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Effect of tempering on microstructure and tensile properties of niobium modified martensitic 9Cr heat resistant steel

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ABSTRACT

The effect of tempering on the microstructure of niobium modified 8.8 wt% chromium steel has been evaluated. Steel has been prepared using the conventional melting and casting route. Homogenization and forging is done at 1100 °C. Dilatometric study shows that the Ac_1, Ac_3 and M_s temperatures are 800, 855, and 131 °C, respectively. Initial cast and forged microstructures consist of martensite/ferrite. The samples are subsequently tempered at 500–800 °C for various intervals of time (1–5 h). The microstructure of the tempered sample is analyzed using optical microscopy, scanning electron microscopy, and X-ray diffraction. High Resolution Transmission Electron Microscopy (HRTEM) is used to identify the precipitate. Nanometer-sized precipitates (50–200 nm) are observed after tempering at 700 °C for 1 h. Niobium rich MC type carbide precipitates and chromium rich $M_{23}C_6$ type precipitates are observed after tempering at 700 °C. Tensile strength decreases with increasing tempering temperature. Maximum tensile strength of 920 MPa is observed after tempering at 700 °C and maximum elongation of ~11% is observed after tempering at 750 °C.

1. Introduction

9% Cr ferritic/martensitic steels are widely used as heat resistant steel because of high thermal conductivity, low coefficient of thermal expansion, good corrosion and oxidation resistance [1–4]. Chromium is a strong ferrite stabilizer. There is an extensive single phase austenite region in Fe–9Cr–C system in the temperature range of 800–1200 °C. Therefore, austenitization of the steel is easier and upon cooling to room temperature the steel fully converts to martensitic structure. A lot of work has been done to improve the creep strength of chromium steel by different alloying additions. Research has been conducted to study the effect of Cu, Al, Mn, Ni, W, and Co in 9% chromium steel [5–8]. Precipitation hardening is one of the most effective ways to strengthen 9% Cr steel. In this method, an alloy is produced by solution treating and quenching to produce a supersaturated solid solution and a second phase precipitates out during subsequent heat treatment [9]. The second phase of MX type carbides, nitrides and carbonitrides provides strengthening of steel by forming very fine and densely distributed precipitates. It is reported that different types of carbides such as MC, M_2C, M_3C, M_7C_3, $M_{23}C_6$ and M_6C precipitates in Cr–Mo–V steel [10]. Shape, size, distribution and stability of the precipitates influence the high temperature properties of the steel. The volume fraction, morphology, and distribution of second phase precipitates can be regulated through tempering treatment [11]. Lu et al. [12] have reported the effect of heat treatment on the microstructure and hardness of T92 steel and observed that hardness decreases with increase of tempering temperature. Golanski et al. [13] have studied the effect of heat treatment on the structure and properties of GX12CrMoVNbN9-1 cast steel and reported that mechanical properties depend on austenizing temperature. It is observed that austenizing and tempering temperature can influence microstructure and properties. Due to its strong carbide forming tendency, niobium can be precipitated in the form of NbC during heat treatment, which can influence the microstructure and properties of 9% chromium steel. The effect of individual alloying element, Nb, has not been reported.

Hence, the aim of the present work is to study the effect of tempering treatment on the microstructure and mechanical properties of niobium modified high chromium steel.

2. Experimental work

The steel investigated is produced in an induction furnace with a batch size of 3 kg. The steel composition is shown in Table 1. It is homogenized at 1100 °C for 4 h in a programmable muffle furnace and subsequently forged to a thickness reduction of around 40%.

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The forged steel is tempered at 500–800 °C for various intervals of time (1–5 h).

The cast, forged and tempered steel samples are polished and etched using 2% Vilella's reagent to study the microstructures by Optical Microscopy (LEICA: DM2500M). High resolution SEM (FEI: QUANTA FEG 250) is used to observe the detailed microstructural features of the samples. X-ray diffraction studies (BRUKER: D8 ADVANCE) are carried out using Co Kα radiation at the diffraction angle (2θ) from 20 to 120° to identify the phases. A cylindrical sample of 4 mm diameter and 10 mm length is used for dilatometric analysis (BAHR THERMOANALYZE GMBH: DIL 805). The rod is heated at 1 °C/s up to a temperature of 1000 °C and subsequently cooled to room temperature. TEM analysis is carried out using FEI: TECNAI G^2 20 S TWIN machine. The sample is mechanically thinned to a thickness of 50 μm and then a 3 mm diameter disc is cut using disc punch. The thin sample is further dimpled to a thickness of around 20 μm using a dimpling machine. Final thinning is done using ion miller (GATAN: PIPS 691). The hardness of the samples is measured using Rockwell hardness testing machine (Brooks Inspection Equipment: MAT10/RAB) at 150 kgf load and 15 s dwell time. Tensile tests are carried out using ASTM E8 standard at a strain rate of 1 mm/min in SHIMADZU-AG-5000G tensile testing machine.

3. Results and discussion

3.1. Dilatometric analysis

The change of phase with respect to the temperature of the steel is evaluated by dilatometric analysis. During continuous heating and cooling of a sample in a dilatometer, the relative change in length ($\Delta L/L$) and temperature (T) as a function of time is recorded. From the recorded data $\Delta L/L$ vs. T is plotted. A typical dilatometric curve for the steel at a heating rate of 1 °C/s is shown in Fig. 1. Ac_1, Ac_3 and M_s temperatures for the steel are 800 °C, 855 °C and 131 °C, respectively. It is observed that Ac_1 temperature is around 73 °C higher than that of binary iron–carbon steel (Fe–0.7C) and also the temperature gap between Ac_1 and Ac_3 is only 55 °C [4,14]. This is due to the presence of chromium in the steel. M_s temperature is ~170 °C less compared to Fe9Cr1Mo steel [15,16]. This may be due to the austenite stabilizing effect of niobium and higher content of carbon. It has been reported that niobium increases the stability of austenite by impairing the martensite nucleation [17]. Hojo et al. [18] have reported that niobium helps to increase the volume fraction of retained austenite. It also decreases the prior austenite grain size due to the precipitation of NbC at grain boundary. A finer grain size of prior austenite itself reduces the M_s temperature. Fine grain austenite provides resistance against the volume expansion that is needed for the austenite to martensite transformation [19].

Table 1
Chemical compositions of the investigated steel in wt%.

Cr	Mn	Si	Co	Cu	Ni	S	P	Nb	C	Fe
8.84	1.17	0.5	0.1	0.04	0.06	0.06	0.09	0.41	0.7	Bal

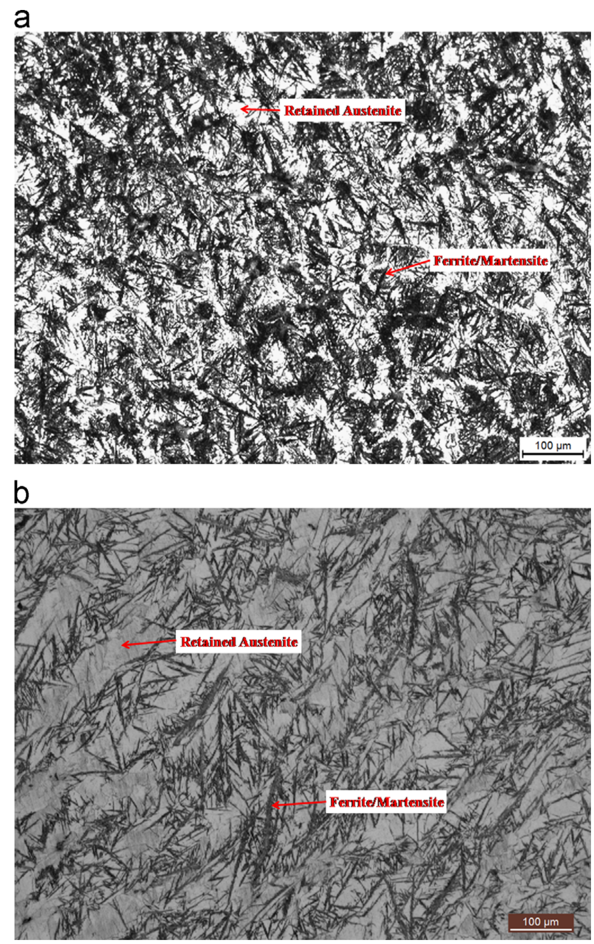

Fig. 2. Optical micrograph of steel (a) after casting and (b) after forging.

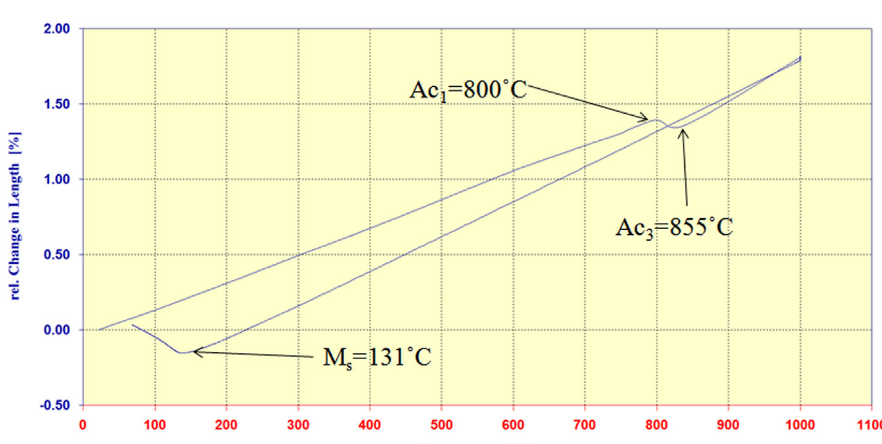

Fig. 1. Dilatometric curve ($\Delta L/L$ vs. T) of the steel.