Available online at www.sciencedirect.com

ScienceDirect

JOURNAL OF IRON AND STEEL RESEARCH, INTERNATIONAL. 2010, 17(4): 45-52

Spheroidizing Kinetics and Optimization of Heat Treatment Parameters in CK60 Steel Using Taguchi Robust Design

Ata Kamyabi-Gol[1], Meisam Sheikh-Amiri[2]

(1. Department of Chemical and Materials Engineering, University of Alberta, Edmonton T6G 2G6, Canada;
2. Department of Materials Science and Metallurgical Engineering, Ferdowsi University of Mashhad, Mashhad 9177948944, Iran)

Abstract: For processing parts made from medium carbon steel, toughness and flexibility are of importance. Therefore, to achieve these properties, the cementite in the steels is spheroidized through heat treatment. Different parameters such as the time and temperature of spheroidizing and the initial microstructure of the steel affect the amount of spheroidized cementite. In the present work, the percent of contribution of two parameters, i. e. initial microstructure and spheroidizing time, to the percent of spheroidization in CK60 steel was investigated using Taguchi robust design. The initial microstructures consisted of martensite, coarse pearlite, fine pearlite and bainite and the chosen spheroidization times were 4, 8, 12, and 16 h. Spheroidizing was done at the constant temperature 700 ℃. After spheroidizing was completed, the samples were prepared in order to observe their microstructure under an optical microscope and to determine the spheroidized percent using MIP™ (metallographic image processing) software. It was found that the spheroidizing time had the most influence (58.5%) on spheroidized percent and the initial microstructure only had a 31.1% contribution. Finally, the instantaneous growth rate of the carbide was also deduced.

Key words: spheroidizing; kinetics; heat treatment; CK60; Taguchi

One of the most widely used methods to improve the properties and workability of metal parts is heat treatment. In fact, by applying certain heat treatments, different properties could be achieved in the same steel. One of the heat treatments applied to steel is spheroidizing. The presence of cementite spheres in a ferrite matrix forms the softest and most workable microstructure in hypereutectoid and tool steels. The majority of all spheroidizing activity is performed for improving the cold formability of steels. It is also performed to improve the machinability of hypereutectoid steels, as well as tool steels. A spheroidized microstructure is desirable for cold forming because it lowers the flow stress of the material. The flow stress is determined by the proportion and distribution of ferrite and carbides. The strength of the ferrite depends on its grain size and the rate of cooling. Whether the carbides are present as lamellae in pearlite or spheroids radically affects the formability of steel. Steels may be spheroidized, that is, heated and cooled to produce a structure of globular carbides in a ferritic matrix[1].

Spheroidization can take place by the following methods[2]:

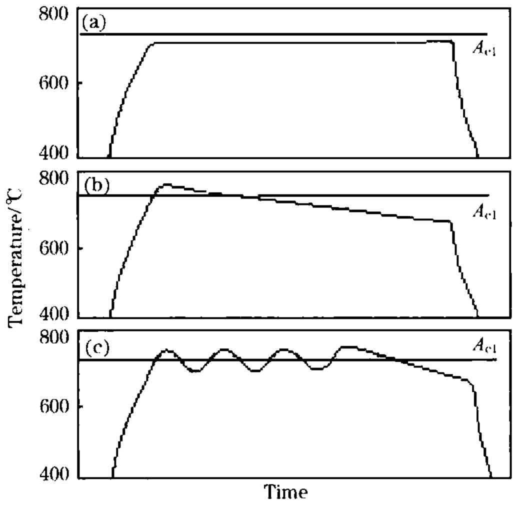
1) Prolonged holding at a temperature just below A_{c1}.

2) Heating to a temperature just above A_{c1}, and then either cooling very slowly in the furnace or holding at a temperature just below A_{c1}.

3) Heating and cooling alternately between temperatures that are just above and below A_{c1}.

Fig. 1 shows the possible temperature-time regimes mentioned above. The swinging regime [Fig. 1 (c)] is used to accelerate the transformation of cementite lamellae to globular form. Increasing the temperature above A_{c1} facilitates the dissolution of cementite lamellae. At subsequent cooling below A_{c1} this dissolution process is interrupted and the parts broken off (which has less resistance to boundary surface energy) coagulate more easily and quickly[2].

Biography: Ata Kamyabi-Gol(1984—), Male, Doctor Candidate; **E-mail**: kamyabig@ualberta.ca; **Received Date**: July 28, 2008



(a) Annealing at 20 ℃ below A_{c1}, for unalloyed steels and for alloyed steels with bainitic or martensitic starting structure;
(b) Annealing at 10 ℃ above A_{c1} (start) and decreasing temperature to 30 ℃ below A_{c1} for alloyed steels;
(c) Swinging annealing ±5 ℃ around A_{c1} for hypereutectoid steels.

Fig. 1 Temperature-time regimes for spheroidizing

Spheroidizing cycles are usually time consuming and hence expensive. Therefore, to achieve better mechanical properties and decrease the cycle time, unstable initial microstructures such as martensite and bainite are used. According to Ref. [1], the shortest spheroidizing time and, at the same time, the most evenly dispersed spheres in the matrix are achieved from the most unstable initial microstructure (mainly martensite) of the steel being processed. The spheroidizing rate in the unstable initial microstructures depends on the diffusion rate of carbon atoms in the microstructure and their coagulation rate.

If the starting structure is pearlite, the spheroidization of carbides takes place by the coagulation of the cementite lamellae. This process can be formally divided into two stages. At first the cementite lamellae assume a knucklebone shape, as shown in Fig. 2. As annealing continues, the lamellae form globules at their ends and, by means of boundary surface energy, split up into spheroids, hence the name spheroidizing. During the second stage, some cementite (carbide) globules grow at the cost of fine carbide particles, which disappear. In both stages, the rate of this process is controlled by diffusion. The thicker the cementite lamellae, the more energy necessary for this process[2].

Spheroidized percent, the number of spheres in a specific area, and the size of the spheres all play a major role in the mechanical properties of the finished part. In general, smaller and more evenly dispersed spheres in the matrix result in better mechanical properties.

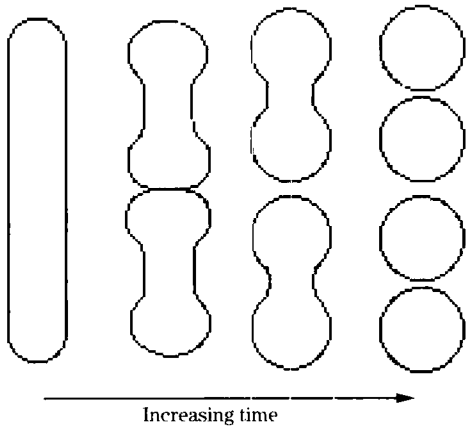


Fig. 2 Schematic presentation of the process of transforming cementite lamella to spheroids during spheroidizing

In order to identify the percent of contribution of the different factors affecting spheroidized percent in this research, Taguchi robust design method was used. Taguchi method is a combination of mathematical and statistical techniques used in an empirical study. It can determine the experimental condition having the least variability as the optimum condition. The variability can be expressed by signal to noise (S/N) ratio[3-6]. The experimental condition having the maximum S/N ratio is considered as the optimal condition. According to literature survey done by the authors there is a literature gap in using Taguchi robust design method on the optimization of spheroidizing heat treatment. Thus the objective of this work is to apply the Taguchi robust design method on the optimization of spheroidizing and to predict the maximum spheroidized cementite percent of the samples.

1 Experimental Details

1.1 Main parameters and their levels

The main parameters affecting the percent of spheroidized cementite in steel are the cycle time, temperature, and the initial microstructure of the steel.

Due to the fact that the temperature range in spheroidizing is relatively closed, this parameter was kept constant and therefore the effect of the two remaining factors on the percent of spheroidized cementite was investigated. The chosen spheroidizing time periods were 4, 8, 12, and 16 h and the initial microstructure of the samples were martensite, bainite, fine pearlite, and coarse pearlite. Table 1 summarizes the parameters and levels used in this research.

1.2 Material and heat treatment

Standard CK60 (1060) steel was used in this research. Two types of specimens were prepared for each experiment. In order to measure the hardness of the samples, disk shaped specimens with diameter of 2.5 cm and height of 1.5 cm were used. The tensile properties of the samples were evaluated by using 6 mm round tension test specimen described in ASTM E8M.

The initial microstructures were obtained by the following heat treatment cycles[5]:

1) Martensitic microstructure: austenitizing at 820 ℃ and then quenching in room temperature water.

2) Bainitic microstructure: austenitizing at 820 ℃, quenching in a salt bath at 315 ℃, holding for 90 min at 315 ℃, cooling in air.

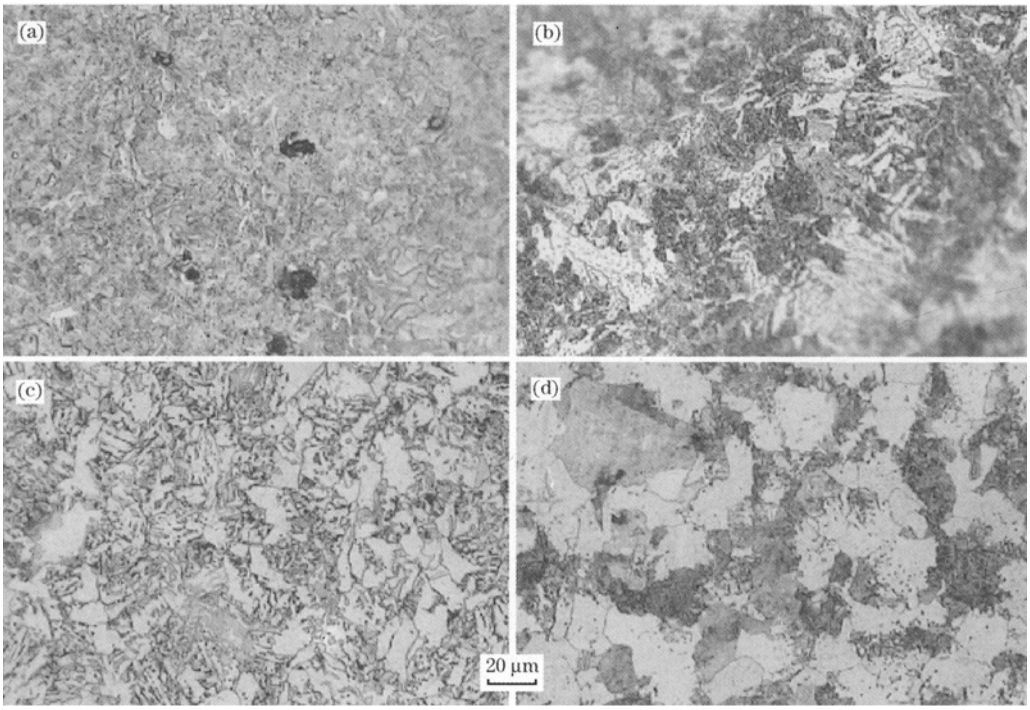
3) Fine pearlitic microstructure: austenitizing at 885 ℃, cooling in non-turbulent air, reaustenitizing at 860 ℃, and cooling in non-turbulent air.

4) Coarse pearlitic microstructure: austenitizing at 885 ℃, cooling to 700 ℃, holding for 20 min at 700 ℃, and finally cooling in air.

After obtaining the initial microstructures, one of each was kept as a reference sample (i. e. one sample of each microstructure with 0 h of spheroidizing). The initial microstructures are shown in Fig. 3. Afterwards, one sample of each microstructure was spheroidized at 700 ℃ for 4 or 8 or 12 or 16 h. Fig. 4 shows the microstructure of the samples after 16 h of spheroidizing.

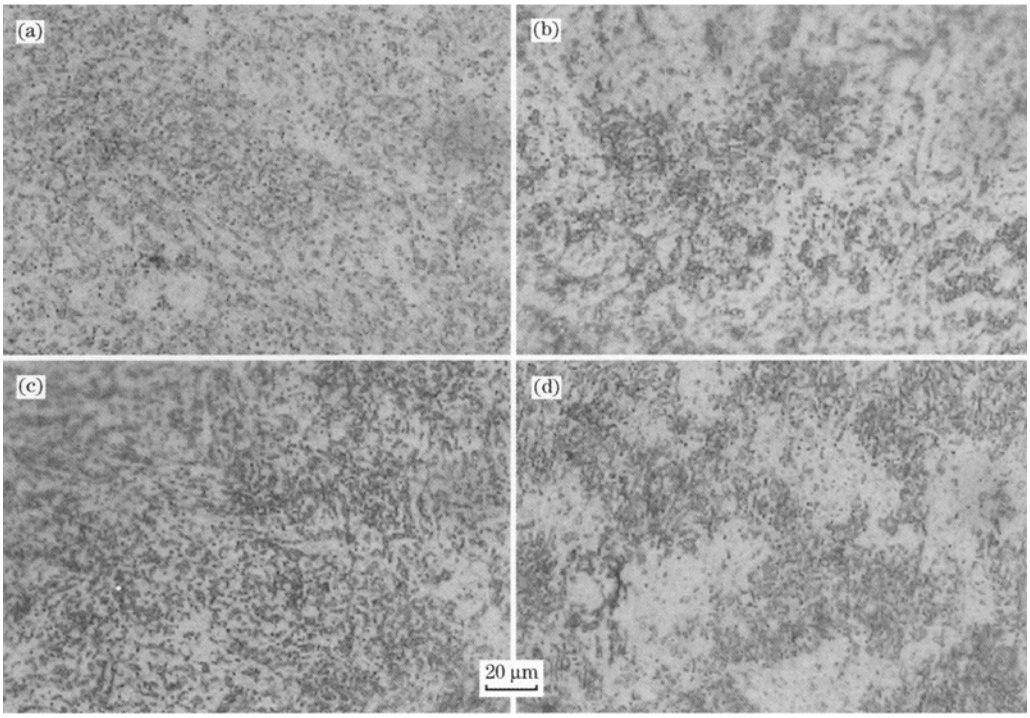
Table 1 Main controlling factors and their levels

Factors	Levels			
	1	2	3	4
Initial microstructure	Martensite	Coarse pearlite	Fine pearlite	Bainite
Spheroidizing time/h	4	8	12	16



(a) Martensite; (b) Bainite; (c) Fine pearlite; (d) Coarse pearlite.

Fig. 3 Metallographic photographs of the initial microstructures, specimens etched in Nital



(a) Sample with martensite as the initial microstructure; (b) Sample with bainite as the initial microstructure;
(c) Sample with fine pearlite as the initial microstructure; (d) Sample with coarse pearlite as the initial microstructure.

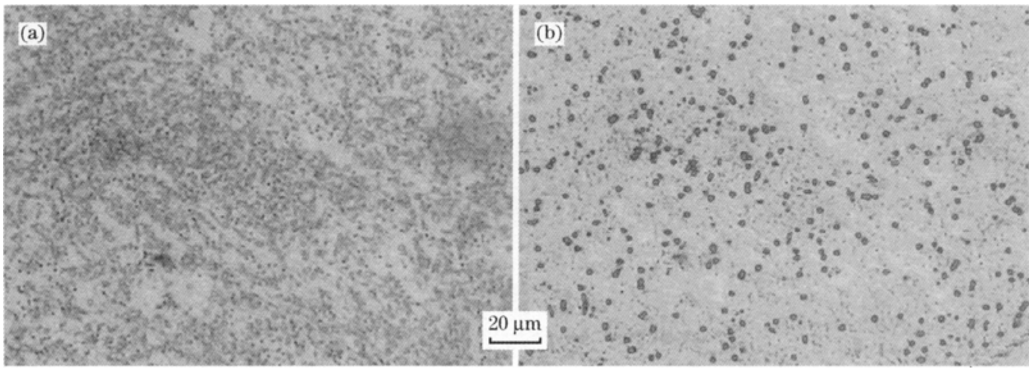
Fig. 4 Metallographic images of samples spheroidized at 700 ℃ for 16 h, etched in Nital

2 Results

2.1 Calculation of spheroidized cementite

After finishing the spheroidizing process, all specimens were polished, etched in Nital and photographed at different magnifications. In order to calculate the percent of spheroidized cementite in the samples, an image processing software entitled "MIP™" (metallographic image processing) was employed. First the percent of cementite in CK60 steel was calculated from the Fe-C phase diagram and then by utilizing the MIP software, the percent of spheroidized cementite was accurately calculated in all of the samples. In this regard, the cementite spheres were first colored green, the image turned into black and white (for better contrast) and finally analyzed via the MIP software. Fig. 5 displays a sample of the primary and modified images used in calculating the cementite percent.

The ratio of spheroidized cementite after sphe-



(a) A normal picture of the sample's microstructure; (b) The same picture with the cementite spheres highlighted (black and white contrast).

Fig. 5 Demonstration of a sample process used to calculate the spheroidized cementite percent

roidizing to initial cementite is defined as the percent of spheroidized cementite. Fig. 6 shows the percent of spheroidized cementite for all the samples.

2.2 Taguchi experiment design

The design of experiments' is an approach for systematically varying the controllable input factors and observing the effects of these factors on the output product parameters. The assumption used in this study was that the individual effects of the input factors on the output product parameters are separable. That means the effect of an independent variable on the performance parameter does not depend on the different level settings of any other independent variable.

The initial microstructure and spheroidizing time where chosen as two independent variables affecting the spheroidized cementite percent and for simplicity named "A" and "B", respectively. According to the Taguchi method, the standard orthogonal array, namely L16 was used. The structure of Taguchi's orthogonal robust design and the results of measurement are shown in Table 2.

The S/N ratios are differently according to the type of characteristic and are classified into three groups[3]. In this study, the S/N ratio was chosen according to the criterion "biggest is best", in order to maximize the spheroidized cementite percent. The corresponding equation for "biggest is best" is displayed below[3]:

$$S/N_B = -10\log_{10}\left[\frac{1}{n}\Sigma\frac{1}{y_i^2}\right] \quad (1)$$

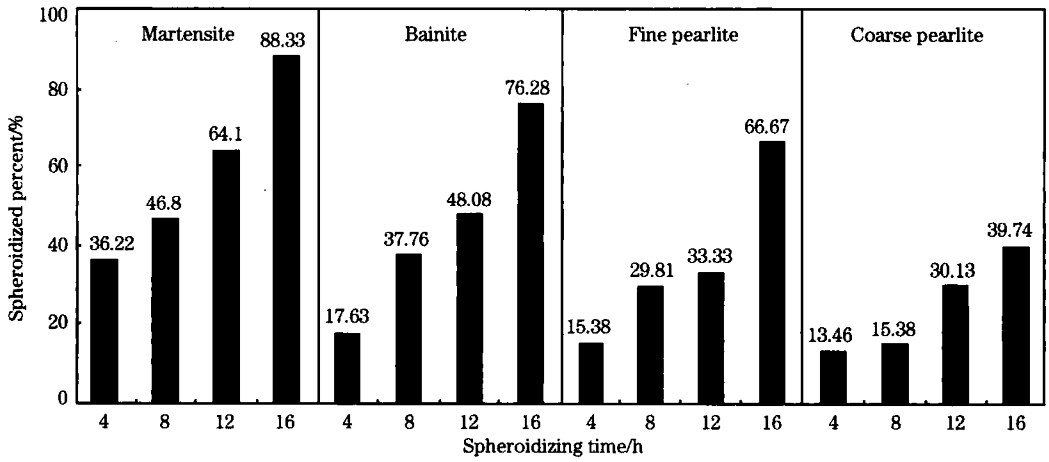


Fig. 6 Spheroidized percent *vs* spheroidizing time for different primary microstructures

Table 2 Taguchi orthogonal array $L_{16}(4^2)$ and the measured values and S/N ratio for mean spheroidized percent

Experiment No.	Initial microstructure (A)	Spheroidizing time/h (B)	Mean spheroidized percent/%	S/N
1	1	1	36.132	−8.846
2	1	2	46.432	−6.669
3	1	3	64.316	−3.912
4	1	4	87.991	−1.155
5	2	1	13.419	−17.560
6	2	2	15.278	−16.461
7	2	3	30.021	−10.563
8	2	4	38.889	−8.291
9	3	1	15.491	−16.357
10	3	2	37.778	−8.455
11	3	3	48.184	−6.353
12	3	4	66.838	−3.508
13	4	1	17.415	−15.392
14	4	2	29.744	−10.549
15	4	3	32.372	−9.878
16	4	4	74.145	−2.789

where y_i is the characteristic property, which is "spheroidized cementite percent" in this case and n is the number of measurements in each experiment. Table 2 shows the S/N ratio for spheroidized cementite percent calculated by the above equation. The average S/N ratio for each level of the parameters is summarized and the S/N response table for spheroidized cementite percent is shown in Table 3.

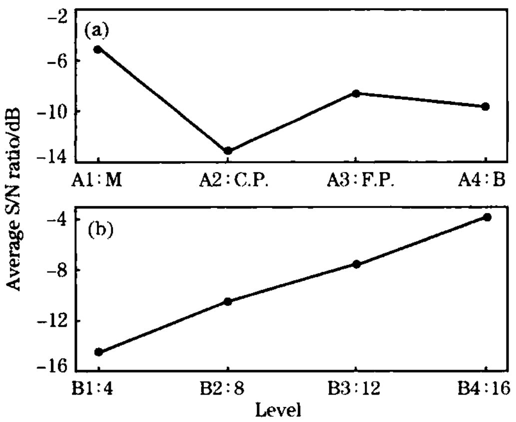
As can be seen in Table 3, the data of (maximum-minimum) of symbol B is the highest value. Therefore, it can be found that spheroidizing time is the significant parameter for affecting spheroidized cementite percent. Fig. 7 shows the average S/N ratio graph for spheroidized cementite percent. It can be noticed from this figure that the optimum levels for each of the parameters are A1 and B4. In other words, based on the S/N ratio, the optimal conditions for increasing spheroidized cementite percent are the A at level 1 and B at level 4.

2.3 Analysis of variance

The purpose of the ANOVA is to investigate which process parameters significantly affect the spheroidized cementite percent of the specimens. This is accomplished by separating the total variability of the S/N ratios, which is measured by the sum of the squared deviations (S_S) from the total mean of the S/N ratio (S_m), into contributions by each process parameter and the error. The equations for calculating the variance for the spheroidized cementite percent can be found in Ref. [3] and have been revised for the current work.

Table 3 Average S/N ratio for each level of the parameters

Factor	Average S/N ratio			
	Level 1	Level 2	Level 3	Level 4
Initial microstructure (A)	−5.146	−13.219	−8.669	−9.652
Spheroidizing time/h (B)	−14.539	−10.534	−7.677	−3.936



M—Martensite; C. P. —Coarse pearlite;
F. P. —Fine pearlite; B—Bainite.
(a) Initial microstructure; (b) Spheroidizing time.

Fig. 7 Average S/N ratio for different levels of the parameters

The analysis of variance (ANOVA) for S/N ratio of the spheroidized cementite percent is shown in Table 4.

As Table 4 shows, the spheroidizing time of the specimens has the largest effect on increasing the spheroidized cementite (58.45%). The initial microstructure of the specimens only has a 31.11% contribution percent which is almost half of the contribution of the spheroidizing time.

2.4 Kinetics of spheroidizing (growth) of carbide

During heat treatment the carbide in the CK60 steel changes into a spherical state. The thermodynamics driving force comes from the decrease of free energy between present carbide and matrix interface. The kinetics driving force is the diffusion of the alloy element on the carbide and matrix interface caused by

Table 4 ANOVA for S/N ratio of the mean spheroidized percent

Source	DF	SS	MS	F	ρ/%
Initial microstructure	3	132.287	44.096	15.894	31.108
Spheroidizing time/h	3	241.240	80.413	28.984	58.449
Error	9	24.969	2.774		10.443
Total	15	398.497			100
$F_{3,9}(0.99)=6.99$					

Note: DF—Degree of freedom; SS—Sum of squares; MS—Mean sum of squares;
F—Variance ratio.

non-homogeneous distribution. Because the size of the carbides is different and the radius of curvature is different from one to another, therefore, the contents of alloy element are non-uniform on the interface of carbide and matrix.

For simplicity let us assume the carbide as a sphere having radius r. When heated, the alloy element will migrate from the carbide tip point to the surface. Because the surface diffusion coefficient of alloy element is much higher than the body diffusion coefficient, here assume that the diffusion of element is only along the interface of carbide and matrix, and then the diffusion fluxes of alloy element from the tip point of carbide to the surface of carbide will be given by[6]:

$$J_1 = -\frac{D4\pi r^2}{\Omega}\frac{\mathrm{d}C}{\mathrm{d}x} \tag{2}$$

where, D is the diffusion coefficient of alloy element; r is the radius of the sphere after coarsening; x is the distance from the tip point of carbide; Ω is the mole volume. The volume of the sphere, that is $V=4\pi r^3/3$, will increase with the increase of radius r. The mole number of the alloy element contained by the circular carbide is $n=4\pi r^3/3\Omega$. Then the rate of obtaining solute of the carbide circular surface will be given by:

$$J_2 = \frac{\partial n}{\partial t} = \frac{4\pi r^2}{\Omega}\frac{\partial r}{\partial t} \tag{3}$$

Suppose that all of the solutes at the tip point of carbide are deposited onto the carbide circular surface, then $J_1 = J_2$, from Eqn. (2) and Eqn. (3) the following equation is obtained.

$$J_1 = J_2 \Rightarrow -D\frac{\mathrm{d}C}{\partial r/\partial t} = \mathrm{d}x \tag{4}$$

Integrate Eqn. (4):

$$\int_{C(r_1)}^{C(r_2)} -D\frac{\mathrm{d}C}{\partial r/\partial t} = \int_{r_1}^{r_2} \mathrm{d}x \tag{5}$$

where $C(r_1)$ and $C(r_2)$ are the concentrations at radii r_1 and r_2, respectively. Assuming that the instantaneous growing rate $\partial r/\partial t$ of carbide is constant, integrating the above expression gives the instantaneous growth rate of the carbide:

$$\frac{\partial r}{\partial t} = \frac{2D\Omega C_0\gamma}{RT(r_2-r_1)}\left|\frac{1}{r_1}-\frac{1}{r_2}\right| = \frac{2D\Omega C_0\gamma}{RTr_1r_2} \tag{6}$$

where, C_0 is the equilibrium contents of alloy element in the matrix; γ is the interface energy between carbide and matrix; R is the universal gas constant; T is absolute temperature.

Similar results have been reported for spheroidizing of eutectic carbide in the twin roll-casting of M2 high-speed steel in Ref. [7].

3 Discussion

Table 2 shows the increase in the percent of spheroidized cementite when spheroidizing time is increased (as was expected). On the other hand, by comparing the samples with different initial microstructures but the same spheroidizing time, a decrease in percent of spheroidized cementite will be noticed when moving from martensite to bainite to fine pearlite and finally coarse pearlite. The martensitic microstructure is an unstable microstructure therefore according to thermodynamic laws it has a high tendency to dissolute in high temperatures and form a more stable microstructure composed of ferrite and cementite spheres. Bainite, on the other hand, has a semistable microstructure and does not impose as much as energy on the thermodynamic system as compared to martensite and therefore the spheroidizing process is accomplished with a slower rate.

Microstructures consisting of fine and coarse pearlite are considered to be stable thus the percent of spheroidized cementite in these initial microstructures is noticeably smaller than the unstable microstructures. Due to the fact that the cementite lamellae in fine pearlite are thinner, dissolution and breakdown of these lamellae occur in a shorter time compared to coarse pearlite. Therefore, the spheroidizing rate in a fine pearlite initial microstructure is higher than a coarse pearlite.

Diffusion activation energy also helps to explain the difference in spheroidization rate in different initial microstructures. In unstable (martensite) and semistable microstructures (bainite), carbon has a high tendency to exit the microstructure and therefore the diffusion activation energy of carbon is smaller than stable microstructures (fine and coarse pearlite). Due to the fact that the cementite lamellae in fine pearlite are thinner compared to coarse pearlite, the required length of diffusion for formation of cementite spheres is shorter and therefore, the rate of spheroidization is higher in fine pearlite. In general, it can be concluded that the more unstable the initial microstructure, the higher the cementite spheroidization rate obtained from spheroidizing the microstructure. This general fact can be seen in Fig. 4.

4 Conclusions

1) CK60 steel may be spheroidized but the required time to achieve a specified percent of sphe-

roidization varies with the initial microstructure.

2) Spheroidizing time has a larger effect (approximately 2 times) on the percent of spheroidized cementite compared to the initial microstructure. Percent of contribution of initial microstructure is 31.11% compared to 58.45% for spheroidizing time.

3) The spheroidization time of unstable initial microstructures such as martensite and bainite is much shorter than stable microstructures such as fine and coarse pearlite.

4) Cementite spheres in specimens with an unstable initial microstructure are more dispersed and smaller compared to semistable and stable microstructures. Better mechanical properties are to be expected from the specimens with unstable initial microstructure.

References:

[1] Arai T, Baker G M, Bates C E, et al. ASM Handbook Volume 4-Heat Treating [M]. Materials Park: ASM International, 1991.

[2] Totten G E. Steel Heat Treatment Metallurgy and Technologies [M]. Boca Raton: CRC Press, 2007.

[3] Bendell A, Disney J, Pridmore W A. Taguchi Methods: Applications in World Industry [M]. London: IFS Publications, 1989.

[4] Taguchi G. Introduction to Quality Engineering [M]. Tokyo: Asian Productivity Organization, 1990.

[5] Unterweiser P M, Boyer H E, Kubbs J J. Heat Treater's Guide Standard Practices and Procedures for Steel [M]. Metals Park: American Society for Metals, 1989.

[6] LI Y, JIANG Q, ZHAO Y, et al. A Dynamic Study on the Spheroidizing of Eutectic Carbide in the Modified M2 Steel [J]. Acta Metall Sinica, 1999, 35: 207.

[7] DI Hong-shuang, ZHANG Xiao-ming, WANG Guo-dong, et al. Spheroidizing Kinetics of Eutectic Carbide in the Twin Roll-Casting of M2 High-Speed Steel [J]. Journal of Materials Processing Technology, 2005, 166(3): 359.