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LIQUIDUS TEMPERATURES OF COMMERCIAL ESR SLABS.(U)
MAY 81 R C ANDREW, G M WESTON

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MELBOURNE, VICTORIA

REPORT

MRL-R-813

**LIQUIDUS TEMPERATURES OF COMMERCIAL
ESR SLAGS**

Richard C. Andrew & George M. Weston

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REPORT

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ABSTRACT

Melting point data for a number of slag systems of particular interest in the electroslag refining of steel have been determined under laboratory conditions. In several instances, the melting point temperatures determined differed considerably from those quoted commercially. Such discrepancies were attributed to the presence of impurities in the raw feed materials, causing their composition to differ considerably from nominal.

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Melting point data for a number of slag systems of particular interest in the electroslag refining of steel have been determined under laboratory conditions. In several instances, the melting point temperatures determined differed considerably from those quoted commercially. Such discrepancies were attributed to the presence of impurities in the raw feed materials, causing their composition to differ considerably from nominal.

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LIQUIDUS TEMPERATURES OF COMMERCIAL

ESR SLAGS

1. INTRODUCTION

The main component of the slags used in the electroslag-refining (ESR) process is calcium fluoride. To obtain the desired slag properties for each melting operation one or more compounds such as lime, magnesium oxide, alumina, silica and rare earth oxides may be added to the fluoride. Information on the physical and chemical properties of an immense range of slag compositions is therefore necessary to optimise slag selection.

One property which is frequently required is the slag liquidus temperature because of its influence on ingot surface finish [1], prefusion characteristics within the slag furnace, and process economics. [2] Accordingly, considerable effort has been devoted to determining this property for a wide range of slag compositions but, because of the high temperatures involved and the highly reactive nature of the fluoride-based slags, many gaps still remain. For example, most effort has concentrated upon the more commonly used binary and ternary slags and little information is available for more complex slags. Furthermore, the liquidus temperatures which have been determined often relate to ideal conditions, involving high-purity components, and as a result the composition quoted is frequently the nominal one rather than the actual one obtained by analysis after fusion and re-solidification. [2,3,5]

In practice, the actual slag compositions can vary significantly from the nominal one selected for use and can change throughout the remelting operation for several reasons. Firstly, whilst the slag components are of excellent quality, because of the large quantities involved they may contain appreciable amounts of other compounds. Adjustments may be made to the composition of the slag charge to take these variations into account, but this is difficult with the result that the nominal composition charged to the slag furnace is only approximate. Secondly, reactions may occur in the slag furnace during pre-fusion which generally lead to an increase in the lime content of the slag bath and a decrease in its fluoride content. These reactions have been discussed in detail by Mills [3], and more details are contained in Appendix I. Again, it is possible to compensate for the changes brought about by these reactions but not always accurately. Thirdly, the

addition of deoxidants to the slag bath throughout remelting influences its composition. Two of the most widely used deoxidants are aluminium and calcium silicide and their continuous addition, either alone or in combination, causes a concomitant gradual rise in the alumina and silica levels of the slag bath.

The Commonwealth Steel Company (Comsteel) realized at an early stage in the working up of its ESR facility that, to gain more precise process control, the liquidus temperatures of slag compositions which corresponded closely with those of the actual slag bath were needed to supplement the data available for nominal slag compositions. To obtain this information work was undertaken at MRL whilst one of the authors (RCA) was attached to Comsteel. The range of slags examined included those being considered for future use by Comsteel as well as those currently in use.

2. EXPERIMENTAL PROCEDURE

Slag samples of the desired nominal compositions were prepared from slag components purchased by Comsteel for their commercial ESR operations. The compositions of these raw components, with the exception of bastnasite, are given in Table I. It is obvious from this Table that most of the components contain sufficient of other major compounds in controlled amounts to make preparation of precise nominal compositions impossible. Bastnasite is a naturally occurring fluorocarbonate with the major elements being cerium and lanthanum. The total rare earth oxide content usually ranges from 27-65 wt%; however a more concentrated form higher in cerium oxide is usually supplied for slag purposes. Analysis of bastnasite in Comsteel's chemical laboratory has proven difficult. The analysis certificate provided by the supplier showed it to contain about 85% by weight of cerium oxide.

The range of nominal compositions examined is shown in Table II. The procedure adopted was to heat 50 g samples of these slags at 850°C for 24 h in silica crucibles open to the atmosphere to remove moisture. These samples were then transferred hot to a graphite container in the apparatus shown in Fig. 1(a). This container was lined with molybdenum sheet to minimise the formation of calcium carbide by reaction between the container and the slag components. The container was designed with a removable base to facilitate extraction of the fused sample for analysis after completion of the liquidus temperature measurement (Fig. 1(b)).

The slag samples were then fused under conditions which allowed reactions similar to those which occur in the slag pre-fusion furnace at Comsteel to proceed. Argon was bled into the apparatus to reduce the violence of the reactions and the accompanying fuming. Each sample was kept molten for several minutes to ensure thorough mixing of the components. Power to the high frequency coils was then switched off and a Pt/Pt+13% Rh thermocouple tip, encased in a graphite sheath was immersed in the fused slag. The liquidus temperature was determined by thermal analysis; the change in the thermocouple output being monitored with an Esterline-Angus Model E11015 recorder. The recorder trace on the first cooling cycle was

frequently irregular which made accurate measurement of the liquidus temperature difficult. To overcome this the sample was re-fused and then allowed to cool. Little fuming was observed on the second fusion and the associated cooling curve normally displayed a well defined change in slope at the liquidus temperature.

Each sample was fused several times and the liquidus temperature recorded each time to ensure reproducibility of results. As a further check at least two samples of each composition were fused, and these samples were frequently combined subsequently to yield a third set of results. Liquidus temperatures were also measured for the Cu-Cu₂O eutectic to establish the accuracy of the experimental procedure. The composition of each slag sample was determined at Comsteel using a Philips model PW1400 x-ray spectrometer.

3. RESULTS AND DISCUSSION

Details of the experimental results together with data provided by other sources are summarised in Table II. In the slag analysis section of this Table the desired (nominal) operating composition for each slag system examined is shown first. To compensate for some of the reactions which may occur during slag pre-fusion (see Appendix I) and so obtain an actual slag bath composition closer to nominal, Comsteel adjust the nominal composition of the slag charged to the slag furnace. This adjusted nominal composition is shown in parenthesis after the desired values for those slags which have been used by Comsteel. Slag bath compositions representative of actual operating compositions are given next. The slag furnace (SF) samples are taken immediately before the furnace contents are poured into the ESR crucible and thus their analyses are effectively those of the slag bath at the start of remelting. The slag cap (SC) samples are taken at the end of the remelting operation.

The compositions of the slag samples produced at MRL to match either the representative SF or SC samples are presented next. Although exact matching of compositions was not possible, mainly because of the complexity of the slag systems examined, they are generally in good agreement except for the bastnasite-bearing slags. The liquidus temperatures determined at MRL and those contained in information supplied to Comsteel are shown against the respective slag compositions for some nominal compositions.

It is evident from Table II that, for three of the slag systems examined, the liquidus temperatures measured at MRL are substantially higher than those nominal compositions provided to Comsteel by other sources. In the remaining systems, the MRL data agree closely with those supplied by the Consarc Corporation.* Because of a complete lack of information on the experimental conditions used to obtain the liquidus temperatures reported by

* Makers of the commercial ESR plant installed at Comsteel.

Consarc Corporation and Barraclough [4], it is difficult to explain the disagreement in results. Several factors, including differences in experimental procedures, quality of slag components and compositional variations may be responsible for this disagreement. Whilst the compositions of both the Comsteel and the corresponding MRL samples were generally very similar, they varied significantly in several cases (first three compositions Table II) from the nominal one. Of particular importance, this variation was most pronounced for the same three slag systems where disagreement in the liquidus temperature values exist. It is most likely, therefore, that the discrepancy in liquidus temperatures is due to differences in slag compositions.

Production experience with both the $[70\% \text{CaF}_2 + 30\% \text{Al}_2\text{O}_3]$ slag and the $[65\% \text{CaF}_2 + 15\% \text{CaO} + 10\% \text{Al}_2\text{O}_3 + 10\% \text{SiO}_2]$ slag at Comsteel suggests that their liquidus temperatures are about 1500 and 1400°C respectively, thus supporting the MRL findings. Other workers [5,6] have reported a liquidus temperature of 1500-1550°C for $[70\% \text{CaF}_2 + 30\% \text{Al}_2\text{O}_3]$, which is also much higher than that determined for this system by Barraclough [4]. Although considerable disagreement exists concerning the structure of the $\text{CaF}_2 - \text{CaO} - \text{Al}_2\text{O}_3$ phase diagram [7,8], a liquidus temperature of about 1400-1450°C for $[60\% \text{CaF}_2 + 20\% \text{CaO} + 20\% \text{Al}_2\text{O}_3]$ appears more likely than the 1240-1260°C reported by Barraclough.

It should also be noted from Table II that short arrests were observed at 1430-1460°C for the $[65\% \text{CaF}_2 + 15\% \text{CaO} + 10\% \text{Al}_2\text{O}_3 + 10\% \text{SiO}_2]$ slag and at 1370°C for the $[46\% \text{CaF}_2 + 15\% \text{CaO} + 7\% \text{MgO} + 20\% \text{Al}_2\text{O}_3 + 12\% \text{SiO}_2]$ slag, indicating in each case that a small amount of primary phase, which was not identified, formed.

A liquidus temperature range is reported for most of the slag systems examined at MRL rather than a single temperature (Table II). Several readings at least were taken for each slag system and this range indicates the spread of liquidus temperature values recorded in the table. The maximum value in each range is believed to be the most reliable one because it is very likely that the lower readings resulted from an inability to locate the thermocouple tip precisely in the centre of the slag sample on each occasion. The closeness of the value obtained at MRL for the $\text{Cu-Cu}_2\text{O}$ eutectic temperature to the standard value [9] tends to confirm the accuracy of the MRL procedure (Table II).

The sum of the analytical results reported in Table II for the individual slag samples generally was less than 100%, considerably so in some instances. One reason for this was the omission of elements such as iron, manganese, carbon and sulphur from the analysis. Furthermore, the analysis procedures are subject to small errors. However, these omissions and errors are not expected to cause any significant problems when comparison is made between the MRL and Comsteel slag sample compositions because they should be of a similar size for samples from the two sources. The analysis results reported for the bastnasite-bearing slags are of greater concern because of the difficulties currently being experienced at Comsteel in obtaining an accurate, reliable analysis procedure for bastnasite. The compositions presented in Table II for the bastnasite-bearing slags must therefore be

treated with considerable caution.

4. CONCLUSIONS

The liquidus temperature of a range of slags used in the electroslag-refining process have been determined by thermal analysis. The compositions of these slags are similar to those either in use or being considered for use at the commercial ESR facility operated by the Commonwealth Steel Company at their Waratah works.

The present work has shown that ESR slags made to a nominal composition using raw components from different sources can have large variations in composition, and therefore melting point, after prefusion. Such variations appear to arise mainly from purity levels in the initial basic slag components. Propriety melting point data may therefore be considerably in error and ESR operators should determine slag melting points using their locally supplied slag components.

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APPENDIX I

MAJOR CHEMICAL REACTIONS WHICH BRING ABOUT CHANGES IN SLAG COMPOSITION DURING PREFUSION

Reactions between either CaF_2 and crucible materials or CaF_2 and other slag components may produce significant changes in the composition of the slag bath. These reactions have been discussed in detail by Mills [3] and only the more important ones are described here. The first reaction of concern is that between CaF_2 and graphite to produce CaC_2 . Normal practice at Comsteel is to prefuse the slag charge in a graphite-lined arc furnace. Formation of considerable amounts of CaC_2 has been observed to occur when the molten slag charge was held at temperature for lengthy periods ("stewing" of the slag charge) or was heated to excessively high temperatures. On these occasions the graphite electrodes in the arc furnace were rapidly consumed, indicating that they were the main reaction site for carbide formation. Excessive heating of the slag charge should be avoided because the presence of CaC_2 in the molten slag poured into the ESR crucible may lead to carbon pick-up in the bottom of the remelted ingot.

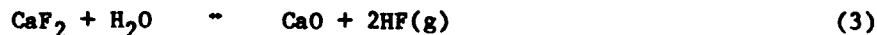
Calcium fluoride will react with alumina to produce a gaseous fluoride, and this leads to an increase in the lime content of the slag bath as follows:



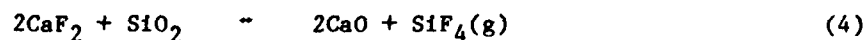
The CaO will form a calcium aluminate by reaction (2) or, if a calcium aluminate is already present, it will contribute to the formation of a calcium aluminate richer in CaO .



Another reaction which will increase the lime content of the slag bath and reduce its fluoride level is that between CaF_2 and water (reaction 3). The water may be present as vapour in the crucible atmosphere or as absorbed water in the slag. This reaction is therefore of particular significance with high lime slags.



Silica also reacts with CaF_2 at high temperatures to produce a gaseous fluoride and lime (reaction 4). The latter reacts with silica to form a calcium silicate by reaction 5 or it converts an existing silicate to a silicate with a higher lime content.



The foregoing description shows that CaF_2 reacts with the other main components of ESR slags to increase the lime content of the slag bath and decrease its fluoride level. In commercial ESR practice, the extent of these changes in each slag system can be determined by monitoring slag compositions over a number of melts. The nominal slag composition charged to the slag furnace can then be adjusted to compensate for them.

TABLE I
COMPOSITION IN WEIGHT PER CENT OF THE INDIVIDUAL SLAG COMPONENTS

Major Component	Composition, wt %												
	CaF ₂	CaO	MgO	Al ₂ O ₃	SiO ₂	P	S	Fe	Cu	Zn	Sn	Pb	Na ₂ O
CaF ₂	94.0	1.8(CaCO ₃)			1.8	0.043	<0.01		<0.005	0.008	<0.01	<0.005	
CaO		96.1	0.3(Mg)		0.40	<0.01	<0.01	0.07	<0.005	<0.001	<0.01	<0.005	
MgO		0.71(Ca)	95.0		2.5	0.01	<0.01	0.06	<0.005	0.005	<0.01	<0.005	
SiO ₂		0.11	<0.01	0.4	97.8			0.08					0.35
Al ₂ O ₃				99% min.	0.016			0.023 (Fe ₂ O ₃)					0.5

Bastnasite : Composition not available - contains approximately 85% cerium oxide.

TABLE II
LIQUIDUS TEMPERATURES OF COMMERCIAL ESR SLAGS

Sample Type †	Slag Composition, Wt%							Liquidus Temperature °C		
	CaF ₂	CaO	MgO	Al ₂ O ₃	SiO ₂	Bast.*	Total	MRL	Consarc ^{††}	Barracclough [©]
Nom.	70(70)	-	-	30(30)	-	-	100			1320-1340
S.F.	63.3	5.9	0.44	28.2	0.9	-	98.7			
MRL	61.3	6.5	<0.1	27.5	0.6	-	95.9	1450-1480		
Nom.	60	20	-	20	-	-	100			1240-1260
MRL	54.4	18.6	<0.1	23.0	0.7	-	96.7	1390-1405		
Nom.	65(70)	15(10)	-	10	10	-	100		1265	
S.F.	54.6	22.4	0.8	10.0	9.4	-	97.2			
MRL	57.8	18.5	0.1	10.4	10.5	-	97.3	1365**		
S.F.	63.7	18.2	0.4	11.1	7.0	-	100.4			
MRL	61.7	13.8	0.1	13.3	9.1	-	98.0	1355		
S.F.	55.8	13.4	3.0	18.6	6.0	-	96.8			
MRL	55.2	14.9	0.1	14.9	12.7	-	97.8	1355-1370		
S.C.	53.3	19.5	0.3	14.0	9.7	-	96.8			
MRL	54.8	16.4	0.1	15.5	13.4	-	100.2	1355-1370		

TABLE II
(Continued)

Sample Type	Slag Composition, Wt%							Liquoidus Temperature °C		
	CaF ₂	CaO	MgO	Al ₂ O ₃	SiO ₂	Bast.*	Total	MRL	Consarc ++	Barracclough ^e
Non.	33(41)	33(25)	-	33	-	-	100		1440	
S.F.	31.7	33.2	0.38	29.9	0.15	-	95.3			
MRL	34.3	30.2	0.1	32.8	1.5	-	98.9	1430-1450		
Non.	49(54)	17(12)	7	17	10	-	100		1280-1290	
S.F.	47.3	17.1	7.2	15.9	10.3	-	97.8			
S.C.	42.5	16.4	5.4	21.2	10.8	-	96.3			
MRL	43.3	16.6	7.2	19.1	10.4	-	96.6	1280		
MRL	42.2	17.1	7.2	20.3	10.9	-	97.7	1285-1300		
Non.	55(60)	15(10)	7	16	7	-	100			
S.F.	55.5	12.0	6.8	15.3	4.66	-	94.3			
MRL	53.5	14.3	6.6	16.2	8.2	-	98.8	1290-1320		
S.C.	49.7	14.9	5.2	16.6	9.3	-	95.7			
MRL	51.3	16.5	7.3	16.8	8.1	-	100.0	1290-1320		

TABLE II
(Continued)

Sample Type +	Slag Composition, Wt%							Liquidus Temperature °C		
	CaF ₂	CaO	MgO	Al ₂ O ₃	SiO ₂	Bast.*	Total	MRL	Consarc ⁺⁺	Barracclough ^e
Nom.	46(51)	15(10)	7	20	12	-	100	1280		
S.F.	43.7	16.1	6.5	18.0	4.8	-	89.1			
MRL	See results for 49F/17/7/17/10									
S.C.	41.2	12.4	6.3	19.0	12.8	-	91.7			
MRL	40.8	15.8	7.1	20.6	12.8	-	97.1			
MRL	41.1	16.4	6.6	23.5	13.0	-	100.6	1290-1310**		
Nom.	54	10	12	19	5	-	100	1295-1305		
MRL	50.4	11.5	12.5	19.2	6.5	-	100.1			
Nom.	55	-	-	25	-	20	100	1460-1490		
MRL	49	1.1	<0.1	19.8	0.8	28	98.7			
Nom.	55(60)	25(20)	-	-	-	20	100	1310		
S.F.	52.5	11.8	0.1	0.7	1.0	31.7	97.8			
MRL	48.4	17.8	0.1	7.8	1.0	16.1	91.2			

TABLE II
(Continued)

Sample Type	Slag Composition, Wt%							Liquidus Temperature °C		
	CaF ₂	CaO	MgO	Al ₂ O ₃	SiO ₂	Bast.*	Total	MRL	Consarc ⁺⁺	Barracough ^e
Cu-Cu ₂ O eutectic								1063(1065 ^{ee})		

* Bastnasite

++ Supplied by Consarc Corporation

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ee E.M. Levin et al, "Phase Diagrams for Ceramicists", The American Ceramic Society, 1964, p.38

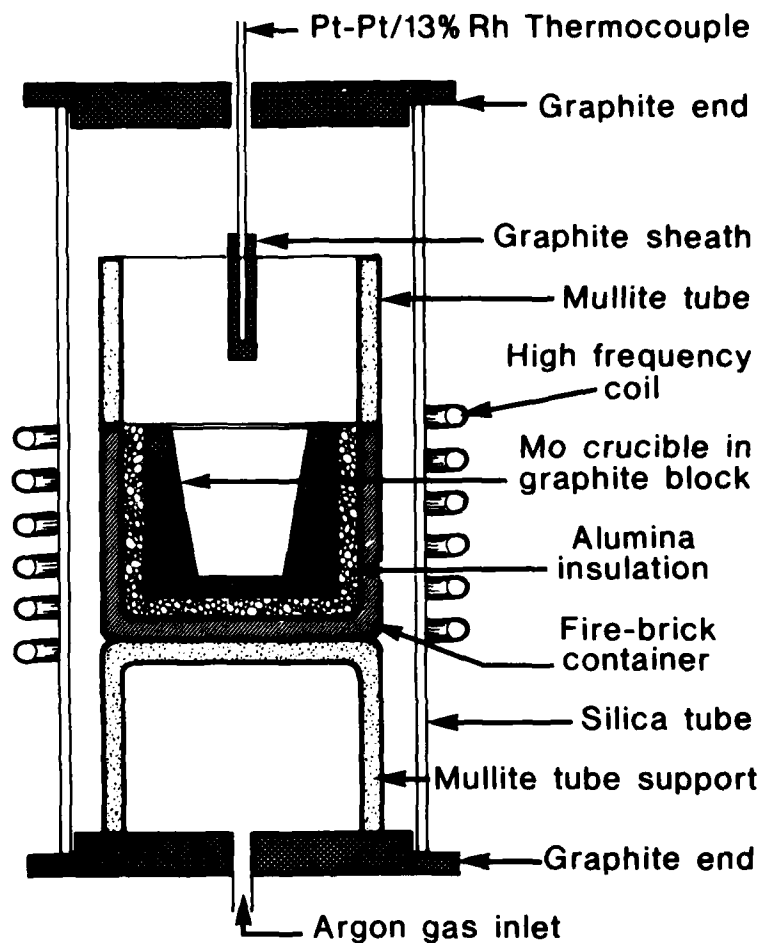
** Very short arrests detected at 1430-1460°C for 65P/15/0/10/10 slag and 1370°C for 46P/15/7/20/12 slag

+ Nom. - nominal composition. Actual composition charged to the slag furnace is shown in parenthesis

S.P. - slag bath sample at the start of the remelting operation

S.C. - slag bath sample at the end of the remelting operation

MRL - laboratory sample

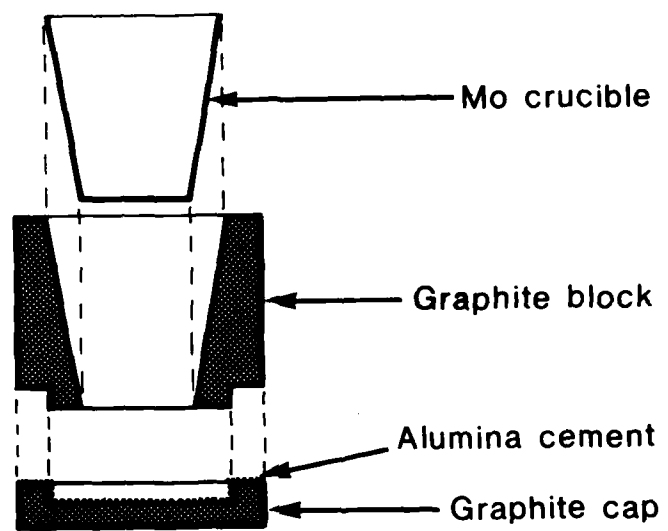


(a)

FIGURE 1 Schematic representation of the apparatus used to determine slag liquidus temperatures.

(a) experimental set-up

(b) design of sample container to facilitate sample removal



(b)

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