The Eurasia Proceedings of Science, Technology, Engineering & Mathematics (EPSTEM)
ISSN: 2602-3199

The Eurasia Proceedings of Science, Technology, Engineering & Mathematics (EPSTEM), 2020

Volume 11, Pages 5-8

IConTES 2020: International Conference on Technology, Engineering and Science

Effects of Deep Cryogenic Treatment on the Microstructural Properties of Medium Carbon Spring Steels

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Abstract: The cryogenic treatment is a complementary procedure that has been applied to a variety of materials to improve their mechanical and physical properties. It was first commercially recognized as an effective method in achieving complete martensitic transformation in the alloyed steels. In this study, microstructural investigations were carried out in order to relate the microstructural properties to the mechanical properties following the cryogenic treatment. For this purpose, the conventional heat treatment (CHT) and the deep cryogenic treatment (DCT: -196°C) procedures were applied to various medium carbon spring steels. Microstructural examinations were carried out by using scanning electron microscopy. The martensite lattice parameters and the amount of retained austenite were measured by using a high-resolution X-ray diffractometer. Rietveld analysis was used to deconvolute the overlapping peaks of martensites.

Keywords: Steels, Cryogenic Treatment, Microstructure, Alloying

Introduction

Martensite is a microstructure that increases the strength and wear resistance of steels. It is generally accepted that martensite has a body-centered tetragonal (bct) structure. The linear relationship between tetragonal martensite and the carbon content is traditionally expressed as in Eq. (1) (Lu, Yu, & Sisson, 2017).

$$c/a = 1 + 0{,}045 \text{ wt. }\%C \quad (1)$$

However, Nishiyama et al. used steels with a carbon ratio of more than 0.6% by weight while demonstrating this equation. Since the c / a ratio could not be determined correctly in steels whose carbon ratio was lower than 0.6% at that time, they assumed that steels containing lower carbon content would also tend to have the same trend.

In the study conducted by Lu et al., the c/a ratios of martensite in the structure were calculated after normalization, austenitization and quenching processes using low carbon AISI 9310, medium carbon AISI 4140, AISI 4150 and AISI 4161, high carbon AISI 1080, AISI 52100 steels. Accordingly, it was determined that as the carbon ratio in steel increases, the angles and the intensity of both (200) and (002) martensite peaks decrease. It was observed that the peaks began to overlap with the decrease of d distance between (200) and (002) peaks in AISI 9310, AISI 4140, AISI 4150 and AISI4161 steels which carbon ratio is below 0.6%. This situation created difficulties in calculating the c/a ratio. It has been determined by SEM examinations that the microstructure is generally lath martensite in AISI 9310 and AISI 4140 steels, plate martensite in AISI 52100 steel and both martensite types in AISI 4161 and AISI 1080 steels. Using the Rietveld analysis, the researchers stated that the traditional c / a calculation model (above equation) can be used in steels containing more than

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0.6% carbon. By deconvoluting the peaks of (002) and (200), it is stated that martensite has bct structure and the c/a ratio is as follows in Eq (2).

$$c/a = 1 + 0{,}031 \, \%C \qquad (2)$$

Although it is generally accepted that martensite lattice structure in carbon steels is bct, it has been reported in the study conducted by Lobodyuk et al. that martensite cage cannot form a smooth bct structure in steels with a carbon ratio of 0.2 to 1.5%. Besides, the mechanism of local distortions in the crystalline structure to change the c lattice parameter is explained using a tetragonal-ish (pseudo-tetragonality) structure. The researchers reported that the crystalline structure of the martensite lattice should be named pseudo-tetragonal because it contains interstitial C atoms.

In the research conducted by (Maruyama, Tabata, & Kawata, 2020), the crystal structure and carbon dispersion behavior of quenched steel with 10 different carbon compositions with carbon ratios between 0.07 and 0.8 were investigated using Rietveld analysis and atom probe tomography (APT). A tetragonal structure has been observed in steels with a carbon composition of 0.1 to 0.7% by weight. It has also been found that during cooling, the carbon atoms tend to remain in the lath martensite regions with dense dislocation. When all steels were examined, it was found that the amount of carbon in solid solution of martensite steels was much more than it could be dissolved in the bcc-Fe structure. It has been stated that since carbon atoms precipitate inhomogeneously by forming carbide or cluster in dislocations due to auto-tempering. The martensite structure provides excess solid solubility in auto-tempered low and medium carbon steels due to tetragonal distortions caused by the slow kinetics of tetragonality decreasing during cooling. Therefore, they stated that it is possible to control tetragonality by changing the cooling rate.

In this study, cryogenic process was applied to three medium carbon spring steels containing different alloying elements and the effect of cryogenic process was investigated microstructurally using SEM. Rietveld analysis was applied to the samples to quantitatively determine the tetragonality of martensite.

Method

Three spring steels with different alloys were selected in the study. The average chemical compositions of spring steels are given in Table 1. Conventional heat treatment (CHT) and deep cryogenic process (DCT) at -196°C were applied to steels and the effect of the cryogenic process on microstructural properties was investigated. The flow chart of the experiment is given in Figure 1.

Table 1. Average Chemical Compositions of Spring Steels

Material	%C	%Si	%Mn	%Cr	%V	%Mo
55Cr3	0,57	0,30	0,85	0,80	-	-
51CrV4	0,50	0,25	0,90	1,10	0,12	-
52CrMoV4	0,50	0,25	0,90	1,10	0,10	0,25

For XRD (Rietveld) analysis, the samples were electrolytically polished in accordance with ASTM E1558 - 99 standard. Experiments were carried out with Panalytical EMPYREAN brand XRD device. Rietveld analyzes were carried out with Maud 2.92v analysis program. The PDF 4+ program containing the ICDD database was used for the "cif" (crystallographic information file) files required for Rietveld analysis.

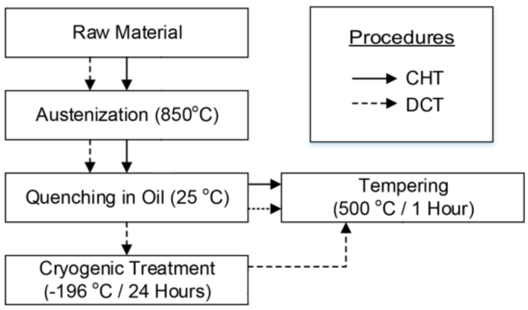


Figure 1. Flow chart of heat treatment procedures

Results and Discussion

In order to examine the formal effect of the cryogenic process on the microstructure, the samples were examined using a scanning electron microscope. SEM microstructures of CHT and DCT samples of 55Cr3 are presented in Fig. 2a and Fig 2b, respectively. It is seen that the DCT sample has a significant increase in the density of carbides as compared to the CHT sample. Besides, a coarsening was observed in the carbide structures. The carbide structure becomes more rounded and is more homogeneously distributed in the matrix.

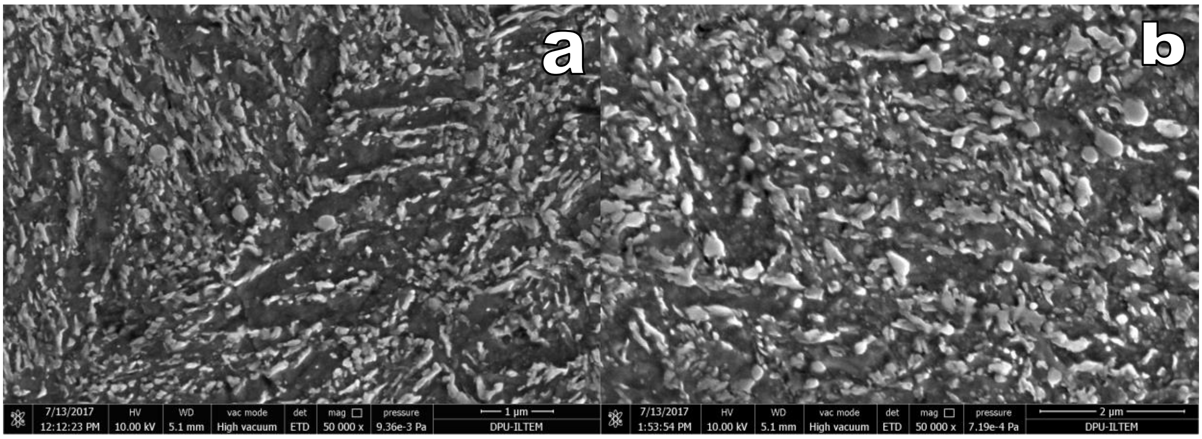
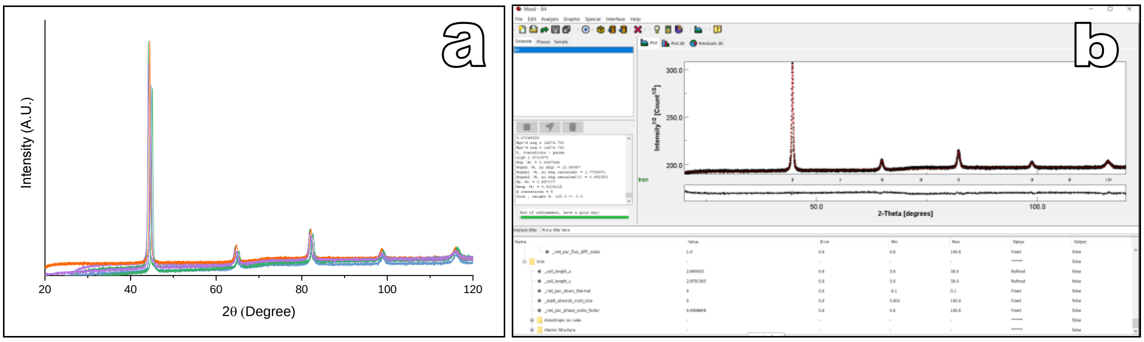


Figure 2. SEM pictures of the samples (a) 55Cr3 – CHT (b) 55Cr3 – DCT

In Figure 2a, XRD patterns of the samples are presented. As expected, the amount of retained austenite in the samples is minimal and the carbide peaks are not evident. The results from the Rietveld analysis performed as seen in Figure 2b are presented in Figure 3.



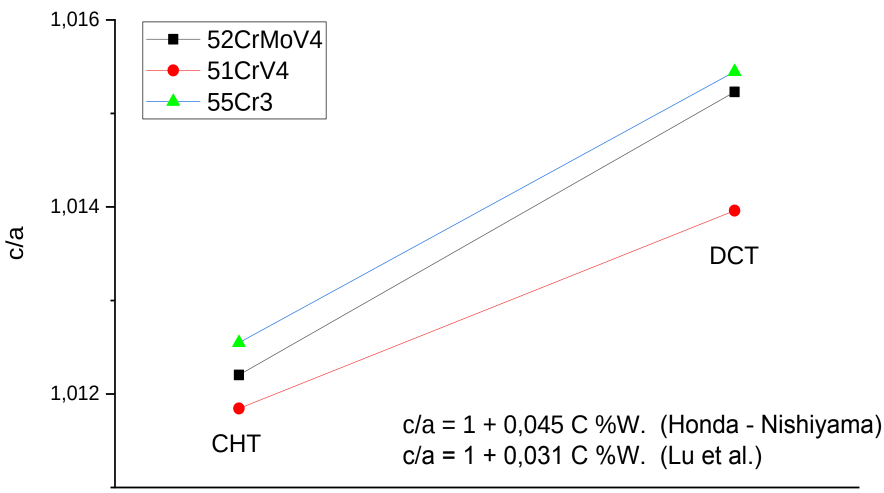


Figure 3. Results of Rietveld analysis performed as seen in Figure 2b

According to the results of Rietveld analysis, an increase in the tetragonality of martensite was observed in all samples after cryogenic treatment. In the case of cryogenic effect, basic parameters that define the change of tetragonality of martensite are mentioned. These are the austenite - martensite transformation that occurs depending on the cryogenic temperature and time (Villa, Hansen, & Somers, 2017), the blurring of the grain boundaries of martensite that changes depending on the tempering temperature and the carbide formation (Su, Chiu, Chen, Lin, & Pan, 2014). It has been reported in our previous studies that these microstructural changes add toughness to the steel material without losing its strength (Özden & Anik, 2020). The results obtained confirm the work of Lu et al. for medium carbon spring steels

Conclusion

Homogenization and increased carbide precipitation were observed in the SEM examination performed as a result of the cryogenic process applied to medium carbon spring steels. It has been observed that this effect increases as the alloying increases. After the Rietveld analysis performed after XRD analysis, it was reported that the c / a ratio in the martensite structure, ie tetragonality, increased.

Acknowledgment

Financial assistance from Eskisehir Osmangazi University Research Fund is gratefully acknowledged (Project No: 201615A230). The authors wish to thank MMD Mechanical and Materials Technologies R&D Consulting Engineering Services Industry and Trade Limited Company for the deep cryogenic treatments.

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