

# ANALYSIS OF HIGH TEMPERATURE COHESION OF ASH PARTICLES USING MODEL PURE SILICA POWDERS COATED WITH ALKALI METALS

Hidehiro Kamiya, Akira Kimura, and Makio Naito\*

Graduate School of Bio-Application System Engineering, BASE, Tokyo University of Agriculture & Technology, Koganei, Tokyo 184-8588, Japan

\* Japan Fine Ceramics Center, Atsuta-ku, Nagoya Japan

**ABSTRACT.** This paper analyzes the mechanism of increasing cohesive behavior of ash powders at elevated temperatures. Model ash powders were prepared from fine pure silica powder coated on the surface with 0.5 wt% alkali metal. The cohesive behavior and deformation properties of model ash powder beds were determined by using a new split type tensile strength tester of powder beds. The behavior of fly ash collected in pulverized coal combustion and model ash powders were compared. In the high temperature range above 1100 K, a rapid increase of tensile strength and plastic deformation of the ash powder beds occurred in both the natural ash and the model silica particles. It is suggested that the coated alkali metal on the silica powders reacted with the amorphous silica phase and formed a small amount of low melting point eutectic materials.

## INTRODUCTION

The increase of stickiness and adhesion force of ash powder at high temperatures hinders the stable operation and scale-up design of various high efficiency coal combustion power generation system, for example, the integrated coal gasification combined cycle (IGCC) and pressurized fluidized bed combustor (PFBC) systems. Dry ash particle deposition and the growth of a deposited ash layer on the water-cooled wall blocked the gasifier in IGCC systems and pulverized-coal combustors<sup>1</sup>. In PFBC systems, the operation of filter cake detachment on rigid ceramic filters by reverse-pulsing has difficulty with the increase of the adhesive force of ash<sup>2</sup>. These cohesive properties of ash particles at these high temperatures depend on many physical and chemical factors.

In our previous paper we measured the tensile strength of fly ash powder beds by using a diametal compression test of ash powder pellets<sup>3</sup> and a new split type tensile strength tester of ash powder beds<sup>4</sup> at high temperature at a range from room temperature to 950°C. We reported that the tensile strength of ash powder beds rapidly increased over a range of 800°C.

This paper analyzes the mechanism of increasing cohesive behavior of ash powders at elevated temperatures by using model ash powders, which were prepared from fine pure silica powder coated on the surface with 0.5 wt% alkali metal. The cohesive behavior and deformation properties of model ash powder beds were determined by using a new split type tensile strength tester of powder beds. Based on the comparison with these results of fly ash and model powders, the increasing mechanism of ash powder stickiness at high temperature is discussed.

## EXPERIMENTAL PROCEDURE

### Model ash powder preparation and characterization

The original powder samples used a commercial pure fused amorphous silica powder and fly ash. Fly ash powder was collected in a commercial pulverized coal combustion plant. The chemical component and particle size distribution of each sample were determined using ICP and a

centrifugal sedimentation method shown in Table 1. The total impurity such as Na, K, etc. in the high-pure amorphous silica powder was about 13 ppm. The concentration of sodium and potassium in the fly ash was about 0.5%. The mean particle diameter of each sample was similar at about 5  $\mu\text{m}$ .

**Table 1 Mean diameter and chemical component of pure silica and fly ash powders**

Powder sample	Mean diameter	Chemical component [wt%]				
		SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	K <sub>2</sub> O	Na <sub>2</sub> O
Pure silica	5.3 $\mu\text{m}$	99.99	13 ppm			
Fly ash	4.75 $\mu\text{m}$	45.0	38.5	4.6	0.3	0.5

Based on the above results, model ash powders were prepared from a commercial pure fused silica powder. Firstly, pure silica powder was added to the sodium or potassium oxalate solution. The amount of silica particles added and the concentration of the oxalate solution was adjusted so that the alkali metal concentration would become 0.5 wt% (silica) after drying. The silica suspension was mixed for one minute by a planetary type mixer and dried in an oven at 120°C. After grinding with a pestle and mortar, dried silica powders with alkali oxalate were heat treated at 620°C for 1 hour, causing thermal decomposition and removal of the oxalate.

#### **Split type tensile strength tester for powder beds**

The detail of a split type tensile strength test at high temperatures was described in our previous paper<sup>4</sup>. The powders were placed in a circular cell 5 cm in diameter with a depth of 1 cm. The cell can be divided equally into a stationary and a movable part; both parts were prepared with high-purity silica glass. After fixing the mobile part using a hook, powder samples were packed into the cell and consolidated by uni-axial pressing (2.5 kPa for 600 s). Tensile testing was carried out at various temperatures ranging from room temperature to 1173 K, with an electric furnace at a heating rate of 600°C/h. After heat treatment at each temperature for 540 s before the tensile test, the hook used to fix the mobile cell is removed, and the mobile cell is pulled in each high temperature conditions. The relationship between load and displacement during loading was then measured.

## **RESULTS AND DISCUSSION**

### **Tensile strength and deformation behavior of fly ash and pure fused silica**

Examples of tensile load and displacement relationship at room temperature and 1123 K are shown in Fig. 1. The brittle failure of both powder beds was observed after elastic deformation at room temperature. In the case of the pure silica powder sample, the brittle failure of the powder bed appeared at 1073 K. On the contrary, in the case of fly ash samples, viscous deformation appeared near the maximum load at 1073 K. Under conditions of relatively low temperatures below 1000 K, the adhesive force of both powders increased gradually in proportion to temperature. The tensile strength of the pure silica powder bed gradually increased, even in elevated temperatures (> 1073 K). However, a rapid increase of tensile strength of fly ash powder beds was observed in the high temperature range (> 1073 K).

### **Model ash powders**

To analyze this rapid increasing tensile strength of fly ash above 1100 K, the relationship between tensile strength and deformation behavior of alkali metal coated pure silica powder beds with

different coating condition at different temperature were measured and shown in Fig. 2. The rapid increase of tensile strength with viscous tensile deformation was observed by a coating of 0.5 % alkali metal on the surface of pure silica powders in a high temperature range above 1073 K.

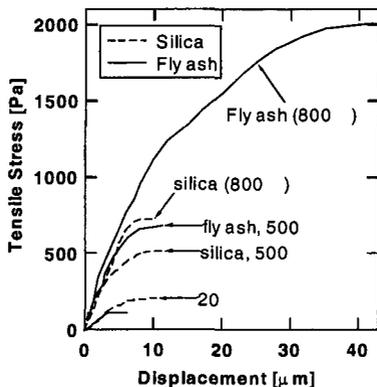


Fig. 1. Examples of tensile stress and displacement relationship of ash and silica.

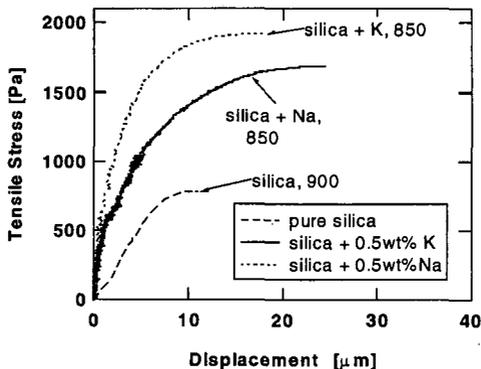


Fig. 2. An example of the tensile stress and displacement relationship of model ash powder beds prepared from pure amorphous silica powder and alkali metals.

The effect of testing temperature on the tensile strength of model ash powder beds with different alkali coating conditions is shown in Fig. 3. Regardless of the alkali metal used, the tensile strength of the model powder beds exhibited an increase with a temperature rise above 1073 K equal to that shown in the fly ash samples. This indicates that the coated alkali metal on the silica powders reacted with the amorphous silica phase and formed a small amount of low melting point eutectic materials. Hence, above 1073 K, a small amount of liquid phase formed between silica particles consisting of amorphous silica and alkali metal. It is suggested that a small amount of low melting point eutectic materials, consisting of amorphous silica and alkali metal, formed a liquid phase and this was responsible for the majority of the increase in the adhesive force mechanism of the ash powders at high temperature. This liquid phase mix was estimated from the increase in rupture displacement during tensile test of powder beds at high temperature.

## CONCLUSION

Model ash powders prepared from pure amorphous silica powder and coated alkali metal were used to reproduce the rapid increasing tensile strength of ash particle stickiness and deformation behavior in high temperatures above 1073 K. We suggest that low melting point eutectic materials consisting of amorphous silica and alkali metal formed a small amount of liquid phase between particles and promoted the increase of ash adhesion behavior.

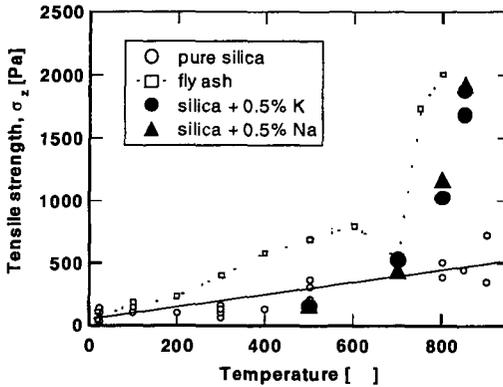


Fig. 3. The effect of temperature on tensile strength of model ash powders prepared from pure amorphous silica powders coated with sodium and potassium

**Acknowledgment:** This work is supported by the Proposal-Based New Industry Type Technology R&D Promotion Program from NEDO of Japan and Grant-in-Aid for Scientific Research (B), Japan.

## REFERENCE

1. D.H. Smith, G.J. Haddad and M. Ferer : *Energy & Fuel*, 11, 1006-11 (1997)
2. H. Kamiya, K. Deguchi, J. Gotou and M. Horio : "High Temperature Gas Cleaning II" p. 111-118 (1999)
3. H. Kamiya, A. Kimura, M. Horio, J.P.K. Seville and E. Kauppinen : *J. of Chem. Eng. Jpn.*, accepted.
4. H. Kamiya, A. Kimura, T. Yokoyama, M. Naito and G. Jimbo : *Pow. Tech.*, to be submitted.