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Rheological properties of candle filter deposits at elevated temperatures and pressures

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**RHEOLOGICAL PROPERTIES OF CANDLE FILTER
DEPOSITS AT ELEVATED TEMPERATURES AND
PRESSURES**

**Thesis
Submitted to the College of Engineering and Mineral Resources
of**

West Virginia University

**In partial fulfillment of the requirements for the degree of
Master of Science in Chemical Engineering**

**by
Brian Robert Jordan**

**Morgantown
West Virginia
October 1998**

ABSTRACT

In order to improve the performance of hot gas ceramic candle filters, the rheological properties of two types of filter deposits were studied. Black powder from the IGCC (Integrated Gasification Combined Cycle) provided consistent data when sampled from two different sources. This powder's mechanical properties were independent of elevated temperature conditions, suggesting that the particle adhesion process is controlled by the rheological properties of the powder. Per US DOE request an apparatus was built to more accurately simulate candle filtration, and it was used to show that the process was unaffected by elevated inlet pressure conditions. The system was modeled by Darcy's law, yielding a permeability coefficient that varied as a function of porosity between 0, at an approximate porosity of 0.885, and $3.7 \times 10^{-11} \text{ m}^2$.

As part of an on-going project, PFBC (Pressurized Fluidized Bed Combustor) simulated filter cakes were prepared, expanding the work of Richman (1997). The new apparatus was used to show that these filter cakes were also unaffected by inlet pressure between the range of ambient pressure conditions and 3 atmospheres. SEM microscopy was used to examine the structure of these cakes, both before and after sintering, revealing a system in which smaller magnesium sulfate particles adhere to the surface of larger calcium sulfate crystals, and apparently densifying more at higher magnesium sulfate compositions.

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CHAPTER I.
INTRODUCTION

1.1 Background

The use and refinement of the application of hot gas ceramic candle filters is presently part of the R&D initiatives in both the Integrated Gasification Combined Cycle (IGCC) and Pressurized Fluidized Bed Combustor (PFBC) processes. Hot gas containing uncombusted particulates leaves the gasifier or fluidized bed combustor and must be cleaned before entering a gas turbine downstream in the process. One of the techniques under investigation for particulate removal is the use of the ceramic candle filter. Hot gas passes through the ceramic membrane and entrained particulates accumulate on the outer shell. These particulates are periodically removed by back pulses of air (every 20 minutes in the case of the IGCC process), but filter cleaning is sometimes incomplete, causing bridging of the space between adjacent filters (Newby 1995), ultimately resulting in mechanical failure of the ceramic filter itself. In order to facilitate more efficient use of the hot gas candle filter, it is necessary to gain a greater understanding of the filter deposit itself, and ideally develop a way to control its adhesive properties. It is for this reason that the Department of Energy contracts agencies to perform filter deposit characterization. The focus this work will be to examine the rheological properties of such deposits.

The powdered black filter deposits from the IGCC process will be studied and characterized. Simulated filter cakes will be prepared and tested, using the deposits removed from the candle filter; measurements of fracture strengths, deformation coefficients, and compression characteristics will be made and potential relations between any of these parameters will be discussed. Experimental conditions will be varied from ambient to elevated temperatures and pressures, in some cases simultaneously, in order to more accurately model those of the

process itself.

Next, a similar analysis of the PFBC process will be performed, which will expand on the work of Richman (1997). In this case, however, the inlet pressure to the simulated filter cake will be varied, as well as the temperature of the process (experiment). Once again using simulated filter cakes, the ratios of the major chemical components will be varied to examine their effect on the rheological parameters, as that the actual deposits themselves are not presently accessible. This portion of the work is intended to complement Richman's; not only is the pressure now being altered, but the use of simulated filter cakes of slightly different origin will assist in gauging the accuracy this technique. This might provide an indication of the variance to expect in comparing such work to other simulations or the actual deposit material.

In this work three major experimental techniques will be employed. The usefulness of destroying powder compacts, or tablets (briquettes), in order to gauge mechanical strength has been common in powder technology for years (sometimes referred to as the 'conventional' briquetting method). Similarly, the split cell apparatus has been in use since the early eighties (Yokoyama et al 1982), and is widely accepted. The packed bed method has been in development by Kono's research group for several years now, and its usefulness was established in the work of a previous graduate student (Richman 1997), not only in measuring fracture strengths, but applicable to the deformation coefficient as well. The uniqueness of this work is that it employs an adaptation of the packed bed technique that allows both the temperature of the simulated filter cake and the inlet pressure performing the compression to be varied simultaneously. This apparatus is introduced for validation and developed in the section on calcium carbonate, used briefly in the portion of this work examining the IGCC powder, and employed extensively in the

part devoted to the study of simulated PFBC deposits (Chapter 5). In these three sections characterization results will be presented, discussed and compared.

1.2 Research Objectives

Experimentation will begin with some preliminary work on calcium carbonate to achieve the following objectives:

- ! To measure the mechanical properties of calcium carbonate, with a focus on the rheological properties fracture strength (σ_f) and plastic deformation coefficient (Y) of the ‘quasi-solid’ body.
- ! To assess elevated temperature effects on these properties.
- ! To examine the effect of increased inlet pressure to the filter cake simulator on these properties, per US DOE request, and then to examine the combined effect of elevated temperatures and pressures.
- ! To compare these properties, showing their relation to one another when applicable.
- ! To compare the rheological properties of calcium carbonate lots 343-FC (Richman 1997) and 905 2-C in order to assess the amount of variance to expect in simulating filter cakes.

- ! To compare the results of the new filter cake simulator with previously established methods.

The IGCC powder will be studied with similar objectives:

- ! To measure the mechanical & rheological properties of the IGCC powder.
- ! To assess elevated temperature & pressure effects on these properties, first independently and then combined.
- ! To compare these properties, showing their relation to one another when applicable.
- ! To model the filter cake system using Darcy's law, solving for 'B,' the permeability coefficient, as a function of the filter cake's changing physical properties.

Expanding on the work of Richman (1997), the sintering magnesium sulfate/calcium sulfate system will be examined:

- ! To measure the mechanical/rheological properties on the same powders, but of different origin and preparation in order to gauge the variance in simulating filter cakes.

- ! To examine the effect of increased inlet pressure to the filter cake simulator on these properties, per US DOE request, and then to examine the combined effect of elevated temperatures and pressures.
- ! To compare these properties, showing their relation to one another when applicable.
- ! To examine the structure of this powder system, before and after exposure to sintering conditions, using scanning electron microscopy.

CHAPTER II.
BASIC CONSIDERATION & LITERATURE REVIEW

Powder technology as a branch of engineering contains a relatively large amount of unanswered questions. For although it has been studied for decades, the behavior of each powder is unique, making the task of forming generalizations to describe powder behavior difficult. On a fundamental level, the question “What is a powder?” still lacks a completely objective answer. One certainly has no difficulty naming an example of a powder, but if asked at what point a collection of particles was so largely and densely agglomerated that it was instead a pile of stones, the answer becomes complicated. Details such as scale or, in other cases, what type of medium the solid is present in are significant.

2.1 Powder Classification

Ironically, one of the most popular powder classifications is also the least stringent. In 1973 Geldhart lumped powders into four categories: A, B, C, and D, based upon density and average particle diameter. Type A powders were characterized as fine particles that were not only aeratable, but displayed a region of homogenous expansion between the minimum fluidization velocity, the point at which the weight of the powder is compensated for by the drag force of the fluidizing gas, and the minimum bubbling velocity, the point at which a bubble first appears in the structure of the aerated powder. Type B powders are generally coarser and bubbling begins as soon as fluidization is achieved. Type C particles, even finer than type A, exhibit strong cohesion forces. Because of the cohesive or “sticky” nature of these particles, they are difficult, if not impossible, to fluidize. Type D are the coarsest particles of all, typically the densest and largest particles, what one might think of as a “pile of stones” in an extreme case.

Some of the implications of this method of classification were considered in 1984 by Rietema, who showed that a two parameter classification was inadequate; citing a variety of other factors which influenced powder behavior (ie. - gas viscosity, gravity, gas adsorption, temperature), he produced results that didn't completely coincide with Geldhart's density/particle size descriptions. Based upon his findings he concluded that it would be more appropriate to describe powders as having A, B, C, or D-like behavior.

Before and since the time that the A/B/C/D classification was introduced, researchers have been trying to describe and predict powder behavior based on a variety of different physical properties. One that commonly shows up in the literature (Fuji, Yokoyama, and Yokoyama 1982, Tsubaki and Jimbo 1984) is the "tensile strength," based on the concept of a powder as an alignment of sub-micron spherical particles. Utilized since powder technology's earliest research (Rumpf 1958, Rietema 1967), tensile strength values were largely theoretical in nature because they could not be measured accurately or reproducibly; advances in the field over the last twenty years, however, have changed that. One example is the swing method cohesion tester (1982) which incorporates a suspended split cell, thereby eliminating any bearing friction that hampered experimental results in previous designs.

Another such property was introduced by Kono in 1994, who treated the bulk powder as a "quasi-solid body," and defined Y , its plastic deformation coefficient. It should be noted that this is not plastic deformation on the particle level, as described in compaction studies, but rather the powder as a whole is treated as one large solid body, beginning at its loosely packed state. The ratio of stress to strain is then measured in the deformation of this body as the "quasi-solid" undergoes particle rearrangement through either compaction or expansion. This type of

deformation is applicable in understanding the flow properties of deformable powders. Kono later produced an experimental relationship between the tensile strength and the deformation coefficient for aeratable powders (Kono 1996), and examined the effects of temperature and particle size on the two rheological parameters.

2.2 Rheological Properties: Tensile Strength and Plastic Deformation Coefficient

The tensile strength of a powder packing structure was originally presented in mathematical terms by Rumpf (1958):

$$\sigma_f = \frac{(1-\epsilon)}{\pi} k \frac{H}{d^{*2}} \quad (2.1)$$

Where:

- σ_f : tensile strength of the compact (Pa)
- ϵ : porosity or voidage of the bulk powder (-)
- k : coordination number, or number of particle contacts for a given particle (-)
- H : contact force at a single contact point (N)
- d^* : the effective particle diameter (assuming a uniform size) (m)

This equation gives an indication of the role voidage, particle diameter, and packing structure play in relation to tensile strength, but due to difficulties encountered in measuring H , it serves a mainly qualitative purpose. This equation is derived from a model that assumes a large number of perfectly spherical particles packed in a random distribution.

As mentioned previously, the plastic deformation coefficient (Y) as defined by Kono, treats the powder structure as a “quasi-solid body” in order to measure flow characteristics. This coefficient increases with decreasing porosity, and can be viewed as a measure of the “stiffness” or “rigidness” of a powder. Powders with large values of Y are dense particulate substances such as sand, which support a load with relatively little deformation or particle rearrangement, whereas those with small values of Y compact quite easily and may be of interest to those in pharmaceutical tableting. Using this criteria, the three generalized types of powders that will be dealt with in this work are shown in Table 1, as well as their associated characteristics and approximate rheological parameters.

Table 2.2.1: Powder Characteristics and Their Corresponding Rheological Parameters

	Coarse Particles	Fine Powder	Very Fine Powder
Approx. d_p range	$d_p > 100\mu\text{m}$	$100\mu\text{m} > d_p > 40\mu\text{m}$	$d_p < 20\mu\text{m}$
Aeratable	Yes	Yes	No
Fluidization Quality	Poor	Good	Normally Impossible
Compressibility	No	Slightly	Yes
Porosity (ϵ)	Constant	Changes Slightly	Changes Drastically
Cohesiveness	Not Cohesive	Slightly Cohesive	Very Cohesive
Bulk Powder	Stable, $\epsilon_{mf} \approx \epsilon_{mb}$	$\epsilon_{lp} \approx \epsilon_{mf} < \epsilon_{mb}$	Fragile, $\epsilon_{comp} \ll \epsilon_{lp}$
Tensile Strength (σ_f)	Approx. Zero	Small to Very Small	Large
Plastic Deformation Coefficient (Y)	Approx. Infinity	Large	Small

Although Table 1 provides a qualitative distinction between these three types of powder,

not all powders fit neatly into one of these categories. There is a great deal of uncertainty in distinguishing the border between fine and very fine powder; for although their behavior is quite different, there is no sudden change between the slight deformability of a fine powder and the drastic deformability of a very fine one. An example of such an exception is starch S (Kono 1994); although it has an average particle diameter of 15 μm , starch S aerates easily and expands homogeneously, a phenomenon which is not typical for such a small size particle. The difference between fine and very fine powder (Geldhart's Type A and C) was considered in great detail by Rietema in 1984. Rietema demonstrated that a powder's conditions could drastically alter its behavior; for example, a Type C powder might become aeratable, but only at an elevated temperature.

In 1996 Kono published an empirical equation that related the tensile strength to the plastic deformation coefficient, found in comparing twenty-some years of experimental work. He found that fluidizable powders lie on a particular line (Figure 2.2.1) when the two rheological properties are plotted, regardless of the powder or its origin. This equation, $\sigma_{f,mb} = 0.11Y^{0.89}$, was later correlated with a theoretical fracture derivation by Griffith and Irwin, and refined to its present form of $\sigma_{f,mb} = 0.1Y$.

Figure 2.2.1 shows the log-log relationship between $\sigma_{f,mb}$ and Y as well the line characteristic of smooth aeration. Kono tested his model and noted that elevated temperatures had the effect of increasing the Y and $\sigma_{f,mb}$ in a ratio that caused fine FCC to intercept the characteristic line and become aeratable. By the addition of fines, the tensile strength of a powder could be increased until particle agglomeration became so significant that it was no longer fluidizable.

2.3 Particle Adhesion in Powders

Particle adhesion in powders proceeds by several different mechanisms, described by Berbner and Löffler (1994) as those with and those without material bridges. Forces without material bridges include electrostatic forces, or the dissimilar charge draw/similar charge repulsion resulting from the build up of static electricity in a powder, and Van-der-Waals forces, which are the result of fluxuating electronic configuration, giving a particle dipolar character (Visser 1989). The significance of Van-der-Waals forces as the prevalent mechanism of adhesion in dry powder was discussed and estimated by Rumpf (1958) who found experimental results comparable to theoretical values calculated by both himself and others.

Forces with material bridges¹² are typically orders of magnitude larger than those without and consist of some sort of contacting liquid or solid interface between the particles. The addition of liquid binder is a common industrial practice; sometimes this binder is allowed to evaporate, resolving the liquid bridge into a solid structure. At temperatures exceeding approximately 60%

Figure 2.2.1: Fracture Strength vs. Deformation Coefficient, Temperature and Adhesion Effects

of a solid's melting point on the absolute scale (Frenkel 1945, Rumpf et al 1976), another phenomena of solid binding, sintering, can take place. In this case there is a substantial increase in tensile strength resulting from the formation of a solid bridge at the area of contact between two particles.

2.4 Sintering Mechanisms and Their Role In the Study of Hot Gas Candle Filtration

Siegell (1984) describes sintering as the migration of holes or lattice vacancies, or as a motion of atoms to a less dense area of the material, depending on how one wishes to view the mechanism. In studying the regeneration of candle filters at high temperatures, the effects of sintering appear to be one of, if not the most significant force in particle adhesion. This effect was analyzed by Berbner and Löffler (1994), Koch, Seville, and Clift (1996), Kono (1997), then later expanded by Richman (1997). Berbner and Löffler noticed an increase in tensile strength with temperature and estimated the force enhancement due to sintering adhesion to be approximately 63 times greater than that of the Van-der-Waals force. Candle filters are often operated under elevated temperatures and compressive forces that increase with filter cake loading; this suggests the possible presence of a sintering enhancement in the filter cake, if not some additional force contribution from viscoelastic flattening and/or chemical effects. According to Frenkel (1945), sintering kinetics are determined only by the lowering of free surface energy and viscous dissipation energy, but in the case of candle filters there is an enhancement from the compressive force of the gas. Also of consideration to fine fly ash particles, Troitskii (1983) has shown that as particle size decreases, the minimum sintering temperature decreases exponentially.

When the sintering growth of a contact point occurs between two particles it typically

proceeds by one of four different mechanisms: surface diffusion, volume diffusion, viscous flow, and vaporization (Siegel 1984). These mechanisms are physically different, and they can all occur simultaneously, but one particular will normally dominate, depending upon the situation. Diffusive sintering occurs when atoms move from regions of high density to those of low ones. Surface diffusion has the lowest activation energy of any of the diffusive mechanisms and typically results in the formation of agglomerated particles. This is then followed by volume diffusion, or the densification of the agglomerate. Material transport by viscous flow is due to the effect of surface tension, which causes material to be transported from convex to concave regions (Siegel 1984, Ristić and Dragojević-Nešić 1987). It is thought to occur by the movement of lattice planes, as opposed to single atoms. Materials that tend to creep (permanently deform due to loads applied below their yield stress) experience viscous flow under pressure (i.e. - the compressive drag force exerted on a loaded filter cake), the rate of which is determined by the viscosity of the material. Transport into the vapor phase is another possible mechanism, but probably the least important in consideration of hot gas filtering.

2.5 Bulk Powder and Powder Compacts

The term “bulk powder” is used to specify a collection of particles as a whole. The same definition could be used to describe the individual particle in the bulk being a collection of smaller, ultimately primary particles, but there is a matter of scale considered by the use of this term. The term “bulk powder” is commonly used to reference a collection of particles that will be handled as a whole or observed as a group.

Treatment of powder as a single entity or “bulk” is useful in powder characterization, the

“bulk powder” can be characterized by certain average properties that describe the collection as a whole, and in some cases (i.e. - average particle diameter in Rumpf’s equation) used to estimate other useful physical properties. Examples include average density, average surface area, average shape factor, and average (or overall) voidage.

In the fluidization theory of Kono (1994), the aerated bulk powder is treated as a single entity or “quasi-solid body” which expands and undergoes deformation under a stress delivered by the drag force of the fluidizing gas. This “quasi-solid body” expands under tension (gas drag pulling in one direction, gravity opposing), in the fluidized bed, until its tensile strength is exceeded and the first fracture forms, at the minimum bubbling point.

Other applications of the idea of “bulk powder” are prevalent in the pharmaceutical industry, where an amount of powder is treated as a whole as it undergoes compression. Calculation of stresses and their effected strains in tableting dies are constantly being studied, as well as die friction, types of deformation, necessary energy input, and performances indices. In this case the idea of the original “bulk powder” is still the same, but reduced in size by orders of magnitude.

In this work properties of bulk powder are studied. Although each powder will be characterized by conventional physical parameters, such as particle density and voidage, special attention will be paid to the rheological parameters of Y and σ_f . In an effort to understand the flow properties of powders, specifically those of candle filter deposits, it is hoped to gain a greater understanding of their behavior and to provide a foundation to improve the performance of hot gas candle filters.

CHAPTER III.
THEORY

3.1 The Packed Bed Method and Its Model

This method has been under development for many years by Kono's group; many of the assumptions and experimental techniques that will be explained are the result of preliminary experiments and years of work, sometimes on a trial and error basis. Based upon these works and their results, the appropriate assumptions were chosen.

In order to obtain a homogenous powder packing, the powder being tested is screened into a cylinder (in some cases a fluidized bed) through a fine sieve so that it rests on a porous plate at its loosely packed bed height H_{lp} . This 'quasi-solid' body is said to consist of N unit layers, specified by its effective particle height d_p^* . Originally considered to be the size of the screen through which the powder was sifted (Richman 1997), this effective particle height is now more accurately determined by extrapolation of briquetting (tableting) and split cell data on a semi-log mechanical strength vs porosity plot (when all three methods are applicable). The necessity of the use of an effective particle height stems from the agglomeration tendency of fine particles. Regardless of the initial sieve size (assuming it was below the effective particle diameter) the experimental results of the packed bed method were consistent, implying that once the experiment had begun some sort of restructuring was taking place, described here by N , the number of effective unit layers. Regardless of the method through which the effective particle size is determined, the number of unit layers is defined as $N_{lp} = H_{lp}/d_p^*$, and it is assumed that N is constant throughout the experiment, or the change in height of the powder bed is directly proportional to the change in effective particle size.

Next, gas flow is introduced downward through the powder bed compressing it against a porous plate. The bed is compressed to a bed height of H_c , with equivalent unit layer height of d_c^* . The compressive gas pressure drop across the powder bed, ΔP_c^* , can be used to calculate the same quantity on a unit layer basis, ΔP_c^{**} , by dividing by the quantity N . By adjusting the gas velocity through the powder compact, the porosity of the resulting cake can be controlled to a limited degree, in preparation for the fracture test.

Once compression is complete, gas flow is halted and the pressure on either side of the powder cake is allowed to equilibrate. Gas flow is then introduced, at a greatly reduced rate, in the opposite direction and an unsteady-state pressure difference begins to build over the length of the powder cake. At some point the ultimate mechanical strength of the powder compact is exceeded and the initial fractures in the bed begin to form. Although it is more practical to measure the mechanical strength (σ_f) and the plastic deformation coefficient (Y) experimentally, these rheological parameters can be expressed theoretically as follows.

During compression of the powder from its loosely packed to a compressed state, the gas flow pressure drop rises to ΔP_c^{**} per unit layer

$$\Delta P_c = \Delta P_c^* / N_c \quad (3.1)$$

where ΔP_c^* is the pressure drop across the bed after compression. N_c , the number of unit layers in the compressed state, is approximately the same as N_{lp} , the number of unit layers in the loosely packed state. N_c can be calculated by dividing the compressed height, H_c , by the effective particle diameter in the compressed state, d_c^* , assuming spherical particles. For the purpose of simplification and consistency, the number of compressed unit layers is approximated as the

number of loosely packed unit layers.

$$N_{lp} \approx N_c \quad (3.2)$$

By introducing the effective diameter d_c^* for powders in their compressed state, the effective pressure drop per unit layer can be expressed by combining the Ergun equation with Equation (3.1)

$$\Delta P_c^{**} = \frac{150 H_c \frac{(1-\epsilon_c)^2}{\epsilon_c^3} \frac{\mu U_c}{(\phi_s d_c^*)^2}}{N_c} \quad (3.3)$$

where ϵ is the bed porosity, μ is the gas viscosity, U_c is the gas velocity, and ϕ_s is the shape factor of the effective diameter. Under these compressive conditions, the plastic deformation coefficient (Y) can be expressed as

$$Y = \frac{\frac{\Delta P_c^{**}}{H_{lp} - H_c}}{H_{lp}} = \frac{\frac{\Delta P_c^*}{N_c}}{\frac{\Delta H}{H_{lp}}} \quad (3.4)$$

During the fracture test the air flow is reversed and the fracture strength can be expressed as

$$\sigma_f = \Delta P_f^{**} = \frac{\Delta P_f^*}{N_f} \quad (3.5)$$

where ΔP_f is the gas pressure drop across the bed when the fracture occurs, and N_f is the number of effective unit layers at that time. As previously mentioned, the number of unit layers is approximated as constant during both the compression and fracture processes.

$$N_f \approx N_{lp} \approx N_c \quad (3.6)$$

Substituting in the Ergun equation for the pressure drop over the length of the powder compact, the fracture strength per unit layer can be expressed as follows:

$$\sigma_f = \Delta P_f^{**} = \frac{150 H_f \frac{(1-\epsilon_f)^2}{\epsilon_f^3} \frac{\mu U_f}{(\phi_s d_f^*)^2}}{N_f} \quad (3.7)$$

Although the rheological properties utilized in this work can be described, and in some cases calculated, with the above equations, these properties will be obtained directly by experiment.

An alternative method exists to measure these properties for fluidizable powders. Details are presented in the M.S.Ch.E. Thesis of Richman (1997).

3.2 Darcy's Law

Originally derived from the study of water flow through sand beds of various thicknesses, Darcy's law relates the average velocity of fluid flow through a packed bed to the pressure driving force and the length of the bed.

$$u_c = K \frac{(-\Delta P)}{l} \quad (3.8)$$

where ΔP is the pressure drop over the length of the bed, l is the length of the bed, and u_c is the average velocity of the fluid as measured over the area of the bed. K , a physical constant depending on the physical properties of the bed and fluid, can be alternatively expressed as B/μ when resistance to flow arises mainly from viscous drag.

$$u_c = B \frac{(-\Delta P)}{\mu l} \quad (3.9)$$

where μ is the viscosity of the fluid. B , termed the ‘permeability coefficient,’ depends only on the physical properties of the bed and it provides an index of the ease with which a fluid will flow through the bed (Coulson et al 1993).

This model will be discussed further and applied to the IGCC process in section 5.2.

CHAPTER IV.
EXPERIMENTAL METHODS

4.1 Mechanical Strength

There are three methods employed in this work to measure the mechanical strength of simulated filter cakes. The first of these is a test of ultimate compressive strength on the powder compact, conventionally referred to as the briquetting method, or tableting, as designated here.

Figure 4.1.1 Acrylic Die w/ Ring Clamp & Piston

Utilizing the die shown in Figure 4.1.1, cylindrical compacts of the substance being tested were formed. To promote particle adhesion during formation of the powder compact, a light molecular weight organic binder was utilized and then allowed to evaporate under ambient, and then under slightly elevated temperature conditions (120 °C). After cooling over desiccant,

tablets were compressed with a Soiltester compression apparatus, and their ultimate compressive strength recorded. The range of tablet porosity was varied slightly by adjusting the amount of liquid used as binder and the amount of compressive force applied during formation, and care was taken so as not to expose the powder compact to elevated temperature conditions long enough to allow sintering (when applicable) as shown by Richman (1997).

Strictly speaking, this compressive mechanical strength is not exactly the same as the ‘tensile strength’ measured by the split cell apparatus or the drag exerted ‘pressure drop per unit layer’ exerted by a flow of gas, but it was shown to be directly comparable in a work by Soltani-Ahmadi (1986). Based upon an equation found by Hiramatsu (1965) for a relationship between

Figure 4.1.2: Split Cell Cohesion Tester Apparatus

the durable static compressive force and tensile strength of a spherical granule, Soltani-Ahmadi verified the equation experimentally with his powders and showed its consistency with Rumpf's equation (Equation 2.1). This resulted in a difference between the compressive and tensile strength of less than an order of magnitude. Because of these findings, the tensile strength in this work (and work previous to this) will be approximated at the durable compressive strength in the lower range of porosities, which show little to no significance on a semi-log plot.

Figure 4.1.3: Typical Split-Cell Read Out

For an intermediate range of porosities, the split cell apparatus shown in Figure 4.1.2 was used. This device, commercially available from Hosokawa, measures the tensile strength of a powder compact by means of a 'split cell,' as indicated by its name. The 'split cell' in which the powder compact is formed consists of a stationary side and a suspended side, attached to a motorized pulley by a string. The string is pulled at a constant rate and it passes through a

FIGURE 4.1.4: PACKED BED FRACTURE METHOD

calibrated force transducer which measures the force required to fracture the powder compact.

The signal from the transducer is amplified and sent to an X-Y recorder for display. An example of a typical split-cell readout is shown in Figure 4.1.3; the maximum in the curve corresponds to the ultimate tensile strength of the powder compact, and the entire force applied can be found by integration of the curve.

In the highest range of porosities, the packed bed method is utilized. As described in Chapter 3, powder is passed through a screen (600 micrometer in these cases) into a quartz column approximately 2 inches in internal diameter. When this is completed, some effort is made to gently level the powder surface and form a uniform area for height measurement (to be discussed in the next section). After the powder undergoes compaction against the distributor

plate with a current of air, the dimensions of the final compact are noted for calculation of the porosity. The air flow is then reversed and the ultimate pressure difference over the simulated filter cake is recorded as it fractures simultaneously in several places. By dividing this quantity by the number of effective unit layers, this ultimate pressure difference can be converted to the fracture strength per unit layer at the calculated porosity. An undetailed schematic of this process, performed with a fluidized bed, is shown in Figure 4.1.4, and was a considerable part of a work by Richman (1997).

4.2 Plastic Deformation Coefficient of the Quasi-Solid Body

Another rheological property to be considered in this work is Y , the plastic deformation coefficient of the ‘quasi-solid’ body. This property was originally measured using the apparatus shown in Figure 4.1.4. As in the case of the mechanical strength measurement (and actually as part of the same experiment), powder is screened into the column and leveled off. Beginning in its loosely packed state, a current of air is applied downward through the column, compressing the powder against the distributor plate. The pressure drop over the ‘quasi-solid’ body is measured by means of a water manometer. This pressure drop is normalized by dividing by the effective number of unit layers. Based upon the change in height with each successive pressure increase, a strain can be calculated, and ultimately used to calculate a deformation coefficient for each new porosity. This plastic deformation coefficient, Y , gives an indication of the rigidity, or stiffness of the powder, at each decreasing porosity. As mentioned previously, when compression is complete the air flow is halted and then reversed in order to measure the mechanical strength of the powder compact.

Figure 4.2.1: Elevated Temperature & Pressure Packed Column Fracture Apparatus

The quartz column (shown in Figure 4.1.4) has optional heating elements which allow for experiments to be conducted at elevated temperature conditions. More recently, however, there has been an interest in examining the effect of the inlet pressure on the rheological properties, and the simultaneous effect of elevated pressure and temperature. It was for this reason the apparatus shown in Figure 4.2.1 was constructed and to which a considerable portion of this work is devoted.

As in the case of the quartz column, powder is screened into the apparatus, this time a steel column of approximately 10 cm in inside diameter. When this is complete, the powder surface is leveled with a straight-edge and the stainless-steel mid-section and lid are bolted onto the base, sealed with high a temperature automotive gasket. This apparatus is unique in that the inlet pressure during compression can be varied by way of the regulator attached to the nitrogen

tank. Also, using the small internal band heater wrapped about the column, the effects of increased temperature and pressure can be examined simultaneously. Unlike the quartz method, the entire apparatus is under pressure; at the beginning of the experiment the apparatus must be slowly pressurized, keeping the initial applied pressure over the cake less than the pressure ultimately to be applied in the experiment. Also, unlike the quartz column, each experiment allows for only one applied pressure/change in height measurement, because the steel column and its casing are not transparent and repetitive opening and resealing of the steel casing jars the powder structure. By performing multiple experiments and noting the height change in the powder compact for each applied pressure (since the starting height is exactly the same in each case), the plastic deformation coefficient can be approximated, as with the quartz column, but with much more effort.

4.3 Microscopic Observation

For the purpose of visually examining the sample powders, two forms of microscopy were utilized. Classic optical observation was performed by using a BH-2 model microscope, manufactured by Olympus, capable of magnifying images up to 500 X. Also Scanning Electron Microscopy was conducted at NIOSH, utilizing a JEOL JSM-6400 scanning microscope, courtesy of Mr. Joel Harrison.

CHAPTER V.
RESULTS & DISCUSSION

The powders tested in this work are significant in not only that they represent the deposits of two candle filter utilizing processes, but three different powder systems are demonstrated. The calcium carbonate powder can be thought of as the ideal or inert system. Calcium carbonate does not adsorb an amount of water significant enough to affect its rheological properties and behaves well in experimental situations. As typical of fine powders, calcium carbonate demonstrates ‘sticky’ or cohesive properties and agglomerates easily putting its bulk mechanical properties in a range that does not require the use of ultra-sensitive instruments to measure. The powder did show some temperature sensitivity, but as that none of the candle filter systems retained calcium carbonate as a deposit it was utilized here mainly for the purposes of instrumental development and in demonstrating method consistency.

Similarly, the IGCC powder demonstrated no change in physical properties in the elevated temperature and pressure ranges, but was not inert. When exposed to elevated temperatures in an open environment the sticky black coating surrounding these particles was burned away, necessitating the need for these experiments to be carried out under a blanket of nitrogen gas. After combustion, the remaining fine, white powder still agglomerated, but adhered to surfaces (i.e. - crucibles, fingers) to a much lesser degree.

The magnesium sulfate/calcium sulfate mixtures originally examined by Richman (1997)

depict a temperature sensitive system. An increase in mechanical strength is observed with exposure to elevated temperatures, stemming from not only a decrease in porosity of the powder compact, but from solid bridge formation as well (as opposed to calcium carbonate, whose increase in tensile strength appears to stem only from a voidage reduction). This work was actually performed as a series of sub-sections, examining the effect of composition as well, but as will be seen in Section 5.3.2, composition did not show a clearly defined effect on the rheological properties over the ranges examined in this work. The pure components are considered, and the effect of 1:1, 1:2, and 2:1 molar ratio mixtures are shown.

In this case, as well as that of the previous two, the effect of elevated inlet pressure to the packed bed apparatus demonstrated no effect. This is of no surprise upon inspection of Darcy's law (Equation 3.9); the only physical property of the compressing fluid that might show significance is that of the viscosity, which varies less than one fifth of an order of magnitude between ambient temperature and 515 °C. This portion of the work is meant to compliment Richman's (1997) and was carried out at the request of the U.S. Department of Energy.

5.1 Calcium Carbonate

Although calcium carbonate is not present in the chemical composition of the filter deposits in either the PFBC or IGCC process, a portion of this work will be devoted to the study of its rheological properties for two important reasons. First, it is inexpensive and easy to handle; calcium carbonate was employed in testing and development of several experimental methods used in this work (i.e. - the original acrylic and then steel column in the elevated pressure casing). Unlike the sulfate powders, it does not readily adsorb water. When testing the tensile strength of calcium carbonate by the split cell method, the experimental procedure does not require working so quickly that accuracy is compromised. Also, in all three mechanical strength methods the powder does not need to be dried before use to produce consistent results. All experimental tests utilized in this work can be performed on calcium carbonate, whereas the unsintered mechanical

Figure 5.1.1: Semi-Log Line Showing the Consistency of Three Mechanical Strength Methods

strength of sulfate tablets is too weak to be measured with the Soiltester compression apparatus, if it was unaffected by water absorption.

A semi-log plot of mechanical strength vs porosity is shown in Figure 5.1.1. As discussed in literature (Tsubaki and Jimbo 1984), a plot of mechanical strength vs porosity forms a linear relation on a semi-log plot. By plotting both the split cell and tableting (briquetting) data, a line can be extrapolated into the range of higher porosities and used to determine the number of effective unit layers. Alternatively, when only two of the methods can be used, as in the case of the sulfate powders, the line can be extrapolated based upon the slope of the split cell data alone, although this method allows more leeway for error and inaccuracy, and requires much more data.

It should be noted that not all calcium carbonate produces well grouped similar results. The mechanical strength of a sample will vary according to powder properties, including particle

Figure 5.1.2: Comparison of Calcium Carbonate Lots

Figure 5.1.3: Temperature Effect On Y for Calcium Carbonate

size distribution, average shape factor, and homogeneity as examples. This deviation is illustrated in Figure 5.1.2 with the packed bed data. Although the mechanical strength lies in the same general area and seems to demonstrate a similar, if not identical slope, lot# 00905 2-C is shifted slightly to the right of lot# 343-FC. Also shown, when the mechanical strength of calcium carbonate is measured at increased temperature, it appears to increase after only 3 hours of heating, moving to the right and gradually up the chart as it becomes less compressible and more adhesive.

A more significant difference is observed in Y, the plastic deformation coefficient of the quasi-solid body, with increasing temperature. Figure 5.1.3 illustrates the change in Y with increasing temperature. In all experiments the powder began at its loosely packed state of 75%

porosity (plus or minus 0.5%). As the temperature of the simulated filter cake increased, the compact became less deformable (compression resulting in particle rearrangement) before reaching at its ultimate height where Y rises to infinity. This behavior is also demonstrated in Figure 5.1.4, where for any given applied pressure (per unit layer), the deformation of the compact decreases with increasing temperature during the experiment. Although the exact mechanism of this (and the increased tensile strength phenomena) is not known, it is suspected to be the result of liquid bridge formation at elevated temperature. An increase in the number (or size) of the contact points would certainly explain a tensile strength increase (as indicated by Equation 2.1), and the increased contact area might impede particle rearrangement, retarding compression.

Figure 5.1.5 shows the deformation coefficient of the two different calcium carbonate lots

Figure 5.1.4: Change In Powder Compact Height With Applied Pressure

Figure 5.1.5: Comparison of Y With Richman's Data (CaCO₃)

to be almost identical. Richman's data (lot# 343 FC) was measured using an acrylic column

(approximately four times the size of the present column) and over the course of a single experiment, as indicated by the smoothness of this curve in comparison with lot# 905-FC. The deformation coefficient for lot# 905-FC was measured using the steel column over the course of several experiments, one for each data point.

Up until this point, the deformation of the simulated filter cake has been discussed in terms of 'applied pressure.' What is meant by this term is actually ΔP_c^{**} , the pressure drop per effective unit layer, not to be confused with ΔP_c^* , the pressure drop over the entire powder body. As this is increased, by controlling the gas flow through the powder compact, the compact

structure rearranges and it becomes smaller and less porous. Thus, within a limited range, the porosity of the filter cake to be studied can be controlled. A plot of ΔP_c^{**} vs porosity, shown in Figure 5.1.6, looks similar to a Y plot in that it climbs to infinity at the lower range of porosities. Some limiting lower value of the porosity is normally attained by this method (gas flow compression), before any significant or observable viscoelastic deformation. Also, as with the deformation coefficient, the curvature of the line decreases with increasing temperature as the compact becomes less deformable.

There is also a semi-log relation between the fracture strength per unit layer over the applied pressure per unit layer, ΔP_c^{**} , vs porosity. Shown in Figure 5.1.7, this line is representative of a stress hysteresis effect in comparing the applied and recoverable energy. Figure 5.1.7 also shows the temperature effect on this line; with increasing temperature the line appears to shift to the right, although at this point it is not entirely clear whether this is the case or that the slope of the line is simply decreasing.

Allowing for a reasonable amount of scatter expected over the course of several experiments, Figure 5.1.8 shows the deformation coefficient to be unaffected by the inlet pressure to the apparatus. This figure also shows the consistency of the previously used quartz column with the present method (steel column) in measuring the deformation coefficient. Figure 5.1.9 illustrates this same behavior with the compressive pressure per unit layer vs porosity graph, both at ambient and elevated temperatures. Although there is a trend for use of the blower at lower compressive pressures and the N_2 tank at higher compressive pressures, this is merely the result of the practical inability to throttle the nitrogen air flow down below a pressure difference of 1 psi over the simulated filter cake and measure it accurately.

Figure 5.1.6: Compressive Pressure Per Unit Layer vs Porosity for CaCO_3

Figure 5.1.7: Temperature Effect on Stored Stress Hysteresis (CaCO_3)

Figure 5.1.8: Effect (or lack of) of Inlet Pressure on Y for CaCO_3

Figure 5.1.9: Compressive Pressure Per Unit Layer vs Porosity, Inlet Pressure Effect for CaCO_3

5.2 IGCC Powder

In the Integrated Gasification Combined Cycle system, coal is converted into a gaseous fuel to remove sulfur. Hot gas exiting the gasifier must be cleaned up before combustion in a gas turbine; ceramic candle filters are one of the methods under study and development for particulate removal. Fine black powder consisting of sub-micron particles accumulates on the surface of these filters and is periodically back pulsed off under ideal operating conditions. Analyzed by Praxis Engineers Inc., composition of this powder from three different IGCC processes is shown in Table 5.2.1 (Praxis Engineering 1996).

Table 5.2.1: IGCC Waste Compositional Analysis as Oxides (Weight Percent Dry Basis)

Sample	I	II	III
Loss @ 105 °C	0.5	0.32	12.0
SiO ₂	34.7	48.77	42.9
Al ₂ O ₃	21.82	18.39	16.5
Fe ₂ O ₃	17.2	19.96	14.2
CaO	3.85	6.39	9.1
MgO	0.93	1.01	1.16
K ₂ O	1.5	1.96	2.17
Na ₂ O	0.43	0.78	0.73
SO ₃	1.61	0.6	1.45
P ₂ O ₅	0.74	0.21	0.13
TiO ₂	1.24	0.94	0.81
Mn ₂ O ₃	0.09	0.15	0.09
Loss @ 900 °C	15.74	0.75	10.3
Carbon	17.94	not specified	not specified
Total	99.85	99.91	99.6

Figure 5.2.1: IGCC Powder Agglomerates

Table 5.2.2, and surprisingly the sample weight was *exactly* the same (within the accuracy limitations of the balance) after the second ignition.

Analysis of samples sent to our lab indicate a higher loss on ignition at 900 °C. Samples were heated initially for 18 hours at approximately 905 °C, and then put back in the oven for an additional 16 hours to ensure they had reached constant weight.

Results of this test are shown in

Table 5.2.2: IGCC Powder LOI (Loss on Ignition) @ 900 °C

Sample	#1 (11/7/96)	#2 (1995)	#3 (1995)
Sample wt.	0.23 g	0.93 g	0.54 g
Wt. of Loss	0.1 g	0.36 g	0.22 g
Ig. Loss	43.5 %	38.7 %	40.7 %

Electron microscopy reveals this powder to be a collection of extremely fine particle agglomerates. Figure 5.2.1 and Figure 5.2.2a show the particles adhering together. In Figure 5.2.2a a roughly spherical particle of less than one micron can be seen just above the size bar. While there is no evidence that this is even on the order of the primary particle size, it is indicative of the powder's wide size distribution and cohesive nature of the light particles. Figure 5.2.2b

Figure 5.2.2a: IGCC Powder Submicron Particles

Figure 5.2.2b: IGCC Powder Agglomerates after LOI

shows these same particles still aggregating, even after their sticky, volatile black coating had been removed during the loss on ignition testing.

As in the case of calcium carbonate, all three methods of determining mechanical strength were applicable to the carbon-like powder, making the task of determining an effective particle diameter relatively easy, regardless of the presence (or absence in this case) of a wide data scatter in the split cell range.

Figure 5.2.3 shows not only a clean fit to the mechanical strength semi-log line, but also how the powder properties were unaffected

in the presence of increased pressure and temperature. Points designating data that are the result of elevated pressure or temperature conditions are designated in the legend, and the first experiment with the combined effect apparatus (steel column) is also shown.

Figure 5.2.3: Mechanical Strength of IGCC Powder

Figure 5.2.4: Deformation Coefficient of IGCC Powder

Figure 5.2.5: Compressive Pressure Per Unit Layer vs Porosity for IGCC Powder

As in the case of the mechanical strength, elevated temperature or pressure bears no reflection on the plastic deformation coefficient. However, unlike calcium carbonate, the deformation coefficient calculated by use of the acrylic dye briquetting apparatus (tableting die) coincides with data from the packed bed methods -- both the quartz and the acrylic column originally in the elevated pressure casing. This is suspected to be the result of lubricative properties of the powder; stemming from its submicron particle size, unlike the larger grains of the sulfate powders which could be heard grinding between the piston and the walls of the die when compressed.

Figure 5.2.5 illustrates how the porosity of the filter cake in the candle filter system can be

controlled as a function of the pressure drop over its length. This plot is normalized to the unit layer basis for comparison to different operating conditions (i.e. - a longer time in between back pulses results in a thicker filter cake). Figure 5.1.5, for calcium carbonate, also becomes linear when converted to a semi-log plot, but for the purposes of comparison to Richman's data and relation to the pressure per unit layer vs porosity figure (Figure 5.2.5), it was not. Regardless of the compression method or operating conditions used, the pressure per unit layer vs voidage values all lie on the same characteristic line when shown on a semi-log plot.

This powder also demonstrates the characteristic fracture/compression hysteresis ratio as a function of porosity, as shown in Figure 5.2.6. Once again, experimental values from several different methods lie on the same characteristic line, regardless of the temperature and/or pressure conditions.

Figure 5.2.6: Characteristic Recovered Over Applied Strain Ratio vs Porosity for IGCC Powder

One practical application of this work is illustrated in the following example. Gas flow (gas velocity) through the filter cake results in a compressive pressure per unit layer (or pressure per unit length, depending on how it is calculated), indirectly controlling the voidage of the filter cake, as presented in Figure 5.2.5. Knowing the pressure drop across the candle filter and height or thickness of the deposit, the porosity of the resulting cake can be calculated.

Given the data set of gas velocity, pressure drop per filter cake thickness, and its corresponding porosity, this filtration system can be described by Darcy's law. Equation 3.8 shows how gas velocity is proportional to pressure drop per unit thickness through K , which is dependent on the physical properties of the bed and the fluid. Figure 5.2.7 shows this plot and how it was modeled over the range of data by a second order polynomial function ($R^2=0.9658$). Similarly, Figure 5.2.8 shows the exponential modeling of the pressure drop vs porosity plot

Figure 5.2.7: Darcy's Law Plot, Determination of Permeability Coefficient

($R^2=0.9379$). Combining the slope of Figure 5.2.7 with the data in Figure 5.2.8, K, the characteristic physical parameter can be calculated, as shown in Figure 5.2.9. Using the viscosity of the gas, the proportionality's dependence on the fluid can be eliminated. B, sometimes called 'the permeability coefficient' depends only on the physical properties of the bed and is shown in Figure 5.2.10.

Using this model and Figure 5.2.6 to estimate the amount of energy that will be required to fracture the deposit, the hypothetical gas velocity necessary to fracture the filter cake can be calculated. This plot is shown in Figure 5.2.11 for the range of porosities investigated.

Figure 5.2.8: Pressure Drop Per Unit Length vs Porosity for IGCC Powder

Figure 5.2.9: Darcy's Law Proportionality K , Dependent On Bed & Fluid Properties

Figure 5.2.10: Darcy's Law Proportionality B , Dependent On Bed Properties Only

Figure 5.2.11: Hypothetical Calculated Fracture Velocity vs Porosity for IGCC Powder

5.3 PFBC Simulated Powder

5.3.1 Particle Morphology

In Measurement of Rheological Properties of Bulk Powder at Ambient and Elevated

Figure 5.3.1.1: SEM Photo of MgSO4 Powder @ 1400X

Temperature, Richman prepared simulated filter cakes consisting of the two main components in the hot gas stream leaving the fluidized bed combustor (magnesium and calcium sulfate). These ratios were varied in molar proportions in order to gauge the effect of each component and a sintering effect was demonstrated through experiments performed at or subjected to elevated temperatures. At the

conclusion of this work there was some question as to whether the inlet pressure would have any bearing on the rheological properties of the powder compact, and that is the focus of this section.

As in the previous work, simulated filter cakes were prepared by mixing chemical grade (99%+ pure, Sigma) magnesium sulfate and calcium sulfate powder in 1:1, 1:2, and 2:1 molar ratios. All preparations were screened and thoroughly dried before and after mixing, and not reused after exposure to elevated temperature conditions.

Figure 5.3.2.1 shows a SEM photograph of the magnesium sulfate powder at 1400X. As would be expected of

5.3.1.2: SEM Photo of CaSO4 Powder @ 1400X

5.3.1.3: SEM Photo of CaSO₄ Powder @ 1400X

5.3.1.4: SEM Photo of IGCC Powder @ 1400X

5.3.1.5: SEM Photo of 1:1 Ratio CaSO₄/MgSO₄ Mixture @ 1400X

such fine particles, they agglomerate into larger clumps. Calcium sulfate, in comparison, seems to be composed of a more crystal-like structure and a wider size distribution. Figures 5.3.1.2 and 5.3.1.3 show small calcium sulfate particles that rival the size of the magnesium sulfate particles, but visual inspection indicates a much larger average particle diameter overall. Both of these

powder's average particle size dwarf the IGCC powder, shown in Figure 5.3.1.4, also at 1400X.

Inspection of the powder mixtures studied in this work, at the same magnification as their pure components, show the same trend. Figure 5.3.1.5 and Figure 5.3.1.6 show smaller particles adhering to the surface of considerably larger ones. The small particles, mainly magnesium sulfate, as judged by the smaller quantity of fines shown in the calcium sulfate pictures, coat the larger calcium sulfate crystals and begin to form agglomerates with other similarly coated particles.

This behavior is even more obvious in Figure 5.3.1.7 and Figure 5.3.1.8. When the magnesium

**5.3.1.6: SEM Photo of 1:1 Ratio
CaSO₄/MgSO₄ Mixture @ 1400X**

**5.3.1.7: SEM Photo of 1:2 Ratio
CaSO₄/MgSO₄ Mixture @ 1400X**

**5.3.1.8: SEM Photo of 2:1 Ratio
CaSO₄/MgSO₄ Mixture @ 1400X**

sulfate content is increased (Figure 5.3.1.7), the larger particles show more coating, and the fine particles bridge the gaps between separate agglomerates. In contrast, when the calcium sulfate proportion is increased, more surface area of the larger crystals are exposed and the agglomeration tendency is less pronounced (Figure 5.3.1.8).

Figures 5.3.1.9 through 5.3.1.13 show the effect of sintering on the material. As in Richman's work, the powders and their mixtures were exposed to elevated temperature (750 °C) for 8 hours. As expected there was some densification and the formation of solid bridges in all five cases, but seemed to be affected to a different degree.

Sintered magnesium sulfate shows even more agglomeration than the unheated sample. The SEM photograph illustrates the not only the effect of the heat densification and larger agglomerate size, but upon close inspection the presence of capillary-like solid bridges can be detected (although this may be difficult in this publication, it is more evident in the actual

Figure 5.3.1.9: SEM Photo of MgSO₄ Powder, Heated @ 750 °C for 8 hrs.

Figure 5.3.1.10: SEM Photo of CaSO₄ Powder, Heated @ 750 °C for 8 hrs.

Figure 5.3.1.11: SEM Photo of 1:1 CaSO₄/MgSO₄ Powder Mixture, Heated @ 750 °C for 8 hrs.

photographs).

In contrast, sintered calcium sulfate showed considerably less of the effects. Figure 5.3.1.10 illustrates the increased agglomeration tendency of the crystals after heating, even though the effects are not as pronounced as in the magnesium sulfate powder.

Comparison of Figures 5.3.1.5 and 5.3.1.6 to Figure 5.3.1.11 reveals the heating effect on the 1:1 powder ratio. The smaller magnesium sulfate powder particles that had previously adhered to the larger crystalline surface of the calcium sulfate powder now seem to be fused with its surface. Also, the 10 micron scale bars indicate an increase in agglomerated particle size.

This effect is more obvious in Figure 5.3.1.12, in which the smaller magnesium sulfate particles appear to not only be adhering to the surface of, but in some cases surrounding the larger calcium sulfate crystals. The edge of the particle shown in Figure 5.3.1.12 displays

Figure 5.3.1.12: SEM Photo of 1:2 CaSO₄/MgSO₄ Powder Mixture, Heated @ 750 °C for 8 hrs.

curvature characteristic of liquid bridge formation, densified to the point that the porosity is almost nonexistent.

In contrast, Figure 5.3.1.13 shows considerable less. There does seem to be an increased agglomeration effect as a result of the elevated temperature conditions, but the agglomerated structure appears more crystal-like and rigid, as opposed to the almost molten

1:2 powder ratio.

The behavior in these photographs shows that magnesium sulfate powder melts and flows easily in comparison to calcium sulfate, suggesting that it should have a direct effect on the rheological properties (more specifically, the tensile strength) of the powder mixtures. Both the smaller particle size of magnesium sulfate and its liquid-like tendency to adhere to larger surfaces

Figure 5.3.1.13: SEM Photo of 2:1 CaSO₄/MgSO₄ Powder Mixture, Heated @ 750 °C for 8 hrs.

and fill void space suggest that the presence of such in a powder mixture will increase the mechanical strength, particularly at elevated temperatures. However, as shown in Figure 5.3.1.14, data collected by Richman (1997), there is no significant difference in the mechanical strength any of the powder mixtures. This and other rheological

properties will be examined in more detail in the following section.

5.3.2 Rheological Properties

Figure 5.3.2.1 shows the mechanical strength of the pure magnesium sulfate powder. Unlike the previous substances, this one can be characterized by only two of the strength methods at ambient conditions. Tablets (small briquettes) created with any of the sulfate powders, pure or mixture, were frail and readily adsorbed water from its surroundings at ambient temperatures. Extrapolation of data from the packed bed and split cell methods suggests that any tablets formed would be well below the lower limit of the Soiltester compression tester; only after sintering could this method provide an indication of a briquette's mechanical strength (or in this case an apparent increase in such). The data appears to be in good agreement with Richman's, though once again slightly offset to the right. There is a slightly larger scatter in the split cell data than with the IGCC powder, due to the powder's tendency to absorb water, as shown to an even greater degree

Figure 5.3.2.1: Mechanical Strength of Magnesium Sulfate

in future examples.

Figure 5.3.2.2 shows mechanical strength data for calcium sulfate. Also in good agreement with Richman's data, it's strength increases less sharply (with decreasing porosity) than magnesium sulfate. Calcium sulfate can be packed more easily than magnesium sulfate, as indicated by the wider range of porosity, perhaps as a result of it's frail strength (making the particles easy to crush) and wider size distribution.

Figures 5.3.2.3, 5.3.2.4, and 5.3.2.5 shows strength data for each of the mixtures. The 1:1 ratio is in good agreement with Richman's data, though once again shifted slightly off to the right. There is slightly more deviation in the 1:2 ratio, and even more in the 2:1 ratio, not surprising when one considers that these mixtures were created by two different individuals approximately

Figure 5.3.2.2: Mechanical Strength of Calcium Sulfate

one and one half years apart. Regardless, the powder mixtures produce no wildly different data or indicate any disturbing differences, and the discrepancies indicate the amount reproducibility to expect in simulating the PFBC filter cakes. In all three cases the effects of sintering are shown on briquettes that were exposed to elevated temperature conditions, approximately 750 °C for 8 hours (Richman 1997).

Inspection of the deformation coefficient of these powders and their mixtures is not quite as easy. The deformation coefficient is measured as a series of small average Y values, with the measured data approaching the true value as the experimental increment decreases. In other words, the plastic deformation coefficient (Y) can be thought of as an instantaneous value; as the incremental pressure increases are reduced (while measuring the corresponding height changes), the measured value of Y becomes more accurate, similar to how a speedometer measures a car's speed by showing a series of small rapid averages. Because time and powder were limited, and because each experiment in the combined effect apparatus involved the tightening and loosening of sixteen large bolts, limited data for the deformation was taken. However, the 'rough averages' shown in Figures 5.3.2.6 through 5.3.2.8 illustrate somewhat more than a qualitative trend. In all five cases the deformation coefficient rose to infinity more quickly at elevated temperatures than at ambient, resulting from the decreased compressibility of the powder compact. The inlet pressure itself once again seemed to have no bearing on powder behavior, as shown by the wide and random scatter of inlet pressures designated on the plots.

1:1 CaSO₄/MgSO₄ Ratio

Figure 5.3.2.3: Mechanical Strength of the 1:1 Ratio Mixture

1:2 CaSO₄/MgSO₄ Ratio

Figure 5.3.2.4: Mechanical Strength of the 1:2 Ratio Mixture

Figure 5.3.2.5: Mechanical Strength of the 2:1 Ratio Mixture

Figure 5.3.2.6: Plastic Deformation Coefficient for CaSO₄ Powder

Figure 5.3.2.7: Plastic Deformation Coefficient for 1:1 Ratio CaSO₄/MgSO₄ Mixture

Figure 5.3.2.8: Plastic Deformation Coefficient for 2:1 Ratio CaSO₄/MgSO₄ Mixture

The only plot in which this was not obvious was that of the 1:2 CaSO₄/MgSO₄ powder ratio. While the pressure independence was demonstrated by this plot, data was inadvertently taken in such a way that the temperature effect was not obvious. The scatter of the ambient temperature data and the elevated temperature data seem to overlap, making it difficult to resolve the distinct curves of the previous figures. As a result of this plot's uselessness (due to poor choice of data points on my part), it has been omitted from this work. To more accurately distinguish the temperature effect on the powder, the compressive pressure per unit layer vs porosity plot must be examined.

The compressive pressure per unit layer vs porosity charts are less corrupted than the deformation coefficient plot. Regardless of the interval between data measurements the curve retains its characteristic shape, and there is an advantage in formulating this chart from more than one experiment: an idea of the data scatter from several experiments and/or columns can be gained. Figures 5.3.2.9 through 5.3.2.13 illustrate not only the effect on porosity of increasing the applied pressure, but temperature and pressure effects as well. Under elevated temperature conditions, the curve can not be approximated with only two points, so a line (not an interpolation) is present for ease of visual comparison to ambient conditions. Once again, inlet pressure demonstrated no effect on the rheological properties; inlet pressures for these data points can be found by comparing porosities with the corresponding deformation coefficient graphs. Unlike the previous case, that of the Y charts, the 2:1 ratio shows a pronounced difference in powder behavior due to the temperature effect (Figure 5.3.2.13).

Figure 5.3.2.9: Compressive Pressure Per Unit Layer vs Porosity for MgSO₄

Figure 5.3.2.10: Compressive Pressure Per Unit Layer vs Porosity for CaSO₄

Figure 5.3.2.11: Compressive Pressure Per Unit Layer vs Porosity for 1:1 CaSO₄/MgSO₄ Powder Mixture

Figure 5.3.2.12: Compressive Pressure Per Unit Layer vs Porosity for 1:2 CaSO₄/MgSO₄ Powder Mixture

The stress hysteresis plots, fracture strength per unit layer over compressive pressure per unit layer ratio vs porosity, are shown in Figures 5.3.2.14 through 5.3.2.18. Examination of the five plots shows that elevated temperatures seem to produce what seems to be a roughly parallel line, shifted slightly down and to the right. Once again, inlet pressure showed no effect, and inlet pressures corresponding to represented data points can be found by comparison of data at a given porosity to the deformation coefficient charts.

Figure 5.3.2.13: Compressive Pressure Per Unit Layer vs Porosity for 2:1 CaSO4/MgSO4 Powder Mixture

Figure 5.3.2.14: Recovered Over Applied Stress Ratio vs Porosity for MgSO₄ Powder

Figure 5.3.2.15: Recovered Over Applied Stress Ratio vs Porosity for CaSO₄ Powder

Figure 5.3.2.16: Recovered Over Applied Stress Ratio vs Porosity for 1:1 Ratio CaSO₄/MgSO₄ Powder Mixture

Figure 5.3.2.17: Recovered Over Applied Stress Ratio vs Porosity for 1:2 Ratio CaSO₄/MgSO₄ Powder Mixture

Figure 5.3.2.18: Recovered Over Applied Stress Ratio vs Porosity for 2:1 Ratio CaSO₄/MgSO₄ Powder Mixture

CHAPTER VI.
CONCLUSIONS & RECOMMENDATIONS

With the new combined effect apparatus (steel column), operating conditions of candle filters can be modeled more accurately. The results produced with this apparatus are consistent with previous methods and the data produced by more accurate modeling instills confidence in our sponsors, keeping the cash stream flowing. There are only two drawbacks to this apparatus: without many experiments it becomes difficult to judge the plastic deformation coefficient of the powder body, and the apparatus itself is subject to physical limitations. Above 300 °C (273 °C in direct contact) the gasket material that seals the apparatus melts, and above pressures of 3 atm (input), gas flow becomes ridiculously difficult to control. These matters might be resolved with the design and construction of a newer apparatus, but at even more elevated temperatures graphite gasketing would need to be used to seal the pressurized casing (and replaced for each experiment), resulting in expensive work.

6.1 Calcium Carbonate

The calcium carbonate utilized in this work exhibited mechanical properties extremely close to those measured by Richman (1997). This is not surprising when it is considered that although they were two different lots, they originated at the same company and were probably subjected to the same standard processing. Tested in its pure form, there was no room for deviation that might result from different mixing times and/or apparatuses.

Although this powder was used as the ‘ideal’ system, this was not completely the case. There was a noticeable fracture strength increase at elevated temperatures, but as that this powder

decomposes well below the elevated temperature conditions of interest in studying candle filter deposition, it was of no concern. This increase in tensile strength at elevated temperatures was also accompanied by a stiffer deformation coefficient (under compression), suggesting that the elevated temperature conditions could have caused the formation of liquid bridges that would cause particles to adhere more under tension and retard packing rearrangement under compression. Alternatively, this elevated temperature effect might have been brought upon simply by evaporation of water adsorbed onto the surface of the particle, decreasing or eliminating lubrication for particles being pushed into rearrangement and causing solid bridges that link the particles together. Regardless of the mechanism, there is an apparent increase in interparticle force with this powder; it occurs rapidly in comparison with the PFBC powders (3 hour temperature equilibration time, as opposed to Richman's 8 hour sintering time) and this phenomenon might be studied further.

This powder was used to demonstrate the consistency of the newer steel column with the previously established quartz and acrylic columns. It was also used to show that there was no difference (for this non-oxidizing powder) in the rheological properties whether the compressing gas was nitrogen or air, at ambient or elevated pressure input.

6.2 IGCC Powder

The IGCC powder demonstrates no pressure or temperature dependence, suggesting that its behavior in adhering to a candle filter is controlled by its rheological properties. Slightly off from our ideal system, this powder oxidized under elevated temperature conditions, necessitating that some experiments be carried out under a blanket of nitrogen. Regardless of this fact, the

IGCC powder's rheological properties were well behaved, all forming nice straight lines on semi-log plots. While our samples seemed to contain more volatile matter than those analyzed in literature (Praxis Engineering 1996), they (three different samples taken over a year apart) behaved consistently.

This system was also well described by Darcy's law. Using a data set of gas velocity, resulting pressure drop per unit length and corresponding porosity, a specific permeability plot was formed as a function of porosity. This parameter is particularly useful in that it describes the filter cake system, independent of the compressive gas's properties, allowing it to maintain significance in different situations, barring any drastic process alterations. Utilizing a semi-log relation found between two of the rheological properties (compressive pressure per unit layer and resulting fracture strength), this parameter was used to back-calculate the hypothetical gas velocity required to remove a candle filter deposit. This portion of the work is fairly recent and ongoing with our DOE agent and as nice as it would be to test the accuracy of our model, it may be a very long time before any form of closure is seen.

If this powder is one of the 'trouble-maker' deposits causing the destruction of candle filters, further research might be conducted into lubricating additives (if such things exist for filter deposits) or perhaps a mechanical approach, such as increasing the gas stream particle size (i.e. - by addition of a cyclone) to decrease the deposit fracture strength. Gauging by the process brochure (U.S. DOE 1997) more success is presently being shown by other particle separation methods and the candle filter option may be abandoned as soon as economically feasible to make a retrofit.

6.3 PFBC Simulated Powder (magnesium/calcium sulfate systems)

Following the beaten path, some of Richman's (1997) work was repeated and then extended, this time under experimental conditions that more accurately those of the process. Richman had already demonstrated the sintering effect at elevated temperature conditions, and in this work several of his experiments were repeated on powders of slightly different origin. With only a few minor exceptions, both sets of experimental results were the same, somewhat surprising since they were measured by two different individuals, approximately one and a half years apart, from two different chemical orders, and there was no way of knowing whether or not the mixing procedures were consistent.

Per DOE request, an apparatus was built to more accurately model the candle filter process, this time combining the effects of both elevated temperature and pressure conditions. As in the cases of the previous two powder systems, an increase in inlet pressure exhibited no effect on the experimental results. Similarly, the use of nitrogen gas to compress the filter cake, rather than air, had no bearing.

Also expanding on the work of Richman, structures of the sintering powder system were examined, this time with the use of SEM microscopy. This technique revealed a system in which the smaller magnesium sulfate particles adhered to and surrounded the larger calcium sulfate crystals. Upon heating the mixtures with more magnesium sulfate appeared to densify more, but surprisingly, there was little to no effect on the rheological properties when molar compositions of the two powders were varied. Intuitively, one would expect an increase in mechanical strength and a decrease in compressibility as more and more smaller magnesium sulfate particles filled empty space between larger calcium sulfate crystals, but this does not seem to be the case. This

might be attributed to the fact that the behaviors of the two powders are reasonably close in their pure form (in comparison to calcium carbonate or IGCC powder), but as of yet the answer is still unclear.

At this point it might be prudent to consider repeating some of this work on the actual deposit (as was done with the IGCC powder). The simulated system has allowed us to reasonably assess the temperature and examine the pressure effects (or lack of in this case), but the origin and preparation of a powder can wildly affect its properties, suggesting that more productive research might be performed on “the real McCoy.” SEM microscopy of an actual sample might alone show the presence of sintering, and here too additives and/or additional processing might reduce wear on the filter.

6.4 Rheological Indexes (Suggested for Further Study)

Following Rietema’s lead in using particle interactions to classify powder, Kono’s ‘quasi-solid’ deformation coefficient can be used to quantify the effect of temperature sensitivity and this is suggested for further research. An Arrhenius plot of some temperature sensitive characteristic, in this case Kono’s deformation coefficient, can be used to calculate an activation energy for that parameter. The ratio of the intercept of this plot to the activation energy provides a measure of the stickiness (or stiffness, or cohesiveness, merely an indication of an increase in interparticle force), varying from zero (no slope, no temperature change in the powder’s cohesion) to some large value where the mass no longer behaves as a collection of individual particles.

The ratio of the activation energy to the measured particle size provides an index of how easily the filter deposit will be removed. The particle radius (in the denominator) accounts for the

drag force exerted on the individual particles as the surface is being cleaned. For temperature sensitive powders (those with large slopes, and therefore large activation energies) this ratio will vary considerably more than nonsensitive or slightly temperature sensitive powders. Large values of this ratio are indicative of small cohesive particles that increase in mechanical strength at elevated temperatures, behavior demonstrated in the PFBC process. At some point this ratio will reach a value, the particular value will be characteristic of the material, that indicates caking has begun and that the powder mass is no longer behaving as a collection of individual particles.

CHAPTER VII.

NOTATION

Symbols

d^*	effective particle diameter
H	height of powder packed bed
N	number of unit layers
U	gas velocity
ΔP^*	pressure drop over bed
ΔP^{**}	pressure drop per unit layer
Y	plastic deformation coefficient
l	length of packed bed
K	Darcy's Law Proportionality
B	Darcy's Law Proportionality (independent of fluid properties)

Subscripts

c	compacted condition
f	fracture condition
lp	loosely packed condition

Greek Letters

ϵ	porosity, voidage, void fraction
ρ	density
σ_f	fracture strength
μ	fluid viscosity
ϕ	particle shape factor

CHAPTER VIII.
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