Spheroidization Cycles for Medium Carbon Steels

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An investigation has been made of spheroidization of medium carbon steels used in the bolt industry. Two process cycles were considered. One was the intercritical cycle, widely used in industry, in which the steel was heated above the lower critical, A1, temperature for approximately 2 hours; then cooled below it; and held for various periods to allow the austenite to transform and carbides to spheroidize. The other process was a subcritical cycle, which involved heating to below the A1 for various times. Wire samples of two steels were studied: AISI 1541, which is high in manganese and considered difficult to spheroidize, and AISI 4037, which is considered easier to spheroidize and is used extensively in industrial applications.

Both cycles produced similar drops in hardness. However, 1 hour of the subcritical cycle yielded greater ductility than 32 hours of the intercritical process, as measured by tensile tests. Results of a new flare test designed to evaluate formability also indicated much faster spheroidization in the subcritical cycle.

The level of spheroidization was defined in this study to be the percentage of carbide particles with aspect ratios less than 3. In 30 minutes, the subcritical cycle produced the same percentage of particles with an aspect ratio of less than 3 as produced by the intercritical cycle in 32 hours. The fast spheroidization in the subcritical process is attributed to the fine pearlite generated by the current practice of rapid cooling off the hot mill. This advantage is lost in the intercritical process as the original pearlite is dissolved above the A1 temperature.

I. INTRODUCTION

HIGH strength bolts are made from alloy steels containing from 0.35 to 0.50 pct carbon and enough alloy to achieve the required hardenability. The alloys studied in this work are two of the most common alloys: AISI 1541 and AISI 4037. Wire rod was produced by hot rolling to the diameter of the bolt shank, cooling by fans, and coiling. After pickling and coating with lime, the coils are spheroidized to achieve the necessary formability. They are then sheared and cold headed, and threads are rolled onto the shank. Finally, the finished bolts are austenitized, quenched, and tempered.

In the cold heading, the shank is held in a set of grips, while the end is struck with a female die with a cavity having the desired shape. The upsetting causes large tensile hoop strains that may cause splitting if the material has insufficient ductility. Figure 1 shows such a fracture. Spheroidizing provides the needed ductility for cold heading.[1] The spheroidization treatment is by far the most time consuming phase of bolt manufacture. Commercial spheroidization of coils usually takes from 10 to 24 hours depending on the alloy and the size of the load.

A number of articles have been written about the mechanisms and kinetics of spheroidization.[2–10] However, very little has been written about the details of the heat treatments. Several articles[11–15] have recommended that the steels be heated into the temperature region between the upper and lower critical temperatures (between A1 and A3) prior to spheroidizing the carbides below the lower critical, but none of these compare the results of such an *intercritical process* with simply heating to below the lower critical (A1) (*subcritical process*), and none give data relating spheroidization and formability.

A survey was made of commercial spheroidization practice by interviewing eight companies. None of the companies would allow their names to be identified. Of these, four were steel producers that spheroidize their own product before shipping to bolt manufacturers. One company simply buys steel wire, cold reduces, and spheroidizes it before selling to bolt manufacturers. The other three companies are bolt manufacturers who spheroidize the wire themselves before cold heading. Of the eight companies, seven use the intercritical process. One company was using a subcritical process, simply heating to below the lower critical and holding. However, that company was considering changing to the intercritical process.

All of the producers agreed that the AISI 1541 steel was the more difficult to spheroidize. For that reason, most of the work in this article was done on that alloy. The cycles used by three of the producers are given in Table I.

The current hot-mill practice of cooling by forced air after hot rolling (Stelmor process)[16] produces very fine pearlite, which should spheroidize rapidly. The intercritical process destroys this fine pearlite by heating into the intercritical region and replaces it by a very coarse pearlite formed at temperatures slightly below the lower critical. Recent work[17,18] confirms that spheroidization occurs more rapidly in the subcritical process. This article compares the two spheroidization processes in two steels.

II. EXPERIMENTAL PROCEDURES:

Two alloy steels were studied. One was AISI 4037, containing 0.37 pct C, 0.80 pct Mn, and 0.25 pct Si with 0.006

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Fig. 1—Split that occurred during cold heading of a bolt.

Table I. Commercial Cycles Used to Spheroidize AISI 1541

	Intercritical Soak		Subcritical Soak	
Company	Temperature	Time	Temperature	Time
Bolt Producer 1	748 °C	6 h	690 °C	10.5 h
Bolt Producer 2	748 °C	6 h	694 °C	12 h
Steel Producer 1	748 °C	4 h	690 °C	14 h

Table II. Experimental Spheroidization Cycles

		Intercritical Soak		Subcritical Soak
Steel	Cycle	Temperature	Time	Temperature
AISI 1541	intercritical	748 °C	2 h	688 °C
	subcritical			704 °C
AISI 4037	intercritical	748 °C	2 h	704 °C
	subcritical			704 °C

pct P and 0.015 pct S. The other was AISI 1541, which contained 0.37 pct C, 1.31 pct Mn, and 0.17 pct Si with <0.003 pct P, 0.012 pct S, and 0.02 pct Al. The calculated A1 temperatures for the two steels are 721.5 °C for the AISI 4037 and 714 °C for the AISI 1541. Both steels were obtained in the form of 0.470 in. (11.9 mm) diameter wire. The cycles used for the two steels are summarized in Table II. The reason for the 688 °C subcritical soak temperature for the intercritical cycle of the 1541 steel is that initial attempts to spheroidize it at 704 °C required excessive times. The subcritical soak time was varied so the changes of microstructure and properties with time could be studied. The wire was cut into 12 in. (30.5 mm) lengths and heat treated in a vacuum furnace. The heating time for the subcritical cycle was 30 minutes, and the cooling time from the intercritical anneal to the subcritical was about 10 minutes. In all cases, the cooling time from the subcritical temperature to 540 °C was 30 minutes.

Fractures in cold heading are caused by secondary tensile stresses in the hoop direction. Figure 2 illustrates a new flare test designed to assess formability by simulating this condition. For this test, specimens were prepared from the heat-treated wire by boring a hole of 0.250 in. (6.35 mm)

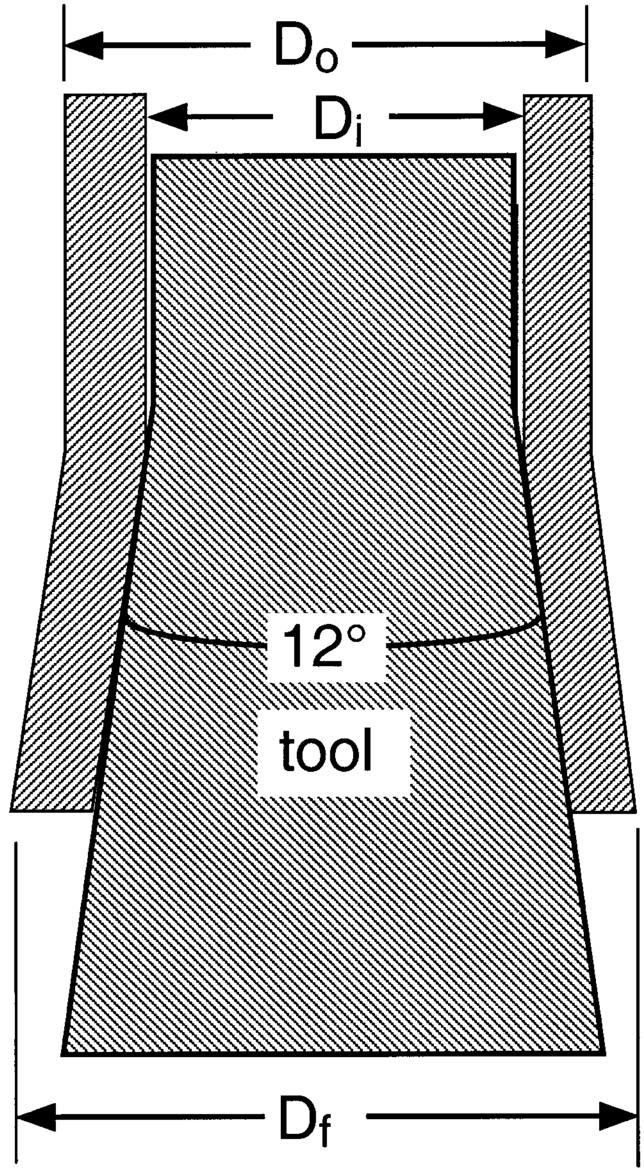


Fig. 2—Flare test used to simulate the hoop strain in cold heading. The circumference of the specimen is forced to expand over a conical tool until splitting is observed.

diameter, turning the outside diameter to 0.370 in. (9.40 mm), and finishing the ends on a lathe. The specimens were then forced over a conical tool at about 1 mm/min, which corresponds to a strain rate of about 4×10^{-4}/s, until a split was observed. Figures 3 and 4 show typical fractures. The fracture strain was taken as

$$\varepsilon = \ln (D_f/D_o) \qquad [1]$$

where, D_o and D_f are the initial diameter and the diameter at the point the split is first observed.

Other mechanical property measurements included the Vickers hardness (200-g load) and the reduction of area in tension tests on bars of 0.25 in. (0.063 mm) diameter and 1 in. (0.254 mm) gauge length.

The changes in the microstructure of the AISI 1541 steel were monitored by scanning electron microscopy. An image analysis program (NIH Image version 1.35, Bethesda, MD)

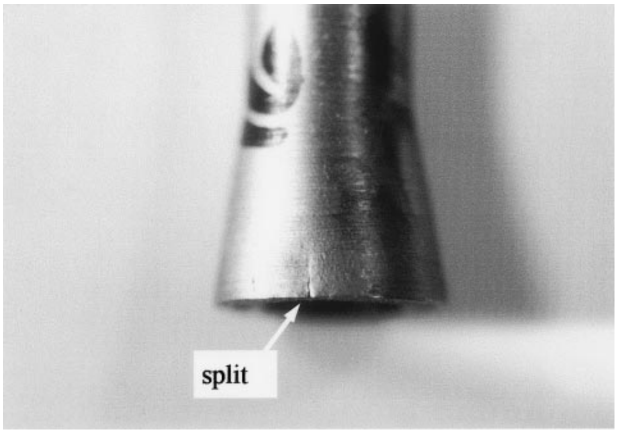


Fig. 3—Side view of flare test specimen showing a split.

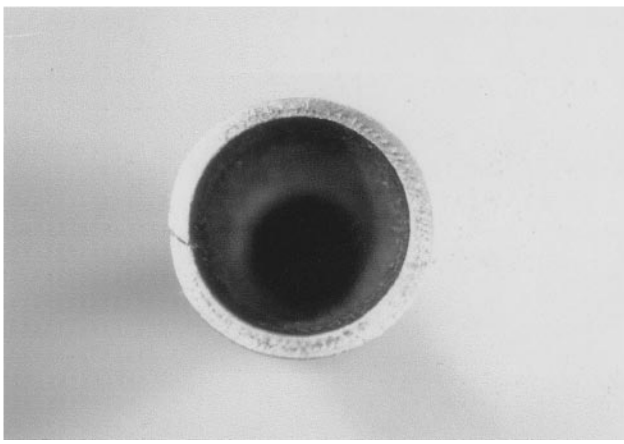


Fig. 4—Split that occurred during a flare test. Bottom view.

was used to determine the aspect ratio of carbide particles and the percent fine pearlite after the intercritical cycle. In industry, the degree of spheroidization is usually estimated by comparing the microstructure with standards. This is very subjective, especially when the size of the carbide particles is very different. Operators tend to underestimate the degree of spheriodization if the microstructure is different from what they are accustomed to. By using an image analysis program, the subjectivity is eliminated. The percentage of particles with aspect ratios less than 3 was used as the measure of spheroidization.

III. RESULTS

In all cases, the times quoted for the intercritical cycle are the times spent below the lower critical. They do not include the 2 hours spent in the ferrite plus austenite region.

A. *Mechanical Tests*

Figures 5 and 6 compare, for both steels, the increase of formability with time for the two processes. The intercritical process requires about 10 hours to reach the same degree of formability as achieved by 2 to 4 hours in the subcritical process. Reduction of area in tension was measured only for the AISI 1541 steel. The reduction of area increased

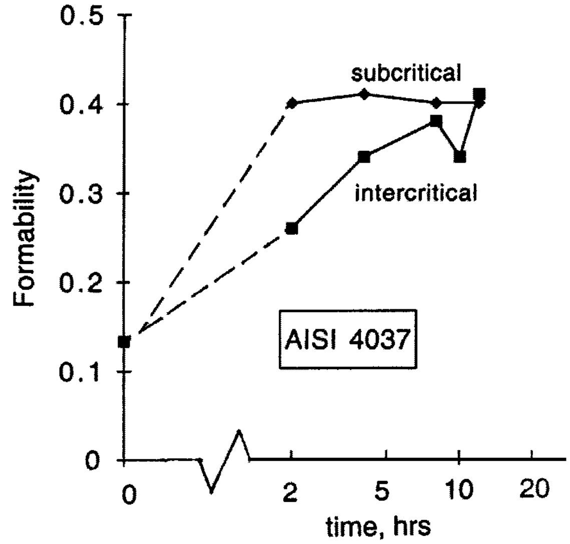


Fig. 5—Flare test results for the AISI 4037 steel. The times plotted for the intercritical cycle are periods spent at 704 °C.

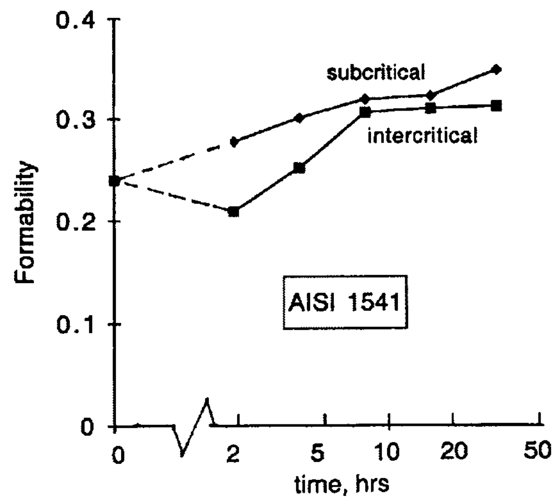


Fig. 6—Flare test results for the AISI 1541 steel. The times plotted for the intercritical cycle are periods spent at 688 °C.

much more rapidly with the subcritical cycle, as shown in Figure 7. The intercritical cycle took 32 hours to reach the same level of ductility as achieved in one-half hour by the subcritical cycle. For the AISI 1541 steel, short times in the intercritical process actually decreased both the ductility and the formability from the hot-rolled condition. This decrease is probably related to the presence of grain boundary carbides and pearlite that is coarser than in the hot-rolled condition.

Figures 8 and 9 show the changes of hardness of both alloys with time below the lower critical. The difference between the two cycles is not very great. Comparing these two figures, it is obvious that much longer times are required for the 1541 steel than the 4037 to achieve the same amount of softening.

B. *Microstructure*

The transformation of austenite to ferrite and carbide in the intercritical cycle is very slow at the 688 °C soak-cycle

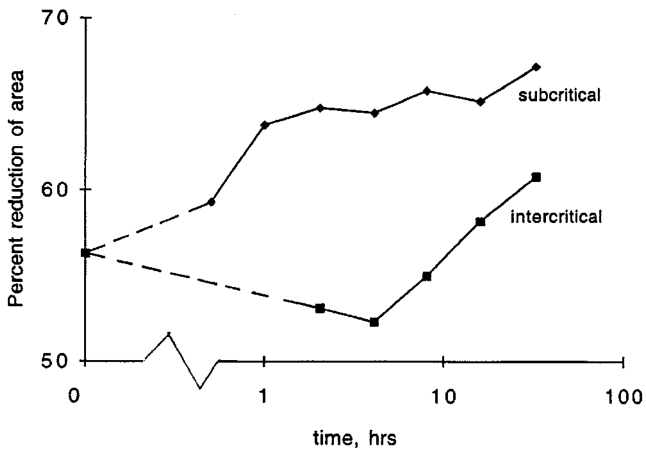


Fig. 7—Increase of ductility of AISI 1541 steel during spheroidization. Intercritical cycles are periods spent at 688 °C.

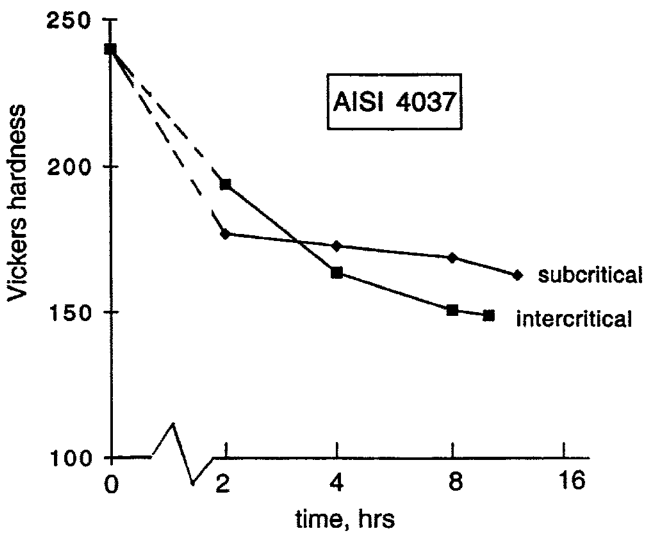


Fig. 8—Hardness decrease during spheroidization of AISI 4037. The times plotted for the intercritical cycle are periods spent at 704 °C.

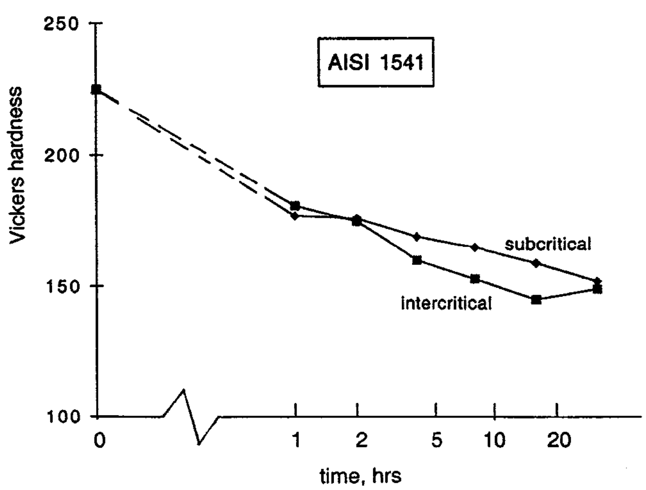


Fig. 9—Hardness decrease during spheroidization of AISI 1541.

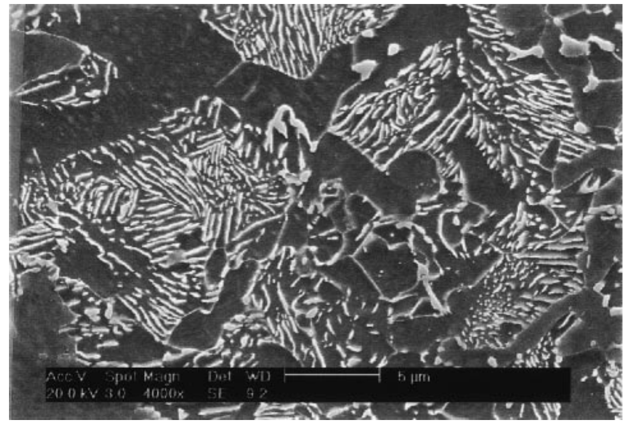


Fig. 10—Microstructure of AISI 1541 from intercritical cycle after 2 h at 688 °C and cooling to room temperature. Note the 5 μm marker. The fine pearlite in this microstructure was formed during the final cooling rather than at 688 °C.

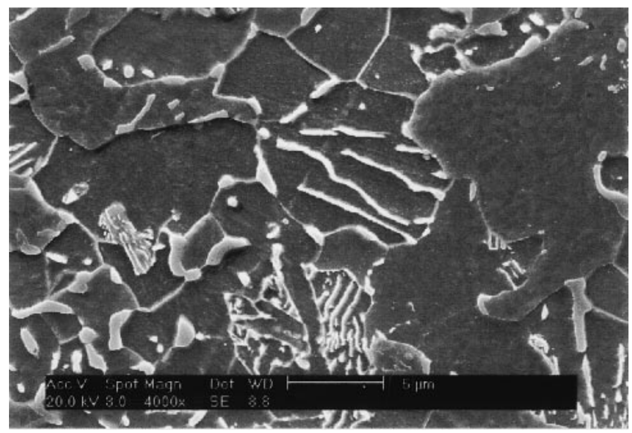


Fig. 11—Microstructure of AISI 1541 from the intercritical cycle after 4 h at 688 °C and cooling to room temperature. Note the 5 μm marker. The coarse pearlite was formed at 688 °C and the fine pearlite was formed during the final cooling rather than at 688 °C.

temperature. Figure 10 shows the microstructure of the 1541 steel held at 748 °C for 2 hours, cooled to 688 °C, held for 2 hours, and then vacuum cooled to room temperature. The pearlite in the microstructure was formed during the cooling to room temperature rather than during the hold at 688 °C. Figure 11 shows a similar sample after a 4-hour hold at 688 °C. The very coarse pearlite and the large carbide particles in the prior austenite grain boundaries formed at 688 °C. There is also some finer pearlite that formed during the cooling to room temperature. The finer pearlite represents the austenite that had not transformed during the hold at 688 °C. Figure 12 shows the microstructure after 16 hours. At this point, there is appreciable spheroidization of the coarse pearlite. The amount of the finer pearlite was assessed using NIH Image, with images enhanced to show only the regions of the fine pearlite. Figure 13 is an example. Using images such as this, it was possible to monitor the austenite transformation at 688 °C. Figure 14 is a plot of the volume percent of fine pearlite (hence, untransformed austenite) as a function of time at 688 °C. Clearly, it takes about 4 hours to convert most of the austenite. In contrast, the subcritical cycle starts with fine pearlite, which spheroidizes rapidly.

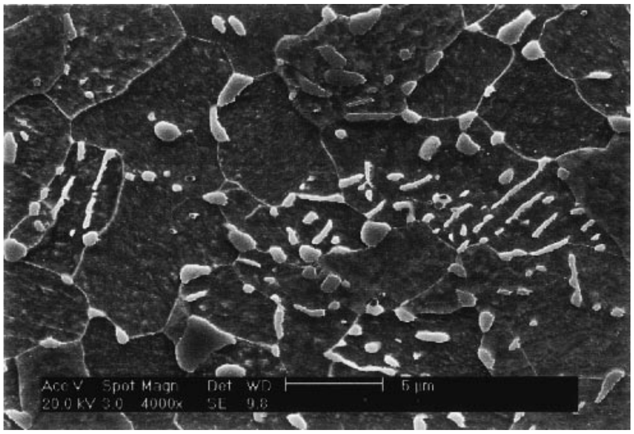


Fig. 12—Microstructure of AISI 1541 from the intercritical cycle after 16 h at 688 °C and cooling to room temperature. Note the 5 μm marker. Some spheroidization of the carbides is apparent.

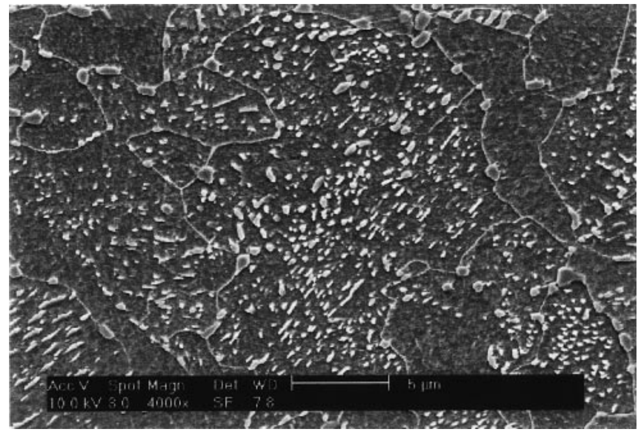


Fig. 15—Microstructure of the 1541 steel after a 4 h subcritical spheroidization. Note the 5 μm marker.

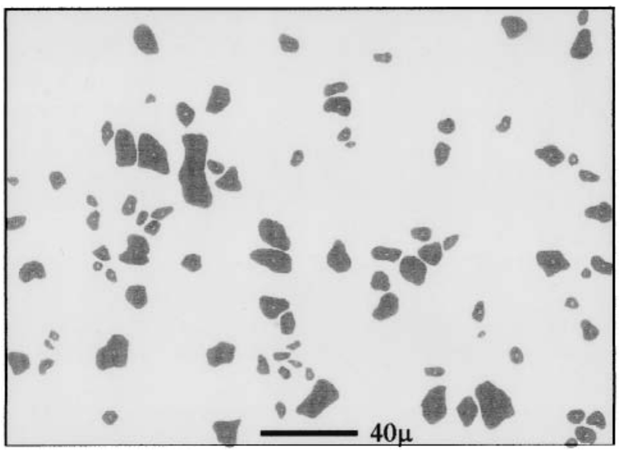


Fig. 13—Enhanced image showing only the areas of fine pearlite in the 1541 steel after 3 h at 688 °C. These areas represent austenite that was not transformed.

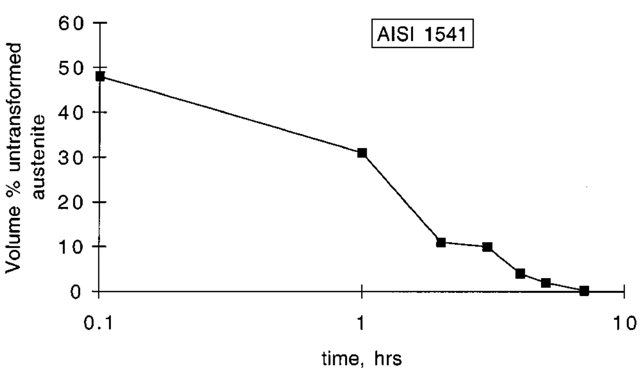


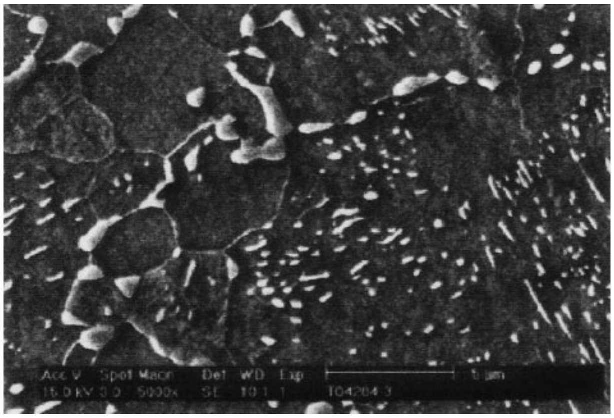
Fig. 14—The volume fraction of the microstructure that consisted of austenite that had not transformed as a function of the time at 688 °C.

Figure 15 shows that a great deal of spheroidization has occurred after 4 hours.

The aspect ratios of the carbide particles were measured by NIH Image from images enhanced to show only the carbide particles. The smallest spheroids analyzed were characterized by approximately 1300 pixels. The procedure for image analysis of the secondary carbide (regions of remaining austenite patches) consisted of digitizing Polaroid images of the microstructure. Then, the images were enlarged and the regions of secondary carbide were blacked by hand to allow the software to identify the whole region. Next, the enhanced image was digitized, and the scale (pixels per micron) was determined, using a micron bar on the image, and entered into the analysis program. The program determined the area fraction pearlite. The results from five randomly selected regions were averaged. Figure 16 shows an SEM micrograph and the corresponding enhanced image. The percent of particles that had an aspect ratio of less than 3 was chosen as a parameter to quantitatively describe the degree of spheroidization. Figure 17 is a plot of this parameter for both cycles.

IV. DISCUSSION

There are two reasons why the subcritical cycle produces much more rapid spheroidization than the intercritical cycle. The first is that with the intercritical process, austenite is slow to transform to ferrite and carbide at temperatures just below the A1, and no spheroidization can occur before the carbide forms. The second reason relates to the nature of the carbides formed. There are several differences. The Stelmor cooled product has finer pearlite with many more kinks and breaks. The intercritical cycle also produces some grain boundary carbides. Table III compares the platelet thickness and break frequency in the pearlite formed in the intercritical cycle with that in the as-received material (Stelmor cooled). The plate thicknesses were determined from five cross sections for each heat-treatment condition. These were polished and etched with a 50/50 nital/picral mix. Five pearlite colonies were taken on each cross section. Since the true plate thickness is only seen where the plate is normal to the cross section, the selected colonies were those with the smallest apparent plate thickness. Four plate-thickness measurements per colony (total of 100 measurements per heat-treatment conditions) were made using an SEM. Reported values are the averages of the 100 readings. It is well documented that the finer the pearlite is, the more rapidly the spheroidization occurs.[1,19,20] One would expect the time, t, to reach a level of spheroidization to be proportional to the square of the



(*a*)

(*b*)

Fig. 16—(*a*) SEM micrograph of AISI 1541 steel after 16 h of subcritical treatment. Note the 5 μm marker. (*b*) Enhanced image of the same region as in (a).

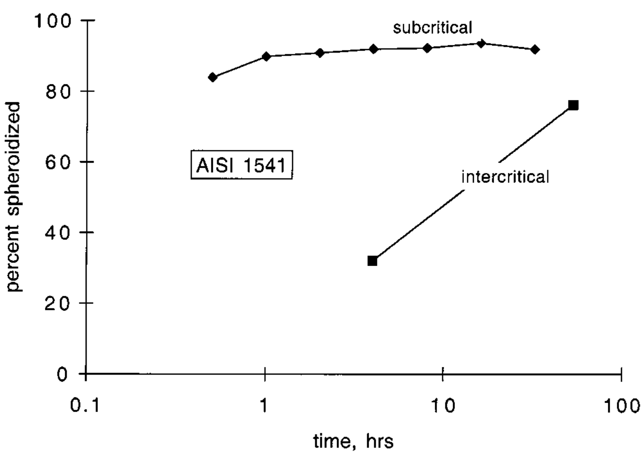


Fig. 17—Effect of time on the degree of spheroidization as measured by the percentage of carbide particles having an aspect ratio of less than three.

platelet size, x. In this case, the ratio of times for two different structures should be given by

$$t_2/t_1 = (x_2/x_1)^2 \qquad [2]$$

The thicknesses in Table I correspond to t_2/t_1 of about 10 and the distances between breaks to t_2/t_1 = about 30. Figure 7 shows that the same reduction of area (59 pct) was found

Table III. Pearlite Platelet Thicknesses and Break Frequency

Cycle	Platelet Thickness, μm	Number of Breaks/μm
Intercritical cycle	0.25	0.32
Subcritical cycle*	0.075	1.70

*Pearlite in the as-received material.

Table IV. Ductility, Degree of Spheroidization, and Average Particle Area in the 1541 Steel after Spheroidizing for 32 Hours

Cycle	Particle Area (μm)^2	Pct Spheroidization	Pct Reduction of Area
Intercritical	0.64	76.2	60.8
Subcritical	0.20	91.9	67.0

after 0.50 hours subcritical and about 20 hours intercritical treatments. This corresponds to t_2/t_1 of about 40.

It is often assumed that the ideal microstructure contains large carbide particles rather than small ones. Table IV compares the ductility with the degree of spheroidization and the average carbide-particle sizes in the 1541 steel for the two cycles after spheroidizing for 32 hours. These data indicate that from a formability standpoint, there is no advantage of a microstructure with a large particle size.

V. CONCLUSIONS

1. Formability of medium-carbon cold-heading steels has been assessed by a new flare test.
2. Spheroidization of medium-carbon cold-heading steels has been characterized by the percent of particles with aspect ratios less than 3.
3. Formability correlates well with degree of spheroidization but not with hardness changes or particle size.
4. The subcritical process requires much less time for spheroidization than the intercritical process.
5. Adoption of the subcritical process by industry would greatly reduce the energy, time and cost of spheroidizing.

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