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OPTIMIZATION OF ALUMINATE CEMENTS WITH 7O% AND 80% OF ALUMINA BASED CASTABLES

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ABSTRACT

This paper will present the methods by which castable systems can be optimized using high quality calcium aluminate cements. Different castable systems will be examined through the use of examples, which show clearly the key aspects and parameters for both conventional and deflocculated castable systems. Conclusions will be drawn as to the key interactions of the castable binder components and the ways in which these can be optimized.

1. INTRODUCTION

Calcium aluminate bonded "refractory castables" have existed since 1856 when H.S.C. Deville prepared a refractory crucible using alumina aggregates and aluminate cement [1]. Calcium aluminate cements and their production had been patented by Lafarge in 1908 [2]. The first industrial calcium aluminate cement production was made by Lafarge in 1913. However, in the United States, refractory castables with calcium aluminate cements were not industrially manufactured before 1929, and the production of castables in Japan was initiated in 1939. Through approximately 70 years of history, refractory castables with calcium aluminate cements evolved from conventional castables with high cement content to the low and extreme-low cement contents, and systems with no cement. Advances in quality of raw materials, innovations in installation techniques and accuracy in the selection and utilization of the refractory castables have made possible and promoted this evolution and development of refractory castables.

Currently, cements with 70 and 80% of alumina are considered referential for these high technology castables.

2. THE CONCEPTIONS OF THE HIGH ALUMINA CEMENTS

The constitutions of 70% and 80% of alumina cements are basically different. Figure 1 summarizes the most important differences. The 70% alumina cements, e.g., Secar 71, are established with pure clinker, which contains the aluminate mono-calcium phases (CA, where C = CaO) and (A = Al2O3) and dicalcium aluminate (CA2) as main components. This pure cement concept allows a flexible and ample series of formulations which will be shown later. The 80% cements, Secar 80, are based on a concept where the main components are cement clinker and pure alumina. In this case, additives are necessary to control the rheology and the features of installation of the castables. This formulated cement concept allows simpler and easier formulation of the castables.

The mineralogy of both high alumina cements is based on the ratio CA/CA2, in a controlled fashion between the two phases. Previous studies [3] show the importance of the cement mineralogy as essential to the subsequent properties of the castables and the specific ratio CA/CA2 is chosen to optimize these properties.

Each mineralogical phase provides specific attributes and it is the combination of these two phases that generate the final properties. This is shown in Figure 2.

It can be verified that phase CA offers a higher degree of hydraulic activity, however, in large amounts, it can cause problems as exaggerated sensitivity to certain additives, aging or limited storage times. The more significant problem is due to extreme heat liberation associated with the hydration of this phase, being able to cause intense water vapor output with cracking of the castable. On the other hand, CA2 shows discrete hydraulic activity at ambient temperature. Normally additives are necessary "to activate", but they can alter the rheology, especially of deflocculated castables or of low cement systems. However, CA2 hydrates at high temperatures and, when combined with CA, it acts as a secondary binder bringing higher mechanical resistance to the castable during the drying process. CA2 can be considered as having an "in situ filler" effect that magnifies the intermediate mechanical resistances of the conventional castables. This fact is attributed to the higher amount and to the type of AH3 generated during CA2 hydration process. Moreover, CA2 is an excellent precursor for the CA6 formation, which acts as binder phase at higher temperatures in many systems.

In this way, through the choice of the ratio CA/CA2, it is possible to optimize the beneficial effects of each phase of the cements and, at the same time, to minimize negative effects. Thus, by controlling these phases, cements can be produced in industrial scale with guaranteed consistency and reliability. This fact is particularly important in the case of deflocculated castables where the controlled dissolution of calcium ions is necessary to obtain fluidity values and adequate working time.

2.1. HIGH ALUMINA CEMENTS IN REFRACTORY CASTABLE APPLICATIONS

Both, 70% and 80% alumina cements are used in extensive series of castable formulations. Due to the conception differences between the two types of cements, it is evident that some applications will be more convenient for a determined application. The choice will depend to a great extent on the logic of the formulation. Figure 3 shows the refractory castables definition.

As illustrated in Figure 3, the castables can be classified by the application technique employed and also by considering the CaO content. In the case of choosing which cement to use, the most important is the classification based on the formulation logic. There are two types of logics, in the first one the cement plays the role of main binder and the CaO dosages in the castable normally are above 2.5%. The second logic consists of systems of dispersed binders [4]. These castables contain fine reactive materials (silica, alumina, chromium oxide, etc.) for better packing of the mass. At the same time, a reduced amount of CAC (calcium aluminate cement) is used for higher intergranular contact. Dispersing additives are necessary to increase the fluidity of fine particles and to allow the castable installation with low amounts of water. The benefits of the incorporation of these fine materials, reducing the amount of cement and using additive are:

- Lower CaO contents in the castable limiting the formation of CAS2 and C2AS phases with improvement in mechanical properties at high temperatures and in corrosion resistance;
- Reduction of the water content reducing the porosity with increase of the density. Corrosion and abrasion resistances are improved through a denser structure of the matrix;
- Improvement of the particle packing improving the thermo-mechanical properties and the corrosion resistance.

Continued on page 8

		Secar 7l	Secar 8O
Mineralogy	CA	xxx	xx
	CA2	xx	x
	α-Al_2O_3	x	xx
Concept Cement		Pure Cement	Formulated
		Without additives	Clinker Alumina Additives
Refractory Applications		Flexibility in Formulation	Simplicity in Formulation

Figure 1. Constitutions of calcium aluminate cements

- Gradual Hydration
- Flexibility in the formulation
- Industrial Reliability

Ideal Position
↓

"Pure CA" ↔ "Pure CA2"

→ • High and fast hydraulic potential ← • Low hydraulic potential at ambient temperature
→ • Tolerance and control of Additive ← • Low green mechanical resistance
→ • Severe heat discharge → • Good precursor for CA6
→ • Problems of aging → • AH3 filler effect in situ
→ • " Nervous Behavior " → • (< 1.5 micron). Good intermediate resistances

Figure 2. Advantages of the mineralogy based on CA/CA2

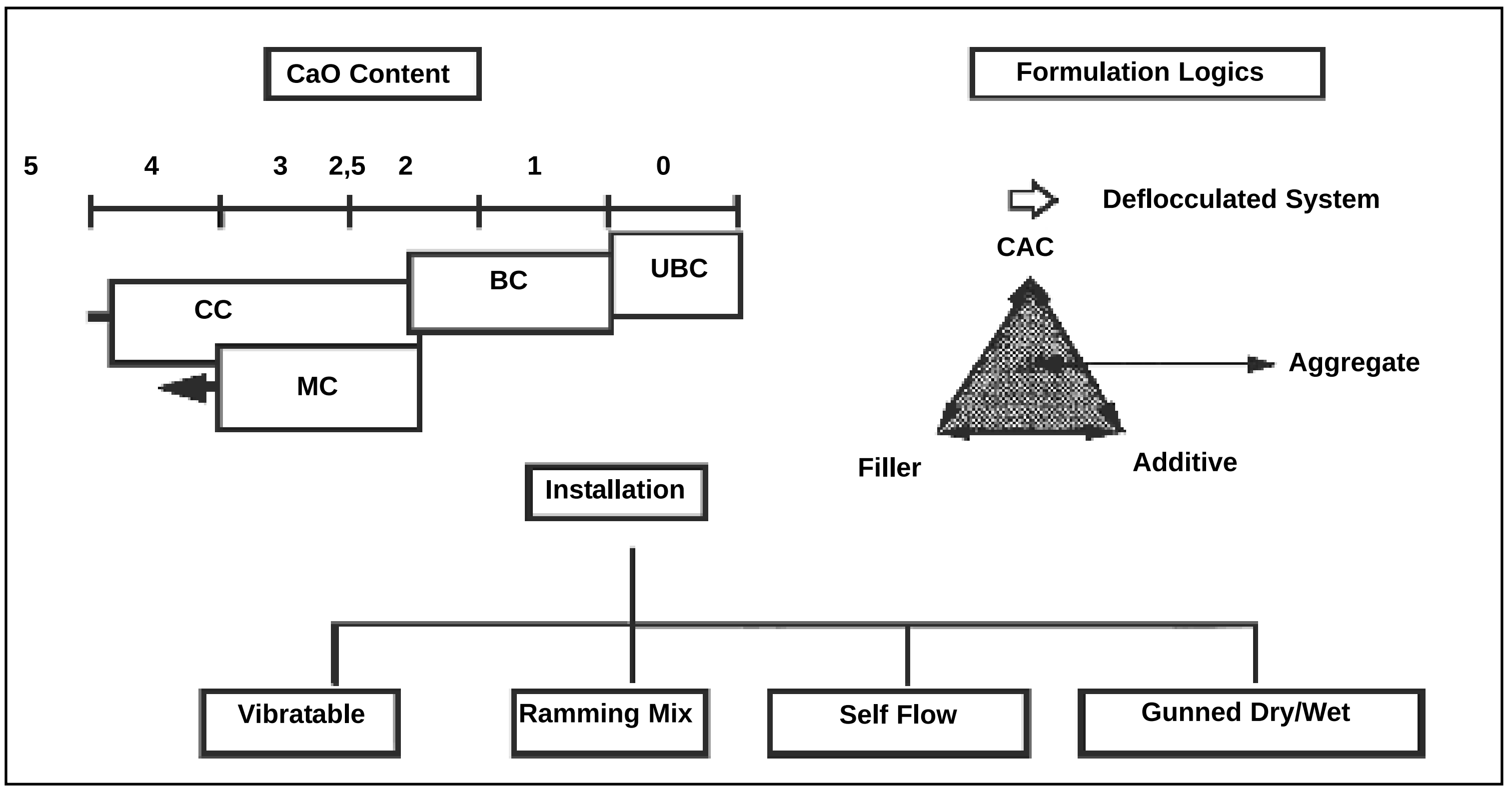

Figure 3. Classification of Refractory Concrete Systems

Continued from page 3

This logic of deflocculated castables can be extended for castables with low cement, below 2%. The key parameter for the formulation is to assure low water consumption to keep a dense structure of low porosity. Otherwise, the benefits of the deflocculated castable will be lost. The active binder can be considered [5] as being a composite of three independent components: calcium aluminate cement, additives and fine reactives. This approach explains why these components are represented as an interdependent triangular system, as seen in Figure 3.

For these dispersed or deflocculated systems, the pure cement concept (70%) is the preferred one. It is a way to prevent undesirable interactions between the cement and the other added elements like the fine materials and the additives. Cement of 80% of alumina is used for conventional castables systems, where the cement acts as basic binder.

This cement is preferred for use in systems of conventional castables with high purity aggregates such as fused alumina. In the following example, a castable is developed on the basis of fused alumina and 18% content of Secar 80. For reference, comparisons are done with another 80% cement source. To allow the comparison, the amount of water was kept steady and the effect on the fluidity and the mechanical properties was evaluated. The results are shown in Figs. 4 and 5. It can be verified that the cement of 80% of alumina promotes high values of fluidity, steady working time and excellent mechanical resistance development.

It can be verified in Fig. 5 that the mechanical resistance decreases after dehydration while firing at 1100°C, as expected, but due to the CA/CA2 mineralogy the resistances are kept high enough and the reduction is minimized.

2.3. EXAMPLE OF APPLICATION OF 70% OF ALUMINA CEMENT

This example shows some of the formulations that should be considered when applying 70% alumina cement to deflocculated castables systems. The active binder phases can be represented in a triangle (as in Fig. 3) while the global behavior is the result of several interactions between individual components as additives and volatilized silica.

The choice of each one of the components becomes a critical factor in the rheology control of the castables. This choice must be made assuring at the same time that the properties of the installed product are retained. Additives employed will depend on the specific choice of the fine materials. Previous studies [6] show the impact and the role of additives and fillers in a variety of systems. The impact of the fume silica and of the type of alumina is extremely important as these materials modify the hydration features of the calcium aluminate cement.

For example, pH and the amount of carbon in the fume silica [7] can have a remarkable effect on the necessary amount of water, on the fluidity and also on the development of mechanical resistance. Values of pH and of high carbon content in the fume silica result in a castable with inferior properties after installation. It was shown [8] that the observed behavior is due to the different interactions between additives, the fume silica and the cement.

In the following example, the control of the installation through modifications of formulation becomes possible through the optimization of dispersing additives. It is possible to simultaneously add diverse additives, each one with different function, in order to modify the rheology of the castable. For example, the use of multiple additive systems allows the optimization of the initial fluidity of the castable, as well as its degradation. This is only possible when Secar 71 is used. This cement shows a considerable tolerance to the additive system that is due to the flexibility of this sophisticated system. However it should be highlighted that it is possible to obtain excellent results in low cement castables containing Secar 71 and a simple additive system. The use of a multiple additive system allows the optimization of the installation properties, that should be understood as consequence of the mineralogical control of the cement based on the relation CA/CA2 ratio.

The example in Table I shows the effect of a multiple additive system on the properties of the castable. In the same example, the use of four additives allows a 20% water reduction while retaining the application properties and mechanical properties. Two dispersing additives are used for dispersion of the fines, sodium tripolyphosphate is used for fume silica and a poliacrylate is used to disperse reactive alumina. Two types of stabilizing/retarding agents are added to assure a working time of 60 minutes even with 4% water content.

The features of castables application are summarized in Fig. 6. It is verified that the use of a multi-additive system simultaneously allows less water consumption with the best properties of flu-

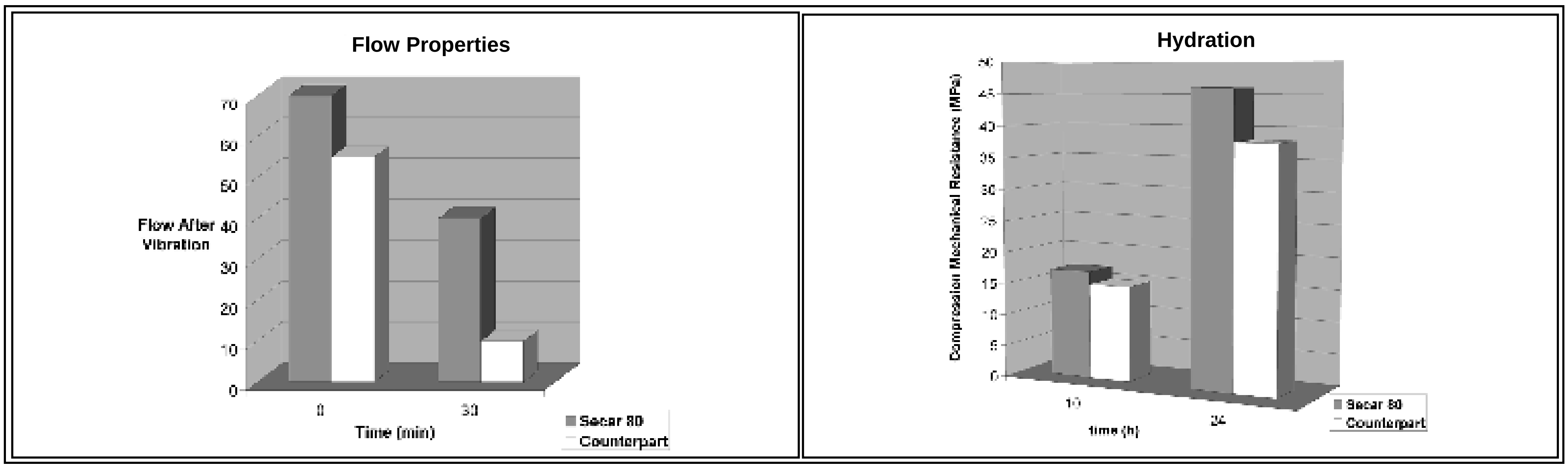

Figure 4. Use of Secar 80 in conventional concretes- Flow and Hydration

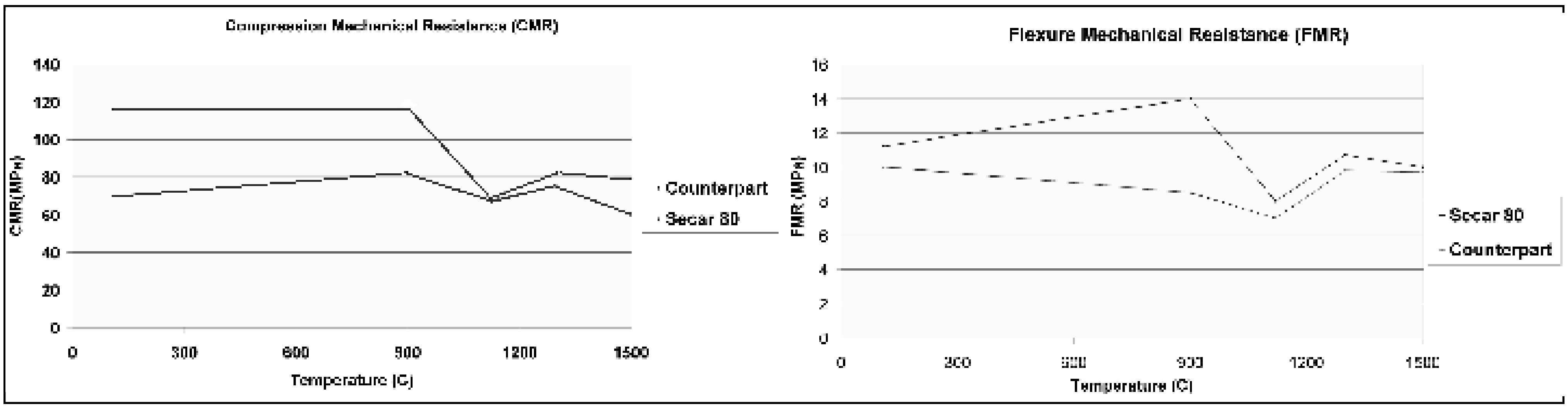

Figure 5. Use of Secar 80 in conventional concretes- Mechanical Properties

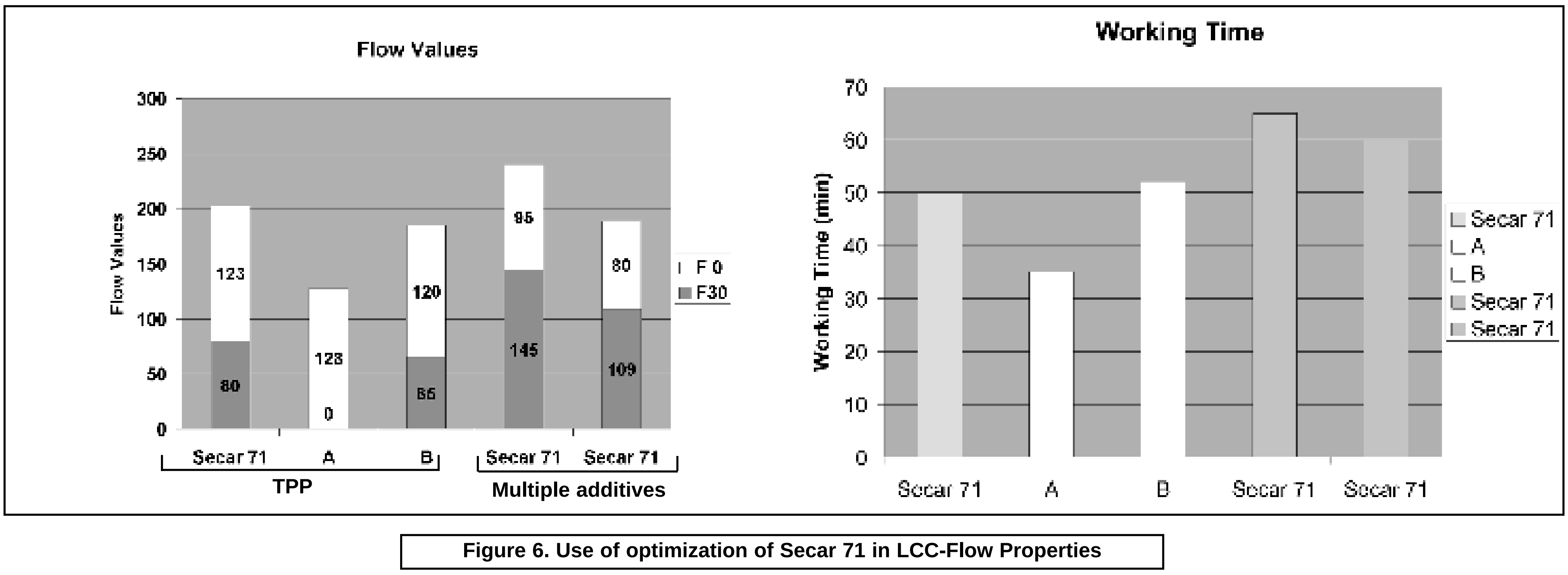

Figure 6. Use of optimization of Secar 71 in LCC-Flow Properties

Table I. Application and optimization of Secar 71 in castable of low cement with fume silica

		Basic LCC		Optimized LCC	
Alumina Tabular	6-10	22	22	22	22
	8-14	10	10	10	10
	14-28	19	19	19	19
	-48	29	29	29	29
Reactive Alumina	P152SB	10	10	10	10
Fume Silica	Elkem 971ND	5	5	5	5
Cement	Secar71	5		5	5
	Counterpart		5		
Additives	TPP	0.12	0.12	0.02	0.02
	D7S			0.03	0.03
	NaBiCarb			0.0015	0.0015
	Citric Acid			0.0015	0.0015
	Water	5%	5%	5%	4%

idity and with longer period of stability of fluidity. They are important characteristics for applications of self-leveling and gunned castables. Mechanical properties and subsequent thermomechanical properties are not compromised by the use of a multi-additive system and, in most of the cases, the results are improved, as it is seen in Fig. 7.

3. CONCLUSIONS

These examples show that the "binder" system in castables, composed only calcium aluminate cements or by a deflocculated system based on them, can be optimized through the use of sophisticated cements as Secar 71 and Secar 80, based in the control of the ratio CA/CA2.

For deflocculated castables, castable optimization can be made simultaneously by considering all three components, any change in one of the components implies a great system change, as the binder system is constituted by the interdependent combination of CAC, fine reactive materials and additives.

In this way, one of the most important formulation parameters is to assure the reliability and repeatability of the raw-materials behavior through systematic and appropriate selection of components.

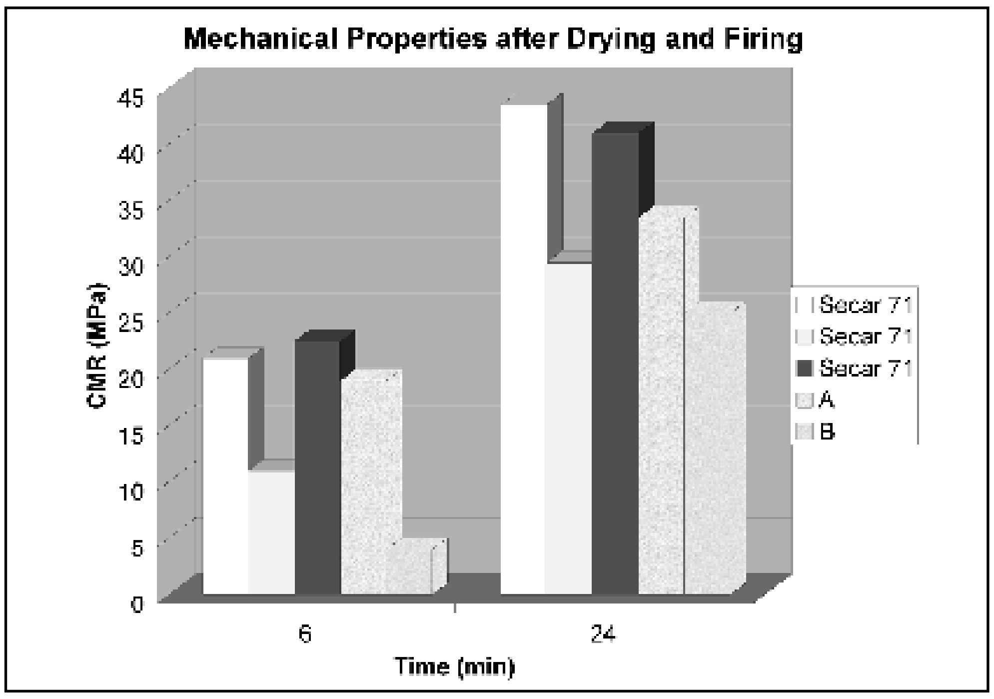

Figure 7. Use and optimization of Secar 71 in LCC-mechanical properties

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Trends in Refractories Technology Highlights of the ACerS Annual Meeting

By Laurel M. Sheppard, Contributing Editor

At the 102nd Annual Meeting held in St. Louis in May of this year, the Refractory Ceramics Division organized a number of sessions under two major categories: fundamentals and industry trends. These sessions included invited and contributed presentations that covered testing, corrosion and thermomechanical properties, modeling, monolithics, and glass furnace refractories. Recycling was discussed in another symposium (Science and Technology in Addressing Environmental Issues in the Ceramic Industry).

As markets continue to shrink due to consolidation, the refractories industry is challenged to continue improving materials and service life. Certain technologies are also becoming obsolete, like ramming mixes, and monolithics continue to replace brick in certain applications.

INNOVATIONS IN MATERIALS

Refractories manufacturers continue to look at ways to improve materials in order to remain competitive. Sometimes these innovations are first developed at universities. For instance, joint research at the University of Alabama and Mexico's National Polytechnic Institute has produced dense mullite by combining micronized kyanite with alumina. This is achieved by attrition milling a mixture of kyanite and alumina for up to 12 hours in water. Submicron size particles are formed after six hours. These particles activate the decomposition of kyanite, which occurs at temperatures 100 degrees lower. Dense mullite is produced after sintering at 1600 degrees C.

Adding graphite to alumina castables can achieve stronger resistance to slag penetration and consequent spalling of linings in iron and steel making vessels. However, how to incorporate enough graphite to improve oxidation resistance and minimize porosity is a challenge. Researchers at Canada's Ecole Polytechnique have developed several promising approaches. Natural flake graphite is added in the form of micropellets (pelletized agglomerates) or briquettes made from crushed aggregates containing alumina, graphite and antioxidants. These approaches have advantages over straight addition of graphite or TiO2-coated graphite.

Using micropellets or briquetts, water demand can be significantly reduced compared to straight flake graphite, Figure 1. This is attributed to the agglomerated state, which has a much lower specific surface area compared to the flake graphite in a dispersed state. Bonding strength after drying, and both cold and hot strengths are also higher for the material with the micropellets or briquettes. Al2O3-MgO castables with up to 30% MgO can be made with optimized properties (workability and thermal expansion) and good slag penetration resistance, as long as anti-oxidants are used.

Cold setting cordierite castables are also under development at CETMIC, a research organization in La Plata. The castables are made with cordierite-mullite aggregates and a fine matrix of silica, alumina and magnesium oxide. The setting bond was achieved using phosphoric acid (PH) or monoaluminium phosphate (MAP) in times ranging from twenty minute to two hours. The setting time was a function of the type of MgO, the phosphate concentration and the MgO/phosphate ratio. The main reaction phase formed was MgHPO4.3-H2O but amorphous phases were predominant. The final crystalline phases at 1100-1350 degrees C were cordierite and mullite.

MODELING IMPROVES PERFORMANCE

Though modeling of refractories has been around for at least a century, only recently has it been more widely used to improve and predict refractories performance. Thermal shock theory has already reached a level where the relative thermal shock resistance of refractories can be accurately predicted and compared using equations involving selected properties, without the need for actual thermal shock testing. Dr. Charles Semler, a refractories consultant, believes that the need for thermal shock tests will eventually be eliminated.

Mathematical modeling is being used to improve refractories performance. Japanese researchers developed and used equations to model the effect of three separate mechanisms on refractory wear, thereby increasing lining life in copper smelting furnaces. Another thermomechanical study of C-MgO refractories used in ladles used finite element analysis (based on room temperature and hot property measurements) to understand the stress effects in linings, and applied the results to improve refractory selection and lining life.

Researchers at Baker Refractories have developed a computer model based on published phase diagrams that graphically maps the slag compositions required for optimal foaming conditions in electric arc furnaces (EAF). The model has been applied to steelmaking operations, with excellent correlation between the predicted and

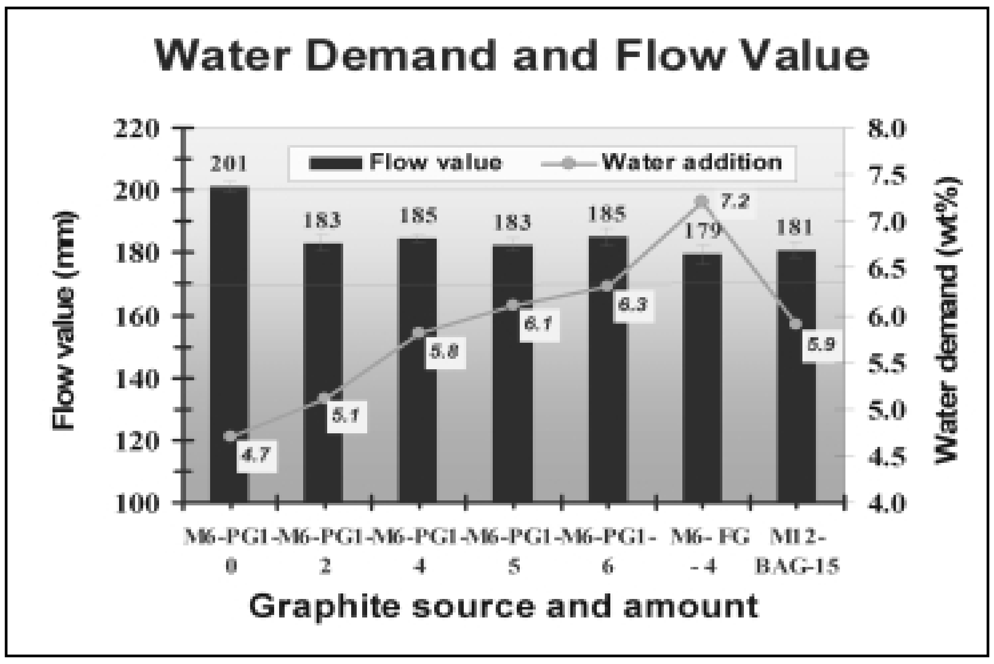

Figure 1. Water demand and flow value as a function of type and amount of graphite. PG=pelletized, BAG=briquette.

actual results. Benefits to these operations include a reduction in both energy and electrode costs, and extended refractory life. Similarly, the Albany Research Center has developed a computer model to calculate the saturated EAF slag chemistry with respect to CaO and MgO, the MgO saturated EAF slag chemistry, and the solid content in solution to optimize spent refractory for slag conditioners.

Other sophisticated technologies being applied to refractories include neural networks and fractal analysis. Neural network technology has been combined with finite element analysis to model inhomogenieties due to chemical reactions, sintering and thermal expansion. However, according to Semler, there are still a few things lacking that is preventing the wider application of modeling. More complete product data sheets and more hot property data are needed. One material lacking in such data (especially creep and high temperature modulus of elasticity) is fusion-cast alumina used in superstructure applications in the glass industry. A closer look at testing methods is necessary to delete obsolete methods, improve existing methods, and develop new ones. New theories, based on proven concepts, as well as improved software for more accurate results, are two other key requirements for moving modeling from the laboratory into the real industrial world.

TOWARD BETTER TESTING

Until modeling is improved, manufacturers will still have to rely on measuring properties and performance using various testing methods. A major problem is developing laboratory tests that accurately simulate real operating conditions. For instance, slag corrosion testing methods-such as crucible, dip or rotary slag tests-can have this limitation. The University of Sheffield compared corrosion results from static crucible and rotary slag tests of MgO-C refractories with Al and Si additions with brick of similar composition taken from a service lining. Different behavior was observed, indicating that conditions varied between the actual furnace and tests. Future work will look at designing rotary slag tests to better match actual service conditions.

Other Sheffield research investigated the corroded microstructure of a steelmaking vacuum degasser unit made of tabular alumina (TA) grain and calcium aluminate cement and compared it to that of a similar castable after crucible testing. The crucible tested material showed complete slag penetration into the refractory, where acicular and platy CA6, dense angular hercinitic spinel and a calcium aluminosilicate glassy phase interspersed with TA were found. The corroded material, on the other hand, showed consecutive layers of slag, porous hercinitic spinel and acicular CA6 before penetration. The slight differences in microstructures are attributed to different conditions of temperature, atmosphere, and slag composition, again indicating that laboratory tests must simulate actual service conditions.

However, data from simulated laboratory experiments has proven useful in interpreting corrosion data acquired from industrial processes, in joint research at the University of British Columbia and Cominco Ltd. (Canada). Refractory bricks used at Cominco's lead smelting operations, made from magnesia-chrome and alumina-chromia, were tested for corrosion behavior in contact with industrial slags. The dissolution of the mineralogical composition were then studied using both slag rotary furnace and crucible methods. This data was correlated with the apparent porosity, air permeability and pore dimensions.

High chrome-alumina refractories have been developed for a new generation of gasifiers that can operate using a variety of fuels. The Department of Energy's Albany Research Center is using a modified cup test in a simulated gasifier atmosphere to identify and characterize refractories/slag reactions. Preliminary results show that refractory wear is due to slag attack and spalling and involves complex interactions between Fe and Ca, accompanied by leaching of Cr.

Simulation of the conditions present in a glass melter is also essential for conducting crown corrosion experiments. Temperature, gas phase chemistry, and furnace pressure and flow must be reproduced if a small scale simulation is expected to accurately reflect the crown corrosion rates realized in industrial applications. This is usually not possible with conventional tests. Therefore, the Refractories Satellite of the Center for Glass Research (CGR) has completed construction of an oxy-fuel simulator (OFS) furnace at the University of Missouri-Rolla.

The OFS furnace has been designed to reproduce the environment of a working commercial glass furnace and it can be modified to accommodate other firing systems and burner arrangements. The OFS has already proved to be a valuable tool for simulating glass melting and for conducting preliminary corrosion studies of silica crown refractories. The furnace has also been designed to test new on-line sensors, such as electrochemical cells that measure the concentration of NaOH in situ. By combining OFS data with existing computer models of glass melting furnaces the evaluation and prediction of furnace life operation is possible as well.

Rheology and permeability are also important properties of refractories that need characterized. Permeability assessment is particularly useful during thermal treatment to evaluate a castable's susceptibility to explosive spalling and thus to mechanical damage. Researchers at the Universidade Federal de São Carlos, (UFSCar) Brazil, have developed equipment to evaluate high-alumina self-flow castables to air flow at temperatures varying from ambient to 800 degrees C. The experimental data correlates well with theoretical calculations. As the temperature increased, the permeability decreased and the flow resistance increased. A major advantage of this method is that it can be used to study fluid dynamic behavior since it can measure both reversible and irreversible changes with temperature.

Since castables are also needed with predictable and controlled rheological properties, researchers at UFSCar have developed a novel rheometer that can evaluate castables more accurately. The system can measure the response of castables to shearing during mixing as a function of time or under cycling revolutions. Both temperature and pH can be evaluated in situ. The rheometer uses planetary motion produced with a 2 hp motor and electronic speedometer. It has been successfully used to design new castables with lower surface areas, thereby reducing the amount of water required. Other parameters that can be optimized include particle size distribution, solid content, and particle shape, as well as type of dispersing and thickening agents.

A CLOUDY FUTURE?

In the last decade, the refractories industry has gone through significant mergers and acquisitions. There are now just two huge conglomerates (RHI and Vesuvius) that dominate the refractories markets. At the same time significant advancements have been made in refractories technologies, including sol gel based materials, pumpable refractories, shotcreting, and pre-cast shapes based on low-cement and sol-gel compositions. In the next century, the industry faces a double-edged sword-how to continue developing new technology to remain competitive in an ever shrinking market.

One solution to this challenge may be advances in computational science. As the cost of computing capabilities continue to decrease, and these capabilities in turn improve, refractories manufacturers will be able to afford to take advantage of these opportunities to develop new products.

Author's note: The papers will be published by the American Ceramic Society in a proceedings of the Refractory Ceramics Division, along with last year's papers.

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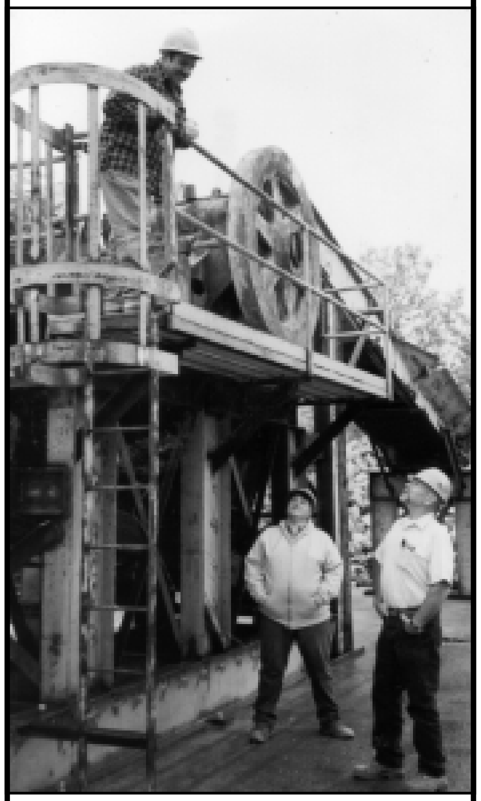
SIDEBAR

RECYCLING CHALLENGES

Though there are many potential applications for recycled refractories and an almost inexhaustible supply of scrap material, there are still few incentives for U.S. manufacturers to implement recycling, according to Robert Oxnard, President of Maryland Refractories Co. (Irondale, Ohio). Landfills are still inexpensive and available, virgin materials usually cost less and there are often no short term payoffs. Higher value materials are also leading to fewer rejects. Those companies willing to pay up front costs associated with recycling are usually high technology firms.

However, Oxnard believes that the U.S. may have to play catch up with its European counterparts (who recycle more due to stricter environmental laws), as international competition increases. In fact, ISO 14000 may eventually level the playing field. Another good reason to recycle is the reduced liability exposure. A more esoteric reason is it is the "right thing to do." Public perception is also changing to view companies that recycle in a more positive light than those who do not.

Even with some of these incentives, Oxnard predicts that the U.S. refractories industry will be slow to change. Only a few of the larger manufacturers have formal recycling programs and there are only a few government programs addressing the issue. For instance, the Albany Research Center is conducting research to recycle spent refractory (especially MgO+C) for use as EAF slag conditioners or as gunning repair mixtures.



“Big Bertha” at Maryland Refractories Co.