Journal of Nuclear Materials 407 (2010) 189–194

Contents lists available at ScienceDirect

Journal of Nuclear Materials

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

Effect of direct and reheated quenching on microstructure and mechanical properties of CLAM steels

J. Qiu [a], X. Ju [a,*], Y. Xin [a], S. Liu [a], Y.L. Wang [b], H.B. Wu [c], D. Tang [c]

[a] Department of Physics, University of Science and Technology Beijing, Beijing 100083, China
[b] State Key Laboratory for Advanced Metals and Materials, University of Science and Technology Beijing, Beijing 100083, China
[c] National/Engineer Research Center for Advanced Rolling Technology, University of Science and Technology Beijing, Beijing 100083, China

ARTICLE INFO

Article history:
Received 29 December 2009
Accepted 15 October 2010

ABSTRACT

Direct and reheated quenching on microstructural mechanical properties of China Low Activation Martensitic (CLAM) steel was investigated. Three direct quenching and tempering (DQ&T) CLAM steels were rolled at the same finish rolling temperature with different quenching rate, and their microstructures and mechanical properties were compared with those of a reheated quenching and tempering (RQ&T) CLAM steel. DQ&T process was proven to strengthen the tensile property but lower the toughness compared to RQ&T process due to the finer austenite grain size formed under RQ&T process. Tensile property first increased with quenching rate and then decreased. The reason for this was discussed.

1. Introduction

Reduced-Activation Ferritic/Martensitic (RAFM) steel has been considered as structural materials for the test blanket modules of International Thermonuclear Experimental Reactor (ITER) and the primary structural materials for DEMO [1]. China Low Activation Martensitic (CLAM) steel is one of the RAFM steel which has been developed by Chinese scientist and is also viewed as a leading candidate for the structural materials in the two blankets to be tested in ITER [2].

CLAM is quenched and tempered steel. Reheated quenching and tempering (RQ&T) is a general heat treatment to produce tempered martensitic steels. Direct quenching and tempering (DQ&T) process is the technical method which is developed as a part in the thermomechanical controlled process (TMCP) [3]. It is also appropriate for quenched and tempered steel. In order to compare the effect of these two different heat treatment processes, direct quenching and tempering (DQ&T) process was applied in this research. In the case of DQ process, the procedure included ingot hot rolling, direct quenching and tempering, while the RQ included ingot hot rolling, self cooling in ambient temperature, reheating to austenitizing temperature, quenching in cooling media and tempering [3–5]. This means DQ reduces energy consumption and decreases environment contamination. But the most important difference between DQ and RQ processes is in austenitizing condition. In the case of RQ sample, reaustenization temperature and time is sufficient and static recrystallization occurs completely, therefore, a homogeneous structure is created. As to DQ process, the microstructure is not homogeneous compared to that of RQ. The reason of this phenomenon is that in the case of DQ procedure, different recrystallization such as dynamic (during hot deformation) and meta-dynamic (between stands) occurred during the process [6].

Although some investigations have been done on RQ process for RAFM steels, no researches can be found in DQ process. In present work DQ and RQ processes were applied to CLAM steel in order to understand the mechanism of DQ and RQ. Correlations between microstructure and mechanical properties of three DQ and one RQ conditions were investigated.

2. Experimental procedures

2.1. Materials

The CLAM steel was produced by University of Science and Technology, Beijing. The steel was melted in a vacuum induction furnace and cast as a 100 kg ingot. The chemical compositions of CLAM steel used in this study were shown in Table 1.

2.2. Thermo-mechanical process

The CLAM steel was austenitized at 1200 °C, and was forged to an 83 mm thick plate. After the forged plate was heated to 1200 °C, the plate was rolled using a pilot plant rolling mill to a final thickness of about 11 mm. The rolling ratio was in the range of 15–20% at each pass, and brought the finish rolling temperature at about 960 °C. After quenching, the tempering procedure was carried out at 760 °C for 1.5 h.

* Corresponding author. Tel./fax: +86 10 62333921.
E-mail address: jux@ustb.edu.cn (X. Ju).

0022-3115/$ - see front matter © 2010 Elsevier B.V. All rights reserved.
doi:10.1016/j.jnucmat.2010.10.012

Table 1
Chemical composition of material studied (wt.%).

Material	C	Cr	W	V	Ta	Mn	Fe
CLAM	0.098	9.08	1.48	0.18	0.097	0.46	Bal.

In the case of direct quenching and tempering (DQ&T) process, after hot rolling, the plate was immediately quenched. Three quenching methods were used: water quench to room temperature, air cooling to room temperature, and spray quenching to 338 °C and then air cooled to room temperature, respectively. Table 2 showed the surface temperature of the plate just before DQ treatment. The plate was tempered at 760 °C for 1.5 h and then air cooling after quenching.

For the reheated quenching and tempering (RQ&T) process, after hot rolling, the plate was air cooled to room temperature. Subsequently, the plate was reaustenitized at 960 °C for 0.5 h and air cooling. The plate was also tempered at 760 °C for 1.5 h and then air cooling after quenching. Fig. 1 is the schematic drawing of hot rolling and heat-treatment of DQ&T and RQ&T process.

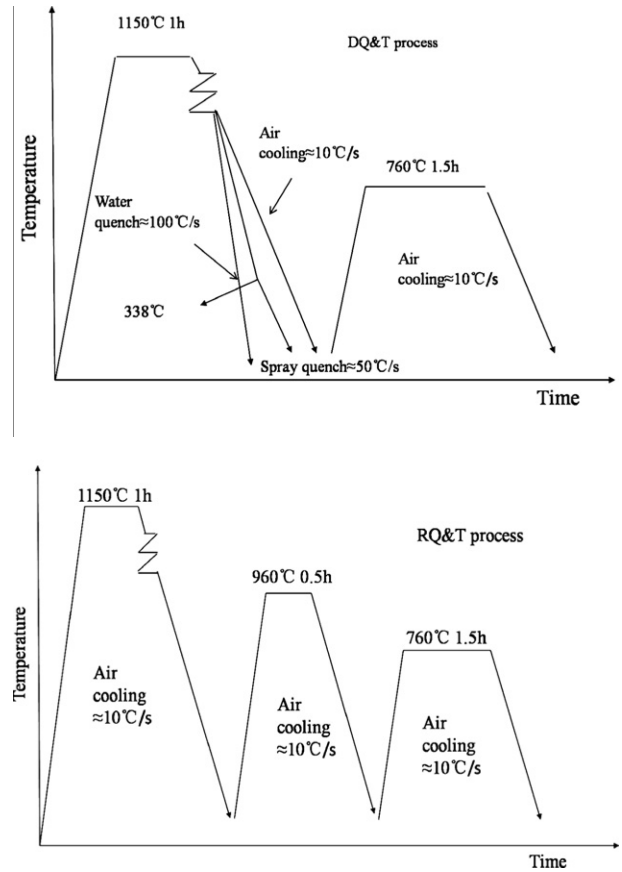

Fig. 1. Schematic drawing of manufacturing processes of CLAM steels with DQ&T and RQ&T.

2.3. Tests method

Cylindrical tensile sample of CLAM steel was 5 mm in diameter and 25 mm gauge length. The tensile tests were conducted in an electromechanical computer controlled machine at a strain rate of 10^{-3}/s. The stresses and elongations reported below are the nominal stress ($\sigma = F/S_0$) and nominal elongations ($\Delta = l/l_0$). The high temperature tests were done under vacuum (<0.01 Pa).

Standard ISO-V Charpy-specimens have been machined from the 11 mm plate material. The specimen size for Charpy impact testing is 10 mm × 10 mm × 55 mm. The specimen orientation was *T–L*. Impact curve was determined over the temperature range from −100 °C to room temperature.

In order to observe the microstructural of fractured specimens, Scanning Electron Microscope (SEM) was used.

3. Results and discussion

Fig. 2a–d are the optical micrographs of DQ&T and RQ&T CLAM steel. All are composed of fine lath-type martensite regardless of quenching rate. No bainitic structure could be identified and there was no evidence for bainitic transformation in CLAM steel. The grey etching regions were due to the existence of interlath carbides within martensite [7]. RQ&T specimens have irregularly spaced laths compared with DQ&T ones. Because in RQ&T process, reaustenization temperature and time were sufficient (960 °C and 0.5 h) and static recrystallization occurred completely, therefore, a homogeneous structure has been generated. In DQ&T specimens, they are elongated in parallel to the rolling direction within which a large amount of deformation is present, whereas many equiaxed grains are observed in RQ&T specimens. This means the influence of the post-quench tempering on the martensite lath morphology was found to be small compared with that of quenching rate.

Fig. 2 showed that austenite grain size (AGS) in RQ&T specimens was finer than that in DQ&T one. The CLAM steel was preheated to 1200 °C and then goes thermo-mechanical hot rolling process. Therefore, coarse austenite structure has been generated.

Fig. 3 showed the variation of tensile properties (TS, YP) of DQ&T specimens with increasing cooling rate at room temperature (RT) and 600 °C. Three specimens were tested under each of these conditions. For comparison, the results of RQ&T were also plotted. Within this quenching rate range, tensile and yield strengths first increased with an increase in cooling rate and then decreased. TS&YP peaked at the value of spray quenching and tempering specimens.

This phenomenon can be explained from two aspects. Firstly, coupled with Fig. 2, lath martensite in RQ&T CLAM steel has a three-level hierarchy in its morphology: martensite lath, block, and packet. Fig. 2 showed little difference in the microstructure of RQ&T specimens, except for the packet size. The martensitic packet size decreased with increasing quenching rate. Tsuzaki et al. [8] researched the effect of cooling of lath martensite in Ni maraging steel. They showed that the packet size and block width decreased with increasing cooling rate for all the alloys examined. The strength of low-carbon steels with lath martensite structure increases as the packet size decreases [9]. The above results suggest that an increasing quench rate may lead to an increase in strength. Therefore, tensile and yield strengths in water quenching sample should higher than that in air cooling and spray quenching ones according to the optical micrographs. But results in tension were not agreement with it. Tensile and yield strengths peaked at the value of spray quenching specimens. Secondly, carbon segregation or carbide precipitation during the quench may also influence strength of full martensite structure. As can be seen from Fig. 2, grey etching regions

Table 2
Surface temperature prior to direct quenching.

Quench method	Surface temperature prior to direct quenching (°C)
Water quench	959
Air cooling	938
Spray quench	961

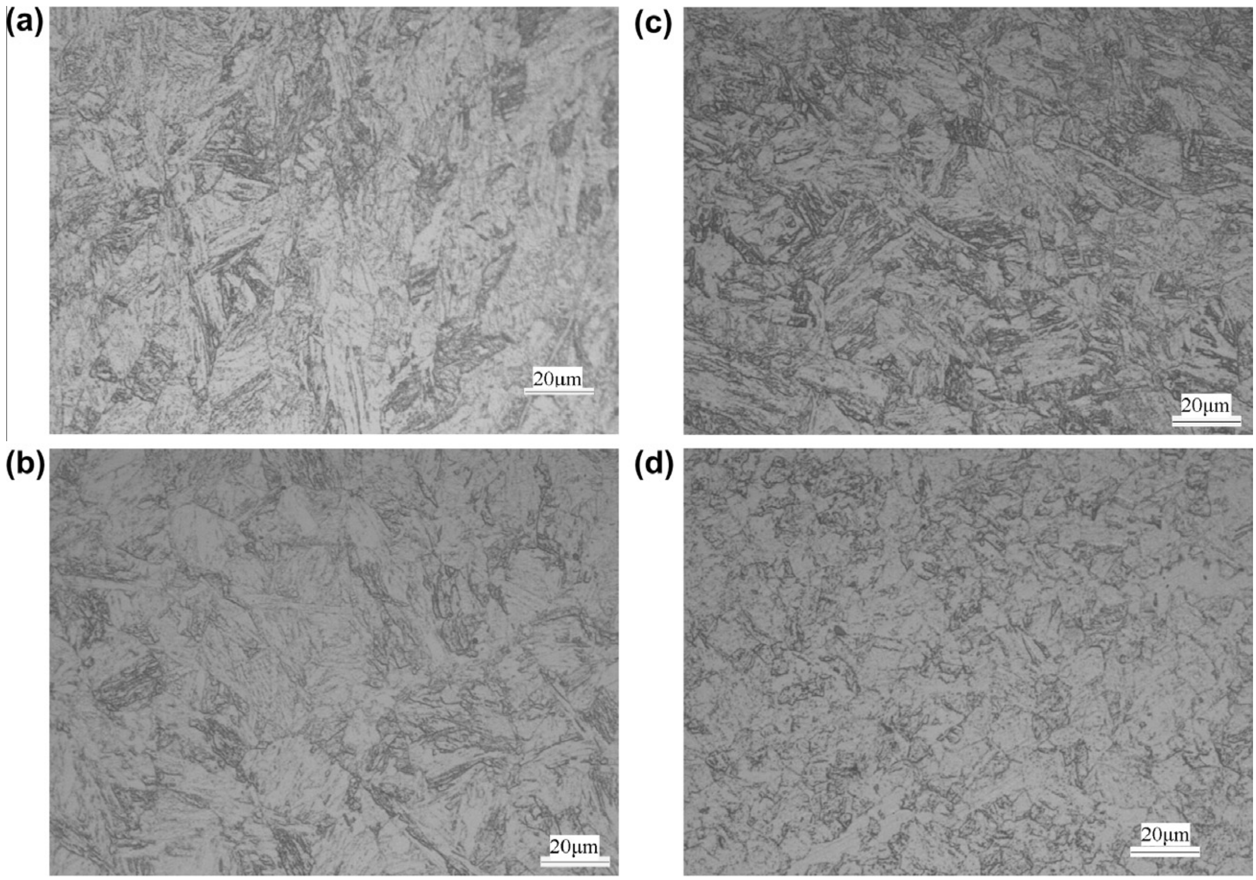

Fig. 2. Optical micrographs of (a–c) DQ&T samples (d) RQ&T sample. (a) Water quenching and tempering. (b) Spray quenching and tempering. (c) Air cooling and tempering. (d) Reheated quenching and tempering.

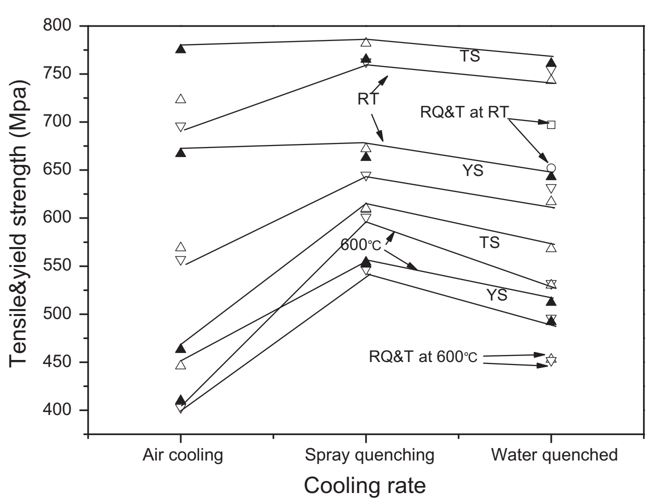

Fig. 3. Effect of the cooling rate on tensile and yield strength of DQ&T and RQ&T samples.

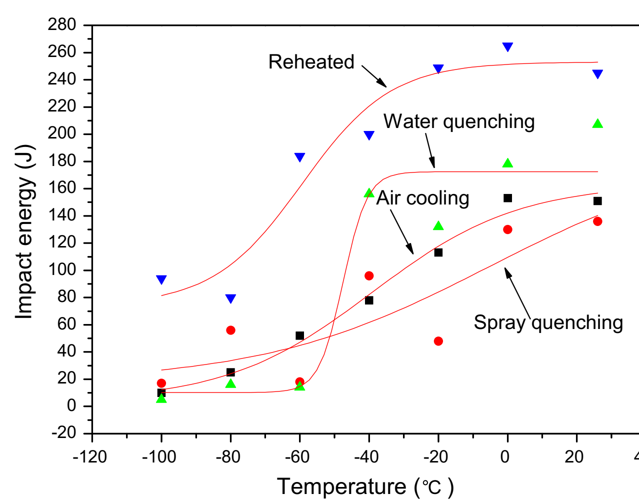

Fig. 4. Impact curves for the DQ&T and RQ&T sample of CLAM steel.

distribution in air cooling specimens were more uniform than that in water quenching and spray quenching ones. As mentioned above, the grey etching regions were due to the existence of interlath carbides within martensite. It suggested that carbides in air cooling specimens were more dispersive distribution. Ansell et al. [10] have investigated the effect on strength of AISI 440A and 4340 steel and demonstrated that the strength increased with decreasing cooling rate. They suggested that the beneficial effect on the strength of the slowly-quenched steel is caused by a dispersion-strengthening effect due to carbon segregation to the structural imperfections in the austenite prior to austenitic transformation. These results suggested that the strength of CLAM steel increased as the quenching rate decreased. So the strength of CLAM steel also related to carbide precipitation. These two reasons affected the strength in two opposite directions and lead to peak value which formed at spraying quenched specimens. As seen in Fig. 3, there was a higher degree of scatter in TS&YP values for air cooling samples than spray and water quenching ones, this maybe due to carbon segregation patterns and extent of carbide precipitation during the slow quenching, as mentioned above. By comparison between DQ&T and RQ&T conditions, it was observed that the TS&YP values of RQ&T specimens were close to that of air cooling ones at any temperatures (RT and 600 °C).

Fig. 4 illustrated the variation of impact toughness of DQ&T and RQ&T specimens. It was observed that DQ&T specimens showed similar upper-shelf energy (USE) and lower-shelf energy (LSE). Klueh investigated the effect of heat treatment on the impact behavior of eight experimental heats of Reduced-Activation Ferritic steels [11], and they found that the quenching rate had essentially no effect on the toughness of high-chromium martensitic steels. The results from this current study are consistent with it. While RQ&T showed a remarkable increase in the value of USE and LSE. With regard to RQ&T condition, this result coupled with the optical micrographs in Fig. 2 agreed with the fact that a finer grain size and higher contiguity ratio is expected to impart higher toughness. As mentioned above, AGS in RQ&T specimens was finer than that in DQ&T. So the impact toughness of RQ&T sample was superior to DQ&T one.

Figs. 5 and 6 showed the fracture surface of each condition. From the fractographs, it is easily seen that the fracture of all the −100 °C specimens of DQ&T and RQ&T occurs by transgranular

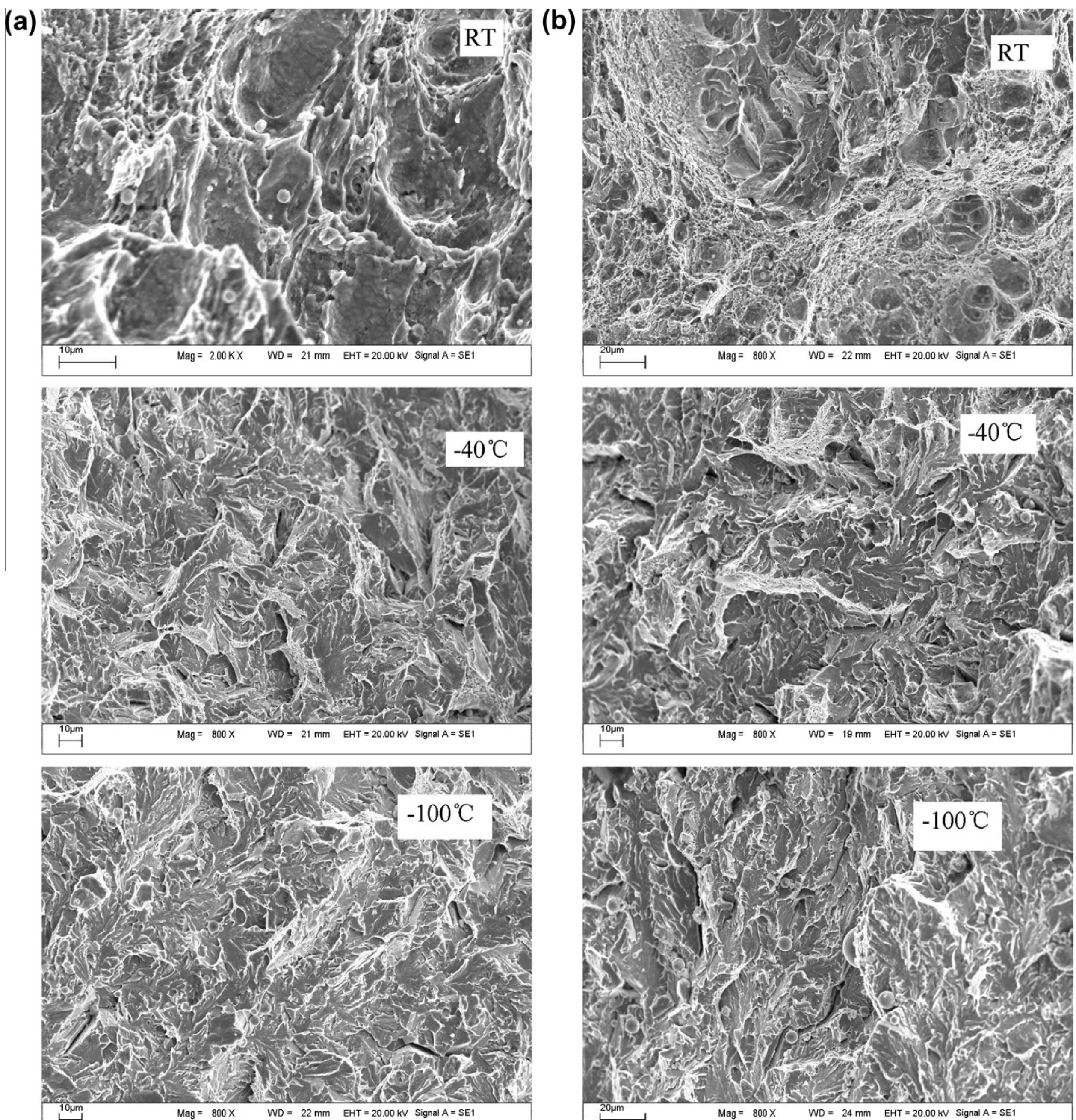

Fig. 5. SEM fractographs of Charpy tested specimens under DQ&T condition. (a) Water quenching and tempering. (b) Spray quenching and tempering. (c) Air cooling and tempering.

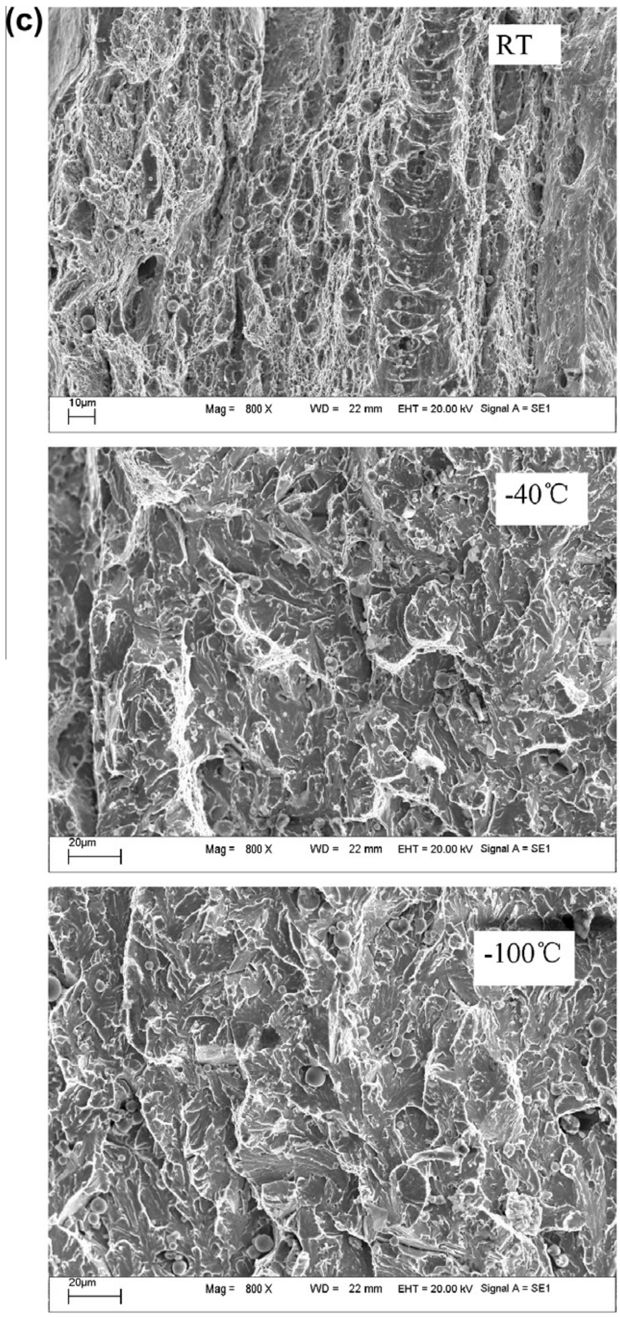

Fig. 5 (*continued*)

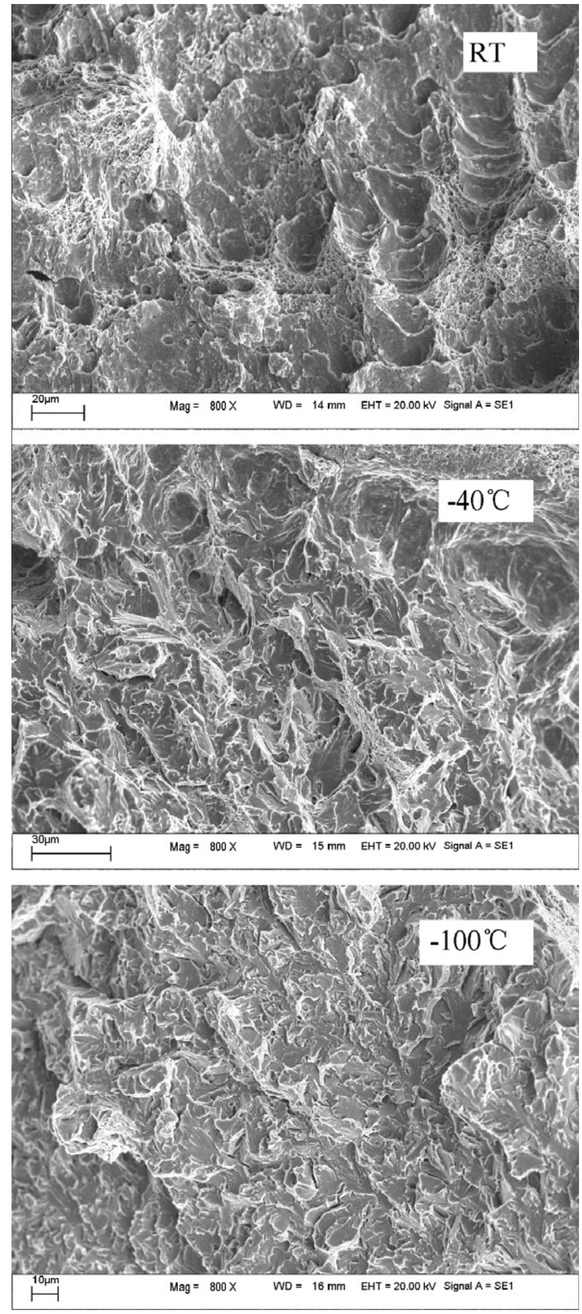

Fig. 6. SEM fractographs of Charpy tested specimens under RQ&T condition.

mode showed no grain deformation, indicating a brittle structure. This fracture is typically brittle. But the RQ&T specimens had higher impact toughness than DQ&T ones, as shown in Fig. 4. On the other hand, cleavage failures were predominant in the −40 °C impact test specimens of DQ&T with small and shallow dimples, while RQ&T specimens with the highest impact value fracture by the mixed mode of localized quasi-cleavage and mainly ductile dimple rupture, which were characterized by cleavage and ductile (dimples) failures. This indicates that the fracture is in the ductile to brittle transition. This change in fracture mode is consistent with the change in impact toughness. So the DBTT of RQ&T specimens was about −40 °C, which is lower than that of DQ&T ones. At room temperature, all specimens showed grain deformation, originating from the plastic structure. The fracture mechanism with bigger and deeper dimples was predominant in the impact test specimens of RQ&T, which had higher impact toughness.

4. Conclusion

In present work, DQ&T and RQ&T processes were applied to CLAM steel. The relationship between microstructure and mechanical properties was investigated. The conclusions are summarized as follows:

(1) Microstructure of DQ&T and RQ&T were investigated. PAG in DQ&T specimens were elongated in parallel to the rolling direction while equiaxed grains are observed in RQ&T ones. This means post-quench tempering on the substructure was found to be small compared with that of quenching rate. In addition, PAG in RQ&T was finer which led to the toughness under RQ&T process was superior to DQ&T. Fractographs of Charpy tested specimens showed that DBTT of RQ&T specimens was lower than DQ&T ones.

(2) DQ&T process can strengthen tensile property compared to RQ&T process. The tensile property increased with quenching rate and then decreased. The YS and TP peaked at the value of spray quenching condition. Increasing quench rate may lead to an increase in strength due to form finer martensitic lath, while decreasing quenching rate caused a dispersion-strengthening effect due to carbon segregation to the structural imperfections in the austenite prior to austenitic transformation, which also strengthened the CLAM steel. So peak value was formed.

Acknowledgement

This work was supported by a grant from the Major State Basic Research Development Program of China (973 Program) (No. 2008cb717802).

References

[1] R.L. Klueh, D.S. Gelles, S. Jitsukawa, A. Kimura, G.R. Odette, B. van der Schaaf, M. Victoria, J. Nucl. Mater. 307–311 (2002) 455–465.
[2] Q. Huang, C. Li, Y. Li, M. Chen, M. Zhang, L. Peng, Z. Zhu, Y. Song, S. Gao, J. Nucl. Mater. 367–370 (2007) 142–146.
[3] Chiaki OUCHI, ISIJ Int. 41 (2001) 542–553.
[4] Woong-Seong Chang, J. Mater. Sci. 37 (2002) 1973–1979.
[5] J. Katsumata, O. Ishiyama, O. Matsumoto, T. Inoue, M. Nakamura, Y. Matsushima, Y. Nakatani, in Proceeding of Physical Metallurgy of Direct-Quenched Steels; Chicago, Illinois, USA. The Minerals, Metals and Materials Society (TMS), 1993, pp. 247–268.
[6] A.H. Meysami, R. Ghasemzadeh, S.H. Seyedein, M.R. Aboutalebi, Mater. Des. 31 (2010) 1570–1575.
[7] M.C. Tsai, C.S. Chiou, J.S. Du, J.R. Yang, Mater. Sci. Eng. A 332 (2002) 1–10.
[8] K. Tsuzaki, T. Maki, I. Tamura, Scripta Mater. 21 (12) (1987) 1693–1698.
[9] Hiromoto Kitahara, Rintaro Ueji, Nobuhiro Tsuji, Yoritoshi Minamino, Acta Mater. 54 (2006) 1279–1288.
[10] G.S. Ansell, S.J. Donachie, W. Messler, Metall. Trans. B 2 (1971) 2443–2449.
[11] R.L. Klueh, P.J. Maziasz, D.J. Alexander, J. Nucl. Mater. 179–181 (1991) 679–683.