Resistance to temper softening of microalloyed low carbon steels

Yongjie Zhang[1*], Chao Zhao[2], Goro Miyamoto[1], Kunio Shinbo[1] and Tadashi Furuhara[1]

[1] Institute for Materials Research, Tohoku University, Sendai 980-8577, Japan
[2] China Iron & Steel Research Institute Group, Beijing 100081, China

Abstract: In this study, the resistance to temper softening of martensite by small amounts of strong carbide-forming elements were investigated. A series of Fe-0.1C-1.5Mn-0.05Si (mass %)-based alloys microalloyed with V, Nb or Ti were used. Comparative study was carried out through hardness measurement of the alloys after quenching and tempering at various temperatures for various times. Different from the monotonous softening by tempering of the base alloy, hardness increment from the base alloy occurs in all the microalloyed steels, especially at higher temperatures or with longer tempering times. Further scanning electron microscopy observation reveals small amounts of V, Nb and Ti additions do not have significant influence on martensitic structure. Through atom probe analysis, it was found that nano-sized alloy carbide is formed in microalloyed steels tempered at higher temperatures, which qualitatively explains their hardness increment from the base alloy. In contrast, although almost no alloy carbide can be observed in those steels tempered at lower temperatures, the microalloyed steels still represent some resistance to softening, which might be caused by the factors other than precipitation hardening by alloy carbide.

1. INTRODUCTION

As-quenched martensite formed in carbon steels possesses uniquely high strength but suffers from low ductility and toughness due to its diffusionless character. Quenching and subsequent tempering to mitigate brittleness is the most basic way to modify the properties of steels for various structural applications. During the tempering of low-carbon and medium-carbon lath martensite, addition of substitutional alloying elements results in some degrees of hardness increment, which is called as resistance to temper softening [1]. Those alloying effects were found to be related to the suppressed coarsening of cementite, retardation of dislocation recovery, and precipitation hardening of fine alloy carbide. However, such hardening is normally obtained when large amounts of alloy elements, e.g. Mn [1-3], Si [1,3-4], Al [2], Cr and Mo [1,5], are added. In this respect, microalloy additions of V, Nb and Ti were also found to be effective against the temper softening [6].

In this study, the roles of strong carbide-forming elements (M; M = V, Nb, Ti) in small amounts on the resistance of temper softening were systematically investigated. Through combination of hardness measurement and microstructural characterization, we try to clarify the origin of hardness change by microalloying during tempering of low carbon steels.

2. EXPERIMENTAL

A series of Fe-0.1C-1.5Mn-0.05Si (mass %)-based alloys were used in this study, whose chemical compositions are listed in Table 1. Hereafter they will be called in shortened names, e.g. the Base, 0.1V, 0.4 alloys, etc. All the alloys with the dimensions of $\sim 5 \times 5 \times 3$ mm^3 were solutionized at 1473 K or 1523 K for 0.6 ks in an electric vacuum furnace to dissolve all the added M into austenite, and then directly quenched into water to obtain full martensitic structure. Afterwards, the specimens were tempered in a salt bath at various temperatures (623 K $\sim$ 923 K) for various times (60 s $\sim$ 43.2 ks). Vickers hardness of these quenched and tempered specimens was measured under the load of 9.8 N, while the microstructure was characterized by optical microscopy and scanning electron microscopy (SEM: JEOL JSM-7001F). In order to reveal the microstructural evolutions in nano-scale of the alloys during tempering, microtips prepared by focused ion beam (FIB) were measured by three-dimensional atom probe (3DAP: CAMECA LEAP-4000HR), with the conditions of 50 K for specimen temperature, 20 % for pulse fraction and 200 kHz for pulse rate.

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* Corresponding author. E-mail: yongjie@imr.tohoku.ac.jp, telephone: +81 22 215 2049.

Table 1.   Chemical compositions (mass %) of the alloys used in this study.

Alloy	C	Mn	Si	V	Nb	Ti	N
Base	0.08	1.50	0.05	<0.003	<0.003	<0.002	0.0009
0.1V	0.08	1.51	0.05	0.10	<0.003	<0.002	0.0007
0.4V	0.07	1.49	0.05	0.43	<0.003	<0.002	0.0010
0.1Nb	0.08	1.50	0.05	<0.003	0.10	<0.002	0.0008
0.1Ti	0.07	1.50	0.05	<0.003	<0.003	0.09	0.0008

3. RESULTS AND DISCUSSION
3.1. Hardness

With similar as-quenched hardness of these alloys, Fig. 1(a) ~ (d) shows their hardness variations with tempering at 623 K, 723 K, 823 K and 923 K for various times, respectively. Compared with the continuous softening by tempering of the Base alloy, hardness of tempered martensite becomes increased by small amounts of V, Nb and Ti additions, especially at higher temperatures or with longer times. In addition, such hardening effects are more significant when more amount of V is added, while secondary hardening occurs in the 0.4V alloy tempered at 923 K for 0.3ks.
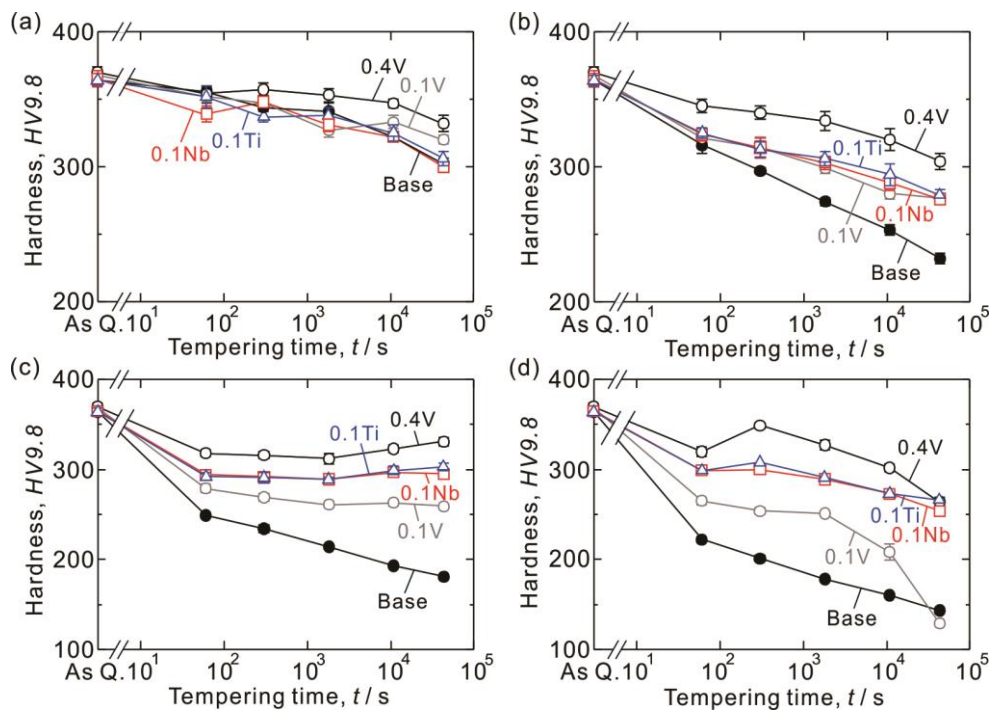

Fig. 1 Hardness variations of various alloys tempered at (a) 623 K, (b) 723 K, (c) 823 K and (d) 923 K for various times.

Fig. 2 plots the hardness against the empirical tempering parameter [7], by considering C content dependence of constant in parentheses (which is taken to be 17.3 according to the C content of 0.1 mass% in this study). Equivalent tempering times at each temperature are also indicated above this figure for reference. The hardness evolution of one alloy under different tempering conditions can be reasonably represented by a single curve. From the overall trend, the descending order of resistance to temper softening can be roughly summarized as: 0.4V > 0.1Nb ≅ 0.1Ti > 0.1V.
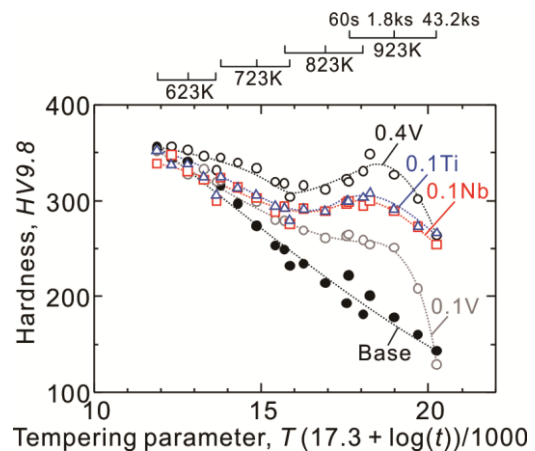

Fig. 2 Hardness variations of various alloys with tempering parameter, where T is in K and t is in s.

3.2. Microstructure

Fig. 3 shows the typical SEM images of the Base and 0.4V alloys in as-quench and tempered conditions, respectively. The as-quenched specimens show similar microstructure of lath martensite (Fig. 3(a) and (b)). After tempered at 723 K for 0.3 ks, densely dispersed submicron-sized cementite is formed in both alloys (Fig. 3(c) and (d)). Similar microstructures are also observed in other M-added alloys tempered at this temperature without any evident difference in the size of cementite. When the temperature is raised to 923 K, the dispersion of cementite in the Base alloy becomes coarsened (Fig. 3(e)). In contrast, cementite appears to be re-dissolved by 0.4V additions (Fig. 3(f)), implying that C is consumed by the formation of MC-type alloy carbide [8]. The amounts of cementite in other M-added alloys tempered at this temperature simply lie between those two alloys.

Fig. 4(a) ~ (d) shows the typical three-dimensional M atom maps of various M-added alloys tempered at 923 K for 0.3 ks (nearly peak in hardness curves), respectively. Consistently with our previous work [9], nano-sized MC is found to be formed by aging from martensitic matrix in all these tempered specimens. Compared with the 0.1V-added alloy, higher number density of MC is formed in the 0.4V, 0.1Nb and 0.1Ti-added alloys due to their larger supersaturation [2], which qualitatively explains their hardness increment from the Base alloy at higher tempering temperatures (Fig. 1(d)). On the other hand, almost no MC precipitate can be detected by 3DAP in the alloys tempered at 723 K for 0.3 ks. Even though, hardness of tempered martensite is still increased by ~ 20 HV with M additions (Fig. 1(b)). In another word, their resistance to temper softening is expected to be caused by other factors, e.g. the difference in dislocation density [10], or presumable the formation of sub-nano clusters beyond the resolution of 3DAP, which will be studied in our future works.
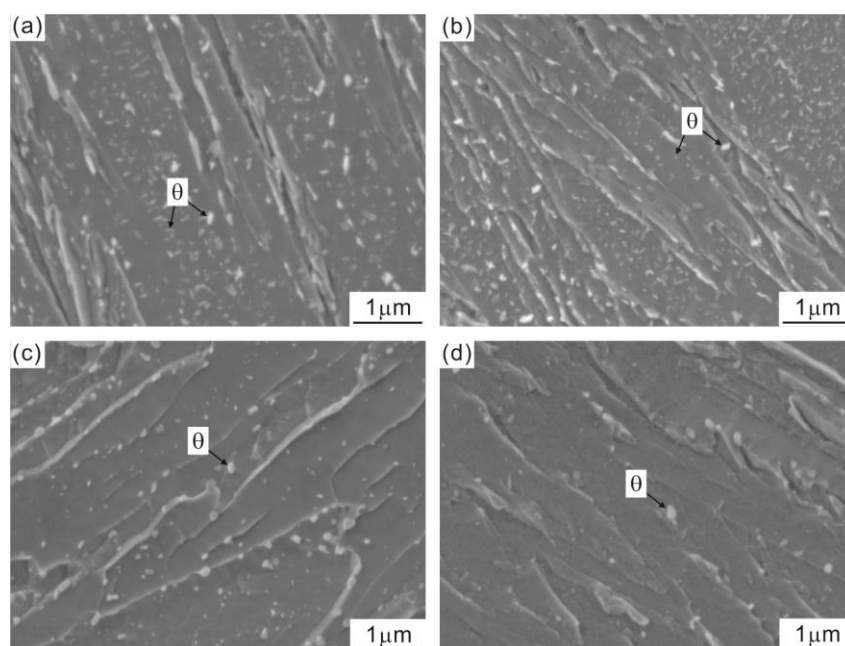

Fig. 3 SEM microstructure of the Base alloy tempered at (a) 723 K for 0.3 ks and (b) at 923 K for 0.3 ks; 0.4V alloy (c) tempered at 723 K for 0.3 ks and (d) tempered at 923 K for 0.3 ks, respectively. θ: cementite.
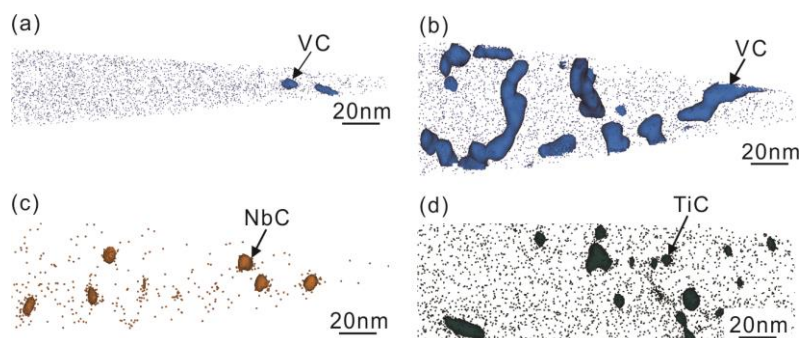

Fig. 4 Three-dimensional M atom maps of (a) 0.1V, (b) 0.4V, (c) 0.1Nb and (d) 0.1Ti alloys tempered at 923 K for 0.3 ks, respectively.

4. SUMMARY

This study investigated the microalloying effects of V, Nb and Ti on microstructure and resultant hardness of tempered martensite in low carbon steels. It was found that large hardness increment in microalloyed steels at higher temperatures is mainly caused by the formation of nano-sized MC precipitates, while their resistance to temper softening at lower temperatures can be hardly explained by the precipitation hardening of MC.

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