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Analysis of Ash Adhesion Behavior at High Temperature Condition by Using Computer controlled FE-SEM with Heat Treatment Unit

To analyze the increasing mechanism of cohesive and adhesive force of ash powders from a coal combustion system at high-temperature conditions, a new observation system, which was composed of computer controlled FE-SEM and chamber unit for the heat treatment, was developed. By using this system, the liquid phase formation on the surface of pressurized fluidized bed coal combustion ash samples was observed after heat treatment at 1123 K, which temperature was corresponding to the rapid increasing temperature of adhesion behavior of ash powder samples.

1. Introduction

The increase of the stickiness and adhesion force of ash powder at high temperatures hinders the stable operation and scale-up design of various high-efficiency coal-combustion powergeneration systems, for example, the integrated coal gasification combined cycle (IGCC) and pressurized fluidized bed combustor (PFBC) systems¹. The cohesive properties of ash particles at these high temperatures depend on many physical and chemical factors. However, these phenomena have been measured and analyzed in few papers^{2, 3}.

In our previous work, we measured the tensile strength of fly ash powder beds by using a diametal compression test of ash powder pellets⁴ and a new split-type tensile strength tester of ash powder beds⁵ at high temperature at a range from room temperature to 1273 K. We reported that the tensile strength of ash powder beds rapidly increased over a range of 1073 K. Furthermore, modified ash powders were prepared from fine pure amorphous silica powder coated and heat-treated on the surface with 0.5 wt% sodium and/or potassium. Both modified ash samples can reproduce the rapid-increase phenomena of tensile strength and the plastic deformation behavior of ash samples collected in pressurized fluidized coal combustion systems in high temperatures above 1073 K as shown in Fig.1. The alkali metal and the amorphous silica phase in ash particles reacted and formed low-melting-point eutectic materials during the coal combustion process, for example, K₂O·4SiO₂, which melting point was about 1040 K, and formed a small amount of liquid phase between particles at about 1073 K. The liquid phase appeared to form a bridge between ash particles and promote the increase of ash adhesion and plastic deformation behaviors. However, since these eutectic materials are evaporated in high vacuum conditions, it is impossible to observe the liquid-phase formation in a SEM system with a hot stage.



Fig. 1 Effect of temperature on tensile strength of ash, pure silica and modified ash powder.

In this present paper, in order to observe the liquid phase formation phenomena on the surface of ash particles at high temperature conditions, a new observation system, which was composed of computer controlled FE-SEM and chamber unit for the heat treatment at the atmospheric pressure, was developed. By using this system, the mechanism of increase of adhesion force of coal combustion ash was discussed.

2. Experimental procedure

The developing system in this study was composed of computer controlled FE-SEM and chamber unit for the heat treatment. The scheme of this system was shown in Figure 2. Firstly, the ash powder sample was put on the computer controlled stage and inserted FE-SEM. Since the surface spattering of Pt and/or carbon on ash sample had a influence on the liquid phase formation at high temperature, high resolution observation of ash samples by FE-SEM was carried out without spattering, and photographic image was memorized with the coordinate in the computer. The stage with ash powder sample was taken out from the FE-SEM, and put into the chamber unit for heat treatment. The ash sample on the stage was given heat treatment at the ranging from 973 to 1173 C for 3 min under atmospheric pressure conditions in this chamber. Finally, the stage with ash powder sample was again inserted in FE-SEM after heat treatment, and moved a memorized position of each sample and observed by FE-SEM. Ash powder samples used were pressurized fluidized bed coal combustion ash.



Fig. 2 Schema of a computer-controlled FE-SEM system with heat treatment unit.

3. Result and discussion

An example of the morphology change of PFBC ash after heat treatment at different temperature up to 1223 K by using this FE-SEM system are shown in Figure 3. After relatively low heat treatment temperature at 1073 K, the shape and morphology of ash sample was almost same. Since the small amount of liquid phase on the surface of some coal combustion ash particles was formed at 1123 K, the surface morphology of some particles became smooth and some ultra fine particles with several ten nm in diameter on micron sized particles were melted after heat treatment. However, other particles were stable after heat treatment. The temperature of liquid phase formation was corresponded to the rapid increasing temperature of adhesion behavior of ash powders.

Furthermore, in the case of high temperature treatment at 1173 K, the sintering and neck growth between micron-sized particles were observed. Since the liquid phase formation and viscous flow sintering took place between ash particles, the adhesion force and tensile strength of ash powder bed continued to increase rapidly with elevating temperature ranging over 1123 K.

4. Conclusion

By using this FE-SEM system with high temperature heat treatment unit, the surface structure and morphology change of some ash powder sample after heat treatment up to 1300 K could be observed. The liquid phase formation on the surface of pressurized fluidized bed coal combustion ash samples was observed after heat treatment at 1223 K, which temperature was corresponding to the rapid increasing temperature of adhesion behavior of both ash powder samples. A part of particles only observed liquid phase, and other almost particles were stable at 1223 K.



Fig. 3 CC-FE-SEM observation of PFBC ash after heat treatment at different temperature

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