

Precipitation Reactions during the Heat Treatment of Ferritic Steels

J. M. VITEK and R. L. KLUEH

The precipitation reactions in two ferritic steels, 9Cr-1Mo-V-Nb and 12Cr-1Mo-V-W, were studied. Analytical electron microscopy, optical microscopy, electrolytic extractions, and hardness measurements were used to determine the types, amounts, and effects of precipitates formed as a function of the heat treatment. The effect of variations in the austenitizing treatment was ascertained. In addition to variations in the austenitizing time and temperature, different cooling rates after austenitization were also used. Air cooling after austenitization (normalization) resulted in little precipitation in both alloys. Precipitation in the 12Cr-1Mo-V-W alloy after furnace cooling was found in all cases examined. Under certain conditions precipitation was also found after furnace cooling the 9Cr-1Mo-V-Nb alloy. However, when compared to the amount of precipitate in the fully tempered state, the 9Cr-1Mo-V-Nb showed a much greater variation in the degree of precipitation following furnace cooling. In addition, the matrix microstructure of the 9Cr-1Mo-V-Nb alloy was very sensitive to cooling rate. The precipitation reactions during tempering after a normalizing treatment were followed as a function of tempering treatment. Tempering temperatures were varied from 400 to 780 °C. The carbide precipitation was essentially complete after one hour at 650 °C for both alloys. Analytical microscopy was used to identify the precipitates. In the 9Cr-1Mo-V-Nb alloy, a combination of chromium-rich $M_{23}C_6$ and vanadium-niobium-rich MC carbides was found. The carbides in the 12Cr-1Mo-V-W alloy were identified as chromium-rich $M_{23}C_6$ and vanadium-rich MC. The results give an indication of the sensitivity of these alloys to heat treatment variations.

I. INTRODUCTION

HIGH-chromium ferritic (martensitic) steels are widely used in the electrical power-generating industries and chemical and petroleum industries.¹ They are currently being considered as candidate alloys for first wall applications in fusion reactor systems.² Two such alloys are 9Cr-1Mo-V-Nb and 12Cr-1Mo-V-W (commercially designated as Sandvik HT9). As part of the United States Department of Energy Fusion Energy Alloy Development Program, a study of the precipitation reactions in these alloys during heat treating was started, and the results are presented here. The objective of this investigation was to develop a better understanding of the precipitation behavior of these alloys as a function of heat treating conditions. Such an understanding should allow for a better interpretation of the results of radiation experiments currently in progress.

The physical metallurgy of the 12Cr-1Mo-type alloys has been reviewed.^{1,3-5} On the other hand, very little information has been reported for the 9Cr-1Mo-type alloys,⁵ especially for alloys that contain strong carbide-forming elements (*e.g.*, vanadium or niobium). Although there have been extensive tempering studies on the 12Cr-1Mo-type alloys, few detailed precipitation studies have been reported. What precipitation studies have been made on 9 and 12Cr alloys generally indicate that the primary precipitate that forms in both is $M_{23}C_6$; small amounts of MC, and in some cases M_2X , have also been reported.^{1,3-10}

These ferritic steels are commonly used in a "normalized and tempered" condition. This heat treatment involves a solutionizing treatment (austenitizing) that produces aus-

tenite and dissolves the carbides, followed by an air cool that transforms the austenite to martensite. The austenitization plus air cool is referred to as the normalization treatment. Typical normalized microstructures, which consist of a martensite lath network, are shown in Figure 1. In commercial practice, the normalized steels are tempered by heating between 650 to 780 °C. Although the optical microstructures after tempering appear similar to the normalized microstructure (Figure 2(a)), transmission electron microscopy shows a variation in structure, depending on the tempering conditions. For the high-temperature tempering treatment generally used (650 to 780 °C), the "tempered martensite" consists of a ferrite matrix with carbide precipitates (Figure 2(b)).

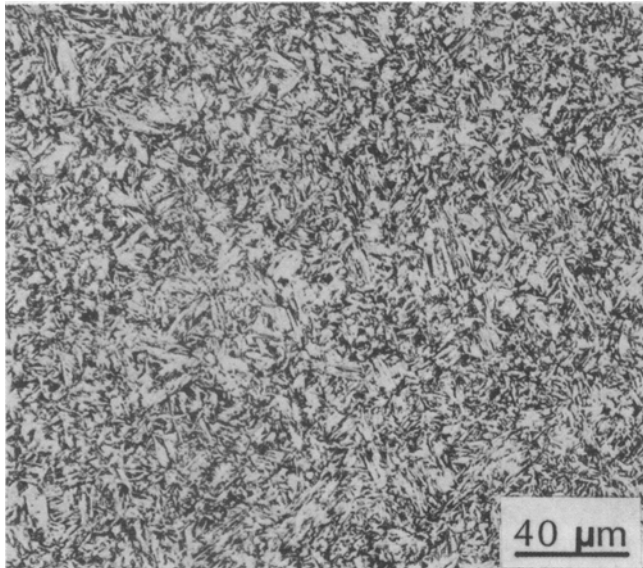
In the present investigation, the carbide dissolution kinetics were studied by varying both the austenitizing time and temperature. In addition, the sensitivity of these alloys to cooling rate after austenitization was examined. The precipitation reactions were followed during tempering by varying both the tempering time and temperature.

II. EXPERIMENTAL PROCEDURES

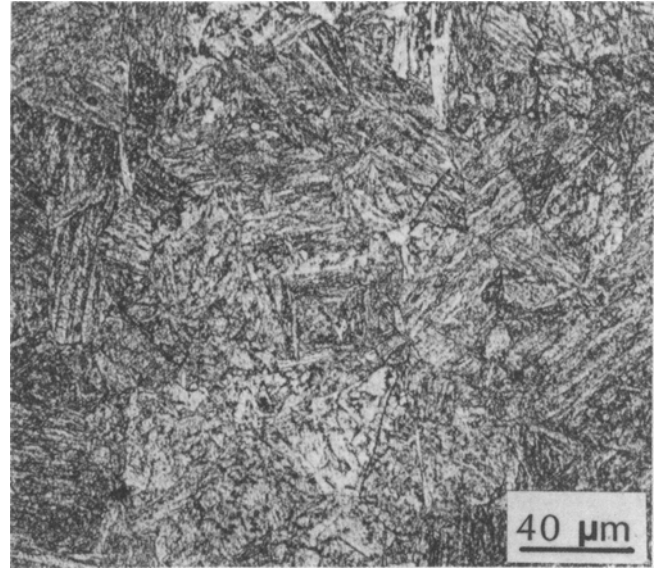
The two alloys investigated were 9Cr-1Mo-V-Nb and 12Cr-1Mo-V-W; compositions are given in Table I. Sheet stock 0.76 mm thick was used in all cases except for transmission electron microscopy, where 0.25-mm-thick sheet was used. The sheet was initially in a cold worked condition. All heat treating was done in a helium atmosphere furnace. Temperatures were monitored to within ± 2 °C. Two cooling rates were employed. For the normalizing treatment, "air cooling" was simulated by a flowing helium atmosphere in a water-cooled jacket. In fact, the cooling rate in the flowing helium atmosphere was somewhat faster than that normally found during an air cool. The time to cool from roughly 1000 to 100 °C was a few minutes. For slow

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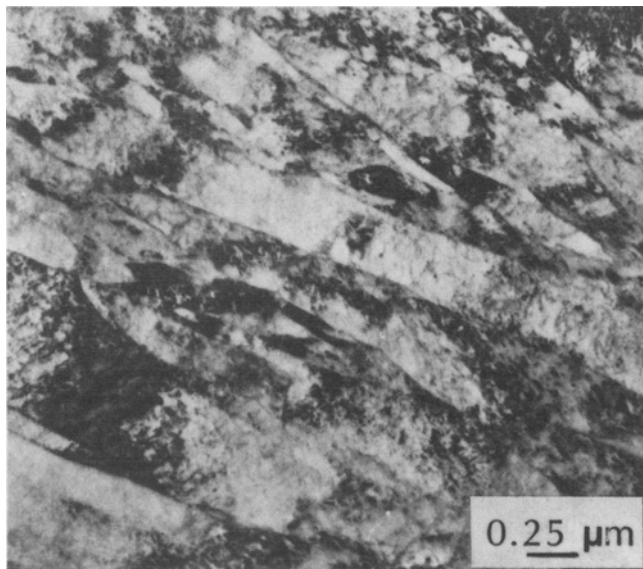
This paper is based on a presentation made at the "Peter G. Winchell Symposium on Tempering of Steel" held at the Louisville Meeting of The Metallurgical Society of AIME, October 12-13, 1981, under the sponsorship of the TMS-AIME Ferrous Metallurgy and Heat Treatment Committees.



(a)

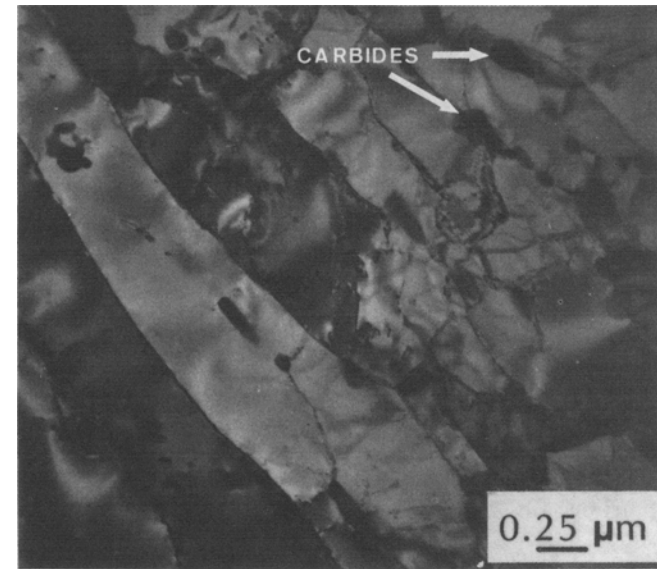


(a)



(b)

Fig. 1—Typical microstructure of normalized (1000 °C/1 h/AC) 12 Cr-1 Mo-V-W, (a) optical and (b) electron micrograph.



(b)

Fig. 2—Typical microstructure of normalized and tempered (1050 °C/0.5 h/AC + 780 °C/2.5 h/AC) 12 Cr-1 Mo-V-W, (a) optical and (b) electron micrograph.

Table I. Compositions (in Wt Pct) of the Alloys Investigated

Element	9 Cr-1 Mo-V-Nb	12 Cr-1 Mo-V-W
Chromium	8.62	11.99
Molybdenum	0.98	0.93
Carbon	0.09	0.21
Nitrogen	0.05	0.02
Nickel	0.11	0.43
Manganese	0.36	0.50
Tungsten	0.01	0.54
Vanadium	0.21	0.27
Niobium	0.06	0.02

cooling rates, the specimens were furnace cooled* in an

*In commercial practice, a slow cool (furnace cool) is called an "anneal" or "full anneal."

essentially static helium atmosphere, with the time to cool from 1000 to 100 °C on the order of 12 hours.

The austenitizing and tempering treatments examined were similar to those being used in the fusion alloy development program. The four different austenitizing conditions studied were: 1000 °C for one hour, 1040 °C for five minutes, 1040 °C for one hour, and 1100 °C for one hour. The specimens were subsequently air cooled or furnace

cooled. Tempering treatments varied from 400 °C for six hours to 780 °C for two and a half hours.

The precipitation (or dissolution) processes were followed by means of optical microscopy, analytical electron microscopy, electrolytic extractions, and hardness measurements. As thin sheet material was used, the hardness measurements were carried out using either a Rockwell F scale on the surface or a diamond pyramid indicator on the polished cross section. Multiple measurements were made on each sample.

Electrolytic extractions were made on samples with an approximate mass of 0.3 g. The extractions were produced in a solution of 10 pct HCl-90 pct methanol at 1.5 V for five hours.¹¹ These conditions insured selective removal of the matrix without any dissolution of carbides. The measurements of precipitate weight were made in air, and the values were converted to weight-in-vacuum in order to correct for atmospheric variations. The accuracy of the extraction results was determined by a series of multiple measurements on similar material. A value of $2\sigma = \pm 0.2$ wt pct was found for the range of 0 to 4 wt pct precipitate.

Electron microscopy was performed on a JEOL 100CX transmission electron microscope, equipped with an X-ray energy dispersive spectrometer (EDS) and modified for analytical work.¹² Thinned foil specimens were made from 0.25 mm thick sheet. Extraction replicas on copper grids were used for precipitate analysis in order to avoid matrix effects when obtaining EDS spectra. Precipitates were identified by a combination of electron diffraction and EDS analysis. Compositional analyses were performed by a standardless technique described in detail elsewhere.¹³

III. RESULTS AND DISCUSSION

A. Austenitization Variations

The results of hardness measurements and electrolytic extractions as a function of austenitization conditions are given in Table II. Considering the air cooled results first, it

Table II. Hardness and Extraction Results on 9 Cr-1 Mo-V-Nb and 12 Cr-1 Mo-V-W Steels as a Function of Austenitization Treatment

Austenitization Conditions		Air Cool		Furnace Cool	
(°C)	Time	Hardness (R _F)	Precipitate (Wt Pct)	Hardness (R _F)	Precipitate (Wt Pct)
9 Cr-1 Mo-V-Nb ^a					
1000	1 h	18.2	0.0	8.3	1.0
1040	5 min	17.9	0.0	^b	1.0
1040	1 h	17.9	0.0	16.3	0.3
1100	1 h	17.5	0.0	18.3	0.0
12 Cr-1 Mo-V-W ^c					
1000	1 h	21.9	0.3	19.4	1.1
1040	5 min	22.2	0.1	20.4	0.3
1040	1 h	22.5	0.0	20.1	0.2
1100	1 h	22.2	0.0	19.6	0.3

^aInitial cold worked hardness = 13.2; pct precipitate = 1.5.

^bToo low to measure.

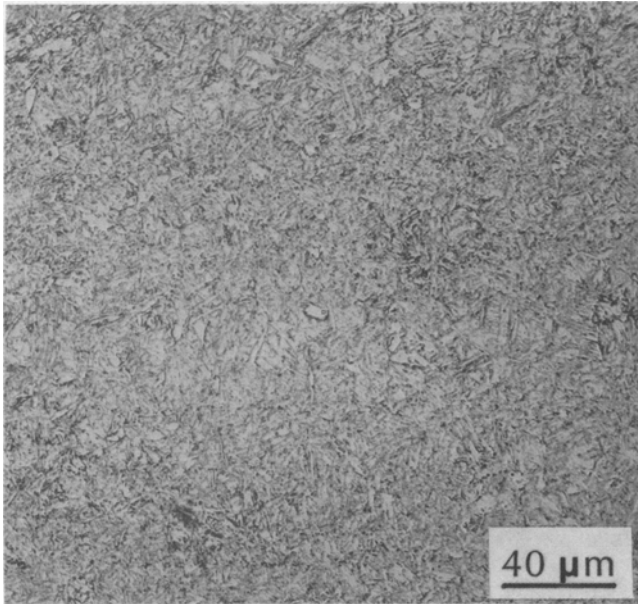
^cInitial cold worked hardness = 17.2; pct precipitate = 3.7.

is apparent that within the accuracy of the extraction technique, all of the conditions examined result in complete carbide dissolution in the 9Cr-1Mo-V-Nb alloy (*i.e.*, no precipitate was extracted). The results indicate carbide dissolution is slightly more sluggish in the 12Cr-1Mo-V-W alloy, with a small amount of carbide still undissolved after the shortest time and lowest temperature treatments. For the conditions investigated, the hardness values are essentially constant and independent of the austenitization treatment. The change in hardness due to incomplete dissolution of carbides in the 12Cr-1Mo-V-W after 1000 °C/1 h/AC is too small to result in a significant difference. Since the carbon level is significantly higher in the 12Cr-1Mo-V-W alloy (0.21 pct vs 0.09 pct), the hardness of the as-normalized martensite in 12Cr-1Mo-V-W is noticeably greater.

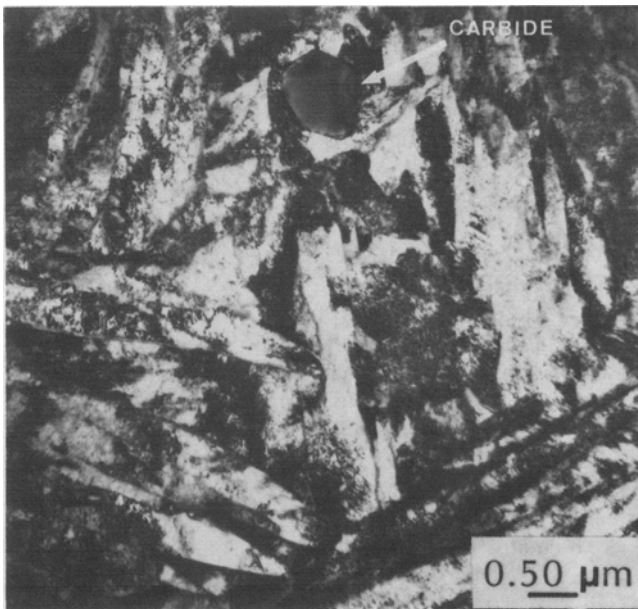
The results from furnace-cooled samples are quite different. Considering the 12Cr-1Mo-V-W alloy first, some precipitation occurs during the furnace cooling, as one might expect. After the 1000 °C/1 h treatment, a significant amount of carbide has precipitated during furnace cooling (1.1 pct). The carbide precipitation during furnace cooling is also reflected in lower hardness values compared to air cooling results. However, even with the very low cooling rates, the microstructure of the 12Cr-1Mo-V-W alloy is essentially unchanged. As shown in Figure 3, the structure still consists of martensite laths with some precipitates. The amount of carbide found after furnace cooling varied as a function of austenitizing time and temperature, decreasing with increasing time or temperature. The same trend was found for the 9Cr-1Mo-V-Nb alloy, and possible explanations are discussed later.

The results on the 9Cr-1Mo-V-Nb alloy are somewhat more complicated. After the lower temperature or shorter time austenitization treatments, a major fraction of the carbide has precipitated during furnace cooling (1 pct compared to a fully tempered value of 1.5 pct). However, longer austenitization times or higher austenitization temperatures resulted in less carbide precipitation during cooling. In fact, after 1100 °C/1 h, no carbide formed during furnace cooling. The hardness values showed a large decrease as the austenitizing time or temperature decreased. This decrease in hardness with lower austenitization temperatures is too large to be attributed only to a smaller amount of dissolved carbon in the matrix. Electron microscopy revealed that in addition, the matrix structure was affected by the austenitizing treatment. Whereas the structure after the 1100 °C/1 h/furnace cooling treatment is fully martensitic (Figures 4(a) and 4(b)), the matrix structure after the shorter time, lower temperature austenitization treatments consists of a ferrite/martensite mixture (Figures 4(c), (d), and (e)). In fact, after austenitizing at 1000 °C/1 h and furnace cooling, the matrix was 100 pct ferrite. The dislocation density in the ferrite matrix also decreased as the austenitization time or temperature decreased. Significant precipitation is also readily apparent.

Analytical electron microscopy was used to identify the precipitated carbides following furnace cooling. For the 12Cr-1Mo-V-W alloy, the carbides found were similar to those found after tempering, and will be described later. In the case of the 9Cr-1Mo-V-Nb alloy, in addition to the same carbides found after tempering (also described later), chromium-rich precipitates were found, in the form of platelets a few tenths of microns in size. These precipitates were



(a)



(b)

Fig. 3—Microstructure of 12 Cr-1 Mo-V-W after austenitizing at 1000 °C/1 h and furnace cooling, (a) optical and (b) electron micrograph.

identified as Cr_2N by electron diffraction. The lattice parameters for the hexagonal Cr_2N structure were found to be $a = 0.280$ nm and $c = 0.445$ nm. A typical EDS spectrum is presented in Figure 5, and indicates some molybdenum is present as a substitution for chromium. The Cr_2N precipitate was not found in the 12Cr-1Mo-V-W specimens, where the nitrogen level is lower than in 9Cr-1Mo-V-Nb (see Table I).

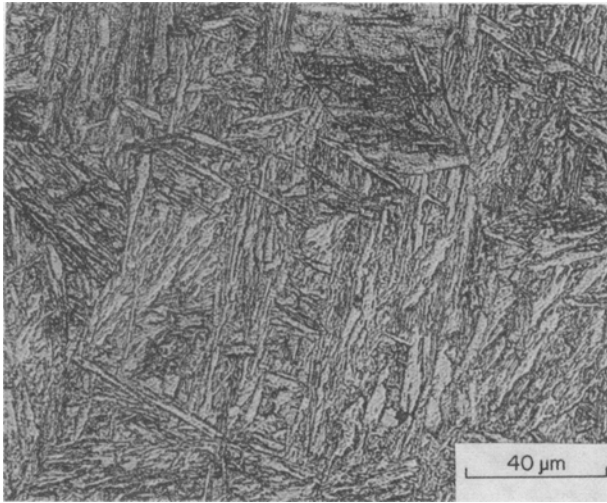
The results of the austenitization treatment variations reveal the rapid dissolution kinetics of the carbide precipitates. Within the accuracy of the extraction results ($2\sigma = \pm 0.2$ wt pct), nearly complete dissolution occurs within

five minutes at 1040 °C (see air cooled results in Table II). However, some carbide precipitation during furnace cooling was found for both alloys. In addition, it was found that the 9 Cr-1 Mo-V-Nb alloy was more sensitive to cooling rate. This is a reflection of the lower hardenability of alloys that contain less chromium and carbon.⁵ For 9 Cr-1 Mo-V-Nb the curve for ferrite formation in a TTT diagram is shifted to shorter times, enabling the formation of ferrite during furnace cooling.

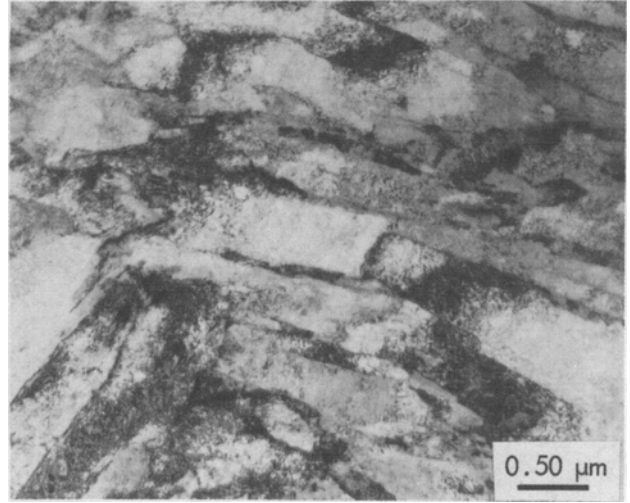
For both alloys, less precipitation occurred during furnace cooling after austenitization treatments at higher temperatures and/or longer times. There are two possible explanations for this result: austenite grain size variations and incomplete homogenization. Since austenite grain boundaries act as inhomogeneous nucleation sites, the austenite grain size can affect the precipitation kinetics during furnace cooling. Figure 6 shows the effect of austenitizing treatment on the prior austenite grain size. As the austenitization time or temperature is increased, the austenite grain size increases noticeably. Therefore, with increasing austenitizing time or temperature, the available preferred nucleation sites for precipitation during cooling decrease, and the degree of precipitation after short time or low temperature austenitization treatments may also be attributed to incomplete homogenization. The extraction results indicate little or no precipitate is present after the short time/low temperature austenitization treatments. However, the extraction techniques used are probably not capable of detecting extremely fine precipitates. In addition, the extraction techniques cannot detect areas where the carbides have dissolved but inhomogeneous concentration distributions still remain. Therefore, it is possible that complete homogenization occurs only at the higher temperatures and/or longer times. In those cases where complete homogenization is not achieved, precipitation during furnace cooling would be significantly faster (*i.e.*, precipitates can nucleate more easily in the regions of inhomogeneity). This hypothesis is supported by the observation of carbide precipitates within the prior austenite grains, not just at austenite grain boundaries (see Figures 6(a) and (b)). These intragranular sites are where extensive precipitation of carbides at martensitic lath boundaries existed prior to austenitization.

B. Tempering Treatment Variations

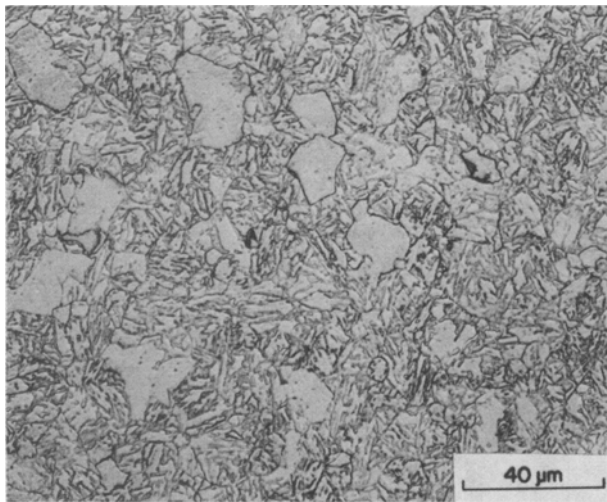
The normalization treatments used before tempering were: 1040 °C/0.5 h/AC for 9 Cr-1 Mo-V-Nb and 1050 °C/0.5 h/AC for 12 Cr-1 Mo-V-W. The earlier results on normalization variations indicate these treatments result in complete carbide dissolution. The weight percent precipitate as measured by electrolytic extractions was determined after tempering at temperatures of 650 to 780 °C. The results for both alloys are plotted in Figure 7 and tabulated in Table III. The amount of precipitate in the fully tempered condition is 1.5 wt pct for 9 Cr-1 Mo-V-Nb and 3.6 wt pct for 12 Cr-1 Mo-V-W, in agreement with the recent results of Sikka *et al.*¹⁰ The amount of precipitate is directly related to the carbon level. The precipitation during tempering is complete within one hour at 650 to 780 °C for 12 Cr-1 Mo-V-W, whereas precipitation is complete within one hour for 9 Cr-1 Mo-V-Nb over the range 700 to 780 °C.



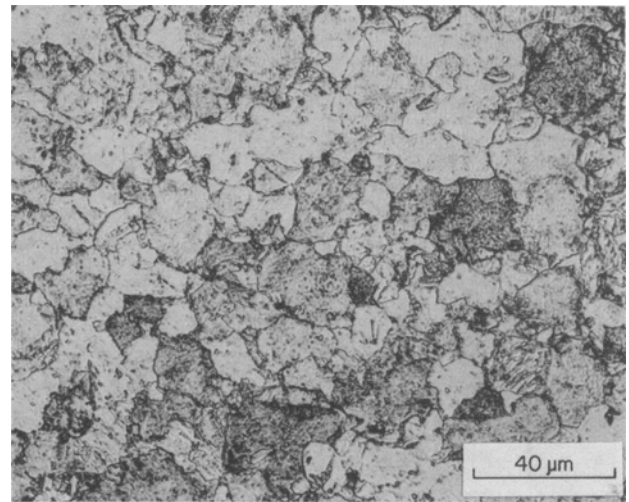
(a)



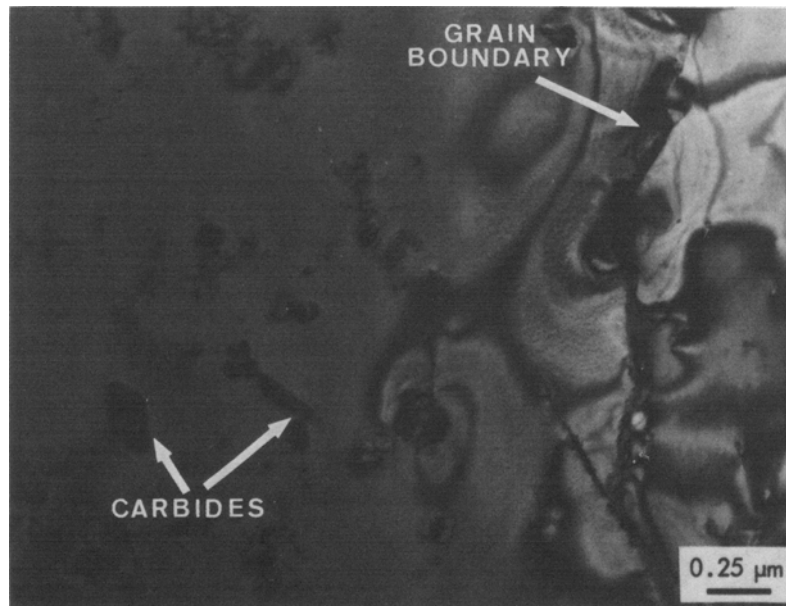
(b)



(c)



(d)



(e)

Fig. 4—Microstructures of 9 Cr-1 Mo-V-Nb after furnace cooling as a function of austenitizing treatment, (a) and (b) 1100 °C/1 h, (c) 1040°C/1 h, (d) and (e) 1000 °C/1 h.

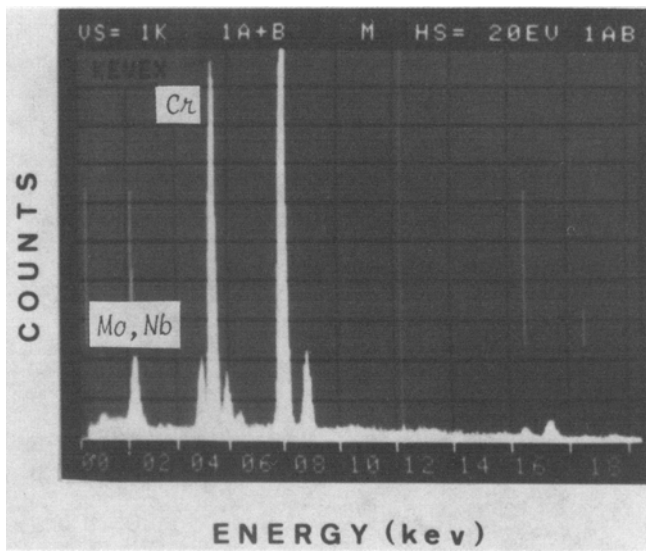
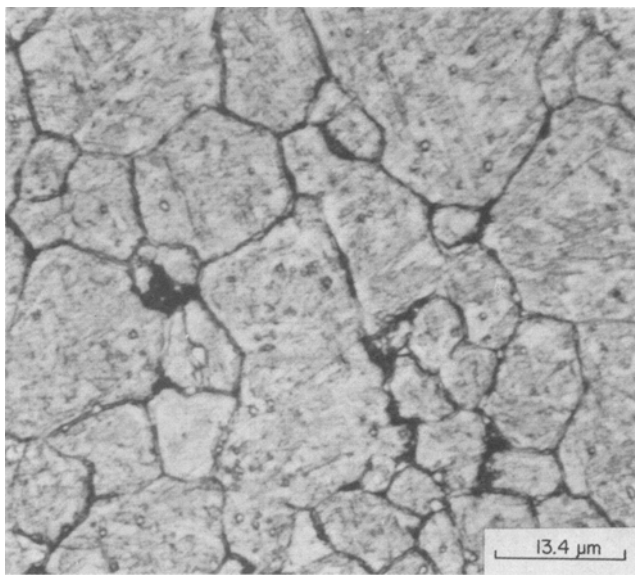
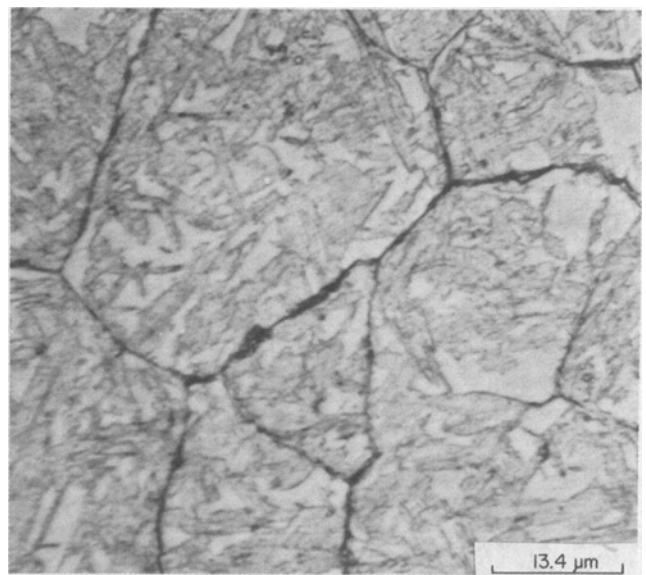


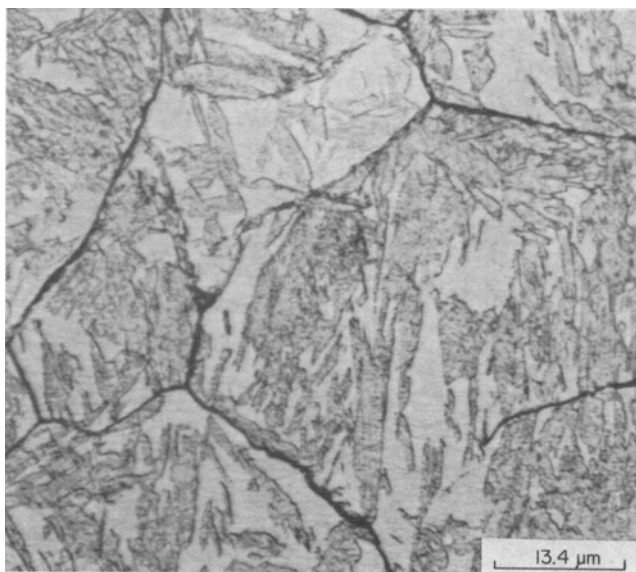
Fig. 5—EDS spectrum of chromium-rich precipitate found in 9 Cr-1Mo-V-Nb after austenitizing 1000 °C/1 h and furnace cooling. Peaks at 8.0 and 8.9 keV are from the copper replica grid.



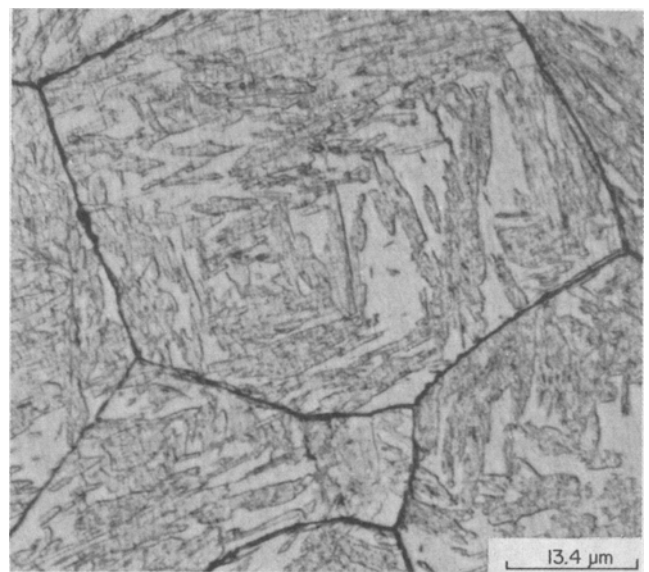
(a)



(b)



(c)



(d)

Fig. 6—Microstructures of 12 Cr-1 Mo-V-W after furnace cooling as a function of austenitizing treatment, (a) 1000 °C/1 h, (b) 1040 °C/5 min, (c) 1040 °C/1 h, and (d) 1100 °C/1 h.

Table III. Extraction Results on 9 Cr-1 Mo-V-Nb and 12 Cr-1 Mo-V-W as a Function of Tempering Treatment

Alloy	Tempering Treatment	Wt Pct Precipitate
9 Cr-1 Mo-V-Nb	650 °C/1 hour	1.0
	650 °C/2.5 hours	1.4
	700 °C/1 hour	1.4
	760 °C/1 hour	1.5
	780 °C/1 hour	1.5
	780 °C/2.5 hours	1.4
12 Cr-1 Mo-V-W	650 °C/1 hour	3.5
	650 °C/2.5 hours	3.6
	700 °C/1 hour	3.4
	760 °C/1 hour	3.5
	780 °C/1 hour	3.8
	780 °C/2.5 hours	3.7

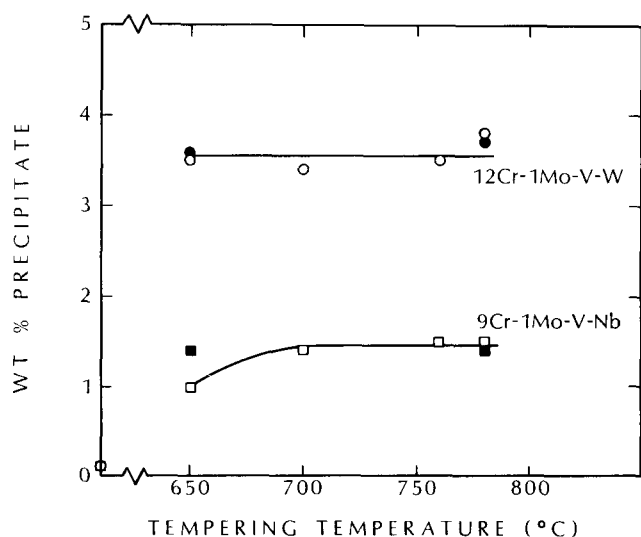
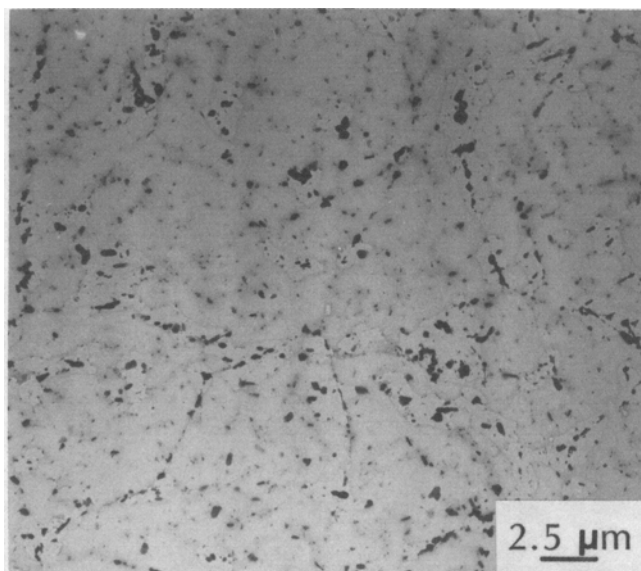


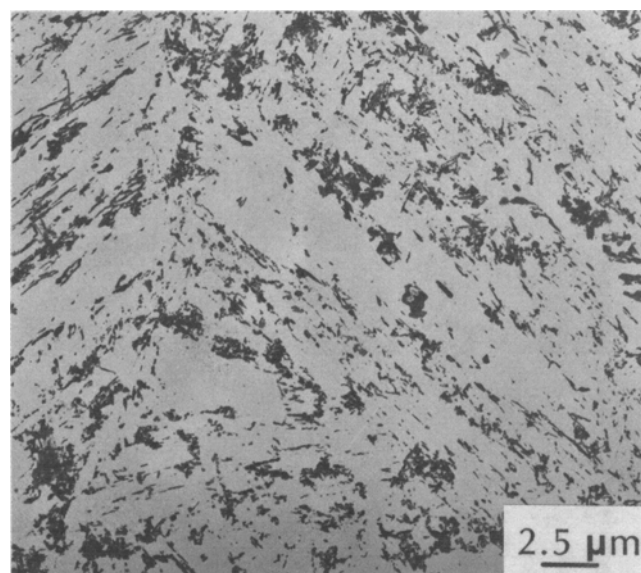
Fig. 7—Weight percent precipitate determined by electrolytic extraction vs tempering temperature. Tempering times were 1 h (open symbols) and 2.5 h (closed symbols). One hour tempering curves are drawn.

Electron microscopy on carbon film extraction replicas was performed to identify and evaluate the precipitates. A typical extraction replica of each alloy showing the size and spatial distribution of precipitates is given in Figure 8. The majority of the precipitates in both alloys, including all of the larger precipitates, were found to be $M_{23}C_6$. These carbides were rich in chromium and iron as shown in a typical EDS spectrum given in Figure 9(a). Analysis of several spectra yielded an approximate composition for the major metal components in $M_{23}C_6$ of (in wt pct): 63 pct Cr, 29 pct Fe, 7 pct Mo. Small amounts (<2 pct) of vanadium, manganese, and tungsten were also detected in the 12 Cr-1Mo-V-W.

In addition to $M_{23}C_6$, niobium- and vanadium-rich MC carbides were found in the 9 Cr-1 Mo-V-Nb alloy. The ratio of niobium to vanadium content varied over nearly the entire range of 0 to 1. A typical vanadium/niobium rich EDS spectrum is given in Figure 9(b). MC carbides were also found in 12 Cr-1 Mo-V-W alloys. However, in this alloy, with a considerably lower niobium content, the MC carbides were all vanadium rich and niobium was a minor constituent. A typical vanadium-rich carbide EDS spectrum is given in Figure 9(c).



(a)

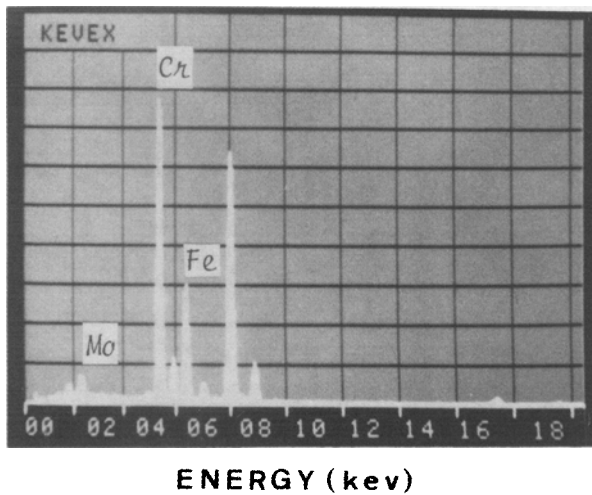


(b)

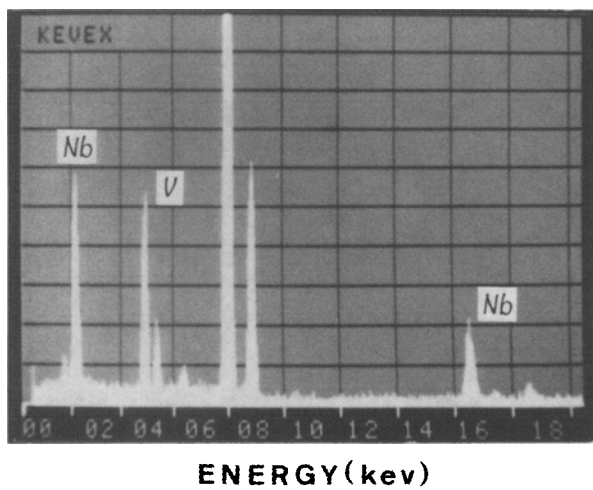
Fig. 8—Extraction replicas of normalized and tempered (a) 9 Cr-1 Mo-V-Nb (1040 °C/0.5 h/AC + 780 °C/2.5 h/AC) and (b) 12 Cr-1 Mo-V-W (1050 °C/0.5 h/AC + 780 °C/2.5 h/AC).

Hardness was measured as a function of tempering treatment for normalized steels tempered between 400 to 780 °C. The results are presented in Figure 10. A Holloman-Jaffe (Larson-Miller) parameter^{2,3,14} may be used to combine time and temperature into one parameter. This allows results from a range of tempering times and temperatures to be plotted conveniently in one curve. Such a plot for the two alloys is given in Figure 11.

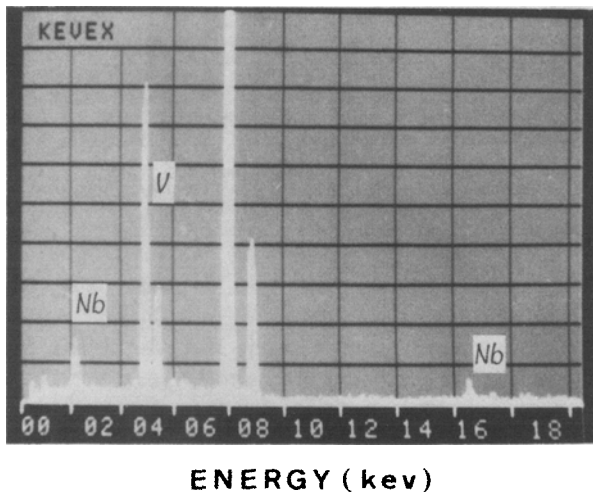
Beyond the small increase in hardness due to secondary hardening, the hardness decreases with increasing tempering time or temperature. However, the extraction results show that the degree of precipitation does not change very much over the range of tempering times and temperatures investigated. Therefore, the decrease in hardness cannot be



(a)



(b)



(c)

Fig. 9—Typical EDS spectra for (a) $M_{23}C_6$, (b) vanadium/niobium-rich MC in 9 Cr-1 Mo-V-Nb, and (c) vanadium-rich MC in 12 Cr-1 Mo-V-W. Peaks at 8.0 and 8.9 keV are from the copper replica grid.

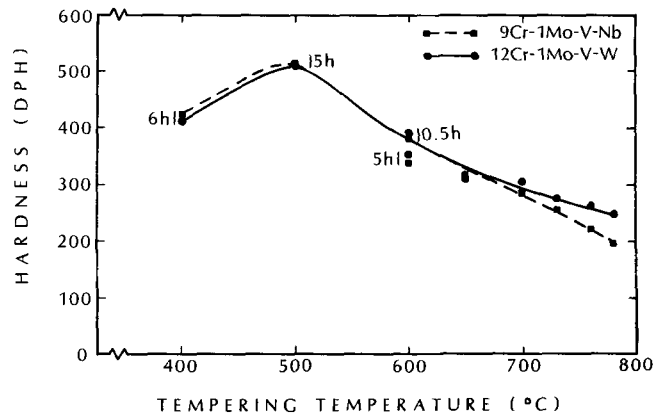


Fig. 10—Hardness vs tempering temperature for 9 Cr-1 Mo-V-Nb and 12 Cr-1 Mo-V-W. Except as noted, tempering times were 1 h.

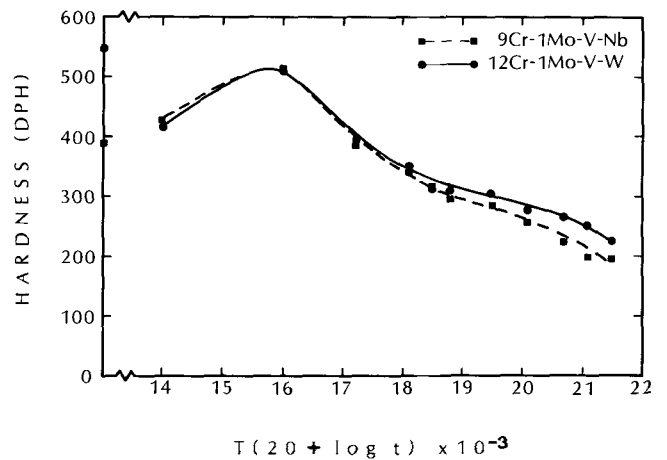


Fig. 11—Hardness vs Holloman-Jaffe parameter for both 9 Cr-1 Mo-V-Nb and 12 Cr-1 Mo-V-W. T is temperature in degrees Kelvin and t is time in hours.

attributed to additional precipitation. Although this effect was not examined further, it is presumed to be due to a combination of precipitate coarsening and cell rearrangement within the lath network.

IV. CONCLUSIONS

The effect of heat treatment on 9 Cr-1 Mo-V-Nb and 12 Cr-1 Mo-V-W was investigated. The following conclusions can be drawn:

1. The kinetics of carbide dissolution during austenitization are quite rapid. Treatments of 1000 °C/1 h or 1040 °C/5 min are sufficient to dissolve essentially all of the carbides.
2. Longer times and/or higher temperatures during austenitization are necessary to avoid significant precipitation during furnace cooling after austenitizing. More complete homogenization and/or a coarser grain size after such treatments help minimize the carbide precipitation during furnace cooling.

3. Completely martensitic microstructures resulted for both 9 Cr-1 Mo-V-Nb and 12 Cr-1 Mo-V-W steels when air cooled from the austenitizing temperature. However, after certain austenitizing treatments, furnace cooling the 9 Cr-1 Mo-V-Nb resulted in ferrite formation, whereas the 12 Cr-1 Mo-V-W alloy was entirely martensitic under similar conditions.
4. Extraction results have shown that the precipitation reactions during tempering are essentially complete in both alloys after 650 °C/1 h. Analytical electron microscopy has shown the majority of carbides in both alloys are chromium-rich $M_{23}C_6$. Additional MC carbides were found. In 12 Cr-1 Mo-V-W, the MC carbides were vanadium rich whereas in 9 Cr-1 Mo-V-Nb the MC carbides were vanadium and/or niobium rich.
5. The decrease in hardness with increasing tempering time or temperature could not be attributed to further carbide precipitation. Rather, carbide coarsening or cell rearrangement of the lath structure are assumed responsible for the continuous decrease in hardness.

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