Materials Characterization 110 (2015) 86–93

Contents lists available at ScienceDirect

Materials Characterization

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

Nano-indentation investigation on the mechanical stability of individual austenite in high-carbon steel

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ARTICLE INFO

Article history:
Received 5 August 2015
Received in revised form 15 October 2015
Accepted 19 October 2015
Available online 20 October 2015

Keywords:
Retained austenite (RA)
Mechanical stability
Nano-indentation
Transmission electron microscopy
Cryogenic treatment
High-carbon steels

ABSTRACT

Quenching (AQ) and cryogenic treatments (QC) were conducted on the high-carbon SAE 52100 steel to investigate the mechanical stability of individual retained austenite (RA) by nano-indentation. The cross-sections of indented RA region prepared by focused ion beam (FIB) were examined by using transmission electron microscopy (TEM). For the first time, it was directly observed that some parts of RA grain, closest to the indent, in AQ specimen had transformed into strain-induced martensite (SIM). However, not any pop-in or transformation was detected in the indented QC specimen. This clearly indicates that the mechanical stability of RA in QC seems significantly enhanced, which is mainly attributed to the cryogenic treatment resulting in a higher carbon enrichment of RA compared to that in AQ. Furthermore, a higher load of external stress may need to trigger its martensitic transformation in QC specimen.

1. Introduction

The high-carbon steels usually contain a significant fraction of retained austenite (RA) after quenching to room temperature. RA is a metastable phase in which further transformation may happen in certain conditions. The transformation from RA to martensite can be triggered when the chemical free energy change $\Delta G^{\gamma\rightarrow\alpha}$ achieves a critical value ΔG_{Ms}, i.e., when a sufficiently large undercooling is reached below the equilibrium transformation temperature [1,2]. This phenomenon is related to the thermal stability of austenite as reported previously [3]. Actually, the RA to martensite transformation can also be stimulated by simply applying an external stress (tension or compression) to supplement the chemical driving force. The additional energy due to the interaction of the applied stress and the shape deformation of martensite phase is the mechanical free energy ΔG_{MECH} [4] and such a phenomenon is related to the mechanical stability of RA which will be discussed in this paper.

The mechanical stability of RA has attracted numerous interests in the automobile industry for fabrication of energy absorption parts, mainly, with TRIP steels [5–8]. However, only little attention has been paid to the stability of RA in high-carbon steels such as SAE 52100 and 100Cr4 which are predominantly based on martensitic microstructure to achieve high strength and hardness and, thus, the RA in those steels is usually considered as an unfavorable and detrimental phase [9–13]. While, the fact is that the high-carbon steels usually contain more RA after conventional quenching or even cryogenic treatment than TRIP steels because of higher carbon content. Those steels are extensively used for bearings, cutting tools, dies and more common applications that require high hardness, strength and dimensional stability, in which the mechanical stability of RA plays a crucial role in optimizing the mechanical properties. However, in the case of high-carbon steels, little systematic work has been done on the mechanical stability of RA, instead, most of the researches were focused on low-carbon and TRIP steels. For example, it has been reported that the mechanical stability of RA is linked with not only its carbon content, but also strain partitioning, grain size and morphology [14–21]. The limited studies concerning the mechanical stability of RA in high-carbon steels were usually performed by macroscopic tensile or compression tests, which can only provide an overall information on the stability of RA [22]. For the individual RA in high-carbon steels, its mechanical stability has not yet been clearly investigated. As far as the authors are aware, the related issue has never been reported; only a few similar studies have been reported by de Diego-Calderón et al. [23,24] and other investigators [25–27] in TRIP steels. It is pertinent to mention here that these results that are found in TRIP steels may also apply for the high-carbon steels, but a further detailed investigation is needed to clarify its correlations with factors such as carbon content, grain size and dislocations and how the fundamental mechanisms operates in RA. Thus, quenching and cryogenic treatments were conducted respectively on the high-carbon SAE 52100 steel to assess the mechanical stability of individual RA and its strain-induced martensite (SIM) transformation behavior by using nano-indentation technique combined with transmission electron microscopy (TEM), focused Ion beam system (FIB),

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http://dx.doi.org/10.1016/j.matchar.2015.10.024
1044-5803/

X-ray diffraction (XRD), scanning electron microscopy (SEM) and electron probe micro-analyzer (EPMA).

2. Experimental procedures

2.1. Material and heat treatments

In the present study, a commercial grade of high-carbon SAE 52100 steel was investigated and its chemical composition is given in Table 1. The as-received steel exhibits spheroidization-annealed pearlitic microstructure as shown in Fig. 1. The heat treatment schedules were designed to get a mixture of martensite and proper amount of RA in the steel. For this purpose, two different treatment procedures i.e. AQ and QC, were performed respectively as Fig. 2 shows. For quenching treatment (AQ), the specimens were fully austenitized at 1100 °C for 10 min and then quenched to room temperature in oil. The QC (quenching with subsequent cryogenic treatment) includes the same austenitization and quenching as AQ, but followed by an additional cryogenic treatment at −196 °C (liquid nitrogen) for 24 h.

2.2. Microstructural observations

Specimens for scanning electron microscopy (SEM, JEOL JSM7600F, 15KV) observation were cut, ground, polished and then etched in 10% sodium metabisulphite solution. The volume fractions of RA in both AQ and QC were determined by a Rigaku D/max-2000 X-ray diffractometer (operated at 35 kV, 200 mA) with Cu K_α radiation in accordance with ASTM E975. For this purpose, the polished specimens were subjected to a further vibratory polishing to remove the deformed surface layer. Electron probe microanalyzer (EPMA-1610) was used to directly measure the carbon distribution within the RA grain in AQ and QC specimens. To locate the measuring lines, several marks were made on polished surface using hardness tip. Then, line-analysis was conducted to obtain the carbon profiles across the nano-indented RA grains. After that, the specimens were etched slightly for SEM observation. By matching the carbon profiles with corresponding SEM image, the carbon distribution of different individual RA grain can be determined accurately. The field emission transmission electron microscopy (TEM, JEOL 2100F, 200 KV) was employed to characterize the phase (martensite and RA) evolution after nano-indentation.

2.3. Nano-indentation tests

Nano-indentation tests were carried out in load control mode on a TI 950 Hysitron Tribolab system at a constant loading rate of 200 $\mu N \cdot S^{-1}$ up to a maximum load of 8000 μN with a Berkovich three-sided pyramidal diamond tip (nominal angle of 65.3° and diameter of 200 nm). The scanning probe microscopy (SPM) equipped on TI 950 can offer an in-situ scanning at high magnification mode to identify the phase of small areas before and after each nano-indentation tests, by which the individual AR grains in AQ and QC specimens can be tested respectively. To ensure the measuring accuracy, the additionally vibratory-polished specimens were used and the Berkovich indenter was calibrated by using a reference specimen before tests.

2.4. Preparations of TEM foils

In order to evaluate the mechanical stability of RA grains by TEM, the focused ion beam (FIB, Carl Zeiss Crossbeam 540, at 30 kV) technique was performed to mill the cross-sectional TEM thin foils beneath the nano-indentation from AQ and QC specimens. Fig. 3 shows the major stages of the FIB preparation processes, in which the thin area of interests containing the indentation axis and one of three sides of the indent was selected to ensure that the cross section was right through the middle of the indent. A protective layer of platinum (~1.5 μm thick) was deposited over the surface of the indents with built-in gas injection source system. In the initial thinning, the Ga^+ beam was accelerated to 30 kV with a current of 5 nA, which was used to mill a staircase on both sides of the plate area. Then, the plate was further thinned to the foil less than 100 nm foil with lower Ga^+ current of 0.1 nA. The damaged layers formed by the initial high energy milling could be removed by applying smaller beam currents during subsequent cleaning and final thinning processes. Finally, the thin foil was lifted out and transferred onto the Cu grid prepared for TEM investigation.

Table 1
Chemical composition of the investigated SAE52100 steel (in wt.%).

C	Cr	Mn	Si	Cu	Ni	Mo	P	S	Fe
0.99	1.42	0.32	0.25	0.03	0.02	0.008	0.02	0.005	Bal.

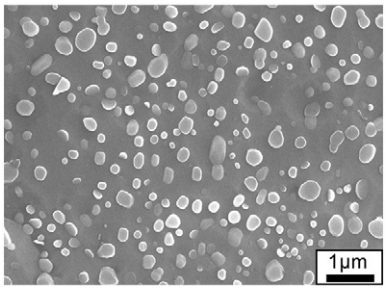

Fig. 1. Scanning electron micrograph of investigated steel in as-received state.

3. Results

3.1. Microstructures

The SEM micrographs of AQ and QC specimens are presented in Fig. 4, in which the bright irregular areas are RA and the gray areas are plate-shaped martensite. Two types of RAs with blocky and film morphologies were found in both specimens. It is seen in Fig. 4(a) that AQ specimen contains a substantial fraction of RA in which most are blocky but few in films. While, when the steel subjected to cryogenic treatment at −196 °C for 24 h (QC treatment), the amount of RA in QC specimen is reduced obviously, and most of them show film-like morphology as seen in Fig. 4(b).

Fig. 5 shows the X-ray diffraction profiles confirming the presence of RA and martensite in both AQ and QC specimens. It is known that the integrated intensity of the peaks for each phase is in proportion to its

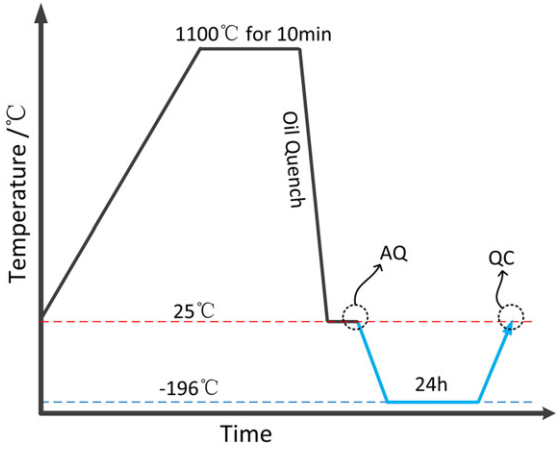

Fig. 2. Schematic AQ and QC heat treatment schedules for the investigated steel.