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Additions of Salt Cake and $KAlF_4$ from Aluminum Production to Steelmaking Slags

Abstract: Fluorspar, a mineral primarily composed of CaF_2 is the most commonly used slag fluidizer in steelmaking. Due to increasing costs for fluorspar steelmakers are seeking alternative means of achieving slag fluidity. The research has concentrated on other minerals or the wastes of other high temperature operating industries. A promising alternative to fluorspar has been identified in a previous work from this laboratory which is the salt cake from secondary aluminum production such as the flux in remelting aluminum scrap and dross. This material is widely available and also considered toxic (meaning that use in steelmaking helps to reduce environmental impacts from disposal). The present work is an investigation of the viability of using mixtures of salt cake and $KAlF_4$ in slag-fluidizing applications by measurements of viscosity and weight loss at high temperatures (to evaluate whether large amounts of potentially harmful gases are formed). In addition, characterization of raw materials and melted slags has been performed. In terms of fluidity the mixtures of aluminum salt cake and $KAlF_4$ are qualified for use as fluidizers. The results of the present study will be used to design plant trials.

Keywords: steelmaking, slag, viscosity, fluorspar, mineralogy, salt cake, potassium fluoroaluminate

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1 Introduction

The role of slags in steelmaking processes is of great importance for the production and refinement of steel products. A slag is a mixture primarily composed of ferrous and non-ferrous oxides. The composition of the slag is responsible for the removal and separation of impurities from molten steel. An ideal slag will have sufficient chemical activity and dissolving power of impurities to remove impurities as well as recover useful metals from the slag, such as Fe and Mn [1]. A basic slag, composed primarily of CaO and MgO, possesses these characteristics but their relatively high melting temperatures exceed the operating temperatures for steelmaking. For this reason SiO_2 is added to achieve a melting temperature within the steelmaking range, about 1873 K. However, high SiO_2 content in slags increases acidity and presents higher viscosity and negative effects on slag-metal reactions [2, 3].

Since the reaction rates at the slag/metal interface increase with the fluidity of a slag, it proves beneficial to fluidize a slag with further mineral additions [3]. The most common fluidizer in the steelmaking is fluorspar, CaF_2. In relatively small amounts, the addition of fluorspar decreases slag viscosity while having no affect on acidity. Therefore, a slag composed of CaO, MgO, SiO_2 and fluidized with fluorspar will have the ability to remove impurities within a sufficient amount of time. However, the decreasing availability of fluorspar and the concern of possibly fluorosilicate off-gasses have led to the desire to replace this fluidizer [2].

Possible replacement fluidizers that have been investigated include potassium feldspar, labradorite, nepheline syenite tailings as well as waste salts from aluminum production [2–4]. Specifically, these waste salts include $KAlF_4$ from primary aluminum production and ‘Salt cake’ from secondary aluminum production, both of which are toxic waste products. The salt cake is composed primarily of sodium and potassium salts which have become oversaturated with alumina [3]. Consumption of these waste products in the steelmaking process would prove to be both environmentally and economically acceptable.

Slags with silica content can be thought of as polymeric liquids, where polymeric chains of varying length are dependent on the acidic or basic nature of the slag. In liquid form, silica forms networks of SiO_2^{2-} linked with O atoms [5]. These highly networked structures are responsible for the relatively high viscous nature of silica-rich slags.

Basic oxide additions break up silica networks by donating O^-. This decrease in chain length increases mobility, leading to a reduced viscosity and higher thermal conductivity [5]. Fluorine additions have a similar but more substantial impact, as they displace oxygen atoms and are incorporated into the slag structure [6].

Rheological work was completed using a rotating cylinder viscometer immersed in slag samples while slag temperature was controlled using a high-temperature furnace. After viscosity trials, samples were sectioned for electron probe microanalysis (EPMA) to determine mineralogy and chemical composition. Additionally, high-temperature mass loss (volatilization) experiments were conducted. Samples were taken before and after weight loss experiments for chemical and mineral analysis.

The purpose of this work is to investigate the effect of additions of salts from primary aluminum production, $KAlF_4$, and secondary aluminum production, spent salt cake on the fluidity of CMS slags. Experiments were performed to investigate the effectiveness of these salts as slag fluidizers at steelmaking temperatures. The objective of the present investigation is to reduce the consumption of fluorspar to a more acceptable level or eliminate it altogether.

In addition to the feasibility of these salts as fluidizers, their volatility at steelmaking temperatures was tested. Weight loss experiments were performed to quantify gas formation. Chemical analyses of these slags were performed before and after mass loss experiments to view the evolution of slag composition.

2 Experimental procedure

Both $KAlF_4$ and the salt cake fluidizers were tested in a master slag (CMS) consisting of 40 mass% CaO, 30 mass% SiO_2 and 10 mass% MgO and compared to a baseline slag fluidized with the conventional fluorspar fluidizer. The decimal compositions of these salt fluidizers are shown in Table 1 [2].

	$KAlF_4$ (wt%)	**Salt cake (wt%)**
Al_2O_3/AlF_3	52.1% AlF_3	46.4% Al_2O_3
CaF_2	0.3%	2.7%
Fe_2O_3	<0.1%	1.9%
KF/KCl	40.9% KF	13.4% KCl
MgO	<0.1%	4.7%
NaF/NaCl	<0.1%	22.4% NaCl
SiO_2	<0.1%	6.4%
Total	93.3%	97.9%

Table 1: Composition of $KAlF_4$ and salt cake fluidizers [2]

Trial #	**Composition (mass%)**			
	CMS Master	**CaF_2**	**$KAlF_4$**	**Salt cake**
1	80	20	0	0
2	80	0	20	0
3	80	0	15	5
4	80	0	10	10
5	80	0	5	15
6	80	0	0	20

Table 2: Slag composition

2.1 Preparation of slag

First, a CMS master slag was blended from a mixture of CaO, MgO and SiO_2 powders. This master slag was then blended with a fluidizer. The composition of the slag was modeled after a typical steelmaking slag and for comparison with previous work, the composition is fixed at 40 mass% CaO, 30 mass% SiO_2, 10 mass% MgO and 20 mass% fluidizer. The fluidizer composition of each slag sample is shown in Table 2. Once blended, samples were compressed in a cold isostaic press (CIP) to enhance particle interaction and to fit them into the crucible.

Reagent grade powders of CaO, MgO, SiO_2 and CaF_2, were used while $KAlF_4$ and salt cake are waste-products received from Steel Dynamics Inc. Weight loss measurements used the identical compositions as those in Table 2, However, due to the metallic aluminum entrapped in the salt cake and its detrimental effect on platinum, a synthetic substitution based on the composition in Table 1 was used.

2.2 Viscometry

A schematic of the viscometry apparatus is shown in Figure 1. The viscometer, a digital rotational Brookfield DV-11, was first calibrated with a variety of silicone oils

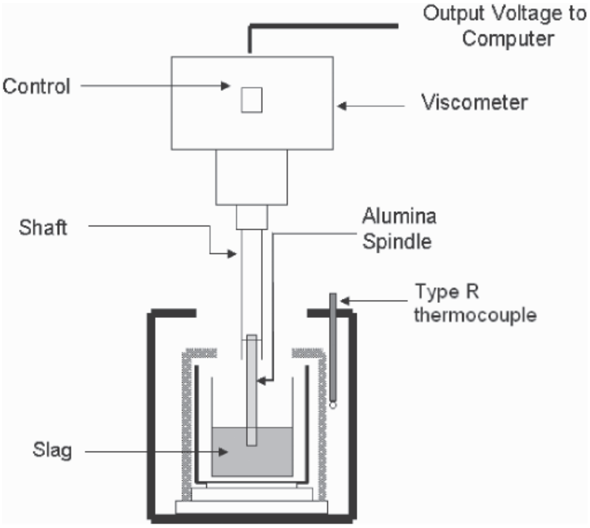

Fig. 1: Schematic of the viscometer

of known viscosities at room temperature. The output voltage recorded, by a Strawberry-Tree aquisition system, of the viscometer could then be correlated to a viscosity value.

Samples were heated slowly in a Lucifer MeltMaster elevator furnace, to 1873 K and held for 10 minutes to ensure thorough heating. The spindle of the viscometer, a 6.3 mm diameter Al_2O_3 cylindr, was then lowered into the slag which was contained in a 64 mm height, 24 mm inner diameter MgO crucible, and rotated at 30 rpm and the sample was allowed to cool at a controlled rate. A computer continuously recorded the viscosity output as well as temperature via an auxiliary thermocouple which is located outside the crucible and calibrated appropriately to reflect the actual temperature of the melt. Once the viscosity exceeded the tolerance of the viscometer data acquisition was terminated. Once cooled, samples were sectioned for electron probe microanalysis (EMPA) examination for phase identification and to examine the interaction of the slag/salt with the crucible/spindle.

2.3 Mass loss

To measure mass loss at high temperature, slag samples were placed into a platinum crucible which was hested in a DT-31-RS-OS-E2404 elevator furnace. Initial mass values were recorded using a Mettler scale accurate to two decimal places covered in fibrous alumina insulation. Samples were then held at 873 K for 30 minutes to allow for dehydration and the mass was re-recorded. Samples were then heated in increments 100 K, held for 30 minutes and the mass was recorded the temperature of 1873 K. The slag was then quenched to room temperature for chemical analysis.

2.4 Chemical analysis

The chemical analysis techniques used in this work include Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) as well as fluorine/chlorine fusion techniques. The chemical difference of a slag before and after heating allows a quantifiable value of volatility.

In ICP-OES, samples were dissolved in a total acid leach, where a mixture of HCl, HF, HNO_3 and $HClO_4$ dissolved all solids. Samples were then diluted to the appropriate concentration for analysis. The results produced in this technique yielded every metal and metalloid oxide present in a slag. However, due to the nature of fluorine and chlorine atoms, these elements required unique approaches to be quantified.

For fluorine, a sample was fused with Na_2CO_3 and KNO_3, and then dissolved in nitric acid. The solution was then tested using a fluorine-specific electrode. For chlorine, a sample was fused with sodium peroxide, and then dissolved in nitric acid. Furthermore, this analysis was carried out by potentiometric titration using silver nitrate.

2.5 EPMA

Electron Probe Micro-Analysis (EPMA), is an electron microscope with the capability of chemical analysis. Fundamentally, this is in principle similar to a Scanning Electron Microscope (SEM), with the addition of wavelength dispersive spectrometry (WDS) to energy dispersive spectrometery (EDS). Coupled with a backscatter detector, this piece of equipment allows for the determination of individual mineral phase determination and imaging. Furthermore, chemical point analysis can be taken on individual mineral grains for exact chemical makeup.

Slag samples from viscometry experiments were examined using this technique. WDS for this instrument was calibrated to optimally detect Al, Ca, Mg, Si, Na, K, Cl, F, O and S. Backscatter images of these samples were taken and individual grain analysis was performed. Furthermore, this technique allowed for qualitative evaluation of any corrosion on refractories.

3 Results and discussion

The results have been categorized under the following titles: viscosity, mass loss and chemical analysis. Under

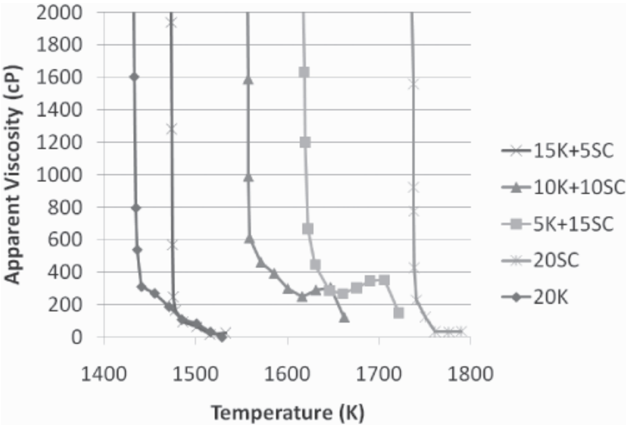

Fig. 2: Comparison of viscosity of various slags with different fluidizers

each heading the results are presented and analyzed. Slags are denoted as containing K, $KAlF_4$, and SC, salt cake.

3.1 Viscosity

Figure 2 shows the apparent viscosity of all the slag compositions shown in Table 2 as a function of temperature. In this figure K represents $KAlF_4$ and SC denotes salt cake in master slag. At steelmaking temperatures, roughly 1823 K, every composition tested appeared to be sufficiently fluid for steelmaking operations. The slag labeled '5K + 15SC' provided a plot very similar to that of the control slag. There are some significant similarities and differences between the slag compositions studied in this project. All slags exhibited a distinct point at which there was a rapid change from high to low viscosity, known as a transition point. However, this transition point, a characteristic of basic slags [5], was different for each slag composition. The transition temperatures for $KAlF_4$ and salt cake fluidized slags are plotted in Figure 3 as a function of the ratio of salt cake over the total amount of fluidizers. A linear trend line resulted. The ratio of salt cake to $KAlF_4$ as a fluidizer appears to have a direct affect on the transition temperature from high to low viscosity. It is presumed this is a direct correlation to the fluorine and chlorine content of the respective constituents. Fluorine, being more electronegative than chlorine, increases the degree of polymer-chain dissociation in a melt. Chlorine, being more electronegative element than oxygen still presents itself as a valuable component to dissociate polymer-chains in a slag, although less potent than fluorine. Overall, as the fraction of fluorine in the slag is reduced, and the chlorine fraction is increased, there is a proportional increase in transition temperature.

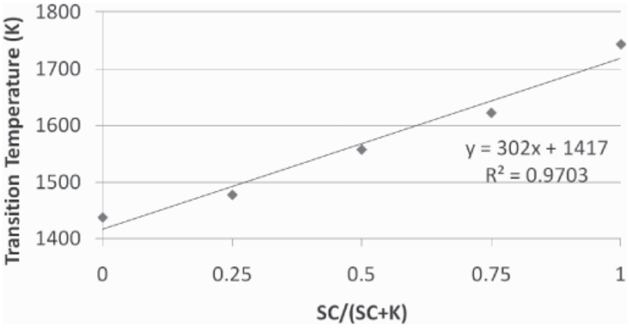

Fig. 3: Trend of transition temperature versus the ratio of salt cake and $KAlF_4$ fluidizers

All samples have an apparent viscosity that is acceptable at steelmaking temperatures. Furthermore, based on the composition of fluidizer, we can estimate the transition temperature in accordance with Figure 3.

3.2 Mass loss

The mass loss of each sample, outlined in Table 2, is important in the investigation of high temperature slag volatility. Figure 4 shows the mass loss of trials all salt cake and $KAlF_4$ fluidized slags versus the temperature at which the mass was recorded. While CaF_2 fluidized slags exhibit less than 1% mass loss at the highest temperatures, the salt cake and $KAlF_4$ fluidized slags suffer a much greater mass loss [2]. This means the volume of gasses produced at steelmaking temperatures are significantly greater than that of the traditional slag. Furthermore, samples appeared to have lost the majority of their mass below 1573 K, where the rate of mass loss decreased rapidly. The sample fluidized with 20% salt cake appeared to lose

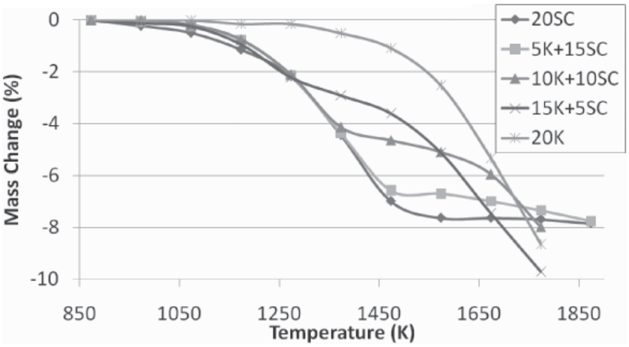

Fig. 4: Experimental mass change for $KAlF_4$ and salt cake fluidized slags

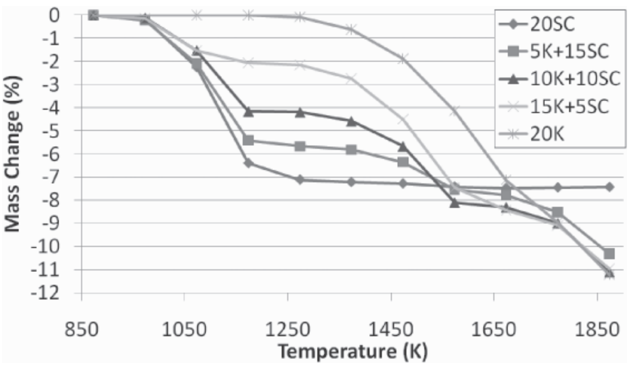

Fig. 5: Mass change for KAlF$_4$ and salt cake fluidized slags calculated using Factsage thermodynamic software

the majority of its mass below 1573 K, and very little at temperatures exceeding this. All other slag compositions showed continuing mass loss with increased temperature.

Figure 5 shows the mass loss of the samples outlined in Table 2 calculated using the Factsage thermodynamic software. The shapes of the curves on both the calculated and experimental results are similar. The primary difference between experimental and computed results is the magnitude of mass change at a specific temperature. This difference could be due to the kinetics of the system. The software delivers results which have achieved thermodynamic equilibrium, where experimental results have not.

3.3 Chemical analysis

Table 3 represents the volatile components of slag systems studied in this work both before and after performing weight loss experiments. Note that due to time restrictions, chloride fusion could not be completed. Chlorine is however included because, as previously seen, chlorine is expected to be a large component of volatile material produced. For all samples, potassium, sodium and fluorine experienced a significant decrease in mass percent.

Potassium was largely volatilized, while sodium commonly remained at a significant level.

3.4 Mineralogy (EPMA)

Table 4 summarizes the mineral content observed during EPMA sessions. In general, fluorine would mineralize into Cusidine, a Ca-Si-O-F mineral. Chlorine, however, did not appear to mineralize in this manner. Rather, chlorine was found in solution in relatively low concentrations throughout the bulk mineralogy. Points numbered on the proceeding figures represent the location of a compositional point-analysis, as described.

3.5 Bulk mineralogy

Bulk mineralogy was performed on viscosity tested samples to observe how their elemental constituents mineralized. To simplify this section, only the slag fluidizer labeled '10K + 10SC' from Table 2 will be discussed. Due to the mineral similarity between all fluidizer compositions, this composition best showed the effects of both KAlF$_4$ and salt cake additions. Figure 6 shows a backscatter image of the bulk mineralogy of this sample. Point 1 likely represents a fragment of the alumina spindle which was

Fluidizer	**Sample**	**Element (mass%)**			
		K	***Na***	***F***	***Cl***
20SC	**Before**	**3.9**	**3.99**	**0.21**	**–**
	After	*0.02*	*0.42*	*0.03*	–
5K + 15SC	**Before**	**4.93**	**3.77**	**1.48**	**–**
	After	*0.11*	*1.72*	*0.99*	–
10K + 10SC	**Before**	**5.76**	**2.92**	**3.52**	**–**
	After	*0.97*	*1.77*	*2.45*	–
15K + 5SC	**Before**	**6.87**	**1.8**	**4.54**	**–**
	After	*0.16*	*0.91*	*2.17*	–
20K	**Before**	**8.91**	**0.19**	**4.93**	**–**
	After	*0.79*	*0.33*	*3.01*	–

Table 3: Composition of slags, as outlined in Table 2, before and after weight loss experiments. Rows in italics represent the elements associated with the volatile phase.

Name	Composition	Liquidus Temperature (°C)
Alumina	Al_2O_3	2020
Magnesia	MgO	2764
Spinel	$MgO \cdot Al_2O_3$	2135
Merwinite	$3CaO \cdot MgO \cdot 2SiO_2$	1575
Monticellite	$CaO \cdot MgO \cdot SiO_2$	1490
Cusidine	$3CaO \cdot CaF_2 \cdot SiO_2$	1407
Gehlenite	$2CaO \cdot Al_2O_3 \cdot SiO_2$	1596
Akermanite	$2CaO \cdot MgO \cdot 2SiO_2$	1436
Melilite	Solution of gehlenite-akermanite	Dependent on composition
Variable alkali calcium aluminosilicates	K_2O or $Na_2O + CaO + Al_2O_3 + SiO_2$	Dependent on composition

Table 4: Nomenclature of minerals found in slag-refractory systems, their compositions and liquidus temperatures. Modified table from [3].

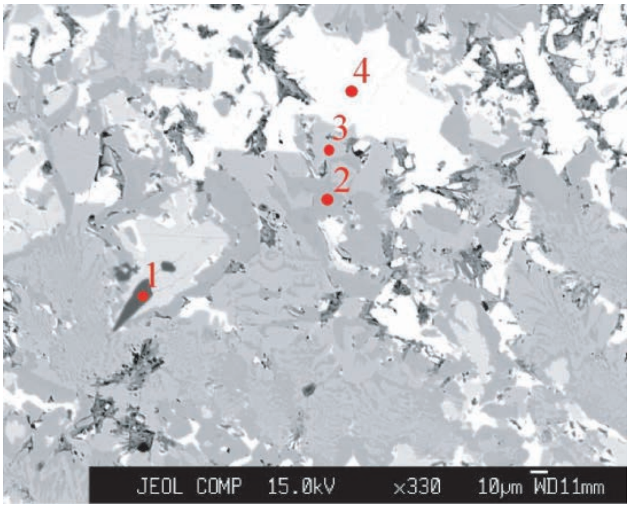

Fig. 6: Backscatter electron image on the bulk material of slag '10K + 10SC'. 1 = Spinel, 2 = Monticellite, 3 = Melitite, 4 = Cusidine.

chemically eroded by magnesia within the slag. Points 2 and 3 represent Monticellite and Melitite, respectively.

The fluorine content of the slag was represented by Cusidine, the lowest melting point compound discovered.

This low-melting point compound is likely responsible for the decrease in transition temperature shown previously in Figure 3. By increasing the amount of fluorine, via $KAlF_4$ additions, the volume fraction of cusidine that will form is increased. By increasing this, and the associated increase of liquids content at a given temperature, the viscosity of a melt will decreases. Therefore, it is logical that an increased $KAlF_4$ content is associated with a decreasing apparent viscosity.

Chlorine did not present itself as a stoichiometric component of any mineral. Rather, chlorine occurred in very low concentrations, rarely exceeding 0.50 atomic percent. It is assumed that any chlorine content added by the fluidizer was largely volatilized during the experiment.

Although there is no accompanying figure, both potassium and sodium were repeatedly discovered in a mineral state. These largely occurred as alkali calcium alumino-silicates [3]. Potassium values ranged from zero atomic percent in the purely Salt cake fluidized slag, to over 10 mass% in the $KAlF_4$ fluidized slag. Sodium was generally lower, ranging from zero to three atomic percent. Akin to chlorine, it is possible that sodium content was a more volatile component of the slag system.

3.6 Corrosion characteristics

Similar to the previous section on bulk mineralogy, viscosity samples were sectioned and investigated to observe any interaction between the slag and the alumina spindle, or the slag and the magnesia crucible. Previous work has been done on the corrosion of a CaF_2 fluidizer on a similar slag system [3] and it is reproduced here for comparison in Figures 7 to 9. For all intents and purposes, we can assume the alumina spindle and magnesia crucible to be representative of refractory brick in industrial applications. The magnesia crucible is analogous to the carbon impregnated magnesia refractory placed in areas that are constantly exposed to slag. Alumina is analogous to the alumina-based refractory which makes up the areas in intimate contact with molten steel.

Figure 7 shows the interface between the magnesia crucible, point 1, and the slag, point 2. In this, we can see a definitive region which appears darker than the majority of the magnesia. Upon great magnification of this darkened region, Figure 8, we can see the presence of a Cusidine phase, point 2, which has infiltrated magnesia particles, point 1. It appears the fluorine content of a slag is responsible for a portion of magnesia refractory wear.

Figure 9 shows the interface between the alumina spindle, point 1, and the slag, points 2–6. The degree of degradation appears more significant than that of the crucible. Of particular interest is the present of point 4, the spinel phase. This represents alumina material which has corroded with magnesia in the slag, liberating itself from the surface of the spindle. Furthemore, alumina may have

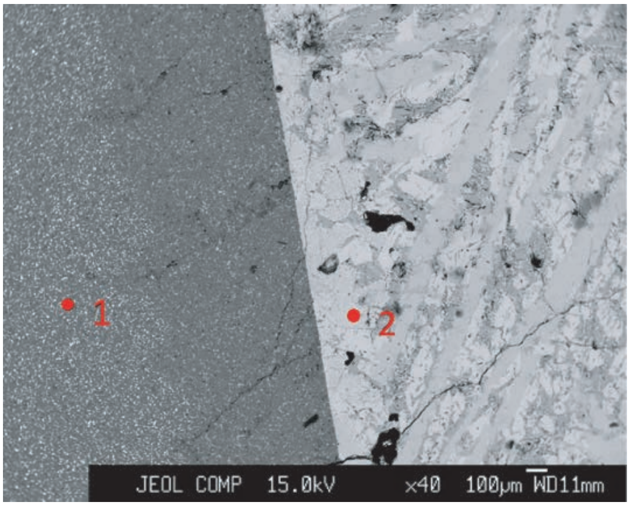

Fig. 7: Backscatter electron image of the ‘20F’ slag and MgO crucible interface. The darkened region in the crucible represents the area infiltrated by the slag [3]. 1 = Magnesia, 2 = Cusidine.

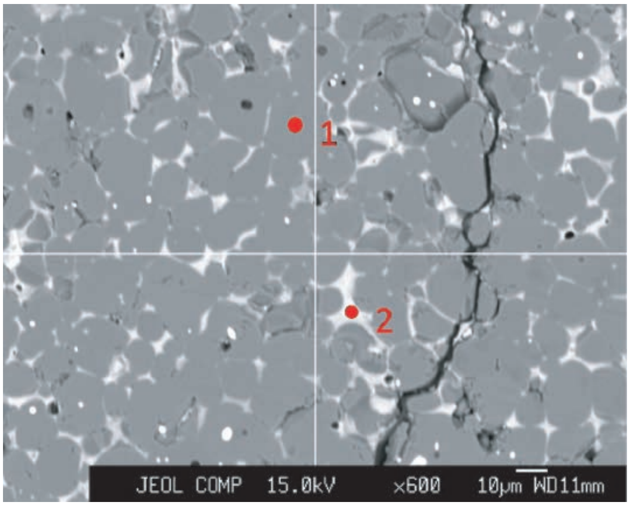

Fig. 8: Backscatter electron image of the MgO crucible infiltrated by the ‘20F’ slag. point 1 represents pure MgO, while point 2, the infiltrate, is Cusidine [3]

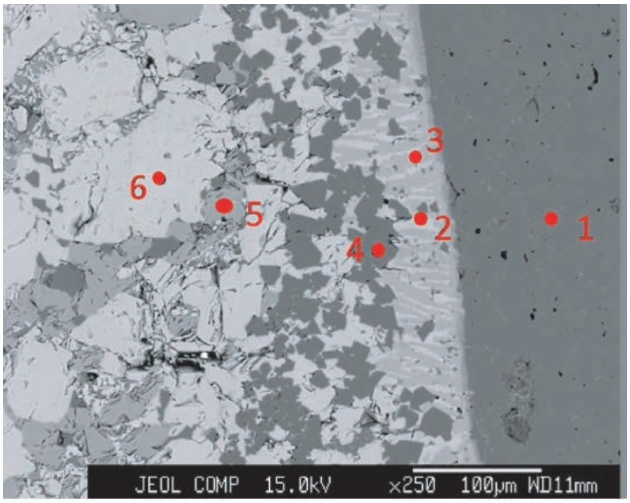

Fig. 9: Backscattered electron image and chemical analysis points of the ‘20F’ slag near the spindle. 1 = alumina, 2 = cusidine, 3 = gehlenite, 4 = spinel, 5 = monticellite, 6 = cusidine.

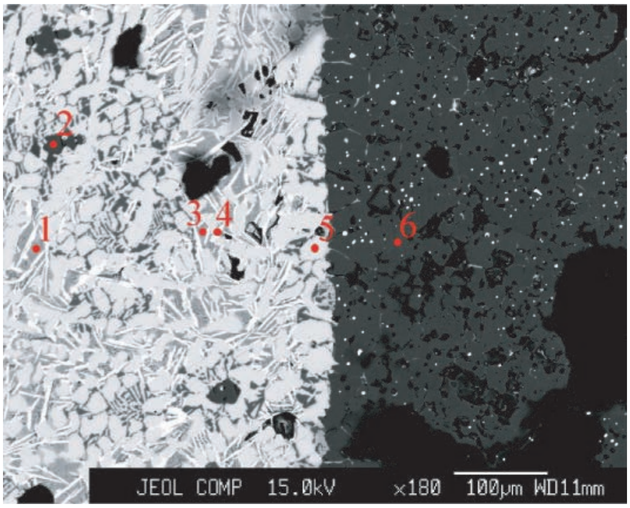

Fig. 10: Backscatter electron image of the interaction between the magnesia crucible and the ‘5K + 15SC’ slag. 1 = Melitite + Cusidine, 2 = Magnesia, 3 = Melitite + Cusidine, 4 = Melitite + Cusidine, 5 = Merwinite, 6 = Magnesia.

been dissolved in the formation of alkali calcium aluminosilicates. Regarding industrial practice, the exposure of a slag to alumina-based refractory is minimal (only during tapping and casting), and is therefore acceptable. Should slag make contact with this refractory, it is expected that a large degree of deterioration should occur.

The corrosion behavior of the slag fluidizer ‘5K + 15K’ as outlined in Table 2 is shown in Figure 10 which depicts the interface between the magnesia crucible, point 6, and the slag, points 1–5. In this, there was no apparent region of infiltration. Fluorine content within the crucible region was measured at 0.00 atomic percent. In comparison to Figure 4–6, this slag composition presents significantly less interaction with the magnesia crucible. Point 2 represents a magnesia fragment, showing that there is still an appreciable amound of degradation occurring, albeit by another mechanism.

Figure 11 shows the slag-spindle interface for the ‘5K + 15SC’ slag system. Akin to the CaF_2 fluidized slag, a significant degree of degradation to the alumina spindle. Similarly, this was due to the formation of spinel, which dissociated from the spindle. The degree of degradation

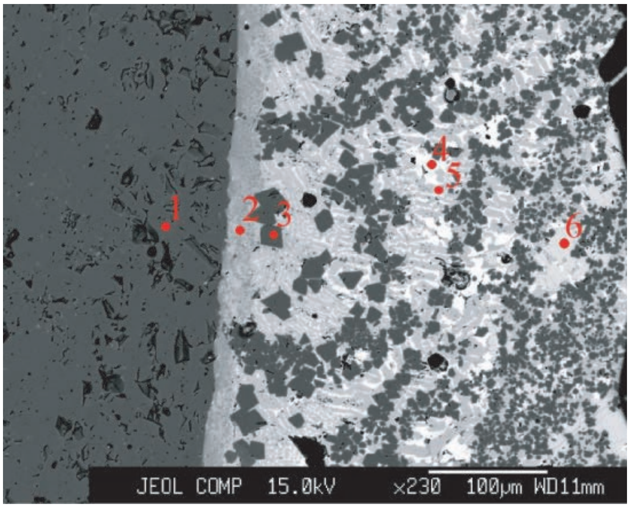

Fig. 11: Backscatter electron image of the interaction between the alumina spindle and the '5K + 15SC' Slag. 1 = Alumina, 2 = Gehlenite, 3 = Spinel, 4 = Cusidine, 5 = Melitite, 6 = Merwinite.

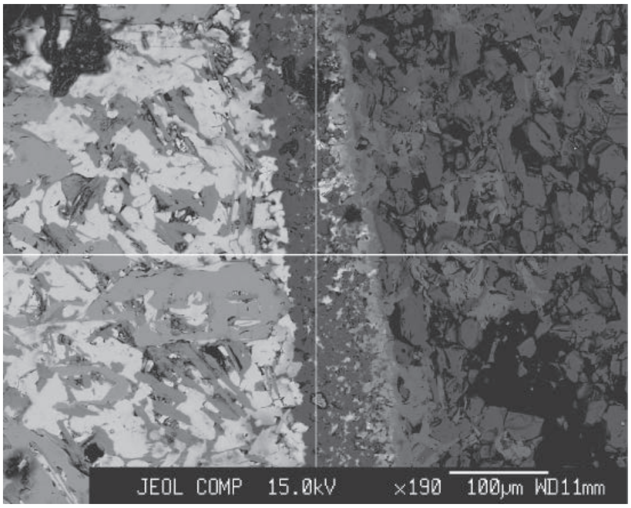

Fig. 13: 20 mass% KAlF$_4$-fluidized slag showing the interface between slag (left) and spindle (right)

of this slag on alumina based refractory is expected to be similar to that of a CaF$_2$ fluidized slag.

The same analysis was performed for the corrosion studies of the 20% KAlF$_4$-fluidized slag on the crucible and spindle. Figures 12 and 13 show the degree of interaction.

3.8 Plant trials

Based on the findings of the present work plant trials were conducted at the Steel Dynamics Flat Rolls facility in Butler, Indiana using a spar replacements composed of 25 mass% KAlF$_4$ and 75 mass% Salt cake. The results of the plant trials are analysed and they will be reported elsewhere.

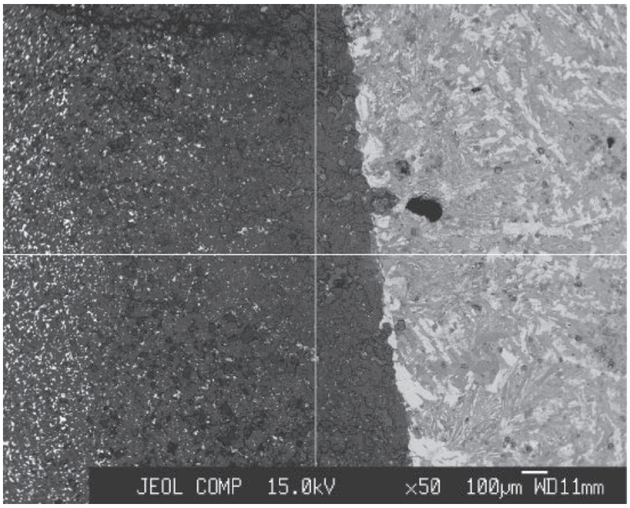

Fig. 12: 20 mass% KAlF$_4$-fluidized slag showing the interface between crucible (left) and slag (right)

4 Conclusions

It was determined through lab experiments that salt cake, KAlF$_4$, and mixtures thereof were all suitably fluid at steelmaking temperatures. As the composition was weighted more towards KAlF$_4$-rich, more cusidine phase would be present in the resultant microstructure when compared to salt cake-rich samples. This low-temperature mineral phase likely explains why compositions weighted heavily with KAlF$_4$ showed a depressed transition temperature. Fluidizers weighted heavier in salt cake displayed the highest transition temperatures. These were however still acceptable in steelmaking conditions. A composition of 25% KAlF$_4$ and 75% salt cake (denoted as '5K + 15SC') proved to have viscosity properties similar to that of a conventional, CaF$_2$ fluidized slag.

The transition temperature was a linear function of the ratio of the salt cake and KAlF$_4$ fluidizer content.

All slags showed degrees of volatility greater than that of fluorspar slags at steelmaking temperatures. Salt cake fluidized slags showed the greatest volatility. As the mass fraction of salt cake was incrementally replaced with KAlF$_4$ the volatility decreased correspondingly. Weight loss experiments showed significant mass loss at high temperatures for all trial fluidizer compositions. These volatile components were largely made of up potassium, sodium and fluorine. It is theorized that chlorine plays a large role in the volatility of slag systems.

The degree of corrosion of the magnesia crucible and alumina spindle in the ‘5K + 15SC’ slag system was compared to that of the ‘20F’ slag system (CaF_2 fluidized slag). Both systems showed equivalently large degrees of corrosion to the alumina spindle. It was apparent that the degree of corrosion to the magnesia in the ‘20F’ system exceeded that of the ‘5K + 15SC’ system. This was due to the presence of a cusidine phase in the ‘20F’ system, which infiltrated the magnesia grains. There was no apparent infiltration into the crucible in the ‘5K + 15SC’ system.

In general, it appeared that all fluidizer compositions investigated in this work proved to be sufficient slag fluidizers in the liquid processing of steel. On an industrial scale, the mixture of 25% $KAlF_4$ and 75% salt cake must be a suitable replacement for fluorspar.

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