Stress corrosion cracking behavior of quenched and tempered 2.25Cr 1Mo steel

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Abstract. For stress corrosion cracking (SCC) study of 2.25Cr-1Mo steel, specimens were initially austenitized at 925°C for 2 hours and quenched in ice brine solution. With this process of heat treatment martensite was formed and banded morphology in the microstructure developed during previous processing was also removed. Tempering at 575°C for 1h was employed to introduce ductility in the quenched martensite of the specimens. The effects of temperature variations on SCC behavior showed a successive decrease in strength and ductility of the steel. Theses variations in tensile properties were correlated with scanning electron microscopic (SEM) examinations. The SEM results clearly revealed that mostly transgranular cracks initiated at the edges of the tensile specimens grown perpendicular to loading axes, and their intensity is increased with the testing temperatures.

1. Introduction
Chromium-Molybdenum ferritic steels (generally referred as Cr-Mo steels) have excellent high temperature properties, formability, weldability and resistance to stress corrosion cracking and other forms of corrosion [1, 2]. These features make Cr-Mo steels the extensively used family of engineering materials for moderately high temperature applications over several decades, such as conventional power and petroleum industries for fabrication of pressure vessels, boiler tubes and coal liquefaction reactors [1-3]. With increasing demand for suitable materials for supercritical and ultra-supercritical steam generators for power plants having much improved efficiencies, Cr-Mo steels have attracted great interest for their potential application in low-temperature parts of such steam generators that will be expected to operate at higher than usual temperatures.

Among Cr-Mo class of steels with specific applications, 2.25Cr-1Mo has its importance in heat treated conditions. In quenched and tempered states of heat treatment these steels show range of mechanical properties due to carbide precipitation specially chromium carbides [4-5]. The nuclear industry considers 2.25Cr-1Mo steel as strong candidate for structural material of the parts due to their better resistance to swelling than stainless steel [6-7]. In the present studies a simulated environment similar to nuclear application of the material was developed to assess the changes or deterioration in the mechanical properties. The optical and scanning electron metallographic observations were added to support the results of these findings. The chemical composition of the 2.25Cr-1Mo steel is given in Table 1.

| Table 1. Chemical Compositions of Steel in wt. percent |
| C | Si | Mn | P | S | Cr | Mo | Fe |
|---|---|---|---|---|---|---|---|
| 0.14 | 0.14 | 0.56 | 0.009 | 0.003 | 2.38 | 0.89 | Bal. |
The as received material was provided in the form of bars having 12x12x100mm dimensions. The initial optical metallographic studies revealed ferrite and pearlite with banded morphology of microstructural contents, shown in Fig. 1.

![Microstructure of as received material showing banded morphology](image1)

**Figure 1.** Microstructure of as received material showing banded morphology

The specimens from as received bars were austenitized at 925°C for 2h followed by ice brine quenching. The specimens were further heat treated by tempering for one hour at 575°C.

For stress corrosion cracking behavior studies, slow strain rate tests (SSRT) was carried out using CORTEST testing machine in PWR simulated environment. Normally the pressure vessel in PWR works at about 290–310°C under a pressure of about 15 MPa. The coolant contains Boric acid and LiOH, which are instantaneously added into the coolant system as per requirement to control fission reactivity and pH, respectively. These conditions were simulated in the laboratory using demineralized water (DMW) having a room temperature pH. Boric acid and LiOH were added to the DMW in amounts giving 100 ppm of boron and 7 ppm of Li in the solution. The tensile samples (25 mm gauge length, 5 mm gauge diameter) used in this study, shown in Fig. 2, were machined from the quenched and tempered ferritic steel test bars.

![Tensile specimen used for SCC testing](image2)

**Figure 2.** Tensile specimen used for SCC testing

The slow strain rate tests (SSRT) were performed on as received and quenched samples in air at room temperature while SCC testing of quenched and tempered samples was carried out in the autoclave having simulated solutions at temperatures of 75, 150, 200 and 275°C. All tests were performed at an initial strain rate of $1 \times 10^{-6} \text{s}^{-1}$. The microstructural analysis of the secondary cracks and fracture surface morphology were studied using Lieca optical microscope and Phillips scanning electron microscope (SEM).
2. Results and Discussion:

2.1. Development of Microstructure after Heat Treatment

The microstructures developed after austenitizing at 925°C followed by quenching in brine solution, and tempering after quenching are shown in Fig. 3 (a), and (b) respectively.

The lath or plate-like morphology of martensite is expected in 0.5 wt.% carbon steel after quenching from the austenite region[8]. The morphology of quenched martensitic microstructure appeared in Fig. 3 (a) looks like small laths with lenticular plates. The hardness of the quenched martensite is Hv = 485. The specimens were tempered at 575°C before proceeding towards SCC testing. The aim of tempering was to introduce ductility in the quenched specimens and to compare the SCC behaviors in both of these conditions. The microstructure revealed after tempering is shown in Fig. 3(b). The carbon diffused out in the form of carbides and in the presence of Cr in the composition, chromium carbides would have been formed. A considerable drop in hardness of Hv = 347 was observed after tempering attributed to the diffusion of carbon entrapped at interstitial positions after quenching. The reduction in hardness after tempering at 575°C indicates that softening effect due to diffusion of entrapped carbon is dominant than temper embrittlement. However, change in toughness which is true indicator of temper embrittlement effects can not be assessed from the reduction in hardness after tempering.

In steels containing chromium, two types of carbides are often formed: Cr7C6 (trigonal in nature) and Cr23C6 (complex cubic). As chromium is weaker carbide former than vanadium, carbides of Cr23C6 are formed only when their contents increased more than 7% and Cr7C6 carbides are formed at1% of chromium level in the steel [8]. However, Cr23C6 were observed in 2.25Cr-1Mo steel after quenched and tempered conditions [9].

2.2. Tensile Properties at Different Temperatures in Simulated Environment:

True stress-true strain curves of specimens to onset of necking in quenched and tempered conditions tested at different temperatures in simulated environment are shown in Fig. 4.
The tensile curves in as received conditions with ferrite & pearlite microstructure and as quenched condition with un-tempered martensite microstructure, tested at room temperature, are included for comparison. A smooth yielding behavior with absence of sharp yield point was observed in all the specimens. The tensile properties of these specimens are shown in Table 2.

| Specimen Code | 0.2% Proof Stress (MPa) | Max. True Stress (MPa) | True Uniform Strain (%) |
|---------------|-------------------------|------------------------|------------------------|
| 75°           | 414                     | 480                    | 8.11                   |
| 25°C          | 433                     | 498                    | 8.13                   |
| 150°C         | 382                     | 457                    | 7.5                    |
| 200°C         | 388                     | 436                    | 7                      |
| 275°C         | 378                     | 419                    | 5.35                   |
| As Received in air | 187                  | 482.4                  | 17.15                  |

|                 | Quenched in air        | 606                  | 711                    | 7.3                    |

In as quenched condition the rise in strength at the expense of ductility is characteristic of hard martensitic microstructure.

**Figure 4.** True stress-true strain curves of specimens to onset of necking tested at different temperatures in simulated environment.
The simulated environment testing at 25, 75, 150, 200 and 275°C resulted in successive decrease in maximum true stresses and maximum true strains with rise in testing temperatures, shown in Table 2. This decreasing trend of strength with rise in testing temperatures is shown as block diagram in Fig. 5.

![Figure 5. Strength Levels of 2.25Cr-1Mo Steel at Different Temperatures](image)

Also included in Fig. 5 is the yield to tensile strength ratio, predicting the work hardening of the specimens. Although yielding in as received specimen was started very early at 187 MPa of stress but it gained a maximum true stress of 482 MPa due to excessive work hardening in this condition. In quenched and tempered conditions maximum work hardening was achieved for sample tested at 150°C and it decreased with further increase in testing temperature. In SCC the specific environment is very importance, and only very small concentrations of certain highly active chemicals can produce catastrophic cracking [10]. Considering this environmental fact and its application in present SCC testing, the cracks would have been initiated at the surface, and at higher temperatures their intensity increased and they propagated more efficiently. Therefore, inter-granular and trans-granular forces have lost the strength to raise the work hardening component with increasing testing temperatures. This decrease in work hardening resulted in losing ductility due to limited extension of stress-strain curves till start of necking in tensile deformation.

2.3. Stress Corrosion Cracking (SCC) in Simulated Environment

SCC is a combined action of stress and a corrosive environment leading to the formation of a crack which would not have developed by the action of the stress or environment alone. The PWR simulated environmental condition created for the present studies in the laboratory are quite effective for the enhancement of SCC. At room temperature (25°C) near the tensile fracture surface (Fig. 6) little micro-cracks on the edges were observed.
The excessive microvoid concentration and severe plastic deformation of the grains along the tensile axes in the necked portion predict the ductile failure with an increase in the ductility. Trans-granular cracking was observed at 75°C. At higher testing temperatures micro-cracks initiated from the edges of the specimens trans-granularly and inter-granularly, and propagated inward due to SCC, shown in Fig. 7.

The severity of SCC increased with testing temperatures and branches of cracks from the initial source advanced with inter-granular zigzag movement as shown in Fig. 8.

**Figure 6.** Microvoid formation and deformation along tensile axes near fracture surface on sample tested for SCC at 25°C.

**Figure 7.** (a) SCC at 75°C showing transgranular cracking perpendicular to tensile axis. (b) SCC at 150°C showing intergranular cracking perpendicular to tensile axis.

The severity of SCC increased with testing temperatures and branches of cracks from the initial source advanced with inter-granular zigzag movement as shown in Fig. 8.

**Figure 8.** (a) SCC at 200°C showing inter and transgranular crack branches normal to loading axis. (b) SCC at 275°C showing inter and transgranular crack branches normal to loading axis.
The inter-granular cracking looked a prefer mode of fracture in the present case at higher temperatures. This may be due to weakening of the forces or the cohesion strength of the grain boundaries due to their embrittlement. Two micro-mechanisms controlled the changes in mode of fracture due to long term thermal exposure and embrittlement:

- Temper embrittlement caused by growth of carbides, precipitated at the grain boundaries changed the transgranular cleavage from dislocation triggered to carbide induced [11, 12].
- Intergranular embrittlement caused by diffusion of impurities and carbide coarsening at grain boundaries [13, 14].

At 75°C in the present studies, there is no considerable indication of precipitation along the grain boundaries and the crack propagated trans-granularly. However, at higher temperatures the mode gradually changed from trans-granular to inter-granular. There is clear indication of inter-granular growth at 275°C with excessive precipitation along the grain boundaries to follow the crack path.

3. Conclusions
Stress Corrosion Cracking (SCC) behavior was studied at 25, 75, 150, 200 and 275°C, in quenched and tempered states of 2.25Cr-1Mo steel. The results are concluded as follow:

- Initially the steel with banded ferrite and pearlite microstructure was austenitized at 925°C and full martensite was obtained by quenching in iced brine solution. Tempering for one hour at 575°C reasonably reduced the hardness due to diffusion of carbon from entrapped interstitial positions.
- Tensile testing in simulated environment clearly demonstrated that severity of SCC certainly increased with increasing the testing temperatures. A smooth decrease in tensile strength and ductility was observed by increasing the testing temperatures. Similarly the work hardening property was also decreased due to enhancement of simulated environment effect at higher temperatures.
- The microstructural observations revealed that at room temperature the material remained ductile and tensile deformation progressed with void formations in the necked region. The SCC initiated from the edge of the specimen at 75°C trans-granularly but at higher temperatures mode of SCC changed to inter-granular. Precipitation along the grain boundaries at 275°C promoted the SCC to progress inter-granularly.

4. References
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