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Improvement of tensile strength and corrosion resistance of high-silicon cast irons by optimizing casting process parameters

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Abstract In this study, the relationship between casting process parameters and mechanical properties in a 14.5%Si containing corrosion resistant cast iron was statistically investigated using Taguchi method, one of the design tools of experiments. Three casting process parameters, which might be thought to be closely related to the determination of mechanical properties of high-silicon cast irons, such as melting temperature, mischmetal addition, and pouring temperature were chosen. Using signal-to-noise (SN) ratio calculated from the ultimate tensile strength (UTS) of each experimental casting run, the relationship between the casting process parameters and mechanical properties was statistically evaluated. The casting condition of high melting temperature of 1,650 °C, 0.2% mischmetal addition, and pouring temperature of 1,350 °C, led to an excellent UTS of 110–150 MPa, which is beyond the industrial criterion. The effects of casting process parameters on mechanical properties and corrosion resistance were further confirmed by combined analysis of fractography, hydrogen content determination, microscopic test, and acid resistance test.

Introduction

High-silicon cast irons have been developed with the growth of the chemical industry for processing and transporting highly corrosive fluids. When the silicon content is 14.2% or higher, these irons exhibit a very good corrosion resistance to sulfuric and nitric acids at all temperatures and solute concentrations. The 14.5%Si iron is less resistant to hydrochloric acid with concentration of above 20%, but this resistance can be improved by addition of chromium and molybdenum, and can be further enhanced by increasing the silicon content up to 17%. High-silicon cast irons have excellent resistance for most acids except hydrofluoric acid, sulfurous and marine atmosphere, some salts, bleach solutions, and etc. Therefore, they are extensively used for drain pipes in chemical plants, laboratories, hospitals, and schools, towers, tubes, and fittings in explosives and fertilizer industries, pumps, valves, mixing nozzles, tank outlets, and steam jets in paper, pigments, and electroplating industries. They are also widely used for sacrificial anodes in impressed-current cathodic-protection systems especially in aggressive environments such as seawater or chloride soils [1, 2].

But high-silicon cast irons have intrinsic difficulties of practical implementation because of their poor mechanical properties. They have high brittleness and hardness, while they have low strength, and thermal and mechanical shock resistance. Therefore, it is difficult to machine high-silicon cast irons. When the shape of castings is complicated or the respective parts of castings have different cooling rates due to a non-uniform casting thickness, they should be shaken out in a red hot state to be immediately stress relieved and furnace cooled for the prevention of cracking.

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Furthermore, since casting defects such as gas porosities, inclusions, and segregation seriously deteriorate strength and corrosion resistance of the castings, casting process parameters should be carefully controlled for commercial applications. Generally in molten cast irons, solubility of gases and thermodynamic stability of inclusions, which directly affect the formation of the casting defects, are known to be strongly dependent on melting and pouring temperatures and molten metal treatment methods [2, 3].

Two types of production methods are widely used presently for high-silicon cast iron products. The first type is the method in which master alloys are made in the 1st melting process to remove inclusions and gas porosities, and then final casting products are made by remelting the master alloys, followed by immediate casting in the 2nd melting process [4]. The second type is the method in which casting products are fabricated after dehydrogenation by inserting stirring equipment or blowing an inert gas into a melt [5, 6]. However these methods have some disadvantages of low productivity, complex production process, and usage of additional facilities.

In the present study, the casting process was optimized to produce high-silicon cast irons components, possessing good mechanical properties as well as excellent corrosion resistance. Cost competitiveness and simplification of the production line were also aimed to achieve. Taguchi method, one of the design tools of experiments [7–10], was applied to systematically investigate the relationship between casting process parameters and mechanical properties, since few reports have been published on the process optimization of high-silicon cast irons. Effects of casting process parameters on tensile strength and corrosion resistance were also confirmed by combined analysis of fractography, hydrogen content determination, microscopic examination of graphite and matrix, and acid resistance test.

Experimental

Specimen preparation and mechanical test

The chemical composition of the alloy examined in this investigation was chosen from the most common commercial alloys (ASTM A 518): C—0.9%; Si—14.5%; Mn—2%; Fe—bal. The relationship between casting parameters and mechanical properties was evaluated by measuring ultimate tensile strength (UTS). The tension test specimens of round cross section were directly prepared by casting owing to the difficulties of machining. Tension test was carried out at room temperature under a constant deformation condition of 2 mm/min, and 10 measured UTS values were averaged after omitting the maximum and minimum values.

Taguchi analysis

In order to produce a casting component of high-silicon cast irons having mechanical properties comparable to currently commercialized products through one-step melting process, melting temperature (A), pouring temperature (B), and mischmetal addition (C) were selected as casting process parameters (factors). The relationship between the casting condition and UTS was statistically analyzed by Taguchi method. All of the three factors have two levels, as given in Table 1. The levels of the factors were selected based on the casting process parameters presently adapted at high-silicon cast iron foundries. Taguchi method provides many standard orthogonal arrays for the appropriate design of experiments. In this study, $L_8(2^7)$ orthogonal array, having the number of experiment of eight and the number of rows of 7, was used. In a two-level orthogonal array, one row has one degree of freedom, corresponding to one effect (main effect or interactive effect). Therefore, if $L_8(2^7)$ orthogonal array is used in the design of the experiment with the three two-level casting process parameters, as shown in Table 2, an experimental condition of entire combination is

Table 1 Casting parameters with constituent level sets used in the present study

Symbol	Casting parameters	Unit	Level 1	Level 2
A	Melting temperature	°C	1,550	1,650
B	Pouring temperature	°C	1,350	1,430
C	Mischmetal content	wt.%	0.0	0.2

Table 2 UTS for each experimental condition according to Taguchi $L_8(2^7)$ orthogonal array

Experiment number	Casting parameter level			UTS (MPa)
	Melting temperature, A	Pouring temperature, B	Mischmetal content, C	
1	1	1	1	36
2	1	1	2	42
3	1	2	1	30
4	1	2	2	68
5	2	1	1	92
6	2	1	2	113
7	2	2	1	25
8	2	2	2	61

constructed, and consequently three main effects (A, B, and C) and three interactive effects (AB, BC, and AC) between the main effects can be statistically evaluated. To analyze the results obtained according to the above design of experiments, a statistical measure called signal-to-noise (SN) ratio was used.

Metallurgical analysis

Fractography, hydrogen content determination, microscopic examination of graphite and matrix, and acid resistance test were systematically carried out to metallurgically identify the effects of melting temperature, pouring temperature, and mischmetal addition on the characteristics of UTS and corrosion resistance. Optical microscopy (OM) and scanning electron microscopy (SEM) with EDS were used for fractography and microscopic examination of graphite and matrix. In order to investigate the effect of hydrogen gas on the formation of porosities, hydrogen content was analyzed using a hydrogen determiner RH-404 of LECO Inc. For this analysis, a cast iron melt was sampled using an evacuated quartz ampoule just before pouring. Corrosion resistance was evaluated by immersion test in an HCl solution. The test specimens with a dimension of 10 × 10 × 50 mm were immersed in a 36% HCl solution for a certain time to measure the mass loss.

Results and discussion

Evaluation of casting process parameters by Taguchi method

Table 3 Response table showing the main effects on UTS for both means and SN ratios

Level	Melting temperature, A	Pouring temperature, B	Mischmetal content, C
Mean analysis			
1	44.1	70.9	46.1
2	72.9	46.1	70.9
Δ	28.8	–24.8	24.8
SN ratio analysis			
1	32.49	36.01	32.07
2	36.04	32.52	36.46

The UTS values obtained as functions of the experimental conditions using the $L_8(2^7)$ orthogonal array are summarized in Table 2. The specimens were all fractured within elastic deformation region, showing zero elongation without any ductility. The experimental results are analyzed to investigate the main effects of casting process parameters and their interactive effects on UTS variation with change of the parameter level, as shown in Table 3 and Figs. 1 and 2. Since the experimental layout was designed orthogonally, the effect of each parameter at different levels can be separated out and also the evaluation of the combined effects between two parameters is available. For example, the means for mischmetal content at levels 1 and 2 are calculated by averaging UTS values for the experiments 1, 3, 5, and 7, and 2, 4, 6, and 8, respectively, and their difference is defined to the main effect (C):

$$C = \left\{ \frac{1}{4}\left(\mathrm{UTS}_2 + \mathrm{UTS}_4 + \mathrm{UTS}_6 + \mathrm{UTS}_8\right) - \frac{1}{4}\left(\mathrm{UTS}_1 + \mathrm{UTS}_3 + \mathrm{UTS}_5 + \mathrm{UTS}_7\right) \right\} \tag{1}$$

And the interactive effect between melting temperature and pouring temperature (AB) is defined as the difference between the main effect A when B is at level 1 and the main effect A when B is at level 2:

$$AB = \left[\frac{1}{2}\left\{ \left(\mathrm{UTS}_7 + \mathrm{UTS}_8\right) - \left(\mathrm{UTS}_3 + \mathrm{UTS}_4\right) \right\} - \frac{1}{2}\left\{ \left(\mathrm{UTS}_5 + \mathrm{UTS}_6\right) - \left(\mathrm{UTS}_1 + \mathrm{UTS}_2\right) \right\} \right] \tag{2}$$

Taguchi method employs a generic statistical measure, i.e., SN ratio, to quantify the variation of various characteristics. In Taguchi method, the terms ‘signal’ and ‘noise’ represent the desirable and undesirable values, respectively, or the mean and the standard deviation for the output characteristics, respectively [7, 8]. There are three kinds of SN ratios: lower is better (LB), nominal is best (NB), and higher is better (HB). Whatever the type of parameter characteristic is, SN ratio is always interpreted in the same way, i.e., the larger SN ratio is the better. UTS is considered as HB characteristic, in the present analysis, SN ratio was calculated by using the following equation:

$$\text{SN ratio} = -10\log\left[\frac{1}{n}\sum_{i=1}^{n}\frac{1}{y_i^2}\right] \tag{3}$$

where n is the number of experiments and y_i is the response of each experiment. Table 3 shows the mean analyses and SN ratio analyses for the effects of the various casting process parameters on UTS.

Figure 1 is the multiple linear graph showing the main effects of the casting process parameters on UTS variation both in the view of mean and SN ratio. It is clear that high melting temperature of 1,650 °C, low pouring temperature of 1,350 °C, and 0.2% mischmetal addition have higher SN ratios leading to an excellent UTS. The UTS value at the optimal combination of the casting process parameters, which corresponds to experiment number 6 in Table 2, 113 MPa, is beyond the industrial criterion (100 MPa).

Figure 2 is the multiple linear graphs showing the interactive effects between the two parameters. A strong interactive effect appeared between melting temperature and pouring temperature, that is, the slopes of SN ratio variation with melting temperature were much different for different levels of pouring temperature. It may be deduced that the significant improvement of UTS by increasing melting temperature to 1,650 °C was achieved when pouring temperature was not 1,430 °C but 1,350 °C. Between pouring temperature and mischmetal addition, a weak interactive effect was observed. Anyway, note that the addition of 0.2% mischmetal can increase UTS even at a high pouring temperature of 1,430 °C. Almost no interactive effect was observed between mischmetal addition and melting temperature, that is, the slopes of SN ratio variation with mischmetal addition were nearly same for different levels of melting temperature. This means that the addition of mischmetal consistently contributes to the remarkable increment of UTS independently on melting and tapping temperatures. It is worth to note that adding 0.2% mischmetal is efficient for the improvement of UTS in all melting and pouring temperatures.

The results of the analysis of variance (ANOVA) are given in Table 4. In Taguchi method, ANOVA using *F*-test is performed to confirm whether the process parameters are statistically significant or not [9, 10]. In this study, *F*-test was carried out with a significance level of 10%, and the *F*-ratios for all factors were larger than the critical value $F_{0.90}(1,1) = 39.9$, as shown in Table 4. This means that all main and interactive effects of the three casting process parameters on UTS were statistically significant. And the *F*-ratios indicate that mischmetal addition is the most significant parameter on the variation of UTS, and melting and pouring temperatures have the strongest interaction. This agrees well with the plots in Figs. 1 and 2.

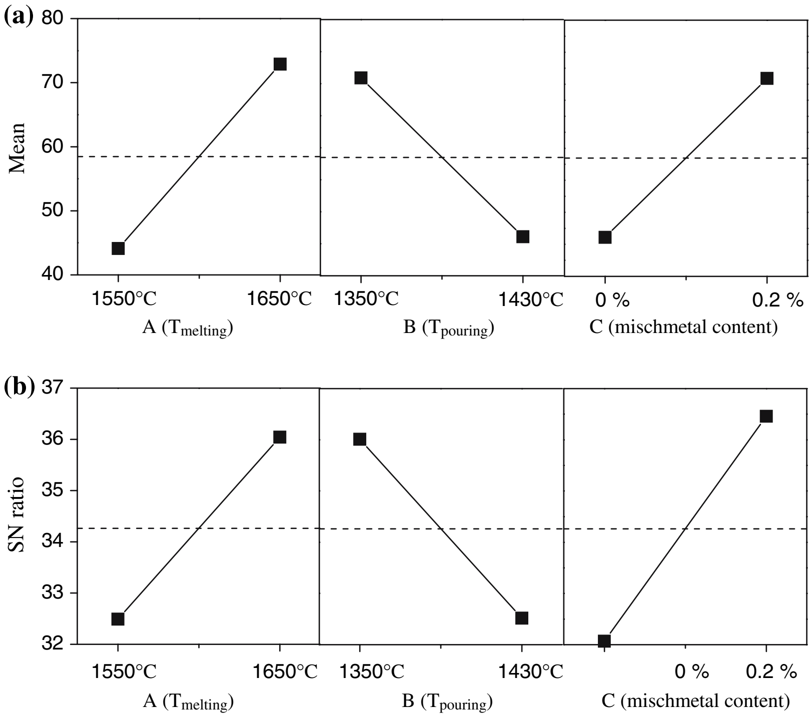

Fig. 1 Multiple graphs showing the main effects of casting process parameters on UTS both for (**a**) means and (**b**) SN ratios

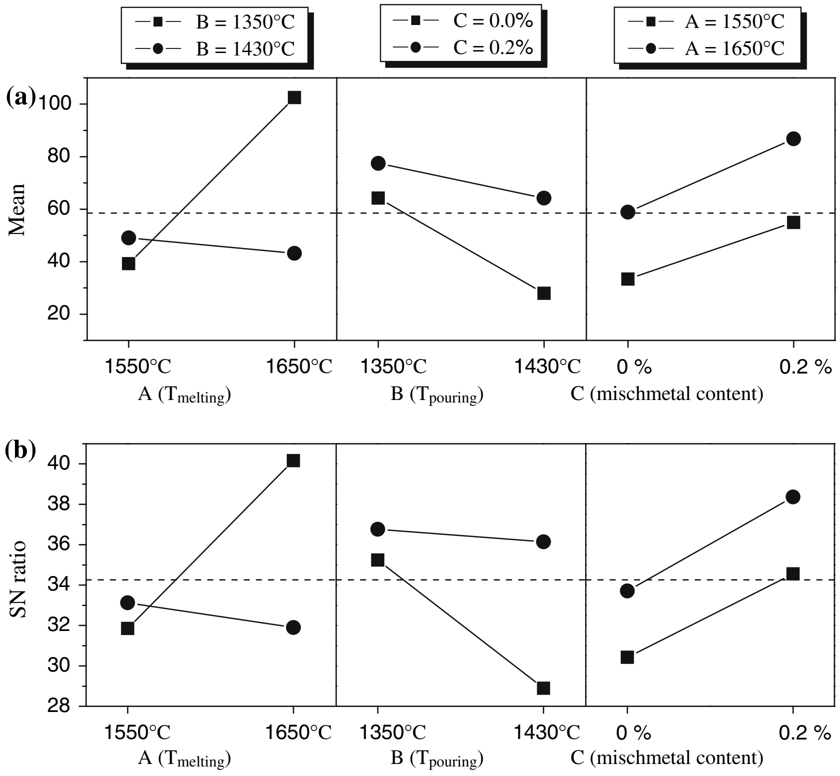

Fig. 2 Multiple graphs showing the interactive effects of casting process parameters on UTS both for (**a**) means and (**b**) SN ratios

Evaluation of casting process parameters by metallurgical analysis

In order to investigate why UTS increased with the increase of melting temperature, the fracture surfaces of tension test specimens were inspected by SEM-EDS. While oxide inclusions are not generally significant in cast irons due to their high carbon content, silicon oxide (SiO_2) may become a problem as silicon content increases, especially, in high-silicon corrosion resistant cast irons [3]. And it is worth noting that SiO_2 is more stable than CO at lower temperature (approximately below 1,550 °C), considering the free energy of formation [11]. Although in Taguchi analysis of evaluation of casting process parameters by Taguchi method, 1,550 °C and 1,650 °C were used as the lower and the higher melting temperatures, respectively, the melting temperature was remarkably varied from 1,350 °C to 1,650 °C to make a sharp contrast in its effect on the change of UTS, holding a mischmetal addition content of 0.2% and a pouring temperature of 1,350 °C. Many inclusions were observed in the specimen melted at a low temperature of 1,350 °C, as shown in Fig. 3a. It is likely that with low melting temperature the silicon oxide was stabilized, fluidity was lowered, the silicon oxide containing impurities included in raw charge materials were not completely removed during melting and remained as inclusions after casting, and as a result they made high-silicon cast irons more brittle and significantly deteriorated UTS.

Table 4 Statistical analysis of variance for UTS (*V*(e) is variance of error)

Factor	Degree of freedom (*f*)	Sum of square (*S*)	Variance (*V* = *S*/*f*)	*F*-ratio (*F* = *V*/*V*(e))
A	1	2419.50	2419.50	8641.07
B	1	2342.43	2342.43	8365.82
C	1	3705.01	3705.01	13232.18
AB	1	4385.94	4385.94	15664.07
AC	1	13.12	13.12	46.86
BC	1	1573.80	1573.80	5602.71
Error	1	0.28	0.28	
Total	7	14440.08		

The effect of pouring temperature on the formation of gas porosities was evaluated by analyzing hydrogen content, because gas porosities due to hydrogen gas have been known as a major reason of the poor mechanical properties of high-silicon cast irons. For the hydrogen analysis, the melt of the 14.5%Si containing cast iron, kept at a high temperature of 1,650 °C with 0.2% mischmetal inoculation, was sampled at various temperatures between 1,450 and 1,300 °C. The analyzed hydrogen contents were

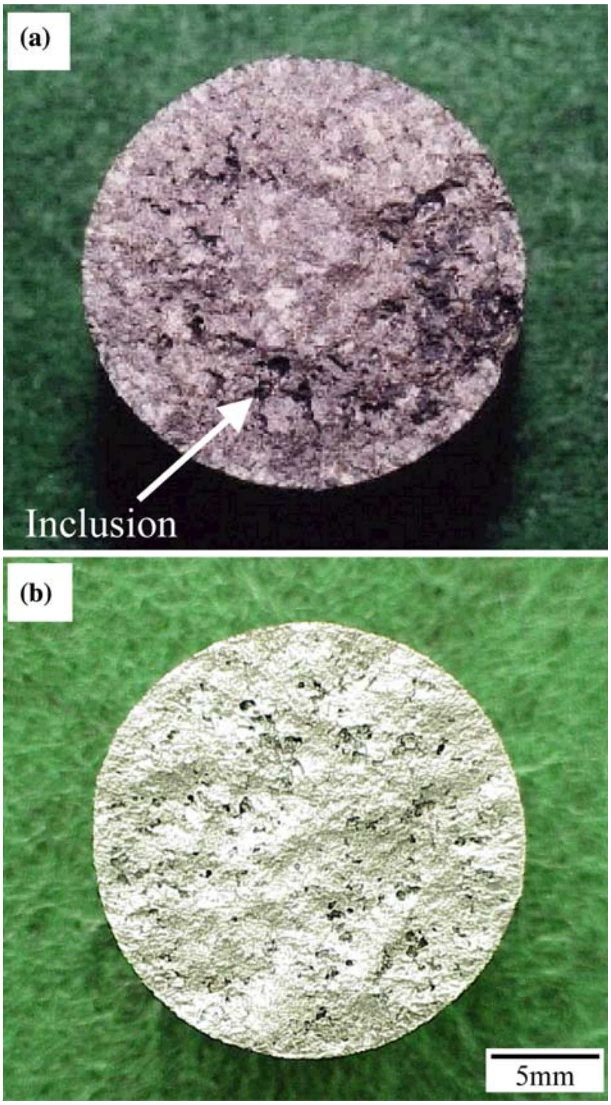

Fig. 3 Photographs of fracture surfaces with the variation of melting temperature; (**a**) 1,350 °C and (**b**) 1,650 °C

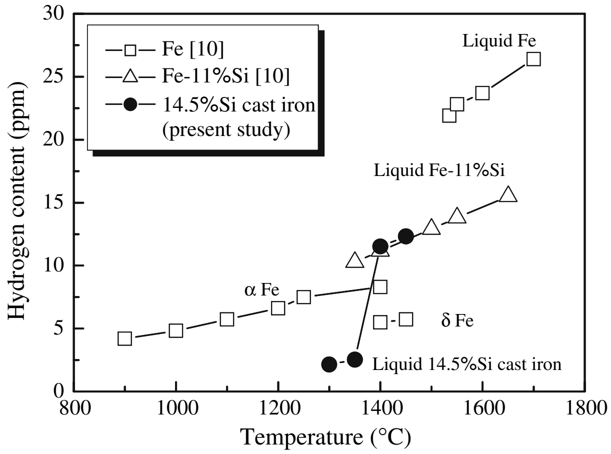

Fig. 4 Variation of hydrogen content with pouring temperature

plotted together with some reference data [12] of Fe and Fe—11%Si alloy in Fig. 4. The hydrogen content significantly decreased with decreasing pouring temperature. It can be deduced that if pouring process was carried out at a low temperature, hydrogen gas was much exhausted during cooling period before pouring after inoculation, and as a result less gas porosities formed during solidification. But a too low pouring temperature makes melt flow distance so short that it is almost impossible to keep pouring temperature below 1,350 °C in practical foundry shops, since castability is reduced and pouring defects are frequently formed.

In Taguchi analysis of evaluation of casting process parameters by Taguchi method, the addition of mischmetal was proved to be always efficient for improving UTS regardless of the other two casting process parameters. Also in the practical casting process, it will consume less electric energy and be more effective than precisely controlling melting and pouring temperatures. In general, only a little amount of mischmetal is added in steel making process [13], but there have been several research reports that in high-silicon cast irons, mischmetal was added up to 0.6% to improve strength and machinability [5, 6]. In the present study, in order to optimize the content of mischmetal addition, tension test was carried out with variation of mischmetal content up to 1.0%, keeping melting and pouring temperatures 1,650 °C and 1,350 °C, respectively. Like usual steel making process, the addition of 0.2% mischmetal was enough to improve UTS of high-silicon cast irons, and no more addition was effective for the improvement of UTS, as shown in Fig. 5. Some difference between UTS values in Table 2 and Fig. 5 under same casting conditions seems to occur due to the variation of raw charge materials, especially steel scrap. Although raw charge material is one of major factor to determine the properties of molten metal by changing moisture content and chemical composition, it will not be dealt because it is beyond the scope of this investigation.

The addition of 0.2% mischmetal was very effective for degassing, as shown in Fig. 6, which shows the cross sections of the riser part of the casting for the tension test specimens. In the sample of 0.2% mischmetal addition (Fig. 6b), only a shrinkage defect due to solidification contraction was observed at the center of the cross section, while in the sample with no addition (Fig. 6a), lots of gas porosities were observed over the whole area.

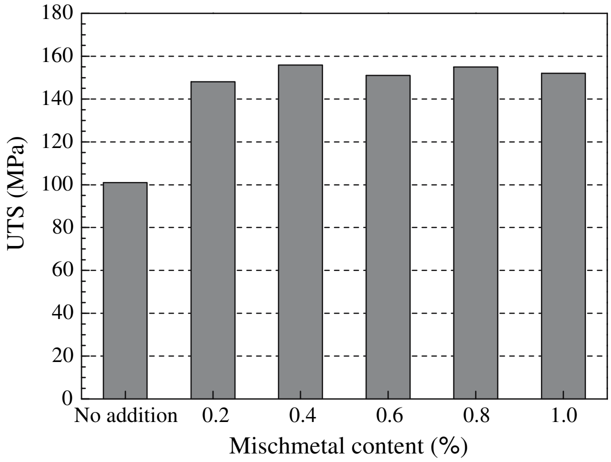

Fig. 5 Variation of UTS with mischmetal content

Figure 7 shows the representative microstructures of the 14.5%Si containing cast irons with variation of mischmetal content. Because high-silicon cast irons contain silicon, one of ferrite former, above 13%, their microstructures compose of ferritic matrix and very small flakes of graphite (Fig. 7a). The above-mentioned ferritic matrix is known as silico-ferrite, which is saturated by silicon and reveals very high hardness and brittleness [1, 2, 6]. When 0.2% mischmetal was added, inoculation effect appeared apparently and resulted in the significant refinement of primary ferrite and eutectic cell, as shown in Fig. 7b. It is also noted that the shape of graphite changed from a flake graphite of A-type to a fine eutectic graphite of D-type. Therefore, considering the degassing effect and the behavior of crack propagation as shown in Fig. 8, where a crack propagates along interfaces between primary and eutectic phases or the graphite nodules in eutectic region, it can be concluded that the inoculation with 0.2% mischmetal improves UTS by refining microstructure as well as by suppressing the formation of gas porosities. It is likely that the refinement of microstructure caused segmenting and shortening of crack propagation paths. When mischmetal was added more than 0.2% (Fig. 7c, d), no significant difference in microstructure was observed. When mischmetal was added below 0.1%, flake graphites were occasionally observed due to fading effect.

The microstructure change due to mischmetal addition had a great influence on corrosion resistance of the 14.5%Si containing cast iron, as shown in Fig. 9. The samples, which were inoculated with mischmetal of 0.2% and above, showed a very low corrosion rate in a 36% HCl solution. Rewording as; this can be understood by the fact that the eutectic fraction was decreased while the primary ferrite fraction increased, as the former has a weak corrosion resistance and the latter, better corrosion resistance. Figure 10 shows that the volume fraction of primary ferrite was much increased from 37 to 50~56% after the inoculation with mischmetal of 0.2% and above. Throughout the experimental results, it can be concluded that the inoculation with mischmetal effectively improves the mechanical properties and corrosion resistance of high-silicon cast irons by modifying microstructure, not being much affected by the mischmetal addition content and the other casting process parameters.

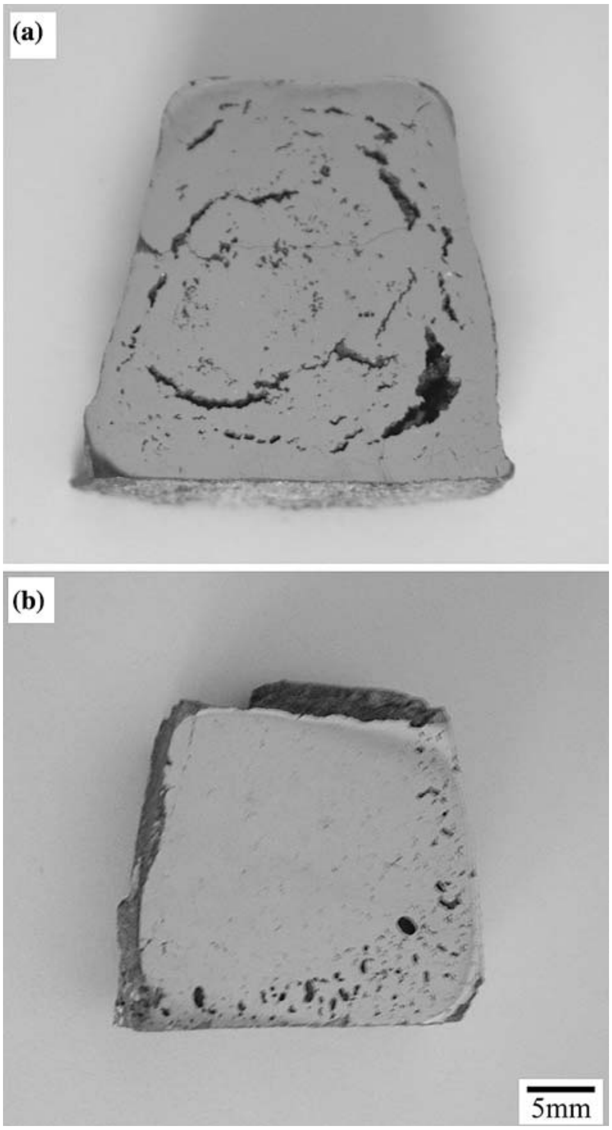

Fig. 6 The cross sectional view of a riser; (**a**) no addition and (**b**) 0.2% mischmetal addition

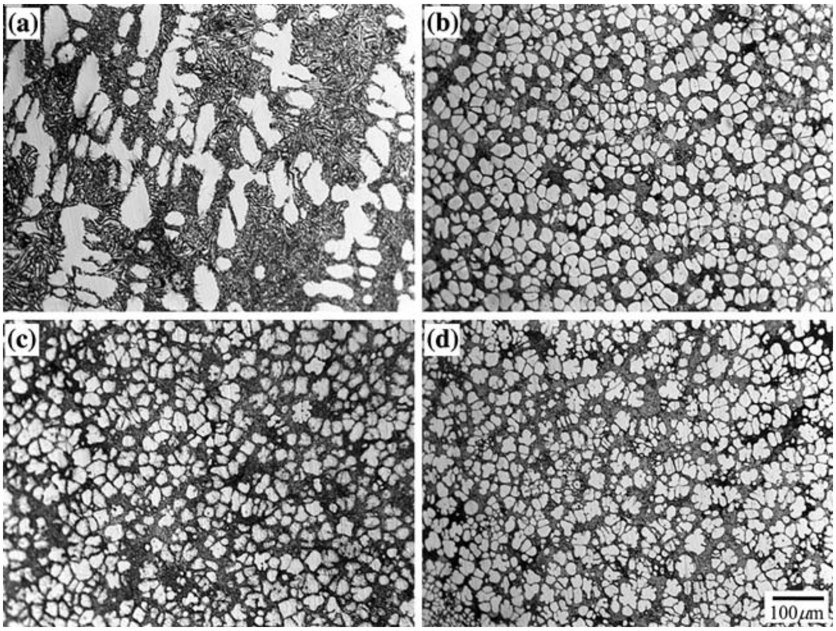

Fig. 7 Optical micrographs of the 14.5%Si cast irons with mischmetal content; (**a**) no addition, (**b**) 0.2%, (**c**) 0.6%, and (**d**) 0.8%

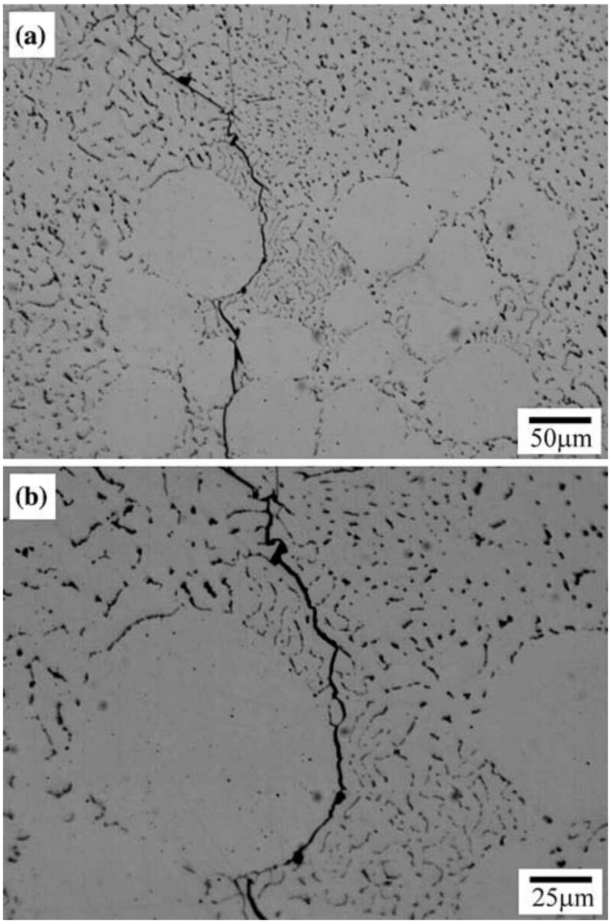

Fig. 8 Optical micrographs showing the behavior of crack propagation; (**a**) a low magnification and (**b**) a high magnification

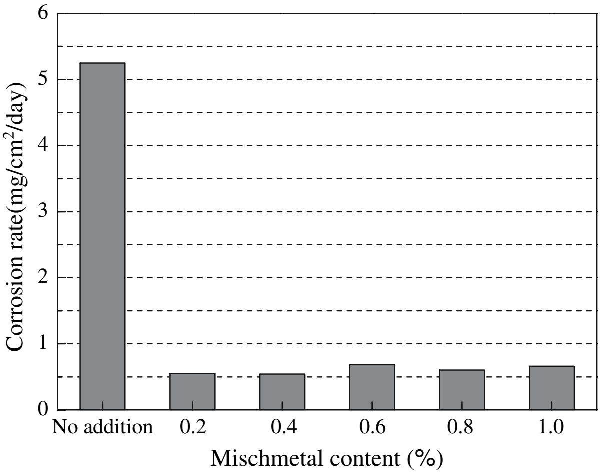

Fig. 9 Variation of corrosion rate with mischmetal content

Conclusions

The relationship between the casting process parameters such as melting temperature, mischmetal addition, and pouring temperature and UTS in the 14.5%Si containing corrosion resistant cast iron was statistically investigated using Taguchi method, and metallurgically confirmed by combined analysis of fractography, hydrogen content determination, microscopic examination of graphite and matrix, and acid resistance test. The obtained results are as follows:

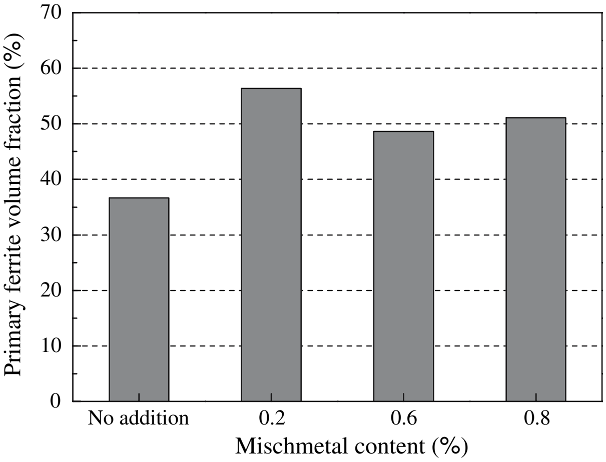

Fig. 10 The volume fraction of primary ferrite phase with the variation of mischmetal content

(1) When the high-silicon cast iron was melted at a temperature of 1,650 °C, followed by mischmetal treatment, and poured at the lowest possible temperature of 1,350 °C, an excellent UTS of around 110–150 MPa, which is beyond the industrial criterion, was able to obtain.

(2) The 0.2% mischmetal addition improved the microstructure of the high-silicon cast iron independently on the other casting process parameters, resulting in the increment of mechanical properties and corrosion resistance.

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