



Characterization of carbon-bonded alumina filters with active or reactive coatings in a steel casting simulator

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Abstract

Carbon-bonded alumina filter materials—with and without coatings of carbon-bonded MgO and carbon-free Al₂O₃—were tested in a steel casting simulator in contact with steel melts containing defined endogenous alumina inclusions. For the evaluation of the filtration efficiency of the different filter surfaces, prismatic samples were immersed in the steel melt under an argon atmosphere at 1650 °C. The carbon-bonded alumina filter material reacted in situ in the steel melt, forming gaseous alumina sub-oxides. These sub-oxides reacted with the dissolved oxygen of the steel and formed a thin crystalline α-alumina layer on the area in contact with the steel. The secondary alumina layer was formed on the carbon-bonded alumina surface as well as on the alumina-coated carbon-bonded alumina substrate. The newly formed layer reflected the roughness of the substrate, influencing the wettability and, therefore, the deposition behavior of the inclusions on the filter material surfaces tested. Sintered fine endogenous inclusions were identified in various shapes on this thin layer. In the case of carbon-bonded magnesia on a carbon-bonded alumina substrate, carbothermal reduction of the magnesia and subsequent reoxidation to secondary magnesia in the form of a thin layer on the contact area with the steel was observed. Based on the carbothermal reduction of alumina and magnesia, MgAl₂O₄ spinel whiskers were formed in situ in the hollow filter struts.

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1. Introduction

The mechanical properties of cast steel products are strongly related to the cleanliness of the melt they come from. Both endogenous and exogenous nonmetallic inclusions influence mechanical strength, fatigue resistance and fracture toughness remarkably. Due to the increasing pressure on the steel industry to produce clean, high-quality steel, the importance of enhancing existing steel filtration techniques as well as the development of materials and methods with higher filtration efficiencies are important goals for the steel and refractory industries. The non-metallic inclusions that need to be removed from steel melts are mainly oxides, carbides, nitrides, and sulfides [1].

Different sources of endogenous and exogenous inclusions include reoxidation, slag entrainment, lining erosion, and inclusion agglomeration on linings. Endogenous inclusions include products of deoxidation or inclusions precipitated during cooling and solidification. If aluminum is used as a deoxidation agent, a high proportion of endogenous alumina particles formed in situ may be identified in a variety of different shapes. Alumina inclusions readily form three-dimensional clusters via collision and aggregation due to their high interfacial energy [2]. In investigations with a confocal scanning laser microscope of the agglomeration behaviors of alumina and other kinds of typical inclusions (such as, for example, spinel, or inclusions that have liquid oxide solution or calcium aluminate phases), behaviors contradictory to the more readily agglomerating alumina particles were observed. Alumina particles attracted each other and agglomerated. Spinel and solid calcium aluminate inclusions, however, did not show any signs of attraction or agglomeration [3].

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Two main morphologies of alumina inclusions are observed: dendrites and clusters. However, faceted, plate-like, and spherical inclusions are also reported. The formation of aggregates and their subsequent growth are attributed to the attachment of single particles whenever inclusions meet. Small spherical inclusions could account for 90% or more of the number of inclusions throughout the secondary metallurgy treatment. However, large inclusions represent from 60% to almost 100% of the oxide volume [4].

The morphology depends on the activities of the materials components in the steel bath and the degree of supersaturation, as well as the aluminum and oxygen contents and the cooling speed of the steel [5,6]. To elucidate the behavior of deoxidation products in steel, it is important to clarify the relationship between primary oxides formed immediately after the addition of deoxidizers and secondary and tertiary oxides, which are formed during cooling and solidification [7]. In Aneziris et al. [8] filter approaches with a surface functionalisation based on so called “active” as well as “reactive” coatings are described. In case of the “active” coatings, the same chemistry as the chemistry of the primary or secondary inclusions that have to be removed are generated on carbon bonded filters. On the other hand, “reactive” coatings react with the dissolved gas in the melt (for instance, oxygen in steel melts) and create inclusions above the liquidus temperature of the melt that are deposited on the filter. With this approach, less tertiary and quaternary inclusions are generated below the liquidus temperature. [9,10]

Steel is deoxidized with various deoxidizers such as Al, Fe–Al, Si, Fe–Si, Ca–Si, Ca–Al, Ti, Fe–Ti, or Zr. The reaction products are fine oxide inclusions, which in spite of their lower density do not completely separate into the slag phase under industrial processing conditions [11]. Particles larger than 30 μm normally float up to the slag in steel melts due to buoyancy forces.

A lot of research work concerning the filtration mechanisms has been carried out in recent decades. For instance, researchers have compared extruded monolithic alumina (400 cells per inch) and alumina packed-bed systems, achieving inclusion removal efficiencies of up to 96% for laboratory melts. The results show that inclusion removal efficiency is a strong function of the melt velocity in the range of 0.08–0.86 cm per second and is weakly dependent on filter length. The filtration mechanisms could be categorized as (a) surface filtration, and (b) depth filtration [11].

For steel filtration ceramic filters in the shape of open porous foams, honeycomb-like structures or spaghetti in different types of stacking are successfully applied today in the steel industry [6,12–16]. Most of the filters used today are open-cell foam filters produced using the replica process [17]. The main filter materials used for steel filtration today are zirconia or carbon-bonded alumina filters. Due to the bonding of the materials, zirconia filters exhibit deformation and shrinkage during the filtration process, which is caused by a glassy bonding matrix and possible reactions with e.g. manganese containing steel. Carbon-bonded alumina material exhibits higher creep resistance [16].

Alumina is often viewed as a chemically inert material under reducing conditions at elevated temperatures. Khanna et al. had

shown that alumina cannot be regarded as chemically inert at steel-making temperatures when carbon, alumina, and molten iron are present simultaneously. Reactions between alumina, carbon, and steel leads to CO gas generation, carbon pickup of the steel, and the formation of new secondary phases. Khanna et al. [18,19] refer to the “catalytic contribution” of melted iron. Carbothermal reduction without any metallic solvent normally takes place in the temperature range of 1727–2200 °C [20,21]. If a “metal solvent agent” such as tin or copper is incorporated in the mixture of carbon/alumina, the carbothermal reaction is completed up to approximately 80% in 60 min at lower temperatures [21].

In the present study, three different surfaces—alumina, carbon-bonded alumina, and carbon-bonded magnesia—were tested to examine their clogging behavior in contact with artificially created endogenous alumina particles using a so-called steel casting simulator. In an oxygen-free argon atmosphere, the prismatic filter samples were dipped into steel melts containing defined particles created in-situ, before being removed and allowed to cool down under inert conditions. This procedure allowed the investigation of filter surfaces after a simulated filtration with adsorbed particles without the presence of solid steel, as in the case under normal industrial trials. Normally, the study of filters from normal castings is hindered by the presence of solid steel all around after use. The influence of oxygen on the properties of, in particular, the carbon-bonded materials (from carbon oxidation) as well as on the properties of the melt (from dissolved oxygen) and the particles contained therein could be avoided. With the use of less and cleaner materials in direct contact (one alumina/spinel-containing crucible per melt), the influence of impurities from refractory materials could almost be eliminated.

2. Materials and methods

For the evaluation of the filtration efficiency of different functional coatings on carbon-bonded alumina filter materials, prismatic 10 ppi filter samples ($125 \times 20 \times 20 \text{ mm}^3$) were produced using the replica process as presented in detail in Emmel and Aneziris [19]. The samples consisted of 30 wt% carbon and 70 wt% alumina after pyrolysis in a carbon grid at 800 °C. Coatings of alumina or carbon-bonded magnesia were applied to these carbon-bonded alumina substrate material, which were then thermally treated at 1400 °C (alumina) or 800 °C (magnesia/carbon) under a reducing atmosphere (a carbon grid). The preparation of the carbon-bonded alumina filter material as well as the coating are explored in detail by Aneziris et al. [22,23]. The compositions of the base material and the coatings are presented in Table 1.

To characterize the different coatings in direct contact with inclusions containing steel melt, a so-called finger test was selected. In Fig. 1 the principle setting of the steel casting simulator used is presented. For each sample, 38 kg of commercially available steel 42CrMo₄ (Mat. no. 1.7225, AISI 4142) in a cylindrical shape (9 cm in diameter, 12 cm long) were melted under a fully controlled argon atmosphere without any detectable oxygen inside the furnace atmosphere.

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