Characterization of the Solidification Structures Within the Dendritic Core of M2 High Speed Steel

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The phases which are present within the dendritic core region during the solidification of AISI type M2 high speed steel (6 W, 5 Mo, 4 Cr, 2 V) were studied and characterized by metallography and quantitative microprobe analysis. A series of samples were quenched from various temperatures during solidification, called "freezing in" the different solidification phases. The first phase observed to solidify in M2 is ferrite which contains very little carbon. As the solidification process continues, most of the liquid surrounding the ferrite transforms to austenite by virtue of a peritectic reaction which initiates at 1330°C. (L + F → A). The ferritic cores also transform at around 1330°C into an austenite plus carbide aggregate. By the time the ingot cools to 1255°C, the carbides at the center of the dendrites dissolve completely, leaving an austenitic phase of uniform carbon and alloy content. At temperatures below the solidus, very fine carbides precipitate from the austenite. No eutectoid decomposition by products such as those commonly observed in Ti1 tool steel were observed in these specimens nor in samples from a commercial ingot.

High speed steels are characterized by their hardness and wear resistance at elevated temperatures. The high hardness quality of these steels is achieved by substantial amounts of carbides which are included in the microstructure. These carbides exhibit thermal stability which allows them to remain hard at temperatures where the surrounding matrix is relatively soft.

The structures which form upon freezing of high speed steels are very important and dictate many of the properties found in the finished product. Several studies have been conducted that explore the solidification process in these highly alloyed steels. The most pertinent investigation, which studied the freezing of type M2 high speed steel, was conducted by R. Barkalow. Employing thermal analysis and extensive metallography on unidirectionally solidified laboratory specimens, Barkalow et al determined the reactions which occur during solidification. For a 0.80 pct C M2 tool steel they are as follows:

1) Delta ferrite starts to crystallize from the liquid at 1430°C, L → F.
2) At 1330°C austenite forms from the peritectic reaction, L + F → A.
3) MC type carbides begin to precipitate from the austenite and M,C carbides by a eutectic reaction. 

Many investigations have been conducted that characterize the carbides which crystallize last from the liquid and outline the dendritic structure of the as cast ingot. However, little attention has been focused on the reactions occurring within the dendritic cores. The purpose of this research is to characterize the phases present and reactions occurring within the core region during the freezing process of the M2 grade of high speed tool steel. Since it has been commonly assumed that the eutectoid reaction which occurs in Ti1 tool steel also occurs in M2, particular attention is focused on the decomposition reactions because they are not well understood in M2.

EXPERIMENTAL TECHNIQUES

Interrupted Quenching

Samples were melted and slow cooled to various temperatures in a vertical furnace under an argon atmosphere. They were then quenched to observe the structures present at the temperature from which the quenching was done. Samples were cooled at 6°C/min similar to the cooling rate observed near the surface of commercially cast tool steel ingots. This technique allowed for the indirect observation of the phases which form during the freezing of M2. This freezing technique differs markedly from that used by Barkalow where reheated unidirectionally solidified samples were used for studying the solidification reactions.

A vertical high temperature molybdenum furnace was employed for the experiments. Samples of annealed M2 bar stock* were placed in 18 mm ID alumina tubes.

* The M2 bar stock was supplied by Universal Cyclops Steel Corporation. The heat analysis of the M2 was: 0.82 pct C, 6.11 pct W, 4.95 pct Mo, 4.18 pct Cr, 1.88 pct V, 0.009 pct S, 0.070 pct P, 0.26 pct Mn, 0.31 pct Si, 0.20 pct Ni, 0.17 pct Co, 0.08 pct Cu, and 0.021 pct Al. The ingot from which the bar stock was made was a round centered square ingot 23 cm (9 in.) on a side at the bottom, 32 cm (12.6 in.) on a side at the top, 76 cm (30 in.) long and weighed 420 kg (930 lb). In addition to the bar stock, 1 cm (0.5 in.) discs cut from the bottom, center and top of the ingot were also supplied. These were cut in the as-received condition (which was sufficient to eliminate ingot cracking and permitted saw cutting) before the balance of the ingot was processed into the bar stock.

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crucibles that were supported by a tungsten basket assembly. The entire crucible and basket assembly was suspended in the furnace by a thin piece of nichrome wire. When the samples had cooled to the temperature of interest, the nichrome wire was melted locally by passing current through the wire. The sample was then quenched in a water bath placed below the sample.

The sample temperature was monitored by a Pt-Pt 10 pct Rh thermocouple placed 1/4 inch above the sample. An adjustable-range and adjustable zero strip chart recorder also was employed to record the cooling rate of the samples during freezing and prior to quenching. To minimize decarburization a protective atmosphere of high purity argon (99.998 pct) was passed through the furnace at a rate of 2200 cc/min and bubbled into oil.

Quantitative Microprobe Analysis

The compositional variations across a dendrite were measured with an electron microprobe. Analyses were performed at an operating voltage of 10 KV and a sample current of 0.05 μA. A 4 μm beam raster was used during the analysis to average secondary carbide precipitates in the matrix. Ten micron steps were taken between point counts to eliminate any chance of measuring overlapping carbon contamination layers.

Pure standards of tungsten, molybdenum, and vanadium were used along with a homogenized alloy of 4.9 pct Cr balance Fe. A meteorite (Canyon Diablo), which contained large cementite (Fe₃C) particles, was used as a carbon standard. The raw X-ray intensity ratios obtained during the analysis were converted into wt pct using a standard computer program which corrects the raw data for atomic number, fluorescence, and absorption effects. Since carbon is heavily absorbed by the other elements in M2, the mass absorption coefficients were carefully chosen and independently entered into the correction program.*

*The mass absorption coefficients for Cₙ were chosen from the data of Henke and Ehsan.

<table>
<thead>
<tr>
<th>Absorber</th>
<th>μ/p for Cₜₚ (cm²/g)</th>
</tr>
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<tbody>
<tr>
<td>Fe</td>
<td>13,300</td>
</tr>
<tr>
<td>W</td>
<td>18,750</td>
</tr>
<tr>
<td>Mo</td>
<td>33,420</td>
</tr>
<tr>
<td>Cr</td>
<td>10,590</td>
</tr>
<tr>
<td>V</td>
<td>8,840</td>
</tr>
<tr>
<td>C</td>
<td>2,373</td>
</tr>
</tbody>
</table>

Carbon contamination, which occurs as a result of electron beam and diffusion pump oil interaction, contributed a significant amount to the carbon background (~50 pct). Two forms of contamination were observed. The first form appeared as a continuous film which adhered instantaneously to the sample as it was placed in the high vacuum sample chamber whereas the second form increased at a constant rate during the analysis. The second form of contamination was reduced during the analysis by employing an air jet which floods the analysis area with a fine stream of dried air. Even though the air jet did not eliminate the contamination build up entirely, it reduced the rate of contamination considerably (~30 pct) and increased the peak to background ratio for carbon.

Since the investigation of carbon content in M2 was critical, a statistical analysis was employed to understand the data and contamination effects. A statistical method outlined by Thiesen* was used to calculate the carbon detectability limit. This limit was determined as ±0.031 wt pct at a 95 pct confidence limit for the operating conditions employed during the analysis.

PRESENTATION AND DISCUSSION OF RESULTS

Metallography

Samples of M2 were melted at 1500°C, cooled at a rate of 6°C/min to a preselected temperature and finally quenched in water as described previously. The temperatures selected for four of the samples are shown on Fig. 1 which is the pseudobinary phase diagram for M2 type tool steels developed by Barklow. The labels on the curves describe the reactions occurring at the time the samples were quenched.

Fig. 1—Plot of reaction temperature vs carbon content (Ref. 7 and 8) illustrating the phases present when the samples were quenched.

Fig. 2—Sample quenched from 1400°C; 1) ferrite, R₂ = 22; 2) austenite, R₂ = 63. Nital etch plus KMnO₄ stain. Field of view, 200 × 175 μm.
The four samples are identified on Fig. 1 as 2, 3, 4, and 5 and typical microstructures are illustrated in Figs. 2, 3, 4 and 5, respectively. Two other samples quenched from 1100°C (Fig. 6) and 900°C (Fig. 7) are not shown on Fig. 1 since they were completely solid at the time of the quench.

Figure 2 illustrates the typical microstructure for a sample which was melted, slow cooled to 1400°C, and quenched. Within the dendritic core region, there are two major microconstituents. At the center of the core, there exists the high temperature delta ferrite which is the first constituent to freeze from the liquid. It is surrounded by an austenitic phase which formed from the liquid upon quenching.

In this discussion and on the photomicrographs the austenite which existed at the high temperatures is labeled as such. After quenching to room temperature the phase had a uniform texture and grain boundary structure. It was also very hard indicating it may have transformed to martensite. It was not considered feasible to perform X-ray diffraction analysis on these small regions. The phase does not appear martensitic to the eye. Ferrite was significantly softer than this phase as determined by microhardness testing with a 100 g load. The Rockwell hardness readings reported herein were converted from the microhardness readings and are used because they are more familiar.

The next two micrographs (Figs. 3 and 4) exhibit a dark etching constituent surrounded by the light etching austenite, which forms from the peritectic reaction initiating at 1330°C, \((L + F \rightarrow A)\). The dark etching constituent at the center of the dendrite is austenite with a fine dispersion of carbides. These carbides precipitate because the alloy rich austenite can no longer accommodate all the W and Mo left by the pre-existing ferrite.

Figures 3 and 4 suggest that the peritectic reaction is divided into two parts. Part one of this reaction occurs at 1330°C when most of the liquid surrounding the delta ferrite transforms into austenite. This part of the peritectic reaction accounts for the thermal arrest which is observed during the freezing process.\(^7,8\) The second part of the reaction occurs within the ferritic cores at the center of the dendrite. As the ingot cools, the ferrite transforms into an austenite plus carbide microconstituent. Since this reaction is diffusion controlled, the alloy rich ferrite first transforms into a low carbon austenite plus carbide aggregate. The carbides must form to accommodate the additional W and Mo which existed in the high temperature ferrite. The carbon necessary for the austenite and carbide formation is obtained by diffusion which occurs at accelerated rates at these elevated temperatures (1330°C to 1270°C).

As the solidification process continues, the carbides dissolve in the austenite. Eventually the carbide phase at the center of the dendrite completely disappears during cooling until only austenite of uniform alloy and carbon content exists in the core region. This is demonstrated in Fig. 5 where the only phase present in the core region is light etching austenite. It should be emphasized that this photomicrograph, like the others, is typical of many which were examined.

The next two photomicrographs, Figs. 6 and 7, characterize the reactions occurring within the dendritic
core below the solidus temperature. It is evident from the two micrographs that as the samples are cooled below the solidus, fine carbides precipitate from the austenite throughout the dendrites and particularly at the high energy sites provided by the austenitic grain boundaries.

The cooling rate prior to quenching used for these laboratory specimens (6°C/min) was selected to produce a dendritic arm spacing similar to that found for specimens taken about 8 cm or 3 in. from the surface of a commercial ingot. The cooling history for both the laboratory and commercial specimens was therefore considered to be essentially the same and as shown in Figs. 6, 7 and 8. The microstructures of the two are also essentially the same. All the photomicrographs display the same variety of carbides within the core region: that is, carbides which precipitate from the austenite along the austenite grain boundaries and within the grains.

The trends during the solidification process of grade M2 high speed steel have traditionally been compared with the trends which exist for grade T1. The same general reactions which occur between the liquidus and solidus for T1 also have been observed for M2. However, a big difference between T1 and M2 that this work emphasizes is that in laboratory and commercial ingots of M2 eutectoid decomposition by products are not observed. In the T1 steel, the core region is composed almost entirely of eutectoid decomposition by products which appear characteristically as a heavy concentration of carbides at the dendrite centers. In contrast the cores of the M2 grade exhibited only isolated areas of heavy carbides. Instead fine carbides precipitate throughout the austenitic cores and particularly at the austenite grain boundaries as shown in Figs. 6, 7 and 8.

Quantitative Microprobe Analysis

The compositions of the various phases which were frozen in during the interrupted quenching experiments were measured with the electron microprobe. The results are given in Table I. The delta ferrite which is quenched from 1400°C (Fig. 2) exhibits, within the statistical variation of the microprobe data, a zero carbon concentration which is indicative of the C solute rejection which occurred during solidification. Figure 9(a) illustrates the variation of C concentration from austenite to ferrite to austenite across the center portion of the dendrite shown in Fig. 2. Since the remaining major alloy elements (W, Mo, Cr and V) are all ferrite stabilizers, no solute rejection occurred and the alloy concentration was observed to be essentially constant across the core region.

Table I also gives the compositions of the constituents which form after an M2 sample is melted, slow cooled and quenched from 1300°C, the structure of Fig. 3. The dark etching constituent which is an austenite plus carbide aggregate displays a slightly lower carbon content and an increased alloy content compared to the surrounding austenite which formed directly from the liquid at 1330°C. The carbon variation is probably not significant in that the expected variation in the measurement is ±0.031 wt%. As shown in Table the austenite generally contains a lesser amount of the alloying elements.

Quantitative Microprobe Analysis

The compositions of the various phases which were frozen in during the interrupted quenching experiments were measured with the electron microprobe. The results are given in Table I. The delta ferrite which is quenched from 1400°C (Fig. 2) exhibits, within the
Table I. Compositional Characterization of Selected Solidification Phases in M2 High Speed Steel (Average Composition 0.82 C, 6.11 W, 4.95 Mo, 1.88 Cr, 1.68 V)

<table>
<thead>
<tr>
<th>Element</th>
<th>Sample Quenched from 1400°C (Fig. 2)</th>
<th>Sample Quenched from 1300°C (Fig. 3)</th>
<th>Sample Quenched from 1255°C (Fig. 5)</th>
</tr>
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<tbody>
<tr>
<td></td>
<td>1) Ferrite</td>
<td>2) Austenite</td>
<td>1) Austenite + Carbide</td>
</tr>
<tr>
<td>C</td>
<td>0.0</td>
<td>0.70</td>
<td>0.49</td>
</tr>
<tr>
<td>W</td>
<td>5.4</td>
<td>5.4</td>
<td>6.74</td>
</tr>
<tr>
<td>Mo</td>
<td>3.7</td>
<td>3.7</td>
<td>4.12</td>
</tr>
<tr>
<td>Cr</td>
<td>3.8</td>
<td>3.8</td>
<td>3.57</td>
</tr>
<tr>
<td>V</td>
<td>1.5</td>
<td>1.5</td>
<td>1.98</td>
</tr>
</tbody>
</table>

Fig. 9—(a) Carbon composition across dendrite in Fig. 2. (b) Carbon composition across dendrite in Fig. 5.

As the solidification process continues, the amount of carbides at the center of the dendrite decreases until only a uniform austenitic core remains. The composition of the austenite phase, which quenched from 1255°C, Fig. 5, appears in the last column of Table I. All the alloying elements and carbon were uniform within very narrow limits across the dendritic core. Figure 9(b) illustrates the carbon concentration across the dendrite in Fig. 5. These chemical analyses show that the peritectic reaction \( L + F \rightarrow A \) which begins at 1330°C continues over a range of temperatures and is completed at 1255°C leaving only austenite in the core region.

The microprobe analysis of the austenite which was quenched from 1255°C illustrates a very important fact about the solidification process of M2. Employing cooling rates which are similar to the rates found in commercial ingots, it is discovered that the peritectic reaction which begins at 1330°C consumes all the ferrite. The ferrite first transforms into an austenite plus carbide aggregate. As cooling continues, the carbides slowly dissolve, and at 1255°C the carbides are dissolved completely leaving a uniform austenitic phase. Since ferrite is not retained during cooling, the eutectoid decomposition reaction reported by Barkalow in unidirectionally solidified specimens, which occurs at temperatures below the solidus, can not proceed. Instead, carbides precipitate from the austenite throughout the matrix and particularly at high energy sites provided by the austenite grain boundaries.

SUMMARY AND CONCLUSIONS

The phases which form during the solidification of type M2 high speed steel were studied and characterized. The first phase to crystallize was the low carbon ferrite, \( L \rightarrow F \). As the freezing process continued, most of the liquid surrounding the ferrite crystallized as austenite at 1330°C due to the peritectic reaction, \( L + F \rightarrow A \). The ferrite which exists at the center of the dendrites transformed at around 1330°C into an austenite plus carbide aggregate. The carbides formed to accommodate all the W and Mo left by the preexisting ferrite. As the freezing process continued, the carbides at the center of the dendrites completely dissolved resulting in a uniform austenitic phase. As samples are quenched from temperatures below the solidus, fine carbides precipitate throughout the austenite with higher concentrations observed at high energy sites provided by austenite grain boundaries. The as cast microstructure observed in the commercial ingot suggests that the laboratory and commercial ingots experience the same freezing reactions.

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