Identification of carbides and phase transformations in sintered Fe-Mo-Mn-C alloys produced under a slow continuous cooling

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Abstract

Different sintered alloys were produced by sintering and slow cooling of powder compacts made from pre-alloyed Fe-0.50Mo-0.15Mn powder mixed with varied graphite powder contents (0.30-1.20 wt. % with 0.15 % increment). According to microstructures, sintered alloys were divided into sintered hypo-eutectoid, near eutectoid, and hyper-eutectoid alloys. By using Groesbeck color tinting and X-ray diffraction technique, the common phase transformation products of these sintered alloys were found to include ferrite and carbides. The sintered hypo-eutectoid alloys had microstructures consisting of polygonal ferrite grains and two forms of ferrite + carbide mixtures, such as ferrite + $M_{23}C_6$ carbide and ferrite + $M_3C$ carbide. In sintered near-eutectoid alloys, only ferrite + $M_3C$ carbide mixtures occupied microstructures. In sintered hyper-eutectoid alloys, large proeutectoid $M_{23}C_6$ carbide formed first and followed by abnormal ferrite, degenerate ferrite + $M_{23}C_6$ pearlite, lamellar ferrite + $M_{23}C_6$ pearlite, Widmanstätten $M_3C$ carbide, inverse bainite (ferrite + $M_3C$) and upper bainite (ferrite + $M_3C$).

Keywords: Sintering, Fe-Mo-Mn-C alloys, $M_3C$ carbide, $M_{23}C_6$ carbide, ferrite + carbide mixtures.
1. Introduction

Phase transformations from austenite to lower temperature phases are important phenomena controlling microstructures and mechanical properties of steels (iron-based alloys) \(^1\). Better physical and mechanical properties of advanced high-strength steels (AHHSs) are required for modern automotive industry \(^2\), \(^3\). Due to steel development trend, different types of thermomechanical processes are developed to manipulate microstructural developments according to phase transformations and to achieve desirable mechanical properties in AHSSs \(^4\)–\(^6\). The common transformation products of steels include solid solution \(\alpha\)-ferrite and martensite, carbides and retained austenite. The arrangements of these products in microstructures determine different types of AHSSs \(^7\).

In powder metallurgy of steels, the performance development of sintered high strength steels (SHSSs) cannot be compared with wrought AHSSs, due to the porous nature of sintered steels. Moreover, some thermomechanical processes cannot be applied for hardening sintered steels because coolant media may be entrapped in their pores. However, strength improvement in SHSSs has been being conducted based on sinter hardening process. To achieve sinter hardening effect, the combined influences of base metal powder compositions and cooling rates of just slightly higher than a cooling rate applied industrially are essential for obtaining hard phases/structures, such as bainite and martensite \(^8\)–\(^11\).

In addition to the application of fast cooling rates to manipulate sintered steel microstructures, the concept of hardening sintered steel by using high carbon contents has not been experimentally tried. The graphite, as the typical carbon source for powder metallurgy steel \(^12\), is employed due to its influences on transformation types and products. As widely known that carbon is one of austenite stabilizing elements, different dissolved carbon contents in austenite during sintering holding are expected to influence different austenite stabilities, which lead to different phase transformations from austenite to lower temperature phases/structures. The different phase transformations mean different transformation products. Since this work employs pre-alloyed Fe-0.50Mo-0.15Mn powder as a starting material, it is also expected that different carbon contents will lead to formations of different carbides. Regarding the carbide formation in molybdenum steels, different types of carbides were previously reported in several works \(^13\)–\(^16\). It is possible that different carbides could form in sintered alloys. A better method for identifying
carbides is introduced in this work. The identified carbides are keys for understanding relevant phase transformations.

2. Experimental procedure

2.1 Materials preparation

Experimental sintered alloys were prepared from mixtures of pre-alloyed Fe-0.5Mo-0.15Mn powder (ATOMET 4001 from Rio Tinto Metal Powders of Canada) and graphite powder. The nominal compositions of sintered steels are given in Table 1. The powder mixtures were added with 1 wt. % zinc stearate as a lubricant. Blending of powder mixtures plus lubricant was conducted in a double cone tumbling mixer for 3.6 ks. The powder mixtures were compacted into tensile test bars, following MPIF Standard 10, with green density of 6.5 Mg/m$^3$. Sintering of the powder compacts was conducted in a vacuum furnace (Schmetz of Germany) at 1,553 K for 2.7 ks. The sintered specimens were continuously cooled by nitrogen gas with the rate of 0.1 K/s.

Table 1 Nominal composition of experimental sintered alloys

2.2 Characterization

Specimens for optical microscopy were prepared according to a standard procedure, including cutting, mounting, grinding (180 to 1200 grit silicon carbide papers), polishing (6, 3 and 1 μm diamond pastes) and etching. The etchants employed in this work included two types, such as Groesbeck’s reagent and 2 % Nital in ethanol. Groesbeck’s reagent (0.1 L water, 0.004 kg NaOH, 0.004 kg KMnO$_4$) was used for color tinting. Color etching was performed by immersing a specimen in the reagent at 333 K for 600 s (modified from the method given by Vander Voort $^{17}$). Microstructural observation was conducted by using Olympus STM7.

Specimens for scanning electron microscopy (SEM) were prepared according to the standard procedure given above. The specimens for SEM observation were etched by using 2 % Nital. Microstructural observation was conducted by using Hitachi SU5000.
Phase identification was carried out by using the X-ray diffraction (XRD) technique. To compare the specimen surface conditions on XRD peak intensities two specimen groups, with a polished surface and with an etched surface after polishing, were experimented. The deep etching was intended to increase the XRD peak intensities of carbides. The deep etching procedure consisted of a solution of 40% nitric acid in water for 40 seconds, followed by cleaning in a solution of 50% hydrochloric acid in water for 5 seconds, all at ambient temperature. XRD was performed by using Rigaku TTRAX III X-ray diffractometer with copper source (wavelength of 0.154 nm) and conditions including step size of 0.2°, time 0.5 s/step and angle of 30-100°.

3. Results

According to microstructural changes with varied carbon contents, experimental sintered Fe-Mo-Mn-C alloys were divided into 3 groups, based on the observed proeutectoid phases similar to those of binary plain Fe-C alloys, such as hypo-eutectoid alloys with proeutectoid ferrite, hyper-eutectoid alloys with proeutectoid carbide and near-eutectoid alloys without proeutectoid phase.

3.1 Sintered hypo-eutectoid alloys

The hypo-eutectoid alloys included those having proeutectoid ferrite as the first product of phase transformations. They included sintered 030C (Fig. 1a), 045C alloy (Fig. 1c), and 060C (Fig. 1e) alloys. They had 3 common microstructural features including polygonal ferrite (PF) grains, ferrite + needle carbide mixture, and ferrite + rod carbide mixture. Different carbide types responded to Groesbeck’s reagent by developing to different color shades, such as violet for needle carbide particles and brown for rod carbide ones. The PF grains showed no response to Groesbeck’s reagent. Thus, they appeared white in OM images. SEM images of sintered 030C (Fig. 1b), 045C alloy (Fig. 1d), and 060C (Fig. 1f) alloys clearly show three different microstructural features, such as PF grains, ferrite + needle carbide mixture and ferrite + rod carbide mixture.

Phase identification by XRD technique was employed on polished and etched specimens for each sintered alloy. The results are given in Fig. 2. Only XRD peaks corresponding to body-centered cubic (BCC) crystal structure of α-ferrite were observed for polished specimens of sintered 030C (Fig. 2a), 045C (Fig. 2b), and 060C (Fig. 2c) alloys. XRD peaks corresponding to
face-centered cubic (FCC) crystal structure of $\text{M}_{23}\text{C}_6$ carbide and to orthorhombic crystal structure of $\text{M}_3\text{C}$ carbide coexisted with strong peaks of $\alpha$-ferrite in deeply etched specimens of sintered 030C (Fig. 2d), 045C (Fig. 2e), and 060C (Fig. 2f) alloys.

The fractions of brown rod carbide particles were higher than those of violet needle carbide particles in sintered 030C (Fig. 1a) and 045C (Fig. 1c). The XRD peak intensities of $\text{M}_{23}\text{C}_6$ carbide were also higher than those of $\text{M}_3\text{C}$ carbide in sintered 030C (Fig. 2d) and 045C (Fig. 2e). The brown rod carbide particles were identified as $\text{M}_{23}\text{C}_6$ type and the violet needle carbide particles as $\text{M}_3\text{C}$ type by matching the fractions of colored carbide particles with XRD peak intensities. In sintered 060C (Fig. 1e), the fraction of brown rod carbide particles was slightly lower than that of violet needle carbide particles. The XRD intensity of $\text{M}_{23}\text{C}_6$ carbide was also slightly lower than that of $\text{M}_3\text{C}$ carbide (Fig. 2f). The brown rod carbide was identified as $\text{M}_{23}\text{C}_6$ type while the violet needle carbide was $\text{M}_3\text{C}$ type.

**Fig. 1** Color-tinted OM images of sintered (a) 030C, (c) 045C, and (e) 060C alloy alloys and SEM images of sintered (b) 030C, (d) 045C, and (f) 060C alloys.

**Fig. 2** XRD patterns of polished sintered (a) 030C, (b) 045C, and (c) 060C alloys and deeply etched sintered (d) 030C, (e) 045C, and (f) 060 alloys.

### 3.2 Sintered near-eutectoid alloys

The sintered near-eutectoid alloys included those having no proeutectoid phase. They included sintered 075C (Figs. 3a and 3b) and 090C alloy (Fig. 3c and 3d) alloys, whose microstructures mainly consisted of ferrite + needle carbide mixtures. Most needle carbide particles were tinted in violet color (Figs. 3a for sintered 075C alloy and Figs. 3c for sintered 090C alloy).

The results of phase identification by XRD technique are given in Fig. 4. Only strong XRD peaks of $\alpha$-ferrite were observed in the polished specimens of sintered 075C (Fig. 4a) and 090C (Fig. 4b) alloys. In contrast, weak XRD peaks corresponding to orthorhombic $\text{M}_3\text{C}$ carbide
coexisted with strong XRD peaks of $\alpha$-ferrite in the deeply etched specimens of sintered 075C (Fig. 4c) and 090C (Fig. 4d) alloys.

**Fig. 3** Color-tinted OM images of sintered (a) 075C and (c) 090C alloys and SEM images of sintered (b) 075C and (d) 090C alloys.

**Fig. 4** XRD patterns of polished sintered (a) 075C and (b) 090C alloys and deep-etched sintered (c) 075C and (d) 090C alloys.

### 3.3 Sintered hyper-eutectoid alloys

The presence of large proeutectoid carbide particles was the criterion for grouping sintered alloys as hyper-eutectoid alloys. Due to such criterion, the sintered hyper-eutectoid alloys included sintered 105C alloy and 120C alloys. The microstructural details of sintered hyper-eutectoid alloys are given below.

The sintered 105C alloy microstructure consisted of three different carbide particle shapes, such as large irregular, rod and lamellar, distributed in ferrite matrix (Fig. 5a). All carbide particles with different morphologies were tinted in brown color. Proeutectoid carbide existed in the form of large grain boundary particles while rod and lamellar carbides were in the form of ferrite + carbide mixtures (Fig. 5b). The XRD pattern of polished sintered 105C alloy (Fig. 6a) shows strong peaks of $\alpha$-ferrite and weak peaks of M$_{23}$C$_6$ carbide. However, the XRD pattern of deeply etched sintered 105C alloy (Fig. 6c) shows moderate peaks of M$_{23}$C$_6$ carbide and weak peaks of M$_3$C carbide in addition to the dominant peaks of $\alpha$-ferrite. The M$_{23}$C$_6$ XRD peaks are corresponding to large irregular, rod and lamellar carbide particles tinted in brown color.

The sintered 120C alloy showed the most complicated microstructure among the experimental sintered alloys. The sintered 120C alloy microstructure consisted of several components, such as large brown proeutectoid carbide particles, white abnormal ferrite regions, fine brown carbide particles, dark violet Widmanstätten carbide particles, and fine dark violet needle particles (Fig. 5c). It was revealed in the SEM image (Fig. 5d) that the fine brown carbide particles had rod and lamellar shapes. The arrangement of ferrite + brown rod carbide mixture was identified as
degenerate pearlite and that of ferrite + brown lamellar carbide mixture was identified as lamellar pearlite. The arrangements of ferrite + dark violet needle carbide mixtures were not in pearlitic style but in upper bainite and inverse bainite (Fig. 5c).

The XRD pattern of polished sintered 120C alloy (Fig. 6b) shows strong peaks of $\alpha$-ferrite and weak peaks of $M_{23}C_6$ carbide. However, the XRD pattern of deep-etched sintered 120C alloy (Fig. 6d) shows peaks of $\alpha$-ferrite, $M_{23}C_6$ and $M_3C$ phases in addition to the dominant peaks of $\alpha$-ferrite. Matching the fraction of each color-tinted precipitate (Fig. 5c) with the corresponding XRD peak intensity (Fig. 6d) revealed that the higher fraction of brown grain boundary carbide and fine brown carbide particles were corresponding to stronger intensities of $M_{23}C_6$ carbide peaks. The lower fraction of dark violet Widmanstätten carbide particles and fine dark violet needle particles were corresponding to weaker intensities of $M_3C$ carbide. The microstructural components of the sintered 120C alloy were identified as follows. The brown grain boundary carbide particles were $M_{23}C_6$ carbide. The ferrite + brown rod carbide mixture was degenerate ferrite + $M_{23}C_6$ pearlite. The ferrite + brown lamellar carbide mixture was lamellar ferrite + $M_{23}C_6$ pearlite. The dark violet Widmanstätten carbide particles were $M_3C$ carbide. The ferrite + dark violet needle carbide mixtures were upper bainite (ferrite + $M_3C$) and inverse bainite (ferrite + $M_3C$).

**Fig. 5** Color-tinted OM images of sintered (a) 105C and (c) 120C alloys and SEM images of sintered (b) 105C and (d) 120C alloys.

**Fig. 6** XRD patterns of polished sintered (a) 105C and (b) 120C alloys and deep-etched sintered (c) 105C and (d) 120C alloys.

4. Discussion

4.1 Carbide identification by combination of color tinting and XRD technique
The OM images of color-tinted carbides are useful for differentiating carbide type. In this work, the condition for color tinting by using Groesbeck’s reagent is modified with longer etching time and under higher temperature. The results show that different carbide types can be distinguished by color shades, i.e., needle carbide is tinted with violet or dark violet while rod and large gain boundary carbides are tinted with brown. The color shades obtained by this modified tinting cannot be compared with some results given in the previous work \(^{17}\) because it was mentioned that \(M_3C\) carbide did not respond to Groesbeck’s reagent.

Phase identification by using XRD technique is a common practice. However, it is demonstrated in this work that the proper preparation of detecting surface is necessary and important. For each sintered alloy, XRD specimens are prepared by two different ways, such as surface polishing and surface deep etching. Although a sintered alloy specimen has plenty of carbide particles as microstructural components, the carbide XRD peaks are hardly detected on a specimen with polished surface. This is probably due to the diffracted XRD beams from carbide particles are masked by those of \(\alpha\)-ferrite. To reduce diffracted XRD signals from \(\alpha\)-ferrite, the specimen surface should be prepared in the way that \(\alpha\)-ferrite is partly removed and carbide particles are maintained. Deep etching is selected for this purpose. It is found that carbide XRD peaks can be comfortably detected in a specimen with deeply etched surface.

This work demonstrates that the carbide identification can be implemented by matching the color tinting information with XRD peak intensity. The fraction of color-tinted carbide is directly related to carbide XRD peak intensity. By using this method, the violet or dark violet carbide particles are identified as \(M_3C\) carbide and the brown carbide particles as \(M_{23}C_6\) carbide.

### 4.2 Phase transformations deduced from identified carbides

Under a slow continuous cooling rate, phase transformations related to carbide precipitations always occur in carbon-enriched austenite zones. In sintered hypo-eutectoid \(030C\), \(045C\) and \(060C\) alloys, carbon enrichment in austenite zones occurs due to carbon diffusion from the growing PF grains to untransformed austenite zones. There are several research works showing evidence of carbon enrichment in austenite due to ferrite transformations even in those occurring at low temperatures \(^{19\text{-}21}\). The carbon concentrations in carbon-enriched austenite zones of sintered hypo-eutectoid alloys are different. Thus, austenite zones with different carbon
saturations transform to different ferrite + carbide mixtures (Fig. 7). The needle carbide particles in ferrite + needle carbide mixture in the SEM image (Fig. 7a), appearing as violet carbide particles in the OM image (Fig. 1a), are identified as M$_3$C carbide. The feature consisting of needle M$_3$C carbide particles formed in thin austenite bands between ferrite plates is according to upper bainite formation given in \cite{22}. The rod and lamellar carbide particles in ferrite + rod carbide mixture in the SEM image (Fig. 7b), appearing as brown rod carbide particles the OM image (Fig. 1a), are identified as M$_{23}$C$_6$ carbide. The arrangement of ferrite + rod carbide indicates that the involved phase transformation is degenerate pearlite transformation while that of ferrite + lamellar carbide indicates lamellar pearlite transformation. The formation mechanisms of degenerate and lamellar pearlite structures are given in \cite{23}. More comments on degenerate and lamellar pearlite structures will be given later.

**Fig. 7** Different ferrite + carbide mixtures formed in carbon-enriched austenite zones in sintered 030C alloy.

Phase transformations in sintered near-eutectoid alloys (sintered 075C and 090C alloys) occur in sintered austenite matrix and result in formation of upper bainite structure (Fig. 8). The microstructural feature of upper bainite structure indicates that carbide precipitation occurs at the second stage after ferrite plate formation cessation \cite{22}. This means that the phase transformation in sintered near-eutectoid alloys occurs with ferrite as the leading phase. In the phase transformation of eutectoid Fe-C steels, ferrite and cementite share equal leading role in pearlite formation \cite{24}.

**Fig. 8** Ferrite + carbide mixtures formed in sintered (a) 075C and (b) 090C alloys.

The phase transformations, deduced from sintered hyper-eutectoid alloys (sintered 105C and 120C alloys), are complicated. With respect to increasing carbon content, the change from M$_3$C carbide in sintered 075C and 090C alloys to M$_{23}$C$_6$ carbide in sintered 105C alloy occurs
abruptly. The $M_{23}C_6$ carbide has larger nucleation driving force among the similar carbides although it has the lowest Gibbs free energy ($\Delta G_0$), which means the lowest phase transformation driving force.

Although $M_{23}C_6$ is the major carbide found in the sintered 105C alloy, indicated by tinted brown color (Fig. 5a) and XRD pattern (Fig. 6c) but this carbide shows three different forms, such as large irregular, rod, and lamellar particles (Fig. 9a). This means that there are three phase transformations, such as proeutectoid carbide precipitation, degenerate pearlite transformation and lamellar pearlite transformation, involved with carbide forms. These three phase transformations continue to occur in sintered 120C alloy (Fig. 9b). The degenerate ferrite + $M_{23}C_6$ pearlite forms due to non-cooperative growth mode while lamellar ferrite + $M_{23}C_6$ pearlite forms due to cooperative growth mode. Mixed degenerate ferrite + $M_{23}C_6$ pearlite and lamellar ferrite + $M_{23}C_6$ pearlite as shown in Fig. 9 are also found as austenite decomposition products in sintered hypoeutectoid alloys (Fig. 7b). To the authors' best knowledge, there are a few reports on ferrite + $M_{23}C_6$ pearlite. The lamellar ferrite + $M_{23}C_6$ pearlite in a Mn-Al steel was reported to form at 923 K.

In addition to the precipitations of large proeutectoid, rod, and lamellar $M_{23}C_6$ particles in sintered 120C alloy, $M_3C$ carbide type is found to precipitate in three forms, such as Widmanstätten $M_3C$ particles (Fig. 9c), fine needle particles in upper bainite structure (Fig. 9c), and fine elongated particles in fan-like ferrite + carbide (inverse bainite) structure (Fig. 9d). The formation of Widmanstätten carbide particles with acicular (ferrite + carbide mixture) microstructure was reported to form during 7.2 ks at 773 K in the 4.12 % Cr 0.88 % C steel (in wt. %). The fan-like inverse bainite formed at 648 K in the 4.12 % Cr 0.88 % C steel and at 673 K in a high-carbon plain steel. Since the conversion of the entire inverse bainitic unit to upper bainite, named the degeneration of inverse bainite is the final stage of the transformation, it is possible that the formation of upper bainite (ferrite + $M_3C$) structure is attributed to degeneration of inverse bainite.

Thus, the cascading phase transformations with respect to decreasing temperatures can be deduced as proeutectoid $M_{23}C_6$ carbide formation, lamellar ferrite + $M_{23}C_6$ pearlite transformation, degenerate ferrite + $M_{23}C_6$ pearlite transformation, Widmanstätten $M_3C$ carbide
formation, inverse bainite (M\textsubscript{3}C + ferrite) transformation and upper bainite (degeneration of inverse bainite) transformation.

**Fig. 9** SEM images of sintered (a) 105C and (b) 120C alloys and color-tinted OM images of sintered (c) and (d) 120C alloy.

**Conclusions**

With increasing added carbon contents, phase transformations indicated by transformation products became more and more complicated. The phase transformation complication could be well-understood after carbide identification was successfully conducted by using Groesbeck color tinting and XRD technique. The phase transformations deduced from carbide information occurred according to carbon contents of sintered alloys as given below.

1. Sintered hypo-eutectoid alloys underwent ferrite transformation followed by both non-cooperative and cooperative eutectoid transformations, which yielded ferrite + M\textsubscript{3}C carbide mixture (termed upper bainite), degenerate ferrite + M\textsubscript{23}C\textsubscript{6} pearlite, and lamellar ferrite + M\textsubscript{23}C\textsubscript{6} pearlite.

2. Sintered near-eutectoid alloys underwent only non-cooperative eutectoid transformation, which yielded ferrite + M\textsubscript{3}C upper bainite.

3. Sintered hyper-eutectoid alloys showed complicated phase transformations including large proeutectoid M\textsubscript{23}C\textsubscript{6} carbide precipitation, abnormal ferrite formation, degenerate ferrite + M\textsubscript{23}C\textsubscript{6} pearlite formation, lamellar ferrite + M\textsubscript{23}C\textsubscript{6} formation, Widmanstätten M\textsubscript{3}C formation, inverse bainite formation and upper bainite formation.

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References


Table title list

Table 1 Nominal composition of experimental sintered alloys
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Figure title list

**Fig. 1** Color-tinted OM images of sintered (a) 030C, (c) 045C, and (e) 060C alloy alloys and SEM images of sintered (b) 030C, (d) 045C, and (f) 060C alloys.

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Fig. 7 Different ferrite + carbide mixtures formed in carbon-enriched austenite zones in sintered 030C alloy.
Fig. 8 Ferrite + carbide mixtures formed in sintered (a) 075C and (b) 090C alloys.
Fig. 9 SEM images of sintered (a) 105C and (b) 120C alloys and color-tinted OM images of sintered (c) and (d) 120C alloy.