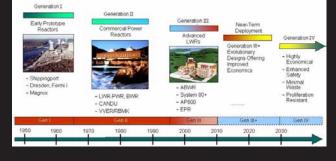
Microsystems for Harsh Environments

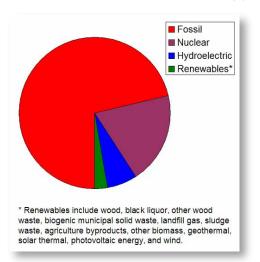
Objective

Our goal is to develop and demonstrate a MEMS-based methodology for evaluating time dependent mechanical properties and reliability of materials that undergo exposure to extreme and harsh environments (e.g., temperature, radiation, corrosive chemistries, etc.). As a first application, we are developing this method to evaluate the effects of long term, high flux radiation exposure on materials used in nuclear power plant construction. Our MEMS-based test platforms will be used to validate the continued use of current facilities and accelerate development of next generation nuclear plants (NGNP). Generation IV: Nuclear Energy Systems Deployable no later than 2030 and offering significant advances in sustainability, safety and reliability, and economics



Impact and Customers

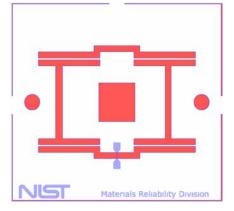
• Currently, the country's 100 operating nuclear power plants supply more than 20 % of our electricity, accounting for over 70 % of our carbon dioxide free electricity production.



- Nuclear power plants emit virtually no carbon dioxide during operation. Even considering "full life cycle emissions," including the mining and transportation of fuel, plant construction, and waste considerations, nuclear carbon dioxide discharges are comparable to wind and hydropower, and less than solar power.
- The average U.S. nuclear power plant has been in operation for over 24 years, already half of the original operational license period.
- To maintain our current capacity through lifetime extension, or to increase capacity through NGNP, a rapid assessment of materials properties under long term irradiation exposure is necessary.
- Small specimen test technology will accelerate materials development and evaluation through the simultaneous testing of large arrays of microspecimens, systematically varied in composition and/or heat treatments, including time dependent effects.

Approach

Classically, material property evaluation is performed with macroscopic specimens and test methods. In contrast, we are developing new MEMS-based test methods to evaluate the properties of bulk materials in environments where macroscopic methods are not applicable, extremely difficult, or exceptionally costly. The properties of the small scale structures will be linked to those of bulk scale structures through comparative testing, coupled with image-based finite element models. Currently, we are developing techniques using focused ion beam (FIB) milling to section microsized specimens from structural materials previously irradiated in test reactors, accelerators, or working power reactors, for insertion into prefabricated on-chip load frames. Ultimately, we plan to fabricate an array of test structures with on-chip control, actuation, and sensing, allowing for long term, in situ, harsh environment testing in high irradiation environments.



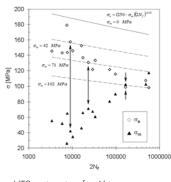


Accomplishments

This project was initiated in FY09 and combines expertise within the Materials Reliability Division in the areas of macroscale and microscale property evaluation.

Microscale Property Evaluation

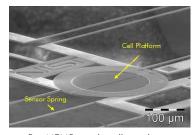
Accessing the as-fabricated properties of materials used in integrated circuits and other related devices is critical to their continued scaling. For the past two decades, the Materials Reliability Division has been developing new methods to test materials reliability at increasingly small dimensions. Initially, we focused on the development of a microscale tensile test system to measure elastic and plastic properties of free standing, patterned thin films. As device complexity increased, however, new methods were needed to address buried layers and interconnects. Because interconnect properties are determined by both the material and the influence of the immediate interconnect environment (which could not be measured directly with the microtensile test), we developed an electrically based method to measure the properties of as-fabricated structures. Through the use of high density current cycling, we have evaluated properties such as ultimate tensile strength and cyclic lifetime of these materials.



UTS estimation for Al interconnects

Fabricated On-Chip Test Methods

In addition to measuring the properties of advanced electronic materials, we also have expertise in the measurement of materials such as living cells. We recently demonstrated a unique bio-MEMS device that stretches a single live adherent cell and measures its corresponding mechanical response. Due to the small size of living cells, this unique approach relied on the use of microfabrication techniques to prepare a protein-coated miniaturized load frame, onto which a cell could be grown, observed through a transparent window, and measured using calibrated on-chip load spring and displacement sensors. This technique provided a proof of principle demonstration of our ability to design and fabricate on-chip materials measurement devices.



Bio-MEMS, single cell test device

Reliability of Structural Materials

In addition to small scale mechanics, the Division has a 50+ year history in measuring fracture and deformation in large scale structural materials. Most recently, we have been applying this expertise to pipeline steels, and adapting the Crack Tip Opening Angle (CTOA) method for characterizing fully plastic fracture. Our research focuses on improving the optical imaging technique for determining the crack tip opening angle, standardizing the measurement method to reduce uncertainty in the data. This method has been successfully employed to measure the effects of aging and operational conditions on the fatigue crack growth rates of steels, spanning five different strengths and more than 70 years of operation.

<u>Evaluation of Materials Properties for Operation</u> <u>in Harsh Environments</u>

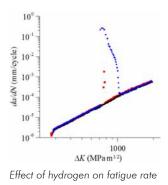
Our research into pipeline safety does not end with aging effects. On the forefront of pipeline research is the issue of materials compatibility



High speed CTOA Load Frame

for distribution of alternative fuels. For example, the future "hydrogen economy" will depend on efficient transport of hydrogen, likely using the existing network of oil and gas pipelines; thus, hydrogen degradation of the pipelines is likely to occur, and quantifying it will be critical.

We conducted tests to evaluate the effects of hydrogen on the fatigue life of pipeline steels. Specimens were prepared with and without surface tinning, and then electrochemically hydrogen charged to determine the effects of surface preparation on hydrogen dissolution. Our results show a two-order-of-magnitude increase in fatigue rate when specimens were hydrogen charged. Data on hydrogen effects is limited, indicative of the challenges associated with performing such tests, and illustrating the need for alternative approaches to fully evaluate materialenvironment interactions under harsh conditions.



Learn More

(Materials Reliability Division)

Publications

Barbosa III N, Keller RR, Read DT, Geiss RH and Vinci RP *Comparison of Electrical and Microtensile Evaluation of Mechanical Properties of an Aluminum Film* Metallurgical and Materials Transactions, 38A: 2160 (2007)

Serrell DB, Oreskovic TL, Slifka AJ, Mahajan RL and Finch DS *A Uniaxial BioMEMS Device for Quantitative Force-displacement Measurements* Biomed. Microdev., 9 (2): 267 (2007)

Darcis PhP, McCowan CN, Windhoff H, McColskey JD and Siewert TA *Crack Tip Opening Angle Optical Measurement Methods in Five Pipeline Steels* Engr. Fracture Mech., 75: 2453 (2008)

Siewert TA, McColskey JD and Ricker RE *Report of NIST Workshop on Materials Test Procedures for Hydrogen Pipelines* NISTIR, 6649 (2007)



Sudook Kim

Nick Barbosa

(303) 497-3445

barbosa@nist.gov

www.nist.gov/reliability