HIGH TEMPERATURE TESTING of ADVANCED MATERIALS in ACTUAL COAL COMBUSTION ENVIRONMENTS

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INTRODUCTION:

This project continues activities with numerous external organizations that are conducting advanced material's R&D to support the Indirect-Fired Cycles Program and Advanced Research & Technology Development Program. These programs have emphasized development of high-temperature ceramic and alloy materials for heat transfer applications, such as slag screens for high-temperature advanced furnaces, and crosscutting applications, such as filters**.** New materials are needed for applications in advanced power systems because of the higher operating temperatures and the severe environment. A new variety of materials have been developed and appear to be viable candidates. Several of these materials have been tested in different environments and different test equipment as well as on probes in operating power plants, but much more testing needs to be done. Tests in boilers are expensive and are limited in operating conditions, such as temperature. As part of a planned testing program, new materials and advanced coatings will be tested in the pilot-scale coal burning facilities at NETL(e.g., CERF) and the combustion and hot gas filter facilities at the University of North DakotaEnergy and Environmental Research Center (EERC). The planned testing will directly support the Fossil Energy Materials Program and materials R&D studies conducted at Oak Ridge National Laboratory (ORNL).

The CERF evaluations are valuable because some researchers under the FE/Oak Ridge-funded projects don't have ready access to pilot-scale facilities, and have only used laboratory muffle furnaces (with ash manually applied to surfaces) that don't realistically simulate actual combustion environments. The CERF tests allow verification of these muffle furnace tests and various thermochemical equilibrium modeling predictions.

RESEARCH OBJECTIVES:

Several new materials systems are potentially applicable to coal-fired combustion, however, because of limited property characterization in actual coal combustion environment additional research to prove their suitability is warranted. The ultimate objective of this research effort a long duration testing of various promising materials and their components in an actual coal combustion environment.

LONG TERM GOALS/RELATIOHSHIP TO NETL's PRODUCT LINE(S):

This reasearch work directly supports the Fossil Energy's Indirectly-Fired Cycles Program and Advanced Research & Technology Development Program. These programs have emphasized development of high-temperature ceramic and alloy materials for heat transfer applications, such as slag screens for high-temperature advanced furnaces, and crosscutting applications, such as filters**.** New materials are needed for applications in advanced power systems because of the higher operating temperatures and the severe environment. The planned CERF testing directly supports the Fossil Energy Materials Program and materials' R&D studies conducted at Oak Ridge National Laboratory (ORNL).

Longer term goals are to evaluate the behavior of numerous new materials (and advanced coatings) behavior in realistic fossil fuel composition environments, compare their behavior in different test stands, and test at several appropriate temperatures and relate the results to utility application.

Build upon NETL's involvement with ORNL, ANL, NCC Engr., UND/EERC and other materials' researchers. Publish at least two papers describing the NETL activities and the significance of results with project partners in an effort to gain more external stakeholder support. Develop new opportunities that lead to the evaluation of successful new materials and/or coatings that can benefit advanced power systems development, including activities that support CO_2/O_2 -enriched combustion and material issues.
Other interested organizations will also be invited to provide high temperature materials for each test in the larger chambers (radiant furnace) of the CERF where the gas temperatures are much higher. We expect the smaller chambers (a convective pass) to be filled with the metals mentioned above to study corrosion in the target 1500°F-1600°F range.

SUMMARY ACCOMPLISHMENTS:

Four Tasks were conducted in the materials project in FY01. These are listed below:
Task1. McDermott Project . In this task metals, welds are currently being exposed in the Ohio Edison Niles Plant in Ohio(high sulfur coal, reheat super heat temperature 1100 F). The samples will be monitored after 1-, 3-, 5-year . The last inspection was in April 2000 and the next one scheduled for April 2002. Same samples are being exposed in 500 lb/hr unit.
Task 2. Collaboration with Argonne National Laboratory. To conduct piggyback materials evaluation tests on alloys supplied by Argonne burning coal/biomass in the CERF.
Task 3. Collaboration with West Virginia University. Thermal and Mechanical properties of samples obtained from McDermott will be conducted.
Task 4. NCC Engg./ Oak Ridge National Lab(ORNL), / Energy and Environmental Research Center (EERC)/ National Energy Technology Lab (NETL). NETL will conduct dedicated CERF test exposure on metals, claddings, ceramics, and coatings at different tempertures for 350-, 750-, and 1000- hours while burning one coal. In Dedicated tests, dozens of samples will be placed in numerous CERF location as identified by the team. Metals will be put at the cooler temperatures of 1500-1600 F. ODS materials will be tested at 1800-2000F. Ceramics and coated ceramics will be tested at 2000-2500 F, and possibly as high as 2800 F.
In FY01, most of the work was conducted on the Task 4. We shall discuss this task below.

Task 4. NCC Engineering Inc./ORNL/EERC/NETL Collaborative R&D Studies of Corrosion for Fossil Energy Materials:

The Combustion and Environmental Research Facility(CERF) was used to assess the performance of solid, liquid, and gaseous fuels in typical pc systems. Commissioned in 1989, the basic design criterion for the 500,000 Btu/hr CERF achieves similarity with full scale utility boilers to study various combustion, emissions, and heat transfer concepts. The CERF has also served as a host site for exposure of ceramic and alloy samples for future combustion applications. To-date, more than 100 ceramic and alloy samples have been exposed in the CERF, including coupons, tensile strips, and ceramic tubes (2-inch ID by 4-foot length) at ambient pressure as well as at 200 psig (for tubes). Samples were inserted in five different CERF locations covering

1700°F-2600°F, with exposures exceeding 1000 hours. CERF tests allow FE/project partners to realistically screen advanced materials, and study corrosion and other gas/ ash/ material interactions that will impact slag screens, heat exchangers, or filters for fossil energy applications.

A key consideration in the planned NCC Engineering Inc./ORNL/EERC/NETL corrosion R&D study is that the target alloy exposures of 1500F-1600F inside the CERF's rear convective pass are lower than in prior work. Initially, dedicated round the clock CERF material's tests are planned so that a lower load (approximately 75% of the full load) operation can be conducted to achieve 1500°F-1600°F gas temperatures where dozens of alloy samples will be inserted in the rear locations of the CERF's convective pass to enable nearly isothermal alloy corrosion studies.

The planned lower load operation will greatly minimize ash deposition behavior (e.g., slagging and fouling) and allow for long duration testing with little or no rodding of the convective pass and thus, minimum disturbances where the target alloy samples will be inserted.However, this low load operation, combined with the fact that only one coal will be used for this project, will require dedicated CERF materials testing as opposed to piggyback testing used previously where materials were exposed to higher temperatures and at varying fuel/combustion conditions.

In this project, the CERF firing rate and other parameters will be adjusted to achieve steady 1500°F-1600°F gas temperatures in the rear locations of the convective section. The planned dedicated CERF testing will thus allow upstream locations (inside the radiant combustor and front convective section) to be utilized for other materials R&D studies at higher temperatures. Project participants will work with NETL to help obtain additional samples that could be simultaneously inserted/exposed in the CERF locations upstream of the 1500°F-1600°F alloy zone.

A comprehensive work plan (in progress) has been developed to expose selected advanced materials appropriate for use in advanced power plants. Other interested organizations will also be invited to provide high temperature materials for each test in the larger chambers (radiant furnace) of the CERF where the gas temperatures are much higher. We expect the smaller chambers (a convective pass) to be filled with the metals mentioned above to study corrosion in the target 1500°F-1600°F range. Several meetings were held with the ORNL partners to discuss the strategy of designing the sample probe.

In the NETL's CERF we expect to test metals, claddings, ceramics, and coatings at different temperatures while burning one coal. This important test will be dedicated to materials testing. Metals will be placed at the cooler temperatures of 1500°F-1600°F. The oxygen dispersed samples(ODS) will be tested at 1800°F-2000°F and ceramic and coated ceramics will be tested at 2000°F-2500°F, and possibly as high as 2800°F, if appropriate. They are to be exposed for 100-, 300-hr, 500-, and 600-hrs in CERF.

We received168 samples of metals, alloys, ceramics, and cladded and coated ceramics for evaluation at high temperature.

A brief description of these samples is given below:

Ceramic materials: The ceramics and the coatings that will be tested in the CERF facility are numerous. Some are uncoated silicon carbide and silicon nitride. SiC will be coated with NZP, spin coated with mullite and CVD(copper vapor deposition) coated with mullite by ORNL. We suggested acquiring some plasma sprayed mullite samples from United Technology. Allied Signal has been requested to coat SiC and possibly silicon nitride with tantalum oxide. Some alumina/silica composite samples from Boeing will also be tested.

Out of 139 samples from ORNL, 69 samples are ceramic based and the rest (70) are assorted alloys. Among the ceramic samples, 21 samples were mullite foam from Beth Armstrong (ORNL), 12 were alumina/laminate and 21 alumina silicate/laminate from Composite Optics Inc., six ring samples of SiC/SiC-ring from Phil Craig (Honeywell), 4 samples were Plasma Spray TaO coated SiC from C.W Li (Honeywell) and finally 5 samples were CVD mullite-coated SiC from Haynes (ONRL)**.** It is difficult to drill holes in ceramic samples. It was decided to build and test a new sample probe. Various ideas were suggested. First a plain mullite tube was tested in CERF for 100 hours. A new probe for ceramic samples is being developed for these tests. Initial test runs using this probe were very promising. Another probe was designed and fabricated for evaluating metals and alloys has already been tested successfully in theCERF.

The higher priority metals. A total of 70 samples of metals and alloys were also received by NETL. Uncoated, coated,and cladded samples are to be tested at 1500°F, may be 1600°F in CERF and at EERC. Some have already been tested at the Niles and Gallatin power station at lower temperatures for longer times. Some of the high priority samples are listed below, which includes ODS materials also.

- INCO803 or HP modified
- Thermie alloy (Fe-25Cr-20Co-Al-Ti-Nb)
- FeAl clad 310 stainless steel
- HR160 (high Si, some Co0)
- Alloy 214 (Alpha former with Ni)

We plan to expose the ODS materials at 1800°F (1000°C, may be 1100°C)

- PM2000 and Iron Aluminide.

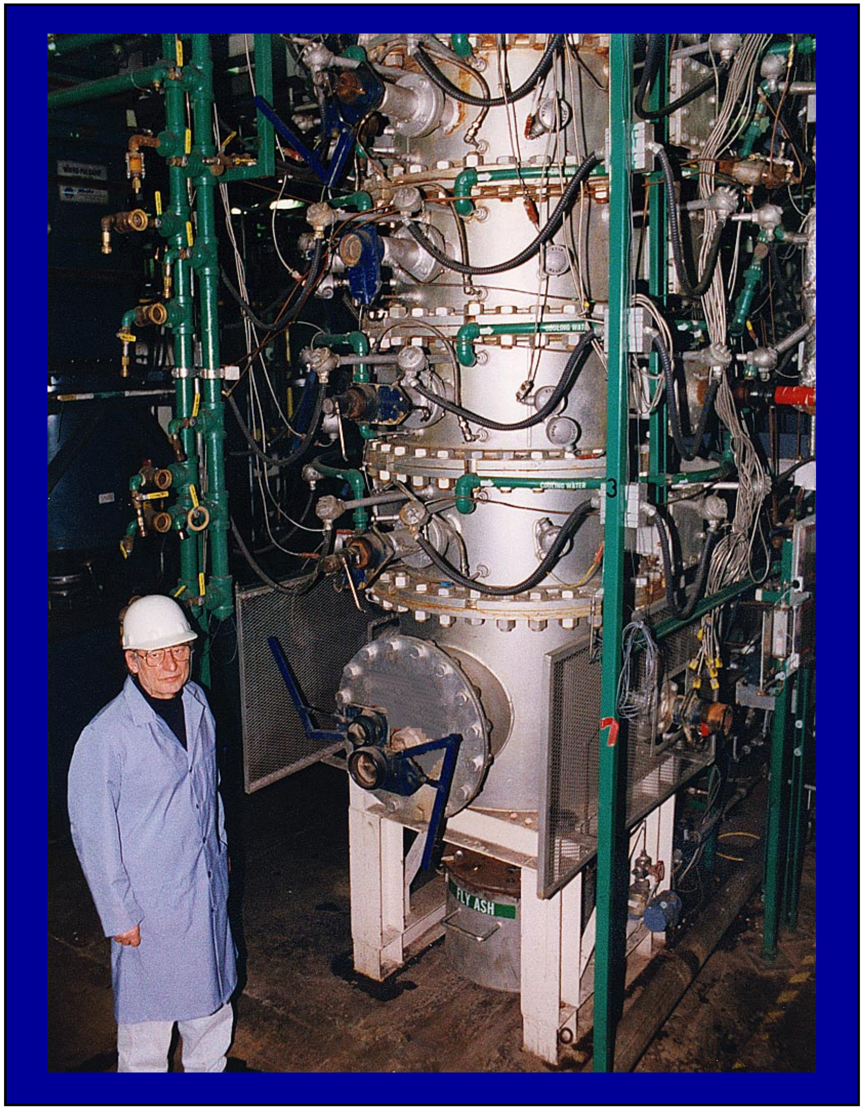
The lower priority metals - uncoated, coated, cladded will be tested if we have room at 1500°F or 1600°F in CERF and EERC. Some of them have been tested at lower temperatures for longer times. Some of the lower priority materials are listed below.

- HR120	Modified 310
- NF709	FeAL strip
- SAVES 25	VDM 45
- VDM 602	671
- Modified 800	347 FG
- Fe-35Cr-2.5 Si	Others

RESULTS:

Most of the year several probe designs were evaluated for the ORNL dedicated tests in CERF. Our Inconel 600 design worked satisfactorily but ORNL thought that Si from Inconel might adversely effect the samples such as ODS alloys becvause of proximity. A ceramic swing design was proposed to alleviate this problem. But unfortunately, the ceramic shattered to pieces while cooling down. This happened two times. This idea was abandoned. The group decided to fabricate the probe out of Inconel 601. This required a custom made Inconel 601 bars for the probe. The cost of such a probe was prohibitive because the vendor will be making the Inconel 601 specially for us. Fortunately, ORNL suggested the use of Haynes HX(Hastelloy). One of ORNL’s contractors had some Hastelloy plates as scraps. Finally, probes were fabricated out of Hastelloy. We were able to acquire Hastelloy in plate form from Specialty Metals, West Virginia. It was cut into bars and then flattened by NETL technicians using brute force. This consumed lot of our time. As a result the testing of the samples in the CERF was delayed. Some results are shown in the photographs. See attached photos.

Combustion and Environmental Research Facility



500,000 Btu/hr Multi-Fuel Solids, Liquids, Gases

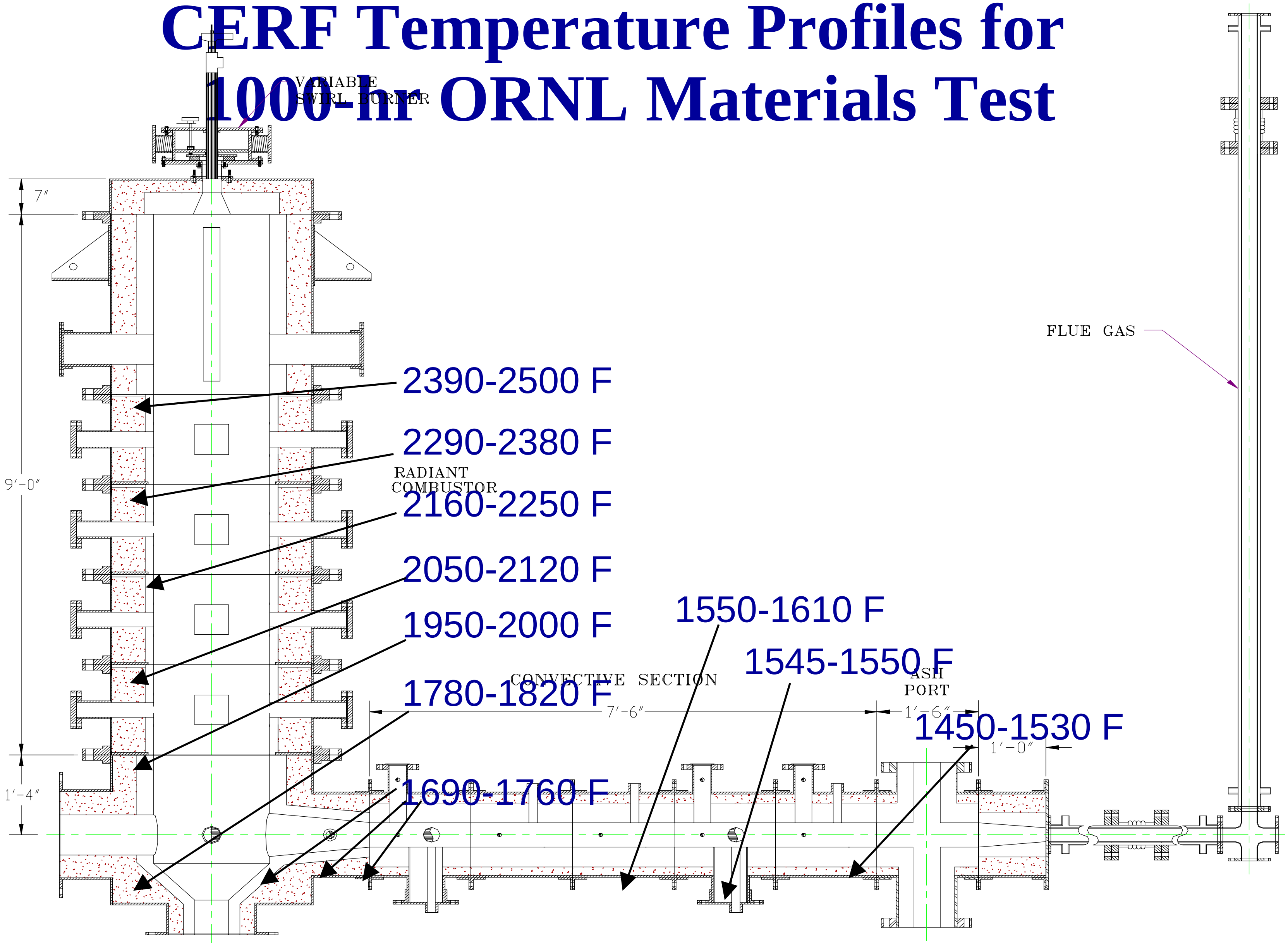
Achieves Similarity With Utility PC Boilers to Study:

- **Fuel Handling**
- **Combustion**
- **Flame Stability**
- **Heat Transfer/Materials**
- **Ash Slagging and Fouling**
- **Flue Gas Emissions**

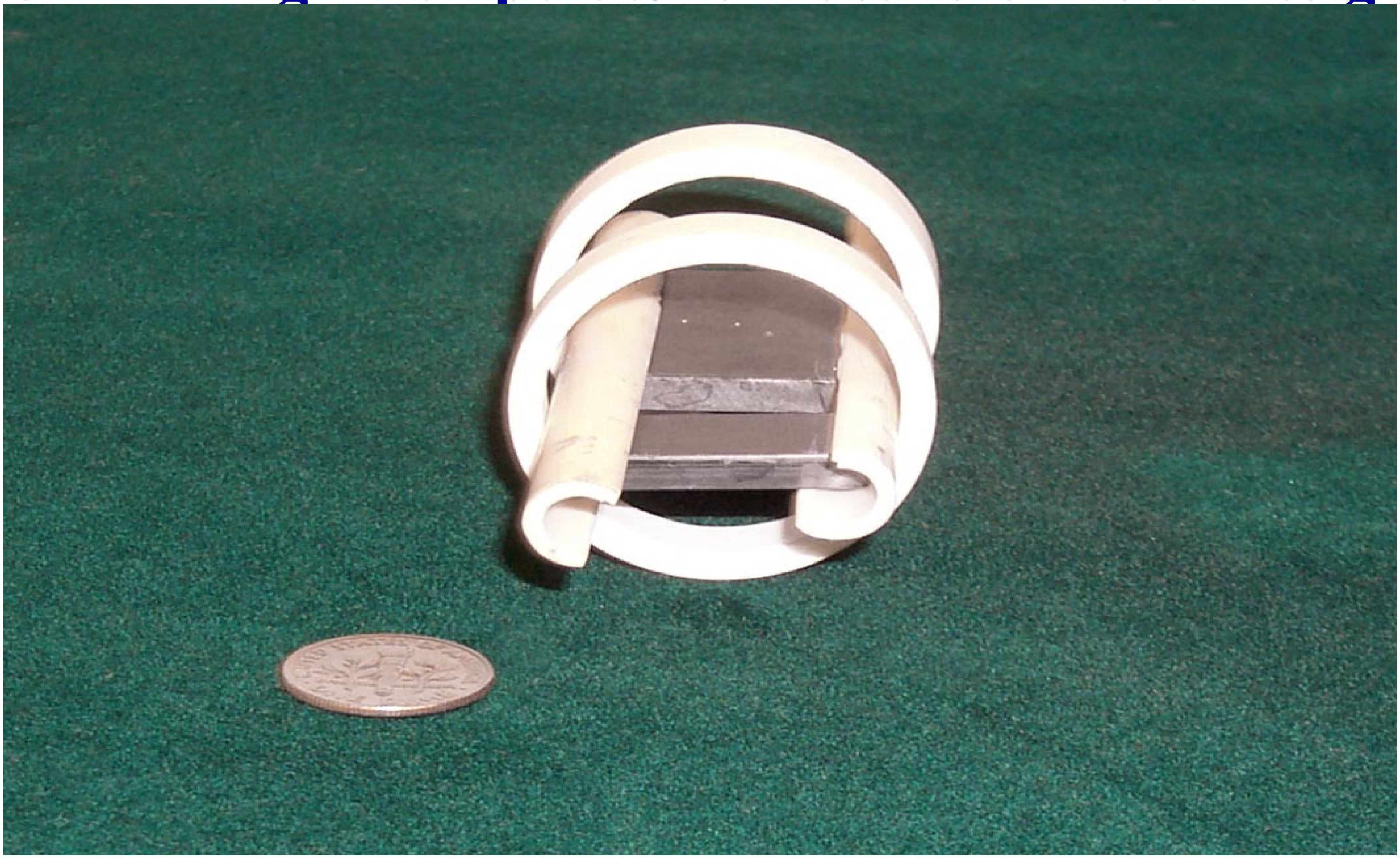
Processes to Improve Combustion and Efficiency, and Reduce Pollution

NETL

CERF Temperature Profiles for 1000-hr ORNL Materials Test

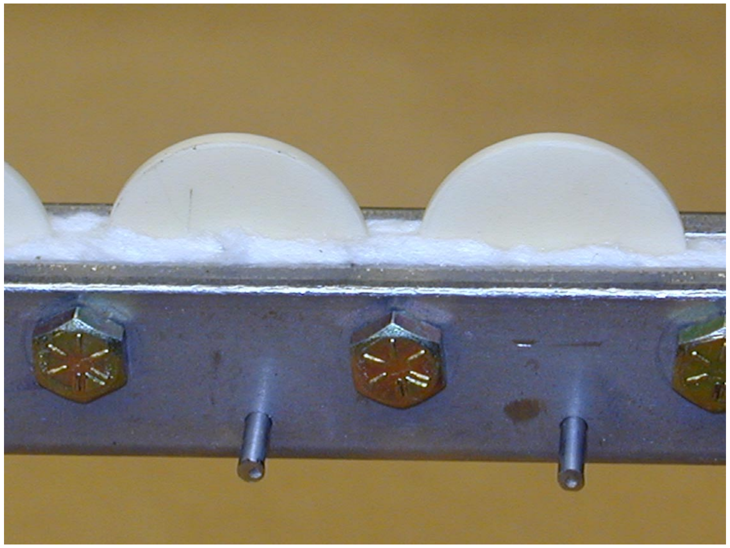


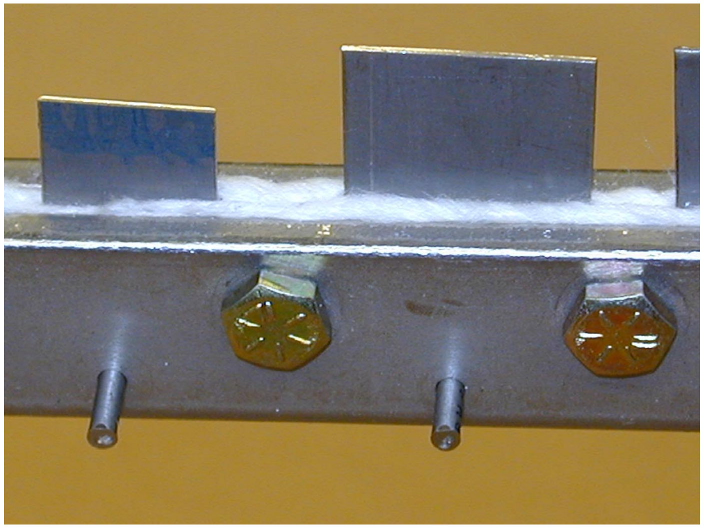
ORNL High-Temperature Materials Probe Design

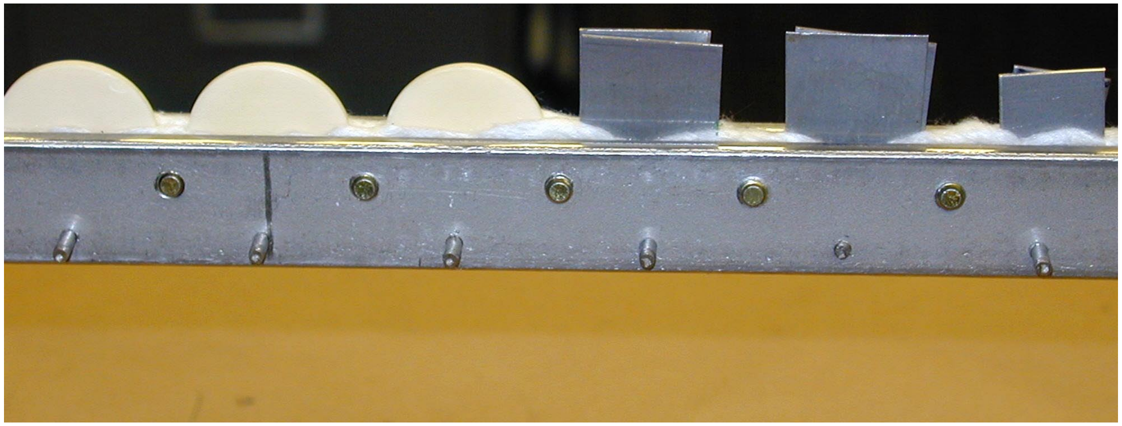



DATE MARCH 5

NETL Inconel Clamped Probe Assembly

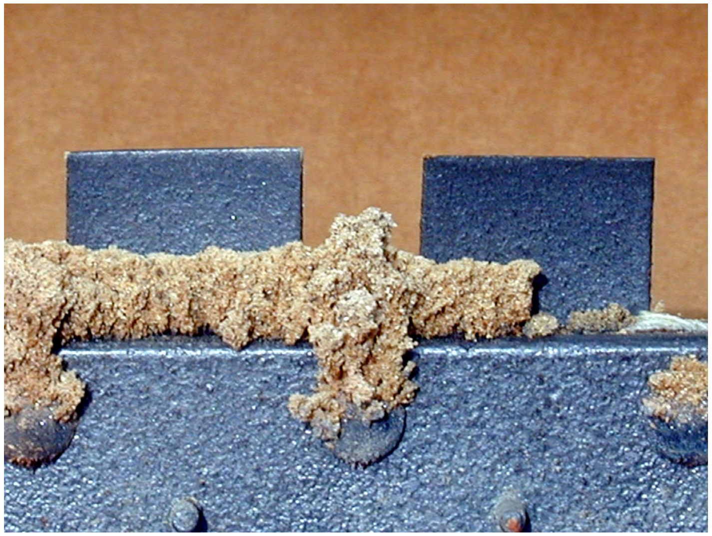


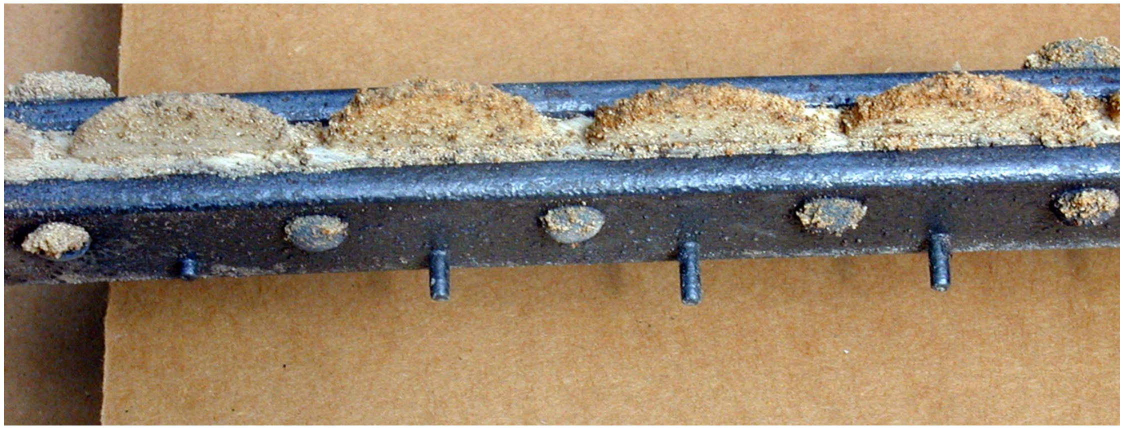




Samples secured from side by two clamped inconel plates, high-temperature yarn and supported underneath by inconel pins

Inconel Clamped Probe Assembly After Test





Probe held-up well during several 100-hr test with no lost samples

Probe exposed at 2220-2320°F during 100-hr tests

CERF Combustor After Test

Top View Looking Down into CERF Combustor & Refractory Walls

<u>note:</u>
inconel clamped probe (63-inches from top of combustor)
short mullite (C-cut ends) tubes (81-inches and 99-inches from top)

NCC/ORNL/EERC-ND/ANL/NETL
FE Materials and Corrosion R&D

- **Alloy Test Samples**:

– Chrome Alloy (Cr-9Ta-5Mo-2Si-0.15La-0.1Ti)	2 sets	2100-2400^{o}F
– Chrome Alloy (Cr-6MgO-0.75Ti)	2 sets	1900-2100^{o}F
– Standard 803 Alloy	1 set	1500-1600^{o}F
– Modified 803 Alloy (Fe-25Cr-35Ni) 3 types	1 set	1500-1600^{o}F
– RA253MA	1 set	1500-1600^{o}F
– INC-625	1 set	1500-1600^{o}F
– NF-709 (0.04 Annealed)	1 set	1500-1600^{o}F
– 310HCBN SS	1 set	1500-1600^{o}F
– 310TAN	1 set	1500-1600^{o}F
– ODS Alloy	7 sets	1700-2100^{o}F
– Thermie Alloy	2 sets	1500-1800^{o}F
– Haynes 230	2 sets	1500-1800^{o}F
– ANL 13 Types	2 sets	1500-1600^{o}F

NCC/ORNL/EERC-ND/SRI International/NETL FE Materials and Corrosion R&D

- **Ceramic Test Samples**:
  - Mullite foam 7 sets 1900-2400ºF
  - SiC/SiC ring (2 types) 1 set 2000-2100ºF
  - Alumina/Laminate 4 sets 1900-2400ºF
  - Alumino-silicate/Laminate 7 sets 1900-2400ºF
  - CVD mullite coated SiC (4 types) 1 set 1900-2000ºF
  - Plasma spray TaO coated SiC 2 sets 2100-2200ºF
  - CFCC from SRI International 1 set 1900-2000ºF (3 Sample Types)

Multiple sample sets allow for corrosion studies at different exposure times (300-,700-,1000-hr) and temperature windows

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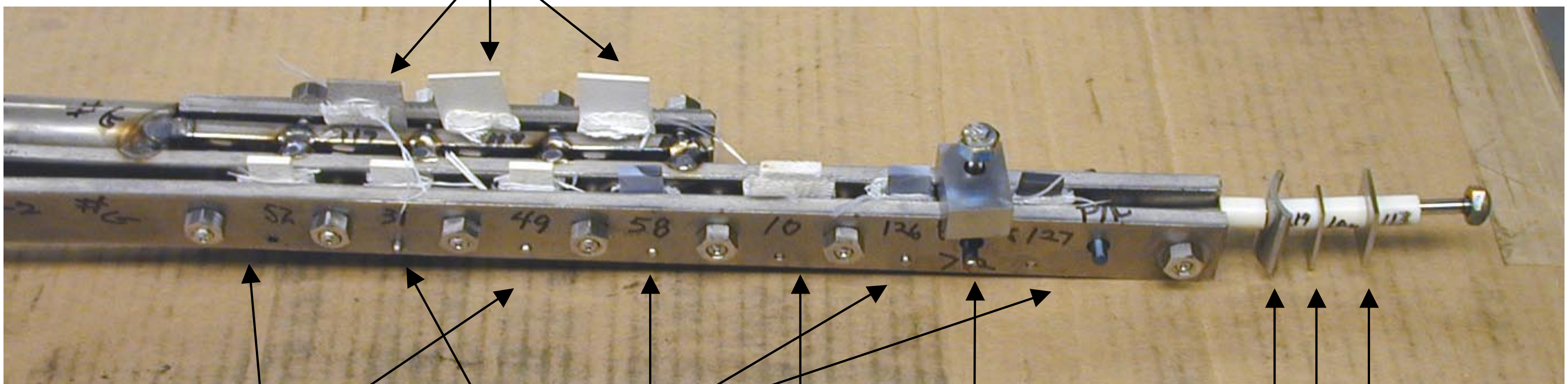
High-Temperature Materials Probe Design

SRI International Ceramic Coupons



ORNL Alumino-silicate/ Laminate 2 Samples

ORNL Alumina/ Laminate

ORNL CVD Mullite-Coated SiC 3 Samples

ORNL Mullite Foam

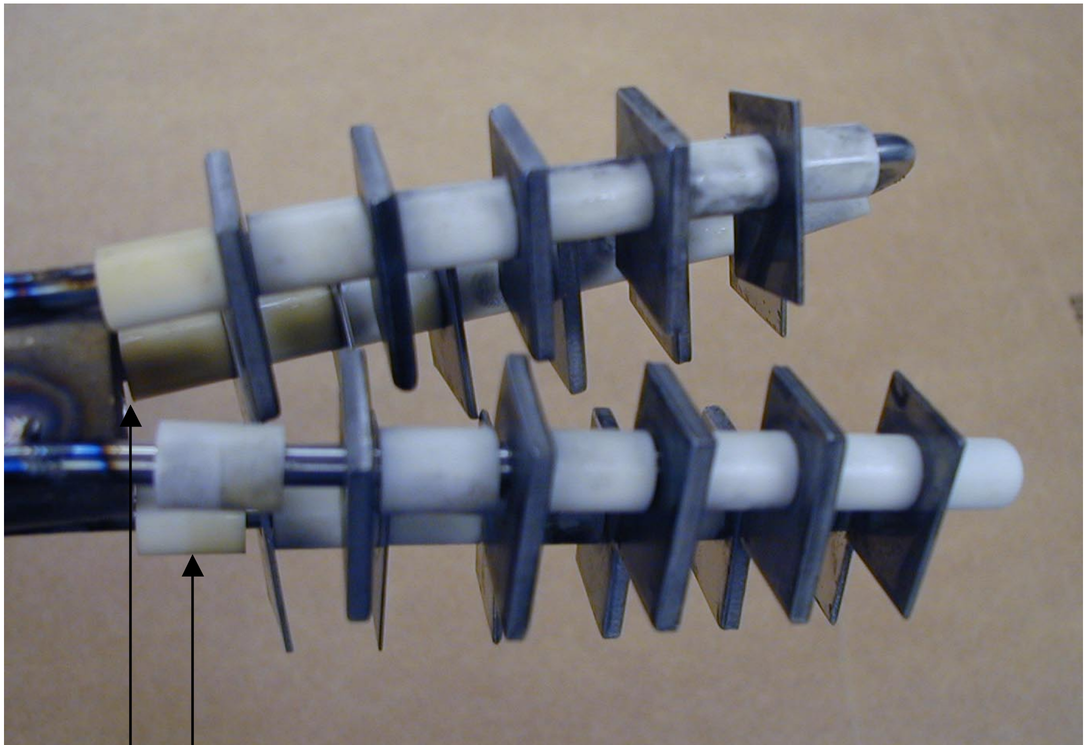
ORNL Chrome Alloy Cr-6MgO-0.75Ti

ORNL ODS Alloy 3 Samples with Alumina Spacers

Hastelloy X (Body, Bolts, Pins) With Cotronics Ultra Temp 391 (Continuous Filament High Alumina Fibers) Tape/Cloth

Samples Exposed at 1900-2000ºF During CERF Testing

High-Temperature Materials Probe Design



<u>Two Sample Sets Each of</u>

- Haynes 230
- Thermie Alloy
- 310TAN
- 310HCBN SS
- Standard 803
- Modified 803 - 3 samples
- INC-625
- NF-709
- RA253MA

Sample Set for 350-hr Test

Sample Set for 1000-hr Test

Hastelloy X Probe with Alumina Spacers

Samples Exposed at 1500-1600ºF During CERF Testing

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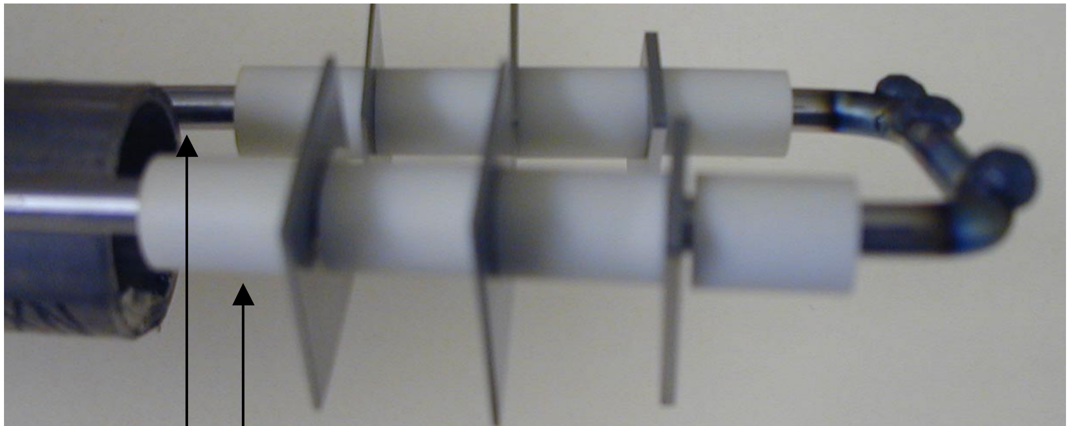
High-Temperature Materials Probe Design



Two Sample Sets Each of

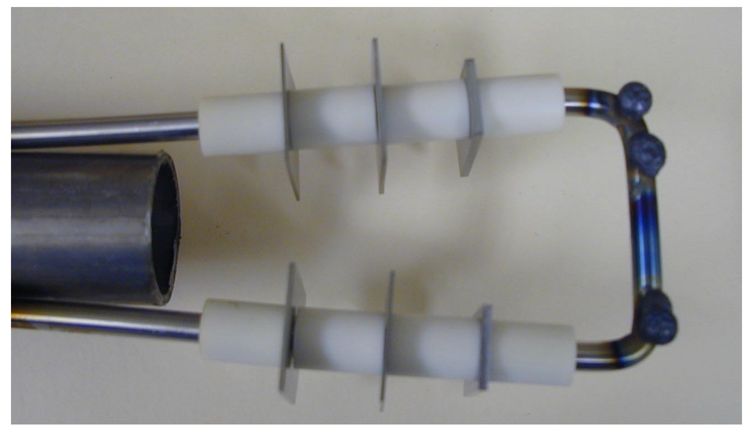
Haynes 230
Thermie Alloy
ODS Alloy

Side View

Sample Set for 350-hr Test

Sample Set for 1000-hr Test

Top View



Hastelloy X Probe with Alumina Spacers
Samples Exposed at 1700-1800ºF During CERF Testing

NETL