



Full Length Article

The influence of crystallization on the flow of coal ash-slugs



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ABSTRACT

Numerous technical applications in the energy and metallurgical industries demand a fundamental knowledge of the flow of slags. In particular, the operation of an entrained flow gasifier is challenging, as the oxide slag has to be reliably discharged. Crystallization in the slag influences strongly the flow behavior of the slag because precipitations occur. In this study, the process of crystallization during flow of two coal ash slags was investigated. Therefore, isothermal viscosity measurements were conducted in order to examine the rheological evolution over time caused by the crystallization. It has been demonstrated that the evolution of viscosity of a sub-liquidus melt depends strongly on time, as well as on temperature and composition. Using a rotational high-temperature viscometer to investigate coal slags, it was found that the crystallization during flow could be separated into three time regimes: a lag-time, in which the undercooled melt behaved as an Arrhenius-liquid; the kinetic-driven crystallization; and, finally, the rheological equilibrium that is represented by a constant viscosity. Furthermore, an increase of relative viscosity caused by crystallization was accompanied by a shift from Newtonian to non-Newtonian flow; here, pseudoplastic flow indicated the existence of precipitations. The results demonstrate that the flow behavior has to be divided into dilute, semi-concentrated and concentrated particle bearing fluids. A view into the morphology of the partly crystallized slag was taken by scanning electron microscope. Differential thermal analysis of the slags was conducted, to underline the results of the isothermal viscosity measurements. The degree of supercooling promotes the kinetics of crystallization. Our results demonstrate that time-dependency has to be considered for an accurate description of flow during crystallization, as well as the influence of degree of supercooling.

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1. Introduction

An effective use of the world's fossil fuels is mandatory in terms of their limited supply and the worldwide increase in demand for energy. Besides the combustion of coal in traditional steam-electric power stations with their limited efficiency and flexibility, gasification technologies are a promising way to convert high or low-grade fuels into a chemical energy carrier like a synthesis gas. During gasification carbon feedstocks are converted at high temperatures and elevated pressures into a syngas. This syngas can be flexibly used either for power generation or as reactant for chemical industry. Therefore, the gasification fulfills future requirements regarding flexibility and efficiency.

Gasification demands a fundamental knowledge of the thermo-physical and thermochemical processes during the conversion of the fuel. Especially the entrained flow gasification requests an essential understanding of the physical properties of the non-

converted inorganic residue. These residues segregate from the gas stream. The high process temperatures up to 1600 °C cause the ash to melt and flow down the inner wall of the gasifier, where it forms a complex slag layer containing liquid, partially crystalline or even solidified phases depending on temperature and composition as shown schematically in Fig. 1. The slag layer protects the refractory material, but an efficient flow has to be maintained to prevent a blockage of the slag tap, which may lead to an unscheduled shutdown. Consequently, the slag viscosity is one of the most important parameters for the operation of an entrained flow gasifier. To ensure a continuous flow of the slag a viscosity below 25 Pa s is desirable [1].

Numerous experimental investigations of the fully molten slag have been conducted to investigate the influence of composition and temperature on the viscosity as summarized for example by Vargas or Allibert [2,3]. The flow of the fully molten slag can nowadays be well described and predicted based on the inner structure of the oxide melts. The dependency on temperature for fluids in general is described by the Arrhenius-law [4] which can be expressed as:

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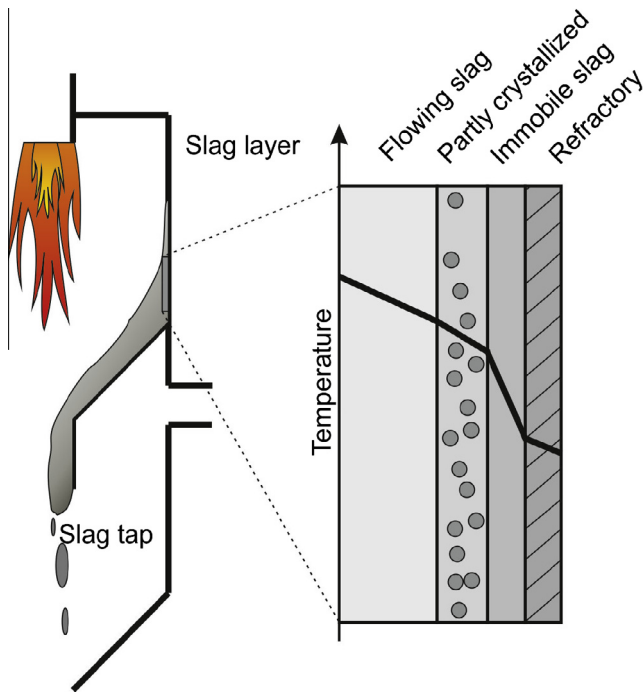


Fig. 1. Slag layer at the wall of an entrained flow gasifier.

$$\eta = A \cdot \exp(E/(R \cdot T)) \quad (1)$$

where η is the dynamic viscosity; T is the absolute temperature; E is the activation energy for viscous flow; R is the gas constant. The decrease of temperature results in an exponential increase of viscosity according to the Arrhenius-law. The influence of pressure is for the most technical applications negligible, because of the low effect of pressure on fluids. Silicate melts behave in this regard similar [5]. The influence of composition on the viscosity is more complicated. Based on the network theory postulated by Zachariassen [6] already at the beginning of the last century, oxides are categorized regarding their role in the network as modifiers, intermediates or formers. Molten slags are treated as a mixture of oxides, such as SiO_2 , Al_2O_3 , CaO , MgO , Na_2O and K_2O . According to their specific role that each oxide plays for the viscosity they are categorized into network former (e.g. SiO_2), network modifier (e.g. CaO , MgO , Na_2O and K_2O) or amphoteric (e.g. Al_2O_3). A network former can increase the viscosity, whereas a network modifier usually decreases the viscosity by breaking the bonds between silicon ions and oxygen ions of the tetrahedral structure. An amphoteric, as the name itself implies, can behave as either a network former or a network modifier, depending on the composition. Since the network theory different empirical and semi-empirical models such as the Urbain-model [7,8] have been proposed to describe the flow of the slag. These models are mostly limited to a specific range of temperature or composition. Using modern thermochemical tools to determine the distribution of associated species that link to the inner structure of the oxide melt, the viscosity of fully molten slags can be described well, including different challenging viscosity behaviors induced by the self-polymerization of silica as well as the charge compensation of an amphoteric by network modifier [9,10].

However, some slags exhibit a very rapid increase of viscosity caused by the appearance of crystalline phases in the sub-liquidus range. The change of viscosity deviates from the Arrhenius-law. The so-called temperature of critical viscosity (T_{cv}) restricts the minimum temperature in the gasifier, as the viscosity strongly increases out of boundaries of operation [1]. The temperature of critical viscosity is defined as temperature at which

the viscosity is strongly increased with temperature [3]. Different mechanisms for the increase of viscosity are postulated, which are referred to a transition from fully molten to partially crystalline slags [11–14]. The temperature of critical viscosity marks the boundary between crystal affected and non-affected flow. The crystalline phases increase the viscosity and also influence the flow behavior, therefore non-Newtonian flow may occur [11,15].

The prediction of partially crystalline slags is the next challenging step in the description of oxide melts. Although a lot of work has been done for fully molten slags, there are less number of publications regarding partially crystalline slags. Oh et al. [16] illustrated the influence and the importance of crystalline phases on the viscosity of coal-ash slags and the difference between glassy and crystalline slags. Nowok [11,17] demonstrated that the viscosity trajectory of ash slag is intimately related to the concomitant microstructure in the viscosity-phase transformation relationship. Phase equilibria needs to be considered to predict the viscosity near and below the temperature of critical viscosity. The appearance of crystals was accompanied by a change from Newtonian to non-Newtonian flow. The same effect was reported by Song et al. [18]. Both results indicate that a transition to shear-thinning flow occurs caused by the precipitates. Groen et al. [19] investigated the gasification slag rheology and crystallization in titanium-rich, iron-calcium-alumosilicate glasses, and showed that in this system the iron content is an important parameter. This observation was supported by Ilyushechkin et al. [14]. Additionally, Groen addressed the crystallization process during flow to the kinetics, thus to the influence of time and temperature onto the mechanics of crystallization. The effect of time and temperature was also observed by Kong, who investigated the effect of the cooling rate on viscosity of coal ash slags [20]. The results demonstrate that the cooling rate influences the viscosity below the temperature of critical viscosity besides the exponential Arrhenius effect. The viscosity of the slag significantly decreased with the increasing cooling rate due to a decreased amount of crystals in the slag. Song et al. [21] postulated results that indicate the evolution of viscosity and flow behavior during the cooling process of coal ash slags at isothermal state is strongly dependent on time.

In this study the crystallization of two coal ash slags during flow was investigated. Therefore, isothermal viscosity measurements were conducted, in order to investigate the influence of crystallization on the flow of coal ash slags. The results indicate that time and temperature are vastly influencing the viscosity of the slag below the liquidus. Both have to be considered to describe the crystallization of coal ash slags during flow. The in-situ observation of crystallization allows the characterization of kinetic parameters of crystallization and the visualization in time-temperature-transformation diagrams, which indicate crystal-affected areas depend on time and temperature. The flow behavior was strongly affected by the precipitations. A transformation to a non-Newtonian flow was observed. Moreover, thermoanalytical investigations of the coal ash slag were conducted to underline the results of the viscosity measurements.

2. Experimental

2.1. Preparation and chemical composition of the samples

Two German coals were chosen for this study the lignite HKT and the hard coal ST-D-2, because of their relevance regarding coal gasification. The coals were prepared firstly by crushing and grinding before the investigation. The ash samples of both coals were prepared by removing the combustible and volatile matter. Therefore, the coals were burned in an ashing furnace at a temperature of 815 °C for 24 h in a well-mixed air atmosphere. The amount of

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