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Ultra-High Porosity Refractories

Brandon Bundy

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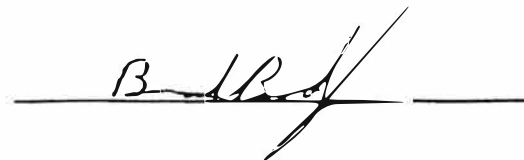
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OURE project: Ultra-High Porosity Refractories

Brandon Bundy

A handwritten signature in black ink, appearing to read "Brandon Bundy", is written over a solid horizontal line. The signature is stylized and cursive.

Abstract

Alumina foams are an attractive alternative to the current fiber board refractories used today by industry. Lower cost and ease of production will improve furnace operation and help lower total cost of furnaces. Many experiments have been conducted to find the optimum foaming system. Ceramic (alumina) slurry is combined with certain surfactants in a mixer. Air is entrained into the slurry using mixer and foam is produced. The green body is dried and fired to produce a finished ceramic (alumina) refractory. Unfortunately, many variables are present with these set of experiments and so the project is not yet finished.

Introduction

High temperature furnaces in industry are expensive. The cost is easily divided between the heating elements, the controller, and the insulation. My goal with this project was to lower the price of the insulation and improve building and maintenance of high temperature furnaces.

There are many types of insulation, from simple bricks to fiber boards; each serves a distinct purpose. The fiber boards are the most expensive and are easily damaged. I can not speak to why the fiber boards are expensive but they are desired when a light weight refractory is needed. They possess a low density which allows them to achieve many desirable qualities such as low mass and a low coefficient of thermal expansion (CTE). With the fiber boards installed in a furnace, the furnace achieves a higher efficiency because it has to heat less mass to temperature. Further more, the low CTE allows the fiber board to go through multiple rapid heating/cooling cycles without permanent damage occurring.

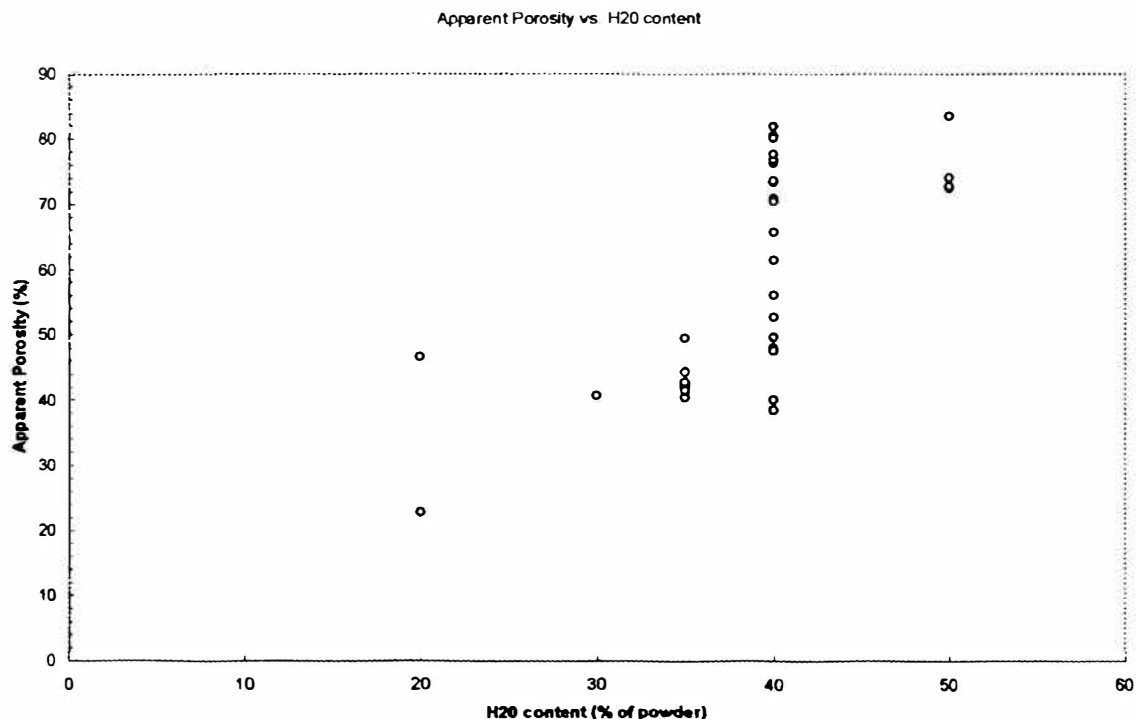
Unfortunately, if the expensive fiber board is damaged, the furnace has to be dismantled until the damaged piece can be removed and replaced. Depending on the piece, a good deal of the furnace has to be rebuilt. The purpose of this project is to replace this fiber board with an ultra low porosity castable. The castable will have the same low density properties that the fiber board possesses, but the castable will be significantly cheaper. Additionally, if the insulation is damaged, the castable can be patched with more castable placed on the damaged part. The low density castable was achieved with manipulating the surface tension and viscosity properties of various alumina slurries.

Body

At the beginning of this section it should be made clear that this research is still on going. The end is far from being seen because there are a multitude of variables that have yet to be identified and controlled. The goal for the project has remained the same but yet there have been so many changes in the experimental procedure. To best show what has been accomplished and what has been failed, this section will show the procedures taken over the past year and some of the results that had influenced the various changes.

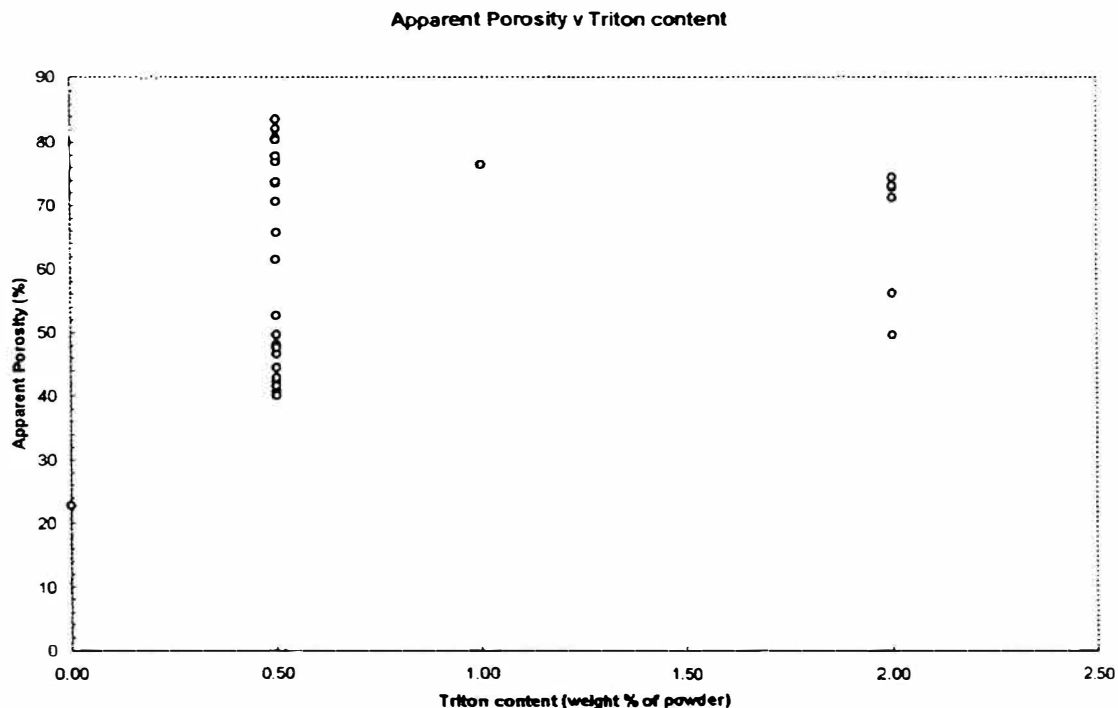
The first foams were started with alumina slurry made of Al 1000 and water, Triton was added and foamed using a hand mixer. Once the foam was formed, it was cast into a set of two

inch cube molds. Unfortunately, the foam was not stable in the molds and would not cure. There was no green strength so the foams would crumble when taken from the molds.



To supplement the Al 1000, Calcinated Alumina (CA) and Alpha-bond Alumina were added in hopes that they would act to set the foam matrix. The cement to alumina ratio was 18/82 but was set arbitrarily. The ratio of CA to Alpha-bond was changed. Alpha-bond alumina was eliminated because shrinkage in the fired samples with Alpha-bond was unfavorable. A few more experiments were conducted varying CA/Alumina ratios. The CA did not hurt the foam and setting was still present so pure CA was foamed. The CA foam increased density so Alumina was re-introduced. A ratio of 50/50 CA/Alumina was chosen to help set the foam but maintain the porosity.

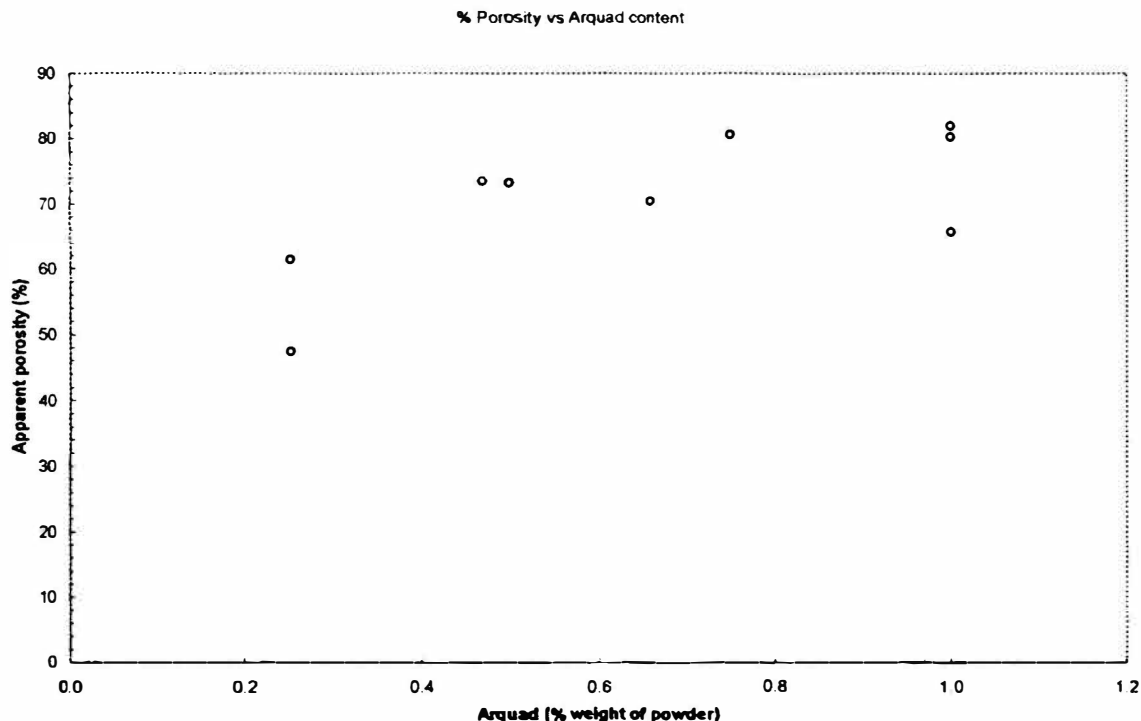
With the ratio set at 50/50, Triton levels were varied to see the effect of Triton level on porosity. The idea was to see the minimal level of surfactants in the foam because any chemical hinders the bonding of the alumina particles. This is an underlying idea that drove a lot of experiments to minimize chemicals and increase bonding within the matrix. The Triton content was only important at one amount. Under a certain amount, around .5 g, the slurry does not entrain much air, causing dense, low porosity foam. Above the .5 g, the additional Triton becomes extra coating on the surface of the slurry.



One variable quickly identified was that of the hand mixer. The hand mixer introduced further chance of the human factor. A suitable automated replacement was an ordinary kitchen aide mixer found in most stores. The new mixer allowed for elimination of the human factor and improved foam volume during mixing.

Following the advancements with the Triton content, the ratio of CA/Alumina was changed to a lower ratio of CA to Alumina. This was driven by the fact that the higher alumina content foams possessed higher porosity, but after systematically reducing the CA content, it was found the setting time was increasingly worse so a 50/50 mix was deemed the best mixture. In this set of experiments, Darvan C was introduced to the mixtures to act as a dispersant for the powders. All remaining experiments were run using Darvan C.

One consistent problem with the foams was that the air bubbles would coalesce to form large pores. Large pores were highly unfavorable so two surfactants, Ammonium Stearate and Arquad T, were chosen to add stability to the small individual air bubbles. The two chemicals were added in small amounts using weigh boats. The ratio of the two was changed to see the varying effects. The Ammonium Stearate added slight porosity to the foams and gave the final samples a nice uniform surface. The Arquad dramatically decreased the amount of large air bubbles.



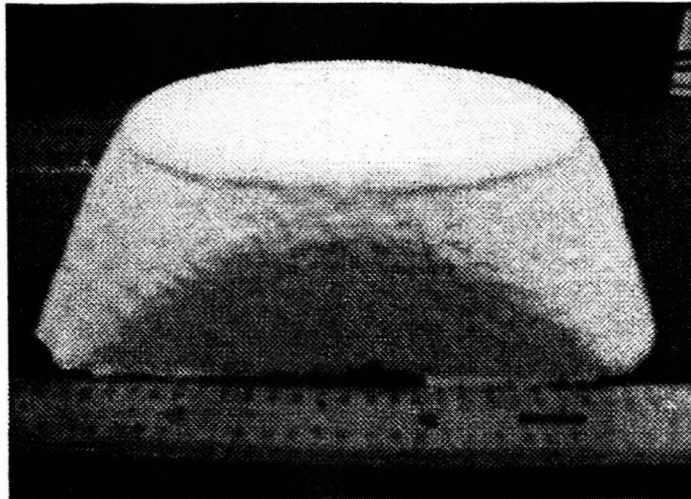
At this point, the decision was made to eliminate the Calcinated Alumina from the foams altogether. It was realized that a pure alumina sample would possess a higher melting temperature and consequently, a higher working temperature. Since the end product will be used as a refractory, a higher working temperature is much more favorable. The Alpha-Alumina was preferred to replace the CA because of high purity of alumina. A ratio of 30/70 Alpha Alumina/Alumina was selected because of the size distribution of the particles in the matrix. It is theorized that the 30/70 distribution will result in the closest packed cell walls. With the reintroduction of the Alpha-Alumina came shrinkage during sintering. Additionally, the Alpha-bond Alumina possesses a far greater surface area than CA so the water levels had to be adjusted.

A few experiments were made using differing water amounts. At this point, one of the best foams was made. The porosity was high and the pores were small. Further experiments were made to replicate this foam but were futile until it was found that the water content was variable. It was found that the water content alternated because the Alpha-Alumina was slowly re-hydrating. Every day that the Alpha-Alumina was exposed to the air resulted in a change. Additionally, the humidity of the laboratory changed the set time of the cement.

Shrinkage after sintering was still a problem so to combat this; the Alumina 1000 was eliminated in favor of tabular alumina. Tabular alumina, unlike the Alumina 1000, had already been sintered. Shrinkage improved but was not eliminated. Alumina spheres were added as well and were found to eliminate the shrinkage problem but introduced large pores and an increase in density.

To further increase the porosity, a product called Expancells were added to our foams. Expancells consist of a pre-expanded hollow sphere of plastic. The problem with the expancells is that they have such a low density; they float and are difficult to incorporate into the slurry. They have to be kneaded in by hand to prevent the particles from becoming air born in the mixer.

This light weight also causes them to float in the slurry and cause a dense layer on the bottom of the samples such as the one pictured below.



As it was stated afore, this project is not done; far from it in fact. I know that this may hurt the overview of this project but I would like to state that during my research, I had found a doctoral thesis on a very similar system. The variable contained in this system may take up to two years to complete.

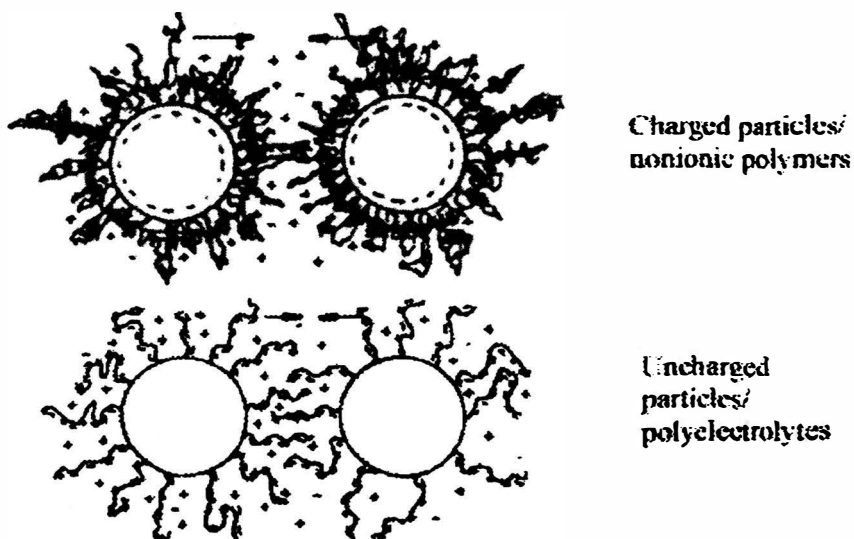
At this point, I am still happy with the results. It seems with each different experiment, a new problem is found which has to be solved. It can be frustrating as well. The next set of experiments is going to be designed such that the dense layer will be eliminated, strength measurements will start to be made, and the ability to replicate will be improved.

Nomenclature

Particles need to be of small uniform size to prevent gravity effects. Van der Waals forces are always present so larger forces are required to prevent the attractive forces, three ways of doing this are electrostatic, steric, and electrosteric stabilization.

Electrostatic stabilization is caused by the zeta potential of the particles in a polar solvent. Each oxide has its own PH at which the zeta potential. This point is called the Isoelectric point (IEP). If the solution has a lower PH than the IEP, then the surface of the oxide particles will adsorb H^+ ions out of the solution causing the particles to be positively charged, however, if the solution is at a higher PH than the IEP, then the surface will be negative caused by the adsorption of the OH^- ions. Since all the particles are of the same charge, then they will repel each other and the particles will remain well dispersed in the solution.

Steric dispersion is simply a charged polymer based chemical (commonly Darvan) that coats the surface of the particle. The coating prevents the particles from agglomerating. Electrosteric stabilization is a combination of the two and can be the most effective for the best dispersed solutions.



Brownian motion is the random agitation of particles due to heat.

Anionic Surfactants—Triton?

The molecules of the anionic surfactants are polarized on one side and negatively non-polar on the other side. The polarized side of the molecules creates H bonds with the water. The non-polar side is repelled out the other side. Since this additive is only semi-soluble in the water, the Triton creates a layer on the surface of the water. The Triton is now the upper layer and lowers the surface tension. The remaining Triton which gets dissolved in the water will bond to any molecules present in the solution. Once the surface is coated with Triton molecules, additional Triton will just be added on top. This explains why after a certain level of Triton, the surface properties do not change.

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