Microstructure and Mechanical Properties of Two API Steels for Iron Ore Pipelines

Leonardo Barbosa Godefroid*, Luiz Cláudio Cândido, Rodrigo Vicente Bayão Toffolo, Luiz Henrique Soares Barbosa

Rede Temática em Engenharia de Materiais – REDEMAT, Universidade Federal de Ouro Preto – UFOP, CEP 35400-000, Ouro Preto, MG, Brasil

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This research compares the mechanical behavior of two API steels (X60 and X70) used in the longest pipeline in the world for the conveyance of iron ore. Tensile tests, Charpy impact tests, CTOD tests and fatigue crack growth tests are performed at ambient temperature. Metallographic examination showed a banded microstructure consisting of polygonal ferrite and pearlite in both steels, with smaller grain size and the presence of a small quantity of bainite in the X70 steel. All the mechanical tests revealed a ductile behavior for the two steels. The X70 steel is preferable for the pipeline project, due to its better mechanical resistance, with no significant loss of fracture toughness and fatigue resistance. Its performance could be even better, if an appropriate combination of thermomechanical processing parameters were able to produce a microstructure with minor amount of pearlite, where acicular ferrite/bainite are present.

Keywords: API steels, fracture toughness, fatigue crack growth

1. Introduction

Over the past 30 years, the world production of oil and gas and the consumption of their products have grown significantly, which has caused an increase in the use of pipelines for their transport. Similarly, the use of pipelines for transporting iron ore over long distances has been a solution adopted by many mining companies. To achieve this demand, it is necessary that the pipes used in transport have larger diameters and work at high pressures. The development of high strength steels, which avoid the use of very high wall thicknesses, makes a significant contribution to pipeline project cost reduction. The manufacture of steel pipelines for oil, gas and iron ore transmission follows the API 5L standard. The requirement of high mechanical resistance, combined with good fracture toughness at low temperatures and also a good weldability, implies the use of high strength low alloy steels (HSLA), obtained by thermomechanical processing. The ultimate goal is to obtain a microstructure with the presence of well-selected phases and refined grain size.

During its operation, defects in pipelines can nucleate and propagate as fatigue cracks while the structure is subjected to internal and external cyclic loading, and fatigue failure can occur in the pipelines. Therefore, a clear understanding of fatigue and fracture toughness properties for pipeline steels is important to provide information for pipeline design during construction and predict pipeline live during operation.

Several studies on the microstructure – basic mechanical properties relationships for pipeline steels have been carried out since 1980s. However, little information is available on fracture toughness and fatigue, mainly on near-threshold fatigue crack growth behavior. Therefore, the present study was undertaken to evaluate the behavior of two commercial microalloyed API pipeline steels manufactured by Brazilian steel plants. The API 5L X60 and X70 grade steels were analyzed and compared in terms of microstructure and mechanical properties. The objective of this study was to verify the safely replacement of an old steel (X60) by a modern steel (X70) in a recently designed transport line of iron ore.

2. Materials and Experimental Procedures

The X60 steel was manufactured by traditional hot-rolling and normalising operations while the X70 steel was obtained by thermomechanical processing, with appropriate choice of rolling parameters: slab reheating temperature, roughing and finishing mill temperatures, degree of final deformation and coiling temperature. For all tests, samples were prepared from cutting, with orientation that maintains the mechanical load for the tests always parallel to the original iron ore flux.

Chemical analyses of the steels were performed by means of a ThermoARL optical emission spectrometer. Specimens taken from the longitudinal plane of the steels were mechanically polished and etched by a 2% Nital reagent, and then microstructures were observed by a JEOL scanning electron microscope (SEM). All the mechanical tests were conducted at room temperature in laboratory air. Hardness and impact tests were performed on WOLPERT machines. Tensile tests, fracture toughness tests (CTOD) and fatigue crack growth tests (da/dN x ∆K) were conducted
on a 10ton MTS servo-hydraulic testing machine interfaced to a computer for machine control and data acquisition. Three-point single-edge bend SE(B) specimens (5 mm thick, 20 mm wide) in T-L orientation were used for all the fracture toughness and fatigue tests. Fracture toughness tests were performed to determine the CTOD (crack tip opening displacement) value at the first attainment of a maximum load plateau, for stable ductile crack extension. Experimental CTOD estimates were made by separating the CTOD into elastic and plastic components, in accordance with the ASTM E1290 standard\textsuperscript{15}. Fatigue crack growth tests were performed under a sinusoidal waveform at a frequency of 30Hz with a load ratio of 0.1, in accordance with the recommendations of ASTM E647 standard\textsuperscript{16}. The fatigue threshold value $\Delta K_{th}$ was defined as the stress intensity factor range at which the fatigue crack growth rate decreased to below $1 \times 10^{-5}$ mm/cycle. This value was estimated by a K-decreasing procedure. The crack closure load was estimated using a crack opening displacement (COD) compliance technique, and was assumed to be the point when the COD-load curve begins to deviate from the linear elastic curve. The constants $C$ and $m$ of the well-known Paris equation are obtained by the linearization of the $da/dN$ versus $\Delta K$ curve between $1 \times 10^{-5} \text{ and } 1 \times 10^{-3}$ mm/cycle. After the tests were completed, the fracture surfaces were examined by a JEOL scanning electron microscope.

3. Results and Discussion

The chemical compositions of the two steels used in the present study are shown in Tables 1 and 2. Comparing these results with the existing specifications for pipelines, it is verified that the steels satisfy the API requirements\textsuperscript{12}. It should be noted the lower carbon content of X70 steel, compensated by the presence of microalloying additions. In this case, the worldwide trend for using Nb-Cr is perceived\textsuperscript{10}, in view of the increasing cost of Mo and V. Contents of S and P are below the maximum allowed by the API standard, to minimize the formation of inclusions (elongated manganese sulfide particles) and segregation (phosphorus segregation to austenite grain boundaries during austenitization), to minimize the tendency for embrittlement phenomena and to avoid the decrease of mechanical properties of the steel (low fracture toughness and low fatigue resistance).

Figure 1a-d shows the SEM micrographs of the two steels studied. It is observed in both steels the presence of polygonal ferrite/pearlite banding (pancake type), a common occurrence in hot-rolled, low alloy steels\textsuperscript{17,18}. Banding is the microstructural condition manifested by alternating bands of quite different microstructures aligned parallel to the rolling direction of steel products, caused by interdendritic segregation (for example, manganese is frequently associated with banding). X70 steel presented a lower ferritic grain size (ASTM 11 to 15) than X60 steel (ASTM 8 to 10)\textsuperscript{19}. This is a consequence of the well-known beneficial presence of microalloying elements (Nb and Ti) and the rolling parameters employed in the X70 steel thermomechanical processing\textsuperscript{20,22}. It’s interesting to note the presence of a small volumetric fraction of bainite / degenerated pearlite and the absence of acicular ferrite in X70 steel. For a pipeline steel, if these constituents can be achieved, it will be with better properties combination\textsuperscript{21-34}, such as high mechanical strength, excellent fracture toughness, good $H_{15}$ resistance, reduction of the Bauschinger effect and superior fatigue behavior, than the polygonal ferrite – pearlite microstructure. The distribution of inclusions in the two steels was also analyzed. According to the specific ASTM standard for steels\textsuperscript{15}, inclusions are classified in the D-globular oxide type, thin, severity level 0.5, scattered randomly in the microstructure. No MnS inclusions or grain boundaries precipitates were observed in both steels.

Table 3 shows basic mechanical properties obtained in this study for the two API steel. This table presents results of tensile, hardness and impact tests. With respect to tensile behavior, the results of both steels are according to API standard\textsuperscript{12}. It’s important to note that X70 steel presented higher tensile mechanical resistance, hardness and impact absorbed energy than X60 steel, without significant loss of ductility. The cumulative effect of observed fine-grained ferrite microstructure, in conjunction with the operation of other hardening mechanisms (solid solution, fine precipitation and dislocation strengthening), provided superior mechanical resistance values to X70 steel\textsuperscript{20-23}. Ductile behavior can still be observed in the two steels by their mechanism of fracture, regardless of the difference in mechanical properties: tensile and impact specimens showed the operation of the mechanism of nucleation, growth and coalescence of micro voids\textsuperscript{16,37}.

Table 1. Chemical composition of the API-5L-X60 steel (wt.%).

<table>
<thead>
<tr>
<th></th>
<th>C</th>
<th>Si</th>
<th>S</th>
<th>P</th>
<th>Mn</th>
<th>Ni</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>0.192</td>
<td>0.149</td>
<td>0.005</td>
<td>0.026</td>
<td>1.324</td>
<td>0.018</td>
</tr>
<tr>
<td>Cr</td>
<td>0.014</td>
<td>0.019</td>
<td>0.010</td>
<td>0.001</td>
<td>0.001</td>
<td>0.063</td>
</tr>
</tbody>
</table>

Table 2. Chemical composition of the API-5L-X70 steel (wt.%).

<table>
<thead>
<tr>
<th></th>
<th>C</th>
<th>Si</th>
<th>S</th>
<th>P</th>
<th>Mn</th>
<th>Ni</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>0.109</td>
<td>0.239</td>
<td>0.004</td>
<td>0.023</td>
<td>1.536</td>
<td>0.011</td>
</tr>
<tr>
<td>Cr</td>
<td>0.024</td>
<td>0.011</td>
<td>0.026</td>
<td>0.016</td>
<td>0.045</td>
<td>0.045</td>
</tr>
</tbody>
</table>

Figure 2 (tensile) and Figure 3 (impact). On the other hand, it appears that the elastic ratio ER is relatively high for both steels. This relationship is assuming growing importance in thick plates intended for the manufacture of large diameter pipes\textsuperscript{38}; the lower is this value, the lower the trend to the development of the so-called “spring-back effect” during the conformation of the pipe. All the mechanical properties could be better for the X70 steel, with adjustments in thermomechanical processing, reduction of pearlite content and the use of a microstructure consisting of acicular ferrite/bainite\textsuperscript{22,24,38-42}. The loss of strength resulting from reduced pearlite content can be offset by precipitation hardening and dislocation hardening. Thanks to the finer grain size and the higher dislocation density of the bainite compared to polygonal ferrite, this microstructure offers higher strength and also improves toughness.
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Figure 1. Microstructural analysis, SEM, longitudinal section. Nital 2% etching. PF = polygonal ferrite; P = pearlite; B = bainite.

Table 3. Basic mechanical properties of the studied steels.

<table>
<thead>
<tr>
<th>Steel</th>
<th>( \sigma_{YS} ) (MPa)</th>
<th>( \sigma_{US} ) (MPa)</th>
<th>ER (%)</th>
<th>( \varepsilon_t ) (%)</th>
<th>AR (%)</th>
<th>( HR_A )</th>
<th>( C_V ) (J)</th>
<th>LE (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>X60</td>
<td>500</td>
<td>576</td>
<td>87</td>
<td>40</td>
<td>65</td>
<td>59</td>
<td>169</td>
<td>14</td>
</tr>
<tr>
<td>X70</td>
<td>586</td>
<td>640</td>
<td>91</td>
<td>38</td>
<td>65</td>
<td>65</td>
<td>184</td>
<td>14</td>
</tr>
</tbody>
</table>

\( \sigma_{YS} \): tensile yield stress; \( \sigma_{US} \): tensile ultimate stress; ER: elastic ratio = \( \sigma_{US}/\sigma_{YS} \); \( \varepsilon_t \): tensile total elongation; AR: tensile area reduction; \( HR_A \): Rockwell Hardness A; \( C_V \): Charpy impact absorbed energy; LE: Charpy lateral expansion.

Figure 2. Fracture analysis, tensile test specimens. SEM, 500X.
Figure 4. Typical load ($P$) versus displacement ($COD$) curves from CTOD tests. Arrows indicate the determination of the plastic component of the CTOD.

Figure 5. Fracture analysis, CTOD test specimens. SEM, 500X.

Table 4. Fracture toughness and fatigue crack growth results.

<table>
<thead>
<tr>
<th>Steel</th>
<th>CTOD (mm)</th>
<th>$\Delta K_{th}$ (MPa(\sqrt{m}))</th>
<th>$K_{cl}/K_{max}$</th>
<th>$C$ ($x 10^{-9}$)</th>
<th>$m$</th>
</tr>
</thead>
<tbody>
<tr>
<td>X60</td>
<td>0.35</td>
<td>9.6</td>
<td>0.35</td>
<td>1.07</td>
<td>3.52</td>
</tr>
<tr>
<td>X70</td>
<td>0.33</td>
<td>9.8</td>
<td>0.40</td>
<td>0.95</td>
<td>3.55</td>
</tr>
</tbody>
</table>

CTOD: fracture toughness; $\Delta K_{th}$: threshold limit, fatigue crack growth in region I; $K_{cl}/K_{max}$: closure effect; $C$: constant of Paris’equation, fatigue crack growth in region II; $m$: constant of Paris’equation, fatigue crack growth in region II.

The fracture toughness test results reveal a similar behavior for the two steels. Table 4 shows the results obtained. Figure 4 illustrates typical load ($P$) versus displacement ($COD$) curves from CTOD tests obtained with both steels. This figure shows the determination of the plastic component of the CTOD. It is interesting to note that although the maximum load supported by X70 steel is higher than the corresponding value for the X60 steel, its value of COD is less, hence the similarity between the CTOD values. Ductile behavior is observed again for both steels\textsuperscript{36,37}, Figure 5.

With respect to the fatigue crack growth resistance, the behavior of the two steels is also similar, in the near-threshold regime (region I) and in the linear regime (region II, the well-known Paris regime) of the traditional sigmoidal curve $da/dN \times \Delta K$. Table 4 shows basic characteristics obtained with these tests. Figure 6a presents the fatigue...
crack growth rates of the two steels as a function of $\Delta K$. In the near-threshold regime, the fatigue crack growth rate of the X70 steel was slightly lower than that of the X60 steel. At higher crack growth rate the sigmoidal curves tended to converge. These behaviors can be explained by the crack closure phenomenon\textsuperscript{13,14,43,44}. Figure 6b presents

![Fatigue crack growth results.](image)

Figure 6. Fatigue crack growth results.

![Fatigue crack growth results.](image)

(a) $da/dN \sim 10^{-7}$mm/cycle.  
(b) $da/dN \sim 10^{-4}$mm/cycle.

Figure 7. Fracture analysis, fatigue specimen. SEM, 2,000X, X60 steel.

![Fracture analysis, fatigue specimen. SEM, 2,000X, X70 steel.](image)

(a) $da/dN \sim 10^{-7}$mm/cycle.  
(b) $da/dN \sim 10^{-4}$mm/cycle.

Figura 8. Fracture analysis, fatigue specimen. SEM, 2,000X, X70 steel.
the crack closure levels of the two steels as a function of $\Delta K$. It is possible to see that the $K_{cl}/K_{max}$ ratio was slightly different for the two steels in the low $\Delta K$ region. Two mechanisms are considered to explain crack closure in this region: roughness or oxide. The fatigue specimens showed, for both steels studied, a transgranular fracture surface, without corrosion deposits. On the other hand, a roughness surface was observed, suggesting the operation of roughness-induced crack closure. The crack closure level decreased with increasing $\Delta K$ for both steels. This implies that crack closure effect is more significant in the low $\Delta K$ region. Fracture surfaces with shear regions in $\Delta K$ next to crack growth threshold ($da/dN = 10^{-3}$mm/cycle), and fracture surfaces with fatigue striations in the linear region of crack growth ($da/dN = 10^{-4}$mm/cycle) are illustrated in Figures 7 and 8 for each steel studied. These different crack growth mechanisms are typical of fatigue cracking in the two regions.

4. Conclusions

Two commercial microalloyed API 5L X60 and X70 grade steels were analyzed and compared in terms of microstructure and mechanical properties. The following conclusions can be drawn from the investigation.

- Both steels satisfy the API requirements for chemical composition.
- Both steels presented a banded microstructure consisting of polygonal ferrite and pearlite. X70 steel also presented a small quantity of banite and a lower ferritic grain size than X60 steel.
- X70 steel presented higher mechanical resistance (tensile, hardness and impact) than X60 steel, without significant loss of ductility, but with high elastic ratio.
- The fracture toughness test results (CTOD) reveal a similar behavior for the two steels.
- In the near-threshold regime, the fatigue crack growth rate of the X70 steel was slightly lower than that of the X60 steel. At higher crack growth rate the sigmoidal curves tended to converge. The crack closure phenomenon can be used to explain these behaviors.
- The X70 steel is acceptable and preferable for the pipeline project, but its performance could be even better, if an appropriate combination of thermomechanical processing parameters were able to produce a microstructure with minor amount of pearlite, where acicular ferrite / bainite are present.

References


