

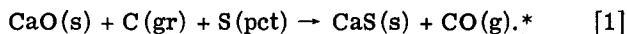
Desulfurizing Iron-Carbon Melts With Lime Under Reduced Pressures

D. C. BOYD, W. C. PHELPS, JR., AND M. T. HEPWORTH

Experiments were carried out to compare the rates of formation of CaS on CaO crystals which were either immersed in a sulfur-bearing, carbon-saturated iron melt or held in the effluent gases just above the melt. Temperatures ranged from 1375°C to 1600°C, and times ranged from one to twelve hours. The CaS layers were measured both metallographically and with an electron microprobe. Data on the time and temperature dependence of the layer thickness are presented. These layers were about 15 microns thick after 6 h at 1460°C, independent of whether or not the crystal was immersed in molten iron. The role of lime in the desulfurization process is to act as a getter for removal of sulfur from the gas stream.

MUCH attention has been given recently to lime as a desulfurizing agent, especially in the cleaning of sour stack gases. The findings reported in this paper may have some bearing upon this problem even though the emphasis is directed exclusively to the desulfurizing of iron.

The desirability of using solid lime during or just after the blast furnace operation, when the carbon content of the iron is still quite high, and the consequent activity of oxygen is low, has been demonstrated by thermodynamic calculations. The reaction which can be used to describe the overall process is



*The nomenclature used in the chemical reactions throughout this paper indicates the standard states or the compounds as follows:

CS(g), CO(g): the gases at 1 atmosphere partial pressure, sulfur or oxygen dissolved in liquid iron with respect to a reference state.

$\underline{\text{S}}(\text{pct}), \underline{\text{O}}(\text{pct})$: the (hypothetical) 1 pct solution in pure, liquid iron.

$\underline{\text{C}}(\text{gr})$: carbon dissolved in liquid iron with respect to graphite as the standard state.

CaS(s), CaO(s): pure, solid calcium sulfide or calcium oxide.

All of the standard states are at the temperature in question.

Equilibrium calculations have shown that with carbon monoxide partial pressures as high as 1 atmosphere the equilibrium sulfur content of carbon-saturated iron can be reduced to as low as 10 parts per million by this reaction.^{1,2}

Iron, especially high-carbon or high-silicon iron, can be effectively desulfurized by reducing the gas pressure at the gas-metal interface and allowing sulfur to vaporize as one or more sulfur-bearing gas species. Many investigators^{3,4,5,6,7} have observed and reported this phenomenon. Zubrev *et al.*⁸ have recently examined the kinetics of desulfurization of steel by blowing powdered lime into the molten steel. They found the reaction kinetics to be controlled by the surface area of the lime powder which was exposed to the steel.

The objective of this work is to compare two proposed mechanisms for Reaction [1] which are illus-

trated in Figs. 1(a) and 1(b). In mechanism (a), lime acts only as a getter for the sulfur and does not enter into the actual desulfurization reaction. In mechanism (b), the rate-limiting step in the desulfurization is the sulfur-oxygen diffusion through the CaS with the subsequent deoxidation of the iron by carbon. Experiments were designed to measure and compare the rates of formation of CaS on CaO crystals which were either immersed in a carbon-saturated iron melt or held at temperature in the effluent gases above the melt.

EXPERIMENTAL PROCEDURE

Time and temperature studies were made on single crystals of fused lime, which were exposed to 700 g of sulfurized, carbon-saturated, liquid iron. A sche-

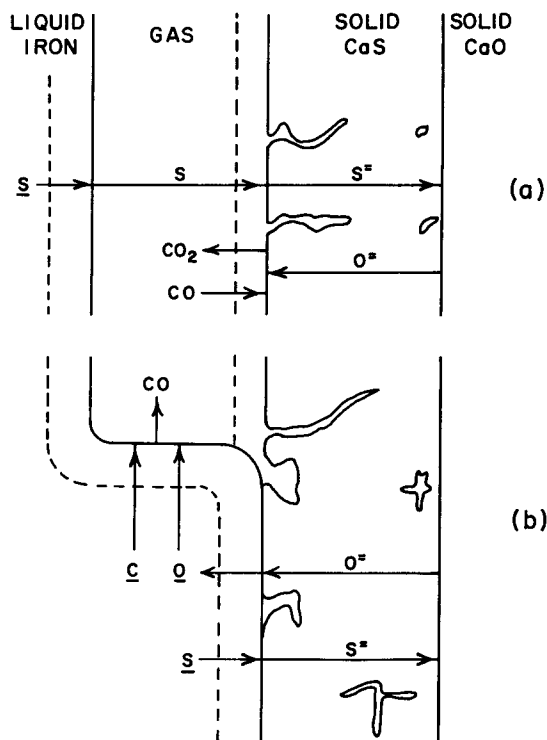


Fig. 1—Diagram of the possible sulfur and oxygen paths for desulfurization of carbon-saturated iron with lime reactions: (a) $\text{S} \rightarrow \text{S}(g)$, followed by $\text{S}(g) + \text{CaO}(s) + \text{CO}(g) \rightarrow \text{CaS}(s) + \text{CO}_2(g)$; (b) $\text{CaO}(s) + \underline{\text{S}} \rightarrow \text{CaS}(s) + \underline{\text{O}}$, followed by $\underline{\text{C}} + \underline{\text{O}} \rightarrow \text{CO}(g)$.

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matic diagram of the furnace used for most of the experimental runs is pictured in Fig. 2. The system consisted of two stainless steel vacuum tanks with an induction-heated crucible made from AUC graphite. The lime was held in position by a net of graphite yarn in a graphite jig which could be manipulated into or away from the crucible by an external control in the lid of the vacuum tank. At the completion of the run the lime-containing jig was allowed to freeze in the iron. When cold, the crucible was sectioned and the iron containing the immersed lime crystal was mounted, polished and examined with optical metallography and also with the electron microprobe. The same procedure was repeated with lime crystals which had been exposed to the atmosphere above the melt but not immersed. Particular attention was paid to the iron-lime or gas-lime interface where a calcium sulfide layer had built up during the course of the experimental run.

The batch materials used for the iron alloy were Electrolytic Glidden Pure Iron Melting Stock (Regular A-101 type),* granular FeS, and graphite chips. The

*Iron supplied by Inland Steel Research Labs.

lime was commercially fused CaO (Dynacel).* Each

*Supplied by Dynamit Nobel Aktiengesellschaft.

crystal was cleaved before each run, having the double advantage of insuring flat, perpendicular surfaces for easy examination and flat, clean surfaces for reproducibility. They were handled exclusively with tweezers.

Fig. 3 shows a partially immersed lime crystal after exposure to a melt but before being polished. When the crystals were polished they were usually completely transparent. This macrograph is of a lime crystal from run 4a. The lime crystal was only partially immersed due to the high buildup of graphite on the melt surface. The remnants of the graphite glisten in this picture. The lighter area on the lower half of the crystal is the CaS layer; however, it has chipped off of the upper half. The slug of metal in which the crystal is embedded is one inch in diameter. The sulfur content of the liquid metal was stabilized by carrying out the experiments under an atmosphere of flowing CO at 10 mm Hg pressure. This suppressed the desulfurization due to the evolution of sulfur-bearing gases (*e.g.*, S, CS₂, CS, COS, and so forth). After the metal and crucible had cooled and the vacuum broken with air, the non-immersed crystal was removed and mounted and examined both metallographically and by electron microprobe. Sulfide layer thickness measurements were made perpendicular to the lime-metal or lime-gas interface on a calibrated, moveable stage microscope and on metallographic photomicrographs. They were confirmed by electron microprobe analysis. Microprobe traverses across the lime-metal interface were generally stepped at 2 micron intervals. The calcium and sulfur contents were measured at each point.

Two supplemental experimental runs, also carried out in a vacuum induction furnace, were made to demonstrate the affinity of CaO for sulfur-bearing liquid iron in the bottom of the two-chambered graphite crucible shown in Fig. 4. The top chamber contained fused lime: either in powder form as in Run #7 or in the form of about 20 half-gram crystals as in Run #8. Any gases given off by the iron in the lower chamber would

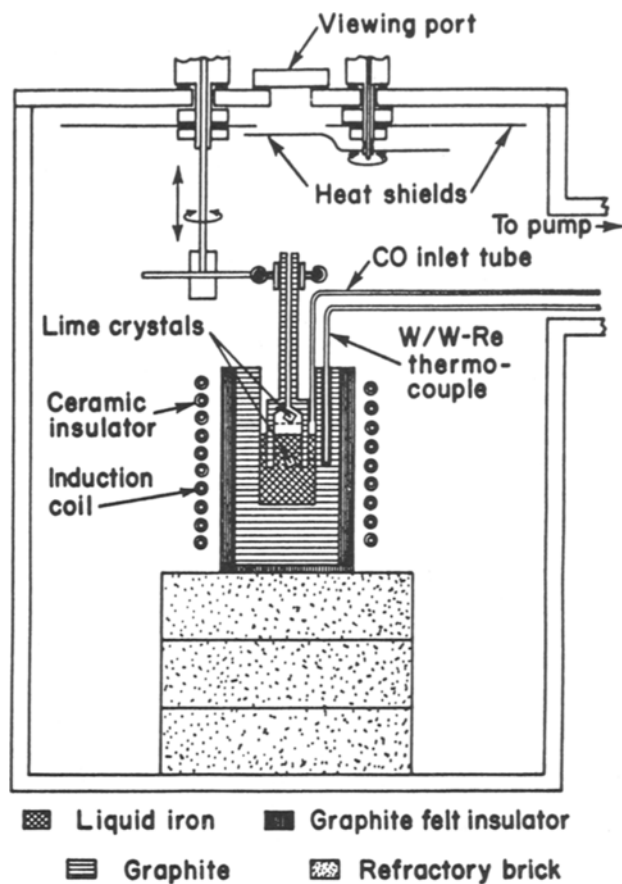


Fig. 2—Desulfurizing furnace.

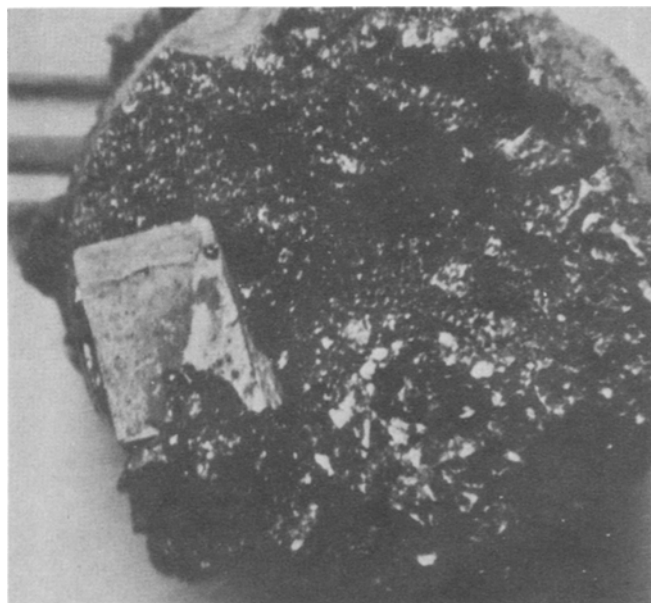


Fig. 3—Macrograph: partially immersed lime crystal.

travel through the holes in the bottom of the upper chamber, through the graphite felt, past the lime in the upper chamber and out through the top. This assured that no liquid iron would touch the lime, but that any effluent gases would have maximum opportunity to be in contact with the lime. After the run the lime was analyzed. The powder was analyzed for sulfur on a

Leco Sulfur Analyzer and one of the crystals was mounted, polished, and examined metallographically and with the electron microprobe as previously described.

EXPERIMENTAL RESULTS

In the course of the present investigation a total of twenty-two desulfurization runs was made. Six of them were made at various temperatures; eight of them were made for varying lengths of time; six were made with varying sulfur and oxygen potentials, and two were made in the vacuum-lime desulfurization crucible. The table summarizes the results of these runs. The reported average sulfur content is weighted slightly in favor of the final sulfur analyses since the bulk of the sulfur lost from the liquid iron was probably lost during the initial stages of the run. Analyses were made

in-house (Leco) and by the Homer Research Laboratories (Bethlehem).

After a lime crystal from a run was examined metallographically, a CaS layer thickness characteristic of that particular crystal was reported. Generally it was the average of five or ten or more measurements. Quite often the layer was not of uniform thickness around the perimeter of the crystal, or the lime-sulfide-metal interfaces were not clearly enough defined to make a meaningful measurement. Such problems as the crystal cracking and chipping away at the interface, or the epoxy not fully hardening and causing the sulfide to erode during polishing were common. In some samples the sulfide layer itself was cracked and separated from itself along the interface. This cracking made measurement difficult. In view of these factors, the degree of confidence with which each CaS layer thickness is reported is indicated with a super-

Table I. Experimental Results

Run No.	Melt ^(a) Temp. °C	Run Time hrs.	Gas Pressure mm. Hg	Melt Sulfur, Pct.		CaS Layer Thickness, microns		Melt Sulfur, Pct.			Oxygen ppm.	Carbon, Pct.	Silicon, Pct.
				Average	Final Initial	Not Immersed	Immersion	Initial (calc.)	Final				
									Leco	Bethlehem			
12a	1360	6	10	0.11	0.92	7 ^D , 7 ^D	(b)	0.12	—	0.11	46	4.5	0.006
14	1370	6	10	0.12	0.93	10 ^B	(b)	0.126	0.115	0.12	30	4.6	0.001
18	1415	6	10	0.12	0.97	12 ^B	15 ^D	0.118	0.110	0.12	26	4.8	<0.01
20	1530	6	10	0.11	0.96	12 ^D , 12 ^D	15 ^B	0.120	0.10	0.11	28	4.5	<0.01
13	1595	6	10	0.11	0.76	70 ^{C(c)}	35 ^D	0.126	0.093	0.10	47	4.6	0.002
9 ^(d)	1460	6	10	0.11	0.95	18 ^C	20 ^D , 16 ^D	0.118	0.113	0.11	52	5.1	0.057
10	1460	1	10	0.11	0.84	12 ^D	12 ^A	0.123	0.107	0.10	44	5.1	0.007
4a	1460	2	10	0.13	0.86	not done	15 ^B	0.14	0.121	—	—	—	—
6	1460	2	10	0.11	0.93	12 ^{A(e)}	10 ^D	0.119	0.111	—	—	—	—
15	1465	4	10	0.10	0.86	17 ^B	15 ^B	0.122	0.10	0.11	54	5.1	<0.01
17	1465	8	10	0.12	0.88	11 ^C	16 ^B	0.125	0.097	0.12	48	5.1	<0.01
19	1465	10	10	0.11	0.79	15 ^D , 15 ^B	17 ^A	0.120	0.08	0.11	24	4.3	<0.01
11	1460	12	10	0.10	0.78	(f)	23 ^C , 22 ^B	0.121	0.100	0.088	60	5.2	<0.002
16	1470	12	10	0.11	0.89	15 ^A	15 ^A	0.123	0.110	0.11	48	4.7	<0.01
1	1460	2	0.1	—	0.18	not done	44 ^A	0.3	0.055	—	—	—	—
2	1460	6	0.1	—	0.037	not done	30 ^B	0.3	0.011	—	—	—	—
3	1460	2	0.07	0.01	0.32	not done	11 ^C	0.025	0.008	—	—	—	—
5	1460	2	1	0.1	0.70	not done	(g)	0.14	0.098	—	—	—	—
21	1470	6	10	0.01	1.00	8 ^D	12 ^B	0.013	—	0.016	30	5.4	<0.01
22	1470	6	100	0.01	1.00	10 ^D	12 ^B	0.013	—	0.016	49	5.3	<0.01
7	1460 ^(h)	2	0.06	—	0.17	not done	not done	0.111	0.019	—	—	—	—
8	1480	1½	0.06	—	0.60	11 ^C	not done	0.121	0.073	—	—	—	—

(a) The reported metal temperature is the estimated temperature at the center of the metal (where the immersed lime crystal is); it is 20°C less than the temperature measured in the crucible wall.

(b) The crystal perimeter was not well-enough defined for meaningful measurement.

(c) The layer thickness varied by about 6 microns but there was a well-defined, measurable layer around almost 100% of the perimeter.

(d) The 16^D micron layer thickness is the one in the middle crystal, which was immersed for six hours but pulled out before the iron solidified. It was mounted in epoxy.

(e) The middle crystal, not immersed for the first hour of the run then immersed for the second hour, had a layer thickness of 11^B microns.

(f) The crystal was inadvertently immersed.

(g) The crystal was unsatisfactorily mounted.

(h) The temperature of Run 7 varied as much as 40°C from the mean temperature that is reported.

The degree of confidence with which each reported layer thickness should be regarded is indicated by a superscript with the following meanings:

A—90% of the measurements made were within 2 microns of the reported value and that the sulfide layer was measurable on 90% of the perimeter.

B—90% within 4 microns and measurable on at least 75% of the perimeter.

C—90% within 6 microns and measurable on at least 50% of the perimeter.

D—The variation was more than 6 microns OR the layer was measurable on less than 50% of the perimeter.

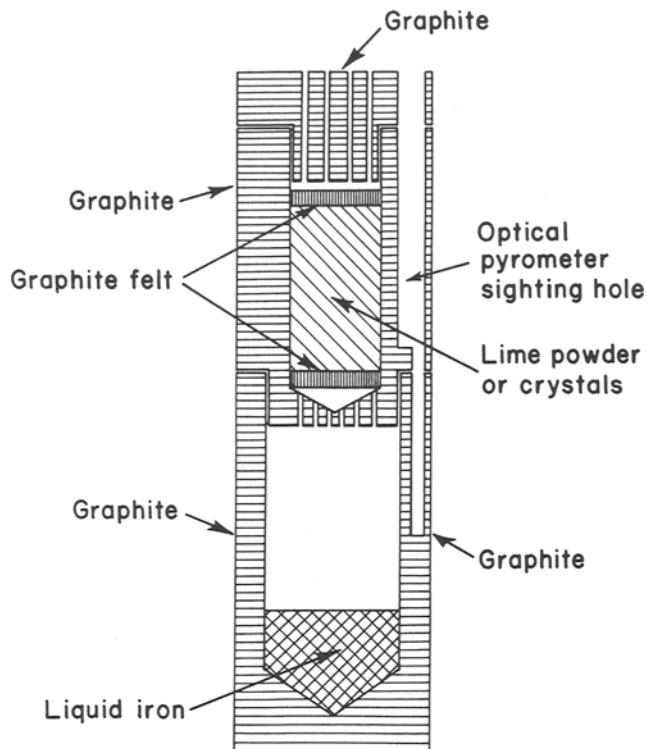


Fig. 4—Two-chambered vacuum-lime desulfurization crucible.

script in the table. The most significant correlations of the data are shown graphically in Figs. 5, 6, and 7.

Effects of Temperature and Time on CaS Formation

Even though there is a scatter in the data, several significant conclusions can be made. Reference to the 7th and 8th columns in the table shows that the formation of CaS proceeds just as rapidly on crystals suspended above the melt as on immersed crystals. As a result of the scatter, it is impossible to tell from Figs. 5 and 6 which set of crystals, immersed or non-immersed, had the thickest CaS layers over the time and temperature ranges studied. Thus all of the points in each figure are represented by one straight line, fit by the least squares method.

The Arrhenius plot in Fig. 5 shows that the rate of CaS formation is sensitive to temperature. The equation of the line is

$$\log x' = 7780(1/T) + 5.67$$

where x' is the thickness of the CaS layer in microns and T is the temperature in K. In more conventional terms this relationship can be represented by:

$$x = 46 e^{-Q/RT},$$

where x is the layer thickness in centimeters, R is the gas constant, 1.987 calories/gm mole-K, and Q is an apparent activation energy equal to 36 Kcal/gm mole. The standard deviation of the slope of the line in Fig. 5 represents a range of about ± 8 Kcal for the activation energy.

Parabolic behavior of the formation of CaS with time may be inferred from Fig. 6. At best it is merely an approximation to the time dependence of the growth of CaS and can by no means be taken as a definitive mea-

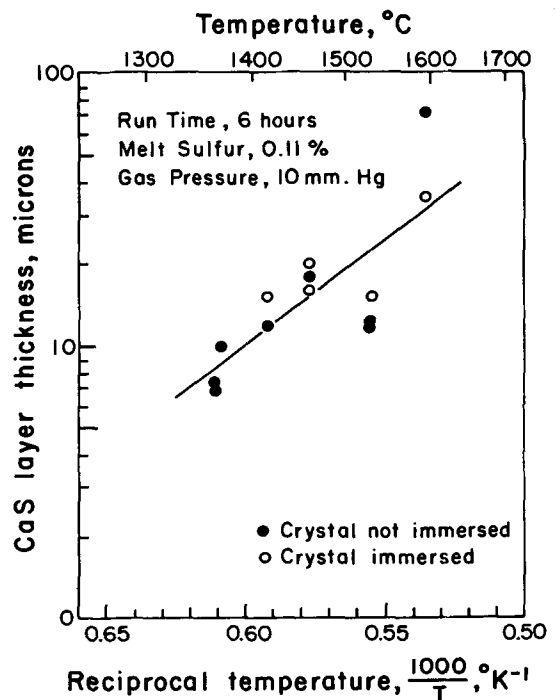


Fig. 5—Effect of temperature on CaS layer thickness.

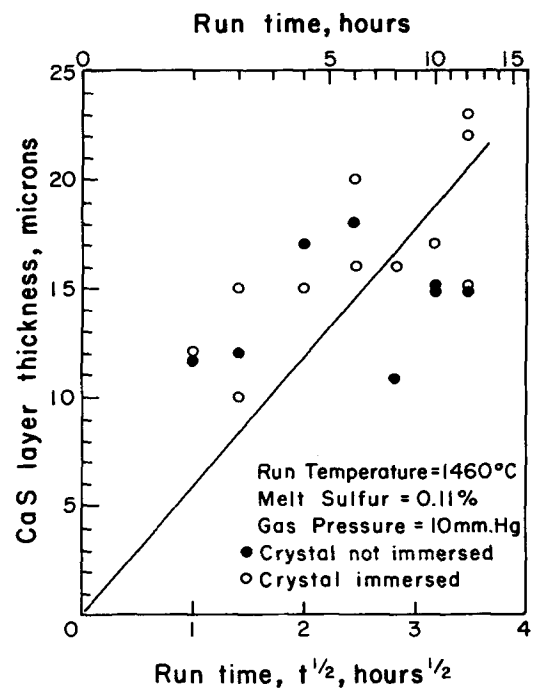


Fig. 6—Effect of time on CaS layer thickness.

sure of it. The erratic behavior is most probably due to the observed chipping, scaling, and porosity in the layers. This premise is substantiated by the results of the metallographic and microprobe studies. The equation of the line of Fig. 6 is

$$x' = 5.917 t^{1/2}$$

where x' is the CaS layer thickness in microns and t is the time in h. In more conventional terms,

$$x = (2Kt)^{1/2}$$

or

$$\frac{dx}{dt} = K/x$$

where x is the CaS layer thickness in centimeters, t is the time in seconds, and K is a rate constant, $4.9 \times 10^{-11} \text{ cm}^2/\text{s}$. The standard deviation of the slope of the line represents a range of about $\pm 2 \times 10^{-11} \text{ cm}^2/\text{s}$ for the rate constant.

Rates of Vacuum Desulfurization: For all of the runs with individual lime crystals, the amount of sulfur which could be accounted for as CaS was stoichiometrically about 1 or 2 pct of the initial sulfur content of the iron. Accordingly, the observed total desulfurization rate of the iron was not controlled by the lime desulfurization reaction but depended instead on the reactions of vacuum desulfurization, *i.e.*, the formation of S, CS₂, CS, S₂, and COS gases.

Fig. 7 gives a semi-quantitative summary of observed vacuum desulfurization during the 16 runs that were made at or near 1460°C. The desulfurization rates are at best characteristic of the experimental apparatus and conditions used. However, the results show clearly the necessity for operating at gas pressures of the order of 10 mm Hg or more to minimize the loss of sulfur due to the formation of sulfur-bearing gases. The lower curve in Fig. 7 agrees well with similar conditions reported by Fruehan and Turkdogan,⁹ indicating that losses of sulfur at these conditions were primarily by evaporation. An analysis of these conditions is given by Belton *et al.*¹⁰

The object of run 7 was the maximum vacuum desulfurization of the liquid iron and the maximum exposure of the effluent gas to the powdered lime. The run lasted 2 hours at 0.06 mm Hg. The 100 grams of liquid metal went from 0.111 pct S to 0.019 pct S, a loss of 92 mg

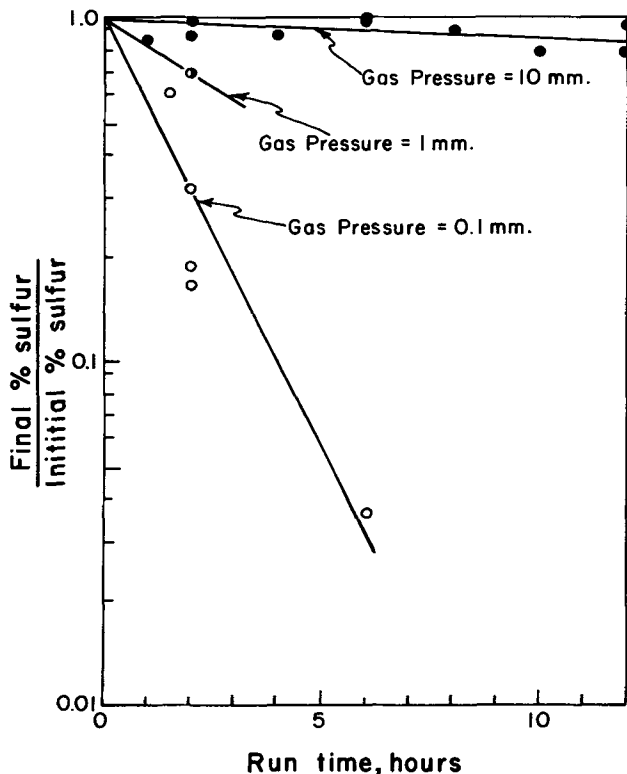


Fig. 7—Effect of total gas pressures on desulfurization at 1460°C.

sulfur. The 20 grams of powdered lime went from 0 pct S to 0.36 pct S, a gain of 72 mg sulfur. About 80 pct of the sulfur lost from the iron was recovered in the lime. All three analyses of the mixture of CaS and CaO which was used as a standard agreed to within 0.03 pct sulfur for an average sulfur content of 0.43 pct. The two analyses on the unknown agreed to within 0.01 pct sulfur for an average sulfur content of 0.36 pct.

In run 8, however, not nearly as much desulfurization was achieved (0.121 pct S to 0.073 pct S in 1-1/2 h). This was probably because a great deal of kish graphite built up on the surface of the previously carbon-saturated iron melt as it was rising to temperatures. Nevertheless a CaS layer 11 microns thick was built up on the lime crystal which was exposed only to the gases coming off of the melt.

Microprobe and Metallographic Results: The results of the microprobe analyses were all plotted as intensity ratios versus distance. A value of 1.00 on the ordinate corresponds to the intensity of the CaS standard minus the background. Intensity ratios from microprobe data are closely related to weight fraction; furthermore, CaO contains a greater weight fraction of calcium than does CaS. Therefore, when the microprobe traverse leaves the sulfide and enters the oxide, the calcium intensity is observed to increase.

Fig. 8 shows an ideal and generally typical traverse. It shows that a 19 micron layer of the same calcium density and sulfur density as the standard CaS has formed on the lime. The traverse also shows the layer to be in direct contact with the lime. The calcium density sharply increases upon passing from the calcium density associated with CaS to the calcium density associated with CaO. The calcium density then becomes constant, and the sulfur density goes to zero.

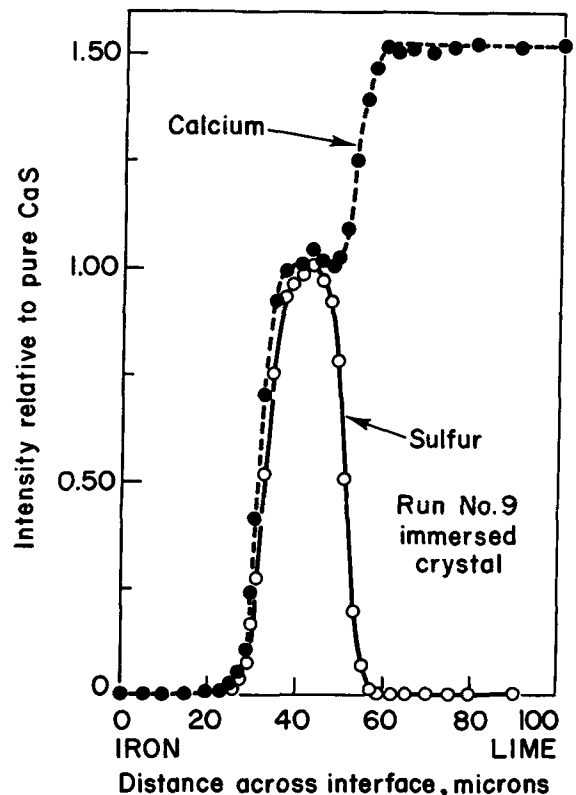


Fig. 8—Electron microprobe traverse.

Fig. 9 is a photomicrograph of the immersed crystal of run 4a. The lime crystal is in the upper right part of the picture, and the iron mounting is the white area in the lower left corner. The CaS layer is white strip that runs along the crystal and back into the crack in the crystal. The grey material filling the crack in the crystal between the CaS layers, and again between the CaS and the iron, is the epoxy used for filling the large voids to facilitate polishing. The lines drawn on the picture are the paths of the traverses shown in Figs. 10 and 11. The calcium sulfide layer can obviously be distinguished quite readily from the lime; however, it is granular and somewhat uneven, thereby making measurement difficult. One of the most interesting features of this picture concerns the crack which developed in the lime crystal near the start of the experimental run. Even though no iron is in the crack, the CaS layer still formed along the sides of the crack. This type of phenomenon, which was noticed in several crystals, suggests high mobilities of sulfur and oxygen ions along the surface of the lime. Figs. 10 and 11 are microprobe traverses which follow the paths drawn on the photomicrograph of Fig. 9. They confirm the white layer to be calcium sulfide.

The results yield evidence to support mechanisms for sulfur transfer from liquid iron to solid lime via a gaseous intermediary such as is illustrated in Fig. 1(a).

Thermodynamic calculations⁷ have shown that monatomic S gas and CS₂ gas are the predominant gas species escaping from carbon-saturated iron; CS, S₂, and COS are of lesser importance depending upon the sulfur content of the iron and the CO partial pressure. Fig. 7 shows clearly that liquid, carbon-saturated iron can be effectively desulfurized by exposing it to reduced pressures. Thus, any mechanical or chemical means which reduce the partial pressures of these sulfur-bearing gases near the iron will aid desulfurization. From a thermodynamic point of view, lime can act as an efficient getter for the sulfur in the gases coming from the iron. The role of lime as a getter for gaseous sulfur was demonstrated by the results of run 7. In this run all of the effluent gases from the iron were pulled through a bed of powdered lime. Eighty-

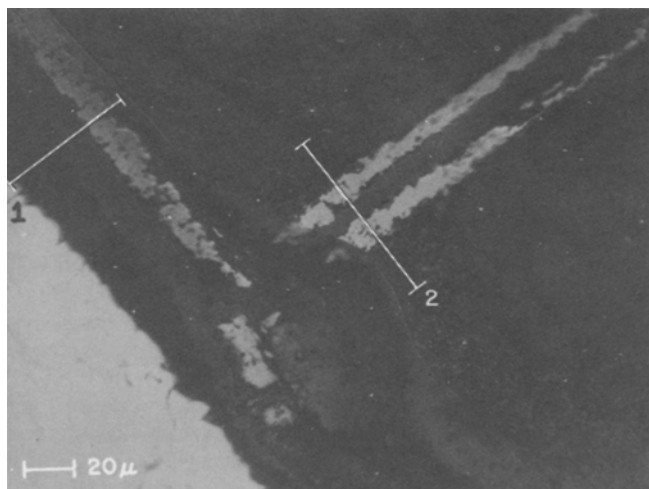


Fig. 9—Macrograph: The CaS layer on the lime crystal of run 4a.

three pct of the sulfur in the iron volatilized in two hours, but about 80 pct of the sulfur that volatilized was recovered in the lime.

Reference to Figs. 5 and 6 shows that calcium sulfide will form at the same rate on calcium oxide whether

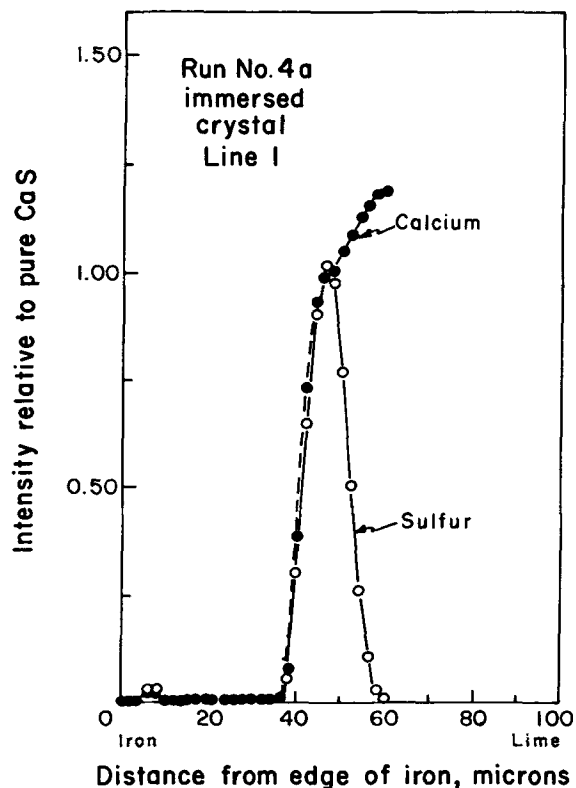


Fig. 10—Electron microprobe traverse of run 4a, line 1.

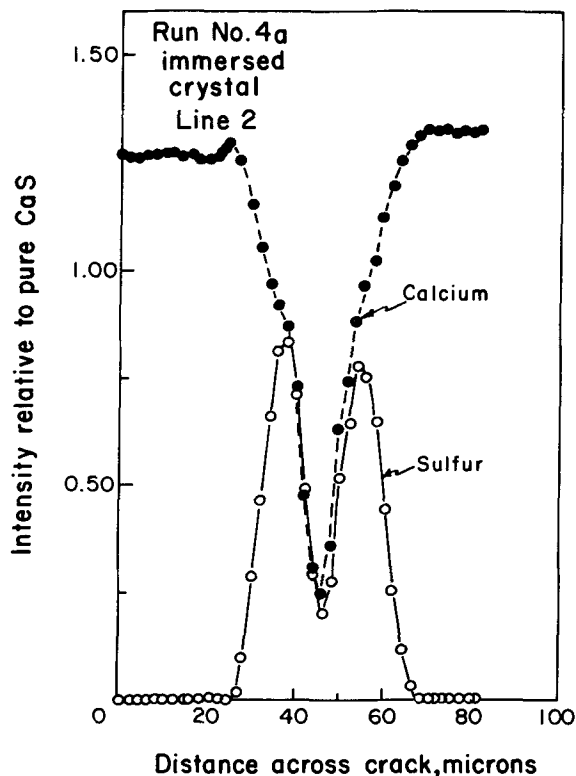


Fig. 11—Electron microprobe traverse of run 4a, line 2.

the CaO is immersed in the liquid iron or held in the gases above it. This similarity in rates indicates that if there is not already a gas intermediary which forms between lime and the iron surrounding the lime, then introducing such an intermediary of relatively large thickness will not diminish the overall rate of sulfur transfer. Hence, the presence of a gas intermediary is at least an important, if not vital, step in the process of desulfurizing iron with lime.

The research has not been able definitely to identify the rate-limiting step for the desulfurization process; however, the apparent rate constant, K , for the thickening of the calcium sulfide layer with time is taken from the slope of the line in Fig. 6 and is equal to 4.9×10^{-11} cm²/s. The apparent rate constant is about the order of magnitude that one would expect if solid-state diffusion was the rate-limiting step. Sulfur and oxygen have relatively high mobilities along the surface of cracks which form in the lime (see Fig. 7) during the experimental runs. Since surface diffusion is several orders of magnitude faster than bulk diffusion, the solid-state diffusion through the CaS may be augmented by surface diffusion along grain boundaries and fissures which develop in the lime.

The data of Fig. 5 may be applied as follows to a system in which sulfur removal is predominantly by gettering with lime. For instance, suppose that 200 mesh (74-micron particle diameter) powdered lime is exposed to sulfur-bearing pig iron for three minutes at 1460°C. The data in Fig. 5 predict that the depth of penetration of sulfur into each lime particle will be about 1.3 microns. This coat of calcium sulfide which builds up on each lime particle (assumed spherical) represents about 8 pct of the total weight of the lime.

To illustrate that this percent conversion is reasonable, consider the work of Trentini, Wahl, and Allard.² They desulfurized 5500 lbs. of liquid pig iron from 0.095 to 0.009 pct S by blowing 112 lbs. of 200 mesh lime through the iron for 3 min. Nitrogen was the carrier gas. The calculated amount of sulfur extracted from the iron was 4.7 lbs., which is equivalent in sulfur content to 10 lbs. of calcium sulfide. Thus, Trentini achieved 9 pct conversion of lime to calcium sulfide.

The agreement between Trentini's pct conversion to calcium sulfide and the 8 pct conversion calculated from the rate constant determined in this work is somewhat coincidental. Trentini carried out his work at 1300°C. However, the error introduced by the temperature difference is apparently counteracted by the assumption of minimum surface area (spherical geometry) for the lime particles. For example, in Trentini's work the depth of penetration of the sulfur into the lime may have been only half that which the equation predicted due to the temperature difference. On the other hand, the specific surface (square centimeters per cubic centimeter) of the lime may have been twice that which was calculated above owing to irregularities in the surfaces of the lime particles.

In spite of the uncertainties inherent in the above comparison, Trentini's results can still be interpreted in light of the results of the present work. The present experiments have shown that the maximum penetration of sulfur into the lime during Trentini's experiments could only have been 1 or 2 microns. Therefore, in

order to achieve the degree of desulfurization that Trentini achieved, nearly the entire surface of the 200 mesh lime had to have been coated with calcium sulfide by the end of the process. However, the entire surface area of Trentini's lime was probably not in intimate contact with the liquid iron for the full three minutes, especially since the lime was carried into the iron in a stream of nitrogen and probably floated to the top with the help of gas bubbles. Hence, a substantial amount of the sulfur must have been transferred from the liquid iron to the solid lime via a gas intermediary.

CONCLUSIONS

1) The mechanisms which incorporate a gas phase intermediary between liquid iron and solid lime are at least as important in the desulfurization process as the mechanism by which sulfur is transferred directly to the lime from the liquid with subsequent carbon deoxidation.

2) The process of desulfurization with solid lime is possibly controlled by the solid-state counter diffusion of oxygen and sulfur through the CaS which builds up and surrounds the lime. The bulk diffusion may be augmented by surface diffusion along grain boundaries and fissures which develop in the CaS.

3) In a commercial system in which a continuous high vacuum might not be attainable the role of the lime would be to act as a getter for the sulfur in the desulfurizing gases in order to keep their partial pressure below saturation level, thus allowing continuous sulfur evolution from the liquid iron.

4) Blowing powdered lime through sulfur-bearing liquid iron is extremely effective since it incorporates the double advantage of having a large iron-gas interfacial area and a large lime surface area near the liquid-gas interface.

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