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RESEARCH ARTICLE

Volume fraction of retained austenite in 1.2842 tool steel as a function of tempering temperature§

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Abstract: Untransformed austenite during quenching process is known as retained austeinite. The quantitative determination of the retained austenite is of great importance to the steel mechanical properties. Its percentage has a large effect on the mechanical properties and service life of components. The amount of retained austenite in through-hardened tool steels should be kept at its optimum level in order to minimize size change, and increase service life. In this study, the influnce of tempering temperature on the amount of retained austenite was evaluated by using X-ray diffraction phase analyses. It was seen that tempering at low temperatures resulted in small amount of retained austenite for the studied steel.

Key Words: Tool steel, Tempering temperature, X-ray diffraction, Retained austenite

1. Introduction

Tool steels are quenched and tempered at different conditions depending upon the type of steel being used and service requirements. The quenching and tempering operations increase the toughness and strength of the material for better service life. The selection of the proper tool steel and the application of the proper heat treatment processes affect the performance of the tools used in engineering applications. [1].

The microstructure of hardened tool steels made up of martensite and retained austenite (RA) when these are quenched to the room temperature. This may be either undesirable or desirable depending upon the application and amount of retained austenite. In some type of applications, the mixture of retained austenite and martensite are desirable due to compensation of the adverse effects of these phases. The amount of retained austenite depends on the chemical composition, austenizing temperature, quenchant temperature, tempering temperature and subsequent mechanical treatments. Generally, a small amount of retained austenite may cause increase in ductility and toughness, but too much retained austenite adversely affects the dimensional stability mechanical properties of steel components. For example, retained austenite at a certain level could be helpful to increase service performance when it is present in the carburized cases of gear teeth.

On the other hand, unstable retained austenite is not desired for components used in the tool and die industry. It leads to a short service life due to the loss of strength, hardness and dimensional stability in fully hardened tool steels [2-4]. Therefore, it is important to control the percentage of retained austenite in order to get optimum service life and mechanical properties for a given application [5-7].

Tolerable amount of retained austenite depends on the type of steel and service conditions. However, it is difficult to define exactly the tolerable amount of retained austenite due to insufficient data [3]. To solve this problem, each research in this aspect has been concentrated to find the effect of untransformed austenite on the performance of a specific steel in service.

These studies generally were concentrated on the evaluation of the effect of retained austenite formed at various heat-treating conditions on the mechanical properties and to find the optimum heat treating conditions and/or composition lead to a better service life for a given application [8-10].

Optical microscopy, scanning electron microscopy, transmission electron, magnetic methods, dilatometer, Mossbauer spectroscopy and X-ray diffraction (XRD) techniques are the common methods that have been used to determine the amount of RA include [11-13]. The suitable method is usually selected based on the percentage of retained austenite, level of accuracy and type of mate-

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rial. With the development of diffractometer technology, quantitative determination of retained austenite content in heat-treated steels by x-ray diffraction has provided a reliable means of controlling properties and ensuring quality [14]. XRD methods are generally gives accurate measurements. This method can used to determine the amount of retained austenite to 0.5% level [15].

In this study, the effect of tempering temperature on the amount of retained austenite was investigated for the hardened 1.2842 (O2) cold work tool steel. X-ray diffraction method was used to estimate the volume fraction of retained austenite in as quenched and tempered specimens. At different tempering temperatures, volume fractions of retained austenite were estimated. Then, obtained results were interpreted to evaluate the influece of tempering temperatures on the volume fractions of retained austenite.

2. Determination of Retained Austenite Content

X-ray diffraction is one of the primary techniques used for the determination of retained austenite in steel. It is based on observing the scattered intensity of an X-ray beam hitting a sample as a function of incident and scattered angle, polarization, and wavelength or energy. One of the most commonly used method to calculate volume fraction of retained austenite is the direct comparison method based on a direct comparison of integrated intensities from each phase. In this method, the austenite fraction is determined from the ratio of the austenite and ferrite diffraction peak intensities and the values of R for each phase [16].

The following expressions needed for the determination of RA phase in steel are given in the American Society for Testing Materials (ASTM) based on direct comparison method. It is a standard practice for X-Ray determination of RA in steel with near random crystallographic orientation. In this practice, if possible, X-ray diffraction peak interference from other crystalline phases such as carbides should be eliminated from the ferrite and austenite peak intensities.

It has been stated that [17], "for a randomly oriented sample, quantitative measurements of the relative volume fraction of ferrite and austenite can be made from X-ray diffraction patterns because the total integrated intensity of all diffraction peaks for each phase is proportional to the volume fraction of that phase. If the crystalline phase or grains of each phase are randomly oriented, the integrated intensity from any single diffraction peak (hkl) crystalline plane is also proportional to the volume fraction of that phase". Based on the ASTM E975-03, for steel containing ferrite (α) and austenite (γ), the ratio of integrated intensity is given by Eq. 1.

$$\frac{I_\alpha^{hkl}}{I_\gamma^{hkl}} = \left(\frac{R_\alpha^{hkl}}{R_\gamma^{hkl}}\right)\left(\frac{V_\alpha}{V_\gamma}\right) \quad (1)$$

Eq.1 holds, if ferrite (or martensite) and austenite are the only two phases present ($V\alpha+V\gamma=1$) in a steel and both phases are randomly oriented.

The volume fraction of austenite ($V\gamma$) for the ratio of measured integrated intensities of ferrite and austenite peak to R-value can be found using Eq. 2.

$$V_\gamma = \left[{}^{I_\gamma}\!/_{R_\gamma} \Big/ \left({}^{I_\alpha}\!/_{R_\alpha}\right) + \left({}^{I_\gamma}\!/_{R_\gamma}\right) \right] \quad (2)$$

For numerous ferrite and austenite peaks, each ratio of measured integrated intensity to R-value can be summed up in Eq. 3.

$$V_\gamma = \left[\left(\frac{1}{q}\Sigma_{j=1}^{q}\frac{I_{\gamma j}}{R_{\gamma j}}\right) \Big/ \left(\frac{1}{p}\Sigma_{i=1}^{p}\frac{I_{\alpha i}}{R_{\alpha i}}\right) + \left(\frac{1}{q}\sum_{j=1}^{q}\frac{I_{\gamma j}}{R_{\gamma j}}\right) \right] \quad (3)$$

$R\alpha$ and $R\gamma$ are the theoretical intensities and they can be calculated by using Eq. 4.

$$R^{hkl} = \frac{1(|F|^2 pLPe^{-2M})}{v^2} \quad (4)$$

Where;

$|F|^2$: structure factor times its complex conjugate,

p : multiplicity factor of the (hkl) reflection,

LP : Lorentz Polarization factor and equals to [(1 + cos22θ)/sin2θ cosθ] where θ is Bragg angle,

e-2M : Debye-Waller or temperature factor which is a function of θ,

v : volume of the unit cell.

3. Material and Method

3.1. Material

The commercial 1.2842 (AISI O2) cold work tool steel was used in this work Cold work tool steels contain high amount of carbon steels and relatively low amonut of alloys. The chemical composition of the steel used in this study is given in Table 1.

This group of steels is relatively inexpensive, and they are widely used for blanking and forming dies that require high surface hardness and minimal distortion during hardening.

3.2. Heat Treatment Process

Hardening and tempering processes have been carried

Table 1. Chemical composition of 1.2842 steel used in the experiments

C %	Si %	Mn %	Cr %	Mo %	Ni %	V %	W %	P %
0.90	0.25	2.00	0.40	-	-	0.10	-	0.03

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Table 2. The selected heat treatment conditions for 1,2842 Steel.

	Temperature (°C)	Time (h)	Medium
Stress Relieving	400	2	Furnace
Hardening	820	0.5	Oil
	150		
	200		
Tempering	250	1.5	Still air
	300		
	400		

out, under the conditions listed in Table 2, according to the technical specification suggested for the heat treatment of 1.2842 steel [18].

Stress relief annealing heat treatment (at 400°C for 2h) is applied to all samples before the quenching process. Then, samples were austenitized at 820°C for 30 minutes and quenched in oil. The samples were stress relieved again before the tempering process at a temperature of 150°C to avoid any crack and distortion. Then, the samples were tempered at different tempering temperatures for 1.5 hours in an electrical muffle furnace and allowed to cool in still air.

3.3. X-ray Diffraction Phase Analyses

XRD phase analyses were performed by using Rigaku Miniflex 2 Benchtop diffractometer (shown in Figure 1) with 40 kV tube voltage, 15 mA tube current.

Six specimens tempered at different temperatures and a specimen as-quenched condition were used for X-ray diffraction phase analyses. The dimensions of each specimen,

Figure 1. Rigaku Miniflex 2 Benchtop diffractometer

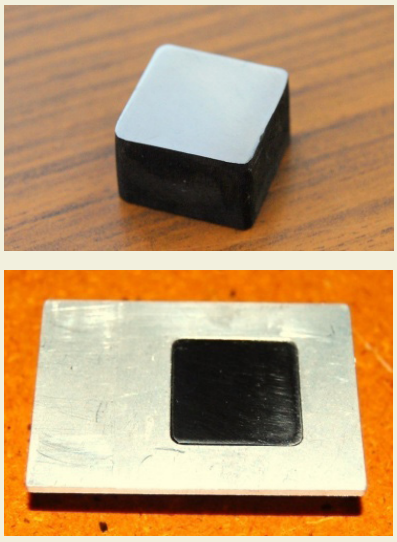

Figure 2. The picture of test specimen and the holder used for XRD phase analyses.

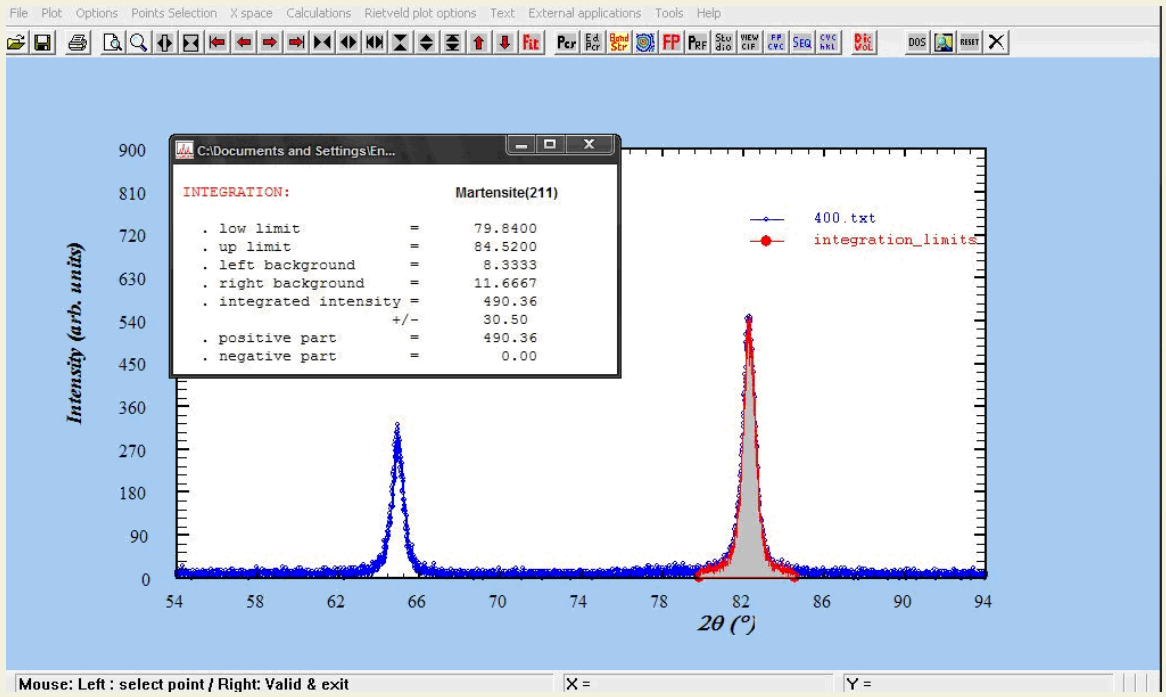

Figure 3. An example of XRD pattern of the tempered sample obtained from WINPLOTRTM software and integrated intensities of each peak.

shown in Figure 2, were 20x20x12 mm. The impruties on face of samples were removed by applying grinding and polishing. Then, each speciemen was located into the holder and subjected to x-ray to obtain XRD patterns for the specimens.

The phases represented by each peak were defined according to the Joint Committee on Powder Diffraction Standards (JCPDS) peak lists. In this study the (200) α, (211) α, (220) γ and (311) γ peaks and their integrated intensities (Iα200, Iα211, Iγ220, and Iγ311) were compared with JCPDS peaks for the definition of the phases obtained in each sample after tempering. WINPLOTRTM software was used to determine the integrated intensities from the XRD patterns by calculating the area under the X-ray diffraction peaks as shown in Figure 3.

The volume fraction of retained austenite (Vγ) was estimated by substituting calculated R values into Eq. 5, which is derived from Eq. 3 [19].

$$V_\gamma = \frac{\left(I_\gamma^{220}/\left(1.42I_\alpha^{200}+I_\gamma^{220}\right)\right)+\left(I_\gamma^{220}/\left(0.71I_\alpha^{211}+I_\gamma^{220}\right)+\left(I_\gamma^{311}/\left(1.62I_\alpha^{200}+I_\gamma^{311}\right)+\left(I_\gamma^{311}/\left(0.81I_\alpha^{211}+I_\gamma^{311}\right)\right)\right)\right)}{4} \quad (5)$$

Finally, the determined volume percentage of retained austenite were plotted as a function of tempering temperatures and results were interpreted in the following section.

4. Result and Discussion

During quenching process, all of the austinite obtained at the austenizing temperature is not transformed into martensite. Depending upon the final temperature reached during cooling stage, some of the austenine will not transform into martensite. Then the final structure consists of austenite transformed into martensite with some amount of untransformed austenite (RA). Mechanical properties of hardened tool steels can be altered during tempering process by controoling the amaunt of RA. In this work, the volume fraction of RA was calculated by comparing the integrated intensities in order to study the effect tempering temperatures on the volume fraction of RA. The results obtained from the calculations are given in Table 3. The same method was also used by Li et al [20]. They have estimated RA content by using the direct comparison techniqueu and they have investigated the effect of RA content on mechanical properties such as hardness and toughness of cold work tool steel. They reported that XRD peaks obtained in the XRD analyses were changed depending on the volume of retained austenite obtained at different tempering temperatures.

Patterns obtained from XRD analyses for as-quenched and tempered samples are presented in Figure 4..

As seen in Figure 4 (a), as-quenched samples were contained the highest volume of RA. Because compared to the XRD patterns of tempered specimens, (220)γ and (311)γ peaks at the 2θ values of about 75 and 90 degrees were hihgher that the (200)α and (211)α peaks obtained in the as-quenched samples.

As shown in Figure 4 (b), (220)γ and (311)γ peaks are lower in compared to (200)α and (211)α peaks and (200) α and (211)α peaks are higher in tempered samples compared to the as-quenched samples. This inidicates that the amount of untransformed austenite content decreases in the saples tempered at the temperature of 150°C.

Similarly, increasing the tempering temperature from 150 to 200°C (Figure 4 c) resulted higher amount of RA. Above the tempering temperature of 200°C, significan decrease was observed at the (220)γ and (311)γ peaks and austenite peaks were not seen very clearly. Almost non-existent austenite peaks are observed in Figure 4 (d), (e) and (f). The reason for extinction after tempering temperature of 200°C is due to the dcrease in the amount of RA

Volume fractions of RA estimated from the X-ray diffraction analyses based on the comparison method are summarized in Figure 5.

As shown in Figure 5, the highest amount of retained austenite (about 11.80%) was obtained after the quenching process. The samples tempered at about 150°C caused a reduction in the amount of RA (about 8.45%). It could be said that the formation of very fine particles of epsilon

Table 3. Integrated intensities and the volume fractions of RA obtained at different heat treating conditions

Tempering at (°C)	I_α^{200}	I_α^{211}	I_α^{220}	I_α^{311}	V_α	RA, % Vol.
As quenched	137.71	213.6	22.24	26.88	0.118	11.80
150	217.54	406.71	29.72	28.98	0.08448	8.45
200	108.16	203.88	18.24	17.25	0.10056	10.06
250	178.31	345.46	6.94	12.06	0.0339	3.39
300	193.04	359.4	6.13	5.59	0.02043	2.04
400	257.02	490.36	3.61	3.76	0.0096	0.96

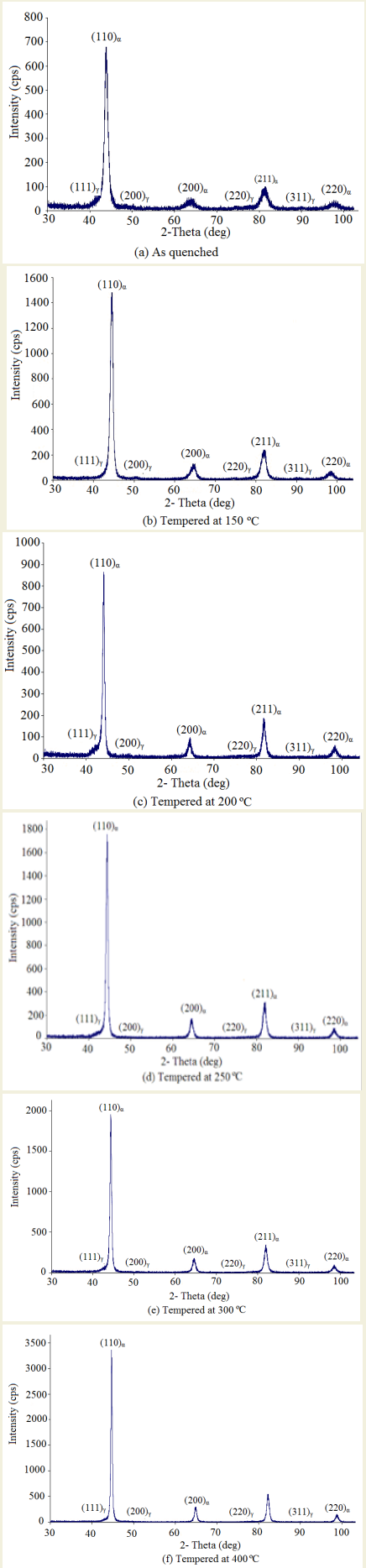

Figure 4. XRD Patterns of as-quenched specimen and specimens tempered at different temperatures

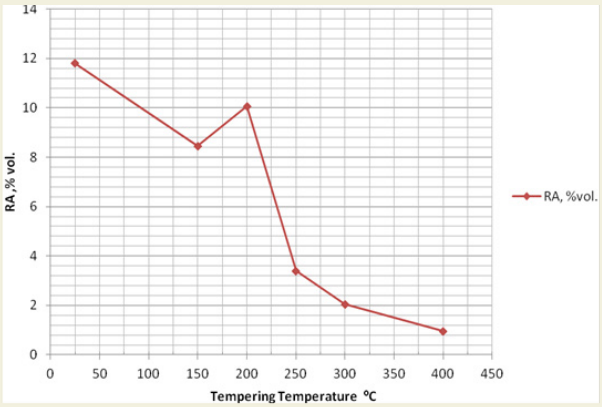

Figure 5. The effect of tempering temperatures on the volume fraction of RA

(hexagonal) carbide as a result of supersaturated carbon precipitation out of the martensite caused the reducion in the volume of RA content up to the tempering temperature of 150°C. Increasing tempereing temperature from 150°C to 200°C resulted a slight increase (about 10.06%) in the amount of RA content. In this tempering temperature range, a slight increase of RA volume was observed due to the decomposition of carbide which resulted the stabilization of austenite. A sharp reduction of RA is observed between the tempering temperatures of 200 °C and 250°C. Amonut of RA continued to decrease in the samples tempered above the temperature of 250 °C.

RA volume dropped suddenly above the tempering temperature of 200°C due to the thermal destabilization of this phase [21]. As stated by Dong et al. [9], Kokosza and Pacyna [22] and Talebi et al [23], after the tempering temperature of 200°C, untransforfed austenite within the structure could be decomposed by a diffusion mechanism and causes precipitation of more carbides. This causes reduction in the austenite stability and results the transformation of austenite to a hard martensitic structure during the cooling process. This could be the reason for the sharp reduction in the amount of RA and carbon concentration of retained austenite. The reduction in the amount of RA was continued when the tempering temperature was increased and nearly no RA was seen at about tempering temperature of 400 °C.

5. Conclusion

Characterization of the RA in quenched and tempered 1.2842 tool steel was evaluated by using XRD phase analyses. The samples were quenched at 820 °C and tempered for 1.5 h at 150 °C, 200 °C, 250 °C, 300 °C and 400 °C. The the amount of RA was calculated for each sample and the results were presented. Following conclusions were drawn from the XRD analses carried out for the quenched and tempered 1.2842 steel.

About 11.8 % of RA was observed in the as-quenched 1.2842 steel. Increasing tempering temperature to 150 °C

resulted a reduction in the amount of RA to 8.45 % level. Amount of RA (10.06 %) started to increase again when the tempering temperature was increased to 200 °C. Above the tempering temperature of 200 °C, the thermal destabilization of austenite resulted a gradual reduction in the amount of RA. Very small amount of RA (0.96 %.) was observed athe samples tempered at about 400 °C.

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