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On the Relation between Growth Characteristic and Graphite Nodule Size of Spheroidal Graphite Cast Iron

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Abstract

To ascertain the growth theory of irreversible graphite migration again, two spheroidal graphite irons of coarse and fine graphite nodules with the same molten iron were heated cyclically in still air and the relation between the growth characteristics and the change of graphite phase of different size of graphite nodule were investigated. Furthermore, the effect of the size of graphite nodules on the heat-resistance of spheroidal graphite iron was considered theoretically.

The results obtained were summarized as follows;

1) The growth theory of irreversible graphite migration became evident again from the quantitative evidence on the redistribution of graphite followed by growth.

2) Growth characteristic of spheroidal graphite iron with the same chemical composition significantly depended upon the nodule size and the growth-rate of coarse graphite iron that was generally greater.

3) It became clear by the theory of irreversible graphite migration that the heat-resistance of spheroidal graphite iron could be effectively increased by reducing the size of graphite nodules.

I. Introduction

When the cast iron is subjected to repeated heating above critical tempesatures, permanent increase in volume occurs and the mechanical properties become brittle and the iron sometimes behaves like a piece of chalk on being cut. This phenomenon has been known as the growth of cast iron[1] and occurs in all grey cast iron and its magnitude depends upon the temperature, the time held at temperature or the length and number of heating cycles, and the nature of the atmosphere or medium surrounding the iron.

There have been many attempts to explain the growth phenomenon from the beginning of the twentieth century and it has been attributed to oxidation[2], graphitization[3], cracking[4] and others, though no theory has been successfully established on the growth mechanism in cast iron. But among these theories, oxidation and cracking were in lead from the observation of microstructures of grown cast irons and the measurements of dilatometric curves.

These two theories insist that causes of growth were due to the internal oxidation by oxidizing atmospheres penetrating into the iron along flake graphite with cracking or bursting occurring around graphite flakes due to stresses caused by volume changes resulting

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from heating and cooling through the critical temperature, and they were also applied to the spheroidal graphite cast iron which was invented in the middle of the twentieth century[5], but there were no adequate explanations for lower growth-rate of spheroidal graphite iron[6].

Nagaoka has induced a new growth mechanism[7-8] due to irreversible migration of graphite from the observation of graphite structures and the peculiarity of dilatometric curves during repeated heatings.

According to his growth mechanism, cast iron grows with the formation of cavities and redistribution of graphite remaining in it because of the irreversible diffusion of carbon occurring in the dissolution and separation of graphite during heating and cooling. Therefore the redistribution of graphite observed in grown cast iron is powerful evidence supporting the theory.

To establish the theory of irreversible migration of graphite, many attempts[9-15] were made by the observation of grown cast iron by optical microscope and scanning electron microscope. The direct observation and XMA analysis of fracture of grown cast iron by scanning electron microscope, the quantitative analysis of graphite phase of grown cast iron by particle analyzer and so on, and the results obtained by these experiments established the basis of the theory and also set forth the prospect to explain various and complex growth phenomena consistently[16]

In this paper, the growth tendency and the change of graphite phase of two spheroidal graphite cast iron of coarse and fine graphite nodules with the same molten iron were analyzed in detail to reevaluate the validity of fundamental growth mechanism by irreversible migration graphite and to show that the sites of redistribution of graphite and its amount depend on the size of graphite nodules from the standpoint of the irreversibility of diffusion mechanism in the dissolution and redistribution of graphite.

Furthermore, the evaluation of heat-resistance for spheroidal graphite cast iron which is unsusceptible to oxidation was investigated from the standpoint of size of graphite nodules.

II. Experimental Procedure

Two specimens for the growth test with coarse and fine graphite nodules were produced by pouring a Mg-treated spheroidal graphite molten iron into a keel block sand mould and a metal mould for a bar to vary cooling rates. Table 1 shows the chemical composition of the spheroidal graphite cast irons and Photo 1 shows the microstructures of graphite nodules in the irons as-cast and ferritized by Figure 1. The ferritized structure is the standard from which graphite phase changes by the growth. The number of graphite nodules measured by particle analyzer was 85 nodules/mm^2 in the coarse graphite sand mould iron and 657 nodules/mm^2 in the fine graphite metal mould iron. The size of growth test piece is 23 mm in dia. and 196 mm in length which was machined from the center of ferritized iron. At

Table 1 Chemical Compositions of Spheroidal Graphite Cast Iron (%)

T.C	G.C	Si	Mn	P	S	Mg
3.62	2.62	2.40	0.56	0.023	0.014	0.038

each end of the test piece, a stainless steel bar of 3 mm in dia. and 10 mm in length was embedded as much as 7 mm to ascertein the standard of the measurement by a micrometer[17].

The test pieces were grown up to 8% linearly by repeated heating in an electrically-heated muffle furnace held at 950°C for 30 min and cooling for 30 min in still air.

The analysis of the grown graphite phase was done quantitatively by particle analyzer[15] (Luzex 450) at the center of the iron which was not affected by the outer oxidizing atmosphere[12]. Optical measuring fields with particle analyzer were $1,250 \times 100\ \mu m^2$ in the fine graphite iron and $5,000 \times 100\ \mu m^2$ in the coarse graphite iron and 30 fields were measured. The magnifications of the objective lens were $\times 20$ and $\times 10$ respectively. The items of the measurements were the total number of graphite nodules, the number of graphite nodules in various sizes and the fractional area of graphite phase.

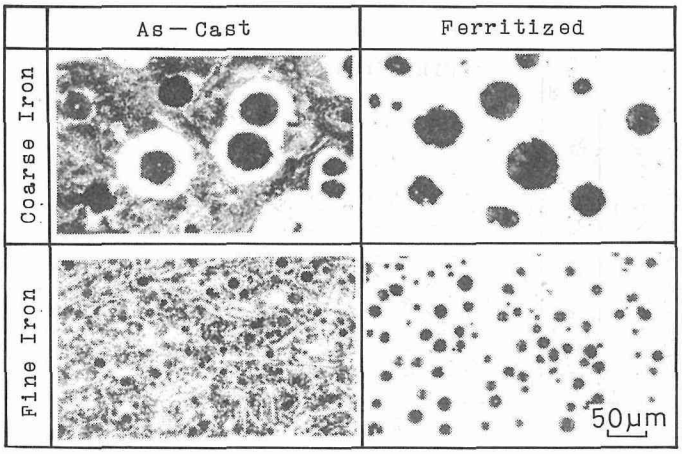

Photo 1 Microstructures of Spheroidal Graphite Cast Irons.

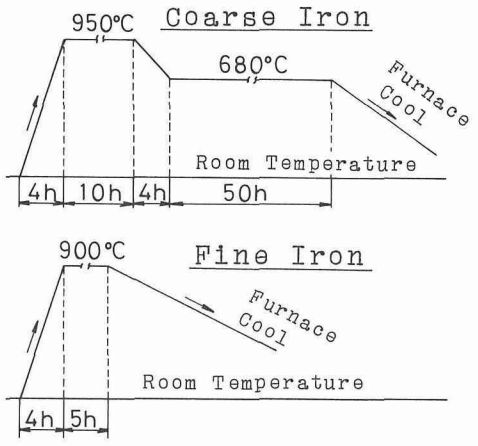

Fig. 1 Ferritizing Method of Spheroidal Graphite Cast Iron.

III. Experimental Results and Considerations

III-1) Growth Curve

Figure 2 shows the linear growth of coarse and fine graphite iron by the test. Both irons grew with the number of heatings, but up to 40 heating cycles or 3% growth, the fine graphite iron grew more than the coarse graphite iron, and after 40 cycles the curves showed an inversion in the growth of the two irons. Finally an 8% linear growth was reached after 87 cycles in the coarse graphite iron and after 109 cycles in the fine graphite iron. The average growth-rate for 5 cycles obtained with the test showed that the rate for fine graphite iron was almost constant at 0.071%/cycle although it was comparatively great at the early stage, while the rate for coarse graphite iron increased continuously from 0.044%/cycle for 5-10 cycles to 0.124%/cycle at 60 heating cycles and then it decreased. But it was noticeable that

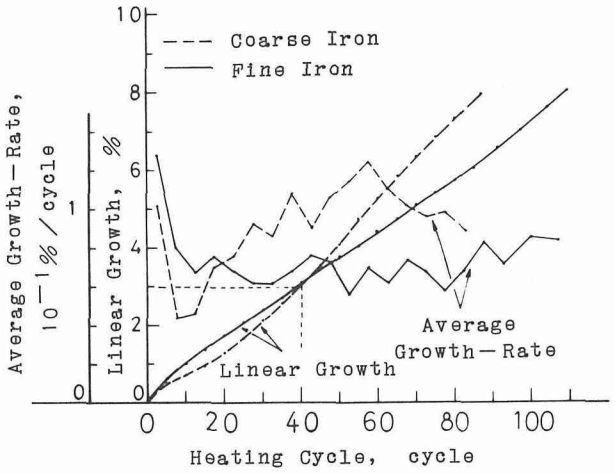

Fig. 2 Linear Growth Curve of Spheroidal Graphite Cast Iron.

at 8% linear growth, the rate of growth of coarse graphite iron was greater than that of the fine graphite iron.

The cause of the rapid decrease of the growth rate of coarse iron after 6% growth was considered as follows; graphites connect with each other and the inner structure of graphite becomes porous due to the irreversible migration of graphite by repeated heatings. Namely the whole structure of spheroidal graphite iron becomes porous such as in sintered iron and the migration of carbon from mother graphite nodules to the matrix was inhibited gradually[12]. Furthermore this was attributed to the decrease in the new sites of redistribution of graphite in the matrix.

III-2) The changes of microstructure of spheroidal graphite cast irons followed by growth

Both ferritized matrixes of cast iron became pearlitic after one heating up to 950°C, and the ferritic amount increased with the growth as shown in Photo 2.

In the coarse graphite iron fluffy graphite grown from the sides of the mother graphite, globular graphite appearing in the matrix apart from the mother graphite and the enlargement of the mother graphite were observed optically[12]. Similar features were also observed in the fine graphite iron, but it appeared that the number of globular graphites redistributed in the matrix decreased. The tendency of enlargement of graphite nodules was remarkable compared to the coarse graphite iron. Further it was noteworthy that the effect of oxidation was not found in the center of both irons even after 8% growth[13].

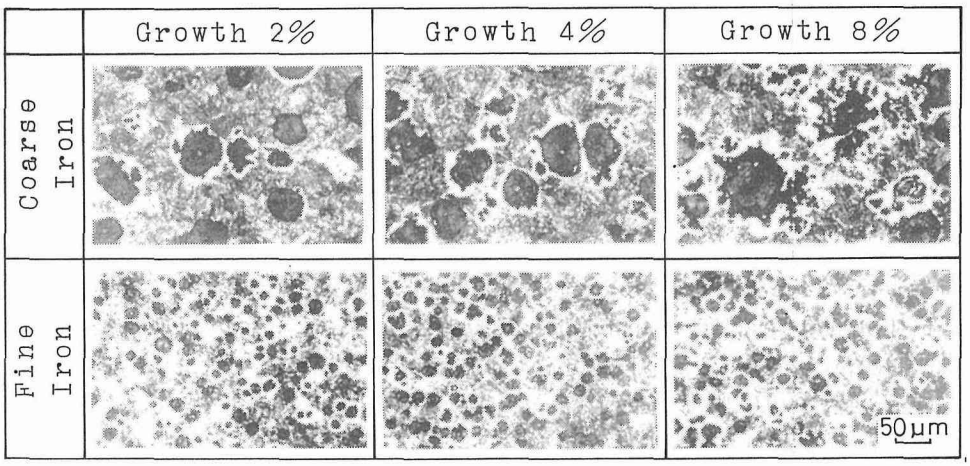

Photo 2 Changes of Microstructure of Spheroidal Graphite Cast Irons followed by Growth.

III-3) The changes of number and the fractional area of the graphite phase followed by growth

Figure 3 shows the changes of the number and the fractional area of graphite phase followed by growth. The number of graphite nodules per mm^2 was obtained by dividing the total graphite nodules by the total area in 30 fields, and the fractional area of graphite phase had an average of 30 fields.

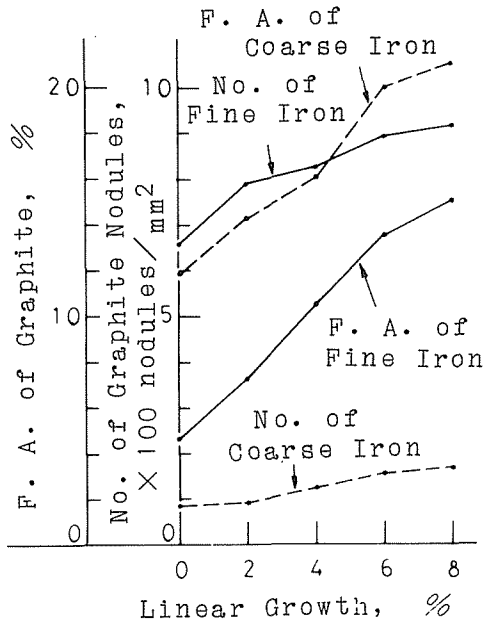

Fig. 3 Changes of No. of Graphite Nodule and Fractional Area of Graphite (F.A.)

The number of graphite nodules increased with the growth, but a big difference could be seen between both cast iron by the increasing tendency. Namely the number of graphite nodules increased almost continuously with the growth in the coarse graphite cast iron and at an 8% growth it reached about twice the growth of 0% growth, while an increasing tendency was inhibited above a 2% growth in fine graphite iron and was about 1.4 times that of 0% growth after 8% growth. Therefore, the relative increasing tendency of graphite nodules was greater in the coarse iron.

The value of the fractional area itself was greater in the coarse iron and increased with the growth. The increasing tendency was remarkable above 4% growth, but the relative increasing tendency was greater in the fine cast iron. Namely it reached about three times that of the 0% growth after an 8% growth, while it was about two times of that in the coarse iron. Furthermore, the noticeable fact was that there was almost a linear relation between the growth tendency and changes of the fractional area of graphite in both graphite irons up to 4-6% growth.

The cause of the increase in graphite phase in the center of spheroidal graphite irons which was not affected by the oxidizing atmosphere was attributed to the redistribution of graphite in the isolated sites far from the mother graphites because the amount of graphite precipitated in the iron was thought to be constant before and after the heating and cooling and there was no outer supply of carbon. The decrease at the latter stage of growth was considered as the vanishing or merging of fine graphite nodules. Thus it can be considered, though indirectly, that the measured graphite phase partly contains some cavities which were optically indistinguishable. Consequently it is concluded that there are some cavities present that were equivalent to the increment of the fractional area of graphite followed by growth and the increment corresponding to the amount of growth.

III-4) The changes in the distribution of the nodule size of graphite followed by growth

Figure 4 shows the changes in the distribution of the nodule size of graphite in the coarse and fine graphite irons followed by growth with histograms in relation of number-ratio and

size of graphite nodules.

The number-ratio of every 10 μm in nodule size was shown in the coarse graphite iron. In the distribution of 0% growth, the number-ratio of 30-40 μm of graphite nodule was at its maximum, and the type showed a close to normal distribution in which the peak declined somewhat to the left.

The size distribution of graphite nodules changed remarkably with the increase in growth. Namely, number of graphite nodule below 10 μm increased remarkably and it reached about 6 times that of 0% growth at 8% growth.

But the increasing tendency was inhibited with nodule size and it became constant over 40 μm. The maximum size of graphite nodule in a 0% growth was 110 μm, but it enlarged to 170 μm in an 8% growth. Therefore, the histogram pattern was continuously deformed with the growth from the 0% growth iron pattern deformed somewhat to a continuous distribution at an 8% growth followed by a number of less than 10 μm graphite size while increasing the maximum size of graphite nodules, namely J-type distribution[18]. The number-ratio below 10 μm at an 8% growth reached about 3 times that of 0% growth.

The number-ratio of every 2 μm was shown in the fine graphite iron. At 0% growth, graphite nodules below 12 μm are 80% of the total nodules and the number of 8-10 μm graphite nodule was the maximum.

The graphite size distribution was also a nearly normal distribution with a graphite size of less than 30 μm. The ratio of fine nodules below 10 μm decreased until a 4% growth in contrast to coarse iron and became almost constant after the 4% growth. On the other hand,

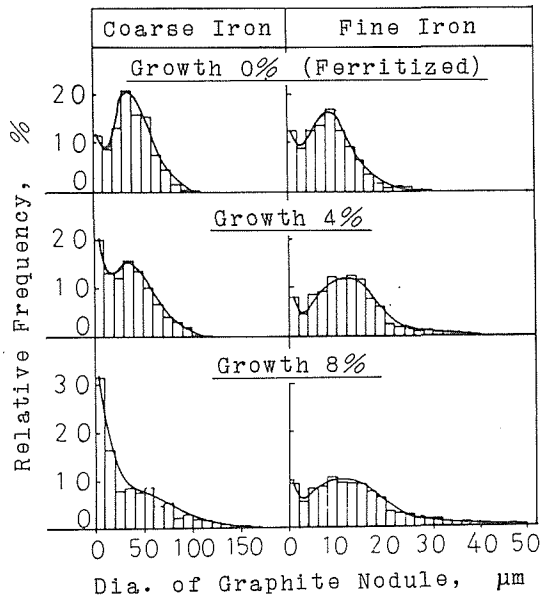

Fig. 4 Graphite Size Distribution.

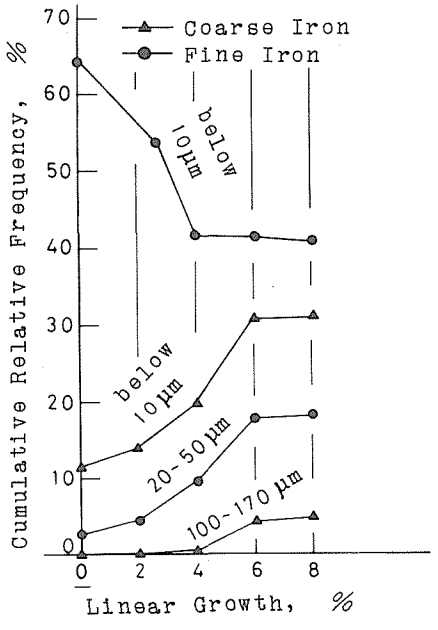

Fig.5 Cumulative Relative Frequency of Graphite Nodule below 10 μm and on the Side of Greater Size.

number-ratio above 10 μm increased until a 4-6% growth and became saturated.

The maximum size of graphite nodules which was 30 μm at a 0% growth enlarged to 50 μm at an 8% growth.

That is to say the histogram pattern deformed to a flatness by the decrease from fine to medium size and by the remarkable enlargement of the maximum size of graphite nodules.

The remarkable differences between the coarse and fine graphite irons were observed clearly in the changes of both graphite size distribution. Namely, the fine particles of graphite in the coarse iron increased with the growth, whereas they decreased in fine iron and the tendency of the enlargement of the maximum graphite nodule was remarkable. Figure 5 shows the results arranged to clarily these phenomena, in which the cumulative relative frequency was shown in the graphite size below 10 μm and greater graphite size of both irons.

It was clearly observed in the Figure 5 that the cumulative relative frequency of graphite below 10 μm increased with the growth in the coarse iron and became saturated in the latter stage of growth, whereas the frequency decreased in fine iron. In contrast, the increasing tendency of the cumulative relative frequency on the side of greater graphite size was remarkable in fine iron compared to coarse iron.

Figure 6 shows the cumulative relative frequency illustrated in a log-probability graph[19], from which the changes of graphite phase in both irons also could be understood clearly as shown in Figure 4.

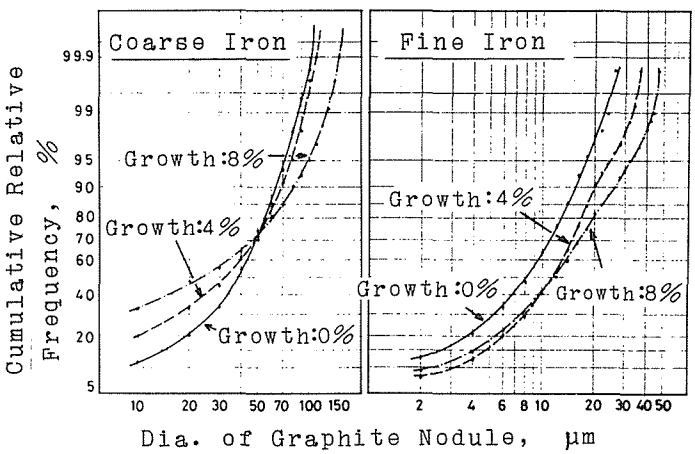

Fig. 6 Cumulative Relative Frequency illustrated on the Log-Probability Graph.

III-5) The changes of various properties of irons followed by growth

Figure 7 and 8 show the changes of various properties of irons followed by growth. The tensile strength of coarse iron ferritized which was 57.2 kgf/mm^2 decreased continuously with the growth and fell to 49.6 kgf/mm^2 at an 8% growth. The Brinell hardness number decreased with a similar tendency of tensile strength. The specific gravity which was 7.1 at a 0% growth fell to 6.1 at an 8% growth. On the other hand the properties of the fine iron

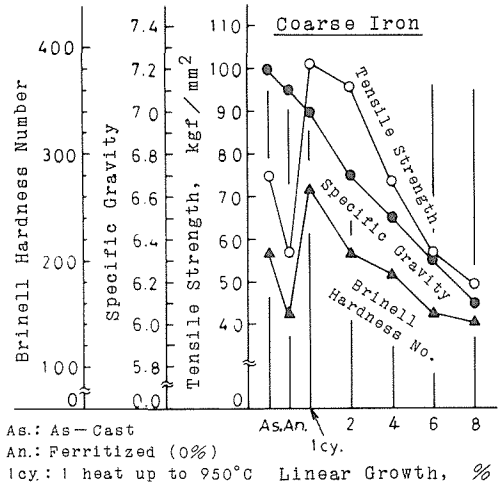

Fig. 7 Various Properties of Coarse Iron followed by Growth.

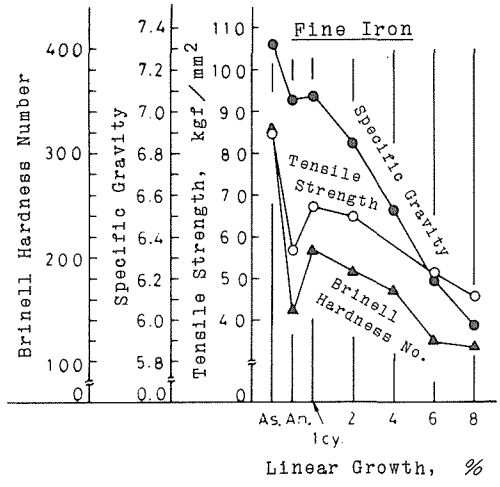

Fig. 8 Various Properties of Fine Iron followed by Growth.

also showed a similar tendency. A remarkable fact was that both irons retained their mechanical property of about FCD 50 although their inner structure became porous linearly after grown an 8%[20].

IV. Discussion

The coarse and fine spheroidal graphite cast irons with the same molten iron were prepared for the growth test, but the growth characteristic significantly depended on the nodule size and the growth of coarse iron was generally greater.

The average growth-rate of the fine iron was almost constant except at the early stage of growth, but that of the coarse iron gradually increased with the growth and reached about 2 times of that of the fine iron at its maximum. Furthermore, the increasing tendency of the number of graphite nodules and the ratio of fine nodulues in the pattern of graphite distribution were remarkable. In contrast, the tendency of enlargement of graphite nodules was remarkable in the fine iron compared to the coarse iron. But there was a common result that the increasing tendency of fractional area of graphite in both irons was about 2 times that of the linear growth and corresponded to the two-dimensional growth.

From the standpoint of the growth theory of irreversible graphite migration[8], the differences of growth tendency by size of graphite nodules were mainly attributed to the diffusion distance of carbon which was dissolved into the austenitic matrix on heating. Namely, as a part of carbon which dissolved into the austenite on heating was redistributed around the mother graphite and other parts were redistributed in the austenitic matrix at a considerable distance from the mother graphite, and if the graphite nodule is coarse and the distance between graphite nodules is long, the graphite is redistributed at an independent site in the matrix and growth is promoted with the increase in the number of graphite nodules as shown in Figure 9. This in fact is the growth mechanism of the coarse spheroidal graphite cast iron. The reason why the growth-rate was lowered in the latter stage of growth was

attributed to the porous tendency of graphite nodule and the decrease in the new sites in which graphite could redistributed. On the other hand, as many fine graphite nodules are distributed closely in the fine iron, redistribution of graphite occurs mainly with nuclei at the surface of pre-existing graphite nodules, namely the mother graphite and the morphology of redistribution of graphite results in an enlargement of graphite nodules. In such a case low growth is seen. The reason for the greater growth-rate at the early stage as compared with the coarse iron was attributed to the larger amount of dissolution of carbon since the surface area of graphite nodule was greater than the coarse iron and furthermore, there were more sites in the matrix where carbon could be redistributed in isolated fashion.

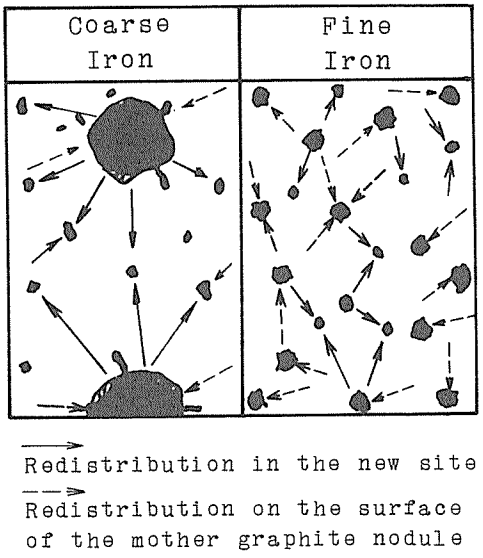

Fig. 9 The Main Mechanism of Redistribution of Graphite in Coarse Iron and Fine Iron.

Nagaoka has suggested a new growth mechanism of cast iron, namely the growth mechanism due to the irreversible graphite migration and he explained various complicated growth phenomena consistently with it after theoretical and experimental substantiation[9-15], and the validity of the growth theory were further clarified from the results of the present investigation. Furthermore, it also became clear that the size of graphite nodules was very important to the heat-resistance of spheroidal graphite iron which was unsusceptible to oxidation, namely the finer the size of graphite nodule the greater the heat-resistance.

V. Conclusions

To reascertain the growth theory of irreversible graphite migration, two spheroidal graphite irons of coarse and fine graphite nodules with the same molten state were heated cyclically in still air and the relation between the growth characteristics and the graphite phase were investigated. Furthermore, the effect of the size of graphite nodules on the heat-resistance of spheroidal graphite iron was considered from the standpoint of the size of graphite nodules.

The results obtained were summarized as follows;

1) The growth theory of irreversible graphite migraition was reconfirmed to be satisfactory from the results obtained regarding the measurement of fractional areas and the number of graphite nodules in grown spheroidal graphite iron.

2) Growth characteristics of spheroidal graphite iron with the same chemical composition significantly depended on the nodule size of graphite and the growth-rate of coarse graphite iron was generally greater than that of fine graphite iron.

3) Growth characteristics of the coarse graphite iron were seen in the number of graphite

nodules and the ratio of fine nodules in the pattern of graphite distribution that increased remarkably.

4) Growth characteristics of fine graphite iron were such that the ratio of fine nodules decreased and the enlargement of graphite nodules was remarkable.

5) From the standpoint of the growth theory of irreversible graphite migration, the difference of the growth tendency between the coarse and fine graphite iron was mainly attributed to the fact that the distance of carbon diffusion in the austenitic matrix during the redistribution of graphite which was long in coarse graphite iron and short in fine graphite iron.

6) It became clear from the standpoint of the growth theory of irreversible graphite migration that the finer the size of graphite nodules were the greater the heat-resistance of spheroidal graphite cast iron was.

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