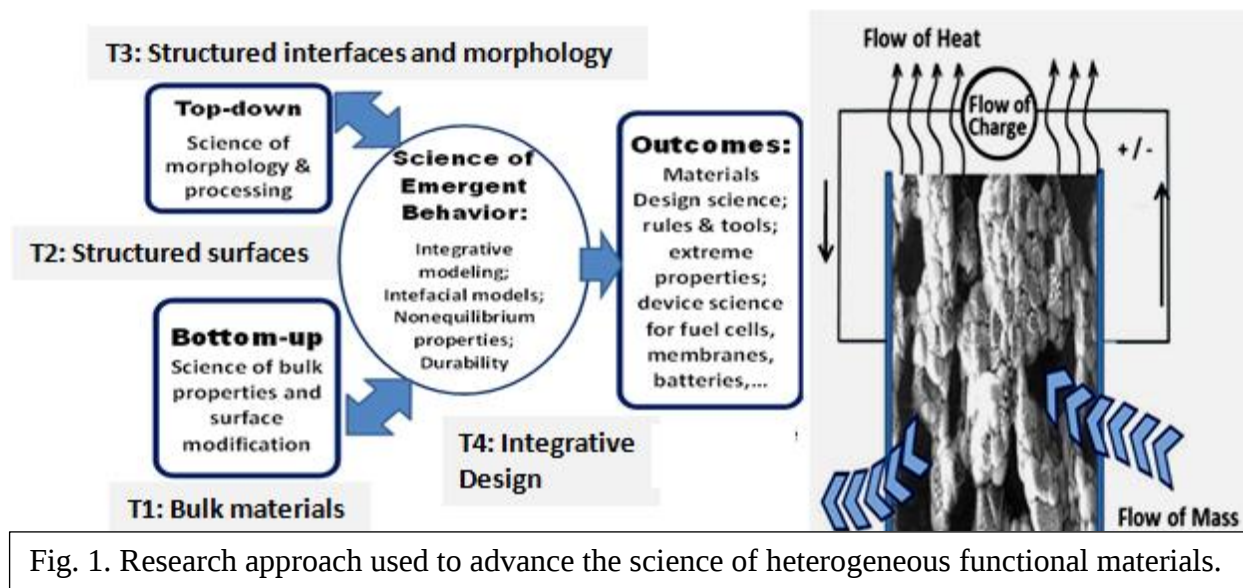


Fig. 1. Research approach used to advance the science of heterogeneous functional materials.

Selected Highlights:

A few highlights and measures of the success of the HeteroFoaM Center will be summarized below. Readers are referred to the "List of HeteroFoaM Publications" provided at the end of this document for further details.

General outcomes:

- Teams in our HeteroFoaM Center demonstrated the HeteroFoaM meso-design concept with successful atoms-to-systems designs at the bulk level (e.g., redox-stable electrodes for SOFC/SOECs and first-ever combined dielectric/redox energy storage), the membrane level (e.g., new MIEC membranes for nuclear fuel waste separation), and surface level (e.g., functionalized electrodes enabling direct hydrocarbon fuel oxidation).
- The HeteroFoaM concept is widely recognized and acclaimed, as evidenced by the fact that our investigators have been **invited** to present our work in 16 countries, to organize and chair several focused HeteroFoaM technical sessions around the world, and to serve on the Editorial Board of the new journal of Multifunctional Composites. The HeteroFoaM Center has been **invited** by the Materials Research Society, the American Physical Society, and the Science Cinema (<http://www.osti.gov/sciencecinema/>) to prepare 5 min. videos specifically highlighting our Center activities as they relate to their societies for featured display at their annual meetings and on YouTube (MRS video circulation ~20,000).
- The Director of the HeteroFoaM Center, Prof. Ken Reifsnider, now Presidential Distinguished Professor and Director of the Institute for Predictive Performance Methodologies at the University of Texas Arlington, will receive a lifetime achievement award from the International Conference on Computational and Experimental Engineering

Sciences for his leadership in multi-scale, multiphysics modeling of multifunctional heterogeneous materials in June of 2017 in Madrid.

The HeteroFoaM EFRC Center structure significantly advanced a small number of **Center-wide canonical themes** that exemplify the paradigm. These basic themes are a foundation for the future structure of science that will build on this work. They leverage some of the fundamental additions to the science of HeteroFoaM that have been achieved by the Center:

- **Non-equilibrium processes – how general potentials drive fluxes and displacements** in heterogeneous functional materials: This theme was one of the strongest collective strengths, and cross-cuts essentially all Center goals. The work of the Center greatly impacted this field, from the mixed conductor work of Chen, zur Loye, Carter, and Brinkman, to the “unbounded local potential” work of Virkar, to the surface characterization breakthroughs of M. Liu, to the surface atomic level analysis (including kinetics) of Heyden, Carter and others, the Center provided world leadership in this area and redefined parts of this field. This is a strong marriage of experiment and analysis; without the new in-situ and in-operando methods developed by this team, the new analysis concepts and methods (from atomic to micro to meso) could not have been validated or correctly formed. Embracing, and providing foundation science for non-equilibrium processes was a Center wide focus resulting in significant advances.
- **3-D Coupling – the reality of morphology effects:** Our Center Team collaborated to provide international leadership in discovering and confirming that morphology effects must be defined by the local coupling of processes in **three** dimensions. A very strong multi-investigator/institutional collaboration in 3-D nano-tomography, rendering, and conformal analysis enabled our team to build the first-ever robust, multi-scale, **conformal** multiphysics analysis of HeteroFoaM type materials. This seriously impacted the general understanding of transport, kinetics, local electrochemistry, and even stability in these materials. Moreover, it changed the design paradigm for conversion, storage, and separation membrane devices, and defined new micro-synthesis paths for such material systems.
- **Emergence – the “systems” part of functional material systems:** HeteroFoaM systems for energy conversion and storage are often open systems, and often operate as non-equilibrium and non-conservative domains. Local functionalities often lead to global property emergence. The HeteroFoaM team discovered that in some cases this emergence can be accelerating (as in material degradation) or decelerating (as in loss of material functionality). The functionality of Material Systems was a major area of emphasis.
- Some specific examples of our progress towards the five year goals of the HeteroFoaM Center follow:

5 Year Goals	Progress
Goal #1: Understand charge transport and storage in heterogeneous functional	▪ Used nano-porous HeteroFoaM architecture to achieve unprecedented super-capacitance, >2500 F/g, demonstrating new storage concept not dependent on redox reactions of cations (NanoLtrs., Jan. 2012, doi: 10.1021/nl300984y) ▪ Demonstrated that a radically different microstructure design for proton conducting anodes with low porosity achieves high performance at 750C (IJHydrogen Energy, 2012, doi: 10.1016/j.ijhydene.2012.09.02) ▪ Demonstrated mixed ionic and electronic conductivity in perovskite

materials (HeteroFoam)	oxides using a p-and n-type co-doping strategy (Solid State Ionics, 228 , 37-45, (2012) doi: 10.1016/j.ssi.2012.09.013)
Goal #2: Establish science of functional surfaces and interfaces	- Created a “direct octane” fuel cell that enables power generation from hydrocarbon fuels as a foundation for portable power in transportation (<i>Nano Energy</i>, (2012). [10.1016/j.nanoen.2012.02.006]) - Designed and demonstrated a surface modification for standard electrode materials that enables direct hydrocarbon fuel oxidation in SOFCs (<i>Advanced Materials</i>, 24, 1439-1443 (2012) doi:10.1002/adma.201104852) - Developed an in-operando XAS testing apparatus capable of controlling temperature, gas environment, and electrical polarization; used synchrotron x-rays to directly observe changes in the electronic state and neighboring atomic structure of Co and Fe cations in an LSCF thin film electrode as a function of temperature, electrical polarization, gas atmosphere, and gas contaminants, such as CO₂ and high amounts of water vapor. (H. Ding, A. V. Virkar, M. Liu, and F. Liu, <i>Phys. Chem. Chem. Phys.</i>, 15, 489, 2013, [DOI: 10.1039/c2cp43148c])
Goal #3: Create synthesis methods to control morphology and functionality	- Developed and demonstrated new electrode morphologies for power generation and electrolysis using a novel freeze casting method. (RSC Advances, 2, 12118-12121 (2012).doi: 10.1039/c2ra21921b) - Used nano-structured synthesis and spark sintering methods to alter the microstructure of MIECs resulting in an order of magnitude increase in electronic conductivity and enhanced separation membrane performance; developed method for controlled functionality design for mixed conductor membranes for nuclear waste (and other oxide) separation, and hydrogen separation methodologies (JES, 160, F470-F481 2013)doi: 10.1149/2.012306jes) - Investigated stability of nano-structured materials in energy systems. The study showed that it is possible to quantitatively determine degradation mechanisms and kinetics of nano-structured materials via novel electrochemical testing. The approach leads to the design of stable catalysts for PEM fuel cells (J. Power Sources, 234 82-90 2013) doi: http://dx.doi.org/10.1016/j.jpowsour.2013.01.115)
Goal #4: Establish a multiphysics, multi-scale methodology for "HeteroFoam by Design"	- Achieved first 3-D multiphysics models set on resolved microstructure of anodes to follow functional changes during operation (ACTA Mater.60, 2012, doi: 10.1016/j.actamat.2012.02.041) - Established new paradigm for design of mixed-electronic/ionic conducting electrodes using new atomic modeling approach to structure-property predictive methodology, validated for SFMO (J.Am.Chem.Soc. 134,2012, doi: 10.1021/ja300831k) - Defined the “HeteroFoam Element” as a meso-platform concept that enables multi-scale multifunctional analysis by relating atomic and nano analysis to meso and global functionality of heterogeneous functional materials (JES 160, F470-F481 (2013) doi:10.1149/2.012306jes) - Created an atomic scale design tool for new SOFC electrodes and their

	surfaces from first principles, and identified basic thermodynamic driving forces for Sr surface segregation and corresponding suppression schemes in LSCF. (Ammal & Heyden, <i>J. Physical Chemistry Letters</i> , 3 (2012); Ding, Virkar, Liu & Liu, <i>Phys. Chem. Chem. Phys.</i> 15 , 489 (2013).
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Summary:

“The aim of exact science is to reduce the problems of nature to the determination of quantities by operations with numbers.”¹ The HeteroFoaM Center founded and advanced a science of material systems that is uniquely capable of creating local rules for heterogeneous constituent interactions with each other and with multi-physical applied fields, making it possible to predict emergent global material properties across multiple scales, from “atoms to systems.” This new conformal science replaces mixture concepts which ignore local interactions and are inherently limited to phenomenological empiricism. The result is a multi-scale, multiphysics methodology for heterogeneous functional material systems that enables the design of constituents, their interfaces, and their morphologies to create disruptive advances in functional materials for electrochemical electrodes and electrolytes, mixed conductors, and dielectric material systems for energy conversion and storage. The HeteroFoaM center created and established the foundations of an “atoms to systems” approach, and demonstrated the value of that approach by achieving breakthroughs in fuel cells, capacitors, and ceramic separation membranes for oxygen transport.

Reference:

1. James Clark Maxwell, 1831-1879

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