

Reaction mechanism between the carbon bonded magnesia coatings deposited on carbon bonded alumina and a steel melt

Anton Salomon^{a,*}, Milan Dopita^a, Marcus Emmel^b, Steffen Dudczig^b,
Christos G. Aneziris^b, David Rafaja^a

^a Institute of Materials Science, TU Bergakademie Freiberg, Gustav-Zeuner-Straße 5, 09599 Freiberg, Germany[†]

^b Institute of Ceramic, Glass and Construction Materials, TU Bergakademie Freiberg, Agricolastraße 17, 09599 Freiberg, Germany[†]

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Abstract

The carbon bonded magnesia (MgO–C) coatings deposited on the carbon bonded alumina (Al₂O₃–C) substrate are intended to be used as reactive filters for filtration of steel melts containing high oxygen levels and inclusions. The aim of this study was to describe the chemical reactions running at 1600 °C at the interface between the MgO–C coating and the liquid 42CrMo4 steel, at the interface between the MgO–C coating and the Al₂O₃–C substrate as well as at the interface between Al₂O₃ inclusions and the liquid steel. The MgO–C/Al₂O₃–C interface was prepared by slip casting. The steel/MgO–C interface was formed between the quickly molten metal and the MgO–C/Al₂O₃–C stack, the steel/Al₂O₃ interface between the quickly molten metal and the Al₂O₃ particles added to the steel melt. In order to imitate the temperature profile during a casting process and concurrently to avoid the metal convection, the spark plasma sintering was used for the steel melting. The chemical reactions at the interfaces were concluded from the phase formation after different reaction times. The results of the electron microscopy, X-ray spectroscopy and electron backscatter diffraction indicate the reduction of MgO and Al₂O₃ by carbon contained in the binder, the formation of CO gas, the evaporation of metallic Mg and Al and the dissolution of the vaporised metals in the steel. The above experimental methods revealed formation of MgAl₂O₄ whiskers and dense MgAl₂O₄ layers at the MgO–C/Al₂O₃–C and at the steel/MgO–C interfaces, respectively. The exogenous Al₂O₃ inclusions inserted into the steel melt were also converted to MgAl₂O₄, which proves that Mg from MgO–C was dissolved within the steel melt.

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1. Introduction

Modern filters for the metal melt filtration are expected to make the metallic melt almost free of inclusions. The surfaces of such filters are based on “active” and “reactive” coatings.¹ Recently, Aneziris et al.² introduced a new generation of reactive filters based on the MgO–C coatings deposited on the Al₂O₃–C substrates, which should: (a) remove the inorganic impurities and inclusions having the size from some millimetres to some sub-micrometres (active), (b) decrease the content of dissolved oxygen in the steel melt due to a reaction with

the oxides deposited on the filter structure (reactive), and (c) promote a non-turbulent melt filling of the mould.

The carbon bonded magnesia (MgO–C) has a lower thermal expansion and a higher thermal conductivity than MgO because of the graphite binder and a low wettability against slags and metal melts.³ These properties predestine the MgO–C to applications in converters, electric arc furnaces, steel treatment ladles and in high duty refractories.^{2,4} The major disadvantage of MgO–C refractories is the direct oxidation and consumption of carbon by oxygen containing atmospheres at steel casting temperatures (1550–1700 °C). When the MgO–C refractory is brought in contact with liquid steel, MgO is reduced by carbon to Mg and CO gas is formed (indirect oxidation).^{2–7} Elemental magnesium reacts with oxygen dissolved in the steel melt and forms secondary MgO as a dense, thin layer at the steel/refractory interface.^{5,8,9} In Ref. 5, this reaction path was

* Corresponding author. Tel.: +49 3731 39 2671; fax: +49 3731 39 2604.

E-mail address: anton.salomon@ww.tu-freiberg.de (A. Salomon).

[†] <http://tu-freiberg.de/fakult5/iww/index.html>.

used for the steel melt deoxidation. However, this reaction was accompanied by the concurrent dissolution of the carbon binder into the steel melt which partly reacts with dissolved oxygen to form further CO gas.²

On the $\text{Al}_2\text{O}_3\text{--C}$ side, the following has to be considered: analogous to the reduction of MgO in the presence of carbon, Al_2O_3 is also reduced to form metallic aluminium and carbon monoxide when carbon is present (Hall–Héroult process).^{10–12} Under atmospheric pressure, this reaction takes place only above 2200 °C. However, low vacuum levels result in much lower onset temperatures for alumina reduction (the onset temperature calculated for 0.1 Pa is 1370 °C).^{10,11} In the presence of iron, this reaction was reported to take place at a temperature as low as 1550 °C¹² even under atmospheric pressure (Ar) leading to the formation of alumina whiskers.

In systems more or less similar to our “three components stack” (steel/MgO–C/ $\text{Al}_2\text{O}_3\text{--C}$), MgAl_2O_4 was reported to grow in form of whiskers according to an oxidation–reduction reaction above 1400 °C in a powder mixture of MgO, C (graphite) and Al under CO/CO₂ atmosphere.¹³ This in situ formation of spinel whiskers was also utilised to increase the strength of MgO–C refractories. The addition of ferrocene ($\text{C}_{10}\text{H}_{10}\text{Fe}$) to a mixture of MgO, Al and liquid Novolak resin (carbon source) resulted in the formation of Fe particles that acted as a catalyst for the growth of spinel fibres at temperatures exceeding 1300 °C.¹⁴ In both cases, the growth of the fibre-like MgAl_2O_4 crystals or whiskers was attributed to vapour–solid (VS) or vapour–liquid–solid (VLS) reactions.^{13,14} Most recently, the formation of amorphous fibres containing Al, Mg and O at the interface between MgO–C and $\text{Al}_2\text{O}_3\text{--C}$ was reported² after dipping tests in 42CrMo4 steel.

In the present article, the MgO–C coating serves as an intermediate layer between steel and $\text{Al}_2\text{O}_3\text{--C}$ in such a way that there should not be a direct contact between the molten metal and the substrate, though, both ceramics ($\text{Al}_2\text{O}_3\text{--C}$ and MgO–C) are porous and the intermediate layer is relatively thin. The resulting “stack” was analysed; the study was focussed on the phase reactions occurring at the interfaces between MgO–C and $\text{Al}_2\text{O}_3\text{--C}$ and between MgO–C and the steel, respectively. Additionally, exogenous Al_2O_3 inclusions added to the steel are exposed to “reactive” and/or “active” filtration mechanisms in order to imitate the aluminium oxide inclusions resulting from deoxidation of the steel melt with aluminium during a real casting process.

2. Experimental

2.1. Utilised materials and production of the ceramic cylinders

Calcined alumina with the grain size (d_{90}) below 3.0 µm (Martinswerk, Germany) and electrofused magnesia (Refratechnik, Germany) with the grain size below 63 µm were used as the raw materials for the production of the hollow ceramic cylinders. Modified coal tar pitch powder 0–20 µm Carbores® P was used as binder as well as a carbon source (Rütgers, Germany), fine natural graphite grade AF 96/97 (<40 µm, Graphit Kropfmühl, Germany) and carbon black powder with a primary particle size

Table 1

Chemical composition of the utilised steel alloy 42CrMo4 as provided by the powder vender.

Element	C	Cr	Mo	Si	Mn	S	Fe
Mass%	0.40	1.00	0.19	0.20	0.70	0.01	Balance

of 200–500 nm (Lehmann & Voss & Co., Germany) were added. In combination with the liquid components based on deionized water, lignisulfonate T11B (Otto Dille, Germany), dispersing agent Castament VP 95L (BASF, Germany) and anti-foaming agent Contraspum K 1012 (Zschimmer & Schwarz, Germany), an impregnation slurry was produced in a high shear mixer, for each of the ceramics, $\text{Al}_2\text{O}_3\text{--C}$ substrate and MgO–C coating, respectively. Additional information about the specific carbon bonded ceramic production is given in Ref. 2. The coated cylinders exhibit the same composition as the AC9MC2 filters in this reference.

The steel powder used for the experiments was 42CrMo4 (TLS Technik GmbH & Co., Spezialpulver KG, Germany). The chemical composition as provided by the powder vender is given in Table 1.

2.2. SPS treatment of the samples

In order to simulate a real casting process, very high heating rates and very short processing times (30 s for the filtration of 1 tonne of filtered steel) are required. Spark Plasma Sintering (SPS) offers the possibility to achieve such process conditions.^{15–17} However, in contrast to the filtration processes, SPS samples are produced without a (macroscopic) flow of the steel melt. The interaction between the stationary counterparts (molten steel with inclusions and coated ceramic filter) was expected to favour the interface reactions against the mechanical interlocking of inclusions in the filter cavities during filtration.

The application of the SPS/FAST for the generation of metal melt had only been described in literature for the infiltration of C/SiC preforms with liquid silicon.^{18,19} The present article relies on a previous contribution and refers to the published results and methods for the reactions of 42CrMo4 steel in contact with $\text{Al}_2\text{O}_3\text{--C}$.²⁰

The sintering was done in a SPS/FAST (Field Assisted Sintering Technique) unit HPD 25 manufactured by FCT Systeme GmbH (Germany). The dimensions of the graphite tools (graphite R7710, SGL Carbon, Germany), of the graphite foil and graphite felt as well as a scheme of the SPS setup used for the sample production are given in Ref. 20. All experiments were performed in the temperature controlled mode, which means that a pre-set temperature profile was used and the electrical power was automatically adjusted to achieve the pre-set temperature (software ECS 2000 and SE607). Electrical direct current pulses with a pattern of 10:5 on:off-pulses (without interval pauses) were applied for the heating.

The $\text{Al}_2\text{O}_3\text{--C}$ mould coated with MgO–C having an outer diameter of 20 mm and a height of approximately 15 mm (hollow cylinder) was placed inside the high strength graphite die with an inner diameter of 20.6 mm lined with graphite foil.

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