THE SOLUBILITY OF CARBON IN MOLTEN IRON-NICKEL-SILICON ALLOY AT 1773K AND 1823K

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ABSTRACT

In the previous study, the authors have presented a new method to produce 300 series stainless steel using electro-silicothermic process and it might be an innovation for stainless steel and ferronickel industry. In the new process, lateritic nickel ore was used to produce different ferronickel alloys with high silicon content (Si＞5.0 wt%) and was named nickel-silicon ferroalloy. The solubility of carbon in molten iron-nickel-silicon (Fe-Ni-Si) alloy is an important design consideration for producing nickel-silicon ferroalloy. A new experimental apparatus was developed in the present study. The molten metal in equilibrium with a graphite crucible, which was kept by an insulated crucible outside, was quenched in a copper mold in order to obtain the solubility of carbon at experimental temperature as accurate as possible. Solubility of carbon in Fe-Ni-Si alloy with different nickel/iron (Ni/Fe) ratio was measured under the temperature of 1773K and 1823K, respectively. The relationship between the solubility of carbon and silicon content between 0 wt% and 15 wt% in molten Fe-Ni-Si alloy at 1773K could be expressed as a function as follows.

$$Ni/Fe = 0.09,\ [\%Si] = 16.72 - 2.01\times[\%C] - 10.72\times\lg[\%C]$$

$$Ni/Fe = 0.27,\ [\%Si] = 15.36 - 1.65\times[\%C] - 14.03\times\lg[\%C]$$

The results showed that the solubility of carbon decreases with an increase in Ni/Fe ratio at the same silicon content in the molten Fe-Ni-Si alloy. A second solid phase appears at silicon content from around 18 wt% to 2 0wt%. The second solid phase was SiC which can be detected by scanning electron microscope.

KEYWORDS: *iron-nickel-silicon alloy, solubility of carbon, lateritic nickel ore, stainless steel, electro-silicothermic*

1 INTRODUCTION

In the previous study, the authors have presented a new process to produce 300 series stainless steel, and the electro-silicothermic metallurgy method was used in the new process[1]. One of the characteristics of electro-silicothermic metallurgy was that the issue of carbon content of the final product was solved by intermediate product. Hence, in order to obtain the primary stainless steel with low carbon content and reduce the decarburization load of AOD or VOD, it is necessary to control the carbon content of nickel-silicon ferroalloy which was the intermediate product to less than 3.0wt% or 1.5 wt%. Authors have developed the experiment method to research the solubility of carbon in molten Fe-Ni-Si alloy which has different composition under the temperature of 1773K and 1823K, respectively.

In the past years, some investigations of the solubility of carbon in molten Fe-Si, Fe-Ni and Ni-Si alloys have been carried out. In 1950s~1970s Chipman[2][3][4][5]、Elliott[6][7]、Darken[8]、Lupis[9]、Kazuhiro[10] et al[11][12][13] carried out many researches on the effect of other element on the solubility of carbon in molten iron, for example, the respective effect of nickel and silicon element alone. Their works were summarized by Sigworth and Elliot et al.[14] and the data of first-order interaction coefficients was calculated, which was the fundamental data for thermodynamic study of iron molten system. However, Fe-Ni-Si alloy also is the iron-based alloy, there was no study on the combined effect of nickel and silicon on the solubility of carbon in this type of molten iron. The authors would like to show in this paper the effect of Ni/Fe ratio on the solubility of carbon in the molten iron with different silicon content.

2 EXPERIMENTAL

The experiment was carried out by equilibrating Fe-Ni-Si melt sample with graphite. The apparatus used for the experiment is pictured in Fig. 1. A resistance furnace used molybdenum-silicon heating units and the temperature was held constant at 1773±1 K or 1823±1 K with $PtRh_{30}$-$PtRh_6$ thermocouple through an automatic controller. A graphite

crucible with a graphite cover was used for experiment and it was kept by an insulation crucible outside this resistance furnace chamber. The details of crucible assembly were shown in Fig. 2. In order to ensure that the temperature does not decrease during the period of sampling process and to obtain an immediate quench, they were both drawn out of the furnace chamber promptly at the end of experiment and the melt was quenched in copper mold (as shown in Fig.3) quickly. The time taken for the sampling process was within 20s. Materials used for the preparation of Fe-Ni-Si alloy samples were shown in Table 1. The samples with different composition were smelt in an induction furnace with the capacity of 10 kg. About 100 g alloy mixture in the graphite crucible was melted up to 1773 K or 1823 K and maintained at this temperature for 4h by a current of purified Ar in each run of experiment. The duration to establish the attainment of an equilibrium state was about 1h, which confirmed in preliminary tests, and the quenching in copper mold was the best cooling method of the sample. The results of preliminary experiments were shown in Fig. 4 and Table 2. As shown in the Table 2, when the melt was quenched with copper mold, the solubility of graphite in molten iron measured by the apparatus described above was more approximate to the value measured by [2], which is in remarkably good agreement with other researchers. The value was 5.21 wt% in this experiment and it was higher than value of 5.15 wt% from the literature [2].

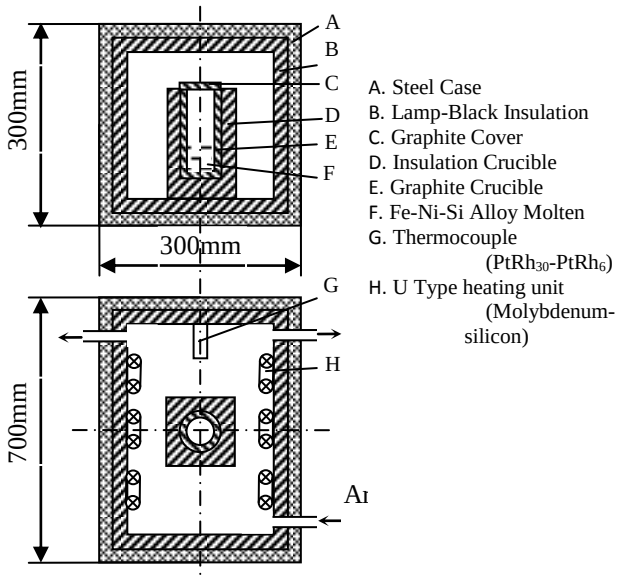


Fig.1. Resistance furnace used for the equilibrium test between Fe-Ni-Si-C melts and graphite

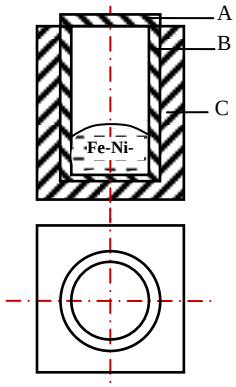


Fig.2. Crucible assembly (A. Graphite cover; B. Graphite crucible; C. Insulation crucible)

Fig. 3. Copper mold used for quenching

Table 1. Raw materials used for Fe-Ni-Si melt

Materials	Composition/wt%											
	Fe	Ni	C	Si	Mn	P	S	Cr	Ni	Cu	Al	Ca
Pure iron	99.90	/	0.02	0.003	0.02	0.0065	0.005	0.01	0.01	0.01	0.01	/
Pure nickel	≤0.30	≥99	≤0.02	≤0.02	≤0.35	/	≤0.01	≤0.2	/	≤0.25	/	/
Silicon	0.5	/		≥99	/	0.16	/	/	/	/	0.5	0.3

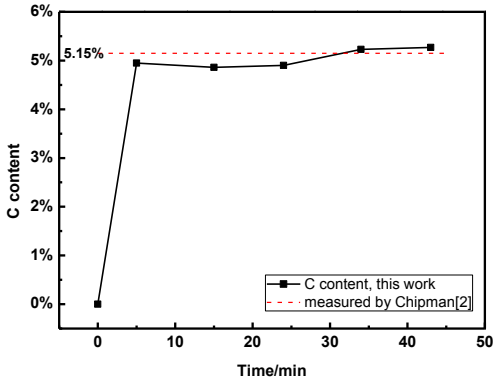


Fig. 4. The dissolved carbon content in iron molten changes with equilibrium test time at 1773K

Table 2. Comparison of solubility of graphite in molten iron at 1773K with different cooling method

Sample cooling	Carbon content, (wt%)	Error
Quenched in the copper mold	5.15[2]	-
Quenched in the copper mold	5.21	1.17%
Sampling by quartz tube and then quenched in water	4.56	-11.46%
Melt with graphite crucible quenched in water	5.37	40.70%

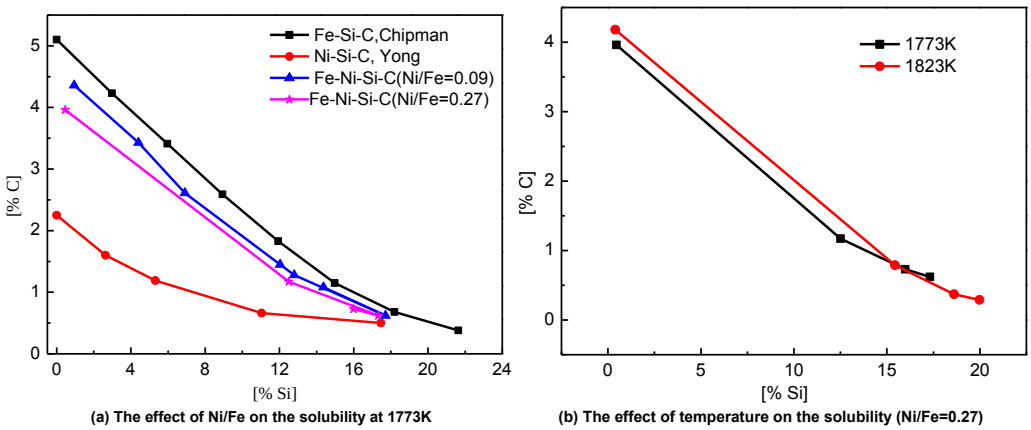
3 RESULTS

The results of fifteen experiment runs are shown in Table 3 and Fig.4. As shown in Fig.4, for each set of solubility of graphite in molten Fe-Ni-Si alloy, the solubility was decreased with the increase in silicon content, where [% C] and [% Si] was 100 times of the mass fraction of carbon and silicon, respectively. The solubility was also decreased by increasing the Ni/Fe ratio when the silicon content was at the same value. The solubility of carbon in molten Fe-Si alloy was measured by Chipman[2] and the molten Fe-Si can be seen as a special system of Fe-Ni-Si with Ni/Fe=0. Similarly, Ni-Si alloy[15] can be seen as another special system of Fe-Ni-Si with Ni/Fe=+ ∞. So that the value of the solubility of carbon in molten Fe-Ni-Si alloy was between that in molten Fe-Si alloy and that in molten Ni-Si alloy when they had the same silicon content. Additionally, the carbon content in the Ni-Si ferroalloy produced by lateritic nickel ores in the new process will be between the value of solubility of carbon in molten Fe-Ni-Si alloy (Ni/Fe=0.09) and that in molten Fe-Ni-Si alloy (Ni/Fe=0.27) because the Ni/Fe ratio in lateritic nickel ores was between 0.09 and 0.27. Hence, to ensure the carbon content of the primary stainless steel in the electro-silicothermic process was less than 3.0 wt% or 1.5 wt%, the silicon content of Ni-Si ferroalloy should be higher than 7 wt% or 12 wt%. However, the solubility of graphite in molten Fe-Ni-Si alloy was increased by increasing the temperature. When the temperature increased to about 50 K, the carbon content increases to about 8%. The smelt temperature could be controlled and kept at a 1773±100 K generally. In order to assure that the carbon content in the Ni-Si ferroalloy was less than 1.5 wt% and satisfied with the requirement of carbon content required in primary stainless steel production, the silicon content of Ni-Si ferroalloy might be

higher than 15 wt%.

Table 3. Chemical analysis of Fe-Ni-Si-C alloy samples

Run No	Temp.(K)	%Fe	%Ni	%Si	%C	Ni/Fe	Run No	Temp.(K)	%Fe	%Ni	%Si	%C	Ni/Fe
1	1773	86.55	8.03	0.94	4.36	0.09	9	1773	68.27	17.92	12.51	1.17	0.26
2	1773	83.46	8.00	4.41	3.43	0.10	10	1773	65.43	17.66	15.99	0.73	0.27
3	1773	80.87	9.30	6.91	2.61	0.11	11	1773	64.60	17.31	17.32	0.62	0.27
4	1773	78.97	7.24	12.04	1.45	0.09	12	1823	74.04	20.69	0.40	4.18	0.28
5	1773	78.51	7.12	12.79	1.28	0.09	13	1823	65.99	17.64	15.44	0.79	0.27
6	1773	77.38	7.00	14.37	1.08	0.09	14	1823	63.90	16.66	18.61	0.37	0.26
7	1773	74.71	6.70	17.71	0.62	0.09	15	1823	63.21	16.16	19.98	0.29	0.26
8	1773	74.35	20.8	0.46	3.96	0.28							



(a) The effect of Ni/Fe on the solubility at 1773K (b) The effect of temperature on the solubility (Ni/Fe=0.27)

Fig. 4. The relationship between silicon content and carbon content in the molten Fe-Ni-Si alloy

A very interesting observation was made during run No.7, No 11 and No 15. The charge contained 22 wt% of silicon but the samples contained only 17.71 wt% to 20 wt%. These three samples presented the limiting silicon concentration which can remain in stable equilibrium with graphite. Analogous behaviours were observed in the Fe-Si-C equilibrium series[2]. Chipman considered that it was because during the second phase it dissolved out from the alloy and he thought it was β-SiC but did not provide the direct proof. Some analysis of the precipitations dissolved out from the alloy in the graphite crucible was carried out and shown in the Fig.5 and Fig.6. The second phase in the equilibrium experiment was confirmed as the SiC.

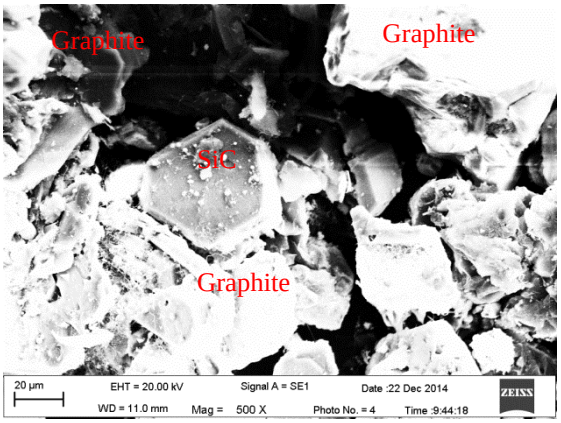


Fig. 5. SEM analysis of educt in the run No.7 of the experiment

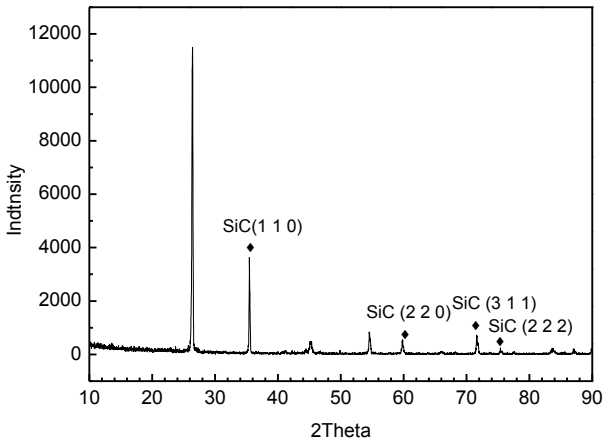


Fig. 6. XRD analysis of educt in the run No.7 of the experiment

4 DISCUSSION

The solubility of solute refers to the amount of solute in the solvent when the solid solute is equilibrating with the solvent at a certain temperature, so the solubility of solute in molten alloy is always expressed as weight percent. According to the difference of the solid phase during which it dissolved out from the molten alloy, the solubility of carbon can be divided into the solubility of graphite and the solubility of carbide. The investigation of the authors and Chipman[2][16] showed that there were two solid phases of graphite and SiC dissolved out from the molten Fe-Ni-Si-C alloy when it was equilibrating with graphite. So the solubility of carbon in the molten Fe-Ni-Si-C alloy can be divided into the solubility of graphite and the solubility of silicon carbide. The carbon content measured in this paper was the solubility of graphite in the molten Fe-Ni-Si-C alloy. Therefore, the chemical equilibrium reaction of these experiments can be expressed as eq.(1)

$$C_{Graphite} = [C]_{sat.,\%} \tag{1}$$

The activity of carbon in the molten alloy can be calculated by eq.(2) when using a weight percent composition.

$$a_C = f_C[\%C] = K^{\circ}_{sol,\%} \tag{2}$$

Where:

$K^{\circ}_{sol,\%}$——Equilibrium constant of reaction (1)；

[%C]——100 times of the mass fracation of C in molten;

f_C——Activity coefficient of carbon;

a_C——Activity of carbon.

Then the activity coefficient of carbon in a quaternary Fe-Ni-Si-C can be expressed in accordance with ε-formalism[14] as eq.(3)

$$\begin{aligned} \lg f_C = {} & e_C^C[\%C] + e_C^{Si}[\%Si] + e_C^{Ni}[\%Ni] + \gamma_C^C[\%C]^2 + \gamma_C^{Si}[\%Si]^2 \\ & + \gamma_C^{Ni}[\%Ni]^2 + \gamma_C^{C,Si}[\%Si][\%C] + \gamma_C^{C,Ni}[\%Ni][\%C] + \gamma_C^{Si,Ni}[\%Si][\%Ni] \end{aligned} \tag{3}$$

Where:

e_i^j——first order interation coefficient, i=C, Si, Ni, j=C,Si, Ni；

γ_i^j, $\gamma_i^{j,k}$——second order interation coefficient, k=C, Si, Ni；

Take the logarithm on both sides of eq.(2) and substitute it with eq.(3). Then the relationship between the compositions of every element in molten Fe-Ni-Si-C alloy can be expressed as eq.(4)

$$\begin{aligned} \lg[\%C] = {} & \lg K^{\circ}_{sol} - e_C^C[\%C] - e_C^{Si}[\%Si] - e_C^{Ni}[\%Ni] - \gamma_C^C[\%C]^2 - \gamma_C^{Si}[\%Si]^2 \\ & - \gamma_C^{Ni}[\%Ni]^2 - \gamma_C^{C,Si}[\%Si][\%C] - \gamma_C^{C,Ni}[\%Ni][\%C] - \gamma_C^{Si,Ni}[\%Si][\%Ni] \end{aligned} \tag{4}$$

Actually, the second-order terms can be ignored because the effect of second-order interaction coefficient on the activity coefficient of carbon in this system was much lower than the effect of first-order interaction coefficient even though the nickel content was in the range of 8 wt%~20 wt% and the silicon content was in 0~20 wt%. The interaction coefficient reported in other papers is shown in Table 4 and the effect of first-order interaction coefficient and second-order interaction coefficient on the activity coefficient of carbon calculated with the data presented in Table 4 and Table 3 is shown in the Table 5.

Table 4. The interaction coefficient between Ni、Si、C and C

e_C^{Ni}	γ_C^{Ni}	$\gamma_C^{Ni,C}$	Temp./K	Ref.
0.010*	1.5×10^{-5}	2.9×10^{-4}	1823	[17]
e_C^{Si}	γ_C^{Si}	$\gamma_C^{Si,C}$	Temp./K	Ref.
0.08	7.9×10^{-4}	6.2×10^{-3}	1773	[14][18]
e_C^{C}	γ_C^{C}		Temp./K	Ref.
0.15	7.6×10^{-3}		1773	[14][18]

Table 5. The effect of first-order and second order terms on the activity coefficient of carbon in molten Fe-Ni-Si-C alloy

Factors		Calculation values			
		Ni/Fe=0.09		Ni/Fe=0.27	
		Min.	Max.	Min.	Max.
First-order terms	e_C^{C}[%C]	0.0868	0.6678	0.0868	0.5572
	e_C^{Si}[%Si]	0.0752	1.4168	0.0368	1.3856
	e_C^{Ni}[%Ni]	0.0670	0.0930	0.1731	0.2080
Second-order terms	γ_C^{C}[%C]^2	0.0029	0.1729	0.1204	0.0029
	γ_C^{Si}[%Si]^2	0.0007	0.2478	0.0002	0.2370
	γ_C^{Ni}[%Ni]^2	0.0007	0.0013	0.0045	0.0065
	$\gamma_C^{C,Si}$[%Si][%C]	0.0254	0.1118	0.0113	0.0907
	$\gamma_C^{C,Ni}$[%Ni][%C]	0.0012	0.0027	0.0031	0.0239

Hence, the eq.(4) can be simplified as eq.(5).

$$\lg[\%C] = \lg K_{sol}^{\circ} - e_C^{C}[\%C] - e_C^{Si}[\%Si] - e_C^{Ni}[\%Ni] \quad (5)$$

Moreover, in the quaternary system Fe-Ni-Si-C, the Ni content was related to the content of silicon and carbon so that the Ni content in the Fe-Ni-Si-C alloy can be calculated by the eq.(6)

$$[\%Ni] = \frac{100 - [\%Si] - [\%C]}{1 + \frac{1}{N/F}} \quad (6)$$

Where, N/F was refers to Ni/Fe ratio in melt of Fe-Ni-Si-C alloy.

Then substitute the eq.(6) with the eq.(5) and eq.(7) will be obtained.

$$\lg[\%C] = \lg K_{sol}^{\circ} - e_C^{C}[\%C] - e_C^{Si}[\%Si] - e_C^{Ni}\left(\frac{100 - [\%Si] - [\%C]}{1 + \frac{1}{N/F}}\right)$$

$$= \left(\lg K_{sol}^{\circ} - \frac{100 e_C^{Ni}}{1 + \frac{1}{N/F}}\right) + \left(\frac{e_C^{Ni}}{1 + \frac{1}{N/F}} - e_C^{C}\right)[\%C] + \left(\frac{e_C^{Ni}}{1 + \frac{1}{N/F}} - e_C^{Si}\right)[\%Si] \quad (7)$$

$$\Rightarrow [\%Si] = \frac{1}{\left(\frac{e_C^{Ni}}{1 + \frac{1}{N/F}} - e_C^{Si}\right)}\left[\lg[\%C] - \left(\frac{e_C^{Ni}}{1 + \frac{1}{N/F}} - e_C^{C}\right)[\%C] - \left(\lg K_{sol}^{\circ} - \frac{100 e_C^{Ni}}{1 + \frac{1}{N/F}}\right)\right]$$

Assuming the N/F was a constant, the relationship between silicon content and solubility of graphite in the molten Fe-Ni-Si-C alloy can be expressed as eq.(8).

$$[\%Si] = a + b \times [\%C] + c \times \lg[\%C] \quad (8)$$

Where a, b, c were both constant which were only influenced by the Ni/Fe and temperature, and their representative was shown in eq.(9), eq.(10) and eq.(11), respectively.

$$a = \frac{1}{(\frac{e_C^{Ni}}{1+\frac{1}{N/F}} - e_C^{Si})} (\frac{100e_C^{Ni}}{1+\frac{1}{N/F}} - \lg K_{sol}^{\circ}) \quad (9)$$

$$b = \frac{1}{(\frac{e_C^{Ni}}{1+\frac{1}{N/F}} - e_C^{Si})} (e_C^{C} - \frac{e_C^{Ni}}{1+\frac{1}{N/F}}) \quad (10)$$

$$c = \frac{1}{(\frac{e_C^{Ni}}{1+\frac{1}{N/F}} - e_C^{Si})} \quad (11)$$

Thus, according for the data shown in Table 3, the statistically obtained relationship equations at 1773K or 1823K are shown in eq.(12), eq.(13) and eq.(14), respectively.

Fe-Ni-Si-C(Ni/Fe=0.09), 1773K:

$$[\%Si] = 16.72 - 2.01[\%C] - 10.72\lg[\%C], R^2 = 0.999 \quad (12)$$

Fe-Ni-Si-C(Ni/Fe=0.27), 1773K:

$$[\%Si] = 15.36 - 1.65[\%C] - 14.03\lg[\%C], \ R^2 = 0.999 \quad (13)$$

Fe-Ni-Si-C(Ni/Fe=0.27), 1823K:

$$[\%Si] = 17.02 - 2.97[\%C] - 6.78\lg[\%C], \ R^2 = 0.999 \quad (14)$$

In fact, the requirement of silicon content for Ni-Si ferroalloy to satisfy the stainless steel production can be calculated by eq.(12) and eq.(13) and the result are shown in the Table 6. The calculated result was well agreed with the experimental result shown in Table 3. Actually, the carbon in the primary stainless steel was less, the load of AOD or VOD was less. It was generally required to be less than 3.0 wt% or1.5 wt% in the traditional production process and only sometimes was required to be less than 1.0 wt% to produce ultra-carbon stainless steel. As shown in the Table 6, the carbon content can be controlled at a level of less than 1.0 wt% when the silicon content in Ni-Si ferroalloy was higher than 15 wt%. It was emphasized that even when the smelt temperature was increased 50 K or 100 K, the carbon content in Ni-Si ferroalloy will maintain less than 1.5 wt% according to the eq.(14).

Table 6. Requirement of silicon content for Ni-Si ferroalloy to satisfy stainless steel production

Requirement for carbon content	Silicon content	
	Ni/Fe=0.09	Ni/Fe=0.27
<3.0%	5.6	3.7
<1.5%	11.8	10.4
<1.0%	14.7	13.7

5 SUMMARY

1) The solubility of carbon was decreased with the increase of Ni/Fe at the same silicon content in the molten Fe-Ni-Si alloy and the relationship between the solubility of graphite and silicon content between 0 wt% and 17 wt% in molten Fe-Ni-Si alloy at 1773K could be expressed as a function as follows.

$$Ni/Fe = 0.09, [\%Si] = 16.72 - 2.01 \times [\%C] - 10.72 \times \lg[\%C]$$

$$Ni/Fe = 0.27, [\%Si] = 15.36 - 1.65 \times [\%C] - 14.03 \times \lg[\%C]$$

2) A second solid phase appeared in the Fe-Ni-Si-C alloy at silicon content from around 18 wt% to 20 wt% and the second solid phase was SiC.

3) The silicon content of Ni-Si ferroalloy might be higher than 15 wt% and satisfied the requirement of carbon content for the new method to produce 300 series stainless steel production.

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