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The development of fine structure superplasticity in cast ultrahigh carbon steels through thermal cycling

E. S. KAYALI*, H. SUNADA†, T. OYAMA, J. WADSWORTH, O. D. SHERBY
Department of Materials Science and Engineering, Stanford University, Stanford, California 94305, USA

Superplastic structures have been developed in an as-cast ultrahigh carbon steel, containing 1.6% C, by heat treatment alone. The heat treatment involves quenching from high temperature (1150° C) and then repeated cycling across the A_1 transformation temperature (727° C). This changes the coarse, as-cast structure to an ultrafine ferrite structure containing fine spheroidized cementite particles. An optimum structure is found after about 10 cycles and strain rate sensitivities of about 0.45 and elongations to failure of over 300% have been achieved.

1. Introduction

Ultrahigh carbon (UHC) steels contain between 1 and 2% C (Fig. 1) and in the as-cast condition have the expected hypereutectoid structures of large (>20 μm) pearlitic grains with coarse, proeutectoid cementite at the grain boundaries. Although traditionally believed to be brittle and difficult to work, in recent years it has been shown that these steels can in fact be made superplastic by any one of a number of thermal-mechanical processing routes [1–6].

The required superplastic structure is usually obtained by a thermal-mechanical treatment involving large deformations (typically the true strain, ϵ, is ~1.5 to 3). In the case of UHC steels, this processing results in a structure of very fine spheroidized cementite particles (0.1 to 0.5 μm diameter) in a matrix of very fine ferrite grains (0.5 to 1 μm). Typically the steels are superplastic in the temperature range of 550 to 900° C [4] (i.e. both below and above the A_1 transformation temperature at 727° C, Fig. 1). Elongations to failure at 650° C of over 1500% are readily achieved in UHC steels [5, 6].

This paper describes a thermal cycling treatment for cast UHC steels as a means of developing fine structure. It will be shown that such cast materials, after cycling, exhibit superplastic behaviour and their properties approach those observed in wrought UHC steels.

The feasibility of utilizing heat treatments alone for refining structures in wrought high carbon materials has been considered by various investigators [7, 8]. Stickels [7] reviewed a number of methods in a paper on the influence of carbide size on bearing fatigue life. Grange [8] has developed uniform dispersions of very fine carbides in AISI 52100 bearing steel through heat treatments and also demonstrated some refinement in a 1.26% C cutlery steel through heat treatment. Basically one of two methods have been used. In the first, the steel is heated to a temperature where all carbides are dissolved, and the steel is quenched. It is then tempered to eliminate retained austenite and to form fine temper carbides. Upon rapid austenitization by induction heating, and further quenching and tempering, very fine structures result. Grange [8] found that additional cycling (up to four times) further refined the fine grain size in some cases. In the second method, the steel is austenitized

*E. S. Kayali is currently Assistant Professor, Department of Metallurgy and Materials Science, Istanbul Technical University, Istanbul, Turkey.
† H. Sunada is currently Associate Professor, Department of Mechanical Engineering Himeji Institute of Technology, 2167 Shosha, Himeji, Japan.

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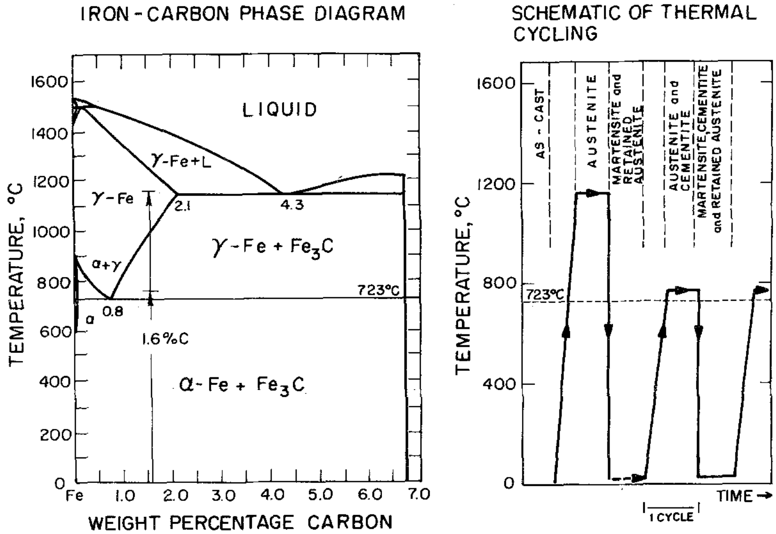

Figure 1 The iron–carbon phase diagram and a schematic illustrating the heat treatment of the as-cast ultrahigh carbon steel (1.6% C). The steel is first quenched from a temperature in the austenite single phase field and then cycled from a temperature just above the A_1 for the desired number of cycles.

to a temperature where all carbides are dissolved and then isothermally transformed to pearlite or bainite and then again austenitized, quenched and tempered.

2. Materials and experimental procedure

An ultrahigh carbon steel casting of the following composition (nominally 1.6% C) was chosen for this investigation: by weight, 1.57% C, 0.73% Mn, 0.28% Si, 0.015% S, 0.020% P, balance Fe. This carbon content corresponds to a volume fraction of cementite of 0.24. All heat treatments were carried out in air on as-cast samples of approximate size 60 x 13 x 2.5 mm. The heat treatments consisted of initially austenitizing in air for 10 min at 1150° C followed by oil quenching. No quench cracks were observed in these samples which had a hardness of $R_c \simeq 55$. A more severe quench, such as a brine quench, led to the formation of microcracks. The next step, which constitutes one cycle, consisted of heating to a temperature just above the A_1 for 10 min and quenching into water, as shown schematically in Fig. 1. Samples were cycled 3, 8, 14 and 20 times.

Following the thermal cycling treatments, samples were annealed at 650° C for 30 min. Tensile coupons were then machined for superplastic testing, care being taken to remove any decarburized surface layers. Two types of tests were carried out at 650° C to determine the superplastic characteristics of the steel. An atmosphere of forming gas (90% N_2, 10% H_2) was used for all superplastic tests. In the first type of test, specimens of 12.7 mm gauge length were tested in tension on an Instron testing machine, at a constant crosshead speed, to failure, at an initial engineering strain rate of 1% min^{-1}. In the second type of test, using tensile specimens of 25.4 mm gauge length, the flow stresses at each of a number of different crosshead speeds were determined. The relationship between the true flow stress, σ, and the true strain rate, $\dot{\epsilon}$, can be used as a measure of the degree of superplastic behavior of a material since they are related through the strain rate sensitivity, m, in the equation $\sigma = K\dot{\epsilon}^m$ where K is a constant. Thus a plot of log σ versus log $\dot{\epsilon}$ yields a line whose slope is m. For perfectly viscous materials, m is equal to unity. Superplastic materials usually have a value that lies in the range $0.4 < m < 0.6$. Non-superplastic materials, in the same temperature and strain rate range, have a value of $m \lesssim 0.2$.

For comparison, a hot and warm rolled steel of the same composition is included in the results*. This method of producing ultrafine structures that are superplastic is well established [1–6].

Optical micrographs were prepared of all the structures using standard polishing and etching techniques. Grain sizes were determined from

*A casting was hot rolled during cooling from 1150 to about 600° C to a true strain of $\epsilon = -1.9$. It was then isothermally rolled at 650° C to a further true strain of -1.9.

optical micrographs. The mean linear intercept, $\bar{L}$, was measured and converted to the average spatial grain diameter, D, using the relationship $D = 1.75\,\bar{L}$ [9].

3. Results and discussion

The warm temperature properties of the as-cast and cycled structures were determined using the tests described in the last section. The results of such tests are shown in Fig. 2. In the case of the as-cast material, in the range of strain rates below $10^{-3}\,\text{sec}^{-1}$, where superplastic characteristics are generally expected, the slope, m, is found to be about 0.17, a typical value for a non-superplastic structure. Fig. 2 also shows the results after cycling as-cast material both 3 times and 14 times, and, for comparison, the material in the warm rolled condition is also illustrated. As may be seen, if the number of heat treatment cycles is increased, the slope of the line decreases reflecting increasing superplasticity. A value of $m = 0.3$ is found after 3 cycles and a value of $m = 0.45$ is found after 14 cycles. By comparison, the same steel processed by the conventional route of hot-and-warm rolling has a slope of $m = 0.5$. The decreasing slope means of course that at a given strain rate, the steel is weaker suggesting considerable refinement of the starting structure. By way of example, at a true strain rate of $\dot{\epsilon} = 10^{-4}\,\text{sec}^{-1}$, the flow stress of the as-cast steel is 138 MPa, after 3 cycles it is 100 MPa, after 14 cycles it is 66 MPa and in the hot-and-warm rolled condition it is 44 MPa.

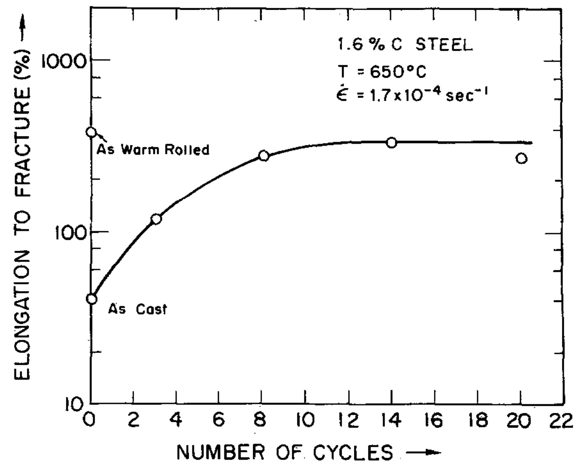

Figure 3 The elongation to failure at 650° C, at an engineering strain rate of 1% min^{-1}, for the 1.6%C ultrahigh carbon steel as a function of the number of cycles. Also shown is the value for the same steel processed by hot-and-warm rolling.

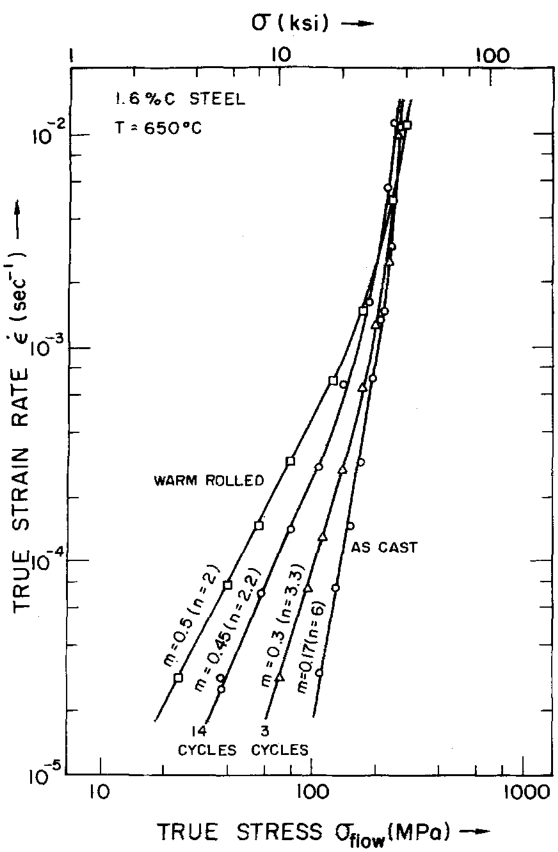

Figure 2 The flow stress, σ, as a function of the applied strain rate, $\dot{\epsilon}$, at 650° C for the 1.6%C steel in the as-cast, after 3 cycles, after 14 cycles, and hot-and-warm rolled conditions. The reciprocal of the slope is m, the strain–rate sensitivity.

The increasing superplasticity with increasing number of cycles is demonstrated by more direct measurement in Fig. 3. In this figure, the elongation to failure at 650° C, at an engineering strain rate of 1% min^{-1}, is shown to increase with increase in the number of heat treatment cycles. The elongation to failure of the as-cast steel is about 40% and increases to 115% after 3 cycles, 264% after 8 cycles, 328% after 14 cycles and decreases slightly to 268% after 20 cycles. These results demonstrate that superplastic properties can be developed in ultrahigh carbon steels by thermal cycling alone. By comparison, the hot-and-warm worked steel has an elongation to failure of 396%.

The development of superplastic structures with increased cycling can be observed by referring to the microstructures in Fig. 4. Typical optical micrographs of the steel in the as-cast, cycled 3, 8, 14 times and the as-hot-and-warm-rolled conditions

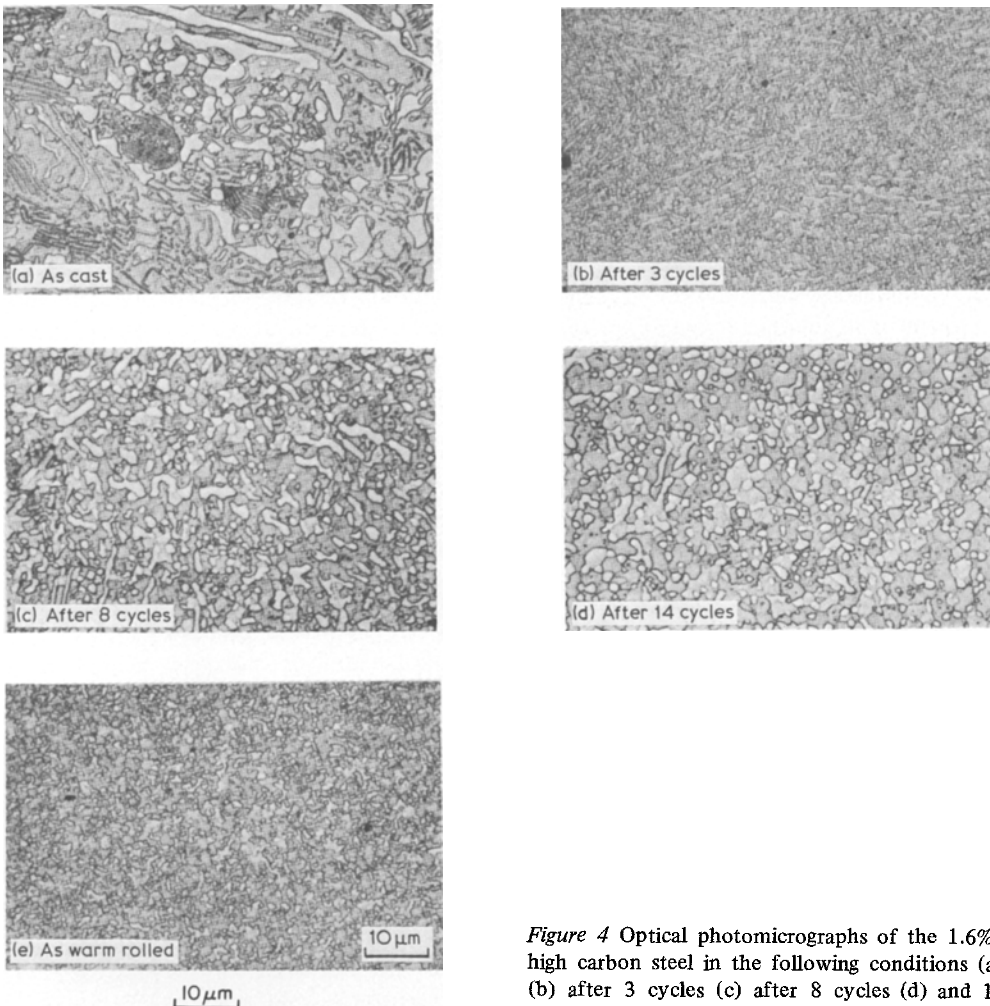

Figure 4 Optical photomicrographs of the 1.6% C ultra-high carbon steel in the following conditions (a) as-cast (b) after 3 cycles (c) after 8 cycles (d) and 14 cycles (e) after processing by hot-and-warm rolling.

are shown. It is clear that increased cycling alters the structure (Fig. 4a → 4b → 4c → 4d). The as-cast structure, exhibiting coarse cementite in a pearlitic matrix of grain size about 20 to 30 μm (Fig. 4a) has poor warm temperature ductility. After 3 cycles the structure is refined considerably (Fig. 4b). Although the structure is fine, the cementite is neither spheroidized nor uniform in shape or distribution. The accompanying warm temperature properties reflect this microstructure in that a low strain rate sensitivity ($m = 0.3$) and low elongation to failure (115%) are found. After 8 cycles, Fig. 4c, the structure is quite uniform and fine (the matrix grain size is about 4 μm). No pearlite remains but some non-spheroidized cementite is still present. At this stage the material is superplastic (elongation-to-failure of ~264%). After 14 cycles, Fig. 4d, the structure is fully spheroidized, fine-grained (2 to 3 μm) and has the best superplastic properties of the cycled materials ($m = 0.45$: elongation-to-failure: 328%). Further cycling does not improve the microstructure or the properties. The structure that was hot-and-warm rolled, Fig. 4c, is seen to be even finer (grain size ~1 μm) than the optimum one obtained by cycling. This also is reflected in the fact that the highest elongation to failure is found in material processed this way (Fig. 3).

The mechanism of refinement of the structure through heat treatment is as follows. A heat treatment from 1150° C, designed to dissolve the massive cementite, Fig. 1, results in a large prior austenite grain size, which in turn leads to a relatively coarse, quenched microstructure. In the first cycle, upon heating to a temperature just above the A_1, the grains of austenite which nucleate are prevented from growing rapidly. This is because not all the cementite is dissolved at this temperature, Fig. 1, and these undissolved carbides serve as pinning points for growing austenite grains. Upon quenching, the resulting structure is a mixture of martensite, cementite and probably a small amount of retained austenite. The martensite is now refined because of the finer prior austenite grain size. Upon cycling such a structure, there is once again an increased number of nucleation sites for austenite so that grain refinement again occurs, leading to further refinement of the transformation products upon quenching. At a certain advanced stage of refinement by cycling, the size of austenite and ferrite grains will be controlled more by grain growth than by nucleation. At this stage the cementite particle size and distribution likely becomes the controlling process for determining the minimum grain size achievable under thermal cycling. This minimum grain size through thermal cycling is not as fine as that produced by the hot-and-warm rolling route (Figs. 4d and e). This is because concurrent straining of pearlite at warm temperatures can lead to even finer cementite particle size and hence finer ferrite grain sizes. The carbide sizes and spacing, in fact, has been shown to be a unique function of temperature and strain rate of working [10].

Although the hot-and-warm rolling leads to a finer microstructure than that achievable by thermal cycling, the latter technique may offer unique applications. As an example, conventional welding of fine grained UHC steels leads to coarse microstructures in both the weld and in the heat affected zones. Thermal cycling would be a way to refine the microstructure of such zones and restore desireable mechanical properties.

4. Summary

An as-cast ultrahigh carbon steel containing 1.6% C, has been made superplastic by heat treatment alone. The heat treatment involves quenching from a temperature where all the carbon is in solution and then repeatedly cycling across the A_1 transformation temperature. The optimum number of cycles was found to be about 10 and an elongation to failure of over 300% was found at 650° C.

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