Materials Characterization 113 (2016) 60–66

Contents lists available at ScienceDirect

Materials Characterization

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

Study of retained austenite and nano-scale precipitation and their effects on properties of a low alloyed multi-phase steel by the two-step intercritical treatment

Z.J. Xie, G. Han *, W.H. Zhou, C.Y. Zeng, C.J. Shang *

School of Materials Science and Engineering, University of Science and Technology Beijing, 100083, China

A R T I C L E   I N F O

Article history:
Received 5 August 2015
Received in revised form 2 December 2015
Accepted 10 January 2016
Available online 12 January 2016

Keywords:
Retained austenite
Nano-scale precipitation
Low alloyed steel
Multi-phase microstructure
Structure–property relationship

A B S T R A C T

Microstructure evolution and properties were studied in a low carbon low alloyed hot-rolled bainitic steel by annealing and annealing plus tempering. Microstructure of the hot-rolled steel consists of lath bainite and martensite. By annealing at 720 °C for 30 min and water quenching, multi-phase microstructure consisting of intercritical ferrite, tempered bainite/martensite, retained austenite and fresh martensite was obtained. With increasing annealing temperature to 760 °C, microstructure of the steel consisted of intercritical ferrite, fresh martensite without retained austenite. After the second step of tempering at 680 °C for samples annealed both at 720 °C and 760 °C, ~8–9% volume fraction of retained austenite was obtained in the multi-phase microstructure. Moreover, fine precipitates of VC with size smaller than 10 nm and copper precipitates with size of ~10–50 nm were obtained after tempering. Results from scanning transmission electron microscopy (STEM) give evidence to support that the partitioning of Mn, Ni and Cu is of significance for retained austenite stabilization. Due to the combined contribution of multiphase microstructure, the transformation-induced-plasticity effect of retained austenite and strengthening effect of nanometer-sized precipitates, yield strength greater than 800 MPa, yield to tensile ratio of 0.9, uniform elongation of ~9% and good low temperature impact toughness of 147 J at −40 °C were achieved.

1. Introduction

There is a strong demand in producing high strength–high ductility–high toughness low carbon low alloyed infrastructural steels with low yield to tensile ratio for environment and safety consideration [1]. Transformation-induced plasticity (TRIP) effect associated with retained austenite has been realized to significantly improve the mechanical behavior of structural steels, especially for excellent strength and ductility combination [2,3]. In addition, stable film-like retained austenite has been found to remarkable contribute to low temperature toughness of steels, especially for lowering ductile–brittle transition temperature (DBTT) [4]. In order to obtain stable retained austenite, numerous approaches have been developed for different alloying steels, such as quenching and partitioning (Q&P) [5], austempering treatments [6], and intercritical annealing [7]. The Q&P or austempering process stabilizes retained austenite by rejection of carbon from martensite or bainite to austenite [8,9] in steels with carbon concentration of 0.15–0.4%. In order to supply sufficient carbon for stabilizing austenite, silicon and/or aluminum usually were added for suppression of carbide formation. Another approach of the intercritical annealing treatment produces retained austenite in high Mn (>5%) and/or high Ni (>5%) alloyed martensitic/bainitic steels [10,11]. Retained austenite is stabilized by partition of Mn/Ni to reverted austenite from martensite/bainite matrix during annealing. However, these steels are mainly used in automobile industry.

In order to develop high performance steels for infrastructure applications, the two-step intercritical heat treatment has been introduced to low carbon low alloyed steels (<0.1C, 2–2.4Mn, 0.5–1Ni) to obtain stable retained austenite [12,13] recently. The first step is the intercritical annealing at the two-phase (ferrite + austenite) region to produce lamellar poor alloyed ferrite and high alloyed reverted austenite. However, the reverted austenite transformed to martensite during the following cooling due to its low stability. The enrichment of austenite with alloying elements in the first step decreases the temperature of nucleation of reverted austenite (A_{c1}), such that the second reverted transformation occurs at a lower tempering temperature during the second step of intercritical tempering. With the benefit of further enrichment of austenite stabilizers and fine size, the second reverted austenite can be stabilized to room temperature.

On the other hand, softening effect caused by recovery of dislocation during holding at high temperatures results in a low yield strength. Based on this consideration, micro-alloying elements (Nb, V) and

---

* Corresponding authors.
E-mail addresses: hangang@mater.ustb.edu.cn (G. Han), cjshang@ustb.edu.cn (C.J. Shang).

http://dx.doi.org/10.1016/j.matchar.2016.01.009
1044-5803/

copper were added to produce precipitation strengthening. In the present study, the one step and two-step intercritical heat treatments were applied to a low carbon low alloyed steel to obtain high strength–high ductility–high toughness. Microstructure evolution and nano-scale precipitation behavior were studied by using SEM, EBSD, XRD and TEM. The structure–property relationship in the steel is discussed.

2. Material and experimental procedure

The nominal chemical composition of the studied steel in weight percent is 0.08C–0.5Si–2.0Mn–0.5Ni–0.9Cu–0.8(Mo + Cr)–0.03Nb–0.1(V + Ti) (wt.%). The steel was prepared by vacuum melting and cast into ingots with a thickness of ~100 mm. The ingots were homogenized at 1200 °C for 2 h, and hot rolled in several passes with a minimum reduction rate of 20% per pass to plates of 16 mm thick, finally water quenched to room temperature. Heat treatment samples with dimensions of 180 mm × 16 mm × 16 mm (length, width and thickness) for tensile properties and Charpy impact testing were cut parallel and perpendicular to the rolling direction of the plate, respectively. The heat treatments illustrated in Fig. 1 were carried out on these samples in a resistance furnace. The directly quenched hot rolled samples were firstly annealed at 720 °C and 760 °C for 30 min, respectively, followed by water quenching. Secondly, the annealed samples were tempered at 680 °C for 30 min, and air cooled to room temperature.

Mechanical properties in terms of strength and elongation of the treated samples (φ10 mm standard tensile test samples) were measured at an extension rate of $2.5 \times 10^{-3}\ s^{-1}$ at room temperature. Impact tests were performed on Charpy V-notch (CVN) specimens with size of 10 mm × 10 mm × 55 mm along the transverse orientation at −40 °C according to ASTM: E2298 test standard. Microstructure of the samples was examined by SEM performed on a ZEISS ULTRA-55 field emission scanning electron microscope (FE-SEM) at an acceleration voltage of 15 kV. To characterize the distribution and morphology of retained austenite, electron back scattering diffraction (EBSD) analysis was carried out using JEOL-7000 field emission scanning electron microscope (FE-SEM) at an acceleration voltage of 20 kV after metallographic mechanical polishing and fine polishing with colloidal silica. A fine step size of 50 nm was used to identify the nano-fine dispersion retained austenite. EBSD data was post-processed by HKL CHANNEL 5 flamenco software to acquire necessary information. Foil specimens were prepared by electro-polishing in a twin-jet polisher using 5% perchloric acid solution for TEM observation. To characterize nano-scale micro-alloying elements precipitates, carbon extraction replica approach was used. The specimens was mounted using a mixture of resin and hardener with ratio of 5: 1. The surface of the mounted specimens was polished and firstly etched with 4% nital (a solution of nitric acid and methanol with volume ratio of 1: 24). Then, carbon was evaporated on to the etched surface. Next, the surface was scored to ~3 mm squares and the sample was etched again with 4% nital. Finally, the extracted replicas were rinsed with distilled water and placed on the copper grid and dried. TEM was carried out using FEI Tecnai F20 and Philips CM12 transmission electron microscopes both in combination with an energy dispersive X-ray spectrometer (EDX), operated at 200 kV and 120 kV respectively. The volume fraction of retained austenite in different samples was determined by using a DMAX-RB 12Kv X-ray diffractometer with Cu K_α radiation under following conditions: acceleration voltage, 40 kV; current, 150 mA; step, 0.02°. The volume fraction of retained austenite were calculated based on the integrated intensities of the $(2\,0\,0)_\alpha$, $(2\,1\,1)_\alpha$, $(2\,0\,0)_\gamma$, $(2\,2\,0)_\gamma$ and $(3\,1\,1)_\gamma$ diffraction peaks.

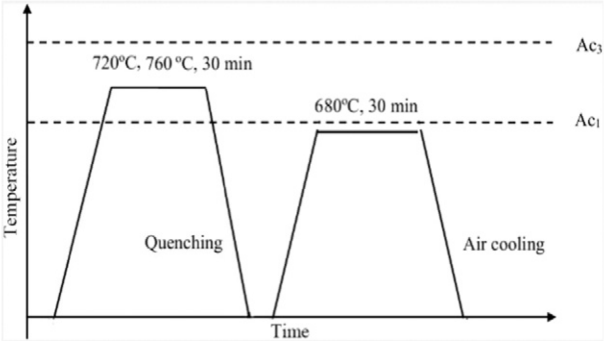

Fig. 1. Schematic diagrams for heat treatment used in the work.

3. Results

3.1. Mechanical properties

Mechanical properties of one-step and two-step intercritically treated samples are summarized in Table 1. The as-rolled sample exhibited high yield strength (945 MPa), ultrahigh tensile strength (1201 MPa) and low yield to tensile (Y/T) ratio (0.79), but a low ductility (uniform elongation: 3.1%; total elongation: 13.8%) and very poor toughness. After one step of annealing, the yield strength and tensile strength dropped dramatically. With increasing the annealing temperature from 720 °C to 760 °C, the yield strength decreased from 748 MPa to 669 MPa, while tensile strength remained as the same level of ~990 MPa. Y/T ratio was very low and ductility was slightly improved for samples one step annealed both at 720 °C and 760 °C. Charpy V-notch impact energy at −40 °C had ~36 J of improvement only by annealing at 720 °C, there was no obvious change for case of 760 °C in comparison with that of as-rolled steel. For the two-step intercritical heat treatment, as annealing temperature changed from 720 °C to 760 °C at a constant tempering at 680 °C, there were ~81 MPa and 60 MPa increase in yield strength, respectively. By tempering at 680 °C, the tensile strength remained to be 924 MPa for sample firstly annealed at 720 °C, while there was ~200 MPa decrease in tensile strength for sample firstly annealed at 760 °C. The Y/T ratio increased by the second step of tempering, but it remained at a desired low level of 0.9 and 0.92. The ductility of the steel was improved largely by the second step of tempering (especially the uniform elongation and total elongation: 8.9% and 22.3% for sample firstly annealed at 720 °C; 11.2% and 25.1% for sample firstly annealed at 760 °C). In addition, low temperature toughness was enhanced largely to be 147 J and 121 J by the second step tempering for samples annealed at 720 °C and 760 °C, separately.

3.2. Characterization of microstructure and retained austenite

General microstructures of hot-rolled and heat-treated samples were characterized by SEM, as shown in Fig. 2. Fig. 2a shows the fine lath microstructure consisting of bainite and martensite for the hot-rolled steel by water quenching. Dispersed carbides can be characterized within bainitic laths. After one-step annealing at a low temperature of 720 °C, tempered martensite/bainite microstructure dispersed with fine fresh formed martensite or retained austenite along grain boundary was observed as Fig. 2b. The tempered matrix remained fine lath-like microstructure. After annealing at a high temperature of 760 °C, dual-phase microstructure mainly consisting of intercritical ferrite and newly

Table 1
Tensile properties and Charpy impact energy of the studied steel after heat treatments.

Processing	σ_s /MPa	σ_b /MPa	A_u/%	A/%	Y/T ratio	CVN/J at −40 °C
As-rolled	945	1201	3.1	13.8	0.79	34
720 °C annealed	748	988	6.2	18	0.76	70
760 °C annealed	669	994	5.6	17.2	0.67	43
720 °C annealed plus 680 °C tempered	829	924	8.9	22.3	0.9	147
760 °C annealed plus 680 °C tempered	729	795	11.2	25.1	0.92	121

Note: σ_s—yield strength, σ_b—tensile strength, A_u—percentage elongation at maximum force (uniform elongation), A—percentage elongation after fracture (total elongation), Y/T ratio—yield strength to tensile strength ratio, CVN—Charpy impact energy.