Fracture Mechanisms of Micro-Alloy Steel at Elevated Temperature

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Abstracts: The objective of this work is to study the hot ductility and fracture mechanisms of micro-alloy steel of industrial production whose initial structural state is a rolling stock. To simulate the thermomechanical treatments imposed we have deformed by tensile our samples after having subjected them to a solution treatment at 1200 °C and a precipitation treatment cycle before deformation. Hot deformations were carried out at a deformation rate of $1.96 \times 10^{-3} \text{ s}^{-1}$ and temperatures from 700 °C to 1050 °C. By observing our tensile-deformed specimens, we can suggest that there is a link between the damage suffered and the type of fracture that results.

Keywords: Micro-alloyed steel, hot tensile, Flow Stress, Fracture mechanisms.

1. INTRODUCTION

Ductility is the result of a trade-off between useful deformation parameters, such as recovery and recrystallization, and various damage mechanisms, which can be local stress increases and/or local energy decreases [1]. Mechanical and thermal treatments of metals are often limited by the occurrence of defects that lead to loss of ductility and affect their further use. In [2], Sellars gives an overview of the physical metallurgy of the heat treatment process and discusses the microstructural changes that occur during heat treatment, including recrystallization and grain growth and their impact on the final material properties. These defects appear as surface or internal cracks. Some materials are less ductile at elevated temperatures within a given temperature range due to their brittleness.

The successful processing of steels at high temperatures, such as forging, rolling, or pressing, requires industrials to have a comprehensive understanding of hot brittleness and a thorough knowledge of the ductility trough. Researchers have conducted multiple studies [3, 4] to analyze the hot ductility of micro-alloyed steels and explore how factors like composition, deformation temperature, and strain rate influence the ductility of these materials. These investigations yield valuable insights for the development and fabrication of such steels.

Several studies have investigated the ductility of materials, yielding various and occasionally conflicting outcomes. This phenomenon arises from a delicate balance between factors that facilitate plastic deformation and factors that exacerbate the damage. Damage is typically characterized by the emergence and expansion of voids or cavities, often occurring during the initial stages of plastic deformation. Notably, the research presented in references [5-8] sheds light on how steels behave in high-temperature and high-strain rate scenarios, revealing their suitability for applications requiring elevated temperatures.

In order to control the use of metals and alloys, it is necessary to study their thermomechanical behavior before subjecting them to deformation treatments on an industrial scale. Zhang et al. [9] studied the effect of heat treatment conditions on the flow behavior, recrystallization mechanism, microstructural changes during hot deformation, and their effects on the final properties. It is characterized by various laboratory tests according to different parameters such as deformation temperature, deformation speed, and microstructural properties of the material.

In this study, the thermoplastic analysis and fracture mechanisms of (C-Mn-S-Al-Nb-V-Ti) micro-alloyed steels were evaluated and performed by tensile tests. The study aimed to simulate the thermomechanical treatment that steel undergoes during production by deforming it in tensile tests after a solution treatment and precipitation 14

treatment cycle at 1200°C. Thermal deformation is carried out at a certain deformation speed at different temperatures between 700°C and 1050°C. Observations of the tensile samples helped to show the correlation between damage and the type of fracture that occurred during the test. his research contributes to a better understanding of the material's behavior and its suitability for use in welded pipelines for gas and oil transportation.

2. MATERIAL AND EXPERIMENTAL TECHNIQUES

In our study, samples were obtained from microalloyed steel hot-rolled coils. The material is characterized by the presence of various additional elements, the respective percentages of which were determined after the precipitation treatment. The chemical composition is as follows: carbon (C) 0.075%, sulfur (S) 0.011%, phosphorus (P) 0.017%, aluminum (AI) 0.047%, silicon (Si) 0.22%, manganese (Mn) 1.67%, 0.051% vanadium (V), 0.062% niobium (Nb) and 0.045% titanium (Ti). Analyzing the initial microstructure, the average grain size was 20 µm (as shown in Figure 1). This microalloyed steel was specially developed for the manufacture of welded pipes for gas and oil transportation. The addition of these alloying elements imparts the required properties to the steel and makes it suitable for such applications.

In order to perform tensile tests on the samples, a heat treatment process was performed in advance (as shown in Figure 2). The main purpose of this heat treatment is to precipitate the alloying elements before the deformation stage. The process starts with the homogenization of the material structure, which is achieved by heating the sample to 1300 °C. Water quenching is then performed to rapidly cool the material. After the quenching step, the samples were machined in the quenched state. These quenched samples were then fixed in a tensile tester. In the next step, the samples were solution treated at 1200 °C.

During this treatment, the material is maintained at a specified temperature to allow further dissolution and distribution of alloying elements within the microstructure. After solution treatment, the samples were gradually cooled to a deformation temperature range between 700 °C and 1050 °C. The purpose of this cooling process is to gradually precipitate previously dissolved added elements within the microstructure of the material. Controlled elimination of these elements is essential to achieve the desired properties and properties of microalloyed steels.



Figure 1. Initial microstructure of specimens



Figure 2. Heat treatment cycle aimed at precipitation before deformation

3. RESULTS ANALYSIS

3.1. Stress-Strain Curves

The stress-strain curves were determined from the force-strain curves and recorded after the elastic components were eliminated after heat treatment cycles with different deformation standards. The tensile curves obtained (Figure 3) show a considerable increase in tension followed by a more or less rapid decrease in tension. This typical appearance indicates the presence of a softening mechanism that increases with increasing test temperature (T = 800°C). This results in a drop in tensile strength with peak stress increasing from over 140 MPa at 800°C to less than 60 MPa at 950°C. It also resulted in a slight decrease in the strain rate at fracture, ranging from 0.6 to 0.5. This trend was observed at all temperatures except 750 °C. At this temperature, we found no bearings with stresses up to 140 MPa. Compared with other temperatures, the ultimate strain rate at the maximum peak is low, about 0.4. These behaviors are often described by dynamic recovery. However, at elevated temperatures (>1100 °C), we observe a sinusoidal shape that characterizes dynamic recrystallization.



Figure 3. Evolution of the tensile stress under heat as a function of temperature at the strain rate of 1.96x10⁻³ s¹

3.2. Fracture Mechanisms

The presence of precipitates hinders intergranular displacement and leads to the concentration of stress around the precipitate sites. Sulfur and phosphorus aid in reducing the cohesion energy at the interface between the precipitates and the matrix, as well as at the grain boundary. The fracture occurs due to the initiation, growth, and coalescence of intergranular cavities, which always result in an intergranular break. In Figure 4, we can see very clearly the priming of the intergranular cavities' triple points. Their alignments and their cyclic aspects suggest that the growth and coalescence of these cavities inevitably lead to fracture.



Figure 4. Longitudinal section of a tensile sample deformed at 900 °C (Priming of the triple point break over the entire width of the sample and intergranular cavities at triple points and their cyclic aspects)

By observing our tensile-deformed specimens we can propose a correlation between the damage incurred and the type of fracture that occurs. The initial category applies to substances that withstand cavity formation, resulting in a ductile cup-to-cup fracture at inter-critical temperatures or a slow, highly ductile fracture with necking at higher temperatures, as illustrated in Figs. 5 and 6.



Figure 5. Observation of a very ductile slow fracture with a pointed necking at a high temperature of 1050 °C)



Figure 6. Very ductile slow fracture obtained at 1050 °C

The second type is evident in materials that are susceptible to cavity germination but are not very resilient to crack propagation, leading to either slow or rapid fractures of the mixed or intergranular type, as shown in (see Figures 7 and 8).







Figure 8. Very ductile slow fracture with pinch necking obtained at 1050 °C

It appears that the additive elements influence cavity germination and crack growth as seen in Figures 9 to 11 since germination and growth of cavities also depend on intergranular slippage and species diffusion rate.

The presence of aluminum nitride at the same time as the sulfur restores the ductility for a given sulfur content at temperatures higher than those observed in the case of steels containing sulfur only [10]. In this case, the ductility is controlled by the dissolution and coalescence of aluminum nitride precipitates. This process makes it possible on the one hand to reach the critical conditions such as the size and the number of precipitates, necessary to unlock the dynamic recrystallization, and on the other hand, to reduce the number of potential sites for priming the cavities.



Figure 9. Presence of manganese sulfide precipitate inside a cup



Figure 10. Presence of manganese sulfide precipitate inside a cup.



Figure 11. Presence of titanium nitride/manganese sulfide duplex precipitate within a cup

3.3. Relationship between Damage and Precipitation

Without taking into account any other considerations, intergranular precipitation is frequently stated as the reason for the reduction in ductility in industrial steels. Our experimental findings show that the simultaneous precipitation and germination of a ferritic phase at the austenitic grain boundaries is what causes the degradation of ductility. As was previously noted, the precipitates dissolve, increasing the ductility should be noted that precipitates often cause a delay in the dynamic recrystallization and precipitation-induced hardness. The dynamic recrystallization is delayed and there is no precipitation hardening, as can be seen on the stress-strain curves shown above. The two primary processes that take place in the event of a hot intergranular cracking nucleation of the intergranular cavities and their growth are caused by the interaction of all these variables.

Metallographic results indicate that the heat-deformed specimens have a considerable portion of cavities. Two phenomena favor the development of cavities. The first is the concentration of stress at the triple points or near the 20

precipitates at the joint. Since the precipitates are observed inside the cavities, the concentration of the stress is probably located in Figures12 and 13.



Figure 12. Several precipitates inside the cavities



Figure 13. Several precipitates on the matrix and inside the cavities. Priming cavities at precipitation sites

The second phenomenon is related to the cohesion of the precipitated interfaces/matrix or grain boundaries. A low cohesion strength of the grain boundary facilitates the nucleation of the cavities and allows the separation of various interfaces during deformation. In the two-phase domain, the weakening of the intergranular cohesion is accentuated by the presence of the germination of the ferritic phase at the austenitic joint. Metallographic observations and tensile test results seem to support this analysis. To observe the intergranular precipitates of the elements such as Nb, V, and Ti, specimens were broken at the temperature of the liquid nitrogen.

Observations using Scanning Electron Microscopy (SEM) (see Figure14), show that the fracture is fragile and intergranular, confirming the presence of precipitates of NbC, VC, and TiC. It is the intergranular precipitation of the additive elements which greatly favors embrittlement by their combined actions with sulfur and phosphorus and by

their opposition to the migration of grain boundaries which should have occurred under the effect of temperature and temperature. the stress of deformation.



Figure 14. Brittle fracture obtained at the temperature of the liquid nitrogen

CONCLUSIONS

The objective of this study was to examine the fractography of microalloyed steel that was subjected to tension at varying temperatures. The SEM investigation of the fractured tensile specimens revealed that different fracture mechanisms were active in each region, depending on the test temperature. The tensile fracture behavior was found to be consistent with the alloy's strength and ductility variations with temperature. A void formation fracture mechanism was observed across a wide range of experimental conditions, while intergranular fracture and cleavage occurred only within specific temperature ranges. The minimum ductility was observed at approximately 900 °C, which is in agreement with previous research on high-purity alloys [11]. In steels containing dispersed elements such as sulfur and phosphorus, this ductility is likely dependent on the competition between grain boundary damage and dynamic recrystallization-induced grain restoration. The level of segregation increases with decreasing deformation temperature during the precipitation treatment when sulfur and phosphorus are present, which weakens the joints and reduces ductility.

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