



Thermal and mechanical stability of retained austenite in high carbon steel: An *in-situ* investigation



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ABSTRACT

High carbon multiphase steels have attracted a lot of interest in industrial applications due to their high resistance against abrasion. The structure of these steels comprises of mixed martensitic structure, plates and laths, and retained austenite. The presence of retained austenite phase plays a very important role in properties of these steels. In this paper, two *in-situ* approaches have been used to study thermal and mechanical stability of retained austenite. By *in-situ* x-ray diffraction, the start and end of phase transformation temperatures of retained austenite were identified. For the sample with higher retained austenite percentage, phase transformation started at lower temperature because by increasing the percentage, required barrier energy for phase transformation will be reduced. Also, different stages of phase transformation were investigated using a nano-indentation method. Phase transformation from austenite to martensite was identified in the mechanical stress range of 60–70 mN.

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1. Introduction

By increasing the carbon content in martensitic steel, the percentage of retained austenite will increase and the hardness will decrease [1]. But by controlling the heat treatment condition, multiphase steel with superior properties can be produced. These high carbon multiphase steels have attracted a growing interest due to their high abrasion resistance. This excellent property mainly arises from martensitic structure which contains metastable retained austenite. Martensite phase is a very hard phase due to straining and distortion of the lattice caused by entrapping of carbon atoms and due to mixed structure of martensitic plates and laths in high carbon steels [2]. These plates and laths act as barriers to dislocation movement which increases the hardness. Also, metastable retained austenite will be transformed to martensitic structure due to application of stress [1–3]. This dual structure and martensitic transformation of metastable retained austenite under the influence of external force makes these steels suitable for high abrasion resistance applications.

Retained austenite phase is a metastable phase at room temperature and would transform to a martensite phase when

sufficient activation energy is given which can be achieved via applying stress or heating the structure. Retained austenite phase can affect the properties of martensitic steels in many ways [4–6]. The work hardening of the material increases, when the material is subjected to high stresses and high temperatures which also increase the toughness [6–8]. When this material is subjected to abrasion, retained austenite on the surface will transform to martensitic structure and hardness will increase. This phase transformation and increase in the hardness will improve the wear resistance of steel [9–10]. This kind of property is desirable in applications where the surface of the material wears due to application of stress but the new surface remains hard.

Due to various industrial applications of high carbon martensitic steels with retained austenite phase present in the microstructure, it becomes an important need to determine the percentage of retained austenite and study its thermal and mechanical stability [11–12]. When these martensitic steels are heated to temperatures more than martensitic transformation temperature and below the austenitic transformation temperature, in the range of 100–500 °C, the unstable retained austenite phase transforms to a stable martensite phase, and the percentage of retained austenite starts to decrease. If the sample stays at this temperature long enough, metastable retained austenite phase will completely transform to martensitic phase [3,8]. It is very important to characterize the stability of retained austenite with both the temperature and mechanical stress in these high

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carbon steels [13–16].

In this work, we used two *in-situ* techniques, *in-situ* X-ray diffraction analysis and nano-indentation, to study the stability of retained austenite. The combination of these two techniques determines the percentage of retained austenite and identifies different stages for complete transformation of retained austenite to martensite.

2. Experimental procedure

Steel samples with the chemical composition as listed in Table 1 were hot rolled and heat treated with different conditions to achieve different retained austenite percentage. Samples A and B with 28.8 and 54.8 percentage of retained austenite have been used for this study. For microstructure investigation, these samples were prepared under standard sample preparation technique and were etched using a 2% Nital solution. Small samples with 14 mm diameter and 3 mm thickness were prepared using diamond cutter (Struers Minimot) for *in-situ* X-ray diffraction measurement and nano-indentation.

In-situ X-ray diffraction was conducted with PANalytical Empyrean XRD instrument equipped with high temperature vacuum furnace and scanning rate (2θ) of $0.4^\circ \text{ min}^{-1}$ over range $2\theta=42.5\text{--}46^\circ$, with unfiltered Cu K_α radiation. The system was operated at 45 kV and 45 mA. The diffraction of X-ray was measured at different temperature from 25 °C to 490 °C with 15 °C intervals and heating rate of $2^\circ \text{ C min}^{-1}$. The retained austenite content at each temperature was calculated using integrated intensities of the 111 austenite peak and 101 peak of martensite and Eq. 1. I_γ , I_α are integrated intensities of austenite and martensite peaks and R is calculated from Eq. 2, which $|F|^2$ is structure factor times its complex conjugate, p and LP , multiplicity and Lorentz factor respectively, e^{-2M} Debye–Waller factor and ν volume of unit cell [17]. After X-ray diffraction characterization, the samples were polished and microstructure of steel, transformed austenite, was investigated using Scanning Electron Microscope (SEM, Hitachi 3400-X) and optical microscope.

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

$$R_\alpha^{hkl} = \frac{1(|F|^2 p L P e^{-2M})}{\nu^2} \quad (2)$$

For nano-indentation, samples were hot mounted using (Struers ProntoPress-20) and polished by using (Struers Tegramin-20). Nano-indentation test was conducted with a 6×6 array and load incremented in steps of 10 mN from 10–360 mN. The indenter used in this test was Vickers diamond pyramid indenter. The Vickers diamond hardness is calculated from Eq. 3, in which P is load applied by the indenter and d is diameter of the tip of the indenter. Eq. 4 can be used to convert GPa hardness to Vickers hardness (HV). After the nano-indentation test, transformed austenite was investigated using Scanning Electron Microscope (SEM, Hitachi 3400-X).

$$\text{VDH} = \frac{2P}{d^2} \sin \frac{136^\circ}{2} = 1.8544 \frac{P}{d^2} \quad (3)$$

Table 1
Chemical composition of steel samples.

Elements	C	Si	Mn	Cr	Cu	Ni	Mo
Percentage	0.83	0.27	0.9	1.0	0.16	0.07	0.02

$$\text{HV} = \frac{\text{GPa}}{9.807} \times 1000 \quad (4)$$

3. Results and discussions

In-situ X-ray analysis was carried out to study the thermal stability of retained austenite with increasing temperature and to study the phase transformation of retained austenite to martensite as temperature increases. Table 2 shows the percentage of retained austenite at room temperature. Fig. 1 shows the percentage of retained austenite versus temperature for both samples A and B as well as the XRD spectrum for each sample at different temperatures. Austenite is a meta-stable phase at room temperature so it transforms to martensite. But this transformation has a barrier energy which can be achieved by the application of stress or temperature. Initially, at lower temperatures, energy is not sufficient for the phase transformation of retained austenite phase for both the samples A and B. For sample A in the temperature range between room temperature, 25 °C, and 160 °C, the change in retained austenite percentage is 2% which is almost negligible. Also, for sample B the change is only 0.72% between room temperature to 100 °C.

In Fig. 1, rate of decrease in percentage retained austenite of sample B is higher than that of sample A to the temperature around 150 °C and both samples attained the same percentage at 300 °C. For sample A, as the temperature increased from 160 °C the percentage of retained austenite started to decrease, which implies that the phase transformation of retained austenite started at temperatures around 175 °C. From the X-ray spectrum, the retained austenite percentage at this temperature was 25.4%. After reaching temperatures around 300 °C, the percentage of retained austenite decreased to below 5% and the phase transformation rate decreased (Fig. 1).

For sample B, as shown in Fig. 1, retained austenite phase transformation starts at 100 °C. Due to higher retained austenite percentage in sample B at room temperature compared to sample A, the phase transformation of retained austenite to martensite takes place at lower temperatures. But in both samples A and B the phase transformation rate decreases around 300 °C and the complete transformation of retained austenite to martensite phase takes place at nearly the same temperatures (400 °C). In both the cases we see that retained austenite gradually transforms to martensite (Fig. 1).

By increasing the temperature the austenite peak starts to shift to the lower 2θ value due to the lattice expansion effect caused by transformation of retained austenite to martensite phase (Fig. 1) which shows the clear phase transformation. Also by increasing the temperature, the shape of the martensite peak changes and becomes sharper which represents the decrease in crystalline size of martensite. This is in good agreement with tempering of martensite and breakdown in the size of martensitic plates.

The microstructures of the martensitic steel samples before and after *in-situ* XRD were studied using a SEM. The difference in the microstructure before and after the *in-situ* X-ray analysis was used to determine the change in the percentage of retained austenite, which is shown in Fig. 2 for samples A and B. Comparison of Fig. 2a

Table 2
Retained austenite percentage of steel samples at room temperature.

Samples	Retained austenite
A	28.80
B	54.8

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