Qualification of Micronized Kyanite and Silica Fume

William L. Headrick
Missouri Refractories Co., Inc.
Formerly University of Missouri-Rolla
Co-authors
Dilip Jain, Kyanite Mining Corp.
Musa Karakus, University of Missouri-Rolla

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Participants

- **West Virginia University (WVU):** Corrosion and Evaluation of Metallic Materials, Dross Buildups/Corrosion in GI/GL
- **University of Missouri-Rolla (UMR):** Selection and formulation of Refractory Materials for Molten metal application, Corrosion testing and Post-mortem analysis
- **Oak Ridge National Laboratories (ORNL):** Design, FEA Modeling, Measurement of Thermo-Physical properties
- Industry Participants: Refractory manufacturers, Specialty metal smelters, Aluminum smelters, Steel manufacturers

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- Introduction
- Samples
- Scanning Electron Microscopy
- pH
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- Trial Mixes
- Preliminary Results
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Abstract

Micronized kyanite is a new raw material available from Virginia Kyanite. It has a mean particle size less than 5 microns and thus can be used to efficiently increase the ultra-fines concentration of self-flow and vibratable castables. Especially those castables used in secondary aluminum manufacturing. The University of Missouri-Rolla has shown that kyanite is more resistant to aluminum attack than silica, mullite and bauxite through post mortems. They have also shown that use of silica fume as an ultra-fine additive and flow aid leads to accelerated reactions with molten aluminum. Characterization of micronized kyanite with a comparison to fume silica is given. It is proposed that micronized kyanite may be used as a partial or total replacement for ultra-fine silica and alumina additions.

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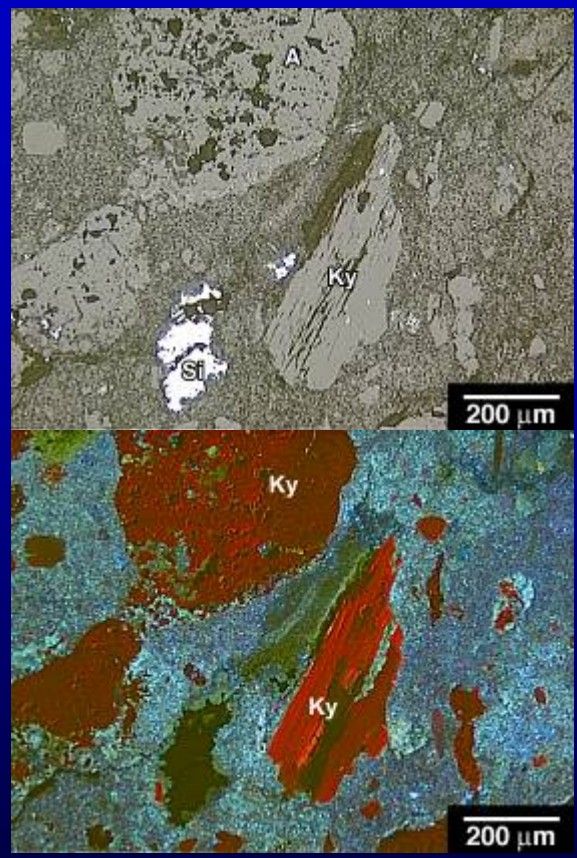
Introduction

- **Musa Karakus, UMR, found that kyanite crystals were least attacked by molten aluminum of any refractory grain.**

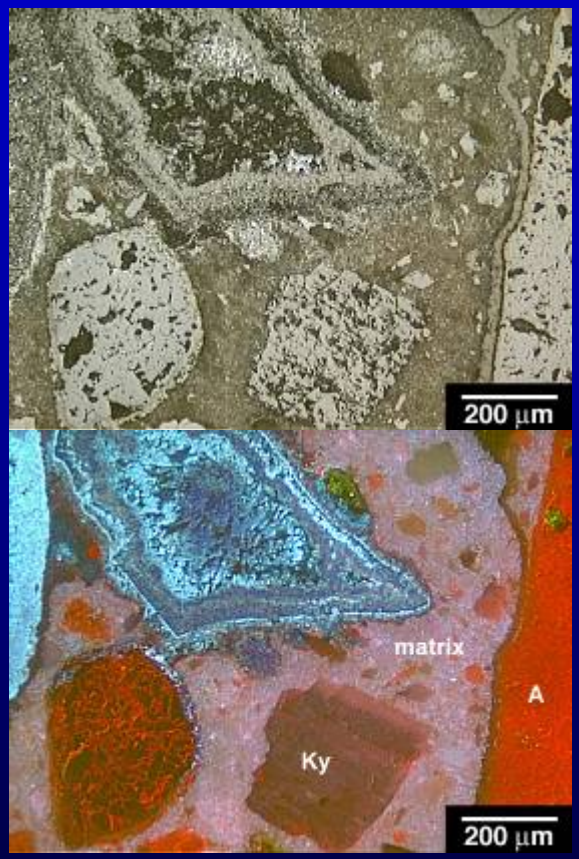
Ref. M. Karakus, W. L. Headrick, and E. Feiner, “Aluminum Melting Furnace Post-Mortem”, pp. 135-156, in Monolithics: Advances, Installation and “Boom”, 41st Symposium on Refractories, The American Ceramic Society, March 30-31st, 2005, St. Louis.



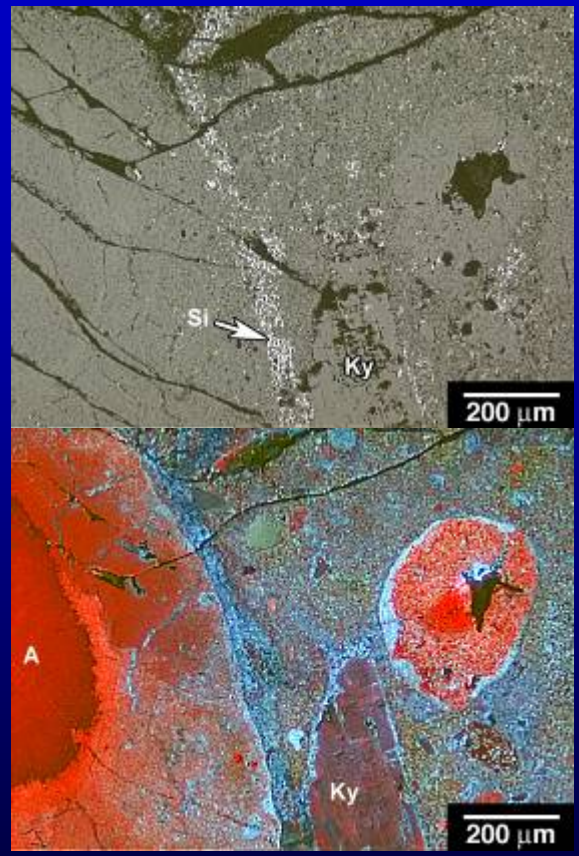
Aluminum Attack Findings

- Purity and type (sintered or single crystal) of refractory fillers play an important role for the reduction of refractory aggregates by molten aluminum.
- Silica fume is the initial path of aluminum attack.
- All bauxite derived alumina aggregates were destroyed by molten aluminum due to Fe-Ti and K-Na-rich glassy grain boundary matrix.
- Selective reduction of mullite aggregates was observed.
- **Single kyanite grains are observed to be unaffected by molten metal penetration into castable.**

Destruction of mullite and alumina



mullite



alumina

Development of castables using micronized kyanite

1. Prepare castables by partial replacement of fumed silica with micronized kyanite such that flow is maintained.
2. Replace bauxite, tabular, flint clay, or mullite fines with kyanite.

Samples

- Micronized Kyanite, Kyanite Mining
- ES 900 W Lot# 217, Elkem
- 965 Fume Silica Controlled Density, Elkem
- T-500 Silica Fume Lot# FZ6A23-1,
- 8010 Globe Silica Fume Regular, Globe Metallurgical
- D1000 Amorphous Silica Fume, Technical Silica Company

pH

Kyanite	**6.5**
ES 900 W	**6.5**
965	**7.5**
T-500	**5**
8010	**11.5**
D 1000	**5**

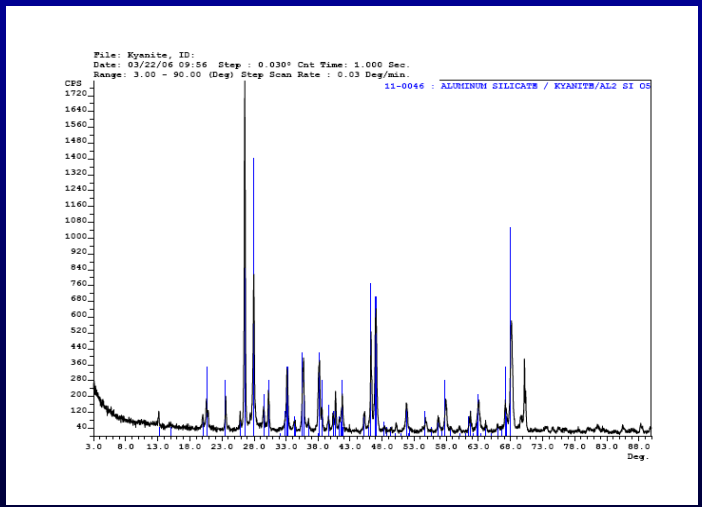
Powders rapidly settled out of water after agitation with the exception of 8010.

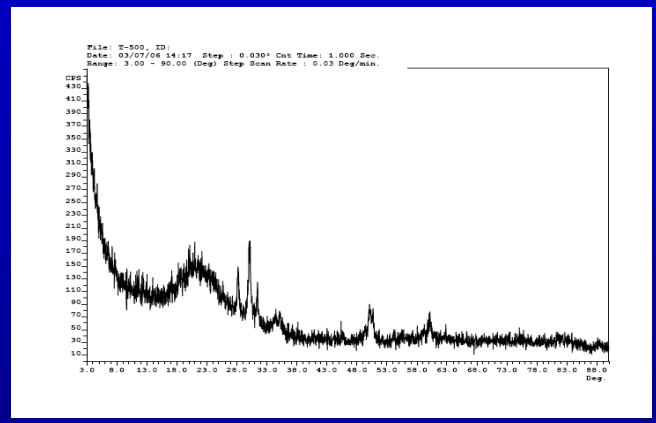
8010 remains suspended in water for days.

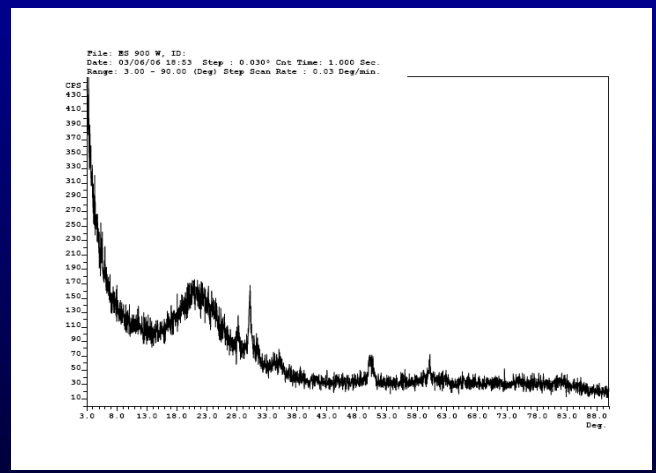
Mix 2 grams powder with 20 ml water. Wait 3 minutes then measure pH.

XRD of Micronized Kyanite and Fume Silica

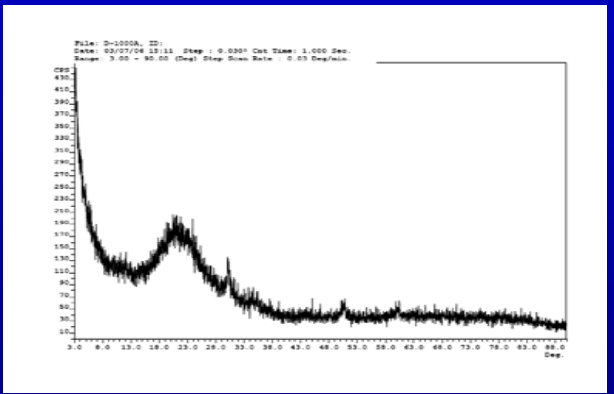
- **Micronized Kyanite contains almost no amorphous phase.**

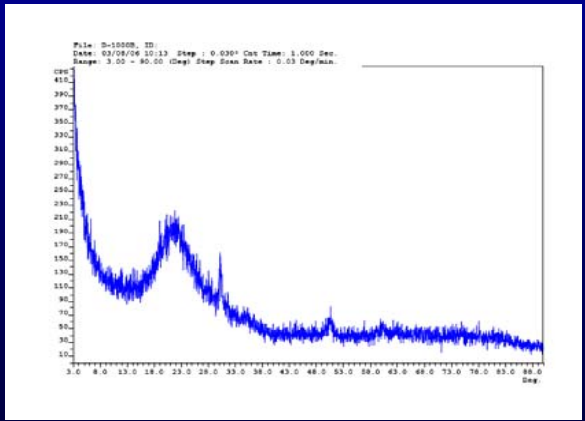


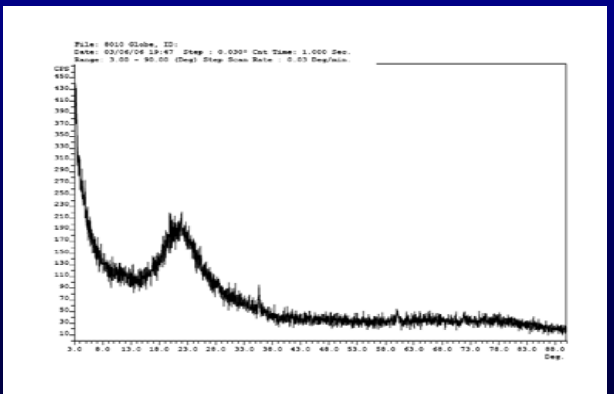


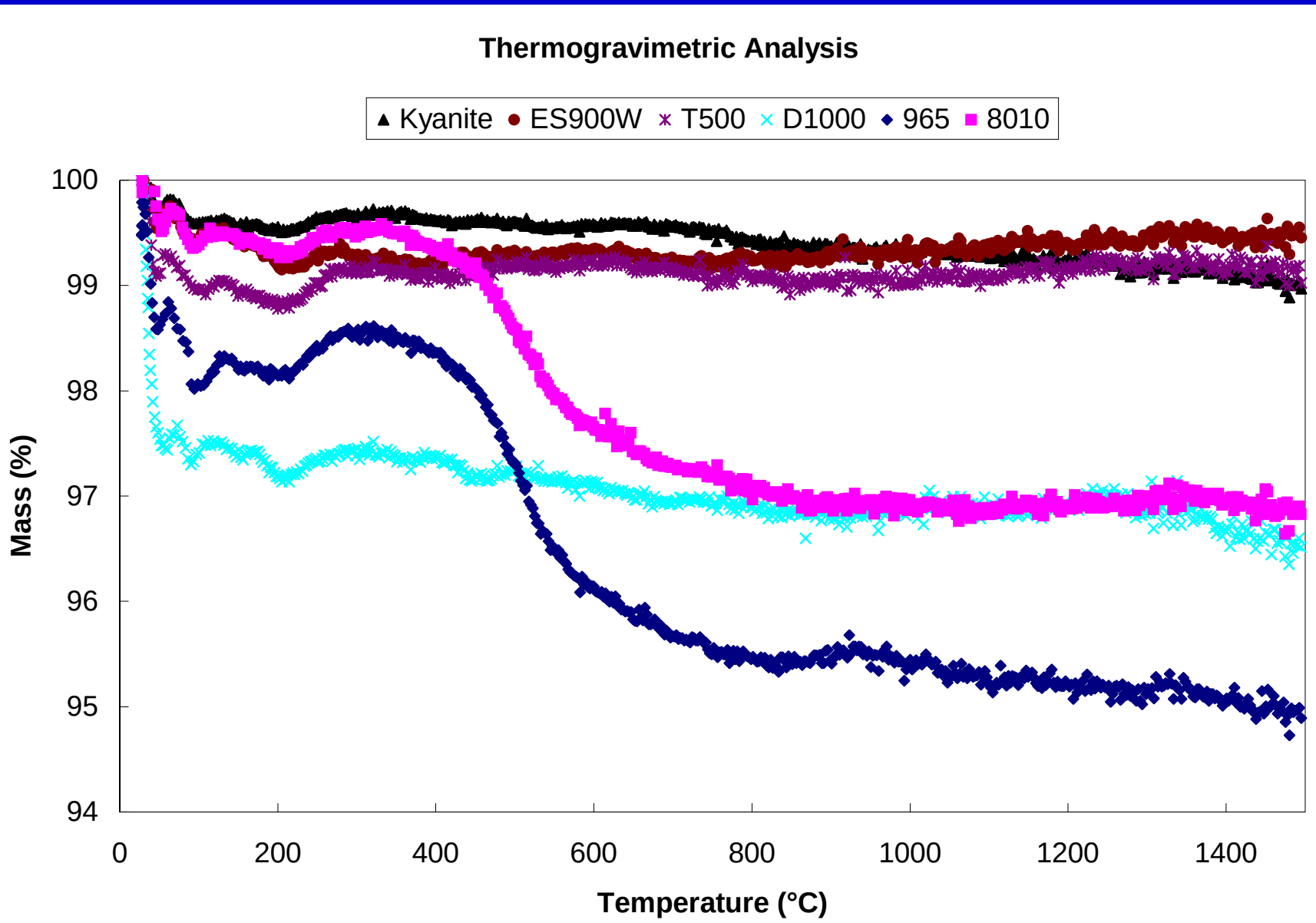


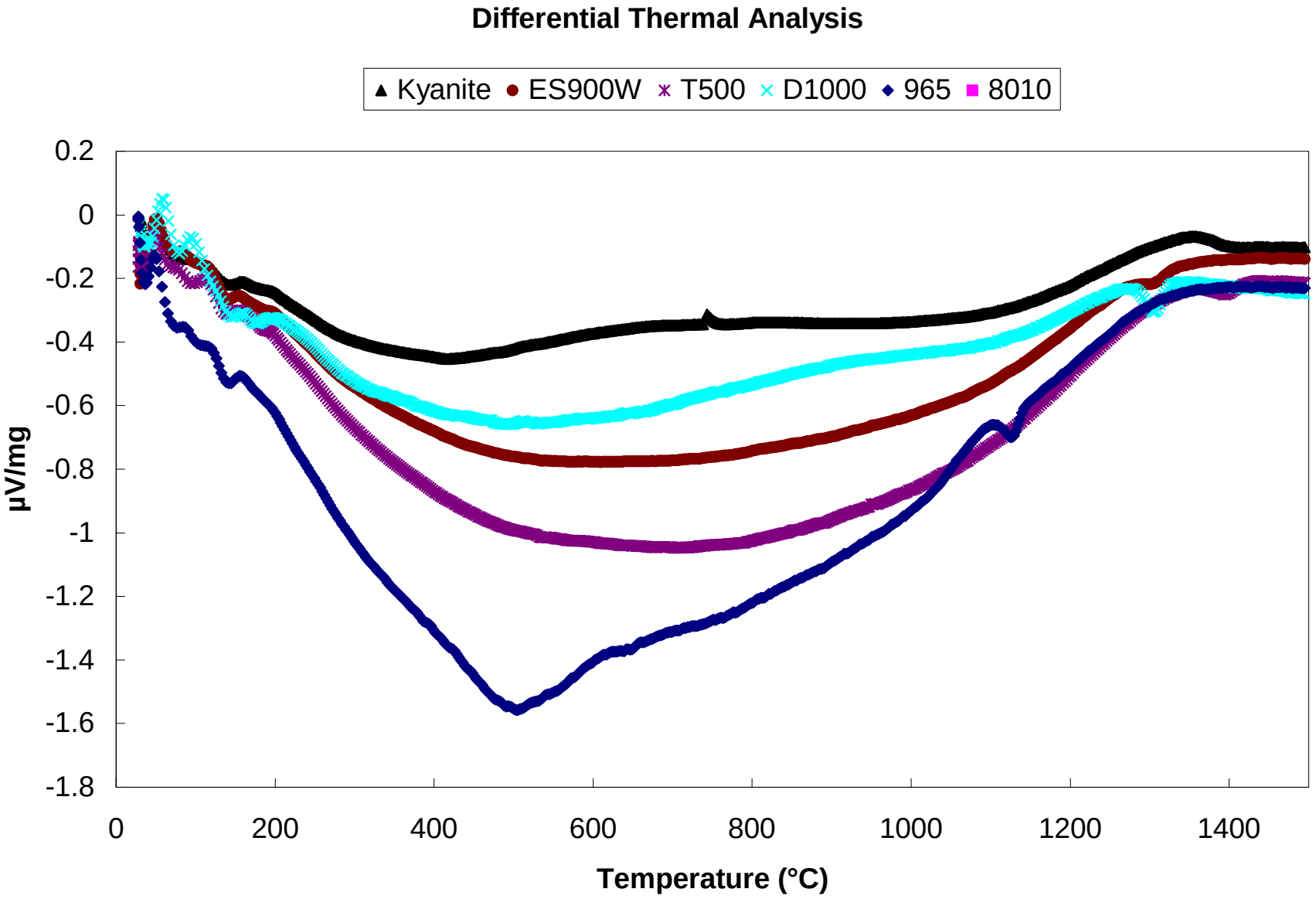
XRD-Silica Fume



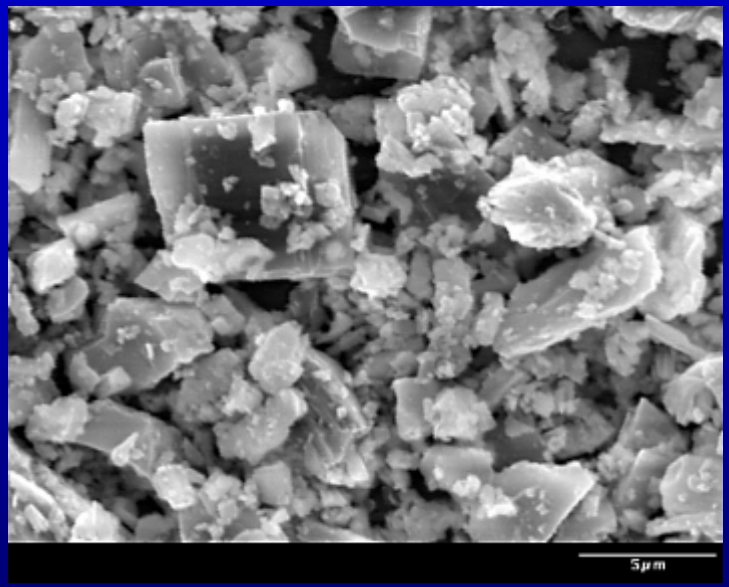




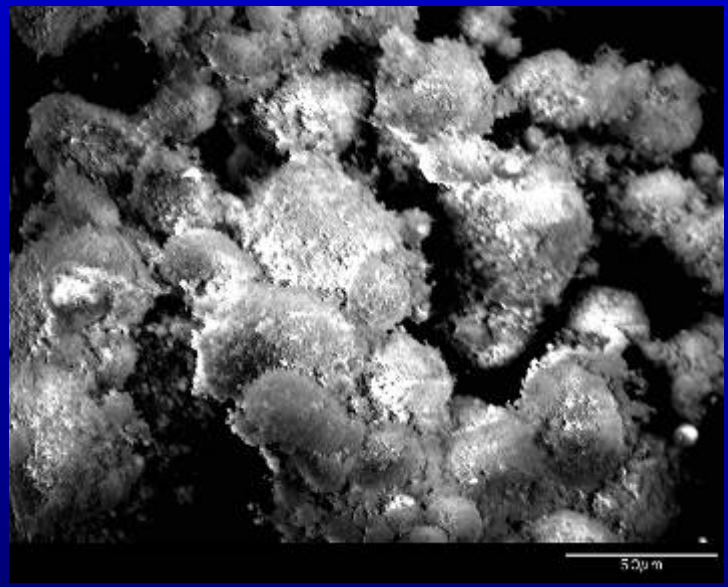

Thermogravimetric Analysis
Kyanite
ES900W
T500
D1000
965
8010
100
99
98
97
96
95
94
Mass (%)
0
200
400
600
800
1000
1200
1400
Temperature (°C)


Differential Thermal Analysis
Kyanite
ES900W
T500
D1000
965
8010
0.2
0
-0.2
-0.4
-0.6
-0.8
-1
-1.2
-1.4
-1.6
-1.8
µV/mg
0
200
400
600
800
1000
1200
1400
Temperature (°C)

Scanning Electron Microscopy



Kyanite



Fume Silica (10X)

Particle Size Distribution

- **Micronized Kyanite**
  - **Mean Agglomerate Size 5 micron**
  - **Mean Particle Size 5 micron**
- **Fumed Silica**
  - **Mean Agglomerate Size 50 micron**
  - **Mean Particle Size 0.1 micron**

Trial Mixes

	Base Mix with Silica Fume	Mix with 4.5 Micron Kyanite	Mix With 4.5 micron & coarser kyanite
M60 3X4	18	18	18
M60 4X8	18	18	18
M60 8X20	13	13	13
M60 20M	18	18	0
K35m	0	0	18
K325M	0	0	13.7
Mullite 325M	8.2	8.2	0
Kyanite 48m	5.5	5.5	0
S.F 971	5.5	2	2
C90 LSB	8	5.5	5.5
Micro Kyanite	0	6	6
Secar 71	6	6	6
SMP	0.1	0	0.2
Additive A	0.1	0	0.2
Darvan 811D	0	0.4	0

Chemistry

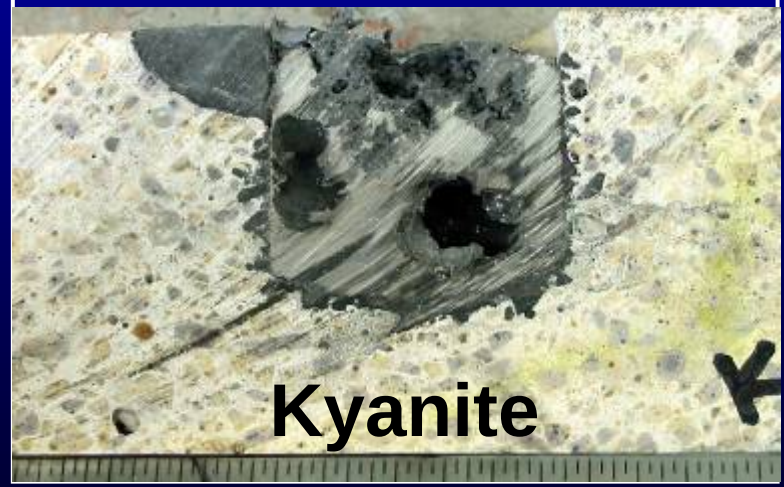
	Base Mix with Silica Fume	Mix with 4.5 Micron Kyanite	Mix With 4.5 micron & coarser kyanite
Al_2O_3	60.2	59.4	60
SiO_2	34.8	35.8	35.3
Fe_2O_3	0.9	0.86	0.87
TiO_2	2.39	2.34	2.07
CaO	1.52	1.32	1.47
MgO	0.03	0.03	0.03
Na_2O	0.01	0.01	0.01
K_2O	0.05	0.07	0.03
P_2O_5	0.13	0.2	0.26

Properties

1090°C Properties		Base Mix with Silica Fume	Mix with 4.5 Micron Kyanite	Mix With 4.5 micron & coarser kyanite
Bulk Density	g/cc	2.45	2.5	2.58
Apparent Porosity	%	14.5	15.4	16.2
CCS	MPa	76	86	55
MOR	Mpa	3.1	3.4	1.4
PLC	%	-0.17	0	0

Cup Test Results

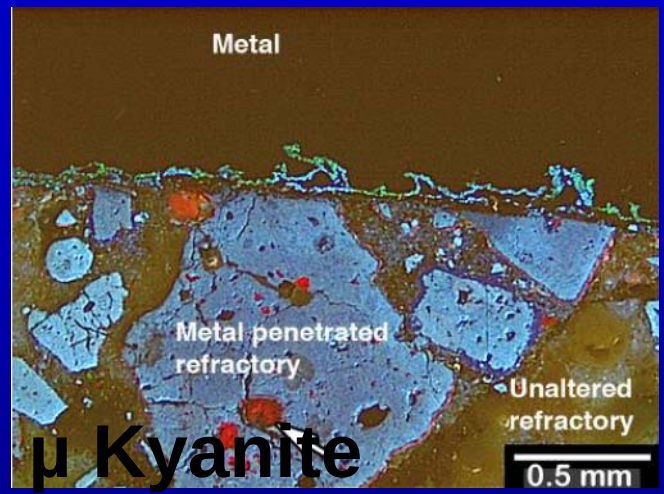
- **Test parameters:**
- **Duration: 240 hours (10 days) at 1000°C**
- **Pre-oxidized to 1200°C for 5 hours**
- **Steam atmosphere maintained during test.**
- **Tested for penetration resistance to molten Al alloy 5083**

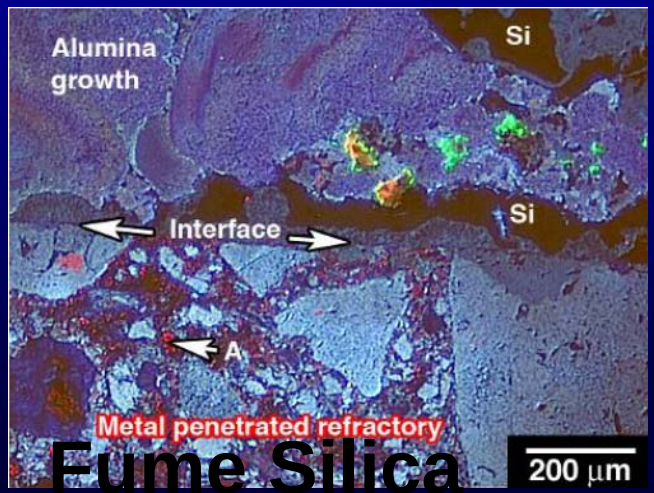


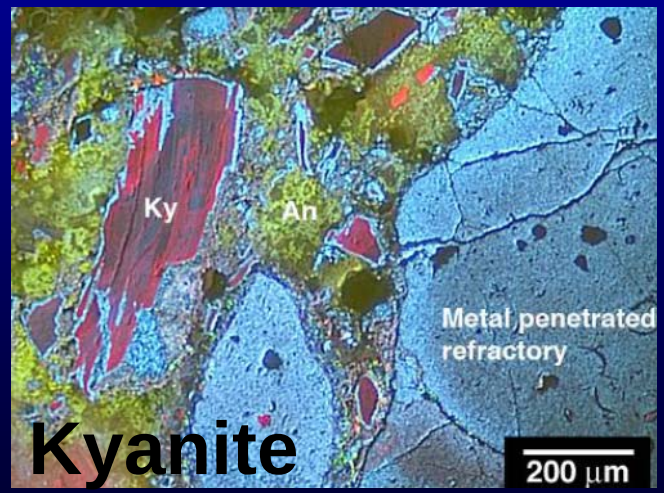
Cup Test Results

Order of attack

1. **Mullite**
2. **Matrix**
3. **Kyanite (grain boundary attack)**
4. **Micronized kyanite**







Conclusion

- **Micronized Kyanite can be used as a partial replacement for fume silica.**
- **Flow is lower with micronized kyanite.**
- **Corrosion resistance against aluminum metal is greatly increased by using kyanite versus Mulcoa 60 fines.**

DISCLAIMER

Funded by

- **DOE Award No. DE-FC36-04GO14038**
- **Kyanite Mining Corp.**
- **Missouri Refractories Co., Inc.**