



## Full Length Article

# Determination of the initial ash sintering temperature by cold compression strength tests with regard to mineral transitions



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## HIGHLIGHTS

- Initial ash sintering temperatures were detected by cold compression strength tests.
- Three different ashes were investigated at oxidizing, inert, and reducing conditions.
- Sintering relevant phases were found by SEM/EDX, high temperature XRD, and TG-DSC.
- Silicates, iron-aluminum-oxides, carbonation, and iron sulfide melts induce sintering.
- Sintering can be connected with melt formation or solid phase sintering.

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## ABSTRACT

Ash deposition, fouling, and slagging are commonly undesirable occurrences in coal utility boilers and gasifiers. Ash deposit formation is usually initiated by sintering of the particles. The sintering characteristics of the ash have to be evaluated to understand the process of deposit formation. Cold compression strength (CCS) tests on heat treated ash pellets produced under defined conditions can be applied for the determination of the initial sintering temperature (IST). Scanning electron microscopy (SEM) in combination with energy dispersive X-ray spectroscopy (EDX), high temperature X-ray diffraction analysis (HT-XRD), and thermogravimetric differential scanning calorimetry (TG-DSC) are analytical methods to characterize the mineral transitions responsible for sintering.

CCS tests were done on ashes of three different coals to determine their IST. For this purpose ash pellets were pretreated at different temperatures. The influence of oxidizing, inert, and reducing conditions at ambient pressure was investigated. The IST were compared with the characteristic temperatures from ash fusibility tests (AFT). HT-XRD, TG-DSC, and SEM/EDX were applied for a more detailed characterization of sintering.

The IST determined by CCS tests are considerably lower than the respective initial deformation temperature (IDT) obtained by AFT. Thus, the AFT is not suitable to detect IST. Major mineral transitions were found which are responsible for initial sintering in the case of the investigated ashes. The formation of silicates or the crystallization of iron-aluminum-oxides were found to induce sintering at oxidizing and inert conditions. Sintering at reducing conditions was initiated by carbonation of calcium compounds, silicate formation, or melting of iron sulfide. These phases can be understood as the relevant phases for initial strengthening of fouling deposits in coal utility boilers and gasifiers in the case of the investigated ashes.

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## 1. Introduction

The thermochemical conversion of coals, e.g. for syngas production or energetic utilization, involves various drawbacks that come along with the coal's ash behavior like slagging and fouling. Hence,

the knowledge of the temperature of agglomeration onset gains high importance. The initial sintering temperature (IST) is commonly used to specify the initiation of ash particle agglomeration [1]. Sintering describes a heat induced mass transport in a loose or packed bed of particles [2]. The particle bed is strengthened and shrinks due to inter-particle material bonds and filling of the void fraction. The driving force is the reduction of the free surface energy, which is kinetically controlled by the temperature [3]. The

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## Nomenclature

### Abbreviations

AFT	ash fusion test (–)
BSE	back scattered electron (–)
CCS	cold compression strength (–)
EDX	energy dispersive X-ray spectroscopy (–)
FT	fluid temperature (°C)
HT	hemispherical temperature (°C)
HT-XRD	high temperature X-ray diffraction (–)
IDT	initial deformation temperature (°C)
IST	initial sintering temperature (°C)
SEM	scanning electron microscopy (–)

ST	softening temperature (°C)
TG-DSC	thermogravimetric differential scanning calorimetry (–)

### Symbols

$A_0$	base area (m <sup>3</sup> )
$F_{max}$	maximum force (N)
$\Delta_R H_m^\circ$	reaction enthalpy (J/mol)
$R_{B/A}$	base to acid ratio (–)
$x_{50,3}$	median particle diameter (m)
$\sigma_D$	compression strength (Pa)

temperature at which the surface energy starts to decrease due to mass transport can be called the initial sintering temperature. Above this temperature the ash particles tend to agglomerate. The determination of the IST and its relation to mineral transitions are the main focus of the present work. A well established experimental investigation to determine ash melting characteristics is the ash fusibility test (AFT) according to international standards (e.g., ASTM D 1857 [4], DIN 51730 [5], ISO 540 [6]). These standards mention the initial deformation temperature ( $IDT_{AFT}$ ) as the lowest characteristic temperature. However, agglomeration may already occur several hundred degrees below the  $IDT_{AFT}$ , as Stallmann and Neavel observed [7,8]. Huffman et al. [9] observed first molten phases already 200–400 K below  $IDT_{AFT}$ , which lead to ash sintering and agglomeration. Hence, a reliable IST cannot be detected by AFT.

Several approaches can be found for the determination of the IST. Neuroth et al. [10] or Bartels et al. [1] present overviews about possible methods: Dilatometry [1,11,3], combined differential thermal analysis and thermogravimetric analysis [12,13], thermal conductivity analysis [14,15], electric conductance [16], thermochemical equilibrium calculations [17] or yield stress [1,18]. The results of the IST vary between the different methods. This makes the IST a value which is specific to the applied method.

In this work the main focus lies on the cold compression strength (CCS) test of previously heat treated ash pellets. Pelletized ash samples are treated at different temperature levels for a specific dwell time. After cooling down of the pellets to ambient temperature they are tested for their compression strength. Drawing the compression strength over temperature an increase in the compression strength indicates the  $IST_{CCS}$ . Similar experiments were carried out, e.g., by Hupa et al. [19], Ots and Zerkowski [20], Nel et al. [12], Skrifvars [21,22], and Al-Otoom et al. [14]. The CCS measurements are widely accepted as a reliable indicator for the IST [1].

For this reason this method is used in the present work for the determination of the  $IST_{CCS}$  of three different coal ashes. The  $IST_{CCS}$  will be compared with  $IDT_{AFT}$  and a self-defined  $IST_{AFT}$ .

Additionally to the temperature, the surrounding atmosphere has a strong influence on the sintering occurrences of ash systems. In iron containing ashes and reducing conditions, e.g., elemental iron and wustite (FeO) are formed instead of hematite (Fe<sub>2</sub>O<sub>3</sub>) [23]. Whereas, wustite is a strong fluxing agent [24,9]. Wustite and iron sulfides, such as pyrite (FeS<sub>2</sub>) and pyrrhotite (Fe<sub>1-x</sub>S), form an eutectic at a temperature of 910 °C [25,26]. Together with calcium sulfide wustite forms an eutectic at 890 °C [26]. Furthermore, iron silicates can be built with relatively low melting temperatures in the presence of silicon oxide. To account for the influence of the atmosphere the following experiments were conducted in oxidizing, inert, and reducing atmosphere. Air was used for oxidizing,

nitrogen (99.999% purity) for inert, and a mixture of 40 vol.% carbon dioxide in carbon monoxide for reducing conditions. The sintering tendencies at different atmospheres were evaluated with the support of high temperature X-ray diffraction analysis (HT-XRD), thermogravimetric differential scanning calorimetry (TG-DSC) and scanning electron microscopy (SEM). The results will be compared with the standardized AFT.

## 2. Material characterization

Three ashes from different coals were analyzed for their sintering tendency. The coals are a Lusatian lignite (LBK), a hearth furnace coke made of Rhenish lignite (HOK) and a South African hardcoal (SAK). The coals were ashed in a muffle furnace at 500 °C to avoid devolatilization of chlorides, alkaline, and alkaline earth metals [27,28,1] and to suppress the decomposition of carbonates and sulfates [29]. Thus, components which essentially affect the sintering behavior remain inside the ash. The resulting ashes consist of fine particles with diameters of  $x < 300 \mu\text{m}$  and median diameters of  $x_{50,3} = 19 \mu\text{m}$  (LBK),  $x_{50,3} = 13 \mu\text{m}$  (SAK) and  $x_{50,3} = 10 \mu\text{m}$  (HOK) (optical measurement with a CamSizer XT, Retsch Technology).

### 2.1. Elemental composition

Table 1 shows the elemental distribution of the three ashes and the resulting base-to-acid ratio

$$R_{B/A} = \frac{\text{Fe}_2\text{O}_3 + \text{CaO} + \text{MgO} + \text{Na}_2\text{O} + \text{K}_2\text{O}}{\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{TiO}_2} \quad (1)$$

**Table 1**

X-ray fluorescence analysis of the 500 °C ashes.

	LBK	SAK	HOK
CO <sub>2</sub>	5.39	6.38	20.10
Na <sub>2</sub> O	0.06	0.68	4.10
MgO	9.12	3.08	14.96
Al <sub>2</sub> O <sub>3</sub>	3.79	21.50	3.78
SiO <sub>2</sub>	13.78	44.24	0.82
SO <sub>3</sub>	21.57	1.75	11.90
Cl	0.23	–	0.42
K <sub>2</sub> O	0.29	0.69	0.60
CaO	23.09	7.35	31.39
TiO <sub>2</sub>	0.19	2.17	0.21
Fe <sub>2</sub> O <sub>3</sub>	21.95	10.20	13.46
BaO	0.11	0.29	0.30
P <sub>2</sub> O <sub>5</sub>	0.03	1.18	0.03
Traces	0.41	0.49	0.39
$R_{B/A}$	3.07	0.32	13.43

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