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LIME-BASED SLAGFORMERS, REFINING & ALLOYING POWDERS, CASTING MOLD FLUXES IN IRON AND STEEL INDUSTRY

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III. TECHNOLOGIES OF CASTING MOLD FLUXES IN STEELMAKING

浇铸保护渣技术

浇铸保护渣技术

中 文 摘 要

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连铸是炼钢过程的一项重要操作。使用浇铸保护渣的主要目的是为了使铸件获得较好的表面质量。模内的热流密度是浇铸的函数，并随着保护渣成分变化（图3、表1）、保护渣的厚度和热导率控制着传热过程，保护渣的熔化速度控制保护渣的供给量，而液态保护渣的粘度又影响着渣层厚度。液态渣的结晶温度影响着渣层的特性。因此，本文着重研究了控制熔化速度、粘度和结晶温度的基本参数。

实验过程：表Ⅰ列出了各种保护渣粉剂的组成、结晶温度和1300 ℃的粘度值。完成了熔化速度、粘度、差热分析、化学分析和熔化行为等诸方面的实验研究。

图4为测量熔化速度所用设备，根据熔化渣滴的重量计算熔化速度。图5为粘度测量装置。在1500 ℃下每下降50 ℃测量一次，平衡时间20－30分钟，至到1100 ℃为止。先由低粘度范围粘度计测量，然后由高粘度范围粘度计测量。差热分析用来确定保护渣体系的凝固温度。

结果和讨论：

①熔化速度：图6表明，熔化速度是保护渣中自由碳含量的函数。图6－8都说明，随着自由碳含量的增加，保护渣的熔化过程受到阻碍。并且随碳粒度的降低熔化速度也降低。

②粘度，保护渣的粘度取决于渣中各氧化物组分的含量。Al_2O_3和SiO_2流动性差，使渣粘度增加；Li_2O和Na_2O流动性好使渣粘度降低。本文用“玻璃体比例”（即碱性氧化物比例）概念来描述氧化物的这种效应：玻璃体比例＝（Na_2O+Li_2O）／（$Al_2O_3+SiO_2$）图9表明，熔融保护渣粘度是“玻璃体比例”的函数，增加玻璃体比例粘度降低（1300 ℃）。

③差热分析：用DTA检测了12个保护渣样，典型的DTA曲线如图10所示。表Ⅰ列出了DTA检测的结晶温度值。图11表明，结晶温度是“玻璃体比例”的函数，随着玻璃体比例的增加温度下降。结晶温度是保护渣的重要性能。希望玻璃体状态保持越久越好或结晶温度越低越好。

总结：对56组不同成分的合成渣进行了粘度、结晶温度和熔化速度检测。合成渣的熔化速度受碳含量控制，二者成反比变化。熔融合成渣的粘度和结晶温度随“玻璃体比例”增加而降低。

文中引入“玻璃体比例”的概念作为解释保护渣性质的化学参数。碱性氧化物含量（即玻璃体比例）对结晶温度、传热速度和拉漏现象的产生有显著影响。

MOLD POWDER TECHNOLOGY

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ABSTRACT

Mold powders are employed in the continuous casting process to optimize lubrication and heat transfer and are therefore an integral part of producing high quality steel in the caster. A series of mold powder compositions were evaluated for fusion rate, viscosity and crystallization temperature. The fusion rate decreased with increasing additions of carbon and decreasing size of the carbon particles. A new chemical parameter, glass ratio, $(Na_2O + Li_2O)/(Al_2O_3 + SiO_2)$ has been introduced to analyse the combined effect of the different oxides in the mold powder, on viscosity and crystallization temperature. Viscosity and crystallization temperature were found to decrease with increasing glass ratio. These results can be used to develop mold powders that match the casting conditions and produce high quality cast product.

INTRODUCTION

Continuous casting is one of the most important unit operations in the overall scheme of steel production for improving the yield and quality of steel. The percentage of steel continuously cast in the United States has grown from less than 10% to more than 60% in the last decade and is projected to be greater than 75% in the 90's. The concept, as the name implies, is to cast the liquid metal directly into a slab, bloom, billet or strip on a continuous basis. The liquid steel is cast by continuously feeding it into an oscillating water cooled copper mold. The partially solidified slab/bloom/billet exits the mold and is then hot rolled (directly or after cooling and reheating). Technologists realized that the use of a powder which is molten while the steel is solidifying acts as a lubricant in the mold and thus protects the solidifying steel shell. Mold powders were developed to be added onto the liquid steel to provide a liquid glass layer of optimium thermal, chemical, and frictional properties at the meniscus in the mold. The quality of steel produced depends on what happened at the solidifying meniscus.

Much work has been done in studying the behaviour and performance of mold powders[1-5]. During the last few years, new automated manufacturing facilities have been developed to produce synthetic powders with exact chemistries.

The purpose of this work was to study the physical and thermal relationships of synthetic mold powders and to study their behavior[6]. Through knowledge of the characteristics of these powders as a function of powder constituents, blending variables and other independent variables, one can optimize the process. This knowledge will enable one to "design" synthetic mold powders for specific casting conditions and steel grades to be cast. Figure 1 describes the approach adopted in the development of these mold powders.

FUNCTIONS OF THE MOLD POWDER

Figure 2 shows the longitudinal section (parallel to the broad face) of a continuous casting mold. The principal objective of using mold powders in continuous casting is to achieve better surface quality of the product.

The various functions of the mold powders are:

(a) To protect liquid meniscus from reoxidation;

(b) To provide thermal insulation for the liquid meniscus, thereby preventing premature solidification or skulling;

(c) To provide lubrication and facilitate strand withdrawal from the mold;

(d) To absorb and dissolve high melting point scum, such as deoxidation products, and thereby prevent its entrapment in the solidifying shell;

(e) To control the heat transfer from the solidifying metal to the mold through the solidified shell;

(f) To control interfacial tension at the meniscus thereby reducing the severity of oscillation marks.

The liquid glass film formation between the strand and the mold has a critical influence on lubrication and heat transfer. Crack formation in the slab is enhanced as the heat transfer rate is increased, or when the heat removal is nonuniform across the strand. Hence it is essential that the liquid glass infiltrate in a continuous and uniform fashion, and that heat transfer at the shell/mold interface is also uniform.

Average heat flux density in the mold is obtained from measurements of the cooling water in the mold[4]. In Figure 3 heat flux density is plotted as a function of casting speed for 3 different mold powders (Table I). The infuence of casting speed and powder chemistry on heat transfer can be seen. Heat transfer through the molten powder layer is controlled by the thermal conductivity and thickness of the molten powder layer. The fusion rate of the mold powder will control the supply of liquid slag. The viscosity of the liquid slag will affect the thickness of the slag layer. The crystallization temperature of the liquid slag will affect the heat transfer characteristics of the slag layer. In this work, an attempt has been made to study the basic parameters that control fusion rate, viscosity and crystallization temperature.

EXPERIMENTAL PROCEDURE

EXPERIMENTAL MATERIALS

All the powders which were examined in this study were obtained from Natco International. Thirteen basic powder formulations were used as listed in Table II. Varying additions of carbonaceous particles were made to each powder to determine the affect on fusion rate. A total of 56 powders were evaluated.

In the design of mold powders, the oxides are put together to have the desired viscosity, and crystallization temperature. Free carbon, in the form of fine, amorphous graphite or thermal black is added to achieve the desired fusion rate. Within a group, carbon levels varying from 0 to 6% were employed. Powders within each group are compared for fusion rates, whereas powders from different groups are compared for viscosity and crystallization temperature. The chemistry of different powders along with their properties are listed in Table II. In a given group, only one or two powders were analyzed for viscosity and crystallization.

TESTING METHODS

The following tests were performed:

- fusion rate
- viscosity
- differential thermal analysis
- chemical analysis
- fusion behaviour

The first three were performed at Drexel University and the last two tests were conducted at Natco International.

FUSION RATE TEST

Fusion rate is defined as the weight of powder thet that melts in unit time per unit area at 1500°C. It is usually of the order of 10^{-3} g/sq.cm./s.

The apparatus used to study fusion rate is shown in Figure 4. The setup essentially consists of a graphite susceptor placed inside an induction coil. The susceptor is placed on a graphite base. A graphite ring is placed on the susceptor. The graphite ring and the base are slit so as not to be heated by the induction field. A thermocouple is inserted into the walls of the susceptor and the assembly is placed inside the coil. A graphite stopper rod is placed above the axial hole of the susceptor. The assembly is then heated to 1500°C and mold powder is slowly added from the top. The powder starts melting and the stopper rod is lifted up. The molten powder flows through the axial hole and is collected in a vessel placed below the coil. Fresh powder is periodically added from the top to replace the molten powder. Thus the ring has 3 layers of powder: a fresh layer at the top, a partly sintered layer in the middle, and molten layer at the bottom. The powder is heated by a surface at 1500°C. Thus the setup simulates the actual casting conditions. After about 20 minutes (for equilibrium), a timer is switched on and the molten powder is collected in a different vessel. Molten powder is collected over 15 minutes, and then the stainless steel vessel is replaced. This is repeated 2 or 3 times.

The surface area over which powder melts is known (17.096 cm^2). The time for which it melts is also known (15 minutes). The powder in the stainless vessel is weighed and the fusion rate calculated. The mean value of 3 or 4 runs is then calculated and reported as the fusion rate for that powder.

VISCOSITY

Viscosity of the powder is critical in powder performance. Usually, the powder viscosity has to be matched agianst the casting speed, through some empirical formulae. The viscosity of the powder at 1300°C in Pascal-seconds or Poise is quoted in the industry.
(1 Pa·s = 10 Ps = 1000 cPs)

The apparatus used to study viscosity is shown in Figure 5. It essentially consists of a Molybdenum crucible kept inside a graphite crucible holder, heated by induction. The Molybdenum crucible contains the mold powder. A molybdenum spindle suspended from the viscometer rotates inside the melt.

At the beginning of the viscosity test, the spindle is calibrated using standard oils at different rpm's. Two standard oils (92.5, 4800 cPs) are used with two viscometers (referred to as low and high viscometers- the former capable of measuring viscosities up to few hundred centipoise and the latter capable of measuring viscosities from few hundred to few thousand centipoise). At a given rpm, for an oil of known viscosity, the viscometer reading is noted. The calibration factor is obtained by dividing the oil viscosity by the reading. For both viscometers, the calibration factors are obtained at different rotation speeds.

During the viscosity test, the decarburised mold powder is added to the molybdenum crucible and heated by induction . The temperature is gradually increased to about 1500°C. Then the low viscometer is placed on a wooden plank above the coil and the moly spindle is suspended from it into the melt through the suspension wires. The spindle is set into motion and the readings noted down at different rpm's. At given rpm, the viscosity is obtained by multiplying the reading by the corresponding calibration factor. Then the mean viscosity at that temperature over different rpm's is obtained. Subsequently, the power is adjusted to reduce the temperature by about 50°C. Approximately 20 to 30 minutes is allowed to attain equilibrium and then the viscosity is measured as explained above. The temperature is decreased in successive steps to about 1100°C. If the viscosity value measured became to be outside the range of the viscometer in place, the latter was replaced by the high viscometer (which covers the apprpriate range of viscosities of interest). Below the crystallization temperature, the reading is very unsteady and the test is terminated. Viscosities at different temperatures are then tabulated. Log viscosity is plotted against reciprocal absolute temperature.

DIFFERENTIAL THERMAL ANALYSIS (DTA)

DTA is frequently used to determine the solidus, liquidus temperatures of metallic systems. The analyzer essentially consists of 2 crucibles, one with the sample to be analyzed and the other with the standard, with thermocouples attached to each of them. The 2 crucibles are placed inside a furnace and heated to the temperature range of interest.

For this study, alumina was used as the standard and small platinum cups (5mm dia., 5 mm ht.) as crucibles. The assembly was heated to 1400°C and then cooled to about 300°C, at the rate of 25°C/min. The temperature difference (between sample and standard) is plotted against temperature of sample. For metallic systems, peaks and valleys in the DTA curve indicate solidus and liquidus temperatures. In the case of mold powders, the exothermic peaks on cooling indicate crystallization.

RESULTS AND DISCUSSION

FUSION RATE

Fusion rate values (@1500°C, $mg/cm^2/s$) are listed in the Table II. Figure 6 shows fusion rate for all powders as a function of the free carbon content. Fusion rate values for different powders belonging to groups G and J are plotted as a function of carbon content in Figures 7 and 8, respectively. Carbon present in the powders comes from two sources, free carbon added to the mixture of oxides; and combined carbon present as carbonates in the oxide bearing minerals. The free carbon particles retard the fusion of mold powders by preventing the coalescence of the oxide particles. This is supported by Figure 6; namely, the fusion rate decreases as free carbon content increases. For example, in group G, addition of 1% free carbon to a matrix without any free carbon, decreases the fusion rate from 8.8 to 4.94 $mg/cm^2/s$; and increasing the free carbon content from 1 to 2%, results in a decrease of the fusion rate from 4.94 to 2.34 $mg/cm^2/s$.

Fusion rate of different mold powders can also be interpreted using molten slag pool depth measurements in the caster. The molten pool depth should be higher than the sum of the mold oscillation amplitude and the mold level variation. If the pool depth is smaller than the desired value, then the optimium amount of lubricant will not be available. In that case, the carbon content of the mold powder should be decreased to increase the fusion rate. Quantitative information relating the fusion rate and carbon content is necessary to make such modifications. Graphs relating fusion rate and free carbon content, obtained from this study can be used to design mold powders. A technical report from Nippon steel lists this as a factor in their methodology for design of mold powders[7].

VISCOSITY

The viscosity values at 1300°C were analysed to deduce the influence of the different constituents of those powders. The viscosity of molten powder is determined by the proportion of different oxides such as alumina, silica, sodium oxide.

Alumina and silica form complex ionic structures and therefore have low mobility and high viscosity. On the otherhand, Li_2O and Na_2O form small molecules, with high mobility and low viscosity. Thus, addition of alumina and silica would increase the viscosity of molten mold powder, whereas sodium and lithium oxides would decrease the viscosity. To get an idea of the combined effect of these oxides, a glass ratio was formulated.

$$\text{Glass ratio} = (Na_2O + Li_2O)/(Al_2O_3 + SiO_2) \quad \text{(all in w/o)} \qquad (1)$$

The viscosity of molten powder as a function of glass ratio is given in Figure 9. It can be seen that an increase in glass ratio results in a decrease in the viscosity. The glass ratio gives an indication of the tendency of the mobility of the ionic structure.

DIFFERENTIAL THERMAL ANALYSIS (DTA)

Twelve mold powders were examined using differential thermal analyzer. A typical DTA plot is shown in Figure 10. The DTA results are listed in Table II. The crystallization temperatures were then interpreted in terms of the powder chemistry. Figure 11 shows crystallization temperature as a function of tha corresponding glass ratio. From Figure 11, it can clearly be seen that as the glass ratio increases, tha crystallization temperature is decreased.

Glass is an inorganic product of fusion which has cooled to a rigid condition without crystallizing. For a substance which can be cooled to the glassy state, no discontinuous volume change is found and there is no exothermic effect corresponding to the change from liquid to solid state.

On cooling, powders which are glassy, will have a glass transition, i.e., no peaks will be found. If the powder forms crystalline precipitates, then it will not show a glass transition; it will have a peak corresponding to crystallization. Table II shows whether the powder is glassy or not. If there is a peak, the peak temperature is also listed. Again, the crystallization temperature is a function of powder chemistry.

To minimize friction in the mold, the molten powder, which is cooling as it flows down the mold, should remain glassy as long as possible or down to as low a temperature as possible. Two factors need to be considered, a)whether the powder will be glassy on cooling or will it form crystalline precipitates, and b)if it forms crystalline precipitates, what is the temperature above which it is completely liquid? Consider a mixture of oxides which forms crystalline precipitates on cooling. Growth of crystalline phases which may differ markedly in thermal expansion coefficient and other thermal properties from the parent glass, can lead to the generation of stresses on cooling. Also, in general, thermal conductivity of pure crystalline oxides are much different from that of glasses[8]. If the mold powder does not completely transform into glass, it would cause locally abnormal thermal properties, resulting in poor performance.

Literature on mold powders [9,10] shows a definite correlation between crystallinity and frequency of breakouts. Thus, crystallization temperature plays a significant role in powder performance, and needs to be studied in more detail to ensure optimum powder performance.

Information such as surface temperature of slab at different points inside the mold, and tha ratio of radiative to conductive heat transfer at high temperatures, for differant powders, is needed to validate/extend the above interpretation. If radiative heat transfer dominates the overall heat transfer process, then the increased conductive heat transfer due to crystalline precipitates would have very little effect on overall heat transfer.

SUMMARY AND CONCLUSIONS

56 synthetic bland mold powders were evaluated for viscosity, crystallization temperature and fusion rate and the properties were compared with tha composition. Fusion rate of synthetic blends is controlled by tha free carbon content. Increasing tha carbon content decreases tha fusion rate. Fusion rate is of the order of $mg/cm^2/s$. Viscosity ranges from 0.1 to 1.1 Pa.s. Viscosity of molten mold powders decreases with increasing glass ratio. Crystallization temperature decreases with increasing glass ratio.

Glass ratio has been introduced as a chemical parameter to interpret the powder properties. Alkaline oxide content (or glass ratio) has a significant effect on the crystallization temperature, and, in turn, influences the heat transfer rate and occurrence of breakouts.

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* pyroscope test

slag No.	Liquid Slag Composition, %										Melting range °C *
	SiO_2	Al_2O_3	CaO+ MgO	CaF_2	Na_2O+ K_2O	Li_2O	MnO	FeO	at 1200°C	at 1500°C	
1	41	10	35.5	9	4	0	0	0.5	2.2	0.24	1195-1210
2	38	7	32	10	11	0	0	2.0	0.5	0.085	1120-1145
3	29	12	20	16	17	0	4	2.0	0.2	0.035	1023-1063
4	36	0.5	12	21	27	2.5	0	0.5	0.04	0.016	840-870
5	30	8	26	14	9	0	12	1.0	0.19	0.038	1100-1140

Table I. Properties of some mold powders evaluated for heat flux density (ref. Figure 3) [4]

MOLD POWDER	Al2O3	Na2O	CaO	SiO2	VISCOSITY @ 1300°C [Pa.s]	CRYSTALLIZATION TEMPERATURE [°C]
A	2.77	14.20	39.49	36.98	0.14	1010
B	0.89	11.56	37.88	43.22	0.32	1060
C1	8.12	14.49	35.66	32.05	0.18	NA
C2	2.39	13.08	39.96	37.91	0.14	NA
D	8.06	12.20	32.86	36.95	0.40	NA
F	0.74	21.14	32.10	36.28	0.08	900
G	7.80	7.63	33.59	38.96	0.30	1075
J	7.96	3.52	23.56	42.41	0.37	1140
M	6.86	6.16	33.43	44.40	0.83	NA
N	2.49	8.52	40.53	38.29	0.11	1230
P	2.21	8.12	40.45	38.59	0.11	1175
Q	5.66	9.34	31.91	46.69	1.08	NA
R	6.14	9.31	39.23	37.19	NA	1070

Table II. Composition and properties of synthetic mold powders

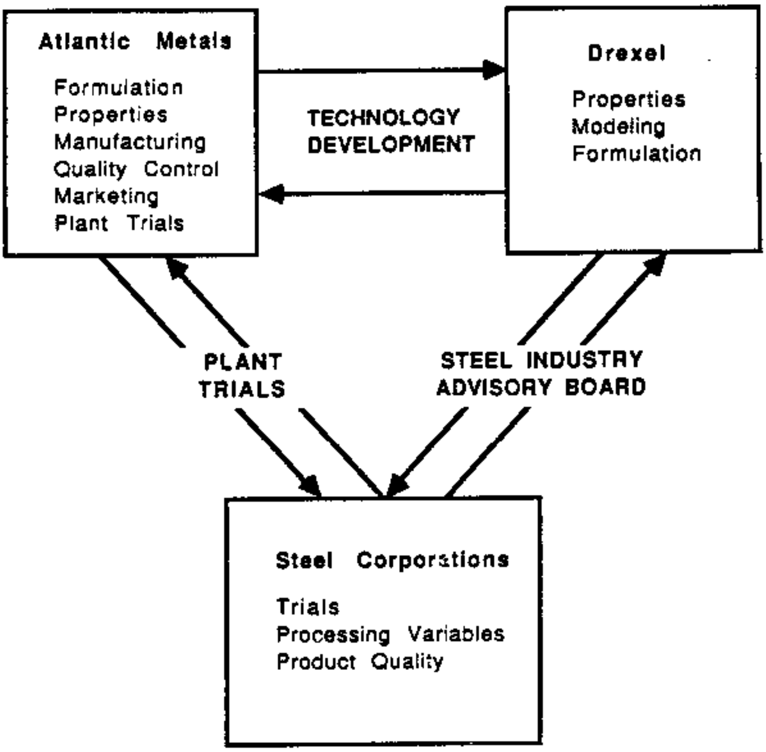

Figure 1. Technology Development and Transfer