# The Spheroidization of Cementite in a Medium Carbon Steel by Means of Subcritical and Intercritical Annealing

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The spheroidization of cementite at subcritical and intercritical temperatures was studied quantitatively by evaluation of a shape factor for the cementite particles. It was found that the degree of spheroidization, as determined by the values of the shape factor, was markedly accelerated by treatments consisting in either (1) subcritical annealing after cold deformation or (2) intercritical annealing followed by subcritical annealing. Nevertheless, some differences were found among the microstructures resulting from the above treatments, which were related to the mechanisms of cementite formation in each of them. Also, the kinetics of austenitization during intercritical annealing was found to be accelerated by previous deformation, and the analysis of the formation of austenite at intercritical temperatures in terms of an Avrami equation was consistent with an "effective" saturation of ferrite-pearlite boundaries with austenite nuclei, followed by a planar mode of growth into the pearlite nodules.

KEY WORDS: cementite; pearlite; spheroidization; carbon steel; shape factor; austenitization.

### 1. Introduction

The spheroidization of cementite in medium carbon steels is one of the most important processes employed in order to provide high ductility and deformability for posterior cold heading applications. Although various reports have addressed the physical metallurgical principles of the spheroidization process,<sup>1,2)</sup> much remains to be known in detail regarding the mechanisms



Fig. 1. Spheroidization treatments for steels.

involved and their adequate control. Figure 1 shows schematically some of the heat treatments which could be used in the spheroidization of cementite. Treatments A, B and C are the most commonly found in industrial practice. Treatment D is not commercially employed, but it has been known for some time that steels with a martensitic microstructure are easily spheroidized.<sup>3)</sup> Treatment E represents an attempt to in-line spheroidization after the finishing mill, which makes use of the hot deformation at intercritical and subcritical temperatures in order to accelerate the spheroidization of cementite.<sup>4)</sup>

In addition to this, a rigorous quantitative characterization of the spheroidization process is practically nonexistent in the technical literature, most likely as a result of the complexity involved in the process itself:<sup>2,5)</sup> a change predominantly in shape, at the early stages, more or less gradually becomes mainly a change in particle size distribution (i.e., coarsening). Some years ago, Tian and Kraft<sup>5)</sup> proposed and used a "shape" factor F (to be described later) for the quantitative characterization of the shape changes occurring as lamellar cementite turned into spheroidal particles. These authors actually showed that such a shape parameter was a convenient way to monitor the progress of spheroidization in eutectoid carbon steels and laboratory Fe-C alloys; however, it was also recognized<sup>5)</sup> that the shape factor F might be slightly dependent on the particle size distribution.

Somewhat surprisingly, we have found no following

up on the work of Tian and Kraft, despite the clear advantages to be gained from it. This is especially important as the variety of spheroidizing treatments will surely increase in industrial practice,<sup>4,6</sup>) with a correspondingly stronger need for an adequate characterization of the (probably different) resulting microstructures.

This work focuses on treatments of the types A and C. Our main objectives have been to characterize quantitatively the spheroidization process, including the effect of previous cold deformation on the results obtained with both types of treatments.

### 2. Experimental Procedure

## 2.1. Materials and Sample Preparation

Commercial AISI 1541 steel rods were used in all experiments. Their chemical composition (in wt%) was: 0.39% C, 1.42% Mn, 0.24% Si, 0.014% P, 0.008% S, 0.07% Cr, 0.033% Al. The samples were divided in four groups. The first group corresponded to the as-hot rolled condition (11.1 mm in diameter). The other three groups were made of samples subjected to 20, 35 and 47% reductions in area, respectively. Deformation of these samples was carried out by cold drawing at the actual production line.

The as-hot rolled samples were given an annealing treatment in order to homogenize their microstructure, by austenitizing at 890°C for one hour and furnace cooling to room temperature, resulting in a ferrite-pearlite microstructure. The other three groups of samples also had a similar, but deformed, ferrite-pearlite microstructure.

The samples for spheroidization and austenitization experiments were cut as 1 cm high cylinders from the original rods. A small bore was drilled along their axis and the hot junction of a chromel-alumel thermocouple was introduced in it for recording directly the sample temperature. Subsequent heat treatments were performed in a vertical tube furnace in which argon gas was continuously flowing. The sample temperature was kept constant within  $+/-2^{\circ}C$ .

### **2.2.** Experimental Determination of $Ac_1$

In order to determine the critical temperature  $Ac_1$  of this steel, samples similar to those described above were heated in a thermomechanical analyzer using heating rates of 1, 10 and 20°C. The extrapolation of the transformation temperatures measured at such heating



Fig. 2.  $Ac_1$  temperatures as a function of heating rate.

## 2.3. Heat Treatments

2.3.1. Subcritical Spheroidization Annealing (Type A) The four groups of samples were subcritically annealed at 700°C for times ranging from 2 to 10 h.

## 2.3.2. Kinetics of Austenitization

Since treatment C (see next section) would involve a previous stage of intercritical austenitization (between  $Ac_1$  and  $Ac_3$  temperatures), with a very important role in the subsequent spheroidization process, it was decided to study in some detail the kinetics of austenite formation. For these experiments the temperature of 732°C was selected. This temperature is close to  $Ac_1$ , in order to avoid the experimental difficulties associated with the very fast rates of austenite formation at higher temperatures.

Samples from the four groups were partially austenitized for times ranging from 0 to 7 200 sec, after which they were quenched in an ice-water mixture and prepared for metallographic observation. The polished samples were etched with Lepera's reagent.<sup>7)</sup> With this reagent pearlite appears dark, ferrite gets a brownwhitish coloration and martensite (the remaining austenite at the time when quenching was applied) presents itself as a white phase. The determination of the volume fraction of austenite was performed with an image analyzer either directly on the quenched samples or from their optical micrographs. In order to minimize possible errors in the measurements due to the existence of retained austenite, this phase was quantified by X-ray diffraction, using standard techniques.<sup>8)</sup> It was found that the amount of retained austenite in our specimens was always less than 1.5% in volume and, consequently, of minor relevance to the results of this study.

## 2.3.3. Intercritical Spheroidization Annealing (Type C)

For this treatment the group of non-deformed samples was intercritically austenitized at 732°C for a time of 1800 sec (enough to dissolve all pearlite, as determined in the experimental results described below) and cooled to, and maintained at, one of four different temperatures (660, 670, 680 and 694°C) by means of lead baths.

# 2.4. Quantitative Measurements of the Degree of Spheroidization

The degree of spheroidization was characterized by means of the shape factor F, used before by Tian and Kraft,<sup>5)</sup> and given by

where  $S^c$  is the specific surface area of cementite particles and  $K_m$  is their average mean curvature. Tian and Kraft<sup>5)</sup> have shown that F is mainly dependent on the aspect ratio of the particle, especially in the sequence of shape transformation starting from a platelet and going to a spherical shape, or vice versa. Also, they showed in two different iron alloys that the value of Fdecreased monotonically with time in isothermal treatments towards the value F=1. Even after  $1\ 000-3\ 000\ h$  at temperatures of  $600-700^{\circ}$ C the value of  $F\sim 1$  remained practically unchanged.<sup>5)</sup>

The actual calculation of the values of F is performed in terms of the quantities  $N_L$  (number of interceptions with cementite particle profiles per unit length of test line),  $V_v$  (volume fraction of cementite) and  $N_A$  (number of cementite particle profiles per unit area on the plane of observation), according to the equation<sup>5,9</sup>

[Notice that the definition of  $N_L$  given in Ref. 3) may be confusing].

In our work, the quantities  $N_L$ ,  $V_v$  and  $N_A$  were measured with the aid of an image analyzer, either directly on the samples or on SEM micrographs printed to suitable size for higher resolution. A total of 30 to 50 fields were used in the analysis in each sample in order to keep the magnitude of probable errors not larger than 10% (95% confidence limits) in all our measurements.

#### 3. Results and Discussion

## 3.1. Subcritical Spheroidization Annealing

Figure 3 shows the microstructural changes which occurred during type A spheroidization treatment of non-deformed and 20% cold-deformed samples, while Fig. 4 shows the corresponding microstructural changes for samples deformed by 35 and 47%. Clearly, the predeformation enhanced the degree of spheroidization, since the non-deformed samples still show some lamelar pearlite after 10 h of treatment at 700°C (Fig. 3(c)), while in deformed samples there is no lamellar microconstituent after the same treatment (Figs. 3(f), 4(c), 4(f)). After two hours at 700°C, samples deformed by 20% showed a large number of very small cementite spheroids (Fig. 3(e)); after ten hours, the spheroids have grown to a size considerably larger (Fig. 3(f)). In the case of samples deformed by 35 and 47%, the eye can not distinguish much of a difference between samples annealed for two and ten hours at the same temperature (Figs. 4(b), 4(c), 4(e), 4(f)). Nevertheless, quantitative measurements of the shape factor, as a function of the



Fig. 3. Microstructures pertaining to annealed samples with spheroidization treatment at 700°C; a) t=0, b) t=2, and c) t=10 h. Deformed samples (20% reduction in area) with same annealing treatment; d) t=0, e) t=2, and f) t=10 h.



Fig. 4. Microstructures pertaining to deformed and annealed samples with spheroidization treatments at 700°C. Samples with 35% reduction in area; a) t=0, b) t=2, and c) t=10 h. Samples with 47% reduction in area; d) t=0, e) t=2, and f) t=10 h.



Fig. 5. Shape factor for samples spheroidized at 700°C for 10 h as a function of amount of area reduction.

amount of deformation, for samples annealed for ten hours do reveal some differences, as shown in Fig. 5.

Tian and Kraft<sup>5)</sup> reported values of F between about 10 and 1 as the cementite changed its shape from lamellar to globular. Only in the case of an Fe–C alloy with a "fine" interlamellar spacing these authors found initial values of F lower than 3; however, they did not present any micrographs to enable us to compare

their microstructures with the ones from our samples. Obviously, taking the value of F as an indicator, the progress of the spheroidization process is markedly enhanced in our deformed samples, as compared to those of Tian and Kraft<sup>5)</sup> and to our own undeformed material (Fig. 5). The enhancement increases with the amount of previous cold deformation; after only ten hours at 700°C, the value of F in the sample deformed by 47% is about one third of the value corresponding to the sample without previous deformation.

The acceleration in the rate of spheroidization observed in the deformed samples, as measured by the shape factor F, may be a reflection of (a) a truly faster rate of the process itself, and/or (b) an effect related to the particle size distributions obtained in these samples. Tian and Kraft<sup>5)</sup> did point out that F is not a "pure" shape factor, although the effect of the particle size distribution on its values is not readily evident. Additional work is necessary in order to clarify this point. Here, we notice that during spheroidization of cementite two concurrent processes are involved. One of them



Fig. 6. Microstructures showing the progress of austenitization at 732°C after different times of isothermal holding; a) t=0, b) t=2, c) t=4, d) t=16, e) t=30, and f) t=120 min. A=austenite, P=pearlite, F=ferrite.

is the globulization process, by which lamellae are transformed into approximately ellipsoidal particles; the other involves coarsening of these particles. From our results, it would seem that previous deformation has enhanced both of these processes. Most likely, the overall effect is due to the combination of the mechanical fragmentation of cementite lamellae and the enhancement of carbon and iron diffusion brought about by cold deformation, as has been suggested in other studies.<sup>10-12</sup>)

### 3.2. Kinetics of Austenitization

Figure 6 illustrates the sequence of microstructural changes observed during the intercritical annealing at 732°C of non-deformed samples. A careful observation of Fig. 6(a) makes evident that, in agreement with previous reports,<sup>13-15)</sup> austenite nucleates at pearlite-ferrite and ferrite-ferrite boundaries, the former being

the most frequently observed nucleation site of the two. Also, it can be observed that the transformation proceeds firstly by the "consumption" of pearlite nodules by the austenite nuclei created at their boundaries, which thereafter grow with a roughly planar front, so that the dimensionality of growth of the austenite must be less than three.<sup>16</sup>

The volume fraction of austenite is indicated in Fig. 7 for each group of samples. At a glance, it is obvious that previous cold deformation has had an accelerating effect on the kinetics of austenite formation. The effect of cold deformation can be shown more clearly by fitting the data of Fig. 7 to an Avrami type equation<sup>16</sup> (normalizing to 100% transformation):

where X is the normalized volume fraction of austenite, t is the time of transformation at constant temperature,

and k and n are constant terms which characterize the kinetics of isothermal transformation. Figures 8(a)-8(d) present the Avrami plots and the calculated values of kand *n* corresponding to the data in Fig. 7. The details of the calculation procedure, which makes allowance for an incubation time, have been given elsewhere.<sup>18)</sup>

The values of n calculated from the data in the present work varied from 0.76 to 0.99. Considering that the number of data is somewhat limited, it may be assumed that the actual value of n should be close to 1.0; lower values for *n* would be difficult to justify in the present situation.<sup>17)</sup> The result  $n \sim 1$  can be interpreted in terms of the nucleation of austenite at interphase and grain boundaries with early site saturation and onedimensional growth.<sup>17)</sup> As a matter of fact, nucleation was indeed observed at ferrite-pearlite boundaries (Fig. 6(a), but apparently without site saturation. Rather, austenite seemed to nucleate only at some of these boundaries, occupying them completely at an early stage, and then to grow into the pearlite nodules with an almost planar transformation front. Such a mode would be consistent with a value of n close to 1 by invoking simultaneously a situation of "effective" site saturation-meaning, in this case, that only a fraction of the existing ferrite-pearlite boundaries effectively acted



Fig. 7. Austenitization kinetics at 732°C in samples subjected to different amounts of prior cold deformation.

as nucleation sites and became saturated with austenite nuclei soon after the start of the transformation.

Speich et al.<sup>13)</sup> claim that intercritical austenitization proceeds in three stages. The first of them involves the nucleation of austenite at ferrite-pearlite and ferriteferrite boundaries and the growth of austenite into the pearlite colonies. The nucleation rate of austenite is not a limiting factor in this process; rather, carbon diffusion in austenite is found to be rate controlling, the diffusion front lying along the pearlite-austenite boundary with a diffusion distance about equal to the interlamellar spacing of pearlite. The same authors consider that owing to the very short diffusion distances, at high temperatures (above 750°C) the growth of austenite is very rapid. Nevertheless, at lower temperatures (as the one applied in the present study) the rate may be limited by manganese diffusion.<sup>13,19)</sup> After completion of the first stage, or concomitant with it, the austenite grows into the surrounding ferrite to achieve the equilibrium volume fraction of austenite, according to the lever rule in the two phase region. This second stage may be controlled at high temperatures ( $\sim 780^{\circ}$ C or above) by carbon diffusion in austenite or, at low temperatures (725-730°C), by manganese diffusion in ferrite, in accordance with the analysis of several other workers.<sup>13,14,19-22)</sup> The third stage in this sequence<sup>13)</sup> involves the removal of manganese concentration gradients within the austenite by diffusion of this element through the latter phase. According to this model, the results in Fig. 7 are related to the first and second stages, the third one probably being too sluggish to become of relevance in the present case.<sup>13,21)</sup> The end of the first stage in our experiments occurs approximately at 1800 and 900 sec, for non-deformed and deformed samples, respectively. This feature of the austenitization process was determined by visual examination and taken as the time when all pearlite had transformed to austenite. Such times are indicated by filled arrows in Fig. 7.

Since our calculated values for n are close to unity (Fig. 8), and we have a situation of rapid site saturation, the value of k is determined primarily by the growth



(b)

k = .0029

Avrami plots of the austenitization process in samples treated at  $732^{\circ}$ C; a) RA = 0, b) RA = 20%, c) RA = 35%, and d) RA = 47 %.

(a)

k=.0022



Fig. 9. Effect of subcritical annealing temperature on spheroidization degree for samples intercritically annealed at 732°C for 30 min and subsequently held for 10 h at; a) 660°C, b) 670°C, c) 680°C, and d) 694°C.

rate of austenite.<sup>16,17)</sup> Austenite growth during the first stage can be controlled, in principle, by the diffusion rate of carbon, in which case this stage would terminate very rapidly, taking total times for completion in the range  $1 \times 10^{-4}$  to  $1 \times 10^{2}$  sec for carbon steels at temperatures from 740 to 900°C.13) However, in our work, as well as those of Garcia and De Ardo15) and El-Sesy et al.,<sup>23)</sup> much longer times have been observed for this stage to be completed. This may be explained if manganese diffusion is the rate-controlling factor.<sup>13,19-22)</sup> On the other hand, at a temperature as low as half the melting temperature, El-Sasy et al.23) observed that recrystallization of cold deformed ferrite was already completed before any austenite had formed. We suggest that the increase in the value of k with the amount of deformation, indicated in Fig. 8, may be related to faster manganese diffusion in recrystallized ferrite (by way of recrystallization) having a small grain size. In other words, when the ferrite grain size is smaller, the larger boundary area can contribute with additional diffusion paths,<sup>22)</sup> the resulting global effect being that the austenitization kinetics are actually accelerated by a higher amount of previous cold deformation, since the latter is known to produce finer recrystallized grain sizes.

### 3.3. Intercritical-subcritical Annealing for Spheroidization

Figure 9 shows the microstructures resulting from the application of intercritical-subcritical annealing to non-deformed samples. As seen, the higher the subcritical annealing temperature is, the rounder and bigger are the cementite particles. In terms of the shape factor F, this type of treatment seems to produce, without



Fig. 10. Shape factor as a function of subcritical annealing temperature in type C treatments.

the need for predeformation, similar results to those observed in deformed samples which underwent subcritical annealing alone (Sec. 3.1). For example, the value of F determined for the samples annealed for 10 h at 660°C (*i.e.*, 2.52) is just somewhat smaller than that found in samples subjected to a subcritical annealing at 700°C for 10 h after receiving 20% cold deformation (3.16). The value of F as a function of the subcritical annealing temperature is shown in **Fig. 10**. It is clear that the higher subcritical annealing temperatures produced lower values of F. However, the effect is much more marked in going from a temperature of 660°C to a higher one of 670°C, than in increasing the subcritical annealing temperature above 670°C.

It is interesting to notice that during these intercriticalsubcritical treatments, when the subcritical temperature used is near  $Ac_1$ , spheroidal cementite precipitates directly from austenite, as can be seen in Fig. 11, taken from a non-deformed sample annealed at 694°C for 3 h. In this micrograph very few cementite lamellae are



Fig. 11. Specimen austenitized at 732°C for 30 min and subsequently held at 694°C for 3 h, in which the direct precipitation of spheroidal cementite particles is observed.



Fig. 12. a) Precipitation of cementite particles on grain boundaries of ferrite in a sample austenitized at 732°C for 30 min and subsequently held at 694°C for 10 h. b) Precipitation of cementite particles on grain boundaries of ferrite in a sample deformed by 47% RA and transformed at 700°C for 10 h.

observed and most of the austenite islands have decompose directly into ferrite and spheroidal cementite particles, an observation seldom found in the literature.<sup>24)</sup>

Figures 12(a) and 12(b) illustrate some differences in the microstructures obtained in the two types of spheroidization treatment used in this study. Figure 12(a) shows the microstructure in a non-deformed sample, after intercritical annealing at  $732^{\circ}$ C for 30 min and an additional subcritical anneal for 10 h at 694°C; in turn, Fig. 12(b) corresponds to a sample colddeformed by 47% and subcritically annealed at 700°C for the same time interval. Careful examination shows that the majority of, and biggest, particles in Fig. 12(a) are found at ferrite grain boundaries, while mostly small cementite spheroids are seen within the ferrite grains. On the other hand, Fig. 12(b) shows a different distribution of smaller cementite spheroids, in which most of them have precipitated and grown within the ferrite grains, and only a small fraction have formed at the grain boundaries.

In the case of intercritical-subcritical annealing, cementite particles precipitate directly at ferrite grain boundaries from a supersaturated austenite (as the latter has had insufficient time to establish equilibrium conditions with non-pearlitic ("old") ferrite, as discussed before in Sec. 3.2); this occurs concurrently with the formation of "new" ferrite. The subsequent growth of cementite particles would be assisted by the grain boundary diffusion of iron. From this, it follows that ferrite grain size will be of major relevance to the kinetics of spheroidization in this type of treatment; given that most particles are situated at the grain boundaries, they will be able to grow at faster overall rates than those other particles precipitated within the ferrite grains. The final effect on the size and number of the particles is evident in Figs. 11 and 12(a).

From the above discussion, it would seem that the faster spheroidization kinetics obtained by means of intercritical-subcritical annealing come about as a result of the rapid globulization of cementite, which has been directly precipitated from austenite, and from the faster diffusion paths offered by the grain boundaries during cementite growth and coarsening.

Additional studies are now in progress in order to determine the effect of the spheroidization treatments on the technologically important properties of steels. At the present stage, we anticipate that the modern technologies of thermomechanical processing and controlled cooling of steels could be employed in order to simultaneously induce and take advantage of the rapid spheroidization treatments and mechanisms explored in this work.

## 4. Conclusions

(1) The shape factor F, first applied by Tian and Kraft to the characterization of the spheroidization process, is indeed a useful measure of the state of spheroidization attained in steels.

(2) Previous cold deformation increases remarkably the spheroidization rate of pearlitic-ferritic microstructures in a 1541 AISI type steel.

(3) Previous cold deformation also accelerates the formation of austenite at intercritical temperatures. This effect increases with the amount of deformation.

(4) The nucleation of austenite was observed to take place mainly at some (but not all) of the ferritepearlite boundaries. However, a situation of "effective" site saturation seemed to apply in this case, according to the analysis of the formation of austenite in terms of Avrami's equation.

(5) In non-deformed specimens, the decomposition of the austenite originally formed at intercritical temperatures produced directly, in addition to new ferrite, spheroidal cementite particles. The spheroidization process in this case was even faster than that observed during subcritical anneal of cold worked specimens.

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