Hot Deformation Simulation of High Nitrogen Valve Steel

E. Erişir¹, U. Prahl², W. Bleck³

¹University of Kocaeli, Turkey, eerisir@kocaeli.edu.tr
²RWTH Aachen University, Germany, ulrich.prahl@iehk.rwth-aachen.de
³RWTH Aachen University, Germany, wolfgang.bleck@iehk.rwth-aachen.de

Abstract—In this study, the effect of microstructure on hot deformability of high nitrogen X50CrMnNiNbN21-9 valve steel was investigated. Phase transformations and precipitation were thermodynamically modeled as well as experimentally determined via microstructural characterization. Hot tensile and compression tests were used to simulate the hot deformation at temperatures between 900 °C-1300 °C. Hot tensile test determined the high temperature properties. The effect of temperature on cracking sensibility during hot deformation was investigated using hot compression test. The results showed hot deformability depends on primary and secondary precipitates of carbides and nitrides.

Keywords—High nitrogen valve steels, Thermodynamical modeling, Simulation of hot deformation, Hot tensile and compression

I. INTRODUCTION

High nitrogen X50CrMnNiNbN21-9 steel is used as a valve material for internal combustion engines [1,2]. In valve materials, there are two factors to be considered; high temperature and corrosive atmosphere [2,3]. The exhaust valves are not cooled and can be heated up to 850 °C. Prolonged operation time might cause precipitates and a decrease in corrosive properties. Therefore, austenite stability is increased by Nb alloying in order to prevent the precipitation of M23C6 at grain boundaries and lamellar M2N in grains [4,5].

However, high nitrogen steels still present several limitations. Edge and surface cracks as well as forging cracks can appear during hot deformation [6,7]. These types of cracks can be generated by precipitates and secondary phases. Lack of data related to hot deformation processes and phase transformations during processing restricts to a large extent the use of high nitrogen steels [8].

The purpose of the present study is to reveal the hot formability in X50CrMnNiNbN21-9 grade of high nitrogen steel with particular emphasis on precipitation. Phase diagrams were calculated with Thermo-Calc software in order to determine precipitation characteristics and phase transformations. The investigation of precipitations after homogenization annealing helps to draw conclusions from appearing phases and dissolving temperatures. Hot formability of investigated steels was determined via hot tensile and compression tests at elevated temperatures between 900 °C and 1300 °C.

II. EXPERIMENTAL PROCEDURE

A. Material

The experimental samples were produced from X50CrMnNiNbN21-9 (DIN EN 1.4882) high nitrogen steel with a nitrogen amount of 0.48 wt.-%. The complete chemical composition is listed in Table 1.

The cylindrical samples for tensile and compression tests are shown in Figure 1. The samples were pre-annealed at 1150 °C for 120 min under a gas mixture of 5 vol.-% N2 and 95 vol.-% CO2 and cooled in a furnace. After the pre-annealing, the samples were tested using the hot deformation simulator.

B. Thermodynamic Modeling

All elements present in the Table 1 were also considered in the calculations with the Thermo-Calc software using the TCS Steel Database TCFE5 [9,10]. The aim of the calculations is the prediction of phase equilibrium for various temperatures.

C. Microstructural characterization

The homogenization annealing was carried out in a Bähr DIL805 plastodilatometer in order to investigate the precipitation and phase transformations. The standard specimen size of 5 mm diameter and 10 mm length was used for the dilatometer experiments. The samples were annealed at 1000 °C, 1100 °C and 1200 °C for 15 min, followed by quenching with cooling rate of 200 K/s and slow cooling with cooling rate of 0.003 K/s. The samples were metallographically prepared. After grinding, they were polished using diamond pastes with particle sizes of 6, 3 and 1 μm. Beraha II and V2A were used as the etchant agents. Scanning electron microscope (SEM) attached energy dispersive spectrometer (EDS) was used to observe the morphology of secondary phase particles and to analyze their chemical compositions. In addition, metallographic observations were made with a light microscope (LM).

D. Hot tensile test

Through the tensile test, the maximum load and the reduction of area were measured. The hot tensile samples in Figure 1a were heated to 1250 °C with a rate of 5 K/s and soaked for 15 min for homogenization annealing. A typical deformation temperature range between 900 °C and 1300 °C.
was chosen (Fig. 2). The deformation rate of 1/s was applied during the tests before the samples cooled down to room temperature with a cooling rate of 50 K/s. Argon was used as protective and cooling gas. The temperature was controlled with a tolerance of ± 5 K using a thermocouple. The reduction of area was plotted as a function of test temperature in order to determine the hot deformability. The fracture surfaces of tensile test samples were also investigated by SEM. Tensile samples after tensile test were given in Fig. 3.

E. Hot compression test

The hot compression tests were performed using a Schenck hot deformation simulator at different deformation degrees up to crack formation on the cylindrical collar samples given in Figure 1b. The aim was to determine a forging window. The collar samples were chosen because of their localized crack susceptibility and thus to reduce experimental effort for identification of cracking locations. The hot compression tests were conducted in the temperature range 900 – 1300 °C at a strain rate of 1 s⁻¹. A thermocouple spot-welded at the surface of specimens was used for the measurement and the control of temperature.

In the beginning of the hot compression tests, an initial degree of the deformation was chosen from the results of tensile tests. The collar samples were compressed with the initial degree of the deformation and examined for the surface cracks with a stereo microscope. If a crack was not present in collar of the samples, degree of the deformation was increased or vice versa. Thus, a limit of degree of the deformation was determined without causing cracks on collar samples.

III. RESULTS

A. Thermodynamic modeling comparison with experimental results

The Thermo-Calc software was used to predict the stability of occurred phases and precipitates. Fig. 4a is an isopleth diagram showing stability of phases as a function of temperature and nitrogen content. The dashed line in the diagram indicates the relevant nitrogen amount for the steel composition used here. According to the thermodynamic calculations, liquid, ferrite, austenite, M(C,N), M₂₃C₆, M₂N and σ phases are in equilibrium. The phase diagram of the steel calculated in Thermo-Calc is in Fig 4b. According to phase diagram, M(C,N) is stable up to 1344 °C. M₂₃C₆ carbides are stable up to 1103 °C. Below 813°C, a small amount of M₂N nitride is also present in the matrix. Sigma phase might also precipitate below 754 °C.

The microstructures of the annealed samples were given in Fig. 5. As calculated in Thermo-Calc, primary M(C,N) on grain boundaries was observed. Cluster of secondary precipitates around the primary M(C,N) phase were also seen. Primary M(C,N) remained undissolved up to 1200 °C. With increasing temperature, the amount of observed clusters decreased and completely dissolved in samples annealed at 1200 °C.

Table 1. Chemical composition (wt-%) of the examined steel.

<table>
<thead>
<tr>
<th>Steel grade/Alloy</th>
<th>C</th>
<th>Mn</th>
<th>Cr</th>
<th>Mo</th>
<th>Ni</th>
<th>W</th>
<th>Cu</th>
<th>Nb</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.4882 / X50CrMnNiNbN21-9</td>
<td>0.48</td>
<td>8.45</td>
<td>20.60</td>
<td>0.21</td>
<td>3.60</td>
<td>0.83</td>
<td>0.15</td>
<td>2.00</td>
<td>0.48</td>
</tr>
</tbody>
</table>
Figure 4: Calculated phase diagrams using Thermo-Calc: (a) isopleth diagram and (b) mass fraction of phases as a function of temperature at the indicated nitrogen content with a dashed line in (a).

Figure 5: Microstructures after annealing and quenching (200 K/s); (a) 1200 °C/15 min, (b) 1100 °C/15 min, and (c) 1000°C/15 min.
In order to determine the chemical composition of primary and secondary precipitates, EDS analysis were performed. Fig. 6a shows the microstructure of sample annealed for 15 min at 1100 °C and cooled at 0.003 K/s. The matrix phase includes mainly Fe and Cr (Fig. 6b). Compare to matrix phase, the EDS analysis of primary M(C,N) indicates Nb in Fig. 6c. Also, the cluster of secondary precipitates was analyzed. According to EDS peaks, Cr as well as W and Mo elements were detected, as shown in Fig. 6d. According to the work by Berns et al, high nitrogen valve steels may include coarse Nb<sub>2</sub>C and Cr<sub>23</sub>C<sub>6</sub> carbides in an austenite matrix phase [5]. With consideration of thermodynamic modeling and literature, the cluster might be a mixture of M<sub>23</sub>C<sub>6</sub> and M<sub>2</sub>N phases. Intermetallic σ phase was not observed in microstructure, although it was predicted in the thermodynamic modeling. The precipitation of σ phase in austenitic matrix could be expected for prolonged annealing time [11].

B. Hot deformation simulation

In order to understand the deformation behavior, hot ductility data obtained from the tensile testing are used. Reduction of area (RA) is defined as:

\[
RA = \frac{A_1 - A_2}{A_1} \times 100
\]

where \(A_1\) = original cross sectional area of specimen before test and \(A_2\) = cross sectional area of specimen at fracture after test. Maximum load in tension could also be determined from the tensile test data.

Fig. 7 shows the curves of RA and maximum load against the testing temperature. According to RA values, poor ductility was observed at temperatures above 1150 °C. The RA is practically zero % at 1100-1300 °C. However, ductility becomes poor outside of this temperature range. At low temperature end, the ductility increased up to 30 %. The maximum load value decreases with increasing testing temperature. The zero value of maximum load at 1300 °C indicates the melting of steel, as predicted by Thermo-Calc. The maximum load increases up to 63 kN at 900 °C.

A forging frame is determined by the occurrence of ductile fracture at a given temperature and therefore limits are set by the appearance of surface cracks within regions that are highly strained due to extensive material flow. In order to determine the effect of deformation temperature to crack formation, hot compression tests with collar specimen were performed. The degree of deformation, \(\varphi\) in compression tests is given as:

\[
\varphi = \ln \left( \frac{h_0}{h} \right)
\]

where \(h_0\) = original height of specimen before test and \(h\) = height of specimen after compression.

Figure 6: Precipitates after slow cooling (0.003 K/s); (a) SEM micrograph, (b-d) EDS analysis for (b) matrix, (c) Nb rich M(C,N) and (d) cluster of secondary precipitates of M<sub>23</sub>C<sub>6</sub> and M<sub>2</sub>N (1100 °C/15 min).
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Fig. 7: Reduction of area and maximum load

Fig. 8 gives the degree of deformation versus deformation temperature. Above the $\varphi$ curve, surface cracks appear on collar of compressed samples. The samples compressed at temperatures between 900-1300 °C show poor hot deformability. The collar samples can be compressed without crack by a deformation degree lower than 0.27.

IV. DISCUSSION

Precipitation and hot deformation behavior were investigated for a wide range of temperature in austenitic grade X50CrMnNiNbN21-9 high nitrogen steel. It was found that the hot deformation behavior is very sensitive to the temperature. Main results can be summarized as follows:

Phase equilibria predicted by ThermoCalc and annealing experiments showed that various precipitates of carbides and nitrides might form. Primary M(C,N) particles have high thermodynamic stability and cannot be dissolved at annealing temperatures up to 1200 °C. Secondary $\text{M}_2\text{C}_6$ and $\text{M}_2\text{N}$ phases were found in cluster form and not stable above 1200 °C. At deformation temperatures, carbides and nitrides present in microstructure. Predicted $\sigma$ phase was not observed because of prolonged equilibrium conditions.

In hot tensile tests, the reduction of area and maximum load values were nearly zero at 1100-1300 °C. Hot compression at temperatures of 900–1300 °C results with a poor in a maximum deformation degree about 0.27.

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REFERENCES