Research Note

The effect of CaF_2 on the contact angle of refining powder on the NiCrMoV steel at high temperature using Image Analysis and Statistical Package for the Social Sciences software

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Abstract

Steelmaking refining powders always play an important role in reducing steel inclusions. These types of powders can be used in steel production processes for all types of low-alloy and high-alloy steels. The compositions of these powders are generally $CaO-Al_2O_3-SiO_2$. Adding CaF_2 values gives special properties to this type of refining powders. In this study, four types of refining powders with 3 to 6% of CaF_2 were melted in induction furnace under high temperature and the surface of NiCrMoV low-alloy steel was impregnated by the molten refining powders. The contact angle between the sample of the molten refining powders and the surface of NiCrMoV steel was determined using Image Analysis (IA) and was analysed using Statistical Package for the Social Sciences (SPSS). The results showed increasing the percentage CaF2 about 6 in refining powder of CaO-Al2O3-SiO2 reduces the contact angle of the molten refining powders on the NiCrMoV steel about 60%.

Keywords: NiCrMoV low alloy steel, Refining powder, % CaF_2, Contact angle, SPSS analysis.

1. Introduction

One of the main industries in the world of economics is the iron and steel industry, which is the raw material for a number of other important industries. After scrap metal is melted in the electric arc furnace (EAF), the molten steel is poured into the ladle and transferred to the next stage of the steelmaking unit by a crane. At this stage, ferroalloys and additives must be added to obtain the final composition of steel. By adding additives and creating special conditions, steel can be refined [1]. These additive materials can be powder and are available in various thermodynamic systems. These materials are melted in the ladle furnace stage and create special conditions for molten steel. The molten refining powders called synthetic slag are always in $CaO-Al2O_3-SiO_2$ multiple systems, and by changing the composition of these multiple systems and adding amounts of CaF_2 and MgO, non-metallic Al_2O_3 and MgO inclusions can be removed. Molten slag will have a significant effect on the removal of non-metallic inclusions by the mechanisms of flotation of non-metallic inclusion in molten metal- slag contact, separation of inclusion from molten metal to slag and dissolution of inclusion in slag [2]. A change in the chemical composition of slag will change the performance of slag. If $MnO-SiO_2-Al_2O_3$ slag is used, the combined inclusions can be predicted from the partial amounts of dissolved aluminium [3]. High-performance slag has better deoxygenating, desulfurization, inclusions removal capacity and leading to the transfer of inclusions with high amounts of Al_2O_3 to the low melting point $CaO-Al_2O_3-SiO_2-MgO$ inclusion system, however high basicity leads to high melting point and low

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fluidity. These high amounts of slag basicity have no effect on the total oxygen content of the molten steel, but lead to the entry of aluminium and magnesium into the molten iron and increase MgO-Al_2O_3 inclusions [4]. Depending on the sampling timepoint during the treatment of the ladle furnace, a different spectrum of inclusion-composition is found. If CaO-Al_2O_3-SiO_2-MgO slag is used under certain conditions, solid alumina (2CaO.2MgO.14Al_2O_3 and CaO.2MgO.8Al_2O_3) may be formed in the steel. It is well known that non-metallic inclusions found in industrial processes are in most cases heterogeneous which reflects their development during the metallurgical treatment [5]. Improvement of slag performance and reduction of slag basicity is done by adding the amounts of CaF_2 in slag, but it should be noted that it will cause environmental pollution and refractory corrosion. Slags based on CaO-SiO_2-CaF_2 also with high basicity have good deoxygenating, desulfurization and inclusion removal capacity. On the other hand, CaO-Al_2O_3 slag creates low oxygenation potential, low melting point, low viscosity and improvement in steel cleanliness. If high levels of Al_2O_3 are present in the slag, due to the slag-steel reaction, high amounts of aluminium inter into the steel melt and Al_2O_3 and Mg Al_2O_3 inclusions make [4]. The physico-chemical properties of metallurgical slags are controlled principally by their structure, basicity, composition, density, viscosity and contact angle between the molten metal and non-metallic solids. These parameters affect the quality of slag in the steelmaking process. The wetting behavior and the adhesion between molten metals and solid ceramics (refining-ladle powder) are important factors which can radically affect the efficiency of any metallurgical process [6]. Examples of situations in which these phenomena can play an important role are; removal of solid inclusions from the melt, either by flotation and filtration; electron beam button melting techniques to assess the cleanness of metals; metal casting and moulding process [4-6]. Practical determination of the wetting and adhesion properties of liquid-solid system at high temperature are generally carried out by either sessile drop or tensiometric techniques. The profile adopted by a liquid drop resting in equilibrium on a flat horizontal surface, is governed by a balance between surface and gravitational forces [6]. Mathematical equations and analysis always help the researcher significantly during the research process. A method called linear regression can be used to match the obtained laboratory data that are prepared in a standard way or obtained from the results of laboratory equipment. In this mathematical method, there are hypotheses that, if the hypothesis is true, there must be a matching of the laboratory sample with the reference sample [7]. To determine the correctness of this hypothesis, it is necessary to use mathematical software. One of these softwares is SPSS, this software can analyse a lot of statistical data on the existing mathematical hypothesis. Using SPSS software with appropriate tools can provide a good analysis to achieve specific parameters in the technical knowledge of steelmaking [8]. In this study, different samples of the slag-making powders with different percentages of CaF_2 were prepared and the effect of different amounts of this compound in the composition of slag-making powder in high temperature induction furnace and melting temperature of these powders on the NiCrMoV steel surface using SPSS software was evaluated. IA software was used to measure the contact surface angle of the molten powder on the NiCrMoV steel surface. The process of determining the contact angle of molten slag-making powders containing CaF_2 (which can be used as refining-ladle powders) on the surface of NiCrMoV steel using hypotheses and mathematical equations of linear regression and with analysis by SPSS software is a new research that can lead to the improvement of research in the steel industry and especially clean steel production.

2. Materials and research methods

Samples of low-alloy steel NiCrMoV were prepared in a cylindrical shape with a diameter of 10 mm and a length of 60 mm (Figure 1). Four samples of refining powder were prepared by combining Table 1 and weigh 15 grams, by 20 ton press in the form of cylindrical samples according to Figure 2. These cylindrical specimens were melted at critical temperature and in high temperature induction furnace graphite crucible (Figure 3). The steel sample according to Figure 1 was immediately immersed vertically and mechanically about 10 mm in the molten powder inside the graphite crucible at a temperature of 1300 °C and in the air atmosphere and went out. This process was repeated 14 times for each sample of molten powder, and using IA, the contact angle of all molten powder samples was measured on the surface of a cylindrical steel sample (Figure 4). The standard angle is determined based on a 100 mg cylindrical specimen on a steel surface and placed at a temperature of 1300 °C in an argon gas environment and when the sample was melted, the standard contact angle of the sample on low-alloy steel NiCrMoV obtained using IA [6]. The figure 5 shows it. The values of contact angle obtained for each sample of molten powder with steel-shaped sample and also steel submersible rod in molten powder samples were evaluated using SPSS software.

Table 1. Chemical compositions of refining powders A, B, C and D
(In terms of weight percentage)

Refining powders	$\%Al_2O_3$	$\%SiO_2$	%(CaO+MgO)	$\%CaF_2$
A	19.11	7.77	50.06	3.08
B	18.72	7.92	49.95	4.04
C	18.88	7.97	49.45	4.98
D	18.84	7.66	49.55	6.01

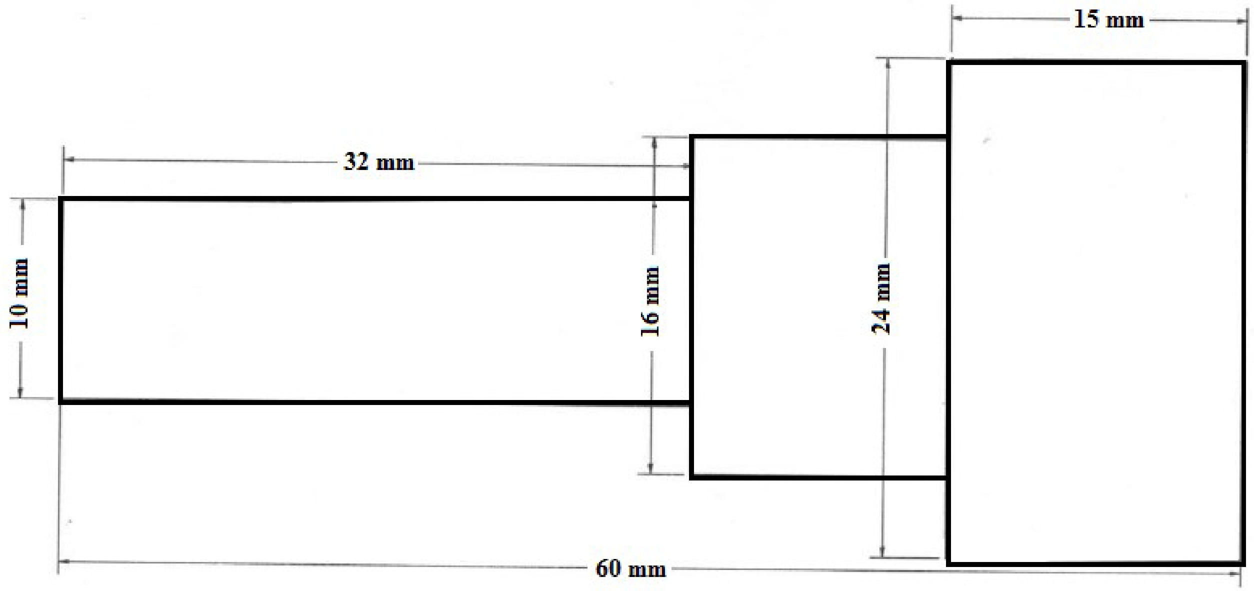

Fig. 1. Sample of low-alloy NiCrMoV steel prepared for submerging in the molten refining powder.

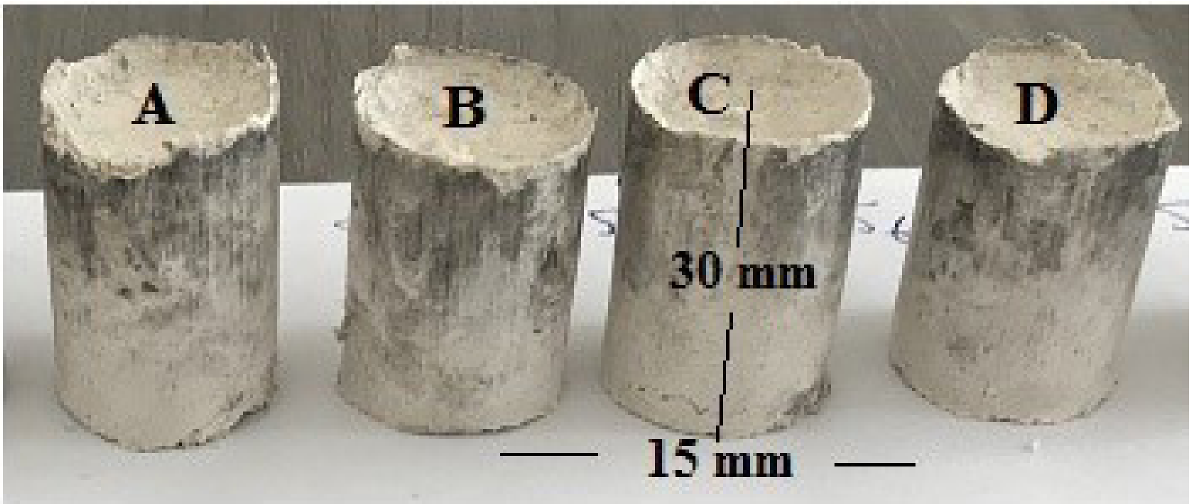

Fig. 2. Cylindrical specimens of 15 g of the pressed refining powders A, B, C and D.

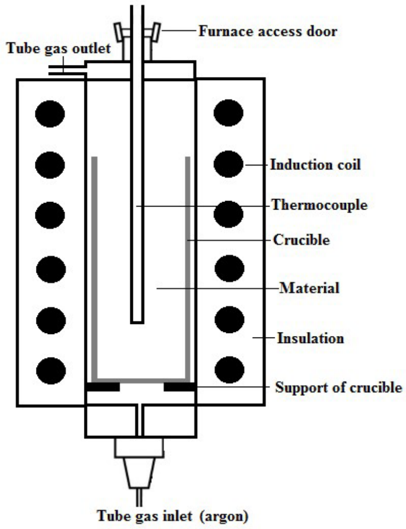

Fig. 3. High temperature induction furnace for melting 15 g and 100 mg refining powder samples.

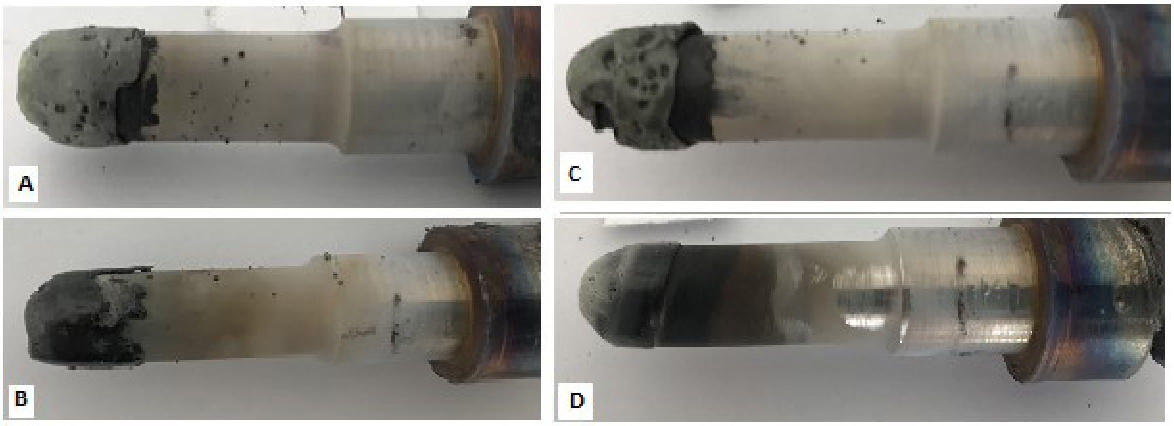

Fig. 4. Samples obtained from submerged steel cylindrical rods in the molten refining powders A, B, C and D to determination of their contact angle.

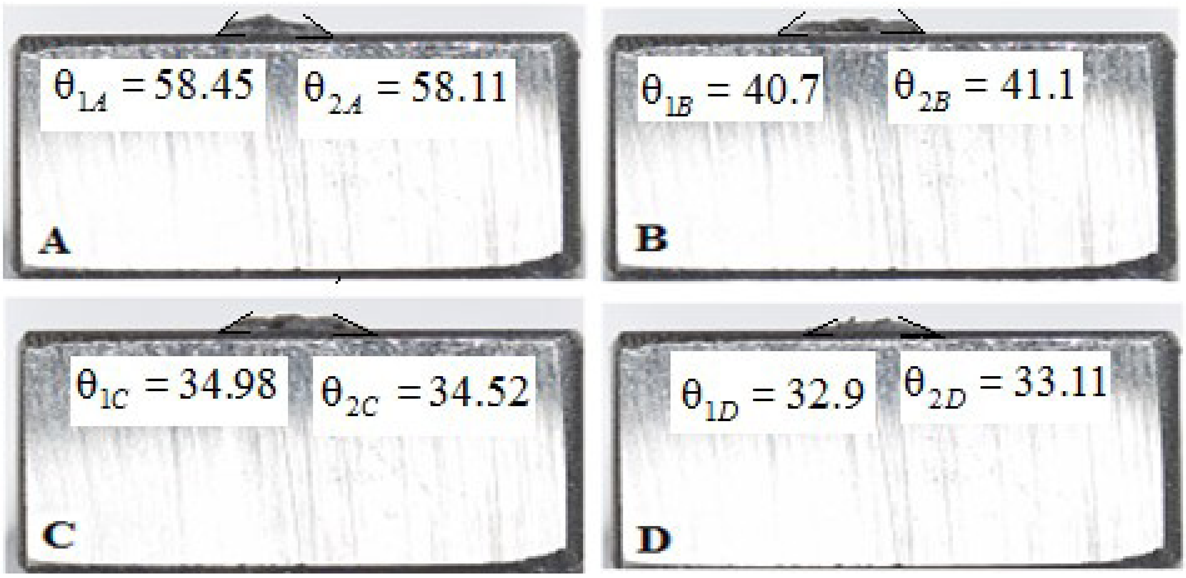

Fig. 5. The molten standard powder samples A, B, C and D on the NiCrMoV steel surface at 1300 ° C.

3. Results and Discussion

3.1. Determining the contact angle of the molten refining powders on the NiCrMoV steel surface by IA software

Four angles of the molten refining powder on the submersible rod surface were measured using IA, and then the average of these four angles for each sample (A to D) was recorded in Table 2. To increase the measurement accuracy, this process was repeated 14 times for each sample. Table 2 shows these values. The results of Table 2 can be used for SPSS analysis. Each sample was tested 14 times to obtain statistical results with high accuracy. According to Figure 5 and Table 3, the contact angles of the standard sample on the steel surface are determined. Each molten powder sample on the steel surface has two contact angles; their average was calculated and recorded in Table 3. Each sample was tested 4 times to obtain statistical results with high accuracy. Tables 2 and 3 show that the standard and measured contact angle decreases with increasing amount of CaF_2. This analysis is suitable for the chemical composition of the tested samples because the contact angle is lower in the chemical composition that has a high amount of CaO+MgO and the powder does not have good fluidity, but with the increase in the amount of CaF_2 in the chemical composition of the samples, the fluidity also increases [6]. This can have a direct effect on the contact angle of the samples.

3.2. Analysis of contact angle of refining powders with different percentages of CaF_2 by SPSS

Analysis of output data from SPSS software is done in two ways. First, it is necessary to determine the normal distribution diagram for each of the output data from the obtained angles, and after the normal distribution for four samples was determined using t-test, the contact angle values of the molten powders with the steel cylinder sample can be determined. Equation 1 shows the value of the t test [7].

$$t = \frac{\bar{x} - \mu_0}{S / \sqrt{n}} \quad \text{Eq.(1)}$$

In equation 1, $\bar{x}$ is the mean value of the data (the standard value), S is the standard deviation value, and n is the number of data.

Table 2. The average angles measured by IA software for four refining samples A, B, C and D with different percentages of CaF_2.

Sample	Angle A	Angle B	Angle C	Angle D
1	72.66	38.34	32	34.56
2	72.75	39.28	33.94	32.96
3	47.89	38.99	34.80	30.37
4	53.42	36.98	32.80	34.08
5	72.89	33.30	35.36	29.74
6	62.48	34.99	35.32	28.82
7	46.45	39.66	31.46	27.40
8	54.24	37.26	33.17	33.11
9	37.23	38.87	34.69	33.60
10	44.70	38.04	33.25	31.82
11	41.38	39.28	34.52	38.66
12	68.19	35.98	35.23	35.07
13	43.91	36.46	33.35	24.44
14	45.23	38.95	34.52	25.52

Table 3. The average angles measured by IA software for four samples of the molten refining powders A, B, C and D with different percentages of CaF_2 on the surface of NiCrMoV steel at 1300 ° C.

Angle A	Angle B	Angle C	Angle D
58.28	40.90	34.75	33.00

3.2.1. Normal distribution diagram

The value of the normal distribution function f (x) is obtained from equation 2 [7].

$$f(x)=\frac{1}{S\sqrt{2\pi}}\times\exp(-0.5\times(\frac{x-\mu_0}{S})^2) \quad \text{Eq. (2)}$$

Figure 6 shows the normal distribution diagram of four samples A to D. As shown in figure 6, all of these graphs have a normal state with a concordance coefficient higher than 95%, so the t-test can be used for them. Samples A to D have the normal distribution diagram (R2>0.95), but they were drawn with different dispersion; the reason can be in the chemical composition of these samples. The distribution diagram of A to D has shifted down and to the left, while the chemical composition of these four samples also showed that amount of CaF_2 are also increasing, so it seems that the increase of amount CaF_2 can reduce the value of f (x) in the equation 2.

3.2.2. Analysis of t-test using SPSS

Table 4 shows the analysis of data extracted from SPSS software. Data analysis by SPSS software shows that the values of the angles of the samples from A to D are 55, 38, 34 and 32, respectively. The t-test according to the standard table with relative error rate of 0.05% and for 14 samples with value of 2.16 determines that this value is greater than equation 1 for all four samples of refining powder, so the hypothesis of equation 3 is correct [7,8].

$$H_0 : \overline{X} = C \qquad H_1 : \overline{X} \neq C \quad \text{Eq. (3)}$$

Therefore, t-test completely indicates this data and the angles obtained in table 4 are in perfect match with the contact angle of refining powders on the NiCrMoV steel.

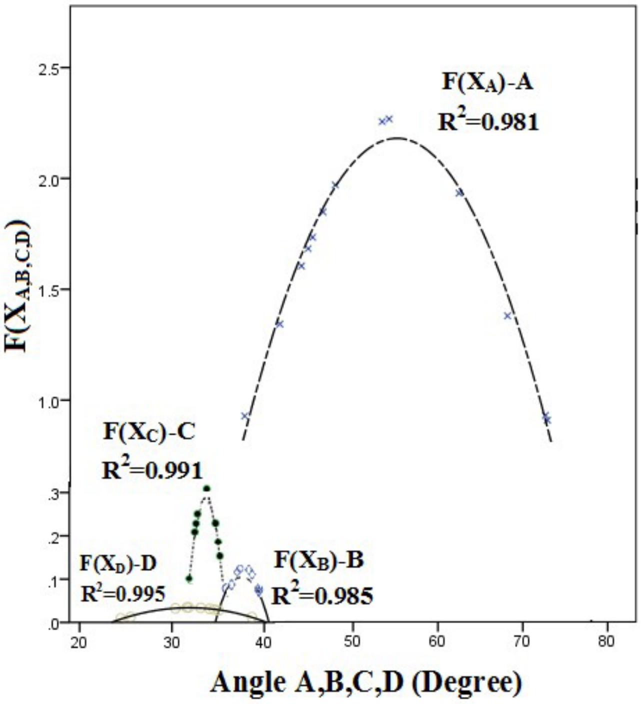

Fig. 6. Normal distribution functions for four powder samples based on the angles obtained.

Table 4. Evaluation of the angle obtained using SPSS software.

	One-Sample Test				
	df	Sig. (2-tailed)	Mean Difference	95% Confidence Interval of the Difference	
				Lower	Upper
Angle A	13	.000	54.963857	47.15394	62.77377
Angle B	13	.000	37.602357	36.51941	38.68530
Angle C	13	.000	33.890714	33.17225	34.60918
Angle D	13	.000	32.015286	29.52649	34.50408

3.3. The effect of contact angle of the refining powder containing CaF_2 on the NiCrMoV steel

The angles obtained between the molten powder samples and the surface of the steel are reduced from A to D, and it is indicated the proper contact angle (wettability) of sample D on the NiCrMoV steel. Table1 shows, by increasing the CaF_2 values, the more suitable adhesion or wettability angle can be achieved on the NiCrMoV steel. Figure 7 shows the reduction of the contact angle by decreasing the percentage of CaF_2 in the refining powder sample.

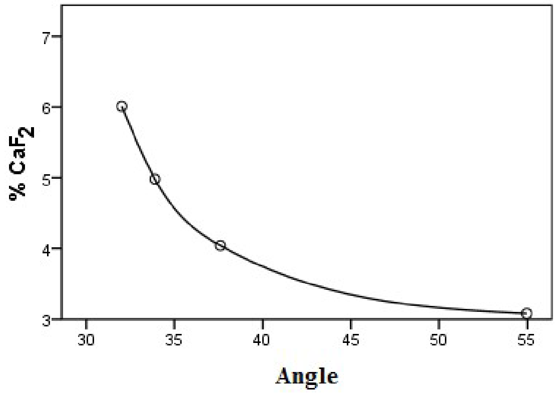

Fig. 7. Effect of percentage of CaF_2 refining powder on the contact angle (wettability) of NiCrMoV steel.

4. Conclusion

The following results were obtained from this study:

- The standardization of the contact angle (wettability) of the molten refining powders on the NiCrMoV steel surface can be done with high accuracy and 14-times repetitions.
- Increasing amount of CaF_2 to more than 5% does not have a significant effect on the contact angle.
- The design of refining powder can be done with proper analysis and the contact angle (wettability) using SPSS and a t-test with the lowest percentage of error and very suitable matching.

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