Materials Science & Engineering A 719 (2018) 21–26

Contents lists available at ScienceDirect

Materials Science & Engineering A

journal homepage: www.elsevier.com/locate/msea

Effects of austenitizing temperature on microstructure and mechanical property of a 4-GPa-grade PM high-speed steel

Hanlin Peng[a], Ling Hu[b], Tungwai Ngai[b], Liejun Li[b], Xianglin Zhang[a,*], Heng Xie[c], Weiping Gong[c]

[a] State Key Laboratory of Material Processing and Die & Mould Technology, School of Materials Science and Engineering, Huazhong University of Science and Technology, Wuhan 430074, China
[b] School of Mechanical and Automotive Engineering, South China University of Technology, Guangzhou 510640, China
[c] College of Electronic Information and Electrical Engineering, Huizhou University, Huizhou 516000, China

ARTICLE INFO

Keywords:
High-speed steel
Carbides
Microstructure evolution
Heat treatment
Compressive strength

ABSTRACT

Effects of austenitizing temperature on the microstructure and mechanical property of a high-strength PM high-speed steel were systematically investigated using a series of heat treatments in the temperature range of 1070–1180 °C. Results showed that the optimal austenitizing temperature to obtain the desired combination of high compressive strength of 3838 MPa and high ductility of 29.7%, the optimal austenitizing temperature was 1180 °C. A maximum strength of 3870 MPa and a fracture strain of 22.7% was obtained by austenitized at 1120 °C. It is evident that the size distribution and carbide contents of both VC and Fe_3W_3C can be significantly changed by heat treatment and triple tempering resulted in slight decline in the amount of cracked carbides. Fractography showed that more broken particles of Fe_3W_3C carbide was observed than that of the VC particles in fractured specimens.

1. Introduction

High-speed steels (HSSs) are important in industries, because they are widely used as cutting tools and fine-blanking tools [1–4]. Casting [5,6], spray forming [7–9], and powder metallurgy [10,11] are the three commonly used methods to fabricate HSSs. Compared to the HSSs prepared by conventional casting method, those obtained by powder metallurgy exhibit a refined grain structure with no segregation, which is critical to fabricating HSSs with high alloying content [10,12].

HSSs are alloys of Fe-C-X multi-component systems, where X represents different alloying elements such as V, W, Mo, and Cr [13]. Its excellent mechanical properties mainly result from the combination of precipitation strengthening and solid solution strengthening. According to previous literatures, the size and amount of carbides can be adjusted by alloy design [14] and heat treatment [15]. Unfortunately, alloy design cannot be achieved by ordinary users, therefore, the desired microstructure and mechanical properties are preferred to be obtained by reasonable heat treatment [16,17] and subzero treatment [18,19].

As a typical HSS, PM Microclean S390 has better mechanical properties than most of the other HSSs. Niederkofler et al. [20] observed lamellar and needle carbides in the annealed PM HSS S390 using 3D-atom probe technique. Torres et al. [21] predicted the fatigue limit of this HSS using a crack growth threshold-based approach. Recently, based on electron back-scattered diffraction, Godec et al. [22] reported that this alloy contains MC and M_6C carbides, and the volume percentage of these carbides reached at approximately 17%. Later, the effects of austenitizing temperature, immersion time of deep cryogenic treatment, simultaneous pulse plasma nitriding and tempering on wear resistance were extensively studied [19,23]. However, in these studies, the temperature range for austenitizing was relatively narrow and reports on the effects of austenitizing temperature on mechanical properties of steels were scarce.

Since the mechanical properties of HSSs is dependent on the heat treatment used [11], therefore, this study aims to reveal the differences in the microstructure and properties of the samples based on various austenitizing temperatures. The influence of heat treatment on carbide evolution and mechanical properties of the HSS was studied and discussed. The results of this study provide the desired microstructure and mechanical properties of HSSs for industrial applications.

2. Materials and experimental methods

Microclean S390 is a PM HSS manufactured by Böhler and has a basic composition (wt%) of Fe-1.6C-4.75Cr-2Mo-5V-10.5W-8Co. Fabrication of the PM HSS S390 mainly involves two steps: (1) gas atomization of steel powders, and (2) hot isostatic pressing (HIP); the

* Corresponding author.
E-mail address: hust_zxl@mail.hust.edu.cn (X. Zhang).

https://doi.org/10.1016/j.msea.2018.02.010
Received 8 October 2017; Received in revised form 1 February 2018; Accepted 2 February 2018
Available online 05 February 2018
0921-5093/

Table 1
Sample codes and corresponding heat treatment.

Sample code	Details of treatment
S1	Quenching (1070 °C)
S1-T	Quenching (1070 °C) + triple tempering (550 °C/570 °C/540 °C)
S2	Quenching (1120 °C)
S2-T	Quenching (1120 °C) + triple tempering (550 °C/570 °C/540 °C)
S3	Quenching (1150 °C)
S3-T	Quenching (1150 °C) + triple tempering (550 °C/570 °C/540 °C)
S4	Quenching (1180 °C)
S4-T	Quenching (1180 °C) + triple tempering (550 °C/570 °C/540 °C)

latter step helps increase the density and mechanical properties of the HSS [24], thus improving the service life. The specimens were received after being treated by HIP. Heat treatments of the HIP specimens were conducted in a vacuum furnace with temperatures ranging from 1070 to 1180°C for 120 min, then followed by nitrogen gas quenching and triple tempering at 550 °C, 570 °C, and 540 °C for 120 min. Detailed heat treatment schedules are listed in Table 1. The heating rate and holding time during heat treatment is kept constant, and only the austenitizing temperature is modified.

The phase analyses were determined by X-ray diffraction (XRD, D/MAX-2500/PC; Rigaku Corp., Japan) with Mo-Kα radiation rather than Cu-Kα radiation in order to obviate strong X-ray fluorescence [12]. Microstructure and carbide distribution were analyzed by energy-dispersive X-ray spectroscopy (EDS, Nova NanoSEM-450). The SEM samples were polished without etching. TEM samples were prepared by ion polishing in a Gatan 691 apparatus. Transmission electron microscope (TEM) observations were conducted on a Tecnai (G2 F20 S-TWIN) transmission electron microscope. The carbides concentration variation with changing temperature was calculated by Thermo-Calc software. Compressive stress-strain curves were obtained using an Instron-5500 testing system, with a sample size of Ø 3 mm × 6 mm and a strain rate of 1×10^{-3} s^{-1}. Three specimens were tested for each alloy.

3. Results and discussion

3.1. Microstructure evolution

XRD spectra of PM HSS S390 specimens after quenching at different temperatures are shown in Fig. 1a, including as-quenched and tempered samples. Only M_6C, MC, and martensite three phases were detected. The diffraction peak intensity of MC and M_6C in triple tempered samples was slightly stronger than that in the as-quenched specimens. RA and other carbides such as M_2C and $M_{23}C_6$ were not detected (Fig. 1a). Fig. 1b provides the microstructure of the experimental specimens subjected to quenching and triple tempering (S2-T). Spherical grey and bright carbides, with an average size of less than 2.5 μm, can be clearly observed. By SEM/EDS microanalysis, the deep-grey particles and bright particles were identified to be vanadium-rich MC compounds and tungsten-rich M_6C (Fe_3W_3C) compounds, respectively.

Transition-metals such as W, Mo, Cr, and V, were added into S390 HSS, with an alloying content up to roughly 22% [23], and these alloying elements promote the formation of different types of carbides, e.g., MC, M_2C, M_6C, M_7C_3, and $M_{23}C_6$ [25]. TEM, EDS and XRD results suggested that only VC and Fe_3W_3C were present in this alloy, as depicted in Fig. 1a and c. Additionally, nano-sized RA in the form of thin films can be observed between the martensite lath, as shown in Fig. 1d. Leskovsek et al. roughly estimated the volume percent of RA in this HSS to be less than 5% [23], which probably accounts for the absence of diffraction peaks for RA in Fig. 1a.

The microstructures of unetched heat-treated S390 specimens obtained by backscattered electrons (BSE) are illustrated in Fig. 2. From Fig. 2, it can be seen that for all specimens, disconnected and spherical carbides were uniformly distributed in the martensitic matrix. The carbides in the triple tempered specimens were slightly smaller than those in the as-quenched specimens, while the carbide contents were slightly higher than those in the as-quenched specimens. These results indicated that carbides (M_6C and fine MC) can be dissolved partially into the matrix during austenitization, and triple tempering contributed to the precipitation of carbides from the supersaturated matrix. After exposure to a temperature as high as 1070 °C for 2 h, M_6C and fine MC particles were metastable and could dissolve into austenite during austenitization. Similar results were also reported by other researchers [26]. However, large particles could not dissolve completely, and some coarse MC particles (rich in V) remained in the matrix. Thermodynamically, smaller carbides have a higher surface energy and hence are preferentially dissolved into the matrix over coarse carbides [27]. Therefore, it could be clearly seen that relatively larger particles remained and fine carbides dissolved in the as-quenched specimens.

When subjected to a higher temperature (up to 1180 °C), it was expected that significant amount of fine V-rich and W-rich carbides would dissolve into the matrix (Fig. 2g). After triple tempering, fine, spherical, and disconnected carbides will be precipitated, as shown in Fig. 2f. The amount of both retained VC and M_6C carbides decreased with increasing austenitization temperature (as shown in Fig. 2a, c, e and g), suggesting that heat treatment is an effective method to refine the microstructure of this alloy.

Since mechanical properties are largely determined by carbides shape, size, distribution and volume fraction, it is very important to characterize all the carbide phases present in the experimental specimens. Thus, Thermo-Calc software was implemented to estimate the equilibrium carbide phase or phases at different temperature. Meanwhile, Image-ProPlus 6.0 software was used to determine the size distribution and volume fraction of the carbides formed in the specimens. Quantitative evaluation results are illustrated in Fig. 3, including both thermodynamic calculation and experimental results.

Fig. 3a shows the mole fraction of carbides at different temperature calculated by using Thermo-Calc software. At temperature of 400 °C, the mole fraction of VC and Fe_3W_3C is 13.74% and 9.28%, respectively (not shown here). The amount of dissolved carbides increased with increasing temperature. Additionally, it was found that around 18.82% VC and 22.82% Fe_3W_3C (mole fraction) were dissolved into the matrix after exposure to the high temperature of 1070 °C. In contrast, 27.22% VC and 36.22% Fe_3W_3C (mole fraction) were dissolved when the austenitizing temperature was increased to 1180 °C, suggesting that this alloy possesses superior thermal stability.

The volume fraction of carbides in this alloy was about 17.4% (as shown in Fig. 3b), which is close to Godec's experimental results [22]. Apparently, triple tempering contributed to the relative increase in the volume fraction of VC, as shown in Fig. 3b. However, changes in the volume fraction of Fe_3W_3C was not obvious, because the volume fraction of Fe_3W_3C was approximately only about one-third of that of VC [22]. The quantitative results were in good agreement with the micrographs in Fig. 2. Roughly over half of the VC and Fe_3W_3C remained undissolved even when heated to the high temperature of 1180 °C, suggesting that the microstructure might effectively inhibited the severe coarsening of the grain and thus avoided the obvious deterioration of mechanical properties. The calculated carbide content at different temperature are in agreement with those obtained by experiments.

The size distribution of both carbides changed significantly after heat treatment. Compared with the as-quenched specimens, the frequency of both carbides with small sizes increased in tempered specimens, as shown in Figs. 3c and 3d. The calculation results agreed with microstructure shown in Fig. 2a-h. Since the tempering temperature was relatively low, the precipitated carbide size remained small. The above reported results indicated that heat treatment can significantly affected the size distribution and volume fraction of carbides in this HSS.