

# *Metallic Filters for Hot Gas Cleaning*



Iver E. Anderson, Brian Gleeson, and Robert L. Terpstra

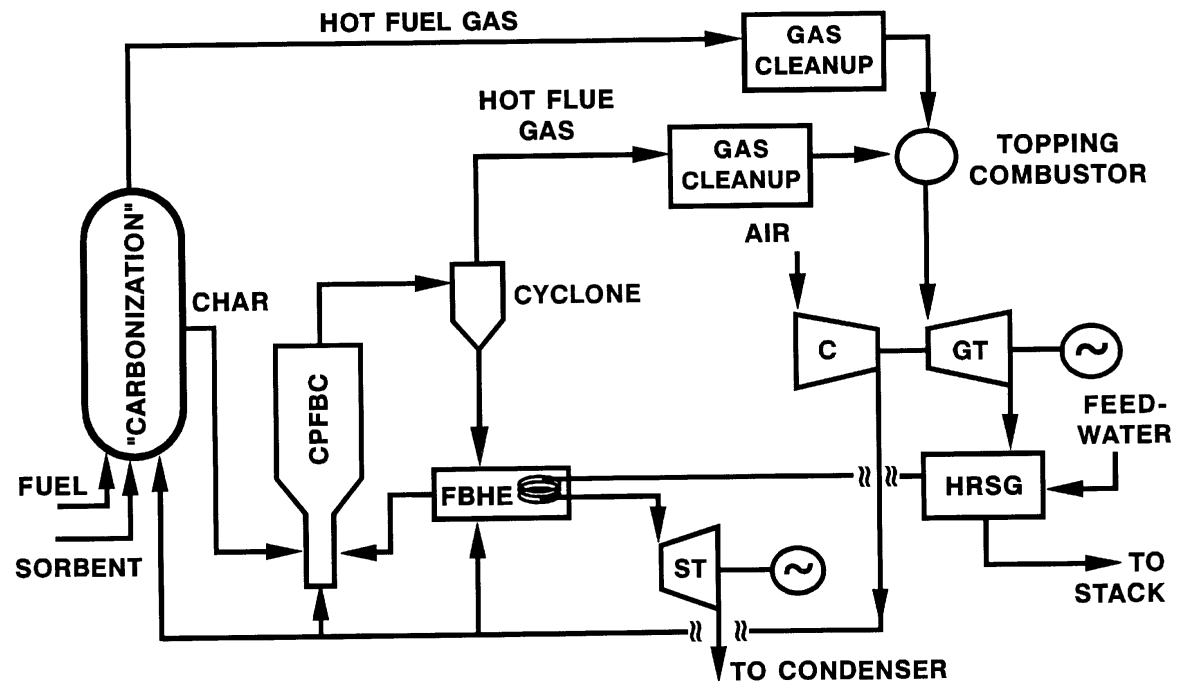
Metal and Ceramic Sciences Program, Ames Laboratory (USDOE),  
Iowa State University, Ames, IA 50011

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# Advanced Coal-Fired Power Generation Systems: Need for Particulate Filtration

- pressurized-fluidized bed combustion (PFBC)
- integrated gasification combined cycles (IGCC)
- both require gas cleanup chambers

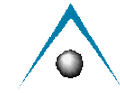


Advanced PFBC cycle

M. J. Mudd, "Pressurized Fluidized Bed Combustion", published by Blackie Academic & Professional, 1995, page 125.



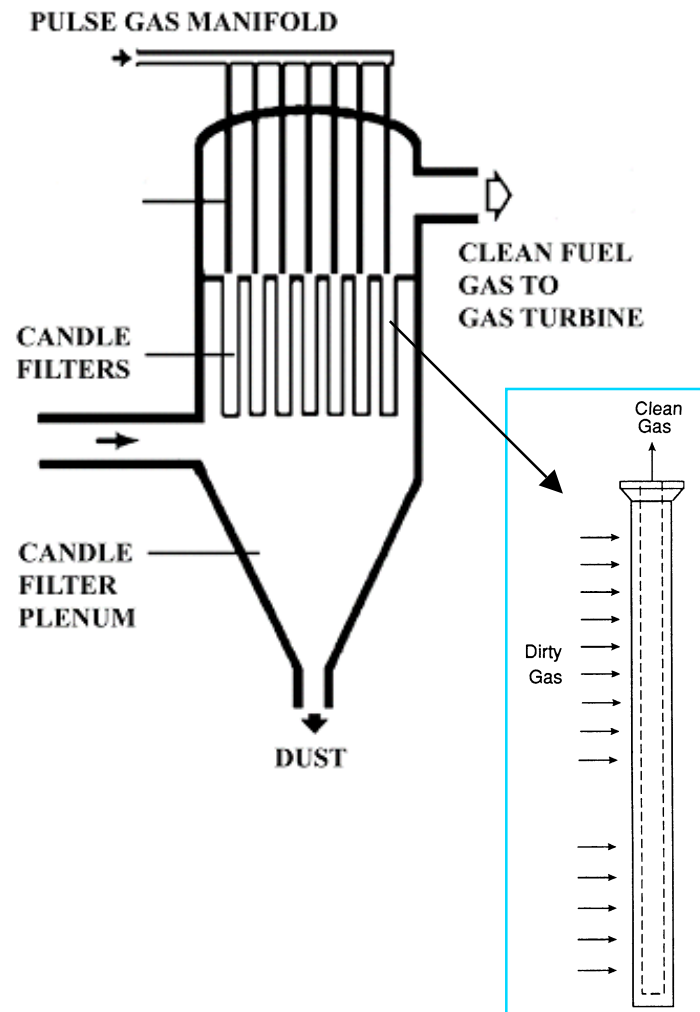
# Hot Gas Clean-up Chamber



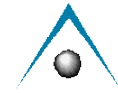
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- Gas clean-up chamber protects the downstream gas turbine
- Porous ceramic “candle filters” strip particles from the hot PFBC gas stream.
- Back flushing with cold gas jets imposes thermal shock/fatigue cycles.

**BARRIERS:** Current ceramic filters are brittle and prone to bending and fatigue failure. New intermetallic ( $\text{Fe}_3\text{Al}$ ) filters have improved HT ductility, but need: RT ductility, HT strength, and weldability.



# Alternative Metallic Filter Technology

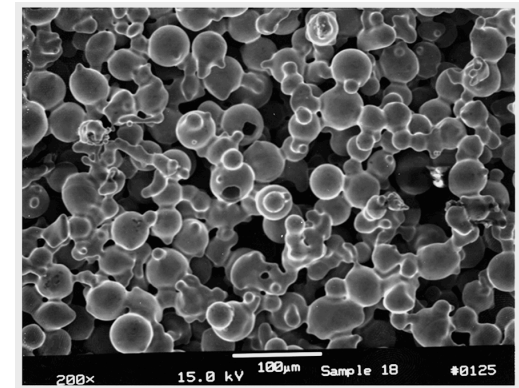


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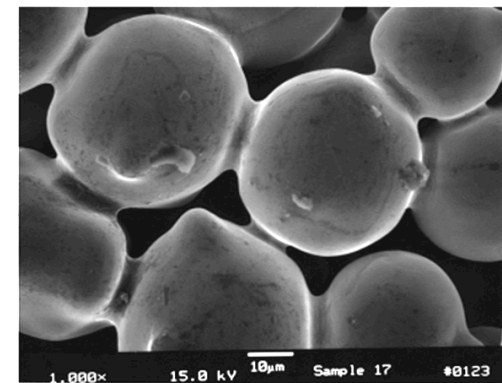
**Goal:** New superalloy metallic filters should offer long term environmental stability and robust mechanical properties (RT to HT) suitable for PFBC and IGCC applications.

## Previous Results:

- Ni-16Cr-9Al-3Fe bulk (cast, pre-oxidized) alloy was found to be corrosion resistant (equal to  $\text{Fe}_3\text{Al}$ ) in a PFBC gas environment and stronger (4 times Y.S.) at 850°C.
- Thin, porous sheets made by vacuum sintering of spherical gas atomized powder of Ni-16Cr-9Al-3Fe were:
  - strong
  - ductile
  - permeable
  - weldable
- Corrosion tests of pre-ox. porous sheets in PFBC gas environment showed moderate oxide surface recession.



200x



1000x

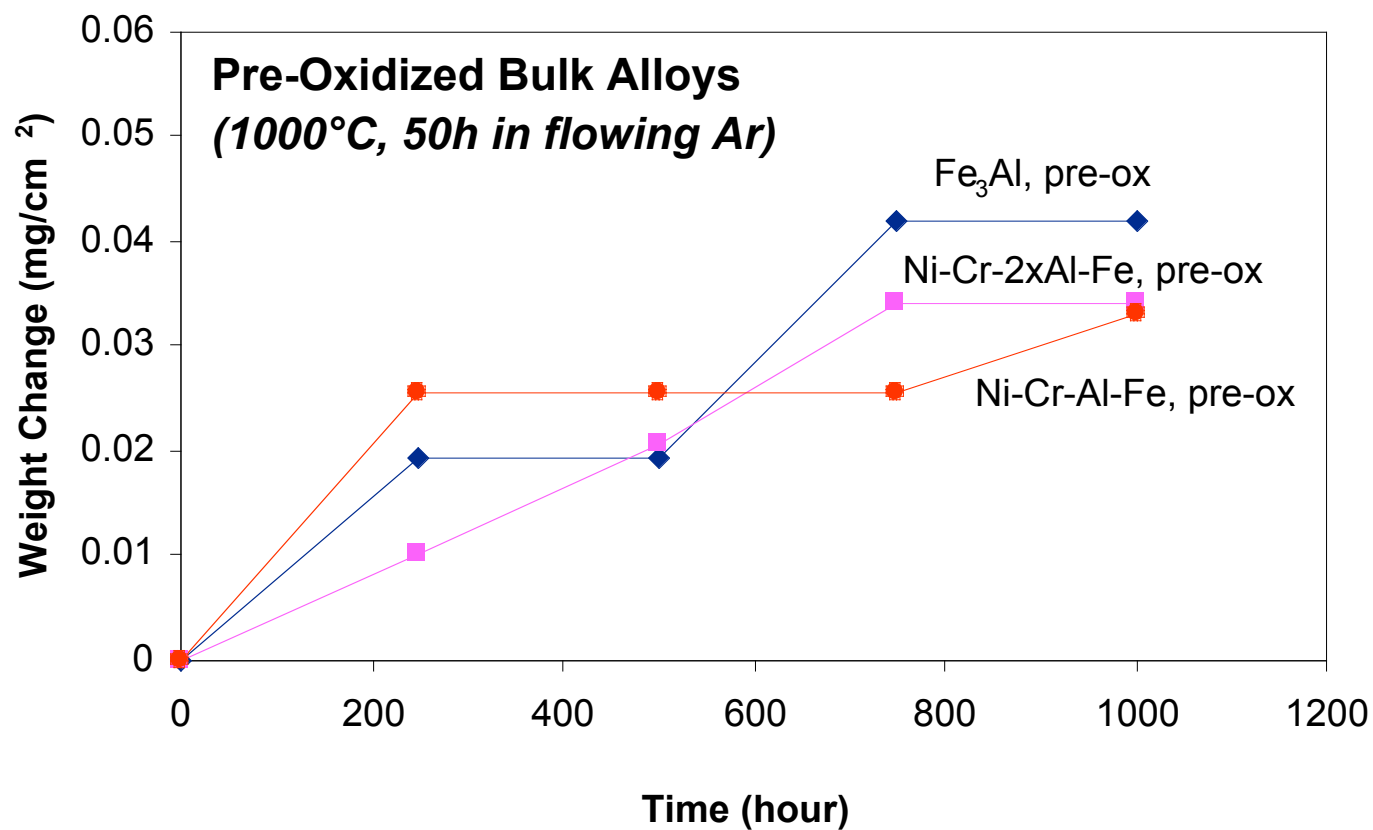
Sintered Ni-Cr-Al-Fe Powder,  
25 µm < dia. < 45 µm

# PFBC Hot Gas Oxidation/Corrosion Tests

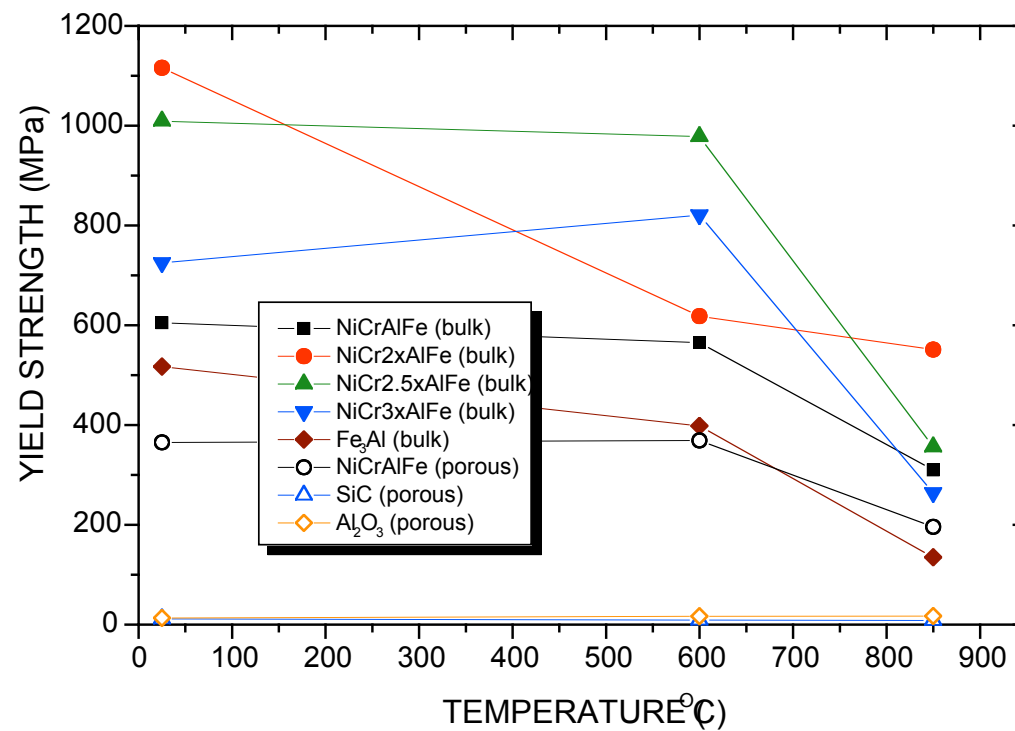


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PFBC Atmosphere@ 850°C = N<sub>2</sub> – 13 CO<sub>2</sub> – 10 H<sub>2</sub>O – 4 O<sub>2</sub> – 250 ppm SO<sub>2</sub>



# High Temperature Asymmetric Four Point Bend Test: Yield Strength Results



# Filter Backpressure Tests

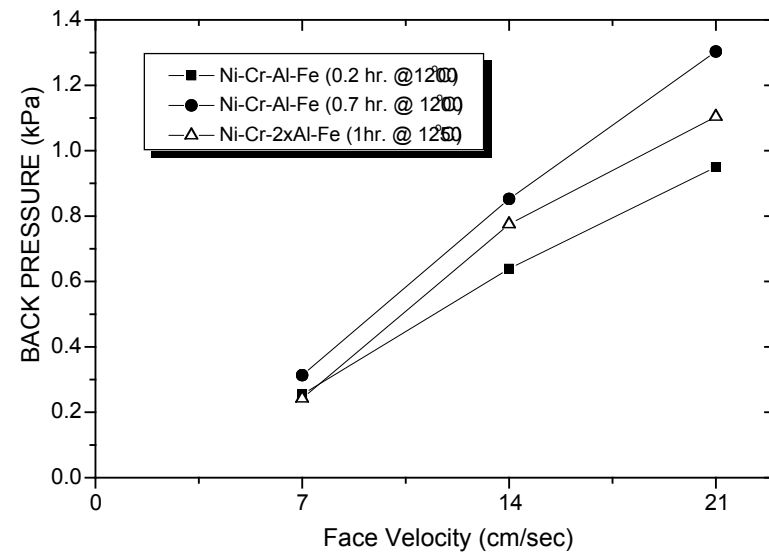
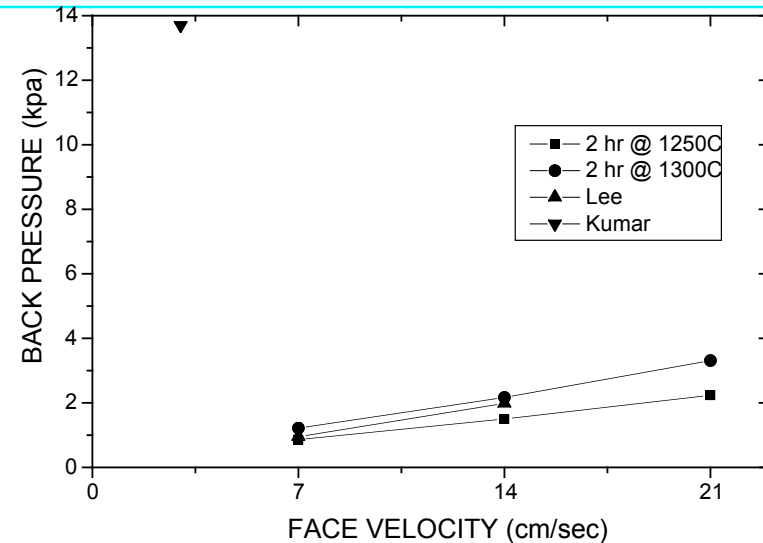


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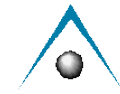
D'Arcy's Law:

$$\dot{V} = \frac{k_D A \Delta P}{L}$$

- With an appropriate pore network (30% porosity) from design of particle size range and sintering cycle, suitable backpressure levels can be achieved for similar sample thickness (10mm).
- Porous sheet with reduced thickness will have a lower pressure drop.



# Porous Sheet Joining by Resistance Welding



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## SAMPLE:

Material: Ni-Cr-2xAl-Fe powder

Particle Size:  $25\mu\text{m} > \text{dia.} < 45\mu\text{m}$

Density:  $\sim 70\%$

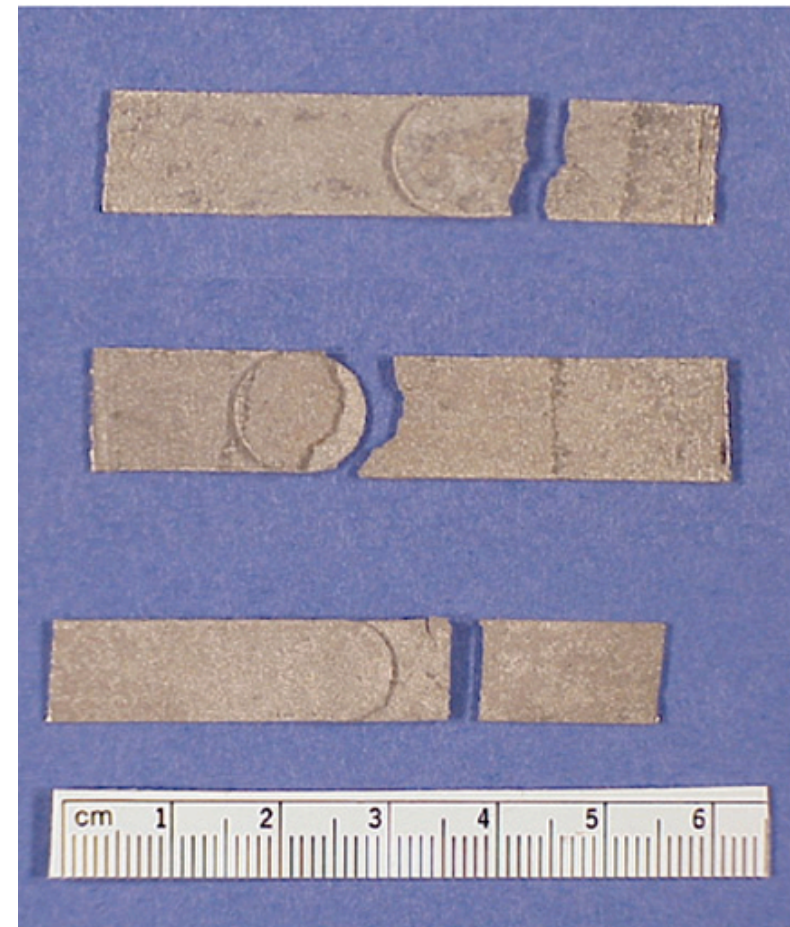
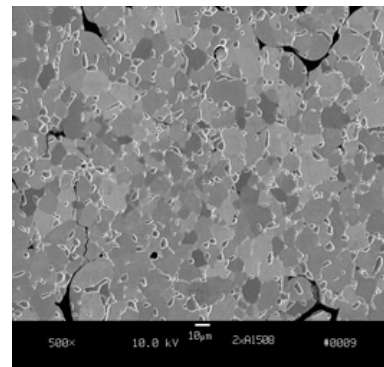
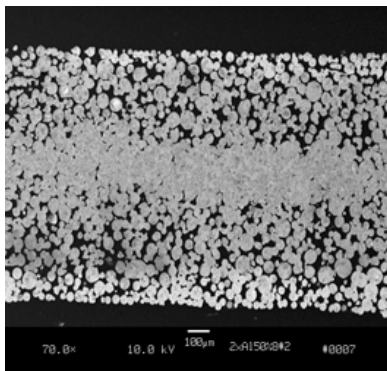
Width: 9.5 mm

Thickness: 0.5 mm

## TEST CONDITIONS:

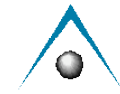
10KVA, 66ms per spot weld

Strain Rate: 0.1 mm/min





# *Rolled & Welded Test Cylinder*



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“O-ring” Test Cylinder:

Dia. = 60 mm

Height = 38 mm

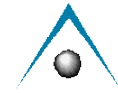
Wall thickness = 0.5 mm

Fabricated using Ni-Cr-2xAl-Fe powder,  $25\ \mu\text{m} < \text{dia.} < 45\ \mu\text{m}$ , vacuum sintered, rolled and resistance spot welded.

Samples in test at EERC under IGCC and PFBC conditions.



# *Purpose and Current Approach*



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## PURPOSE:

Select alloy candidates for use in IGCC environments to enable further development of porous metallic filter processing from a series of alloys with both chromia and alumina scale forming characteristics to investigate possible use in both PFBC and IGCC applications.

## APPROACH:

- Perform corrosion resistance tests IGCC gaseous atmosphere using chromia-formers and Mo-modified alloys, along with previous Ni-Cr-Al-Fe alloy modifications (including porous samples).
- Enhance understanding of microstructure and property effects during porous sheet sintering process and develop improved sintering mold materials to facilitate eventual scaling of process to produce material for candle filter fabrication.
- Analyze results of extended exposure testing of porous samples in PFBC and IGCC environments.



# *Alloys Selected for IGCC Test*



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## **Commercial**

Fe-15.8 wt/o Al-2.2wt/o Cr-0.2wt/o Zr  
Fe-22.0wt/o Cr-20wt/o Ni-18wt/o Co-3wt/o Mo  
Ni-30wt/o Co-28wt/o Cr-3.5wt/o Fe-2.75wt/oSi  
Ni-16wt/o Cr-4.5wt/o Al-3wt/o Fe

## **Experimental**

Ni-16wt/o Cr-9.0wt/o Al-3wt/o Fe  
Ni-16wt/o Cr-13.5wt/o Al-3wt/o Fe  
Ni-16wt/o Cr-9.0wt/o Al-3wt/o Fe-20wt/o Mo  
Fe-17.6wt/o Mo-9.4wt/o Al

## **Designation**

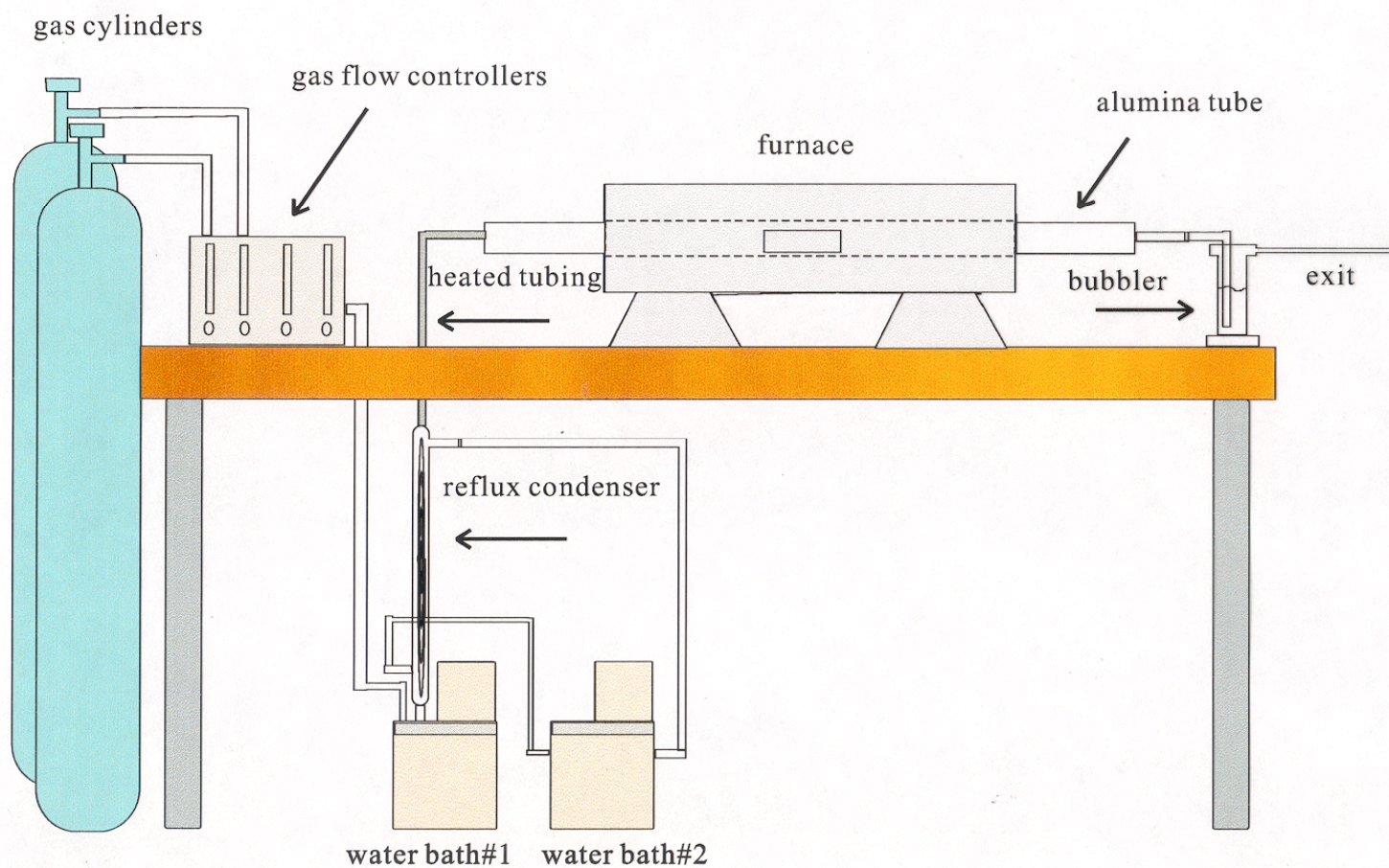
Fe<sub>3</sub>Al □  
Fe-Cr-Ni-Co-Mo-W  
Ni-Co-Cr-Fe  
Ni-Cr-Al-Fe  
  
Ni-Cr-2xAl-Fe  
Ni-Cr-3xAl-Fe  
Ni-Cr-2xAl-Fe-Mo(20%)  
Fe-Mo-Al

# Hot Gas Corrosion Test System



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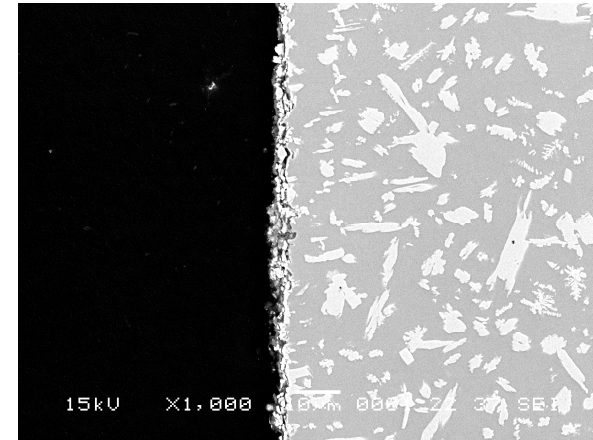
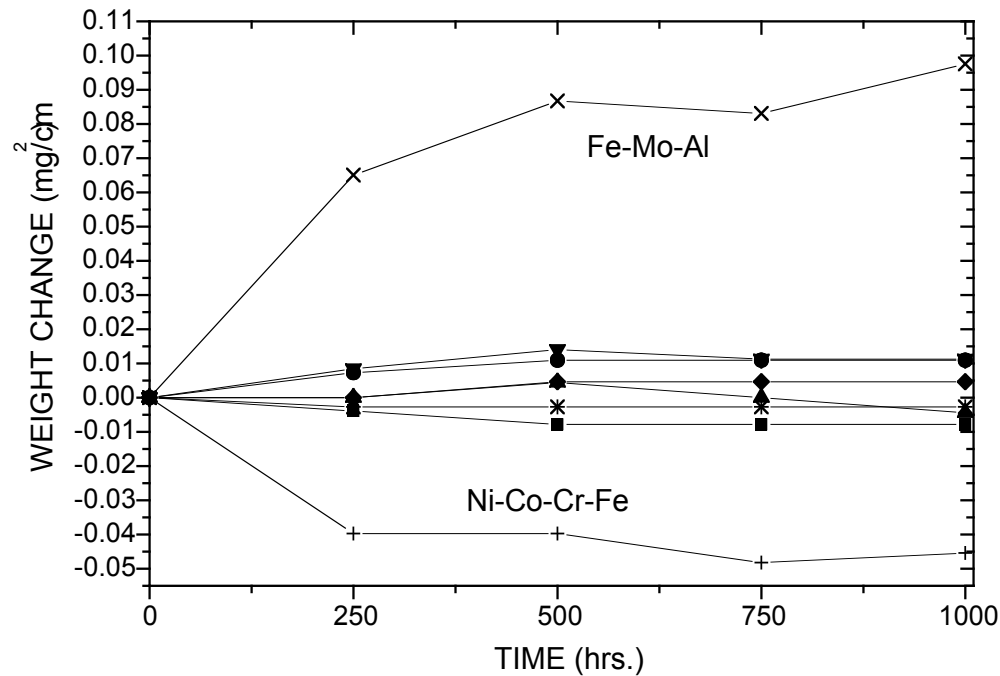
**IGCC Atmosphere @600°C = N<sub>2</sub>-24%CO-5%CO<sub>2</sub>-1.3%CH<sub>4</sub>-20ppm H<sub>2</sub>S**



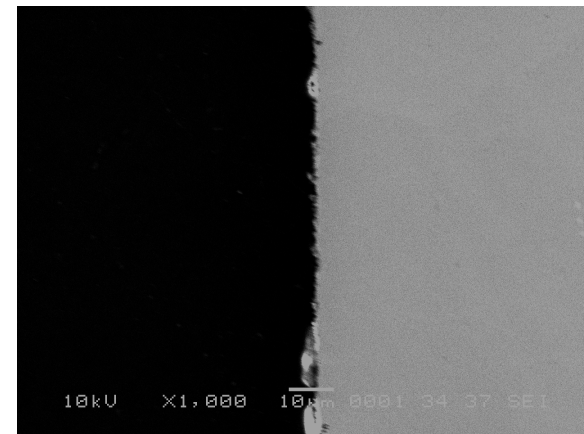
# Initial Down-Selection of IGCC Alloys



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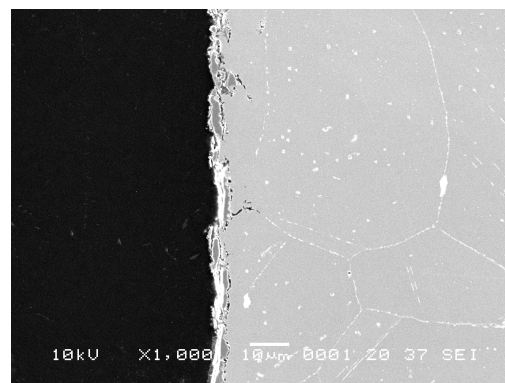
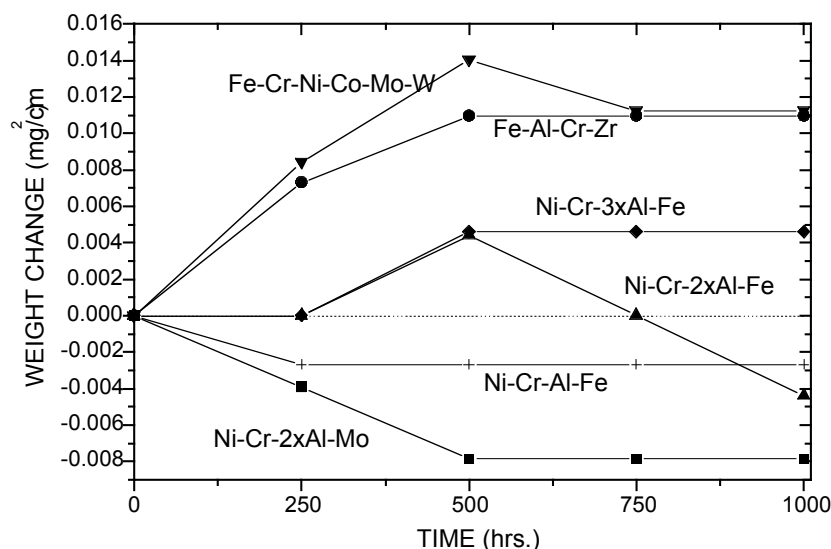
Fe-Mo-Al



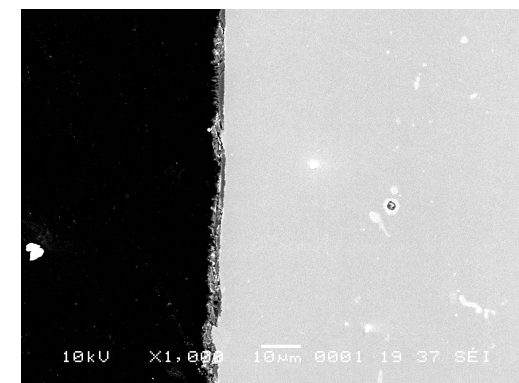
Ni-Co-Cr-Fe



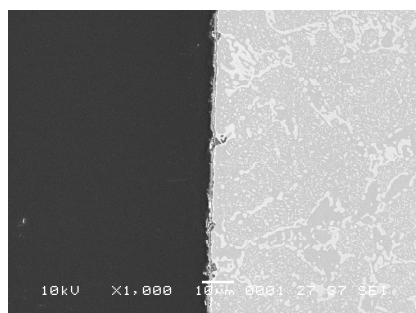
# Cross-Section Micrographs of Alloy Surface Layers after 1000h Exposure to IGCC Atm.



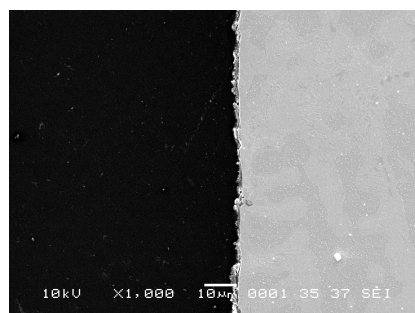
Fe-Cr-Ni-Co-Mo-W



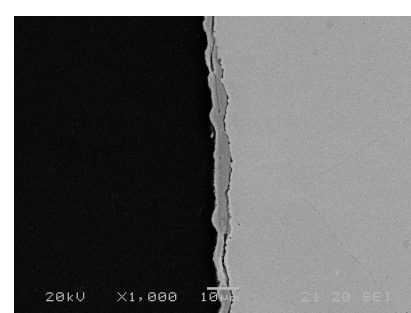
Fe-Al-Cr-Zr ( $\text{Fe}_3\text{Al}$ )



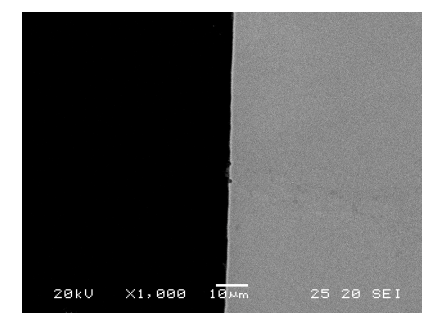
Ni-Cr-2xAl-Fe-Mo(20%)



Ni-Cr-3xAl-Fe



Ni-Cr-Al-Fe

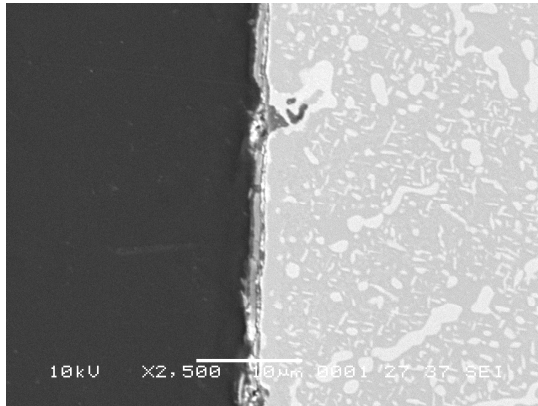


Ni-Cr-2xAl-Fe

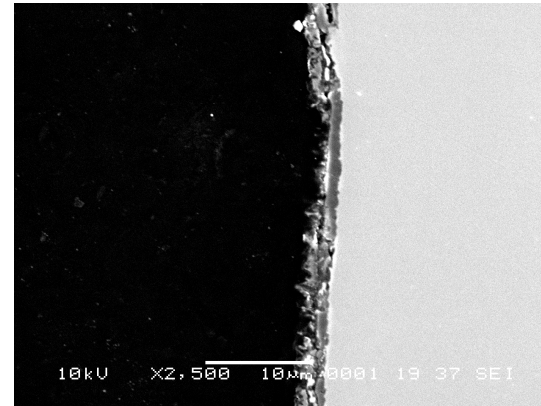
# Surface Appearance of Promising Alloys



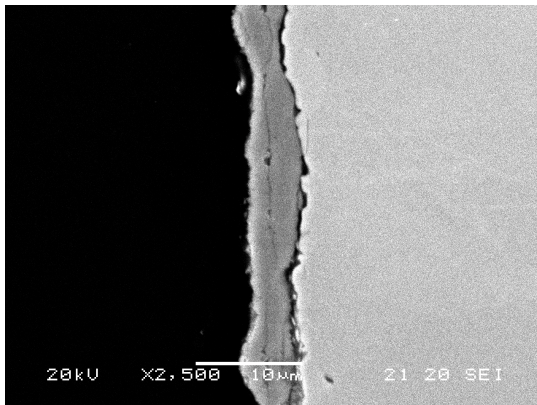
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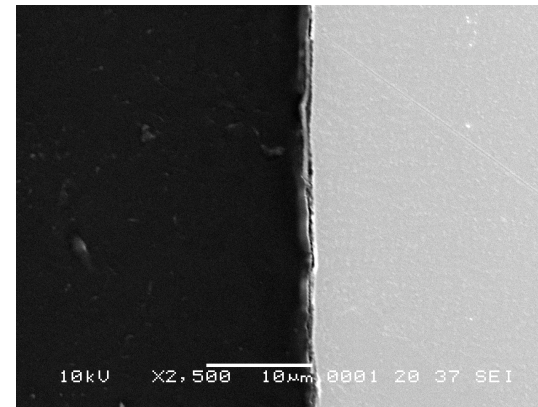
Ni-Cr-2xAl-Fe-Mo(20%)



Fe-Al-Cr-Zr ( $\text{Fe}_3\text{Al}$ )

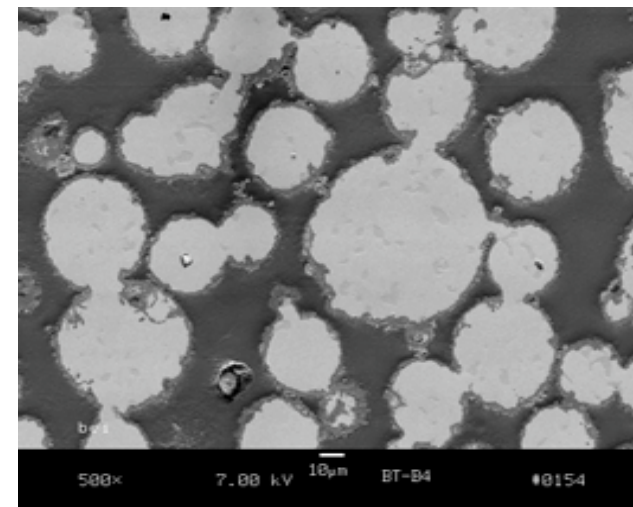
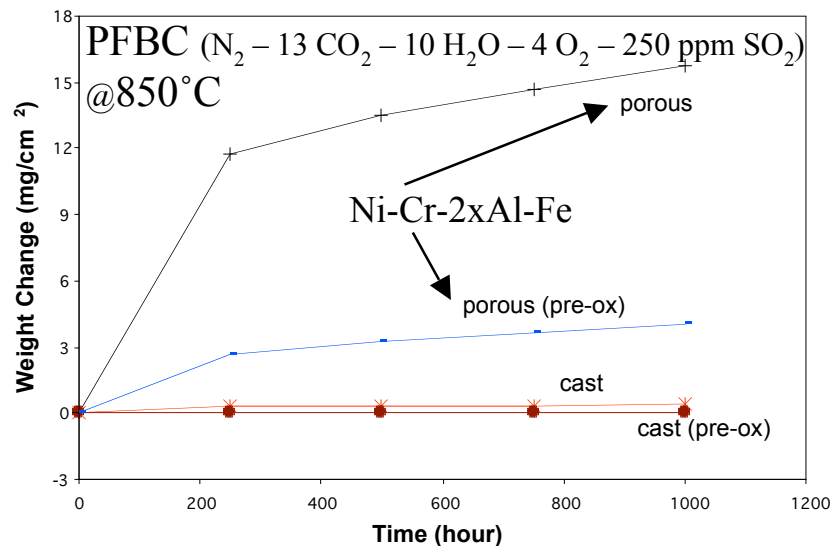
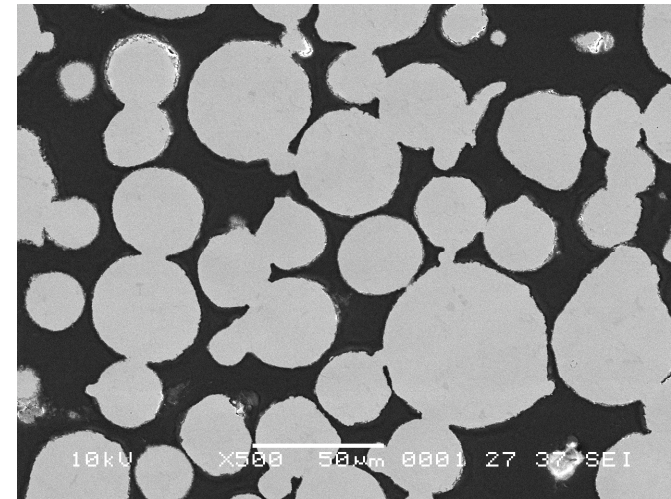
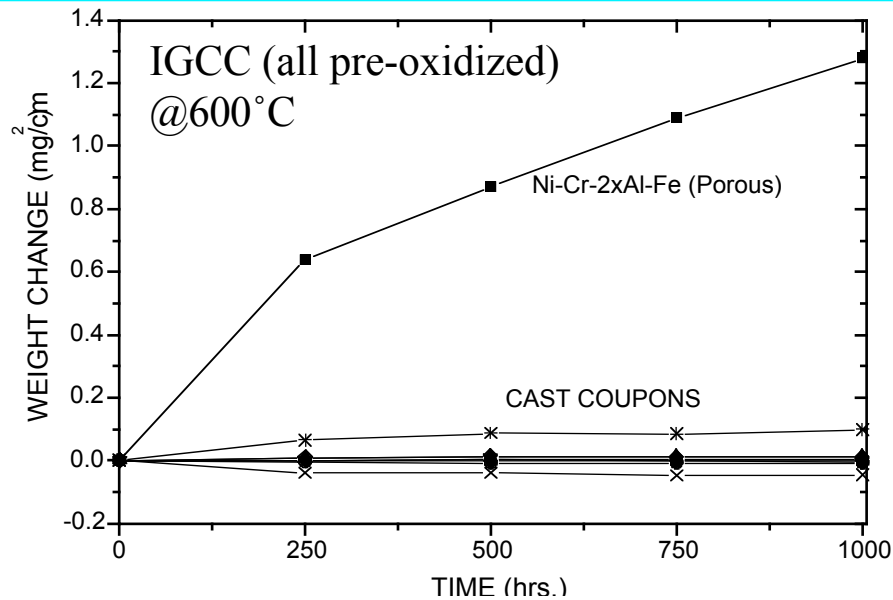


Ni-Cr-Al-Fe



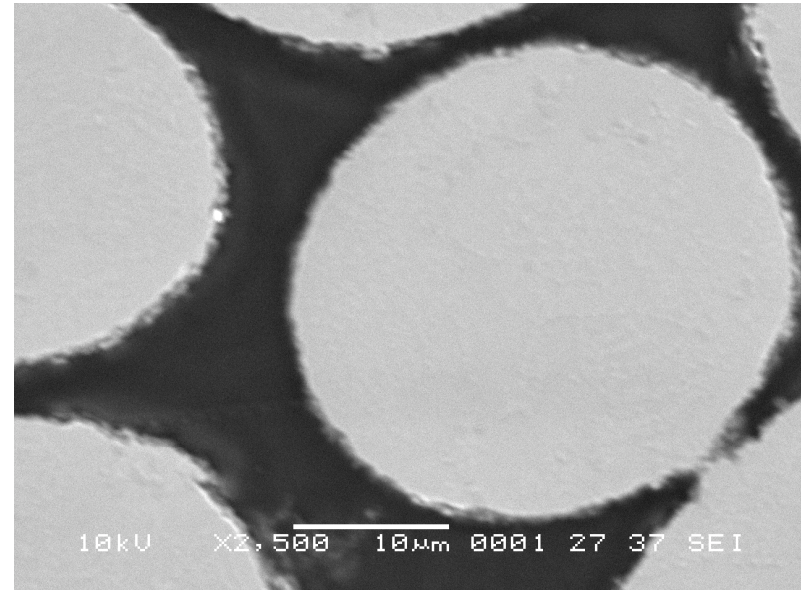
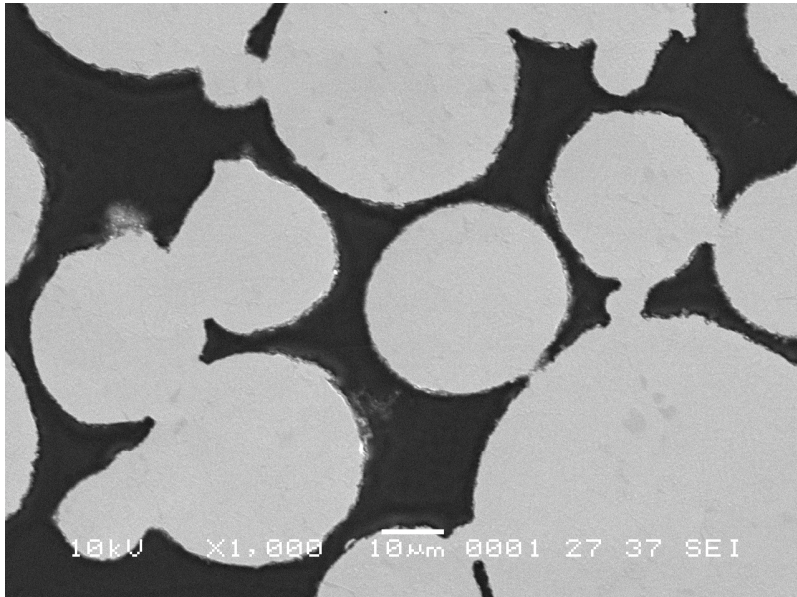
Ni-Cr-2xAl-Fe

# Comparison of Porous Sample Surfaces after Exposure to IGCC and PFBC Atm.

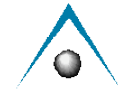




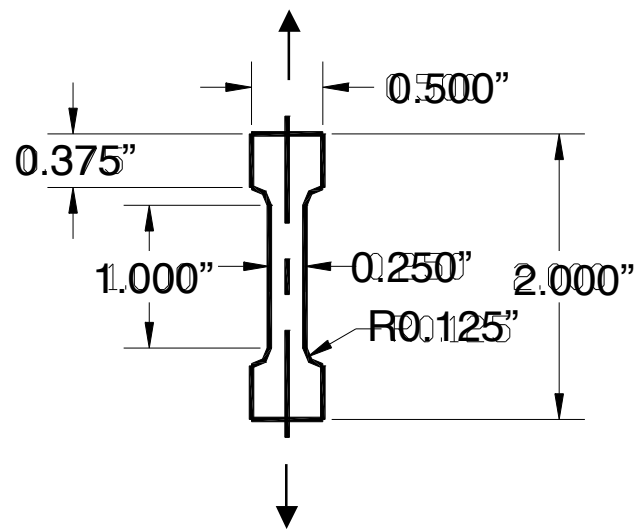
# *Cross-Sections of Porous Ni-Cr-2xAl-Fe Alloy Sheet after 1000h in IGCC Atm.*



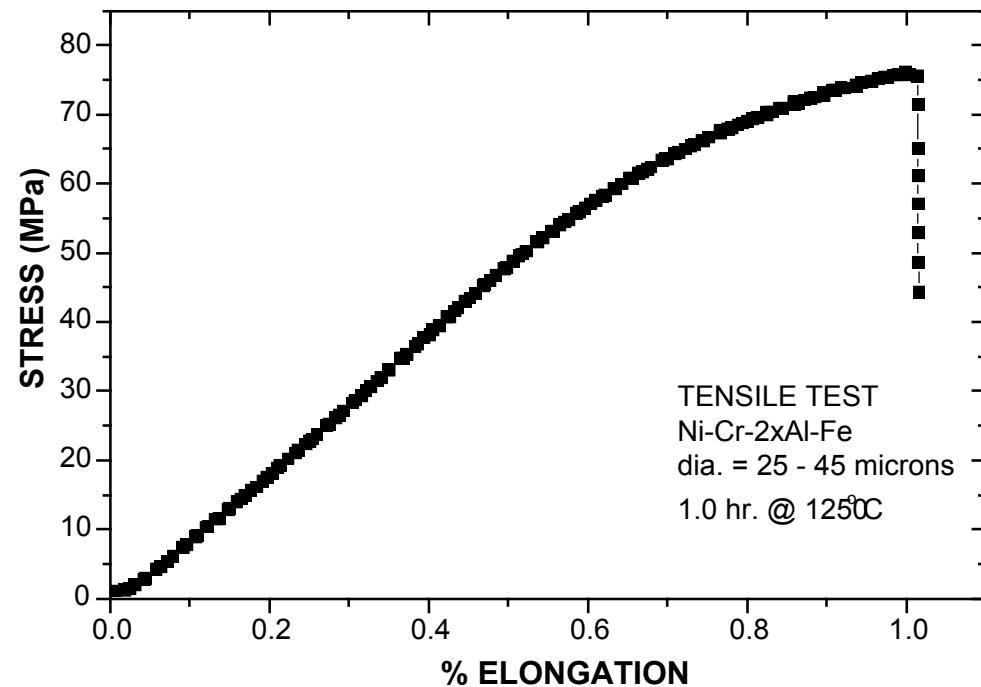
# Schematic of Tensile Sample and Example of Tensile Test Result



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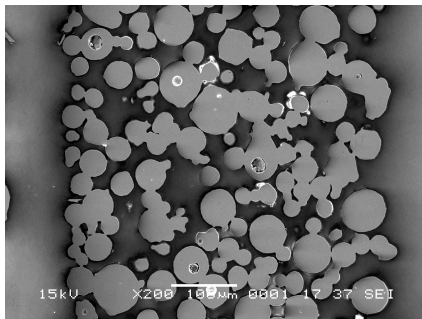
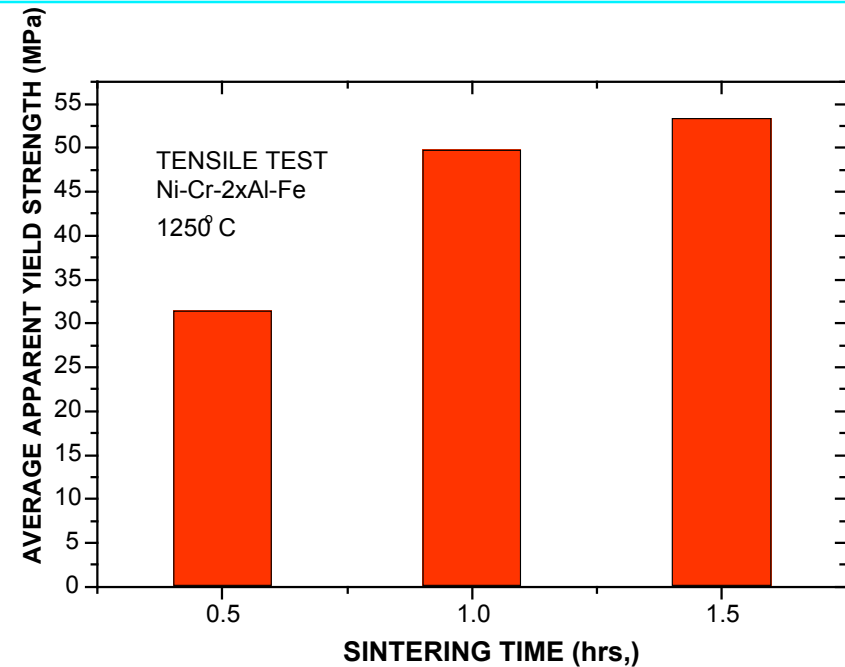
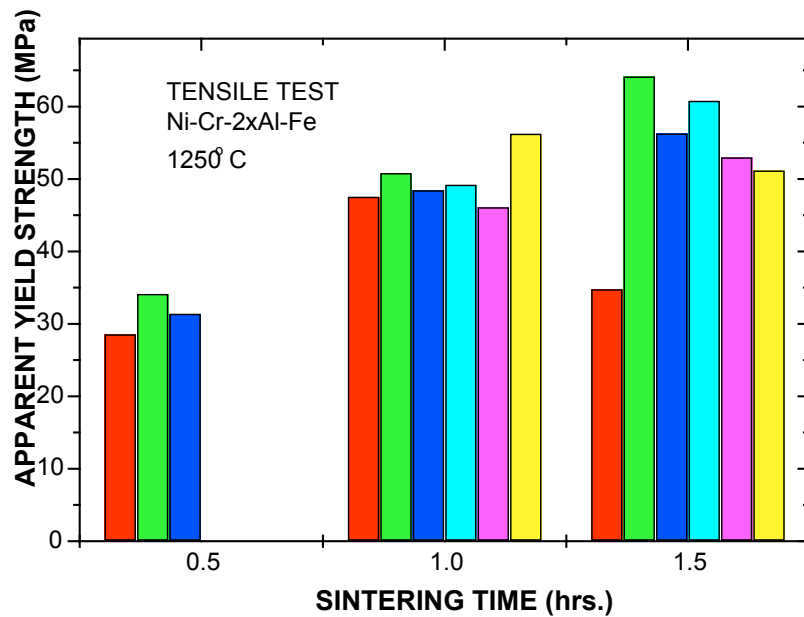


thickness = 0.020" (0.5mm)

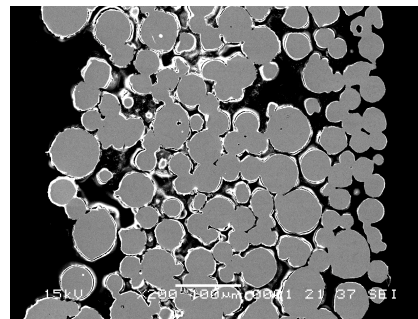




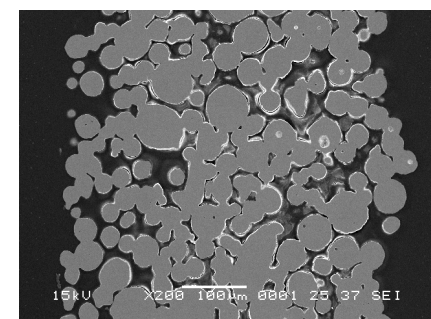
# Effect of Sintering Time on Tensile Strength of Ni-Cr-2xAl-Fe Porous Sheet



0.5 hrs.

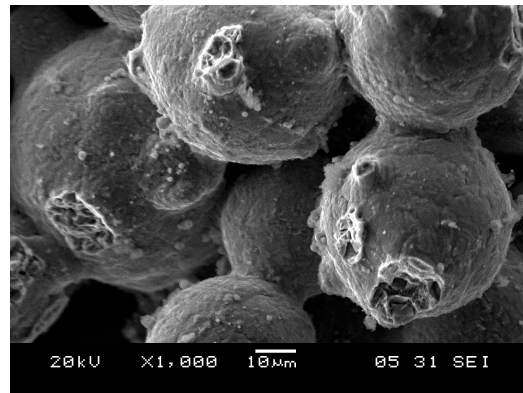
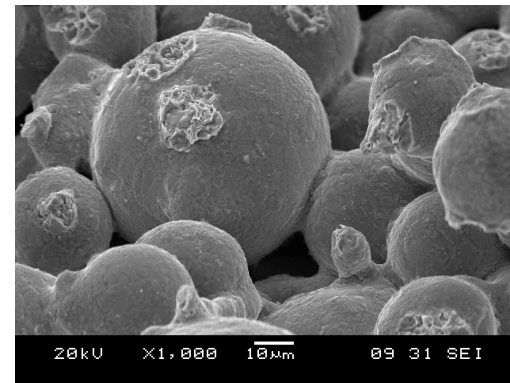
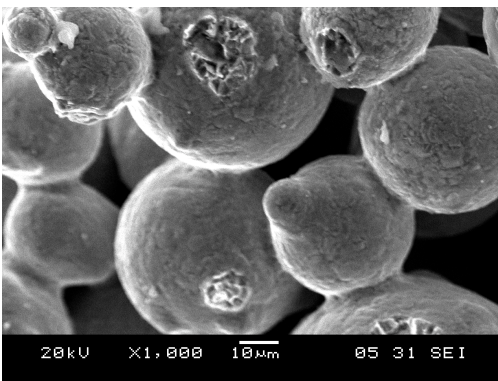
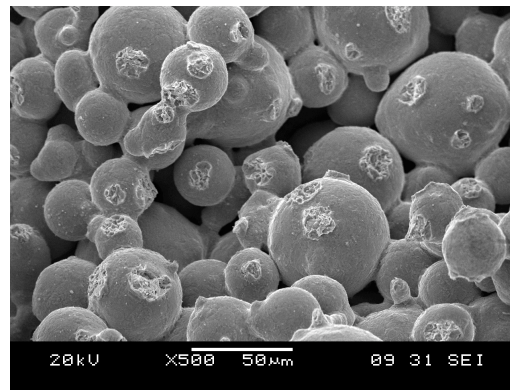
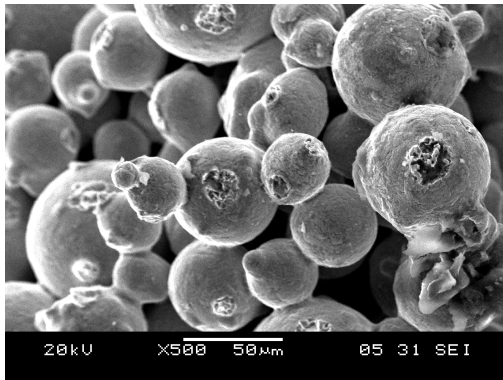


1.0 hrs.



1.5 hrs.

# *SEM Fracture Surface Examination, Comparison of Sintering Times*

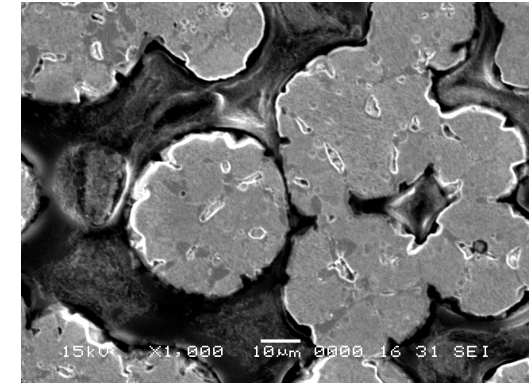
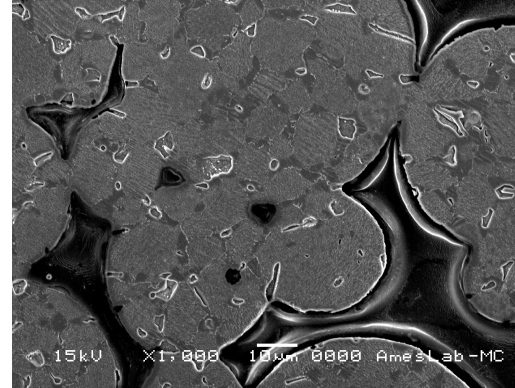
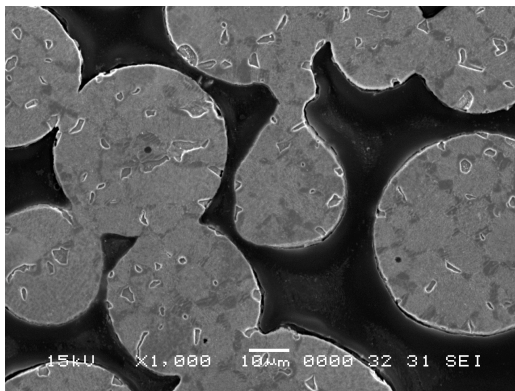
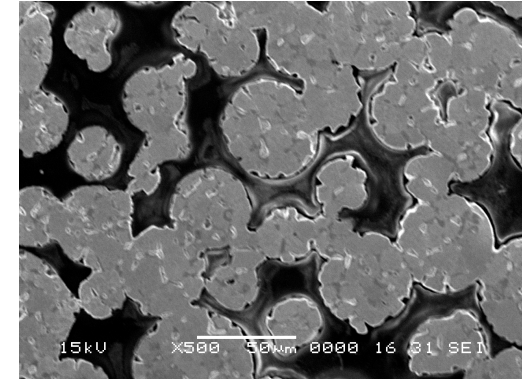
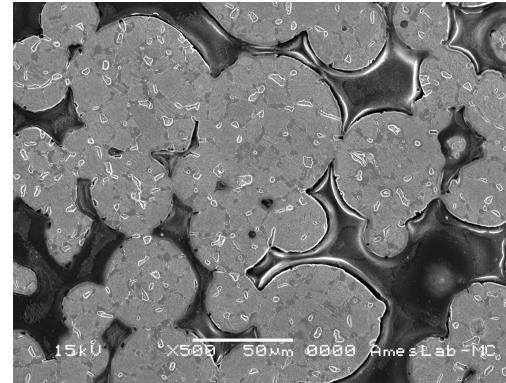
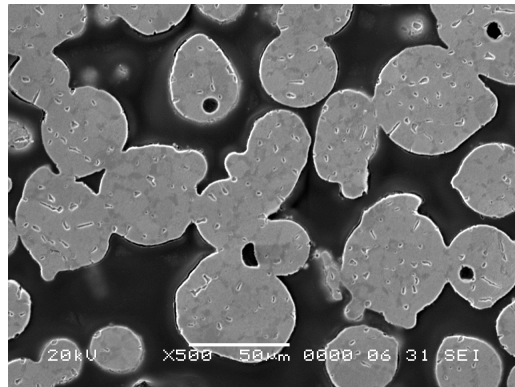


0.5 hr.

1.0 hr.

1.5 hr.

# *Etched Cross-Section SEM Micrographs of Sintered Tensile Samples*



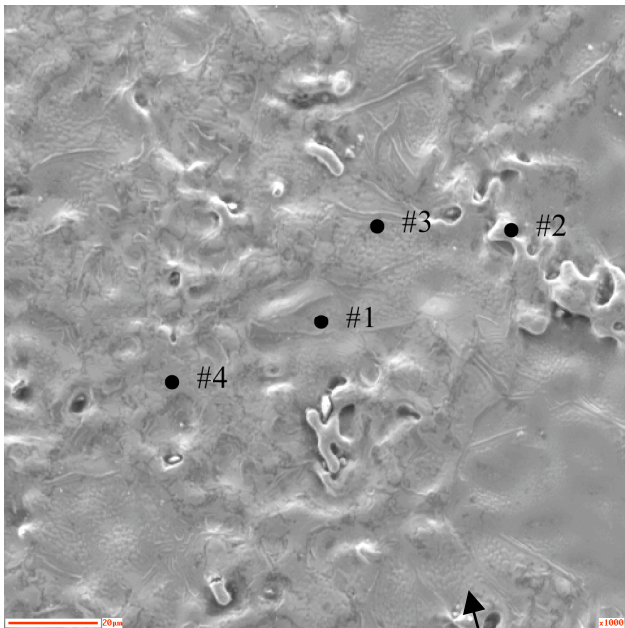
0.5 hr.

1.0 hr.

1.5 hr.

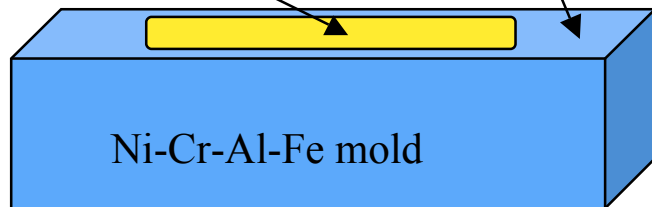


# Auger Depth Profiling Identified Problem with Alternative Mold Concept

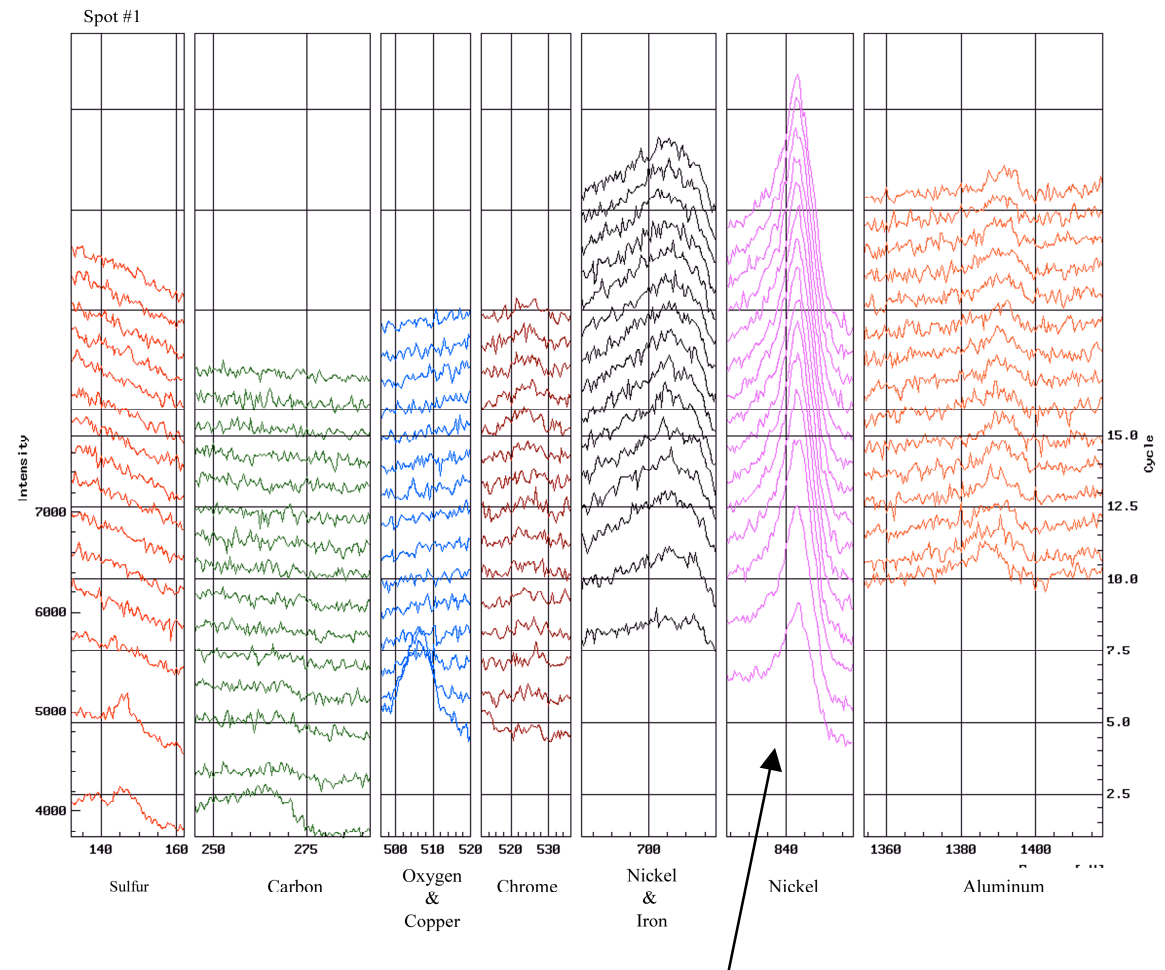


Pre-oxidized mold surface,  
after vacuum sintering.

Mold cavity, 0.5mm deep.

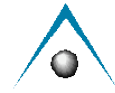


Ni-Cr-Al-Fe mold



Ni-enriched mold surface promotes porous  
sheet sticking after vacuum sintering.

# Conclusions



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- Results of IGCC corrosion tests indicated that Ni-Cr-2xAl-Fe alloy has most desirable surface film formation, i.e., excellent passivation behavior. Also, the same alloy had the best behavior in a previous series of PFBC tests.
- Three other alloys Ni-Cr-Al-Fe, Ni-Cr-2xAl-Fe-Mo(20%), Fe<sub>3</sub>Al, showed promise for IGCC applications, based on IGCC corrosion tests.
- Tensile testing of thin porous sheet samples is helpful for assessment of suitable sintering conditions, along with permeability testing.
- Microstructural analysis of tensile samples suggested preferred combination of grain growth and second phase coarsening for optimum sintered condition, which will help in rapid development of full scale manufacturing process for filter sheets.
- Additional development of sintering mold material is needed before large porous sheets can be processed.

# *Future Work*



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- Extend exploration of surface reactions in selected alloys during lower temperature oxidation/sulfidizing (IGCC) conditions, including response to down-time corrosion, i.e., in presence of HCl.
- Expand study of mechanical (tensile and bend strength) properties of porous sheet materials to include HT tensile testing.
- Initiate analysis of long term PFBC and IGCC trials (e.g., at EERC) and expand scope of testing to include through-flow conditions.
- Continue work on filter fabrication techniques including mold design, sintering, porous sheet forming, and welding and, hopefully, fabrication of full scale candle filter elements.

# Full Scale Porous Sheet Processing Concept for Metallic Hot Gas Filters

## Conceptual Manufacturing Process for Metal Candle Filters

