Fatigue strength evaluation of a 13%Cr supermartensitic stainless steel by the thermographic method

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Abstract. The fatigue limits of a 13Cr super-martensitic stainless steel from a hot rolled seamless tube were determined for three heat treatment conditions, including the as received quenched and tempered (QT) condition, quenched (Q), and quenched and double tempered (Q-DT). These heat treatments were applied to produce different microstructures and different tensile mechanical properties. The microstructures were characterized by microscopy and magnetic methods. The fatigue strength was accessed through the utilization of the thermographic technique. The results show that fatigue limits measured were between 38% and 44% of the ultimate strength. The as quenched condition gave the higher fatigue limit (444 MPa). However, the material has the lower ductility at this condition. The results aid to decide the best heat treatment condition and give support to design in applications where dynamic loadings are expected.

1 Introduction

The so-called supermartensitic stainless steels (SMSSs) are corrosion resistant alloys developed initially to the oil and gas industry [1-4]. Usually containing 12-13%Cr, they have extra low carbon (<0.03%) to improve toughness, corrosion resistance and weldability. Nickel is added as austenitizing element and to increase toughness and corrosion resistance

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in high CO₂ environment [4]. Also, molybdenum is added to enhance the pitting corrosion resistance [4] and mechanical strength after tempering. UNS S41426 is one of the supermartenitic grades with basic composition 13%Cr-5%Ni-2%Mo (wt.%). Ti and V are added as micro-alloying to this grade to precipitate as carbides and carbonitrides which refines the microstructure and intensify secondary hardening.

Pipes and forgings of supermartensitic UNS S41426 are used in oil country tubular goods (OCTG) below the Christmas tree in oil and gas offshore production. For this application they are purchased as classes 95 (655 MPa) and 110ksi (758 MPa), which are the minimum yield limits (σ₀) specified. For pipeline application, in which the pipes must be joined by weld, SMSSs with minimum yield strength 80 ksi (552 MPa) were developed [5].

Mechanical and corrosion resistance of SMSSs may vary considerably with heat treatments [6-8]. Quenching may be carried out in liquid media from temperatures around 1000°C. Tempering parameters are fundamental to adjust the final properties of the steel. Usual tempering temperatures for 13%Cr-5%Ni-2%Mo (wt.%) steels ranges from 550°C to 650°C. The increase of tempering temperature in this interval in general causes the decrease of mechanical resistance and the increase of toughness. Double tempering is reported in the literature as heat treatment sequence that increase the austenite content, which can promote an increase in the low temperature toughness of the steel [9-10].

The influence of chemical composition and heat treatments on tensile properties and toughness of SMSSs have been extensively studied in the literature. However, the fatigue strength was not reported till now. The fatigue strength is mostly required if the pipe is used as risers for oil and gas transportation. The fatigue limit is an important parameter for design of pipes and accessories. In the present paper, the fatigue limits of a SMSSs UNS S41426 with different heat treatments were determined by thermographic method. This contactless method correlates the stress amplitude applied on a specimen with the heat generated in the surface due to crack initiation. The temperature variation (ΔT) with the applied number of cycles (N) is measured for growing stress amplitudes (Sₐ), using a unique specimen. Plotting ΔT/N against Sₐ, the fatigue endurance limit (Sₑ) is determined in the point where an abrupt increase of ΔT/ΔN is observed.

2 Experimental

The UNS S41426 steel used in this work was obtained from a seamless pipe with chemical composition shown in Table 1. Specimens were cut and pre-machined. A set of specimens were heat treated by quenching, and another set was quenched and double tempered. These two sets of heat treated specimens were compared to the as received material, which has been already quenched and tempered by the manufacturer. Table 2 describes the heat treatments and specimens identification.

| C   | Mn   | S    | Cr   | Ni   | Mo   | Ti   | V   | Fe |
|-----|------|------|------|------|------|------|-----|----|
| 0.028 | 0.454 | <0.001 | 12.1 | 6.20 | 2.01 | 0.145 | 0.044 | Bal. |

The three sets of specimens (Q, QT and Q-DT) were machined for tensile and fatigue tests. The specimens for tensile tests were subsize with gage length 6.25 mm (ASTM A-370 [11]). The fatigue specimens were carefully machined with the same roughness, with dimensions shown in Fig.1.

Tensile tests were performed in triplicate with velocity 0.5 mm/minute. Vickers hardness tests with load 10 kgf were also performed (8 indentations per specimen).
The fatigue tests were performed by rotating beam fatigue machine operating at 8500 rpm (frequency of 141.7 Hz). The contactless thermographic method correlates the stress amplitude applied on a specimen with the heat generated in the surface due to crack initiation. Fig.2 shows the experimental apparatus with the thermographic camera used to measure the temperature of the specimen. The specimen was painted with black ink. The temperature variation (ΔT) with the applied number of cycles (N) is measured for growing stress amplitudes (S_a), using a unique specimen. Plotting ΔT/ΔN against S_a, the fatigue endurance limit (S_e) is determined in the point where an abrupt increase of ΔT/ΔN is observed.

In this work, each ΔT/N versus S_a was constructed with 7 or 8 levels of S_a. The ratio between the minimum and maximum stress (S_{min}/S_{max}) was R=-1, which implies in mean stress equal to zero.

The microstructures were investigated by light optical (LOM) and scanning electron microscopy (SEM) and magnetic measurements to quantify the austenite volume fraction (AVF). Magnetization curves were constructed in a Vibrating sample magnetometer (VSM) with maximum applied field of 2T. The saturation magnetization (m_S) was used to determine the AVF with equation (1):

$$AVF = 1 - \frac{m_s}{m_S(i)}$$

Where m_S(i) is the magnetization intrinsic of martensite. This value was the magnetization saturation of a specimen quenched and cold rolled to obtain 100% of martensite.

| Identification | Heat treatment description |
|----------------|---------------------------|
| Q              | Water quenching (WQ) from 1000°C (1h) |
| QT             | Quenched and tempered by the steelmaker (as received) |
| Q-DT           | Quenched (1000°C/1h, WQ) and double tempered (670°C/2h + 600°C/2h) |
3 Results and discussion

Table 3 presents the tensile properties (yield strength ($S_y$) ultimate strength ($S_u$), elongation) and hardness of specimens representative of Q, QT and Q-DT.

Figs. 3(a-e) show the microstructures of specimens representative of the three tested conditions. Sample as quenched (Q) has a soft martensitic structure with very fine particles of Ti carbonitrides (Figs. 3(a-b)). The austenite volume fraction (AVF) measured by magnetism is 0.043. This condition presented the highest hardness, yield limit and ultimate strength, and the lowest elongation between the three tested conditions. Sample QT has mechanical properties compatible to grade 110 ksi ($S_y \geq 758$ MPa), with a microstructure consisting of tempered martensite, with small Ti carbonitrides and AVF = 0.081. Sample QT-DT was the softest material, with higher elongation and lower hardness than the other conditions. The microstructure is composed of tempered martensite and austenite fraction equal 0.154, produced by the double tempering treatment. In all heat treatment conditions the material contains square and relatively coarse TiN particles as those observed in Figs.3(c) and 3(e). Delta ferrite, which is detrimental to toughness [12] and act as initiation sites for fatigue cracks [13], was not observed in none of heat treatment conditions.

Table 3: Tensile properties and Vickers hardness (HV10) of Q, QT and Q-DT samples (average of three tensile tests and 8 Vickers hardness indentations).

| Specimen | $S_y$ (MPa) | $S_u$ (MPa) | Elongation (%) | HV 10 |
|----------|------------|------------|----------------|-------|
| Q        | 1004       | 1008       | 13.1           | 334±11|
| QT       | 832        | 881        | 22.1           | 301±4 |
| Q-DT     | 693        | 846        | 23.9           | 289±3 |
The method proposed by Risitano [12] was chosen to determine the $S_e$ from the $\Delta T/\Delta N$ versus $S_a$ curves. As an example, Figs. 4 (a-b) show the curves constructed for 2 specimens quenched (Q) and the determination of $S_e$. Table 4 shows the values of $S_e$, $S_e/S_u$, elongation and AVF of each specimen.

**Table 4: Fatigue endurance limits ($S_e$), $S_u$, elongation and AVF.**

| Specimen | Elongation (%) | $S_e$ (MPa) | $S_e/S_u$ | AVF  |
|----------|----------------|-------------|------------|------|
| Q        | 13.1           | 444         | 0.44       | 0.043|
| QT       | 22.1           | 297         | 0.34       | 0.081|
| Q-DT     | 23.9           | 324         | 0.38       | 0.154|
Analysing the results of Table 4, we can conclude that the SMSS as quenched has the higher $S_e$ and the higher $S_e/S_u$ ratio. At this condition the material has the higher mechanical resistance, but the lower ductility. The Charpy impact toughness of as-quenched SMSS UNS S41426 is high and the fracture is ductile in temperatures as low as -46°C [7-8], because the martensite with extra low carbon is soft. The specimen quenched and tempered (QT) for class 110 ksi presented the lower $S_e$ and $S_e/S_u$ ratio. An optimum combination of ductility, toughness and fatigue endurance was obtained in the specimen Q-DT. With this heat treatment the material achieves the requirements of class 95 ksi. The high AVF contributes to the high toughness [7-9] but does not decrease significantly the endurance limit ($S_e$).

4 Conclusions

The fatigue endurance limit ($S_e$) of a SMSS UNS S41426 was determined by thermographic method using the Risitano methodology [14]. The material was tested in 3 heat treatment conditions: quenched (Q), quenched and tempered (QT) and double tempered (Q-DT). Specimen Q has a microstructure of soft un-tempered martensite with 4.3% of retained austenite. Specimen QT has tempered martensite with 8.1% of reversed austenite, while the specimen double tempered has 15.4% of austenite. In all conditions the steel contains TiN and Ti(C,N) small precipitates which is believed to improve the mechanical properties. The highest $S_e$ and $S_e/S_u$ ratio was obtained in the as-quenched (Q) material, but with lower ductility. High ductility with intermediate $S_e$ and $S_e/S_u$ ratio was obtained in the specimen double tempered (Q-DT). The results of this work can be used to select the best heat treatment for design of SMSS structures subjected to dynamic loadings.

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