# TAILORING OF MECHANICAL PROPERTIES OF AISI 410 MARTENSITIC STAINLESS STEEL THROUGH TEMPERING

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## Abstract

Martensitic stainless steels are broadly used in industrial applications where demands for higher strength, hardness and wear resistance are high. Tempering treatment plays a crucial role on performance of such steels. The present work is focusing on the effect of tempering on carbide precipitations and properties of AISI 410 SS. Samples were austenitized at 980 °C for 30 min and tempered over the range of 300 - 732 °C. During tempering the carbides precipitated in the form of $M_{23}C_6$ and $Fe_2C$ in varying amount. During tempering at 300 °C, the ultimate tensile strength (UTS) increased to 1100 MPa from 741 MPa (as-received). Tempering in the range of 500 - 550 °C yields segregation of carbides at the grain boundaries and overall matrix. UTS was found to be reduced gradually from 944 MPa to 641 MPa when tempering temperature increased from 550 to 732 °C attributed to the coarsening and spheroidization of carbides. The optimized tempering condition was found out to be 550 °C for enhanced strength-ductility ratio, i.e. UTS 944 MPa with fracture strain of about 11 % and hardening effect (515 Hv).

**Keywords**: Martensite, tempering, segregation, hardening

## 1.    INTRODUCTION

AISI 410 martensitic stainless steels are prominent materials for industrial applications in steam and marine turbine blades, steam generators, pressure vessels, cutting tools, pumps, valves and diesel engines [1-2]. Heat treatment can be adopted in this type of steels to acquire good mechanical properties and corrosion resistance. It can also be used in low or high operating temperature conditions. They are cheaper and cost effective as compared to duplex stainless steels. The mechanism of hardening of steel requires austenitizing and quenching to room temperature [2]. In austenitizing, the material is heated above the upper critical temperature to form full austenite phase and in quenching, the austenized material is subjected to rapid cooling to room temperature. This results in phase transformation from austenite to martensite metastable phase. The martensite obtained after hardening is extremely brittle with high internal stresses which can result in heavy distortion and cracking during service. To improve the properties of the hardened steel, normally tempering is carried out. Tempering can be achieved by heating a hardened steel to a temperature below the lower critical temperature followed by cooling in air to room temperature. Depending upon required mechanical properties the tempering temperature can be varied from 100 to 750 °C [3]. The tempering time for steels are generally 1 to 2 hours and can be increased up to 4 hours for bigger sections. During tempering process, structural and thermal stresses are the two common internal stresses formed. The structural stresses resulted from increase in volume of the specimen due to transformation of austenite to martensite and whereas thermal stresses are because of rapid decrease in the temperature of the samples. The control of austenitizing temperature and cooling rates are very vital to avoid the internal stresses which in turn can cause severe distortion and cracking. In this tempering process there are two different types of carbide precipitates occur out of parent martensite phase, one is iron rich ($Fe_2C$) and other is chromium rich carbides ($Cr_{23}C_6$). During tempering the unstable phases obtained after hardening changes to stable phases such as tempered martensite and ferrite carbide mixture. The type and mechanism of precipitation is very vital for research as this is an operating temperature

range often used in industrial applications. It has been reported that the plain 13 Cr steel shows slight secondary hardening at about 500 °C due to heterogeneously distributed $Cr_{23}C_6$ carbides [1]. Although there are several reports on tempering behaviour of such steels, yet there is no clear understanding about microstructural variation and its correlation with properties, particularly on 410 SS steels, during tempering. There has been a drastic variation from one report to another. One of the major issues is with very less amount of fracture strain (ductility). This is still an active area of research and present investigation attempts to address some of these issues. Objectives of the present investigation is to study the microstructural evolution during tempering process on 410 SS. Attempts have been made to obtain optimized condition to enhance the fracture strain while having relatively higher tensile strength.

## 2.  EXPERIMENTAL PROCEDURE

### 2.1.  Material and heat treatment process
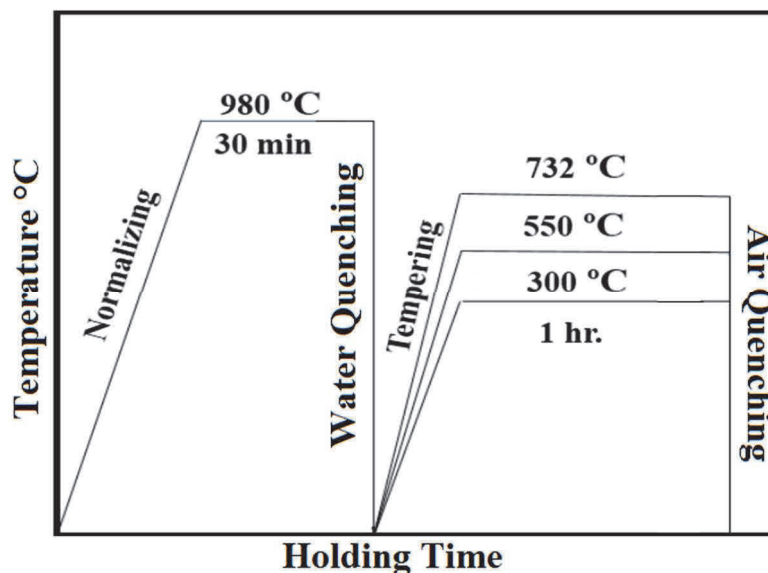


**Figure 1** Schematic diagram of conventional tempering heat treatment

AISI 410 martensitic stainless steels specimen with the dimensions of 250 × 250 × 12 mm$^3$ was received from IGCAR Kalpakkam, India. As received material had a history of normalized (980 °C) and tempered (730 °C) condition. Chemical analysis was carried by ICP OES with test method IS-228. The chemical composition of AISI 410 SS: 0.098 wt. % C, 12.12 wt. % Cr, 0.97 wt. % Mn, 0.41 wt. % Si-0.011 wt. % P 0.01 wt. % S and balanced Fe. The samples were heated to austenitization temperature at the rate of 10 °C/min in a muffle furnace at 980 °C for a soaking period of 30 min to obtain homogenous austenite microstructure. Then, the samples were quenched in water to ensure fully martensite microstructure. After quenching samples were specifically tempered separately at 300 °C, 550 °C and 732 °C for 1 hr. followed by air quenching - see **Figure 1**.

### 2.2.  Characterization

The metallographic samples were prepared carefully by polishing techniques and then etched with the reagent mixture of 100 ml ethanol and 5 g FeCl3 (tempered martensite). The microstructures were investigated by an optical microscope. The base martensite phases were analyzed by using X-ray diffraction (PANalytical, model: X'pert PRO) with Cu kα radiation. The selected specimen was investigated for carbide precipitates in more detailed manner by using transmission electron microscopy (TEM 2100 JEOL with LaB6 filament) equipped with energy dispersive X-ray spectroscopy (EDS). The thin foils for TEM were prepared by twin-jet

electropolishing in a solution of 10% perchloric acid and 90% methanol operating at 25 - 28 V for about 8 seconds at room temperature. TEM specimens were observed using an acceleration voltage of 200 kV. Microhardness measurement was carried out using Vickers hardness tester (Durascan) under a load of 200 g and dwelling time of 5 s. The result includes after averaging over hardness values (Hv) taken at ten different locations on each samples. The tensile specimen was prepared according to ASTM standard E8 with dimension of 100 mm overall length, 32 mm gage length, 6 mm width and 4 mm thickness, were machined and then tempered in different temperatures. The strength and fracture strain were evaluated through tensile testing by using computer-controlled servo hydraulic material testing system (MTS model no 370.10) with capacity of 100 KN. All specimens were pulled to fracture at a nominal strain rate of $5 \times 10^{-4}$ s$^{-1}$ under ambient conditions. The load-displacement curves were recorded to determine various tensile properties such as 0.2% yield strength (YS), ultimate tensile strength (UTS) and fracture strain [4].

## 3. RESULTS AND DISCUSSION

The XRD analyses of tempered samples at different temperatures were shown in **Figure 2**. Three major martensite peaks α' {101}, {200} and {211} were observed (**Figure 2**). The martensite α' {101} peak is shifted to lower values of 2θ (**Figure 2**) for all tempering temperatures with respect to as quenched samples.
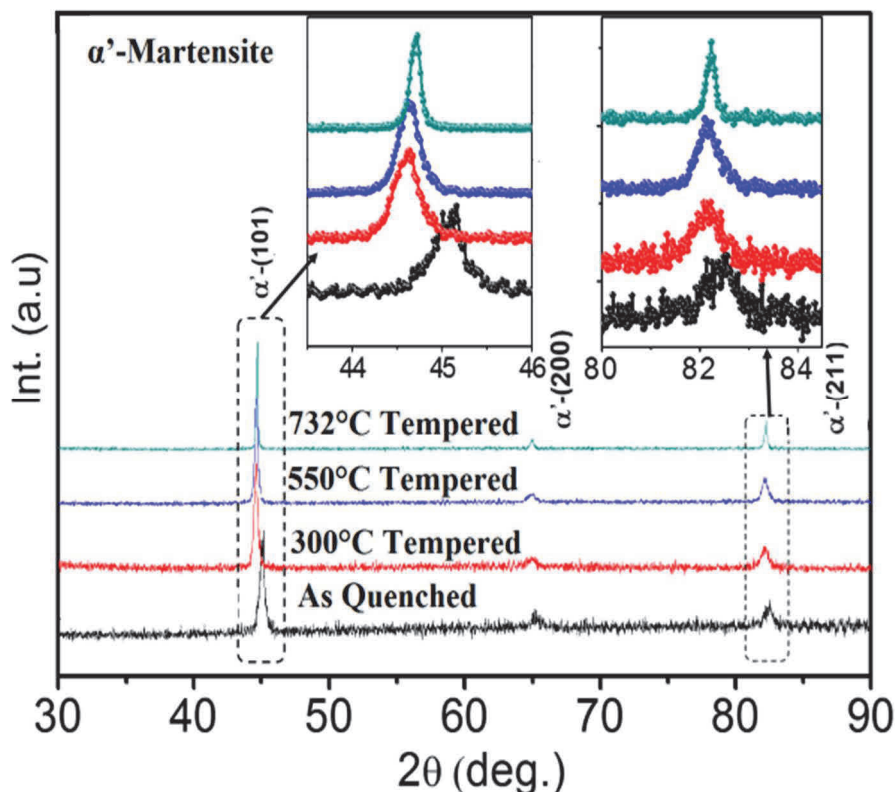


**Figure 2** XRD patterns for 410 SS for different tempered conditions

For tempering conditions of 300 and 550 °C have lower angle peak shift with respect to as quenched samples and which can be considered that the lattice parameter was increased due to inadequate carbon diffusing out of martensite matrix at lower tempering temperatures [5]. Peak shift to higher value of 2θ with respect to lower tempering temperatures (300 and 550 °C) suggests lower lattice parameter indicating stress relaxation with compressive residual stress of the martensite matrix [6]. Sharpening of the peaks was also reported and as there was no lattice strain during high tempering temperature (732 °C).
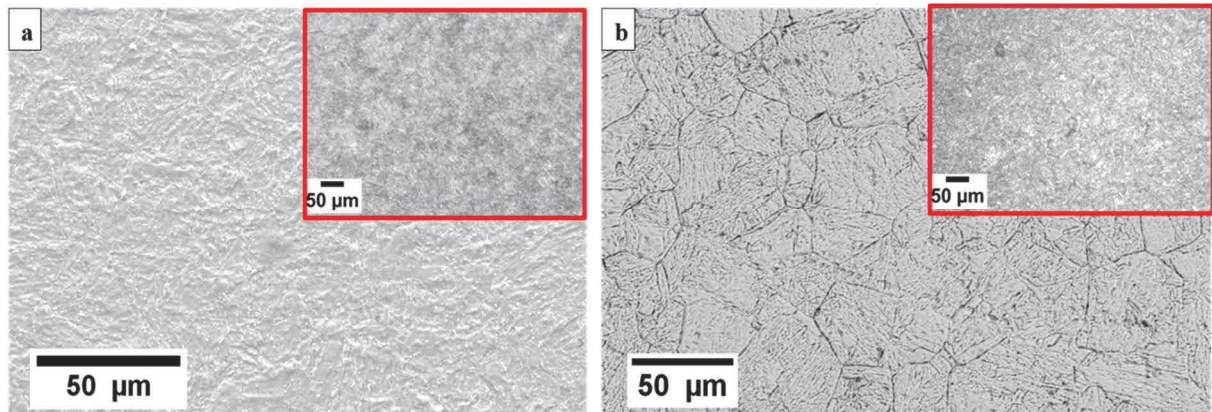
**Figure 3** SEM pictures, in BSE imaging modes and optical micrograph, of a) As Received b) As Quenched

FESEM and Optical microscope exposed the microstructure of AISI 410 martensitic stainless steel in **Figure 3** for as received and as quenched conditions. **Figure 3b** shows as quenched condition in which more needle like structure with predominant laths and grain boundaries of martensite phase were visible as compared to as received samples (**Figure 3a**). **Figures 4** shows bright field TEM micrographs of the microstructure of the samples after conventional tempering at 300, 550 and 732 °C respectively. The two different types of precipitate morphology were observed i.e. globular marked as A and rod like structure marked as B as shown in **Figure 4b**. Globular precipitate were identified as $M_{23}C_6$, in which M is indicated as strong carbide-forming element as Chromium (Cr) and $Fe_2C$ precipitate as rod like structure which was identified by energy dispersive X-ray spectroscopy (EDS) shown in **Figure 4b**.
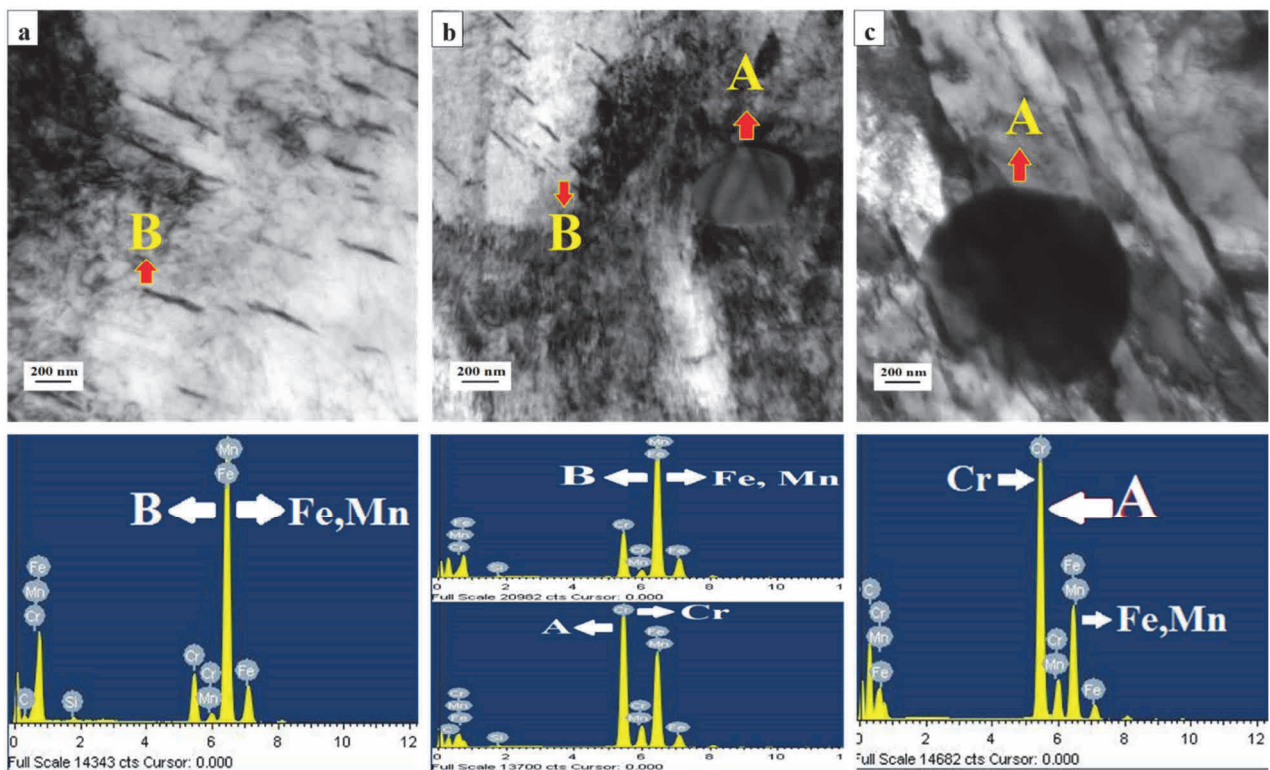


**Figure 4** Bright-field TEM of tempered AISI 410 martensitic stainless steel a) 300 °C showing marked B-ferrite carbides (rod like structure) with EDX, b) 550 °C showing both Marked A-coarse $Cr_{23}C_6$ (globular structure) and B-ferrite carbides with EDX, c) 732 °C showing Marked A-coarse $Cr_{23}C_6$ with EDX spectra

The martensite nature changes when tempering takes place. The process of tempering at 300 °C relieves internal stresses due to structural and thermal stresses. For tempering treatment at 300 °C only small amount of iron rich carbides could be identified as shown in **Figure 4a**. Iron rich carbides such as $Fe_2C$ predominately co-exist at this low tempering temperature [7]. But whereas for further tempering at 550 °C segregation of both iron rich carbides ($Fe_2C$) and chromium rich carbides ($Cr_{23}C_6$) coexists in the material (**Figure 4b**) with more dislocation density. The EDS shown in **Figure 4b** proves about the types of carbide present in 550 °C tempered condition. Both $Cr_{23}C_6$ and $Fe_2C$ types of carbides precipitate together in the range of 500 - 600 °C tempering temperatures [8]. In this tempering range the hardening takes place due to uniform dispersion of carbide particles with in martensite lath structure that hinder dislocation motion, thereby strengthening [9]. The bright field images of TEM shows high dislocation densities with the martensitic lathes and needle like structure all over the matrix. Domination of chromium rich carbides takes place at higher tempering temperature of 732 °C. In **Figure 4b** and **4c**, the size of the globular precipitate ($Cr_{23}C_6$) marked as A increased from 500 nm to 1000 nm diameter. The coarsening of chromium carbides takes place due to ease of diffusion of carbon at higher tempering temperatures from martensite matrix (**Figure 4c**). Also, it has been found by TEM in *Figure 4c* that the $Cr_{23}C_6$ carbides precipitating preferentially at the martensite lath boundaries but whereas iron rich carbides are all over the martensite matrix shown in **Figure 4a**. The precipitation preferably co-exists along the martensitic lath boundaries due to the greater ease of diffusion in these regions which was caused by tempering treatment. The release of strain, which causes high density of dislocation all over the martensite matrix, can be seen in TEM bright field image (**Figure 4c**).

The graphical representation in **Figure 5a** shows hardness variation as function of tempering temperature. The hardness of as received samples was obtained as 223 Hv. In as quenched it shows higher hardness value of 512 Hv. Hardness of the steel usually decreases during tempering. But the hardness was almost retained till 550 °C (**Figure 5a**) which could be attributed to the increased amount of carbides as shown in **Figure 4b**. At higher tempering temperature of 732 °C the hardness dropped drastically, attributed to the softening of matrix due to out diffusion of carbon and coarsening of carbides.
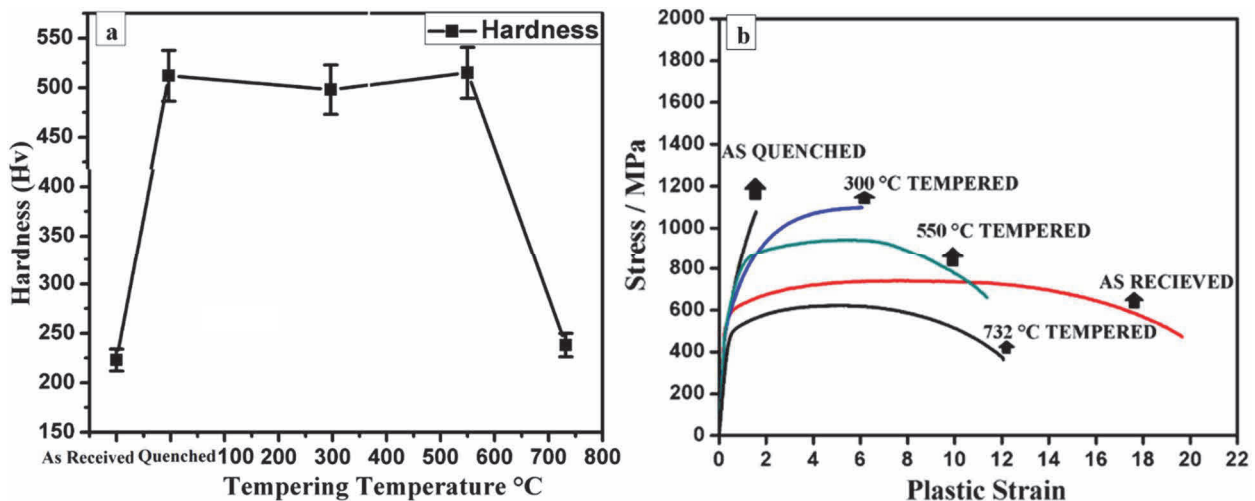


**Figure 5** a) Microhardness profiles variation as a function of tempering temperatures, b) Nominal stress-strain curves of different tempered conditions

The tensile test results have been shown in **Figure 5b**. As received samples shows yield strength of 583 MPa and UTS (Ultimate tensile strength) of 741 MPa and 20.1 % strain. For as quenched condition the strength was increased to UTS of 1079 MPa. During tempering at 300 °C, showcased higher UTS of 1108 MPa but material almost lost its ductility with diminished strain of 6.51 % as compared to 550 °C tempered with insignificant change in UTS of 944 MPa with higher strain of 12.03 %. The tensile strength fracture was retained

without losing the strain (12.03 %) for 550 °C which could be associated to the finer and higher amount of precipitates (**Figure 4b**). The finer precipitates always help in strengthening mechanism. Due to softening of the matrix at higher tempering temperature of 732 °C showcased lower UTS of 641 MPa with higher ductility. This softening was caused by coarsening of carbides and carbon diffusing out due to high temperature.

## 4. CONCLUSION

Air hardenable AISI 410 martensitic stainless steels have lath structure in as quenched condition. The existence of both type of carbide precipitates found to be stable at 550 °C tempering treatment which gave significant strength to ductility ratio. The TEM bright field images confirms precipitation of carbides in the form of ferrite carbide ($Fe_2C$) and chromium carbide ($Cr_{23}C_6$) is attained together at intermediate tempering temperature of 550 °C. It showed superior tensile strength of 944 MPa with 12 % fracture strain (ductility) and supreme hardness of 515 Hv. The AISI 410 SS exhibits the best comprehensive mechanical properties when samples were tempered at intermediate temperature of 550 °C.

## ACKNOWLEDGEMENTS

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