Coal Ash Sintering Model and the Rate Measurements

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INTRODUCTION

In spite of innumerable laboratory investigations and the wealth of practical experience, there remains some enigmatic facets in the formation of sintered ash and slag deposit on heat exchange surfaces in coal-fired boilers. That is, occurrences of massive build-up of ash deposits can take boiler design and operation engineers by surprise. This would suggest that the engineers are receiving incomplete or incorrect information on the deposit-forming propensity of ash in different coals.

Traditional methods of assessing the behavior of deposit-forming impurities in high temperature boiler plants are based on ash fusion tests as described in different national standards for coal analysis and testing, e.g., ASTM (1968), British Standard (1970), DIN (1976) and Norme Francaise (1945). These ash fusion tests have been developed from refractory material technology, and are based on observations of the change in shape and size of an ash sample on heating. It has been realized that the results of ash fusion tests are frequently imprecise, and can lead to a mistaken assessment of the likely severity of boiler fouling and slagging.

There has been a number of suggestions made for ash fouling and slagging indices to predict more accurately the rate of deposit build-up with different coals as reviewed by Winegartner (1974). The empirical formulae, e.g., silica ratio, basic to acidic oxide ratio, and sodium and sulphur contents of coal, are based on the chemical analysis and have a limitation that they apply to particular coals. That is, there is no universal formula of predicting the severity of boiler fouling based on ash fusion tests and chemical analysis with all types of coal.

It is therefore evident that further research is necessary on the mechanism of ash particle-to-particle bonding at high temperatures. This work sets out to redefine an ash sintering model in terms of measurable parameters, surface tension, viscosity, electrical conductance, temperature, particle size and time. In the experimental work, novel methods of measuring the rates of ash sintering were applied, in order to test the validity of the sintering model.

SINTERING MODEL

Frenkel (1945) has derived an equation relating the growth of the interface between two spherical particles:

$$x^2 = \frac{3r\gamma t}{2\eta} \qquad (1)$$

where x is the radius of the interface, r is the original radius of the spheres, γ is the surface tension and t is the time. Rearranging the equation in terms of x/r and t, it becomes:

$$\frac{x}{r} = 1.225 \left(\frac{\gamma}{\eta r}\right)^{1/2} t^{1/2} \qquad (2)$$

and it is applicable when $\frac{x}{r} < 0.3$.

Sintering by viscous flow is the principal mechanism for the formation of deposits in coal fired boilers and the rate of sintering of different size particles for a given viscosity (10^8 N s m^{-2}) can be seen in Fig. 1 where the ratio of x/r is plotted against time on a logarithmic scale. The surface tension of fused ash was taken to be 0.32 N m^{-1} as measured previously by Raask (1966). Fig. 2 shows plots where the ratio of x/r represents the degree of sintering of 5 μm radius particles having different viscosities. Table 1 lists four arbitrary stages of sintering of ash deposit on boiler tubes from the initial contact between the ash particles to the formation of fused slag where the shapes of initial constituent particles are no longer distinguishable.

Table 1. Degree of Sintering Based on the Ratio of Neck Bond Radius to Particle Radius ($\frac{x}{r}$)

Ratio of x/r	Degress of Sintering	Comment
0.001	Onset of sintering	Deposit of this degree of sintering on boiler tubes would not have significant cohesive strength and would probably fall off under the action of gravity and boiler vibration.
0.01	Slightly sintered matrix	The deposit on boiler tubes would probably be removed by soot blowing.
0.1	Strongly sintered deposit	The deposit on boiler tubes would be difficult to remove by soot blowing.
>0.3	Slagging	The ash particles lose their original identity and the deposit on boiler tubes cannot be removed by soot blowing.

Rapid formation of sintered boiler deposits and slags is usually explained by the presence of a liquid phase or molten surface layer on ash particles. In high temperature glass and slag technology (blast furnace slag), a liquid phase is considered to have a viscosity value below 10 N s m^{-2}. The plots in Fig. 1 and 2 show that with small particles it is not necessary to evoke the presence of a liquid phase for a rapid sintering. For example, particles 0.1 m in diameter would require about 10 milliseconds to form a substantial sinter bond, when the viscosity has a high value of 10^8 N s m^{-2}. With the same viscosity 10 μm particles would require about 10 seconds to achieve the same degree of bonding.

The two parameters which govern the rate of sintering, namely the surface tension and the viscosity, both decrease with temperature as shown in Fig. 3. The temperature coefficient of surface tension is small (Curve A) and it is approximately proportional to the inverse of square root of temperature as discussed by Boni and Derge (1956) whereas the viscosity changes exponentially with temperature as shown by Curve B. It is therefore evident that the rate of sintering will show an inverse relationship with the viscosity and will increase exponentially with temperature.

PARTICLE-TO-PARTICLE NECK GROWTH MEASUREMENTS

The model for coal ash sintering discussed in the previous section is based on the viscous deformation and flow at the contact points between spherical particles. It would, therefore, be logical to consider determining the rate of sintering by a technique where the measurements are based directly on Frenkel's equation. Kuczynski (1949) has

carried out such sintering measurements by placing spherical particles on the surface of a glass slab of the same composition. Raask (1973) has reported some results of sintering rate measurements with coal ash slag particles based on a similar technique. The method requires spherical particles of ash and these can be prepared by passing ground coal minerals or slag through a vertical furnace as described by Raask (1969). Subsequently the particles were placed in a narrow groove on a platinum foil as shown in Fig. 4a. The particles were then introduced into a preheated furnace and kept at a constant temperature in air, or in a gas mixture for a period of five minutes to several hours. The radius of the sinter neck between the particles (Fig. 4b) was measured microscopically at the ambient temperature.

Fig. 5 shows the rate of neck growth between spherical particles of slag, 60 μm in radius, when heated in air. The spherical particles were prepared from boiler slag of a typical British bituminous coal ash which has caused some boiler fouling. The time for a firm degree of sintering, ($x/r = 0.1$, Table 1) was 135 seconds and 70 seconds at 1375 K and 1425 K, respectively. From these measurements the time required for a given degree of sintering can be calculated for the ash particles of different sizes. For example, the ash deposited on boiler tubes in pulverized coal fired boilers contains a larger number of particles of 0.5 to 1.0 μm in diameter, and these particles require only a few seconds to form a strongly sintered deposit in the same temperature range.

MEASUREMENTS OF ASH SINTERING RATES BY SIMULTANEOUS DILATOMETRIC AND ELECTRICAL CONDUCTANCE TECHNIQUES

Particle-to-particle sinter bonding usually results in a shrinkage of the external dimensions of a powder compact, and the dilatometric shrinkage measurements have been extensively used to determine the rate of sintering of glass and refractory materials. Smith (1956) has used a dilatometric shrinkage technique to study the sintering characteristics of pulverized fuel ash and an intercept of the shrinkage curve on the temperature axis was taken to define the sinter point. The measurements can give useful information and the results can be related to different degrees of sintering as outlined in Table 1. With some coal ashes, however, anomalous results can be obtained where the shrinkage measurements show no change although a significant degree of sintering has taken place. This deviation in the sintering behaviour from the Frenkel model makes it necessary to monitor another parameter which relates to the process of particulate ash coalescence.

Viscosity measurement by the rod penetration method has been applied by Boow (1972), Raask (1973) and Gibb (1981) to assess the sintering characteristics of different coal ashes. However, the rate of initial sintering cannot be measured by this technique, and Raask (1979) therefore considered the use of a method of electrical conductance measurements for monitoring the rate of sintering of coal ashes. Previously Ramanan and Chaklader (1975) had used the same technique to study sintering of glass sphere and nickel powder compacts.

The essential premise of this method is that the particulates are of an electrically conductive material, e.g., nickel, or that the glassy and ceramic materials contain some cations, e.g., alkali-metals which constitute an ionic conductance path when an electrical potential is applied. The method is, therefore, not applicable to measure the rate of sintering of nonconductive powders, e.g., alumina. This is not a limitation with coal ashes as all ashes contain sufficient amounts of cationic species; 0.1 per cent by weight quantity of sodium, potassium or calcium is likely to be adequate for the purpose of providing a conductance path.

A powder compact before sintering has a low conductance because of lack of particle-to-particle contacts. As the cross-sectional area of sinter bonds grows on heating the conductance is increased according to the equation:

$$\Lambda = A \left[\left(\frac{D}{D_o}\right)^{2/3} - 1 \right] \exp\left(\frac{-E}{RT}\right) \qquad (3)$$

where Λ is the conductance, D_o and D are the densities of sample before and after sintering, E is the energy of activation of sintering, R is the thermodynamic (gas) constant, T is the temperature and A is a constant. When the degree of sintering does not change, e.g., on cooling after the process of particle coalescence has reached the stage of density D, the equation (3) reduces to:

$$\Lambda = A_1 \exp\left(\frac{-E}{RT}\right) \qquad (4)$$

Rask (1975) has described a furnace assembly sketched in Fig. 6 for simultaneous measurements of the electrical conductance and the dilatometric shrinkage measurements. Fig. 7a shows the furnace in its down position for exposure of the sample well; sample crucible and three pellets of sintered ash from previous runs are shown at the well. Fig. 7b shows the furnace in the operation position. The heating rate of 0.1 K s^{-1} (6 K min^{-1}) was the same as that used in the ASTM (1968) ash fusion tests and the sinter tests can be carried in air or in simulated flue gas. Care is needed to stop heating when the ash sample has decreased 30 per cent in height to avoid slagging; once slag is formed it is difficult to remove the frozen material from the crucible.

Initial experiments were made with a soda glass, ground below 100 μm in particle size, of known viscosity/temperature characteristics published by Napolitano and Hawkins (1974). This was done to establish the validity of the simultaneous dilatometric and conductance measurements for determining the rate of sintering of powder compacts. The results are shown in Fig. 8 where Line A_1 depicts thermal expansion of the alumina support tubes and the sample, and Curve A shows the linear shrinkage of 10 mm high sample of powdered glass. The intercept of Curve A on the temperature axis, 875 K can be defined as the sinterpoint temperature.

The conductance plot (Curve B gives the same sinterpoint temperature and the results increase exponentially with temperature. On cooling Curve C shows a large hysteresis effect, that is, these are significantly higher than the corresponding results on heating. On reheating, however, the conductance measurements fit closely to Curve C. This behavior is in accord with the sintering model and the measurements on first heating when sinter bonds are formed, fit equation (3). Since on subsequent cooling and reheating, the process of particle coalescence is "frozen," the conductance change is governed by the exponential temperature as defined by equation (4).

Fig. 9 shows that there was in inverse relationship between the viscosity and the electrical conductance with respect to temperature. The conductance is dependent on the mobility, i.e., on the rate of diffusion of sodium and calcium ions in the glass matrix, and thus an inverse relationship between viscosity and self-diffusion is established as stipulated by Frenkel's sintering model. The results of conductance measurements plotted in Fig. 9 cover the viscosity range of 10^6 to 10^{10} N s m^{-2}, and it has been suggested previously by Raask (1973) that this is a relevant viscosity range for the formation of sintered deposits in coal fired boilers. That is, strong sinter bonds can form in this viscosity range within a few minutes or several days depending on the particle size (Figs. 1 and 2).

A number of British and US bituminous coal ashes have also been investigated for their sintering characteristics by the simultaneous shrinkage and conductance measurements. Fig. 10 shows typical shrinkage curve and the conductance plots on heating and on cooling which were obtained with an Illinois coal ash. It was evident that with

this ash and other bituminous coal ashes tested, sintering proceeded according to equation (3). The conductance data can be examined in more detail on the log n against 1/T plot as depicted on Fig. 11. The conductance plot on heating is nonlinear as expected from equation (3), whereas on cooling the plot was linear in accord with equation (4).

The conductance plot on heating can be divided into three sections where the logarithmic conductance values in S (micro-Siemen) are as follows: 1 to 10, 10 to 100 and 100 to 1000 units. Fig. 12 shows schematically the conductance path and shrinkage in three different degrees of sintering. The strength of the sintered pellet to crushing was determined at room temperature at the end of the sinter run. The presentation shows that an increase of 155 K from the initial sinter point temperature of 1125 K to 1180 K resulted in a high degree of sintering where the conductance readings were above 100 μS.

There are some coal ashes which exhibit in their sintering behavior a remarkable degree of divergence from the particle coalescence model as defined by equation (3). Fig. 13 shows that the nonbituminous coal ash, Leigh Creek, Australia, commenced to sinter at 1100 K according to the conductance measurements (Curve B). There was, however, no shrinkage of the ash pellet before temperature reached 1350 K (Curve A). That is, there was a gap of 250 K between the ash sinterpoint temperature indicated by the conductance measurements and that deduced from the shrinkage measurements. The reason for this nonconformist behavior must be that there was a significant degree of particle-to-particle bonding of the infusible material, e.g., quartz by a low viscosity phase at temperatures of 1100 to 1350 K. This may be an explanation for severe boiler fouling with high sodium coal ashes as discussed by Boow (1972) which is inconsistent with the results of conventional ash fusion tests. Leigh Creek ash had another unusual sintering feature; after an initial rise in conductance with temperature there was a decrease with an inflection point at 1325 K (Fig. 12). This was probably because the high amount of sodium in ash, 6.3 per cent of Na_2O by weight, resulted in crystallization of sodium alumino-silicate or aluminate in the temperature range of 1275 and 1325 K thus reducing the concentration of sodium ion in the glassy matrix.

Laboratory prepared ashes can be categorized according to the results of simultaneous measurements of shrinkage and the electrical conductance on sintering of an ash compact. The majority of ash compact coalesce and shrink according to the model as defined by equation (3) and particle-to-particle bonding is accompanied by the external shrinkage and a change in shape of an ash sample in early stages of sintering. With these ashes the conventional ash fusion tests, e.g., ASTM-Method (1968) usually give meaningful results. There are, however, some coal ashes rich in sodium which can give anomalous results when sinter tested as exemplified by the conductance and shrinkage curves in Fig. 12. That is, there can be a high degree of the internal particle-to-particle adhesion resulting in the formation of ash compact or deposit of high strength without the corresponding external shrinkage and these ashes should be tested by the electrical conductance technique to give meaningful results in the early stages of sintering.

NEED FOR AUGMENTING CONVENTIONAL ASH FUSION TESTS BY SINTERING RATE MEASUREMENTS

Ash fusion tests are based on the external change in shape, deformation, shrinkage, and flow of a pyramidic or cylindrical pellet of ash when heated in a laboratory furnace. A pyramidic shape is often used because it is easier to observe rounding of the pointed tip of the specimen than that of the edge of a cylindrical pellet. The methods are empirical, and strict observance of the test conditions is necessary to obtain reproducible results and these are laid down in the US ASTM (1968), British Standard (1970), German (DIN 1976) and French (Norme Francaise, 1945) procedures.

It has been recognized by many researchers that although ash fusion tests can given useful information regarding the fouling and slagging propensities of coal ashes, there are serious shortcomings. First, the tests are based on subjective observations and not precise scientific measurements, and they have a large margin of error. For example, the ASTM-method which is widely used in many countries allows for a 55 K margin of reproducibility in the initial deformation, softening hemisphere temperatures in an oxidizing atmosphere, and a 70 K margin of uncertainty in determining the initial deformation temperature in a reducing atmosphere. Within that 70 K margin the viscosity can change by an order of magnitude and consequently the rate of ash sintering will change by the same factor.

Another, more serious, shortcoming with the ash fusion method is that when testing some coal ashes there occurs an extensive degree of particle-to-particle bonding without any visible sign of deformation in the shape of an ash pellet. It is, therefore, evident that an additional method is needed to assess the sintering characteristics of coal ashes. A choice of sinter measuring techniques is given in Table 2.

TABLE 2. REVIEW OF ASH SINTERING TECHNIQUES

Measuring Technique	Equipment	Comments
Neck-growth measurements between spherical particles (Kuczynski, 1949), Raask, 1973)	A furnace and a microscope	This is suitable for homogeneous material when available in the form of spherical particles. It is not suitable for routine sinter testing of coal ashes.
Simultaneous shrinkage and electrical conductance measurements (Raask, 1979)	Needs a purpose-built furnace assembly for more accurate measurements. A simpler version uses platinum wire electrodes as described by Raask (1979) and by Cumming (1980)	Each ash could be provided with sintering rate curves. The method needs to be tested and assessed by different researchers.
Crushing strength measurements of sintered ash pellets (Atting and Barnhard, 1963; Gibb, 1981)	A furnace and a crushing strength measuring device	The method has been used by several researchers but there is no agreed procedure
Ash agglomeration by sieving test (Stallman and Neavel (1980)	A furnace and a sieving machine	This is one of the simplest methods of testing for initial sintering, and it warrants more systematic tests
Ash plug flow method	A tubular furnace tube with perforated plate to support an ash plug	So far no experimental results have been found in literature

The brief review in Table 2 shows that there are several laboratory methods of sinter testing coal ashes, which can give some useful information regarding their deposit

forming propensity. However, none of these techniques could be written as recommended methods, and a coordinated test program involving specialist researchers in different laboratories would be required to assess their general applicability.

CONCLUSIONS

1. Frenkel's sintering model is a useful introduction to understanding of the mechanism of formation of boiler deposits in the crucial early stages of particle-to-particle bonding. The model sets out unequivocally the rate controlling parameters in sintering, namely surface tension (the driving force for particle coalescence) viscosity (the temperature sensitive parameter) and particle size.

2. Measurements of the rate of neck-growth between the spherical particles demonstrate the validity of the sintering model, but the technique is not suitable for routine assessment tests of the sintering characteristics of different coal ashes.

3. A method of simultaneous measurements of dilatometric shrinkage and electrical conductance has been developed for assessing the deposit forming propensity of coal ashes. The measurements are based on a sintering model which stipulates that the formation of particle-to-particle bonding leads to enhanced conductance and increased density of ash test samples and boiler deposits.

4. There are some coal ashes, rich in sodium which do not behave as predicted from sintering models. With these ashes the sinterpoint temperature defined by the electrical conductance measurements can be over 250 K lower than that indicated by the results of conventional ash fusion tests.

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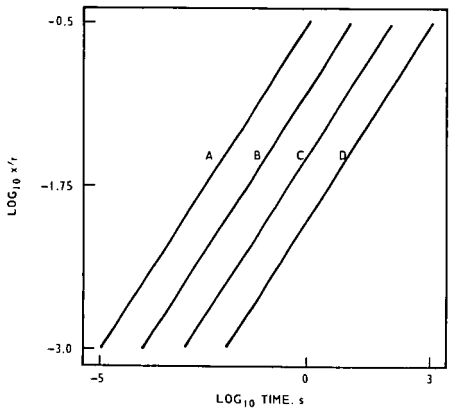


FIG. 1 LOG-LOG PLOT OF DEGREE OF SINTERING (x'/r) AGAINST TIME VISCOSITY – 10^8 N s m^{-2}

PARTICLE RADIUS: A – 0.05 μm B – 0.5 μm
C – 5.0 μm D – 50 μm

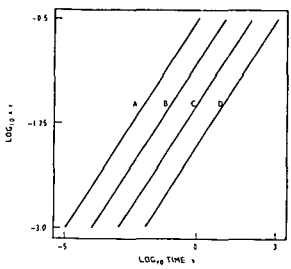


FIG 1 LOG-LOG PLOT OF DEGREE OF SINTERING (x r) AGAINST TIME VISCOSITY - 10^8 N s m^{-2}

PARTICLE RADIUS A - 0.05 μm B - 0.5 μm
C - 5.0 μm D - 50 μm

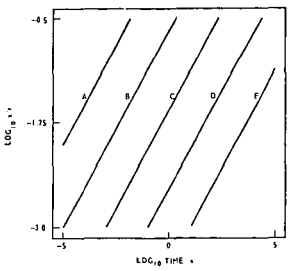


FIG 2 LOG-LOG PLOT OF DEGREE OF SINTERING (x r) AGAINST TIME PARTICLE RADIUS - 5 μm

VISCOSITY (N s m^{-2})

A - 10^4 B - 10^6
C - 10^8 D - 10^{10}
E - 10^{12}

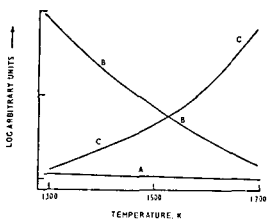


FIG. 3 THE EFFECT OF TEMPERATURE ON ASH SINTERING PARAMETERS

A - SURFACE TENSION B - VISCOSITY
C - RATE OF SINTERING

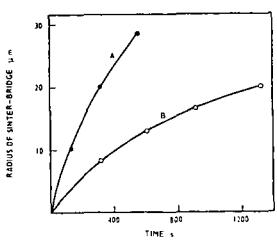
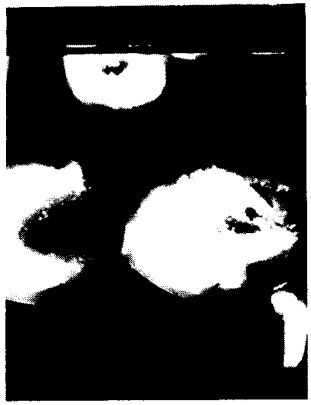


FIG 5 GROWTH OF SINTER BRIDGE BETWEEN PARTICLES, RADIUS - 60 μm

A - AT 1435 K B - AT 1375 K

10 μm

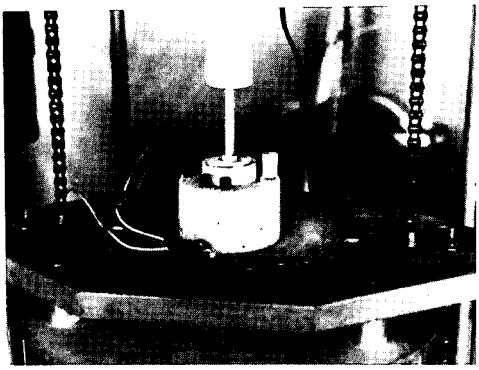
(a) SLAG PARTICLES ON PLATINUM PLATE BEFORE SINTERING



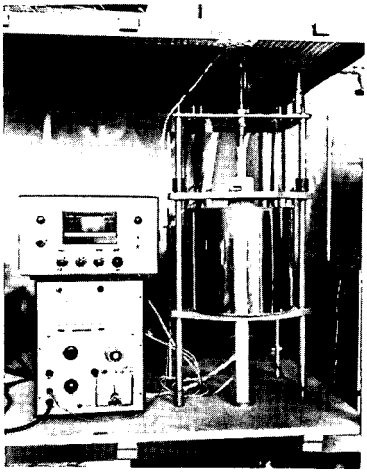
20 μm

(b) SINTER BRIDGED PARTICLES

FIG. 4 SINTERING OF SPHERICAL PARTICLES OF COAL ASH SLAG



(a) DOWN POSITION FOR SAMPLE CHANGE



(b) OPERATION POSITION

FIG. 7 FURNACE ASSEMBLY FOR THE ASH SINTERING MEASUREMENTS

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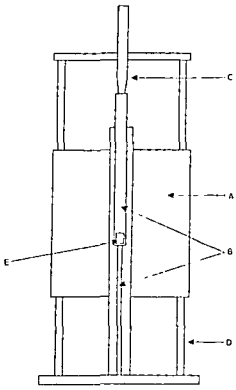

A FURNACE
B CONDUCTANCE LEADS AND THERMOCOUPLE
C DISPLACEMENT PROBE
D FURNACE HAS BALANCED LOWERING AND RAISING ACTION
E SAMPLE

FIG 6 SCHEMATIC OF FURNACE FOR SIMULTANEOUS SHRINKAGE AND CONDUCTANCE MEASUREMENTS

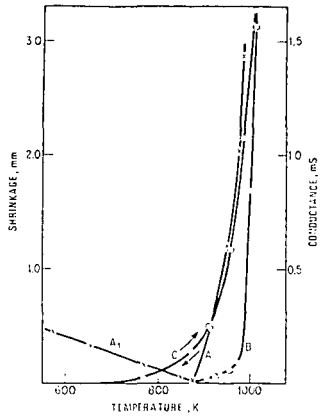

FIG 8 SIMULTANEOUS SHRINKAGE & ELECTRICAL CONDUCTANCE MEASUREMENTS OF SODA GLASS

A_1 - THERMAL EXPANSION
A - SHRINKAGE
B - CONDUCTANCE ON HEATING
C - CONDUCTANCE ON COOLING & REHEATING

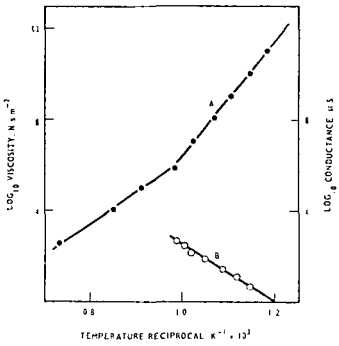

FIG 9 ARRHENIUS PLOTS OF VISCOSITY (A) AND CONDUCTANCE (B) OF SODA-GLASS

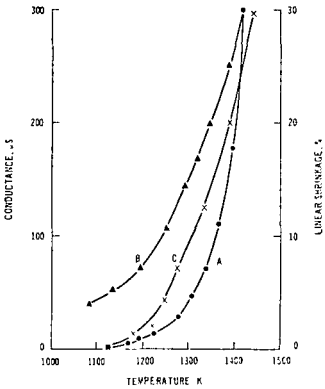

FIG 10 CONDUCTANCE AND SHRINKAGE PLOTS ON SINTERING OF ILLINOIS COAL ASH

A - CONDUCTANCE ON HEATING 5 K min^{-1}
B - CONDUCTANCE ON COOLING 5 K min^{-1}
C - SHRINKAGE ON HEATING 5 K min^{-1}

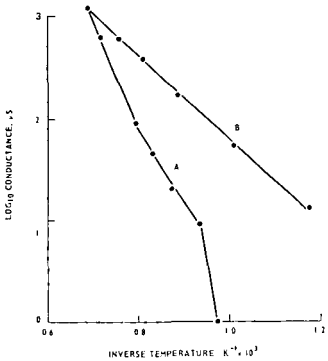

FIG 11 CONDUCTANCE LOGARITHM/INVERSE TEMPERATURE PLOT ON SINTERING OF COALITE BREEZE ASH

A – HEATING 5 K min⁻¹
B – COOLING 5 K min⁻¹

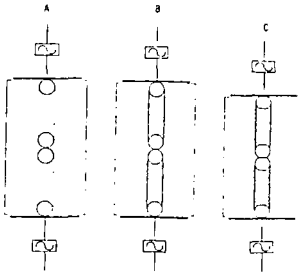

FIG 12 THREE STAGES IN THE SINTERING OF ILLINOIS COAL ASH – 12 mm HIGH, 8 mm DIA PELLET BETWEEN PLATINUM PLATES

	TEMP., K	COND, μS	SHRINK., %	STRENGTH, MN m⁻²
A FIRST CONTACT	1125	1.0	0.07	0
B INITIAL SINTER	1195	10	2.0	0.1
C RAPID SINTER	1280	100	10	1.3

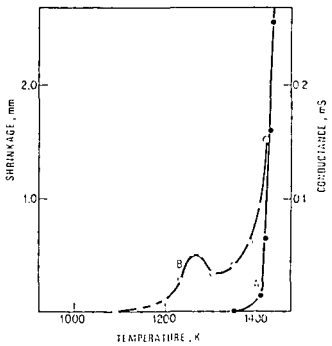

FIG 13 SIMULTANEOUS SHRINKAGE (A) & CONDUCTANCE MEASUREMENTS (B) LEIGH CREEK, AUSTRALIA COAL ASH