X-Ray Diffraction Evaluation of Dislocation Density and Crystallite Size in the HAZ

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Abstract

The aim of this work is to study the effect of successive welding process on microstructure and mechanical properties of the heat affected zone (HAZ) in welded HSLA steel, employed in petroleum and gas transport. The analysis of the diffraction pattern profiles by the Rietveld refinement method (X-ray Diffraction Line Profile Analysis XRDLPA), allows obtaining detailed information on the evolution of the microstructural parameters such as the sizes of the domains consistent with diffraction (crystallite size), micro-deformations and dislocation densities. Based on the X-ray diffraction analysis (XRD) using Material Analysis Using Diffraction (MAUD) software, the results show an outstanding evolution of microstructural parameters in the HAZ, which means an increase in the crystallite size and a decrease in the dislocation density according to the number of welding process.

Key words: HSLA, XRD, Dislocation density, HAZ.

I. Introduction

The welding process is generally the simplest, fastest, and most cost-effective technological process widely used in various industries and engineering fields such as pipeline construction, shipbuilding, gas production, offshore oil and others. Different physical metallurgical processes that occur during welding induce appearance of significant residual stresses that may have considerable influence on service behavior of welded structures and components, to cause even failures of welded joints [1-3].

Dislocations are responsible for most aspects of plastic deformation of metals. The dislocation density (ρ) is a microstructural feature extensively studied in the scientific community and can be investigated by several instrumental techniques, such as transmission X-ray diffraction and electron microscopy. The determination of the dislocation density using XRD is based on the broadening of the diffraction lines. Over the years, methods and programs have been developed to assess efficiently ρ by the X-ray diffraction profile analysis.

There are many methods of residual stress measurements but one of the important is the X-ray diffraction method that permits to carry out detail analysis of residual stress state in weld
region. X-ray diffraction line profile analysis applied to study of HSLA steel weldment allows measuring of dislocation densities in the HAZ. Difference of physical and mechanical properties of component is additional factor that contributes appearance of residual stresses after welding of HSLA steel pipe. Residual stresses measured in HSLA steel pipeline jointed by welding are caused by temperature gradients between weld zone and base metal which induce an important dislocation densities in HAZ.

In the literature, only a few studies report on the influence of successive repairs on the mechanical behavior of HSLA steel welded joint without highlighting the effect of the microstructural parameters on the mechanical properties in the HAZ, which is considered as the most vulnerable zone in the welded joint. In the present paper we aim at assessing the effect of successive repairs on the microstructure evolution using XRD with Rietveld refinement method to determine the relationship between dislocation density and crystallite size in the HAZ according to the number of welding process.

II. Material and Methods

Grade API5LX70 steel commercialised in pipeline was used with 8 " diameter and 14.2 mm of thickness, surface finish prior to welding, using welded SMAW process in 07 passes with E6010 and 7010 electrodes (Figure1).

The results of Chemical analysis of the X70 showed 0.073C, 1.65Mn, 0.61Nb, 0.066 Mo, 0.044Ni, 0.01Ti and 0.06 Si

![Figure 1. Welded assembly](image)

The sequences of repairs which consist in grinding the welded joint until a certain depth to completely remove the weld metal (WM), and repair the assembly again. We proceed to repair the welded joint three times and identify the specimens for each repair. A radiographic control is required for each specimen to verify the quality of the assemblies after each weld repair according to the requirements of API-1104 standard [4]. In order to identify the macro- and micrographics of the welded joint, the specimens were longitudinally cut and polished until mirror state and subsequently etched with Nital at 2%.
The XRD technique was performed to characterize the HAZ of the different welded samples using BRUKER D8 diffractometer with copper λKα radiation. In this work, MAUD software (Material Analysis Using Diffraction), based on a Rietveld refinement procedure was used. This method was discussed at length in literature [5].

III. Results and Discussion

3.1 Macrostructure evolution as function of the number of repair

Macrographics of the different weld repair conditions are given in Fig. 2. These macrographics show three different zones: base metal (BM), weld metal (WM) and heat affected zone (HAZ) for all samples.

![Macrographics of different weld repair conditions](image)

Figure 2. Cross section of the different weld repairs conditions. (Wo) as-welded, (R1) first repair, (R2) second repair, (R3) third repair.

From the results of microstructural examination shown in Fig. 3, it was seen the HAZ is formed of Bainite and Acicular Ferrite mixture, which exhibits a typical grains-coarsened heat affected zone (GCHAZ) adjacent to the weld metal (WM) where, the grains are relatively larger than the ones of BM. Further, in the interface between the HAZ and the BM, grains-fined heat affected zone (GFHAZ) are detected.
In accordance with the number of repairs, grain size evolution and grain size measurements are illustrated in Figs 4. It is noted that the measurements are carried out in three different regions of the HAZ: top, middle and bottom. It can be clearly seen that the higher the number of repairs, the larger the grain size. In the first repair, the grains are finer than the as-welded ones; their sizes shrink from 12 to 10 µm in the middle region; and, this is due to the refinement of the grains which may be attributed to the presence of micro-precipitates NbN, TiN [6][7]. Yet, from the first to the third repair, the grain growth is observed in the top region of the HAZ. The same tendency is recorded in the two other regions. Furthermore, O.E Vega [8] observed that the grain size measurements increase with the number of weld repairs, which produce grain growth in the HAZ, mainly in the middle and bottom areas as compared with the as-weld condition.

Figure4. Grain size in HAZ as function of the repair number.
3.2 X-ray diffraction line profile analysis using MAUD software

Fig. 5 corresponds to the diffractograms of X-ray diffraction of the base metal and HAZ for each of the samples of the welded joints. After identifying the ICDD reference spectra, the three peaks correspond to the diffraction plane (110), (200) and (211) of the ICDD card 00-006-0696 characteristics of Fe α body-centered cubic structure. The R1 peak is the highest one in the plan (110) yet the smallest in the plan (200) comparatively to the other samples. This orientation would be due to the effect of crystalline texture and the microstructure developed in this process.

![XRD Diffractograms of BM and HAZ of different samples](image)

**Figure5.** XRD Diffractograms of BM and HAZ of different samples.

Table 1, shows the results obtained from the Rietveeld analysis for X70 steel HSLA welds. The lattice parameter evolution with the number of repairs is mainly caused by the presence of both tensile and compressive residual stresses which causes XRD peaks displacement.

<table>
<thead>
<tr>
<th></th>
<th>BM</th>
<th>Wo</th>
<th>R1</th>
<th>R2</th>
<th>R3</th>
</tr>
</thead>
<tbody>
<tr>
<td>a (Å)</td>
<td>2.862</td>
<td>2.864</td>
<td>2.868</td>
<td>2.867</td>
<td>2.869</td>
</tr>
<tr>
<td>D (Å°)</td>
<td>1154.089</td>
<td>3864.485</td>
<td>2058.045</td>
<td>3215.580</td>
<td>3325.952</td>
</tr>
<tr>
<td>R.m.s</td>
<td>7.955x10^{-4}</td>
<td>4.647x10^{-4}</td>
<td>5.143 x10^{-4}</td>
<td>6.127 x10^{-4}</td>
<td>4.550 x10^{-4}</td>
</tr>
<tr>
<td>p(m²)</td>
<td>2.638x10^{-10}</td>
<td>0.460x10^{-10}</td>
<td>0.955x10^{-10}</td>
<td>0.728x10^{-10}</td>
<td>0.570x10^{-10}</td>
</tr>
</tbody>
</table>
This result shows that, after the welding process (Wo) the material is under a low local strain, leading to minimum value of micro deformation ($r_{ms}$) and an important value of crystallite size (D). Fig. 6 clearly shows that, after the first repair (R1) the strain is more important which causes an increase in both the micro-deformation and the dislocation density. After the second repair (R2), a decrease in microstrain and dislocation density in the HAZ, this can be explained by the partial relaxation of the local strain and residual stress distribution[25].

![Figure 6. Dislocation density variation with the number of repairs](image)

In Fig. 7, we notice an increase in values of crystallite size as function of number of repair; this result is due to the refinement of grains and recrystallization phenomena. It is accompanied by a decrease of microstrain and dislocation density.

![Figure 7. Crystallite size variation with the number of repairs](image)
Conclusion

The concluding remarks drawn by this work ascertain that

- The successive repair has a significant influence on the grain size. The higher the number of repairs, the larger the grain size.
- The successive repairs affect the microstructures of the assemblies and they also generate an increase in the crystallite size, which is accompanied by a decrease in the dislocation density in the HAZ. This can be attributed to the repetitive thermal cycles.

References