RESPONDING TO THE REFRACTORY INDUSTRY'S NEED FOR FULLY GROUND MATRIX ALUMINAS

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ABSTRACT

In recent years as the refractory industry has worked to develop monolithic refractories for ever more critical applications, the need for fully ground matrix aluminas has expanded. For many years, very low soda, premium grade fully ground aluminas such as Alcoa's A-16SG and A-17 have been available. They have performed exceptionally where their cost could be justified. A number of years ago, A-3000FL was developed as an alternative to A-17 and its use has grown dramatically. In recent years a series of multi-modal particle size fully ground aluminas, some containing magnesia-alumina spinel, were developed.

This paper reviews the various superground aluminas, spinels, and Tabular alumina that are available and introduces a new refractory grade matrix alumina. The new alumina is becoming available as a result of the new high efficiency batch ball milling facility completed by Alcoa in early 2001 in Leetsdale, PA, USA. The particle size of the new fully ground matrix alumina allows the knowledgeable refractory designer to provide the rheology required for today's specialty installation methods such as low moisture, self-leveling and pumpable systems when used in combination with Microsilica. Data is presented demonstrating effectiveness over a broad range of service temperatures when combined with mullite or Tabular alumina aggregates in low cement castables.

INTRODUCTION

The need for fully ground matrix aluminas has grown with the development of low cement, ultra low, and no cement castables. These aluminas are used in achieving the specialized particle size distributions that are required to achieve the desired physical properties and placement rheologies. Many papers and articles have been published[1, 2, 3] documenting the need for controlled particle size distribution (PSD) in both the refractory aggregate materials (down to ~75 microns/200 mesh) and the fine refractory matrix materials. It is by controlling the intricacies of the particle size distribution and wise selection of additives that the various placement rheologies are achieved. Otherwise high mixing water may be required, the mix will not be pumpable, and may suffer from inadequate physical properties and water or aggregate separation after casting.

Refractory designers have seen the benefits in water reduction and ceramic sintering of appropriate size fully ground calcined aluminas and ground Tabular aluminas as compared to –325 mesh ground calcined aluminas. The partially ground –325 mesh calcined aluminas consist primarily of partially sintered agglomerates of individual alumina crystals. The agglomerates have significant open porosity that soaks up water during mixing and casting. During firing, the porosity limits crystal growth at ceramic sintering temperatures.

The highest performance refractory systems (for hot strength, creep resistance, slag corrosion resistance, etc.) have been based on Tabular alumina and magnesium-aluminate spinel and are Microsilica free. Addition of Microsilica to these high purity refractory systems can improve intermediate temperature strength (to 1,000+oC) but low viscosity glasses begin to form in sufficient quantity to cause softening by 1250oC. However since there are still many refractory applications that do not require the >1500oC performance of the highest purity systems, there are thousands of tons of Microsilica containing monolithic refractories consumed annually.

HISTORICALLY AVAILABLE FULLY GROUND MATRIX ALUMINAS

Refractory designers have had a range of particle size fully ground aluminas for a number of years. Those that have been available the longest were developed to meet the particle sizing, chemical purity, and controlled ceramic sintering requirements of the technical ceramics and electronic ceramics industries. Examples of these are Alcoa's premium Reactive Aluminas A-16SG, A-15SG, and A-17. Fully ground aluminas of this time period were at or below 0.10% Na_2O.

The next group to be developed was entirely satisfactory for all but the most demanding applications. The lower cost of these products and their consistent, excellent performance caused them to be utilized in many refractory applications. Examples of these are Alcoa's Realox Aluminas A-1000SG, A-152SG, A-3000FL, and A-3500SG. A recent addition to this group was RG100.

During the period of time that this series of fully ground calcined aluminas were being applied, refractory designers also started to apply ground Tabular aluminas much more extensively. The ground Tabular aluminas filled the portions of the particle size distribution above 5 microns with very thermally stable, non-porous particles. Examples of these are Alcoa's T-64 –20 micron, -325 mesh, and –100 mesh.

The most recently developed fully ground matrix aluminas are the extremely broad particle size, multi-modal, high purity aluminas. Their particle size has been optimized over a wide particle size range to minimize the number of matrix components needed(4). Their use minimizes the formulation design time and effort necessary to achieve extremely good physical and rheological properties. Magnesium aluminate spinel is utilized in some of the multi-modal matrix aluminas to provide both enhanced high temperature (>1200oC) strength and resistance to corrosive steel slags(5,6,7). Examples of these are Alcoa's CTC-30, CTC-50, and CTC-55 (contains spinel).

A summary description of these materials is provided in Table I. A series of plots is also included in Figures 1 and 2 to provide typical particle size distributions for them.

Table I. Examples of Fully Ground Matrix Aluminas - Typical Data

Product	PSD Type	Top Size*	d90*	d50*	d10*	% Na_2O
A-1000SG	Mono-Modal	7.7	1.6	0.49	0.2	<0.10
RG-100	Mono-Modal	31	7.5	0.7	0.2	<0.10
A-152SG	Mono-Modal	5.5	2.7	1.2	0.5	<0.10
A-20SG	Mono-Modal	13	6.0	3.5	1.5	<0.30
A-3000FL	Bi-Modal	13	6.5	3.1	0.4	<0.10
CTC-30	Multi-Modal	13	5.0	1.34	0.3	<0.10
CTC-40	Bi-Modal	26	7.0	0.9	0.3	<0.10
CTC-50	Multi-Modal	44	17	1.8	0.3	<0.20
CTC-55	Multi-Modal	44	18	1.9	0.3	<0.10
Other Typical Ground Matrix Aluminas and Spinels						
T-64 –20 micron	Broad	52	22	5	0.7	<0.30
T-64 -325 mesh	Broad	74	35	12	1.6	<0.30
T-64 -100 mesh	Broad	350	145	60	16.5	<0.30
AR-78 0-0.020 mm	Broad	52	22	5	0.7	<0.15
AR-78 0-0.045 mm	Broad	74	36	15	1.7	<0.15
AR-78 0-0.090 mm	Broad	250	86	30	6.3	<0.15

* Microns by Microtrac X100 - laser light diffraction

Figure 1: New Fully Ground Reactive Alumina & Tabular Matrix Aluminas

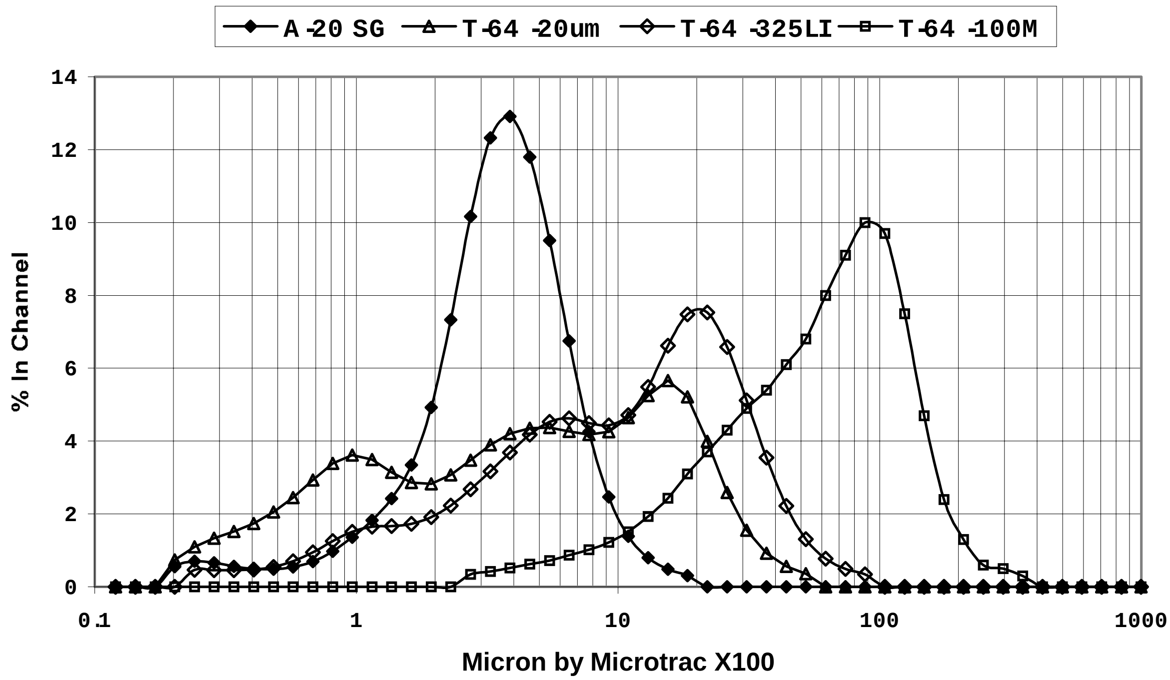
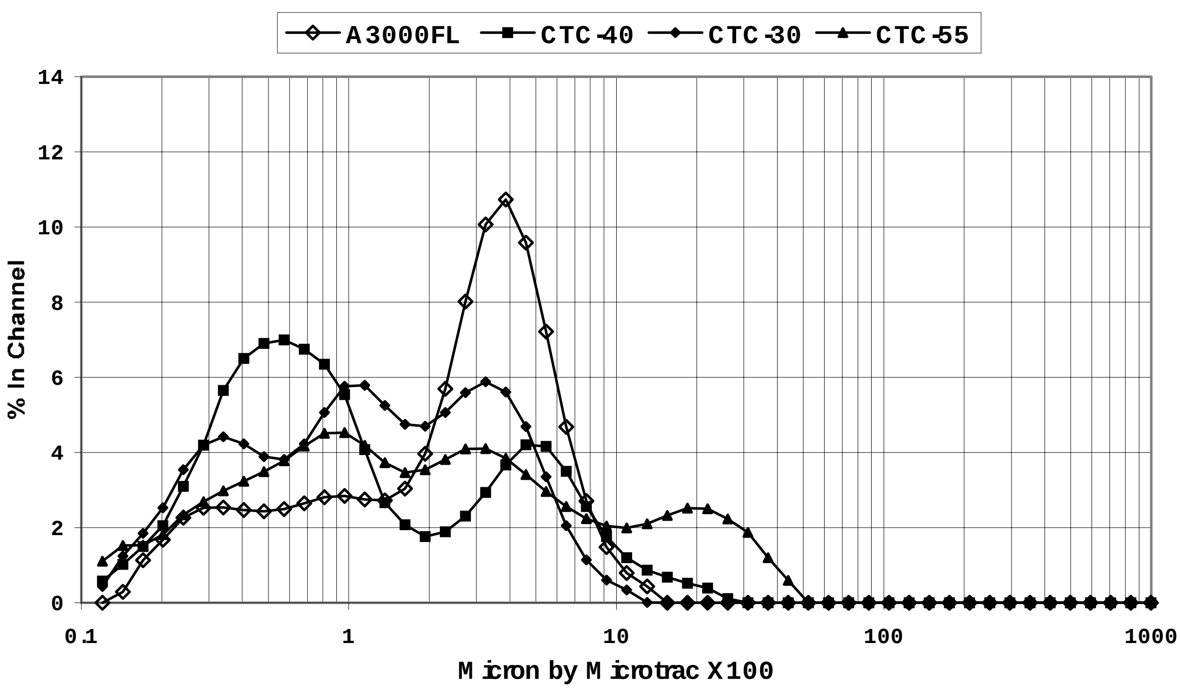


Figure 2: Typical Multi-Modal Matrix Aluminas



NEW FULLY GROUND MATRIX ALUMINA

Why another matrix alumina when the array of particle size distributions now available is very large? The primary missing product type has been a 3.5 median micron fully ground alumina designed to compliment the use of Microsilica in the refractory matrix. Until this year Alcoa World Chemicals has been limited in what products could be supplied by batch milling capacity. With the installation of additional grinding capacity at Leetsdale, PA, this limitation has been eliminated. The first new product to be offered is A-20SG. The chemical and physical properties for A-20SG are presented in Table II.

Table II. A-20 SG - Typical Product Data

Chemistry, %			PSD, Microtrac X100		Microns
	Al2O3	99.6		d90	6.0
	Na2O	0.25		d50	3.5
	SiO2	0.01		d10	1.5
	Fe2O3	0.01			
	CaO	0.02			
			PSD, Sedigraph 5100		Microns
Surface Area, m2/gm		1.6		d90	6.0
Powder Water Demand		17.5%		d50	3.3
Green Density		2.34		d10	1.4

A-20SG is mono-modal alumina with a low surface area and water demand. It is designed to be utilized in combination with Microsilica, low water demand ground Tabular aluminas, calcium aluminate cement (CAC) binders, or other low water demand matrix fines to achieve the overall particle size distribution required for optimal particle packing and good flow behavior. The combination of low open porosity materials and optimized particle packing can be utilized to achieve self-flow properties at <5% casting water additions and vibration flow at <4.5% water. A-20SG is an excellent choice for the mullite and calcined bauxite aggregate containing castables that typically contain Microsilica.

But why is the 3.5 micron particle size important? See Figure 3 for the particle size distributions of Elkem 971 Microsilica and a –325 mesh Tabular alumina. These products are typical of the matrix fines that have been in wide use for a number of years for low cement castables. The Microsilica is 80% < 1 micron and the –325 mesh Tabular alumina is only 25% less than 4.5 microns. To achieve low casting water requires the void space in the matrix to be minimized. With only these two materials, more Microsilica than the castable designer wants, if the matrix refractoriness is to be maintained, often will be required to fill the matrix voids. By also having A-20SG with 80% <5 microns to fill in the matrix PSD below the –325 mesh product, less Microsilica is needed to minimize the voids that increase the required casting water.

But is the A-20SG limited in application to just these simple matrix formulations? The simple addition of A-20SG does offer the ability to significantly improve the performance of the refractory matrix but its addition only may not provide the optimal performance. The addition of the broad particle size T-64 –20 micron Tabular alumina allows the refractory designer to optimize the matrix PSD even more. See Figure 4. With this combination of a –325 mesh alumina, a –20 micron alumina, a 3.5 median micron alumina, and 0.3 median micron Microsilica the ability to optimize water demand and castable flow properties is available.

Figure 3: A20 SG with Conventional Matrix Fines Materials

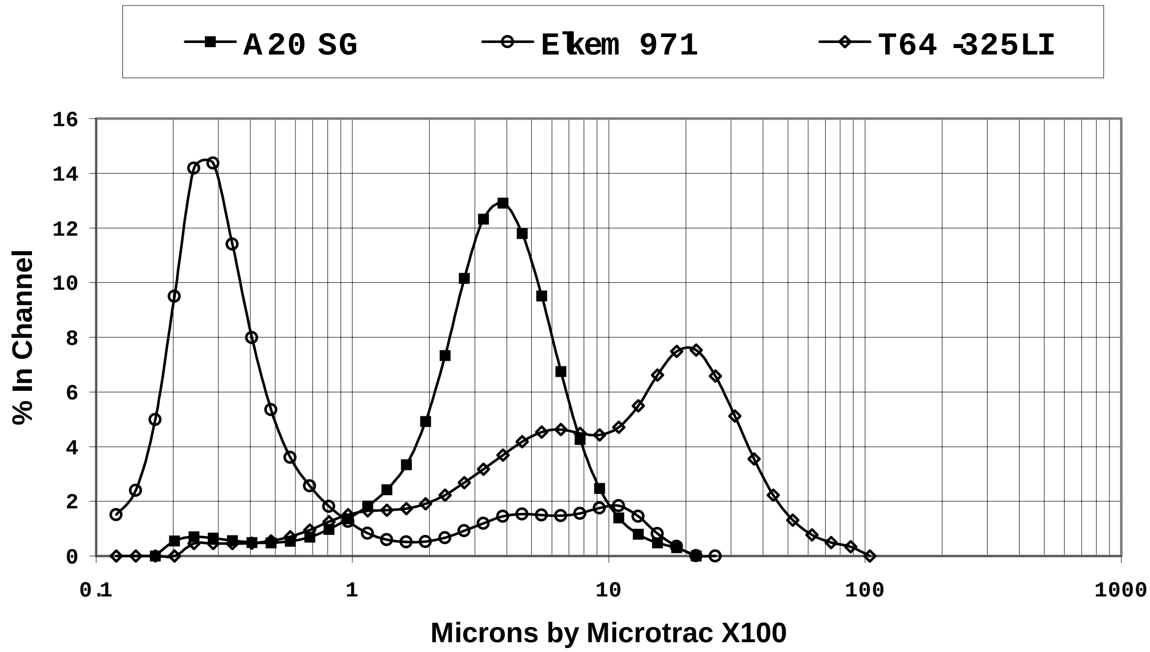
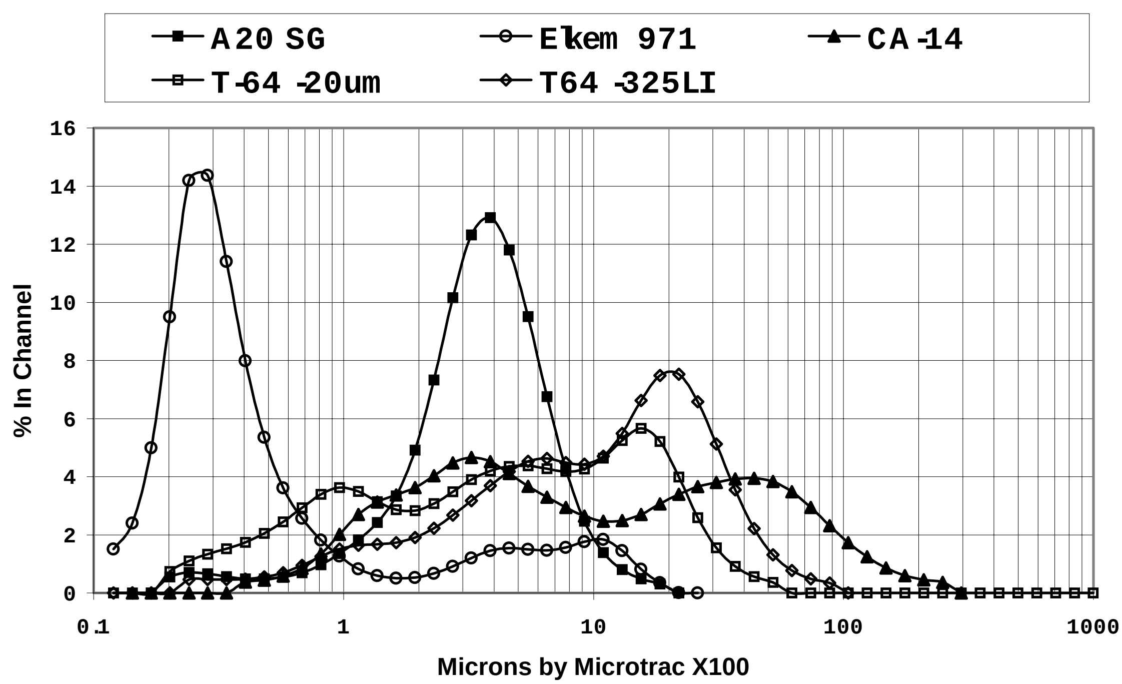


Figure 4: Matrix Fines - CA-14 and T64 -325LI Added



TEST CASTABLES USING A-20SG

To demonstrate the effectiveness of A-20SG three test castables were developed. Two are self-flowing ultra low cement castables containing 3% Microsilica. The first is based on Mulcoa 60 aggregate and aggregate fines. Both A-20SG and T-64 –20 micron have been utilized with 3% Microsilica and 2% CA-14 to achieve excellent self-flowing consistency and high strength at 4.8% water.

The second Microsilica containing castable is based on Tabular alumina aggregate. Again both A-20SG and T-64 –20 micron have been utilized with 3% Microsilica and 2% CA-14. In this self-flowing castable raw Kyanite has been utilized to minimize high temperature shrinkage. Excellent self-flow and strength are achieved at 4.3% water.

The final test castable was designed as a vibration placement system with no Microsilica in the low cement classification. It is based on Tabular alumina aggregate with only 3 sizes be utilized; 3 to 6 mesh, -6 mesh, and –14 mesh. A small amount of T-64 –20 micron is used with the A-20SG and 5% CA-14 to achieve excellent vibration flow and high temperature hot strength and creep resistance at 4.5% water.

The Alcoa Dispersing Aluminas were utilized in all three castables to achieve set control and minimize casting water at the various levels of matrix PSD optimization. The ratio of the retarding “S” type dispersing alumina to the accelerating “W” type is varied to achieve set control. The total of the two types is set based on the dispersing agent need. Typically, 0.7% to 1.2% total dispersing alumina is used.

The castable descriptions and testing results are presented in Table III.

CASTING PROPERTIES

The casting water level for each formulation was adjusted to achieve the desired casting consistency. At this casting water level both the Mulcoa aggregate formulation and the Tabular alumina formulation was described as being a soft, easy flowing castable that was easy to mix. There were no problems with dilatency during mixing. Neither was there any water or aggregate separation. Overall the casting consistency of both the vibration castable and the self-flowing castables were typical of high performance commercial formulations that utilize Microsilica.

Each test castable was evaluated for flow at 10 minutes after adding water to the mixer and at 40 minutes. For the self-flowing castables, the European mold was used and the results are reported as the average diameter of the material after allowing to spread for 2 minutes after the mold is removed. The mold is 80 mm high, 70 mm diameter at the top and 100 mm diameter at the bottom. Target flow is 220 to 250 mm after 2 minutes.

Excellent self-flow was achieved at 4.8% water for the Mulcoa aggregate formulation, 240 mm at 10 minutes and 236 mm flow at 40 minutes. The formulation de-aired very well. Demolding strength was reached at 3 hours with the dispersing alumina combination chosen.

At 4.3% casting water, the Tabular alumina aggregate self-flowing formulation achieved 249 mm flow at 10 minutes and 255 mm flow at 40 minutes. This formulation would have achieved the self-flow targets at 4.2% casting water. This formulation also de-aired very well. Demolding strength was reached at 4 hours with the dispersing alumina combination chosen.

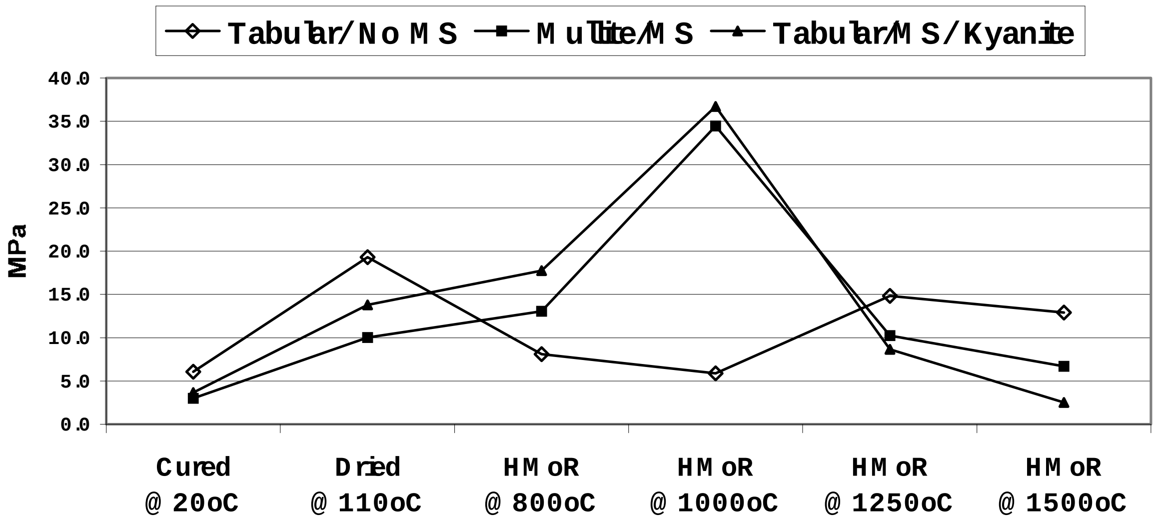
The vibration placement castable was tested using the ASTM flow mold. The sample is vibrated for 30 seconds after the mold is removed. Vibration flow is reported as percent increase in diameter. Target flow was 120 to 140% at 10 minutes. Excellent vibration flow was achieved at 4.5% water for this Tabular alumina aggregate formulation, 135% at 10 minutes and 4% flow at 40 minutes. The formulation de-aired very well. Demolding strength was reached at 2 hours with the dispersing alumina combination chosen. The dispersing alumina ratio chosen for this formulation is typical of the performance desired for a pre-cast shop that will pour the mixed batch quickly and wants to use it is molds twice in an 8-hour day.

Table III. A-20SG Castable Data

General Description	Mullite/MS	Tabular/MS/ Kyanite	Tabular/ No MS
Classification by Cement Content	ULCC	ULCC	LCC
Placement Type	Self Flow	Self Flow	Soft Vibration
Tabular Aggregate Content (%)		62.0	65.0
Mullite Aggregate Content (%)	59.0		
Tabular Matrix Fines Content (%)	15.0	18.0	15.0
(% <20 micron T-64 in Matrix Fines)	(15)	(15)	(5)
Mullite Matrix Fines Content (%)	11.0		
A-20 SG Content (%)	**10.0**	**10.0**	**15.0**
Microsilica Content (%)	3.0	3.0	
Raw Kyanite Content (%)		5.0	
CA14 Content (%)	2.0	2.0	5.0
Dispersing Alumina Content	0.7%M-ADS1 0.1%M-ADW1	0.6%M-ADS1 0.2%M-ADW1	0.5%ADS3 0.5%ADW1
Casting Water	4.8%	4.3%	4.5%
Castable Flow	Self Flow	Self Flow	Vibration Flow
10 minutes after adding mixing water	240mm	249mm	135%
40 minutes after adding mixing water	236mm	255mm	4%
Modulus of Rupture (MPa)			
Cured @20oC	3.0	3.7	6.1
Dried @110oC	10.0	13.8	19.3
HMoR @800oC	13.1	17.8	8.1
HMoR @1000oC	34.4	36.7	5.9
HMoR @1250oC	10.2	8.7	14.8
HMoR @1500oC	6.7	2.5	12.9
ASTM C-832 Expansion Under Load and Creep			
(1 Speciman Each Castable, Dried @110oC, 172 kPa Load)			
Maximum Dilation, %	0.51	0.82	1.22
Maximum Dilation, oC	1450	1231	1468
Creep from 20 hrs to 50 hrs, %	Failed	-0.88	-0.12
Permanent Change in Length, %	Failed	-3.29	-0.46
ASTM C-20 (Density (gm/cc) & Percent Apparent Porosity) and PLC with no load			
Bulk Density / Porosity / PLC (800 C Firing)	2.66/ 11.4%/ -0.11%	3.17/ 12.3%/ -0.06%	3.17/ 11.5%/ -0.01%
Bulk Density / Porosity / PLC (1000 C Firing)	2.67/ 11.5%/ -0.19%	3.18/ 12.0%/ -0.05%	3.19/ 8.5%/ +0.01%
Bulk Density / Porosity / PLC (1250 C Firing)	2.68/ 11.9%/ -0.31%	3.17/ 12.9%/ -0.11%	3.17/ 10.5%/ +0.01%
Bulk Density / Porosity / PLC (1500 C Firing)	2.63/ 11.3%/ +0.36%	3.20/ 11.0%/ -0.29%	3.08/ 15.8%/ +0.37%

MODULUS OF RUPTURE

Figure 5: Modulus of Rupture



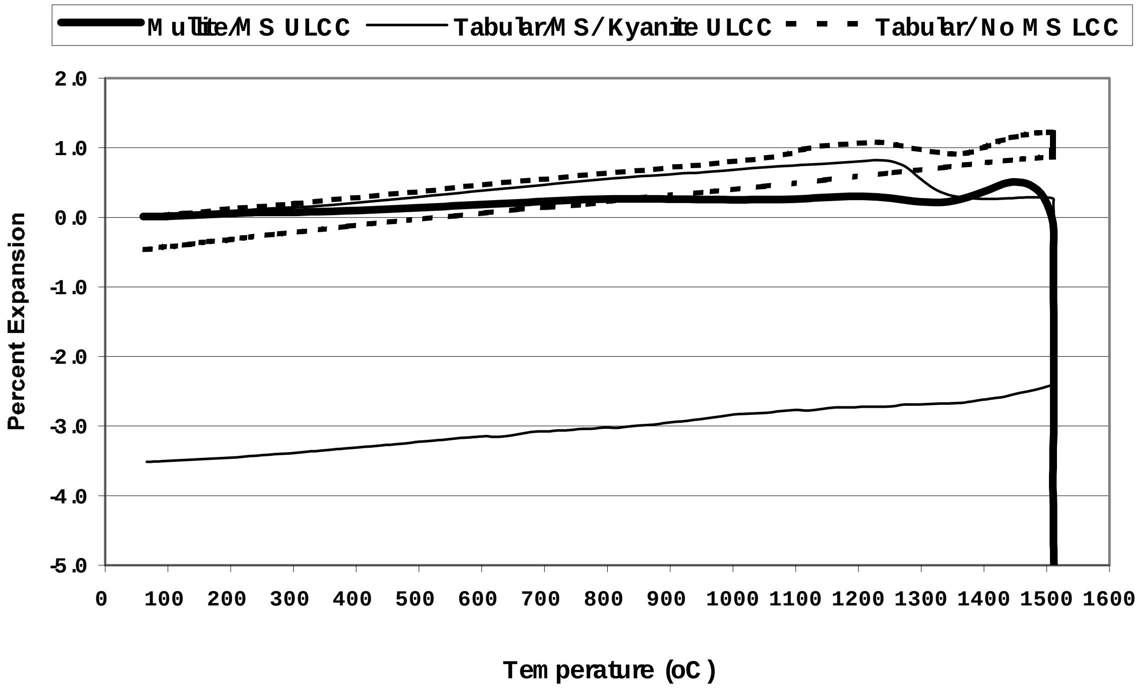
ASTM C-133 was used to determine Modulus of Rupture (MoR) after (1) curing 24 hours at ambient (20oC) temperature in a sealed plastic bag and (2) drying 24 hours at 110oC. Hot Modulus of Rupture (HMoR) was evaluated using ASTM C-583 at 800, 1000, 1250, and 1500oC. The results are plotted in Figure 5.

The dried strength of the ultra low cement, Microsilica containing, self-flowing formulations is very high considering only 2% CA14 and 3% Microsilica is used. Dried strength for the low cement, no Microsilica formulation is even higher at more than 19 MPa. It is obvious that the greatest difference in HMoR for the castables tested is at 1000oC. For both Microsilica containing formulations, the 1000oC HMoR is approximately 3 times the dried strength and is 6 times the 1000oC HMoR of the no Microsilica formulation. The strength enhancement of the Microsilica has started sufficiently in the 800oC data to overcome the strength loss related to the decomposition of the cement's hydration bonding that is seen in the no Microsilica formulation at 800 and 1000oC.

In the 1250oC and 1500oC HMoR it is evident that the Microsilica has started forming glassy phases as the hot strength drops from ~35 MPa at 1000oC to less than 10 MPa. At these temperatures the no Microsilica formulation hot strength has increased to the 13 to 15 MPa range. While this HMoR is more than adequate for many applications it is lower than the hot strengths seen for true high performance, high purity refractories. The 1250 and 1500oC hot strengths of this castable could be increased significantly with the addition of sub-micron Reactive Alumina such as A-1000SG or RG-100 and the inclusion of AR-78 fines in the matrix design if required. Of course the same can be accomplished with the use of bi-modal or multi-modal Reactive Aluminas such as A-3000FL or CTC-30.

ASTM C-832 EXPANSION AND CREEP UNDER LOAD

Figure 6: C-832 Expansion Under Load - Dried Samples



ASTM C-832 was used to determine Expansion Under Load (EUL) and Creep Under Load (Creep) for each test castable. These tests were run at the Orton Foundation Laboratory on dried samples. The results for EUL are plotted in Figure 6 and in Figure 7 for Creep.

As expected the rising temperature expansion rates for the two Tabular alumina aggregate test castables are very similar to ~1000oC. At ~1250oC the expansion under load for the Microsilica and raw kyanite containing Tabular alumina aggregate castable becomes shrinkage as glassy phases are formed but at ~1370oC the conversion of the raw kyanite causes the shrinkage to stop until ~1450oC is reached. Significant shrinkage under load does occur during the 50-hour creep hold at 1500oC. After ~1450oC is reached again during cool down the shrinkage rate becomes very linear for this Microsilica containing castable. Maximum dilation was 0.82% at 1231oC, creep from 20 hours to 50 hours at the 1500oC hold temperature was -0.88%, and the total shrinkage from beginning to end of the test was 3.29%.

For the no Microsilica, Tabular alumina aggregate test castable there is minor shrinkage from ~1250 to ~1350oC and some shrinkage does occur during the 50 hour creep hold. Maximum dilation was 1.22% at 1468oC, creep from 20 hours to 50 hours at the 1500oC hold temperature was -0.12%, and the total shrinkage from beginning to end of the test was 0.46%.

For the mullite aggregate, Microsilica containing test castable the expansion rate appears to be linear until ~750oC when the reactivity of the Microsilica becomes apparent. The expansion rate is relatively consistent to 1450oC when the glassy phases are formed in sufficient quantity to cause significant shrinkage. In after testing review of this castable’s performance it was apparent that the creep testing should have been done at 1400 or 1450oC not at 1500oC. These temperatures would have better demonstrated the performance for both of the Microsilica containing test castable formulations. For the mullite aggregate test castable the maximum dilation was 0.51% at 1450oC.

Figure 7: C-832 Creep Under Load at 1500oC for 50 Hours

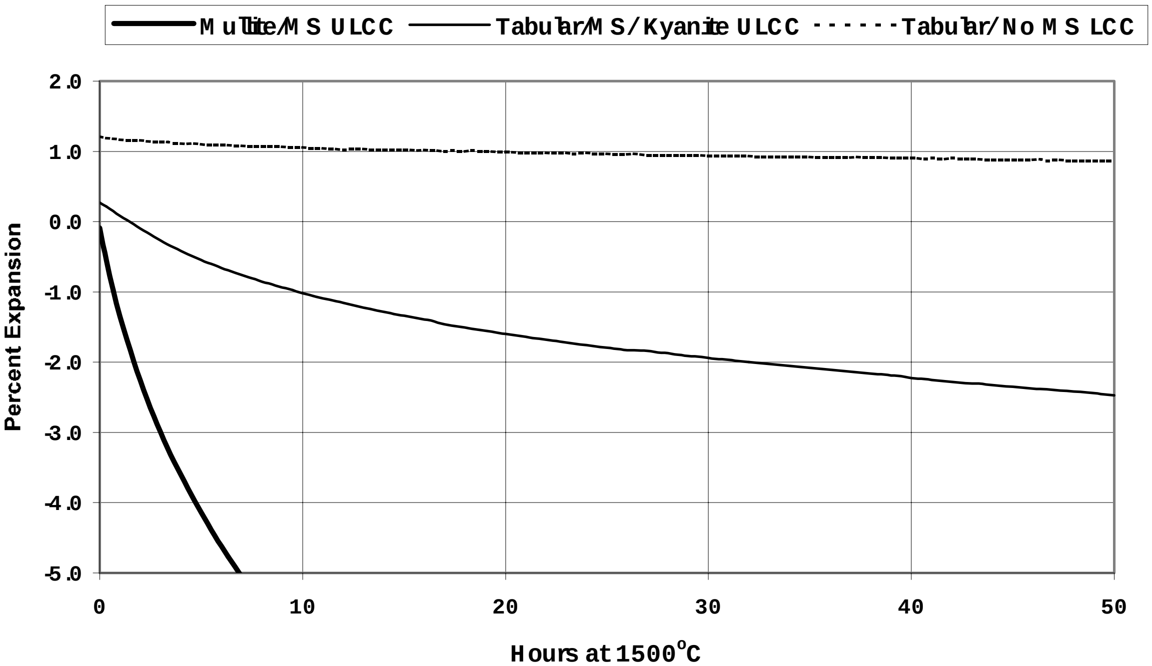


Figure 7 is a plot of the expansion (shrinkage) of the test castable samples during the 50-hour 1500oC hold. The ASTM Creep Under Load is reported as the change from the 20th hour at temperature to the 50th hour. Creep results are reported for the two Tabular alumina aggregate test castables. No creep was calculated for the mullite aggregate test castable because the sample shrinkage exceeded the equipment's range of operation prior to the end of the 50-hour hold. Creep at 1500oC for the Microsilica system was –0.88% and –0.12% for the no Microsilica system.

PHYSICAL PROPERTIES

ASTM C-113 was used to determine PLC (Permanent Linear Change) from the dried condition to after firing with no load. ASTM C-20 was used to determine Bulk Density and Apparent Porosity. Both tests were run on all 3 castable formulations after firing for 5 hours at 800, 1000, 1250, and 1500oC.

The C-20 densities and apparent porosities of the Microsilica containing formulations are representative of excellent commercial products. For the mullite aggregate density is near 2.66 grams/cc at all conditions and the porosity is less than 12% at all conditions. The formulation shrank after firing at 800, 1000, and 1250oC firing but expanded 0.36% after the 1500oC firing. For the Tabular alumina aggregate formulation with Microsilica and raw kyanite, density is ~3.18 grams/cc at all conditions and the porosity is less than 13% at all conditions. The shrinkage was ~0.10% except after the 1500oC firing it was up to 0.29%. The shrinkage at 1500oC was limited by the mullitization of the raw kyanite designed into the formulation. For the no Microsilica, Tabular alumina aggregate formulation, density is ~3.18 grams/cc except for the 1500oC fired sample that is down to 3.08 grams/cc. Porosity was less than 12% except for the 1500oC fired sample that is at 15.8%. This no Microsilica formulation had approximately zero change after the 800, 1000, 1250oC firings and 0.37% expansion after the 1500oC firing.

SUMMARY

A broad range of fully ground matrix aluminas has been available for a number of years. They include mono-modal, bi-modal, and multi-modal particle size distributions. They have allowed refractory designers to achieve almost any desires matrix particle size distribution. What has been missing in the Americas is a fully ground matrix alumina specifically designed for use with Microsilica.

Alcoa World Chemicals is now producing A-20 SG, a mono-modal, 3.5 median micron, narrow PSD, fully ground alumina. Test castables were developed using the A-20SG. Testing demonstrated that:

- Excellent self-flowing placement rheology can be achieved with 4.8% water using mullitic aggregate and 4.3% water with Tabular alumina aggregate with optimized aggregate PSD.
- Excellent vibration placement rheology can be achieved with 4.5% water using a total of 7 components; 3 T-64 aggregate sizes, T-64 –20 micron, A-20SG, CA-14, and dispersing aluminas.
- With 3% Microsilica and 2% CA-14 excellent hot MoR is demonstrated up to 1000oC; >10MPa dried, >13MPa at 800oC, and >34MPa at 1000oC.
- Thermo-mechanical testing demonstrated that the Microsilica containing test castables begin to soften just above 1200oC. The mullite aggregate test castable failed less than 10 hours into the creep under load hold at 1500oC but the Tabular alumina aggregate castables show higher creep resistance
- The Microsilica free formulation shows the highest creep resistance of all the A-20SG containing castables tested. However the strength and creep resistance in the temperature range around 1000 to 1500^{o}C and above can be improved significantly by using more sinter-reactive sub-micron Reactive Aluminas or bi-modal and multi-modal Reactive Aluminas[3,6].

Overall each of the A-20SG test castables proved to provide excellent properties with only minimal limitations. The A-20SG proved to be very beneficial in reducing the water and Microsilica content requirements for self-flowing castables.

OUTLOOK

In addition to the fully ground matrix aluminas, mono-modal or multi-modal, Alcoa World Chemicals has developed combined castable matrix formulations. Examples are the AFL aluminas for the INFILCAST® technology(8) or the very recently developed Alcoa Integrated Matrix (AIM) product family(9). These product families can be utilized as high performance tools for the refractory producer to simplify their life when formulating high performance, high alumina castables.

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