SINTERING CHARACTERISTICS OF LOW-RANK COAL ASHES

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Abstract – The electrical resistance and compressive strength were measured to gain a better understanding of the sintering characteristics of low-rank coal ashes involved in deposit formation in combustion systems. Low-rank coal ashes were prepared by the standard ASTM ashing procedures at 750°C and then separated into three different particle size fractions. The sinter point determined by the electrical resistance method decreased with decreasing particle size at three different particle size fractions of each coal ash. The compressive strength test was made as a function of temperature in the range 750-950°C. At a given sintering temperature, strength of the sintered ash was inversely proportional to particle size. For any given particle size of each coal ash, the strength increased with increasing sintering temperature. X-ray diffraction of the sintered coal ashes showed that, as sintering temperature increased, there was an inverse relationship between sinter strength and the amount of anhydrite in the sintered ash, and a direct relationship between strength and the amount of hauyne.

Key words: Sintering of Coal Ash, Sinter Point, Compressive Strength, Ash Deposition

INTRODUCTION

Ash deposition in coal combustion systems is important since the deposits act initially as insulation material to reduce heat transfer in the radiant zone of the boiler and thereby upset the overall thermal balance within the boiler. Furthermore, large deposits in the upper furnace may fall and damage tubes in the bottom of the furnace [Raask, 1985; Borio et al., 1986; Singer, 1981]. Ash deposits accumulated on convection tubes can reduce the cross sectional gas flow area, increasing fan power requirements and also creating higher local gas velocities which accelerate fly ash erosion. Problems of this fouling are most likely to occur with coals having high sodium contents, usually found in low-rank coal deposits. These problems can result in reduced steam generating capacity, unscheduled outages, reduced availability, and costly modifications [Borio et al., 1986].

Manifestations of ash behavior include clinkering on stoker grates, agglomeration of ash in fluidized beds, slagging and fouling in pulverized coal combustors, and slag flow in cyclone units [Reid, 1981]. The key feature linking these situations is the ability of the liquid phase to act as a glue to bond together particles which are still solid. With rising temperatures in the combustor, the solid particles would be those ash components which have not yet melted; for systems which are cooling, the solids may be crystallized from a melt. In either case, the bonding of particles is governed by the sintering equation developed by Frenkel [1945].

Frenkel's equation describes the rate of coalescence of particles in terms of measurable parameters: viscosity, surface tension, and particle size.

$$(\mathbf{x/r})^2 = 3 \,\gamma t/2\eta \mathbf{r} \tag{1}$$

where r is the radius of the particles, assumed to be spheres; x is the radius of the interface or "neck" between particles, assumed to be circular; γ and η are the surface tension and vis-

cosity of the liquid, respectively; and t is the sintering time. It is clear from the above equation that square of the radius of the contact area is proportional to surface tension and sintering time and inversely proportional to viscosity of the liquid phase at a given size of particle.

An evaluation of ash sintering characteristics is useful to predict whether a given coal ash will result in strongly bonded ash deposits during combustion. The several major parameters such as ash composition, ash particle size distribution, sintering temperature and time, and reducing or oxidizing nature of the atmosphere will influence sintering behavior.

Several laboratory methods have been developed to evaluate specifically the sintering characteristics of coal ashes. Barnhart and Williams [1956] developed a laboratory test for determining the tendency of an ash to form hard, bonded deposits in the convective tubes of coal fired boilers by measuring the compressive strength of sintered ash pellets. Fouling coals with high sodium content were found to have lower sinter points and higher sintered strengths than non-fouling coals with low sodium content. Dering et al. [1972] examined the sintering behavior of different size fractions of fly ash from brown coal. They reported that the finest fraction (0-5 µm) of fly ash sintered more strongly and sintering began at a lower temperature than for the coarse fractions. This was explained in terms of the increased activity of the sintering surfaces between the particles and the higher concentration of water soluble alkalies in the finest size fraction of fly ash. Smith [1956] used a dilatometric shrinkage technique to determine the sinter point of coal ash. This measurement gives useful information, and the results can be related to the different degrees of sintering as in the neck growth measuring technique.

Raask [1979] developed a laboratory method for simultaneously measuring the electrical conductance and the dilatometric shrinkage for soda glass of known viscosity and temperature characteristics. According to his results, seda glass with a viscosity of 10^{10} Nsm⁻² showed the beginning of shrinkage and the increase of electrical conductance at 600°C. He was also found that the viscosity range of 10^{6} to 10^{10} Nsm⁻² was a relevant viscosity range for the formation of sintered deposits in coal fired boilers.

Electrical resistance measurements have been made by Cumming [1980] on coal ash specimens for studying the onset of melting. He considered an electrical resistance measurement as a possible candidate for measuring onset of melting because of the uncertainty of initial deformation temperature as determined by the ash fusibility test. The reasoning was that while the ash was completely in the solid state, its resistance would be very high, but with increasing temperature caused the first appearance of ash in a liquid phase, ionic conduction as in an electrolyte could occur, with a sudden fall in resistance.

Conn [1984] reported the sintering behaviors of coal ashes prepared by ASTM procedures, fly ashes, and model materials (soda-lime-silica glass and synthetic mineral mixture) based on the measurement of the ash shrinkage and electrical resistance simultaneously. Most coal ashes showed both simultaneous shrinkage and a slope change at the sinter point in the relationship between electrical resistance and inverse absolute temperature. Benson [1986] measured the strength of deposits produced in the vertical tube furnace as a function of height (distance from the base of the deposit). The growth of strength in the deposit related to the amount of liquid available for sintering and to the liquid viscosity.

The approach in this study was to sinter two low-rank coal ashes as a function of temperature under an oxidizing (air) atmosphere to gain a better understanding of the sintering behavior involved in ash deposit formation in coal combustion systems. The sinter point of coal ashes was determined by the electrical resistance method. Different particle size fractions of each coal ash were used to determine the effect of particle size on the sinter point. The compressive sintered strengths of each coal ash were measured as a function of temperature to test the qualitative aspects of the Frenkel [1945] equation and to interpret and evaluate the physical and chemical changes of the sintered ash particles as related to deposit formation process. Different particle size fractions of each coal ash were used to determine the effect of particle size on the compressive sintered strength. The chemical changes of the sintered ashes were ascertained from X-ray diffraction analysis after measuring the compressive sintered strength.

EXPERIMENTAL

1. Sintering Tests

The furnace used for the sintering studies was an ATS (Applied Test Systems Co.) electrically heated tube furnace, rated for use to 1600°C, as shown in Fig. 1. The furnace interior was protected by a mullite tube. The furnace temperature was monitored by a B-type thermocouple attached to the inside of the furnace and was controlled by a microprocessor control system.

Details on the electrical resistance method to determine the sinter point have been described and discussed by several workers [Raask, 1979; Cumming, 1980; Conn, 1984]. Briefly, an alumina crucible with a flat base, 10 mm internal diameter and 25



Fig. 1. Experimental apparatus for coal ash sintering.

mm high was used to contain the ash sample to determine the ash sinter point by the electrical resistance method. Disks of platinum foil 10 mm in diameter and 0.5 mm thick were used as the electrodes by placing it both in the bottom and on the top of the ash sample, as part of an electrical circuit to measure the ash resistance.

Platinum wire leads 0.2 mm in diameter were spot welded to the platinum foil electrodes. The ash pellet sample was placed between two platinum electrodes inside the furnace to determine the sinter point. These electrodes were connected to a 15 volt stabilized d.c. source by way of a 2000 ohm resistor in one of the leads, as shown in Fig. 2. and a potentiometer was connected across the resistor. The ash resistance is related to the voltage drop across the fixed resistor and the temperature from a R-type thermocouple situated adjacent to the sample pellet.

As the sample was heated, its electrical resistance decreased and an increased current flowed in the electrical circuit. Since most of voltage drop in the circuit was across the sample, the resistance of the sample could be related to voltage drop read on the potentiometer as follows: $E=I(R_f+R_{ast})$, $I=V/R_f$, and $R_{ast}=$ E/I since $R_{ast} \gg R_f$. E is the voltage of the d.c. power supply, I is the calculated current flow in the circuit, V is the voltage drop from the potentiometer, R_f is the fixed resistance, and R_{ast} is the calculated resistance of the sample. A plot of log (resistance) versus inverse absolute temperature normally gives a straight line, and the slope of this line changes as sintering commences; the temperature at the point where the slope changes is called the sinter point. At the sinter point, the formation of a liquid phase or increased particle contact resulted in a change in the slope of the line relating resistance to inverse absolute tem-



Fig. 2. Electrical circuit for determining coal ash resistance.

perature.

2. Compressive Strength Measurements

The compressive strength test of Barnhart and Williams [1956] was modified in the present study by preparing a small ash pellet having a bulk density comparable to that of the deposit in a boiler. To determine the compressive strength, the ash sample was compressed in a die where enough pressure was applied to give a cylinder of 8 mm diameter by 8 mm height having a bulk density of 1.2 g/cm³. The pellet was put in an alumina crucible of 25 mm diameter and 50 mm height which was subsequently heated in the sintering furnace to the desired temperature at 6-8°C/min in air, and held at that temperature for an one hour. The pellet was left inside the furnace to cool to room temperature within about 2 hours. The compressive strength of the sintered ash pellet was measured at room temperature after removal of the cold sintered ash pellet from the furnace. To determine the compressive strength at room temperature, the sintered ash pellet was placed on a flat stainless steel plate in the Instron apparatus and crushed by rasing it vertically against the load cell attachment. The load cell can supply loads in the range of 20 to 1,000 pounds and the output (the force required to crush the sample) was recorded in a potentiometric chart recorder. An X-ray diffraction analyzer (Rigaku ME200DX) was used to identify the chemical changes of the sintered ash pellets.

3. Sample Preparation

The coal ashes studied were produced from Eagle Butte (Wyoming) subbituminous coal and Beulah (North Dakota) lignite. The proximate and ultimate analyses of these two coal samples are shown in Table 1. The ashes were prepared using the standard ASTM ashing procedure (D2795-86) [1990] at 750°C. The compositions of the two ashes are shown in Table 2. Three different particle size fractions of each coal ash were used: $53 < D_p$ (ash particle size)<75 µm, $38 < D_p < 53$ µm, and $D_p < 38$ µm.

RESULTS AND DISCUSSION

1. Determination of Sinter Point by Electrical Resistance Method

Several methods of determining the onset of coal ash sintering by electrical conductance [Raask, 1979], electrical resistance [Cumming, 1980; Conn, 1984], and shrinkage measurements [Smith, 1956; Conn, 1984] have been utilized. These measurements are based on the sintering model developed by Frenkel [1945] which stipulates that the formation of particle-toparticle bonding leads to enhanced conductance, or decreased

Table 1. Proximate and ultimate analyses of coal samples

	Eagle Butte subbituminous	Beulah lignite
Proximate(wt%)		
Moisture	25.9	30.0
Volatile matter	32.9	31.0
Fixed carbon	35.8	33.0
Ash	5.4	6.0
Ultimate(dry, ash-free, wt%)		
Carbon	54.0	48.6
Hydrogen	6.3	3.2
Nitrogen	0.6	0.7
Sulfur	0.4	0.7
Oxygen(by diff.)	38.7	46.8

Га	ble	2.	Ast	i com	positions	of	coal	sampl	es
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	Eagle Butte subbituminous	Beulah lignite		
Component	Ash	Ash		
SiO ₂	28.5	27.6		
Al_2O_3	15.5	15.8		
TiO_2	0.8	0.7		
Fe_2O_3	10.2	10.8		
CaO	22.7	19.8		
MgO	5.6	5.4		
Na ₂ O	3.9	5.5		
K ₂ O	0.1	0.1		
SO ₃	12.7	14.3		

resistance, and increased density of the sintered ash samples. In this study, a simplified method for determining the sinter point by electrical resistance measurement was used to evaluate the sintering tendencies of low-rank coal ashes. The electrical resistance versus inverse absolute temperature curves at three different particle size fractions of Eagle Butte subbituminous coal ash are shown in Fig. 3. As shown in Fig. 3, the sinter point increases with increasing particle size, from 480°C for the $D_p < 38$ µm ash and 510°C for the $38 < D_p < 53$ µm to 565°C for $53 < D_p$ <75 µm ash. The sinter point of the coal ash decreased as a function of particle size since the surface area to volume ratio of the ash particles increases with decreasing particle size. As particle size decreases, an increase in surface energy would occur along with an increased rate of sintering as predicted by the Frenkel equation.

The electrical resistance versus inverse absolute temperature curves showed a nonlinear relationship. An ash compact before sintering would have a high resistance because of lack of particle-to-particle contacts. As the cross sectional area of sinter bonds between particles grows, the resistance of ash particles exponentially changes with increasing temperature. This nonlinear relationship between plot of log resistance and inverse absolute temperature is in accord with Raask's results [Raask, 1979]. Conn [1984] reported a laboratory method for simultaneously measuring shrinkage and electrical resistance to determine the sinter point of coal ashes prepared by ASTM procedures. He found that sinter points of several coal ashes determined by shrinkage measurements were a little higher than those from



Fig. 3. Effect of particle size on the sinter point determination of Eagle Butte ash by electrical resistance measurement.

electrical resistance measurements. For such materials of a wide particle size range, the electrical resistance technique appeared to be more sensitive to the onset of sintering of very small particles.

In the case of the Beulah lignite ash, as shown in Fig. 4, the sinter point curves of three different particle size fractions of ash determined by the electrical resistance method showed that the sinter point also increased with increasing particle size, from 448°C for the D_p <38 µm ash and 500°C for the 38<D_p<53 µm to 550°C for 53<D_p<75 µm ash. The rate of sintering also increased as the particle size fractions of the each coal ash were decreased from 53<D_p<75 µm to less than 38 µm.

Several sinter points (for instance, 480°C, 550°C and 625°C in the case of Eagle Butte ash having particle size of $D_p < 38$ μ m) have been found at each particle size fraction of the each coal ash, as shown in Figs. 3 and 4. In the electrical resistance versus inverse absolute temperature curves, the initial sinter point was defined as the point at which an initial slope increase in sudden slope change was observed due to the onset of sintering of very small particles having a relatively low melting point. The observations of several sinter points might be possibly due to another phase transformation or development of extensive sintering of very small particles which had already been sintered at the initial sinter point. The deposit of this degree of sintering which was formed at the initial sinter point temperature on boiler tubes might have no significant cohesive strength and would probably fall off under the action of gravity and boiler vibration.

These lower electrical resistances and initial sinter points compared with those of bituminous coal ashes [Conn, 1984] were probably due to the higher alkali metal oxide content of the low-rank coal ashes, which gave greater electrical conductance than the clays and other components present in bituminous coal ash. The Beulah lignite ash, containing relatively high amounts of sodium oxide (5.5 wt% of ASTM ash), showed the lower electrical resistance and sinter point in comparison with the Eagle Butte coal ash. There may be a high degree of the internal



Fig. 4. Effect of particle size on the sinter point determination of Beulah ash by electrical resistance measurement.

particle-to-particle adhesion in some coal ashes rich in sodium. With these ashes the electrical resistance measurements give more meaningful results in early stages of sintering than those of any other technique.

In the electrical resistance method, the tendency to exhibit a gap between sinter point temperature and initial deformation temperature could be due to the formation of a small proportion of liquid phase well below this overall softening temperature, sufficient to cause tackyness, but not to cause deformation of the test pyramid. Conn [1984] found that the variation of electrical resistance with temperature above the sinter point was not due to changes in growth of sinter necks, but due to the normal decrease of liquid or solid-state resistance as temperature was increased.

2. Measurement of Sintered Strength of Coal Ashes

Barnhart and Williams [1956] and Gibb [1981] have used crushing strength measurements of laboratory-prepared specimens to access the sintering characteristics of coal ashes. The compressive sintered strength determination also appeared to rate the fouling tendencies of coal ashes. Barnhart and Williams [1956] found that the compressive sintered strength having about 10,000 psi below 1000°C did not make a severe fouling in a pulverized coal fired boiler. The compressive sintered strength of coal ashes is usually a function of heat treatment temperature and time.

Tangsathitkulchai [1986] showed that appreciable compressive strength was achieved using one hour heat treatment time in most tests. In this study, the one hour heat treatment time was chosen as a sintering time in the following sintering tests as a function of temperature. The sintering behavior of ashes produced from two low-rank coals was studied as a function of temperature in the range of 750-950°C. This temperature ranges relate to the formation of ash deposit layer on the boiler tube surface because there is a temperature difference between flue gas (-1000°C) and steam tube (-500°C) in an actual boiler.

The results of compressive strength of the sintered Eagle Butte and Beulah low-rank coal ashes having three different particle size fractions are given in Figs. 5 and 6, respectively. For any given particle size (i.e., before sintering) of each coal ash, the compressive strength of the sintered ash increases with increasing sintering temperature. At a given sintering temperature, the compressive strength of the sintered ash increases with decreasing particle size. Dering et al. [1972] have found the inverse relationship between the compressive sintered strength and the particle size by the results of compressive strength measurements with size-separated fly ash fractions. The effect of an increase in compressive sintered strength with decrease in particle size can be explained both by the increase in the total surface of contact between particles and by the difference in chemical composition of the different fractions of ash from the same coal. According to the test results of the compressive sintered strength, these two low-rank coal ashes may not be expected to make severe ash deposits in convective sections of a pulverized coal fired boiler, but a periodic cleaning by sootblower is needed to prevent strong deposits.

After determining the compressive sintered strength, samples of the crushed ash pellets were taken for analysis by X-ray diffraction to identify and to try to quantify the minerals present. Approximate quantitative analyses of minerals present in the three different particle size fractions of Eagle Butte and Beulah coal sintered ash $(53 < D_p < 75 \ \mu m, 38 < D_p < 53 \ \mu m, and D_p < 38$ um) are shown in Tables 3 and 4, respectively. Analyses of the phases present in the three different particle size fractions of sintered Eagle Butte and Beulah ashes show the same trends. As shown in Tables 3 and 4, respectively, results of the X-ray diffraction of each sintered ash at each particle size showed that, as sintering temperature increased, there was a depletion of anhydrite (CaSO₄) and increase in the amount of calciumcontaining aluminosilicate phases. The melilite series of minerals, (Na,Ca)₂(Mg,Fe,Al)(Si,Al)₂O₂, contain gehlenite (Ca₂Al₂- SiO_{2} , sodium melilite (NaCaAlSi₂O₂), and nepheline (NaAlSiO₄). These minerals contain calcium and sodium cations within a silica-deficient aluminosilicate structure. Nepheline is an anhydrous framework silicate which forms where there is an abundance of alkalies (Na and K) and a lack of SiO₂. In the case of the particle size less than 38 µm, the quantity of plagioclase also sharply increased at 950°C. Plagioclase, (Ca,Na)(Al,Si)Al-Si₂O₈, is the group name for the solid solution series between anorthite (CaAl₂Si₂O₈) and albite (NaAlSi₃O₈). In the Eagle Butte and Beulah sintered ash, the presences of pyroxene, (Ca,Na) (Mg,Fe,Al) (Si,Al)₂O₆, and melilite were identified in addition to the nepheline and plagioclase. In the case of the particle size less than 38 µm, the pyroxene in both cases increased in quantity at 950°C.

There was an inverse relationship between the compressive sintered strength and the amount of anhydrite in the sintered ash, and a direct relationship between the compressive sintered



Fig. 5. Effect of particle size and temperature on compressive strength of Eagle Butte sintered ash.

Table 3. X-ray di	iffraction anal	vses results ((area%) of	Eagle Butt	e sintered as
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Temperature.ºC

Fig. 6. Effect of particle size and temperature on compressive

strength of Beulah sintered ash.

< 0.1

61.8

ble 4. X-ray diffraction analyses results (area%) of Beulah sintered ash								
emperature	Particle size	Anhydrite	Quartz	Hematite	Nepheline	Hauyne	Pyroxene	Plagioclase
750°C	53 <d<sub>ρ<75 μm</d<sub>	62.6	20.0	10.3	6.6	0.5	<0.1	<0.1
	38 <d<sub>ρ<53 μm</d<sub>	73.8	15.2	4.2	5.8	1.0	< 0.1	<0.1
	D _p <53 μm	61.3	17.5	9.3	10.4	1.5	<0.1	<0.1
850°C	53 <d<sub>p<75 μm</d<sub>	46.9	10.6	7.4	0.2	34.9	<0.1	< 0.1
	38 <d<sub>p<53 μm</d<sub>	41.4	15.9	3.0	1.2	37.0	1.5	< 0.1
	$D_p < 53 \mu m$	45.9	14.3	2.6	1.9	32.5	2.8	<0.1
950°C	53 <d<sub>p<75 μm</d<sub>	34.6	5.3	1.9	<0.1	48.7	6.3	0.9
	38 <d<sub>p<53 μm</d<sub>	33.7	3.2	<0.1	< 0.1	51.0	4.0	1.2

0.4

3.2

Ta T

20.8

strength and the amount of hauvne, (Na,Ca)₈(Si,Al)₁₂ O₂₄(SO₄)₂, in the range of sintering temperature (750-950°C). The sodalite type minerals contained hauvne and nosean (Na₈Al₆Si₆O₂₄SO₄). These phases are similar to the melilites, but contain minor sulfate concentrations and have lower melting points. The presence of hauyne having a relatively low melting point (700-800°C) aluminosilicate may have played a role in the sintering of these ashes. This suggests that the increase of compressive sintered strength may be due in part to a reaction of anhydrite with quartz or clays to form calcium aluminosilicate "glue".

 $D_a < 53 \mu m$

Deposit strength was found to be a function of sodium melilite (sodium aluminosilicate) concentration [Honea, 1983]. In two separate investigations [Honea, 1983; Hein, 1983] it was shown that deposit strength increased with both decreasing calcium concentration and increasing sodium content. The sodiumcontaining phase was once again found to be an aluminosilicate. This increased particle-to-particle bonding would lead to much higher sinter strengths than just the freezing of liquid phases on the surface of ash particles. However, it may be possible that some of the liquid phase formed at the heat treatment temperature was quickly removed by interaction of the abundant alkalies, alkaline earth sulfates, and aluminosilicate glass to form crystalline phases. It appeared, therefore, that the strength of these pellets resulted mainly from the properties of the newly formed crystalline phases, that is, it was due to the formation of crystalline bridges between particles.

Previous work on ash fouling has indicated that the formation of melilites and sulfates contributed to severe fouling [Rindt et al., 1981]. Plagioclase that was observed in the sintered ash at 950°C is a normal constituent of superheater deposits [Nicholls, 1932]. Formation of the melilites and the sodalites is thought to be the result of complex reactions between alkalies, alkaline earth sulfates, and aluminosilicates [Rindt et al., 1981]. Sintering ashes in the laboratory possibly give a more realistic representation of the minerals that are formed on the tubes due to greater particle-to-particle contact. Thus, the build-up of boiler deposit is closely related to the changes in the nature of mineral matter taking place in the flame. However, many of these various low melting aluminosilicate compounds may not be present during the initial stages of sintering and maybe formed after the deposit has remained in the furnace for a period of time.

CONCLUSIONS

were used to evaluate the sintering characteristics of low-rank coal ashes having three different particle size fractions in a sintering furnace. The sinter point decreased with decreasing particle size due to the increased surface area to volume ratio of the ash particles. The results of sinter point determination using the electrical resistance method indicated that this method demonstrated the onset of sintering due to the presence of a liquid phase from the lowest melting point component in coal ash. In the compressive strength test of low-rank coal ashes in the range of 750-950°C, there was an inverse relationship between sinter strength and the amount of anhydrite in the sintered ash, and a direct relationship between strength and the amount of hauyne. The compressive strength test results of coal ashes also showed the qualitative aspects of the Frenkel equation.

10.7

Melilite < 0.1 <0.1 <0.1 < 0.1 < 0.1<0.1 2.3 2.9

1.5

1.6

NOMENCLATURE

- D, : ash particle size
- Е : voltage of d.c. power supply
- I : calculated current flow in the circuit
- r : radius of particle
- : calculated resistance of sample R_{ash}
- \mathbf{R}_{ℓ} : fixed resistance
- : sintering time t
- : voltage drop read from potentiometer v
- : radius of interface or "neck" between particles x

Greek Letters

- : surface tension of liquid γ
- : viscosity of liquid n

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