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The effect of magnesia and alumina crucible wear on the smelting characteristics of titaniferous magnetite

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Synopsis

Test work was conducted to investigate the effect of magnesia and alumina crucible wear in laboratory-scale smelting tests on titaniferous magnetite (titanomagnetite) in terms of iron and vanadium recoveries as well as slag properties. The study was motivated by the desire to develop capacity to provide technical support to the minerals industry in the establishment and continuous optimization of industrial processes for the efficient extraction of Fe, V, and Ti from titanomagnetite. The tests were conducted using the smelting recipe used by Evraz Highveld Steel and Vanadium Corporation (EHSV); that is, the same titanomagnetite feed material and a dolomite flux to achieve a typical CaO:MgO ratio of 16:14 in the slag. The results of the test work demonstrated that chemical attack by the titania-bearing slag on the magnesia and alumina crucibles is inevitable. The crucible wear was more pronounced with magnesia crucibles than with alumina crucibles. The Fe and V recoveries to alloy in alumina crucibles were higher than those obtained in magnesia crucibles. However, the recoveries in both cases were comparable to the EHSV data. The phase chemical compositions of the best slags, in terms of Fe and V recoveries to alloy, from each crucible type were significantly different. In a magnesia crucible, the slag was composed mainly of perovskite ($CaTiO_3$), forsterite (Mg_2SiO_4), and monticellite ($CaMgSiO_4$). However, the phase chemical composition of the slag produced using an alumina crucible was similar to that of the typical EHSV titanomagnetite, comprising pseudobrookite solid solution ($MgTi_2O_5$-Al_2TiO_5), spinel [$(Mg)(Al,Ti)_2O_4$], and perovskite. The results have shown that both magnesia and alumina crucibles can be used to conduct laboratory-scale test work using the reviewed slag chemical composition. In addition, the more readily available alumina crucibles are better than magnesia crucibles in terms of Fe and V recoveries as well as the slag phase chemistry.

Keywords
smelting, titaniferous magnetite, crucible wear, slag characteristics.

Introduction

Titaniferous magnetite (titanomagnetite) deposits are found in many countries throughout the world, including Russia, China, and South Africa. In South Africa, the titanomagnetite deposits are found in the Bushveld Complex. The titanomagnetite deposits typically contain appreciable reserves of iron and vanadium with concentrations in the range of 38–58% Fe and 1.2–2.2% V_2O_5, as well as significant titanium concentrations of 12–21% TiO_2 (Gwatinetsa, 2013). Titanomagnetite is typically processed by smelting in the presence of a carbonaceous reductant and flux to produce a valuable vanadium-bearing pig iron and a slag containing significant concentrations of titanium dioxide (TiO_2, titania). The titanomagnetite slag is generally discarded on waste dumps. The metal is processed further to produce vanadium and steel products (Steinberg *et al.*, 2011). The global depression in the market and prices of metals, and increased operational expenses as a consequence of excessive electrical energy costs in some countries, including South Africa, have prompted the established titanomagnetite smelters to intensify their process technologies or close down. Furthermore, prospective smelters apply caution through comprehensive process feasibility studies before committing to an operational technology. In such cases, laboratory-scale test work is generally adopted as a cost-effective approach for testing a wide range of parameters under controlled conditions (Geldenhuys and Jones, 2011).

Mintek gained expertise and experience in titanomagnetite smelting processes as early as in the 1960s (Jochens *et al.*, 1969) and thereafter continued with innovative work on the subject (Boyd *et al.*, 1993). Substantial titanomagnetite processing work is included in numerous reports on collaborative projects conducted by Mintek and its clients over more than 45 years.

The intensification of, and likely innovation in, titanomagnetite smelting processes typically involves laboratory crucible tests to maximize vanadium and iron recoveries to metal and the deportment of titania to the slag phase. Laboratory test parameters controlled for the optimization of the extraction of vanadium, iron, and potentially titanium from titanomagnetite

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generally include reductant addition, operational temperature, and fluxing regime, which subsequently affect the characteristics of the by-product titania-bearing slag such as basicity, viscosity, activities of slag components, and liquidus temperature. The laboratory-scale smelting of titanomagnetite generally entails numerous challenges relating to test crucibles, *viz.* crucible compatibility in terms of the effects on smelting characteristics and temperature limits (Geldenhuys and Jones, 2011). Titania-bearing slag is known to be corrosive towards all refractories, hence commercial smelting furnaces operate with a freeze lining to assist with the protection of the sidewall against corrosion (Pistorius, 2004). In their study of the interaction of MgO- and Al_2O_3-based refractories with titania-bearing slag, Garbers-Craig and Pistorius (2006) showed that both refractory systems are attacked by low- and high-titania slags. At the laboratory scale, chemical attack of both the MgO- and Al_2O_3-based refractories by the titanomagnetite slag is inevitable. The crucible wear generally results in changes in the slag composition, and thus the slag characteristics and overall smelting characteristics. Magnesia crucibles are not readily available in South Africa and generally come at a high cost compared to alumina crucibles.

The focus of the current laboratory-scale study was therefore to investigate the smelting characteristics of titanomagnetite in both MgO and Al_2O_3 crucibles in terms of the vanadium and iron recoveries to metal, and slag properties such as phase chemical composition and basicity. The smelting recipe used by Evraz Highveld Steel and Vanadium Corporation (EHSV) was adopted to allow the laboratory test results to be compared with commercial plant data.

Experimental

Materials and equipment

Titanomagnetite from the Main Magnetite Layer (MML) of South Africa's Bushveld Complex deposit was supplied by EHSV. The typical bulk chemical composition of the titanomagnetite feed to the EHSV process was acquired from the literature (Steinberg *et al.*, 2011), and that of the supplied sample was determined at Mintek. These compositions are given in Table I. As desired, the chemical compositions show that the feed samples are in fact from the same orebody. The elemental compositions of the samples are also included in Table I to facilitate the determination of the elemental deportment after smelting.

A low-sulphur carbon (LS Sascarb from Sasol) was used as reductant. Commercial MgO, Al_2O_3, SiO_2, and CaO chemicals supplied by Associated Chemical Enterprise were used in synthetic fluxes to simulate the dolomite and quartz flux used by EHSV. The compositions of the reductant and synthetic fluxes are included in Table II. High-purity MgO and Al_2O_3 crucibles were purchased from Kayla Africa (South Africa) and Tateho Ozark Technical Ceramics (Germany) respectively. The smelting test work was conducted in a 100 kW induction furance that was set up by LH Power. A schematic diagram of the induction furnace is shown in Figure 1.

The bulk chemical compositions of the raw materials and smelting test products were determined by CCD simultaneous inductively coupled plasma optical emission spectrometery (ICP-OES) (Varian Vista-PRO), and the total carbon and sulphur concentrations in the metals by LECO techniques (CS–230 and CS-744). The phase chemical compositions of the slag samples were analysed by scanning electron

Table II

Composition of the reductant and synthetic fluxes (mass %)

	C	MgO	Al_2O_3	SiO_2	CaO	Ash	Loss on ignition
Sascarb	98.7				0.33	0.97	
MgO		99.0					
Al_2O_3			98.0				
SiO_2				98.5			
CaO					96.0		3.50

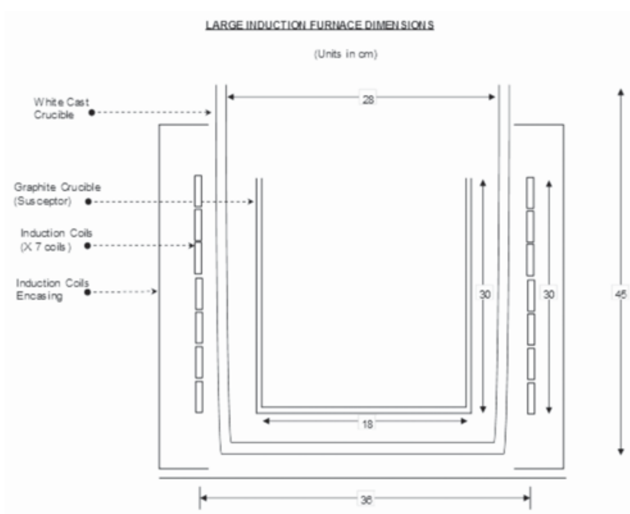


Figure 1—Schematic diagram of the 100 kW induction furnace

Table I

Bulk chemical compositions of the feed titanomagnetite materials (mass %)

	Fe_3O_4	MgO	Al_2O_3	SiO_2	CaO	TiO_2	V_2O_5	Cr_2O_3	MnO
Mintek test work	77.7	1.48	3.97	1.34	0.07	13.31	1.67	0.20	0.24
EHSV operation	76.5	1.60	4.80	2.00	0.10	12.7	1.65	0.40	0.30
	Fe	Mg	Al	Si	Ca	Ti	V	Cr	Mn
Mintek test work	55.3	0.88	2.07	0.62	0.05	7.88	0.92	0.14	0.19
EHSV operation	54.8	0.96	2.54	0.93	0.07	7.61	0.92	0.27	0.23

microscopy (SEM) (Zeiss MA15) equipped with energy dispersive spectrometry (EDS) (Bruker) and X-ray diffractometry (XRD) (Bruker D8 Advance).

Smelting recipe design

Laboratory-scale smelting tests were carried out to study the effect of wear in two crucible types (high-purity MgO and Al_2O_3) on the smelting characteristics of titanomagnetite. The principal variable in the test work was the reductant addition. Since iron is the major reduceable component in the titanomagneitite, the carbon reductant requirement was calculated based only on the stoichiometric reduction of magnetite, as shown in Equation [1]. The carbon additions in the tests were varied at 100%, 110%, and 120% of stoichiometric with the intention of studying the influence of carbon addition on the recovery of the valuable elements (Fe and V) to the alloy phase and preferential Ti deportment to the slag.

$$Fe_3O4_3 + 4C \rightarrow 3Fe + 4CO \qquad [1]$$

The fluxing strategy aimed to achieve and work with a slag similar in composition to that of the EHSV operation. At EHSV, fluxing of the titanomagneite is effected by the addition of quartz and dolomite, a natural mineral with a CaO:MgO ratio of about 16:14 – this assumes that dolomite would constitute 30% of the subsequent slag as predicted by Jochens *et al.*, (1969). The liquidus temperature of the EHSV titanomagnetite slag can be predicted using the phase diagram that was developed by Jochens., *et al.* (1969) and shown in Figure 2. The concentrations of SiO_2 and Al_2O_3 as well as the overall flux slag to control the dilution of TiO_2 were controlled in order to be able to reference Jochens' phase diagram with the compositional range of CaO 30–0%, MgO 0–30%, SiO_2 19.69%, Al_2O_3 13.12%, and TiO_2 37.19% (Jochens *et al.*, 1969). As shown in the phase diagram, the slag liquidus temperature for the smelting tests with CaO and MgO contents of 16% and 14% respectively was estimated to be about 1390°C. A summary of the smelting recipes is given in Table III.

Smelting test procedure

The test recipes, shown in Table III, were prepared by mixing the respective components and subsequently milling the mixtures in a ring mill to produce a homogeneous mixture. The recipes were weighed and densely packed in MgO and Al_2O_3 crucibles. The charged crucibles were placed in the reaction chamber of the 100 kW induction furnace. As shown in Figure 1, the furnace chamber is made up of a graphite susceptor. The chamber containing the test crucibles was closed by a graphite lid equipped with three drill-holes for (1) an alumina tube used to deliver argon gas into the furnace chamber to create an inert environment throughout the duration of each test, (2) an alumina off-gas duct to avoid pressurizing the reactor, and (3) an alumina sheath for encasing the B-thermocouple used to control the heating mechanism of the furnace and monitor the sample temperature.

The smelting tests were executed in two sets (for each crucible type) of three tests to complete a total of six tests. The furnace was heated gradually by incremental power input adjustments until a target temperature of 1600°C was reached. As in commercial operations, the target smelting temperature includes a superheat of about 200°C to the predicted slag liquidus temperature of about 1390°C – this action is typically conducted in order to ensure that the viscosity of the melt is low enough to allow for efficient slag and alloy separation. The furnace operates by inductive heat generation inside a graphite suscepting crucible, created by the water-cooled induction coils around the furnace. Each test run was allowed to remain at the test temperature for 1 hour, after which the crucibles were left inside the reactor to cool overnight to room temperature under argon atmosphere. The products were then weighed and separated into alloy and slag. The alloy samples were analysed by ICP-OES and LECO techniques. The slag was analysed by ICP-OES, SEM-EDS, and XRD.

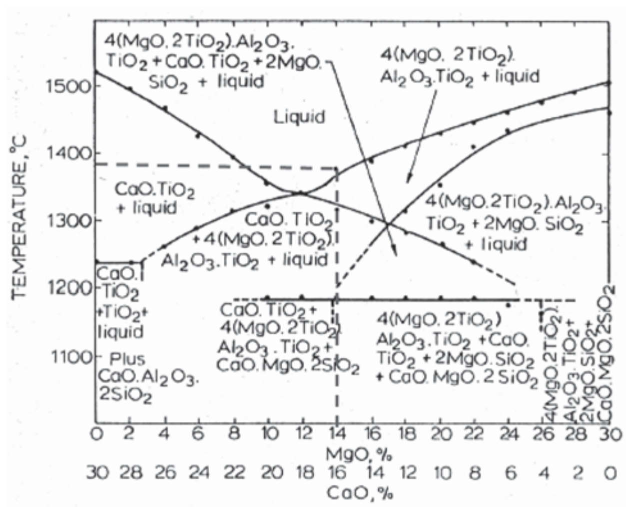


Figure 2—Phase equilibrium for the slag system: CaO 30–0%, MgO 0–30%, SiO_2 19.69%, Al_2O_3 13.12%, and TiO_2 37.19% showing the liquidus temperature of a slag with a CaO:MgO ratio of 16:14 (Jochins *et al.*, 1969)

Table III

Summary of smelting test recipes (mass %)

Test	Crucible	Stoichiometric C	Ore	CaO	MgO	Al_2O_3	SiO_2	C
1	Magnesia	100%	75.50	4.50	2.75	0.62	4.50	12.15
2		110%	74.67	4.42	2.70	0.58	4.44	13.19
3		120%	73.70	4.38	2.70	0.59	4.41	14.22
4	Alumina	100%	75.52	4.47	2.78	0.58	4.50	12.14
5		110%	74.60	4.43	2.72	0.61	4.46	13.20
6		120%	73.59	4.38	2.69	0.59	4.42	14.34

Results and discussion

Product masses

The typical appearance of the smelting products is shown in Figure 3. In general, good slag and metal separations were observed. The product alloy masses, which are calculated as the ratio of the mass of the collected alloy to the mass of the ore (without additives), expressed as a percentage, are plotted as a function of stoichiometric carbon addition in Figure 4. These results show that the mass % alloy produced was consistent at 100% and 110% stoichiometric carbon additions, but dropped significantly with 120% stoichiometric carbon addition. This was attributed to the presence of unreacted solid carbon in the molten system, which adversely affected the melt viscocity and subsequently the efficiency of slag and metal separation. Although the effects of stoichiometric carbon addition are similar, the alloy masses produced in the alumina crucibles are generally superior to those produced in magnesia crucibles.

Alloy characteristics

The chemical compositions of the alloys produced are given in Table IV. The chemical composition of the typical alloy produced by EHSV is also included for the purpose of comparison. At EHSV, the Ti and C concentrations in the alloy were used as indicators of the strength of the reducing conditions in the system. High Ti and C in the alloy indicated highly reducing conditions in the furnace, which called for the addition of a corrective material to lower the reducing conditions in the furnace (Steinberg, 2008). The results of the current test work show significant contamination of the alloys by Ti as the stoichiometric reductant addition was increased – at the respective carbon additions, Ti contamination was more severe in magnesia crucibles than in alumina crucibles. The carbon contents in alloys produced with 100% and 120% stoichiometric carbon addition in both crucible types deviate significantly from the EHSV specification of about 3.2%. This deviation may result in alloys with significantly different liquidus temperatures. The vanadium-bearing alloy is used mainly for producing vanadium and steel products, thus the characteristics of the alloy should allow for recovery of the valuable products in downstream processing. The Fe and V grades of the alloys produced in alumina crucibles are generally high compared to those produced in magnesia crucibles. It was expected that the iron and vanadium grades would improve with increased carbon addition. However, the Fe and V contents were diluted by undesirable elements (Ti, Si) as conditions became more reducing in both crucible types. A high deportment of Ti to the alloy is undesirable as this would require further processing of the alloy to remove the titanium prior to the recovery of the valuable products lest their quality is compromised.

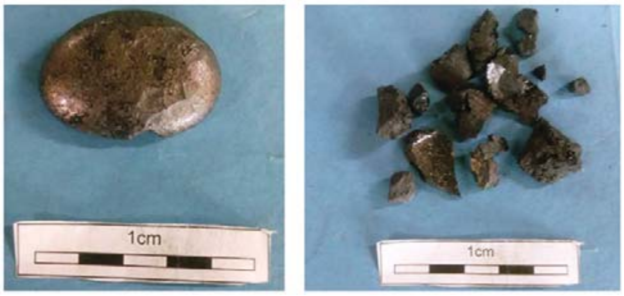


Figure 3—Typical appearance of the slag and metal produced from the smelting tests

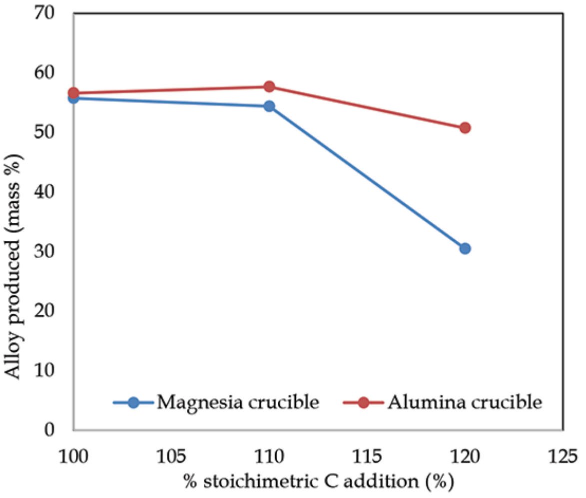


Figure 4—The mass % alloy produced plotted as a function of stoichiometric carbon addition

It is typical in laboratory-scale investigations to evaluate or investigate the relationship between test parameters and elemental recoveries and/or alloy grades. Small-scale test work is, however, subject to large errors due to uncertainties in the analyses and masses of the products, as small quantities are collected, and a small error can easily skew the mass balance and recovery calculations.

Table IV

Bulk chemical compositions of the alloys (mass %)

Test	Al	Ca	Cr	Fe	Mg	Mn	Ni	Si	Ti	V	C	Total
1	0.22	0.14	0.44	95.5	0.15	0.18	0.11	0.48	0.17	1.24	2.27	100.96
2	0.24	0.17	0.39	95.0	0.19	0.24	0.11	0.45	0.81	1.46	3.79	102.90
3	0.22	0.19	0.39	94.6	0.21	0.24	0.10	0.32	1.23	1.37	4.67	103.57
4	<0.005	<0.005	0.42	96.8	<0.005	0.10	0.12	0.19	0.04	1.34	2.48	101.51
5	<0.005	0.008	0.46	96.2	<0.005	0.14	0.11	0.35	0.15	1.66	3.28	102.36
6	<0.005	0.332	0.41	95.8	<0.005	0.21	0.10	0.69	1.46	1.63	4.36	104.99
EHSV	-	-	0.34	94.5	-	-	-	0.20	0.20	1.29	3.20	99.73

In the current test work, the elemental Fe and V recoveries were calculated according to Equation [2]. Figure 5 presents the elemental recoveries of the critical elements Fe and V to the alloy phases in the magnesia and alumina crucibles. In the EHSV operation, the Fe and V recoveries to alloy were estimated, using the data in the open literature, to be about 95% and 77% respectively (Steinberg, 2008). The recovery results in the current work showed that the best smelting conditions were obtained in alumina crucibles, with the highest Fe and V recoveries (recoveries higher than 100% were attributed to measurement uncertainty) obtained at 110% stoichiometric carbon addition. However, the Fe and V recoveries with 100% stoichiometric carbon addition in both crucible types were fairly close to the typical recoveries achieved at EHSV. Although the recoveries were better in the alumina crucible tests, the Fe and V recoveries (with both 100% and 110% stoichiometric reductant additions) in magnesia crucibles are still comparable to the EHSV recoveries. In any case, commercial titanomagnetite smelting is conducted in furnaces lined with magnesia refractories.

This observation suggests that both the magnesia and alumina crucibles are suitable for studying titanomagnetite smelting characteristics for Fe and V production, It should be noted that this observation is applicable to the target smelting recipe, and thus the subsequent slag chemistry.

% Recovery of element to alloy=
(% Mass of element in alloy × mass alloy)/ [2]
(% Mass of element in feed ×mass of feed)×100

Slag characteristics

The smelting tests targeted a slag with a melting point as identified in the phase diagram in Figure 2. Table V shows the typical chemical compositions of the EHSV slag (Steinberg *et al.*, 2011) and the slags produced in the current smelting study. The main slag components were Al_2O_3, MgO, CaO, SiO_2, and TiO_2. In the magnesia crucibles (Test 1 to Test 3) the target slag chemistry of CaO:MgO ratio of 16:14 was not attained due to the wear of magnesia crucible, which resulted in the dilution of other slag components by the increased MgO content in the slag. Crucible wear also resulted in an increase in the basicity of the slags. However, at 120% stoichiometric reductant addition (Test 3), the concentrations of the slag components, including CaO and MgO, were severely diluted by high iron in the slag. The high iron in the Test 3 slag is attributed to the possible increase in the viscocity of the melt due to the presence of unreacted solids, resulting in the entrainment of Fe metal in the slag. The extent of metal entrainment in the slag was, however, not investigated. As shown by the generally good recoveries of Fe and V in the alloys produced with 100% and 110% stoichiometric reductant addition, the increased basicity of the slag did not appear to adversely affect the Fe and V recoveries in the reviewed slag composition.

In the alumina crucible tests (Test 4 to Test 6), the crucible wear was still severe and resulted in the dilution of slag components. However, as the current definition of basicity (explained in Table V) does not include Al_2O_3 as a component, the alumina crucible wear did not affect the basicity. The basicity with alumina crucibles was consistent and comparable to the targeted basicity of about 1.5 (CaO 16%, MgO 14%, and SiO_2 19.69%). The slag from Test 6, which was conducted with 120% stoichiometric reductant addition, contained relatively high iron due to the increase in the viscocity of the melt, which was due to the presence of high unreacted carbon in the melt, which resulted in the entrainment of Fe metal in the slag.

In general, the residual iron and vanadium with specific reductant additions was relatively high in magnesia crucible tests compared to alumina crucible tests. The dilution of the slag components in alumina crucible tests was slightly less prominent compared to that in MgO crucibles. In particular, the TiO_2 slag concentrations in magnesia crucible tests were significantly lower than in alumina crucibles, due to the dilution of the TiO_2 by the relatively high Fe alloy entrainment in the magnesia crucible slags.

It is evident from the results that dissolution of the MgO and Al_2O_3 from the respective crucibles had occurred, resulting in higher concentrations of MgO and Al_2O_3 in the slags than anticipated.

The phase chemical compositions of the slags obtained from the best conditions (high recoveries) in both crucibles were subjected to SEM-EDS analysis for comparison with EHSV slag. Figures 6–8 show the backscattered electron images and EDS and XRD results. As reported by Goso *et al.* (2015), the EHSV slag is composed primarily of pseudobrookite ($MgTi_2O_5$-Al_2TiO_5) or spinel [(Mg)(Al,Ti)O_4] phases; it is not clear which of the two phases forms first. The perovskite in this slag crystallized as a ternary phase.

Figure 7 shows the microstructure of the best slag produced in magnesia crucibles (110% stoichiometric carbon addition). The slag is composed mainly of perovskite and forsterite, with monticellite forming as a finer-grained ternary phase. A significant portion of the slag that occurs in the Mg-Al-Ti-O system was not confirmed by XRD – the phase could be either pseudobrookite or spinel. Although this slag and the EHSV plant slag were produced using basic refractories, they have different phase compositions, but the reason for this is not well understood. It is unlikely that the difference in phase composition resulted from different cooling regimes, as neither of the slags was subjected to quenching.

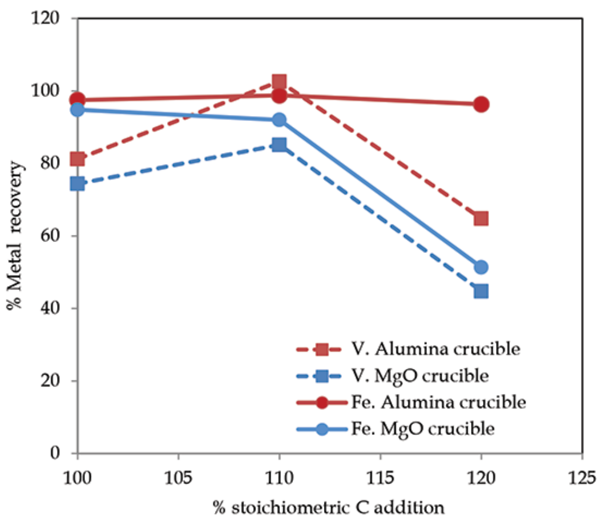


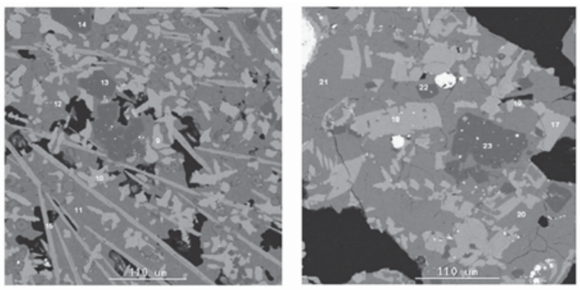
Figure 5—Relationship between stoichiometric carbon addition and recoveries of Fe and V to alloy

Table V

Bulk chemical composition of the slags (mass %)

Test	Al_2O_3	CaO	Cr_2O_3	FeO	MgO	MnO	SiO_2	TiO_2	V_2O_5	Total	#Basicity
1	10.30	13.13	0.16	10.98	20.50	0.2.	15.85	24.80	0.88	+96.82	2.12
2	10.70	14.70	0.08	5.03	20.60	0.19	17.60	25.70	0.37	+94.96	2.01
3	6.52	8.04	0.31	55.97	13.70	0.24	9.50	16.90	1.49	*112.66	2.28
4	22.00	16.03	0.10	1.01	11.05	0.41	18.45	30.75	0.39	100.19	1.47
5	21.70	16.66	0.08	1.21	11.90	0.36	18.30	31.00	0.27	101.48	1.56
6	19.30	16.24	0.09	10.97	11.90	0.25	17.00	29.20	0.43	*105.38	1.66
EHSV	18.00	14.10	0.20	1.00	14.10	0.40	16.20	35.60	0.90	100.50	1.74

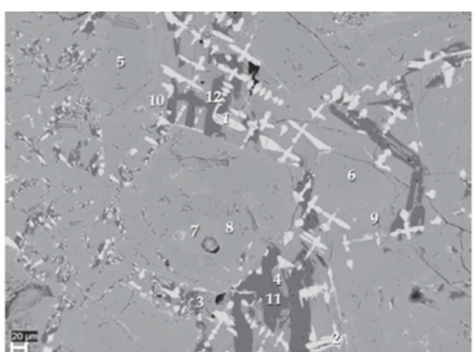
Basicity = (%CaO+%MgO)/%SiO_2
* High totals due to Fe entrainment in the slag
+ Deviation from 100% attributed to measurement uncertainty



Point	MgO	Al_2O_3	SiO_2	CaO	TiO_2	V_2O_5	FeO	Phases
9,10	0.1	0.2	0.1	39.6	55.8	0.1	0	Perovskite
11	18	11.3	30.5	18.5	15.3	0.3	0.3	*
12	13.9	8.2	38.7	22.1	12.6	0.1	0.2	*
13, 14	28	60.6	0.1	0	11.3	1.7	0.3	Spinel
15, 16	8.9	2.1	0.3	0.7	82.3	1.1	0.2	Pseudobrookite
17, 18	5.8	2.7	0.1	0.2	90.3	0.2	0.1	Pseudobrookite
19	0.2	0.3	0.1	39.5	56.1	0	0.1	Perovskite
20, 21	10.5	9.7	34.8	23.4	16.7	0.1	0.2	*
22, 23	28.1	65.2	0.3	0.1	7.9	0.7	0.4	Spinel

x Phase not detected by XRD

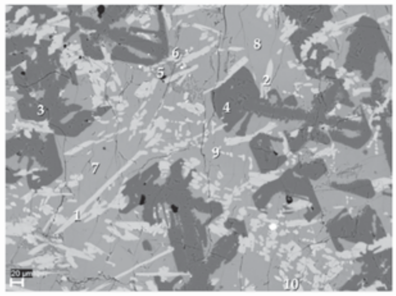
Figure 6—Microstructure, chemical composition (mass %, by EDS), and phase composition (by XRD) of the EHSV slag



Point	MgO	Al_2O_3	SiO_2	CaO	TiO_2	Phase
1, 2		0.45		39.46	60.09	Perovskite
3, 4	27.97		33.8	38.22		Monticellite
5	35.58	18.71			45.71	MgAlTiO
6	33.44	15.49			51.07	MgAlTiO
7	22.34	17.35	16.9	15.42	27.99	*
8	32.11	17.72			50.17	MgAlTiO
9	10.83	11.61	34.1	26.92	16.58	*
10	11.14	15.55	32	25.96	15.35	*
11, 12	58.77		37.4	2.622	1.18	Forsterite

x Phase not detected by XRD

Figure 7—Backscattered electron image, chemical composition (mass %, by EDS), and phase composition (by XRD) of the slag produced with 110% stoichiometric C addition in a magnesia crucible



Point	MgO	Al_2O_3	SiO_2	CaO	TiO_2	MnO	FeO	Phase
1, 2	6.62	3.86		0.84	88.14		0.54	Pseudobrookite
3, 4	28.50	65.60			5.90			Spinel
5, 6	2.03	27.18	47.61	11.06	2.93	2.63		x
7, 8	7.90	19.29	26.28	23.71	22.81			x
9, 10		0.84	1.12	38.99	59.05			Perovskite

x Phase not detected by XRD

Figure 8—Backscattered electron image, chemical composition (mass %, by EDS), and phase composition (by XRD) data of the slag produced with 110% stoichiometric C addition in an alumina crucible

The slag that resulted in highest Fe and V recoveries in the alumina crucible tests (Figure 8) consisted of pseudobrookite as the primary phase, spinel as the secondary phase, and perovskite as the ternary phase. This phase composition is similar to that of the EHSV slag, though in this case inclusions of pseudobrookite were observed in the spinel, suggesting that the pseudobrookite crystallized first.

As detailed above, the difference in the phase compositions of the slags produced using magnesia and alumina crucibles did not adversely affect the Fe and V recoveries, as in both cases the recoveries were comparable to those in the commercial EHSV process. Based on the phase chemical compositions, the titanomagnetite slag produced in alumina crucibles is similar to the EHSV slag. Hence, for the studied slag composition the alumina crucible appears to be the best type to use in laboratory-scale test work.

Conclusion

Laboratory-scale carbothermic reduction smelting tests on titanomagnetite have demonstrated that chemical attack of the magnesia and alumina crucibles by the titania-bearing slag is inevitable. The aggressiveness of the titania-bearing slag led to dissolution of the respective crucible components into the slag phase. The crucible wear was more pronounced with magnesia crucibles than with alumina crucibles. The Fe and V recoveries to the alloy were higher in alumina crucibles than those in magnesia crucibles, but the recoveries in both cases were comparable to the EHSV data. The phase chemical compositions of the best slags in terms of Fe and V recoveries were significantly different for each crucible type. The slag produced using an alumina crucible had a similar phase chemical composition to that of the commercial EHSV titanomagnetite slag. The results of the test work have shown that both the magnesia and alumina crucibles can be used to conduct laboratory-scale test work with slags in the reviewed chemical composition range. However, since alumina crucibles are relatively easily obtainable, it is preferable to use alumina crucibles.

Further work is recommended to investigate the effect of magnesia and alumina crucibles on the smelting characteristics of titanomagnetite at various CaO to MgO ratios in the compositional range covered in the available phase diagram, and to study the changes in liquid phase formation as a function of MgO and Al_2O_3 addition.

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