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Interface modification of tempered martensite utilizing nano-scale transition carbide via isothermal sub-zero treatment

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ABSTRACT

The present study investigated the influence of isothermal holding time on characteristics of nano-scale transition carbide and interfaces of tempered high-carbon martensitic steel processed by sub-zero treatment (SZT). We interpreted a stress-induced activity of carbon atoms through SZT prior to tempering, which was crucial for obtaining desirable precipitation of transition carbide. The modification upon interfaces under the influence of transition carbide characteristics was recognized for the first time and discussed based on mechanism of precipitate-interface interaction in relation to the nanohardness value.

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1. Introduction

Advanced development of ferrous alloys with excellent mechanical performance was required to satisfy the need for a variety of practical applications [1]. This could be accomplished by implementing microstructural strengthening induced by the formation of BCT (body-centered tetragonal) martensite through quenching followed by structural relaxation via tempering [2–4]. Upon tempering at 523–773 K, high-carbon steel might regain acceptable ductility due to the beneficial effects of stress relaxation and precipitation of stoichiometric cementite in the microstructure [2,4]. Such cementite would inevitably undergo a coarsening process, resulting in the microstructure susceptible to transgranular fracture [1,5]. Moreover, the occurrence of recrystallization and grain growth might lose strengthening while ductility was improved.

In fact, a non-stoichiometric transition carbide occurred at temperature below 523 K, was known for several advantages over cementite, such as nano-meter scale, lower diffusivity of carbon, and resistance against coarsening [2]. Up to date, the inherent characteristics of the transition carbide were still underestimated due to a robust control of their population, size, and uniformity (not massively clustered) were hard to be achieved [1,2], which was in contrast to cementite counterpart. To the best of authors' knowledge, a specific processing design to overcome such issue has been lacking in the literature, implying that the optimum use of transition carbide remained unexplored. It was believed that proper control on transition carbide might eventually give a rise to the structure with a number of interfaces through mechanism of precipitate-interface interaction [1,2,5]. This would lead to an improvement in microstructural stability and mechanical performance based on the modification of interfaces. Thus, the present work investigates the unexplored contribution of transition carbide to the interface characteristics triggered via isothermal SZT and low-temperature tempering. The correlation between mechanical properties and related structural mechanism is discussed in reference to the conventional counterpart.

2. Experimental procedures

The as-received samples were high-carbon steel with a chemical composition of Fe-0.85C-0.4Mn-0.2Si-0.15Cr (wt%). A sample $(25 \times 25 \times 3 \text{ mm}^3)$ was austenitized at 1173 K for 10 min and immediately quenched via SZT isothermally at 233 K for 1–24 h to form low-tetragonality martensite (LTM) structure. Another sample was water-quenched down to room temperature, resulting in ordinary martensite (OM) structure. The M_s and M_f (starting and finishing temperature of martensite transformation) were





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estimated to be 414 K and 250 K [5]. Low-temperature tempering at 473 K for 2 h were performed subsequently on OM and LTM to produce ordinary tempered martensite (OTM) and tempered martensite with unique distribution of transition carbide (TMTC) structures, respectively. The microstructural observations were performed on the central regions of each sample via electron back-scattered diffraction (EBSD). Kernel average misorientation (KAM) maps were used to analyze the extent of microstructural lattice distortion [6]. The details of structures were observed via bright-field transmission electron microscope (TEM). The compression and related change in BCT lattice were investigated using X-ray Diffraction (XRD) with Cu K α radiation. The nanohardness were evaluated by Berkovich-equipped nanoindentation instrument. All observations and indentations were performed at room temperature.

3. Results and discussion

Fig. 1a, b present TEM images of the OM and LTM after 24 h holding time. The average widths of martensite plates were \sim 177 nm and \sim 110 nm in OM and LTM, respectively. The dislocation density of LTM was higher than that of OM. The LTM exhibited a degree of dislocation density that might be comparable to that obtained via severe plastic deformation (SPD) methods [1,7]. The

excess energy stored after SPD generated microstructural instability which would be compensated by a formation of ultrafinegrained structure having remarkable degree of dislocation density [7–9]. Similarly, the exceptional amount of dislocations in the LTM might be associated with high degree of microstructural instability generated during martensitic transformation [2–4].

Fig. 2a shows the nanohardness decrease of LTM throughout 24 h of holding time. Since no phase transformation was observed, the softening behavior was believed to be associated with the continuous crystallographic shrinkage triggered during the immersion. Such extreme conditioning would cause accumulation of compressive stress on highly-distorted BCT, increasing its structural instability [4,10]. Unlike that occurred after SPD, relaxation through release of carbon atoms from lattices would be favorable, whose phenomenon was called 'cold stress relaxation'. This was confirmed by KAM maps shown in Fig. 2b. c. A low fraction of high distortion area (\sim 5°, red color) after 24 h holding (Fig. 2c) implied the occurrence of massive lattice relaxation and solid-solution weakening responsible for low-tetragonality martensite [6,11]. Such structural phenomena were also indicated by XRD pattern displayed in Fig. 2d, e: (i) shifting of martensite peak suggested a huge compressive stress upon the structure; (ii) minor peak splitting might imply a change in tetragonality [3,4,12]. After being released, the 'free' carbon atoms would migrate with ease due to



Fig. 1. TEM images of (a) OM and (b) LTM after 24 h of isothermal SZT.



Fig. 2. (a) Nanohardness of LTM after isothermal SZT for 1–24 h. KAM maps of LTM after (b) 1 h and (c) 24 h. (d, e) Representative XRD peaks of BCT (o') for LTM.

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