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Correlation between Tetragonality and the Residual Stress in Cryotreated Spring Steels

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Abstract: In the conventional heat treatment process (CHT), a significant amount of compressive stress occurs in the material as a result of the quenching process. After the tempering process, it is seen that the materials lose those compressive stress. This loss can be explained by the formation of carbide structures and the loss of tetragonality of the supersaturated martensite structure. The cryogenic treatment is a complementary process that it has been given to a miscellany of materials to improve their mechanical and physical attributes. 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. The martensite lattice parameters and the amount of retained austenite were measured by using high-resolution X-ray diffractometer. Rietveld analysis was used to deconvolute the overlapping peaks of martensites.

Keywords: Steel, Cryogenic, Residual stress, Rietveld

Introduction

The notion of chilling below zero degrees was proposed in the mid-1920s and subjected to extensive research in the 1940s, which created the groundwork for the complementing Cryogenic process (Villa et al., 2017). In particular, the Deep Cryogenic Treatment (DCT) procedure, which is carried out below -190°C, is frequently used to enhance the mechanical characteristics of steels (Preciado & Pellizzari, 2014). The conversion of retained austenite to martensite, a rise in carbide deposits, and enhanced homogeneity in the matrix are microstructural factors that account for this improvement in mechanical characteristics (Özden & Anik, 2020).

The quenching procedure in the conventional heat treatment method (CHT) causes the material to experience a sizable amount of compressive stress. It is observed that the samples substantially lose these compressive stresses during the tempering process. (Bensely et al., 2008). The precipitation of nano-fine carbides in the structure and the elimination of tetragonal supersaturated martensite are the causes of this reduction (Preciado & Pellizzari, 2014). Molybdenum and other carbide-forming elements are crucial in preventing grain development in the primary carbide structure. Additionally, they promote the precipitation of secondary carbide structures, which preserve compressive stresses in the structure (Michaud et al., 2007). Since spring steels operate under a variety of loads, it is extremely desirable to keep these compression stresses in the structure (Myeong & Yamabayashi, 1997).

In this work, three medium carbon spring steels (55Cr3, 51CrV4, and 52CrMoV4) with various alloying components were subjected to a cryogenic procedure. In order to quantify the tetragonality (c/a ratio) of martensite and the impact of alloying elements on residual stress, the effect of the cryogenic process was examined using X-Ray Diffraction (Rietveld Refinement and Residual Stress) technique.

- Selection and peer-review under responsibility of the Organizing Committee of the Conference

Method

Medium carbon spring steels with compositions of 55Cr3, 51CrV4, and 52CrMoV4 were used in the investigation. ÇEMTAŞ (Bursa) produced the steels in a 55 mm diameter using the continuous casting method. After the casting, they were hot rolled to a diameter of 19 mm and then cut into 20 cm lengths. Table 1 displays the results of optical emission spectrometry analysis in weight %.

Table 1. Optical emission spectrometry analysis results (Weight %)

Steel	C	Si	Mn	Cr	Mo	V	P	S
55Cr3	0,57	0,30	0,85	0,91	-	-	0,010	0,005
51CrV4	0,52	0,28	0,84	0,93	-	0,13	0,008	0,004
52CrMoV4	0,54	0,26	0,87	1,12	0,19	0,11	0,009	0,003

XRD experiments were carried out with a Panalytical Empyrean diffractometer at a speed of 0.25° min^{-1} between the 2θ range of 20° - 120°. The PDF 4+ program containing the ICDD database was used for the CIF files required for Rietveld analysis. Rietveld analyzes were carried out with MAUD (materials analysis using diffraction) 2.92v program. The program was capable to refine the material structure and the microstructure simultaneously with the Marquardt least-squares method (Gasan & Erturk, 2013). The refinement was continued with new steps until the goodness of fit (GOF) coefficient stopped converging to 1.

XStress 3000 diffractometer was used to determine the residual stresses of the samples. Cr-Kα X-ray source was used throughout the experiment. 156.4° diffraction angle, 0.3 Poisson ratio, and 211 GPa Young's Modulus were selected as the experimental parameters. Average Diffraction data were measured in seven tilt angles between -40° and +40° with 20 second exposure time. These seven tilt angles were averaged to determine the residual stress and FWHM values of the sample.

Results and Discussion

X-Ray Diffaction and Rietveld Analysis

Phase evaluations of conventionally hardened and cryogenically treated spring steels used in the study were carried out using XRD technique. XRD patterns of all samples are presented in Figure 1. In the XRD examination, all the structures were characterized as tempered martensite. Steels have a very high hardenability with the effect of their carbon ratios and alloying elements. Therefore, the low amount of residual austenite in the samples could not be detected in the XRD analysis. This indicates that the amount of austenite phase is below the minimum XRD phase detection percentage of 3%. The 2θ° angles of the detected peaks are presented in Table 2.

Table 2. 2θ° angles of the detected peak

Sample	2θ° - α'(110)	2θ° - α'(200)	2θ° - α'(211)	2θ° - α'(220)
CHT-52CrMoV4	44,159	64,432	81,791	98,453
CHT-51CrV4	44,214	64,422	81,806	98,442
CHT-55Cr3	44.255	64,462	81,741	98,386
DCT-52CrMoV4	44,574	64,769	82,080	98,715
DCT-51CrV4	44,619	64,839	82,237	98,807
DCT-55Cr3	44.606	64,830	82,184	98,821

After the cryogenic treatment, the samples have a shift of about 0.4 degrees compared to the conventionally treated samples. This shift can be associated with a change in the lattice parameters. Although it cannot be determined due to the % amount, the plastic deformation effect of the new martensite structure formed by the transformation of the retained austenite structure can be shown as the reason for this shift.

Looking closely at the XRD patterns, it can be seen that there is a noticeable expansion effect at the peaks of the cryogenically treated samples as a secondary effect of cryogenic treatment. This expansion effect has been reported in the literature as an extension of the increasing strain in the structure (Warren & Averbach, 1950). It is also possible to think that this strain indicates an increase in the dislocation density. These stresses created by the dislocation density are directly related to heterogeneous stress distributions that can act as a driving force for carbide formation during the cryogenic process.

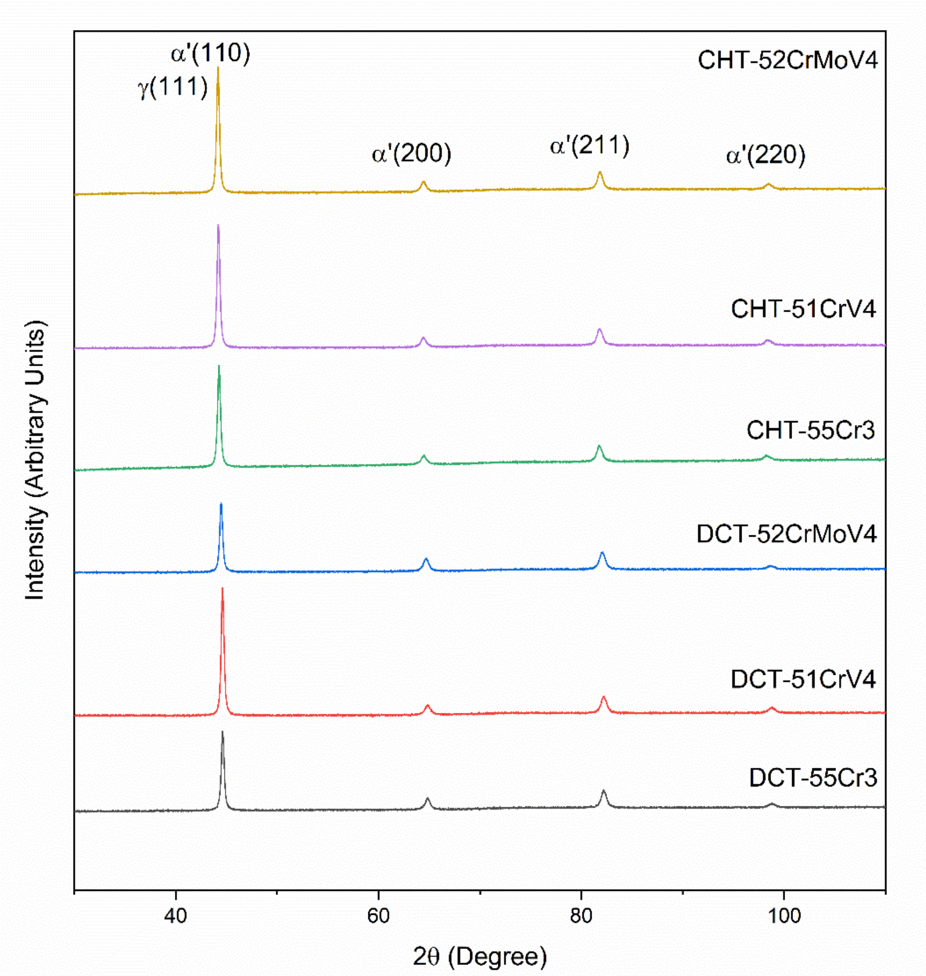

Figure 2. XRD patterns of the samples

In order to quantitatively investigate the effect of cryogenic treatment on the lattice structure of tempered martensite, Rietveld analysis was performed on the samples. The lattice parameters (a, c) and tetragonality (c/a) obtained as a result of the analysis are presented in Table 3.

Table 3. Rietveld (Space Group P42 n:1 a_M: 2.8665 Å) PDF Card - 04-003-1451

Procedure	Sample	a (Å)	c (Å)	c/a
Q	52CrMoV4	2.865949	2.921633	1.019429
	51CrV4	2.865861	2.923466	1.020100
	55Cr3	2.865536	2.918508	1.018485
CHT	52CrMoV4	2.866171	2.894268	1.009803
	51CrV4	2.865433	2.894786	1.010244
	55Cr3	2.866215	2.892435	1.009148
DCT	52CrMoV4	2.865857	2.898057	1.011236
	51CrV4	2.866244	2.899036	1.011441
	55Cr3	2.866919	2.895714	1.010044

Considering Table 3, it can be said that the deep cryogenic process causes plastic deformation in the cage, as in the XRD results, and plays an active role in preserving the tetragonality of the martensite. The deformation caused by the transformation-induced fresh martensite and carbide formation and the effect of locking the slip planes increased the tetragonality of the martensite structure.

Residual Stress Analysis

Residual stress measurement was applied to all heat treated samples to measure the effect of the deformation created by the cryogenic process on the microstructure, which can be directly related to the mechanical properties. Tests were also applied to the quenched samples without tempering (Q) as the base sample, aiming to determine the compression stresses created by the transformation itself. Residual stress results are presented in Table 4.

Table 4. Residual stress results of samples

Sample	55Cr3		51CrV4		52CrMoV4	
	MPa	FWHM	MPa	FWHM	MPa	FWHM
Q	-102,9 (± 9,3)	3,23 (± 0,05)	-147,3 (± 5,4)	3,36 (± 0,07)	-154,46 (± 6,80)	3,23 (± 0,08)
CHT	42,3 (± 10,9)	2,22 (± 0,06)	43,1 (± 10,6)	2,12 (± 0,06)	32,47 (± 4,75)	2,70 (± 0,05)
DCT	-51,7 (± 7,3)	2,47 (± 0,07)	-67,0 (± 10,0)	2,76 (± 0,07)	-108,10 (± 6,56)	3,43 (± 0,07)

When the table is examined, it is seen that a significant amount of residual compression stress occurs as a result of the quenching (H) process, as expected. After the tempering process, it is seen that the specimens lose their compressive stress to a great extent. This reduction can be attributed to the precipitation of nanofine carbides in the structure and the removal of tetragonal supersaturated martensite (Bensely vd., 2008; Preciado ve Pellizzari, 2014). Conversion of retained austenite to martensite in cryogenic samples creates compression stresses in the internal structure and causes crystal defects in the form of dislocations and twinnings. During the martensitic transformation at cryogenic temperatures, plastic deformation is accompanied by the volume effect created by the martensitic transformation. This resulting dislocation and/or twinning slides in the plane and engulfs the inert carbon atoms and causes carbon clusters to form. During tempering, these carbon clusters serve as nucleation sites for the precipitation of fine carbides. The increase in the density of both crystal defects and nanocluster carbides caused greater locking of the already dense dislocations and therefore kept the residual stress in the system, limiting the level of relaxation in cryogenically treated samples. In particular, 52CrMoV4 steel, which contains the most intense carbide-forming element, is the steel group that loses the least compressive stress in the sample (DCT-52CrMoV4) structure.

Conclusion

In the study, the effects of cryogenic treatment on the residual stress and tetragonality of different medium carbon spring steels were investigated. Accordingly, it was observed that the efficiency of deep cryogenic processing (DCT) increased in the presence of carbide-forming alloying elements, and the desired compressive stress could be maintained in the samples. It is possible that the plastic deformation and dislocation density created by increasing tetragonality values can be associated with the persistence of these stresses in the structure.

Scientific Ethics Declaration

The authors declare that the scientific ethical and legal responsibility of this article published in EPSTEM journal belongs to the authors.

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