

## Role of Nb in low interstitial 13Cr super martensitic stainless steel

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### ABSTRACT

The effect of adding 0.1 wt% Nb to low interstitial (N 0.01 wt%, C 0.01 wt%) 13Cr super martensitic stainless steel (SMSS) on solid phase transformation and microstructures achieved by normalizing and tempering was investigated using dilatometer, electron backscattered diffraction (EBSD), transmission electron microscope (TEM), X-ray diffraction (XRD), and its consequence on mechanical properties was examined to clarify the role of Nb in low interstitial martensitic stainless steel. Nb was found to retard kinetics of reversed austenite formation during tempering and to suppress the occurrence of Cr rich precipitates. The measurement of mechanical properties shows that while the strength properties were significantly increased by nano-scale precipitates enriched in Nb in the steel with 0.10 wt% Nb, the ductility and toughness properties were restored by optimum volume fraction of retained austenite. Excellent strength and adequate toughness properties were obtained by tempering the steel with 0.10 wt% Nb and low interstitial (N 0.01 wt%, C 0.01 wt%) steel at 600 °C.

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### 1. Introduction

Super martensitic stainless steels (SMSSs) have been increasingly applied to produce oil country tubular goods (OCTG) – seamless pipes for drilling, casing and tubing for the application in oil and gas fields with more corrosive environments [1]. They present an economical alternative against carbon steels and duplex pipes as they offer improved corrosion resistance in relation to carbon steels, being free from the use of coatings and inhibitors, and lower production costs when compared to duplex grades. Recently, a number of new weldable 13Cr martensitic grades, called “Super 13Cr [2–5]”, have been developed and are also being applied to flow lines from the wellheads to the treatment stations, transporting unprocessed fluid (crude oil or gas) at very high pressure and temperature. They are based on the Fe–Cr–Ni–Mo system with 4–6 wt% of Ni, 0.5–2.5 wt% of Mo, low amounts of carbon, nitrogen, phosphorus and sulfur ( $C \leq 0.02$ ,  $N, P, S \leq 0.03$ ). Typical values of mechanical properties range within the following: 650–750 MPa of 0.2% yield strength, 880–950 MPa of tensile strength, elongation at rupture up to 20% and impact energy up to 100J [5,6]. These properties are strongly dependent on the microstructure of martensite with retained austenite film occurred at martensite lath boundaries and within laths obtained by normalizing from austenite field temperature and tempering above Ac1 temperature. Low carbon content and precipitation of reversed austenite are the main reasons for low

strength properties of SMSSs. Micro alloying elements of Ti, Nb and V are usually employed to combine with residual C and N to form carbo-nitrides, thereby increasing the strength. The studies [7] on Ti microalloying of super martensitic stainless steel have shown that Ti as a “stabilizing and refining element” raises strength and toughness effectively. Due to the different solubility and precipitation behavior of carbo-nitride of micro alloying elements during processing and heat treatment, the effect of different microalloying element is different.

The objective of this study is to investigate the effect of high Nb (0.10 wt%) addition to low interstitial (C 0.01%, N 0.01%) 13Cr–5%Ni–1%Mo base chemical composition on phase transformation, microstructure and mechanical properties of normalized and tempered super martensitic stainless steel.

### 2. Materials and methods

The chemical compositions of two tested SMSSs with and without Nb are summarized in Table 1. Laboratory steel samples were made in the form of ingots, using a 100 kg vacuum induction furnace. The ingots were hot rolled at 1200 °C into plates with 12 mm thickness. Normalizing was carried out at 1050 °C for 0.5 h, followed by 2 h tempering heat-treatment at temperatures that ranged from 550 °C to 700 °C. After tempering, the samples were quenched in oil. The heating rate of tempering treatment is about 60 °C/min.

The dilatometer technique was performed to study the solid state transformation of the SMSSs. The continuous heating treatments were carried out by heating samples from room temperature at a rate of 60 °C/min to the austenitizing temperature of 1100 °C.

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**Table 1**  
Chemical compositions of two SMSSs (wt%).

Steel grade	C	Si	Mn	P	S	Cr	Ni	Mo	N	Nb	Ti
SMSS	0.022	0.3	0.57	0.022	0.001	12.84	4.44	0.69	0.01	–	–
Nb-SMSS	0.008	0.39	0.63	0.010	0.007	13.35	5.10	1.08	0.01	0.11	0.008

The isothermal treatment was conducted by heating at equivalent rate of 60 °C/min to 600 °C. The samples were maintained at this temperature for 3 h to simulate the phase transformation of the SMSSs during tempering. The mechanical properties were evaluated by means of tensile and impact tests. The tensile tests were performed on a tensile testing machine CMT5105 with an extensometer at strain rate of 0.001 s<sup>-1</sup>. Cylindrical test specimens with a uniform gauge cross-section of  $\varnothing$  5 mm  $\times$  25 mm and machined parallel to the plate rolling direction were used. The impact tests were performed on full size Charpy V notch impact specimens (10 mm  $\times$  10 mm  $\times$  55 mm) at room temperature. All mechanical properties value reported in the results are the average of three tests. All specimens for microstructure characterization were cut from residual portion of Charpy tests samples. Thin foils for transmission electron microscopy (TEM) analysis were prepared by mechanically grinding the samples with initial thickness of 400  $\mu$ m to the thickness of 100  $\mu$ m and electropolished with a double jet Tenupol Struers machine at -40 °C, 15 V in an electrolyte containing 27 mL perchloric acid and 273 mL alcohol. Carbon extraction replica were prepared on samples surface well-polished and lightly etched using electrolyte containing 2 mL HCl + 4 mL HNO<sub>3</sub> + 12 mL alcohol. The precipitate morphology, size and dispersion were examined using conventional transmission electron microscopy (TEM-Philips CM12 120 kV) as well as scanning transmission electron microscope (STEM-FEI-Titan 80–300 kV). The micro chemical compositions of precipitates were analyzed using Energy Dispersive Spectrometer (EDS) attached to TEM. The crystallographic microstructures were characterized by electron backscattered diffraction (EBSD). The volume fraction of the retained austenite was measured at room temperature by X-ray diffraction using Cu-K $\alpha$  radiation on tempered specimens. The evaluation of volume fraction was determined by measuring the integrated intensities of (1 1 1) $\gamma$  and (1 1 0) $\alpha$  peaks of X-ray pattern, using the procedure outlined by Leem et al. [8].

### 3. Results

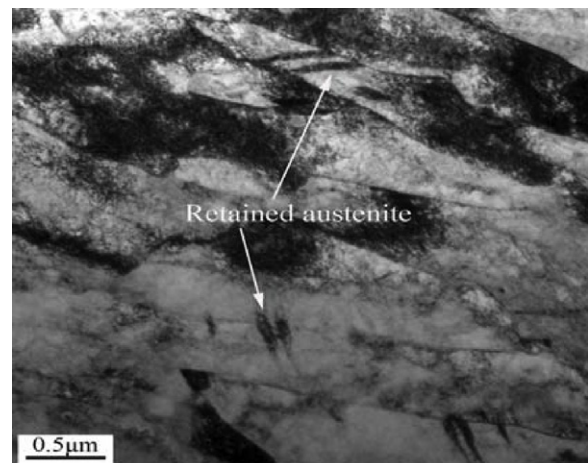
#### 3.1. Microstructural characterization

Fig. 1 shows the TEM image of highly dislocated lath martensite with small amount of retained austenite microstructure in the Nb-SMSS normalized by air cooling from austenite field temperature of 1050 °C. The individual martensite lath is found about 0.2–0.3  $\mu$ m thick. No precipitate was found in the as normalized sample using TEM, which reflects the absence or very small size of precipitates. Fig. 2 shows the EBSD characterization of crystallographic microstructure of as normalized SMSS (Fig. 2(a)) and Nb-SMSS (Fig. 2(b)). The prior austenite grain boundaries are found with misorientation above 15° (below 45°), while “martensite lath packet” boundaries exhibited misorientation above 45°, and the lath boundaries are low angle boundaries [9]. Comparing the size of prior austenite grains of SMSS with that of Nb-SMSS, prior austenite grains were not refined by Nb addition.

Fig. 3(a) shows the austenite transformation volume fraction as a function of heating temperature calculated from the continuous heating curves of dilation diagrams of SMSSs with and without Nb, using the procedure outlined by Lee et al. [10]. At same temperature, volume fraction of austenite transformed from martensite in SMSS is higher than that in Nb-SMSS. It can be con-

cluded that Nb retards the austenite transformation. It is known that the measurement of austenite transformation start temperature (Ac1 temperature) relates to the heating rate [8]. Although the Ac1 temperature is further above 600 °C up to 860 °C at heating rate of 60 °C/s, the phase transformation from martensite to austenite would occur if the samples were maintained at 600 °C. As shown in Fig. 3(b), the continuous reduction in length of samples during maintaining at 600 °C is attributed to the formation of austenite, since the C content in SMSSs is low and the influence of carbide precipitation on length reduction can be ignored. For the same holding time, the reduction in length of SMSS samples is more than that of Nb-SMSS sample, indicating that the volume fraction of austenite in SMSS is higher than that in Nb-SMSS. The initial contraction in length is due to the stress release of high dislocated martensite resulted from normalizing. The continuous variation of austenite volume fraction with increasing time indicates that the formation of austenite follows nucleation and growth mechanism associated with concentration of Ni into reversed austenite [8,11], other than shear mechanism without diffusion.

In agreement with many studies carried out on microstructure evolution during tempering of SMSS [8,11,12], long time tempering of martensite slightly above equilibrium Ac1 temperature, about 550 °C in present study results in transformation of martensite to reversed austenite, which is relatively stable on oil cooling to room temperature and can be partially retained at room temperature. Table 2 gives the volume fraction of retained austenite in two SMSSs after tempering at different temperatures calculated from X-ray diffraction results. Due to the low thermodynamic potential and kinetics for the formation of reversed austenite at lower tempering temperature, retained austenite cannot be detected by X-ray diffraction because of its low volume fraction after tempering at or slightly above 550 °C. Fig. 4 shows schematically the variation of volume fraction of retained austenite in two SMSSs as a function of tempering temperature. As tempering temperature is increased above 550 °C, the volume fraction of retained austenite in two SMSSs increases first and then decreases, indicating that the stability of reversed austenite becomes lower with increasing tempering temperature. It has been reported that the stability of



**Fig. 1.** TEM pictures of microstructure of the Nb-SMSS as normalized showing highly dislocated martensite laths with retained austenite.

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