Metallographic, structural and mechanical characterization of weld nuggets in Fe-Mn-Al-C low-density steels microalloyed with Ti/B and Ce/La by GTAW process

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This article has been accepted for publication and undergone full peer review but has not been through the copyediting, typesetting, pagination and proofreading process, which may lead to differences between this version and the Version of Record. Please cite this article as doi: 10.1002/srin.202100229.

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Abstract

The use of low-density steels has become increasingly important due to the advantages they offer, such as high-strength, high-ductility and principally weight reduction. However, there are few studies regarding their weldability by conventional processes due to the metallurgical complexity of these alloys, which include kappa carbides precipitation and hot-cracking associated with the high contents of Mn, Al and C. The main objective of this research work is the characterization of the fusion zone (FZ) and different heat-affected zones (HAZ-1 and HAZ-2) of austenitic Fe-Mn-Al-C LD steels microalloyed with Ti/B and Ce/La. For this purpose, weld nuggets were obtained by autogenous GTAW process using heat inputs of 660 and 975 J. The weld nuggets were characterized metallographic, structural and mechanically by light optical microscopy, scanning electron microscopy, X-ray diffraction and Vickers microhardness. In general, weld nuggets showed three well-defined zones (FZ, HAZ-1, HAZ-2) where γ-austenite is the main phase and AlN and MnS particles act as nuclei of complex precipitated particles of microalloying elements such as TiC and (Ce, La)O. Low-density steels microalloyed showed a considerable decrease in hot-cracking. HAZ-2 displayed the highest hardness values (up to 450 HV) due to strong precipitation of kappa carbide with a modulated structure.

Keywords: Low-density steel; Ce, La, Ti, B microalloying elements; Weldability, GTAW process; Heat-affected zone; Kappa carbide.

1. Introduction

The use of steels in the industry has spread completely worldwide. However, due to the current environmental conditions, the energy efficiency of the systems and/or structures in which low-density (LD) steels intervene must be increasingly promoted. LD steels of the Fe-Mn-Al-C system were developed in the 50’s to replace stainless steels using Cr instead of Al and Ni instead of Mn [1]. However, its potential as a structural steel in the automotive industry was soon noticed due to its low density and high mechanical resistance, which attracted the attention of the industry. Among other benefits, the Fe-Al-Mn-C system steels exhibit good cryogenic properties and corrosion resistance [1]. The addition of Al to Mn-rich austenitic steels generates two important effects: on the one hand, the increase of the stacking fault energy (SFE) and, on the other hand, the precipitation of kappa carbides. This last feature is the most important reinforcement mechanism in austenitic Fe-Mn-Al-C steels that contain high amounts of Al and C. However, these carbides produce a complex system that makes difficult to join these steels by conventional welding processes. The kappa phase is produced by spinodal decomposition (γ → y₀ + γ' → y₀ + L1₂ → y₀ + L'1₂/E₂₁ = y₀ + k) [2], where y₀ is a carbon lean γ-phase, γ' is a carbon rich γ-phase, L₁₂ is a...
short range ordered phase with Al atoms at the corners of the cube and Fe/Mn at the faces. $L1_2$ phase undergoes further ordering of C atoms, which settle at the octahedral site of the cube lattice, resulting in the formation of $E2_1$ and $L'1_2$ structures, where $L'1_2$ finally has the same crystallographic structure of austenite, in order to accomplish the requirement of the spinodal decomposition, i.e., same crystal structure of parent and product phases. This last phase $L'1_2/E2_1$ turns into k-carbides [3]. This phase transformation generates intragranular cuboidal carbides of nanometric size, by the concentration of C and Al, which directly affect their formation [1]. On the other hand, aging heat treatment is the most common process that triggers the spinodal decomposition of austenite [3]. However, the driving energy which promotes its precipitation can also be obtained from the hot-rolling process and the own chemical composition [2]. Such carbides can also be formed after the welding process [4], since the steel is exposed to a severe thermal cycles of rapid heating/cooling transformations, which can promote a complex field of internal stresses. Accordingly, the heat-affected zone (HAZ) and the fusion zone (FZ) of LD steels present defects such as stress concentration or residual stress as a result of thermal cycles.

It is well-known that one of the main problems related to welding is the generation of cracks, which can occur due to residual stresses induced by expansions or contractions at the time of the δ-ferrite and γ-austenite transformations achieved in the welded joint, exceeding the stress or shear resistance of the base metal (BM) or weld metal (WM), which can be generated during cooling, when both BM and WM have low hot ductility. Thus, hot cracking and kappa carbide precipitation represent important factors to assess in LD steels weldability. Studies by Bartlett et al. [5] have shown that small additions of Ce to LD steels produce more nucleation sites during solidification, which in turns promotes a reduction in the grain size. Also, rare earth elements (Ce/La) are highly reactive and rapidly combine with O and P to form and modify non-metallic inclusions [6, 7]. On the other hand, Ti additions to steels have a well-known function as deoxidizer and desulfurizer, but also as grain size refiner. Additions of B to steel have been used in order to improve its mechanical resistance through the formation of BN (less harmful) instead of AlN and its grain growth inhibition effect [8]. It is worth mentioning that the literature available on welding of LD steels is very limited. Some exemptions can be found in the works by Saha et al. [9] and Lee et al. [10] who carried out laser welding (LW), gas tungsten arc welding (GTAW), continuous laser welding (CLBW), pulsed laser welding (PLBW) and spot welding (SW), in different LD steels compositions. Some of the most relevant effects produced by fusion welding of LD steels are chemical segregation, hot cracking, variations in hardness, formation of inclusions and phase transformations. Although this topic has already been studied in the literature, the information is still scarce. This work is focused on the problems generated by using a welding process that is
widely used in the industry (GTAW), without the need to use scarce and expensive processes such as laser welding (LW), continuous laser welding (CLBW) and pulsed laser welding (PLBW).

2. Experimental details

Ingots of three austenitic LD steels were prepared using an induction furnace, one cast without microalloying elements (named LD-NM), and another two casts microalloyed with Ce/La and Ti/B (named LD-REM and LD-Ti/B, respectively). The chemical composition of the studied steel is shown in Table 1. The steels were microstructurally conditioned by a three-stage hot-rolling thermo-mechanical treatment at 1200 °C then cooled down to room temperature within the furnace, followed by a solutioning heat treatment at 1150 °C and finally a water quenching to room temperature. The reduction in area of the three stages of hot rolling was 60%, 33% and 50%, respectively.

Table 1. Chemical composition of the studied low-density steels (wt.%).

<table>
<thead>
<tr>
<th></th>
<th>Fe</th>
<th>Mn</th>
<th>Al</th>
<th>C</th>
<th>P</th>
<th>S</th>
<th>Ce</th>
<th>La</th>
<th>Ti</th>
<th>B</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td>LD-NM</td>
<td>Bal</td>
<td>31</td>
<td>8</td>
<td>1.9</td>
<td>0.01</td>
<td>0.001</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>0.015</td>
</tr>
<tr>
<td>LD-REM</td>
<td>Bal</td>
<td>31</td>
<td>8</td>
<td>1.9</td>
<td>0.01</td>
<td>0.001</td>
<td>0.074</td>
<td>0.046</td>
<td>-</td>
<td>-</td>
<td>0.015</td>
</tr>
<tr>
<td>LD-Ti/B</td>
<td>Bal</td>
<td>27.3</td>
<td>7</td>
<td>1.2</td>
<td>0.012</td>
<td>0.001</td>
<td>-</td>
<td>-</td>
<td>0.019</td>
<td>0.005</td>
<td>0.005</td>
</tr>
</tbody>
</table>

Steel plates of 3.5 mm thickness were obtained, from which samples of 3.5 mm x 10 mm x 12 mm were machined to obtain the welding nuggets. The autogenous GTAW process was then employed using the following process parameters: i) constant electric arc length of 1 mm, ii) current intensity of 20 and 25 A, iii) potential difference of 11 and 13 V, iv) holding time of 3 s, v) heat input of 660 and 975 J, vi) direct polarity, vii) electrode of tungsten (3.1 mm), viii) Ar as shielding gas, and ix) non external clamping. The operative parameters were selected in previous bead on plate and weld nuggets experiments in the LD steel in as-cast condition with different heat input, varying mainly the current intensity.

The microstructure of the weld nuggets was revealed as follows: firstly, the primary roughing was carried out with sandpaper from 180 to 2000 grit, and then polishing with diamond paste from 9 until 0.1 µm and cleaning in an ultrasonic bath with ethanol, and a subsequent chemical attack with 5% HNO₃ and 95% ethanol solution (50 ml). The average grain size was determined by the linear intersection method according to ASTM E 112 - 96 standard [11]. Linear elemental chemical analysis was performed in the FZ with a JEOL 6400 scanning electron microscope equipped with a thermionic tungsten filament and energy dispersive spectroscopy (EDS) detector. The structural characterization by X-ray diffraction (XRD) was done in a D8 Advance Bruker diffractometer.
(detection limit of 1%, and 0.5 % in high-symmetry materials) with the following operating conditions: Cu Kα radiation, angle range (2θ) from 20 to 120°, step 0.03° and time of 3 s per step. Three microhardness profiles (one on the surface, and two on the cross-section) were performed using the Vickers method applying a load of 500 g for 15 s according to ASTM E 384-17 standard [12]. The transverse hardness profiles were made at 25 and 50 μm from the surface. All hardness profiles were performed along a total length of 5000 μm from the center of the weld nugget with a distance between indentations of 150 μm.

3. Results and discussion

3.1. FZ and HAZ’s characterization by light optical microscopy (LOM)

Figure 1 shows the LOM micrographs of the cross sections of weld nuggets using 20 and 25 A of current intensity. It is apparent that different zones were generated by the current welding process: a FZ produced by high focaled temperatures, and two different HAZ’s associated with the heat extraction through the interior of the samples (the two different HAZ's are better observed in Figure 2, as commented later). It is worth noting that only the LD-NM steel is showing hot cracking in the FZ irrespective of the current intensity used, while the other two steels displayed a lesser degree of affectation.

Figure 1. LOM micrographs of the cross sections of weld nuggets. a, b, c) 20 A and 3 s, and d, e, f) 25 A and 3 s.
According to Borland [13] hot cracking is caused due to the inability of the solidified WM to hold stresses in a critical temperature range during cooling. This can be the case during differential solidification, i.e., when the WM is melted and the solidification begin, a certain microstructure solidifies first and other later, according to the equilibrium phases diagram. This is found in steels with a large liquid-solid interval. It is important to note that cracking phenomenon is more important in butt or lap joint conditions where external clamping forces are involved, which influence the cracking mechanism. However, in this work welding nuggets were made without clamping, and therefore the effect of this force on the cracking is negligible. The crystalline structure of δ-ferrite is BCC and γ-austenite is FCC, so, when the cooling process of steel causes a transformation from liquid phase to δ-ferrite and then to austenite, these contractions may cause defects such as cracks and flaws. This type of cracks is characterized by occurring at high temperatures between approximately 800 and 900 °C. On the other hand, impurities like P and S can also cause hot cracking. For instance, Suutala et al. [14] have reported that steels that undergo austenitic solidification are prone to hot cracking when the sum of the P and S content is above approximately 0.02 wt.%. As well, recent works [15] have reported that S is more harmful than P to hot cracking, due to the stronger segregation of S. It has also been reported that impurity-forming elements such as S and P and alloying elements such as B, Si, Ti and Nb in low percentages, promote hot cracking, particularly in austenitic steels [16], since they can segregate to grain boundaries and even remain in liquid state in the later stages of solidification. According to Shankar et al. [17] the cracking susceptibility decrease due to the addition of Ti/B, can be caused by stabilization of the ferritic field at high temperatures, a phase more resistant than austenite. This would explain the absence of hot cracking in the present LD-Ti/B steel.

High-Mn austenitic steels are sensitive to hot cracking due to the wide range of solidification temperature and the phase transformations undergone in this range. It should also be noted that, austenite compared to ferrite, is characterized by a lower thermal conductivity and a higher coefficient of thermal expansion. The combination of a large solidification temperature range and high coefficients of thermal expansion plays an important role in cracking sensitivity [18]. Solidification cracking is generated along the grain boundary in the final solidification stage during welding, which depends on the segregation of solute elements at the grain boundaries or the concentration of impurities. In the present case, the greater presence of cracks in the LD-NM steel may also be associated to high C content, which produces a wide range of solidification temperatures. In the case of LD-REM steel, although its C content is the same as in LD-NM steel, it is important to take into account the presence of Ce and La. It is well-known that Ce or La addition to steel eliminates the segregation at grain boundaries of elements such as S or P, as well as the associated embrittlement [19]. On the other hand, the addition of Ce and La or other rare earths

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metals have been found to be highly effective binding P and S as stable compounds [20]. Rare earths reduce the possibility of formation of S or P rich particles that act as stress concentrators in FZ, which can initiate cracks. This would also explain the lack of hot cracking in the LD-REM steel.

The solidification crack morphology is related to the liquid fraction along the grain boundary and its condition in the final solidification stage, where the presence of Al promotes the presence of δ-ferrite and contributes the increase of grain boundary by refining grains and dispersing the remaining liquid fraction [21]. The detrimental effects of S and P have led to the addition of other elements that counteract their effects. Mn additions are known to decrease cracking in high S content steels by forming MnS eutectics with a higher melting point and less damaging than FeS [17]. On the other hand, it has been found that N has negative effects regarding cracking in weld metals. N gets into the system from the atmosphere during GTAW process when a poor gas protection may occur. N has a reducing effect on δ-ferrite, thus causing greater sensitivity to cracking, as well as promoting the segregation of P and S impurities towards the grain boundaries [17]. Similarly, harmful AlN formed at very high temperatures from the steel manufacturing may remain present during and after the welding procedure. On the other hand, some beneficial effects of the ferritic-austenitic (FA) solidification mode, such as a smaller solidification temperature range, can lead to less susceptibility to solidification cracking in steels with higher Al contents [17].

Figure 2 show different areas produced in the welding nuggets when observed in the same direction than the electrode positioning. Here, the FZ can be easily noticed, i.e., the area where the fusion is achieved. Then a first HAZ (named HAZ 1) can be appreciated, corresponding to the area adjacent to the FZ where a grain refinement occurred. Finally, a second HAZ (named HAZ 2) is observed, a zone where the grain size remains equal to the one of the base material or in some cases slightly larger. It will be shown later that, in the HAZ 2, there is a significant increase in hardness due to kappa phase precipitation, reason why even though the grain size remains practically constant, heat extraction is promoting aging.
Figure 2. LOM and SEM micrographs of the surface of weld nuggets. a, b, c) 20 A and 3 s, and d, e, f) 25 A and 3 s.

The HAZ 1 is produced by a phase transformation of γ-austenite to δ-ferrite during heating. When high cooling rates occur later, the alloy get into the austenitic field again, where nucleation of new austenitic grains takes place, but because they do not have enough time to grow, a fine grain is produced. In the HAZ 2, no phase transformation occurs because the only possible change is a slight grain growth due to the temperatures reached. **Figure 3** shows the average grain size obtained in both HAZ’s for each composition and each welding condition.

![Graph showing average grain size](image)

Figure 3. Average grain size in the heat-affected zones. a) 20 A and 3 s, and b) 25 A and 3 s.

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As the solidification process occurs, there is a rejection of solute towards areas far from the center of the weld button, forming a concentric circle that delimits the FZ. It will be shown later that this is a very narrow ring rich in carbides, nitrides and sulfides. It is important to note that the studied LD steels contain microalloying elements and high C, which also contributes to the formation of TiC and the precipitation of kappa carbide. The HAZ 1 exhibits a very similar fine grain size for the three LD steels studied, while the HAZ 2 shows an important increase in grain size. It is worth mentioning that neither HAZ 1 nor HAZ 2 reached a temperature too high to allow phase transformations, as discussed below.

3.2. Second phases and kappa carbide precipitation by scanning electron microscopy (SEM)  
Figure 4 shows the elemental chemical mapping of a particle in the LD-REM steel in the 25 A condition. One can notice a high concentration of Ce and La located on precipitates as AlN particles, which facilitates their nucleation. Many of these particles were found in the FZ, among columnar dendrites.
**Figure 4.** Elemental distribution maps of LD-REM steel particle in 25 A condition. a) AlN-(Ce,La)O complex particle, b) elemental chemical mapping, c) kα Ce pattern, d) kα La pattern, e) kα Al pattern, f) kα N pattern.

It is clear that particles may nucleate above other impurities as MnS or AlN, hence, the presence of nitrides or sulfides act as a nuclei for rare earth particles requiring less energy due to heterogeneous nucleation [6]. The resulting particle according to its stoichiometry is Ce and La oxide, whose high concentration causes a decrease in its content in solid solution, reducing the purpose for which it was added to the alloy. **Figure 5a** shows a small group of particles composed, among other elements, of Mn, Al, Fe, Ti and C, found in the LD-Ti/B steel.

![Figure 5](image.png)

**Figure 5.** Particles found in the LD-Ti/B steel, a) TiC, b) AlN-TiC.

The size of the TiC particles is approximately 100 nm. These particles strongly contribute to improve the mechanical properties by a precipitation hardening mechanism. However, they can also promote stress concentration in the solidified metal zone, increasing hot cracking susceptibility. Matsuda et al. [20] studied fully austenitic steels which content less than 0.05 wt.% Ti, which reduced susceptibility of cracking. They also showed that higher percentages increase the possibility of formation of low-melting point eutectics that increase cracking. On the other hand, **Figure 5b** shows the presence of larger complex particles, with similar percentages of Ti, which are nucleating in AlN particles.

**Figures 6a** and **b** show the FZ in the LD-NM steel at the 20 A condition. It is possible to observe a severe cracking affectation in the FZ. These cracks spread along the columnar dendritic zone, oriented to the zone of maximum heat extraction. As already mentioned, the most severe cracking was observed in the LD-NM steel while the LD-REM steel shows less sensitivity to hot cracking.
but greater than LD-Ti/B steel. It is very important to highlight that LD-Ti/B steel shows the highest resistance to cracking of three studied LD steels. An explanation of this behavior could be partly associated with the higher N content in both LD-NM and LD-REM steels, which forms nitrides capable of stress concentration and forming defects.

**Figure 6.** Localized cracking in the FZ in the LD-NM 20A steel nuggets, a) cross-sectional FZ, b) crack spread along interdendritic spaces, c) chemical line scan, d) EDS from line scan.

Figures 6c and d show the variation of chemical elements in different areas of the crack. It can be seen how Mn varies across the crack. At the edge there is a high level of Mn, but inside, it falls abruptly. Al increases at the edges and could be associated with N due to its high affinity to form nitrides. O and C increase importantly within the crack, which can be associated to the presence of oxides and carbides. It would also promote a greater range of localized solidification temperature, which allows the presence of liquid phases in the final stage of solidification. Elements such as P, S and N at low contents show slight increases in the edges, which could indicate the existence of interdendritic segregation and the possible formation of harmful metallic sulfides and phosphides. Also, N promotes a lower stability of the δ-ferrite at high temperatures, which makes the area more susceptible to cracking [17].

**Figure 7** shows micrographs of different zones generated in the weld nugget of the LD-NM steel at 25 A.
Figure 7. Microstructures produced in the weld nugget of the LD-NM steel at 25 A. a) FZ, b) precipitation zone, c) HAZ 1, d) HAZ 2.

**Figure 7a** shows a dendritic microstructure of the FZ with some embedded carbides. **Figure 7b** shows the thin precipitation zone, formed mainly by a strip of particles rich in Al, such as AlN and some other impurities as MnS. This zone is the "belt" formed between FZ and HAZ 1, where AlN precipitation mainly occurred, reaching a high hardness value. **Figure 7c** shows the HAZ 1 where the precipitation of Al-rich phases was dispersed in small quantity. **Figure 7d** shows the HAZ 2 where a large number of second phases, nitrides and carbides are observed. Each of these zones are responsible for producing important variations in hardness as will be described in section 3.4.

**Figure 8** shows the modulated structure of the kappa carbide of the three studied LD steels in the welding condition of 25 A. This microstructure was found in the HAZ 2, that is, the zone of the greatest hardness and farthest from the center of the weld nugget.
Figure 8. Kappa carbide in modular structure in HAZ 2 in cross-sectional view of weld nugget, a) LD-NM steel, b) LD-REM steel and c) LD-Ti/B steel.

The modulated structure was enhanced by the presence of AlN in the same area. According to Sato et al. [22] the modulated structures in the Fe-Mn-Al-C alloys are considered to be formed by the fluctuation of the chemical composition in the austenitic matrix, acting as an intermediate stage of the decomposition of austenite ($\gamma$) → austenite ($\gamma$) + kappa carbide in a particular temperature range and creating a periodic arrangement in the austenitic matrix. Figure 8a shows the LD-NM steel with the most density of nucleated kappa phase compared to the LD-Ti/B steel (Figure 8c). This is agrees with the prediction of the equilibrium phase diagrams shown in Figure 9. According to JMatPro, under equilibrium conditions, kappa phase should be higher for the LD-NM steel than LD-Ti/B steel.
Figure 9. Phase diagram under equilibrium conditions calculated by JMatPro® for a) LD-NM 0-100 wt.%, b) LD-NM 0-0.05 wt.%, c) LD-Ti/B 0-100 wt.% and d) LD-NM 0-0.05 wt.%. If the cooling process was slow enough, a combination of ferrite and austenite could be observed at room temperature. However, under conditions of fast cooling, the transformation does not have enough time to occur. In other words, under a metastable condition, austenite appears as the main phase at room temperature, with small traces of second-phases.

Figure 10 shows the HAZ 2 of the LD-Ti/B steel. Figures 10a and b show the lines that form a network of small periodic oriented voids, where cuboidal kappa phase particles have been detached by the metallographic preparation.
Figure 10. Modulated structure of kappa phase formed in the HAZ-2 of LD-TiB steel. a) Kappa phase nucleated in grain boundary, b) nanometric voids, c) nanometric particles, and d) B deconvolution.

The same types of marks can be observed within the grain in Figure 10c, which can significantly decrease the tensile strength and ductility of the steel due to the nucleation of intergranular kappa carbides and AlN. Figure 10c shows traces formed by nanometric voids, where intergranular kappa carbides were nucleated and grown. On the other hand, a deconvolution of the spectrum obtained by EDS was carried out in order to corroborate the presence of B in the steel, which is presented in Figure 10d. B was found concentrated in the form of particles or segregated in grain boundary. The presence of large B particles precipitated was not detected, so it is assumed that, these particles have nanometric size, as reported by Mejía et al. [23].

3.3. Phase analysis by X-ray diffraction (XRD)

Figure 11 shows the X-ray diffraction patterns of the studied LD steels. $\gamma$-austenite (111) was found as the main phase, being the higher peak of intensity. Other peaks of lower intensity correspond to $\delta$-ferrite (111) phase, as well as the kappa (111) and $\beta$-Mn phase (510).
Figure 11. X-ray diffraction patterns in the welding nuggets of the studied LD steels. a) 20 A, and b) 25 A.

It is important to note that after the precipitation of the kappa carbides within the austenitic matrix, the formation of \( \beta \)-Mn usually occurs due to over-aging [24] by the permanence of the steel at high temperatures. In this work it is evident that during the generation of the welding nuggets, favorable conditions of time and temperature are presented for the formation of kappa carbides and \( \beta \)-Mn, despite not presenting an isothermal condition, particularly in areas where the cooling rate is low and the holding time is longer, e.g., HAZ 2 regions. The X-ray results indicate the presence of \( \beta \)-Mn in a very small quantity; however, it was not possible to observe it in the micrographs by SEM. The \( \beta \)-Mn phase is totally detrimental to the mechanical properties because it causes brittle fracture and reduces impact energy [25]. This phase is produced by the lack of C in the surrounding austenite after the kappa carbide precipitation. The enrichment of Mn of a particular zone in austenite causes the formation of a Mn-rich phase. The formation of a Mn-rich phase significantly decreases the Mn content of the adjacent austenite phase. Consequently, the austenite phase contiguous to the Mn-\( \beta \) phase is depleted of Mn and the Mn-poor region readily transforms to ferrite phase. As a result, a
\( \gamma \rightarrow k + \alpha + \text{Mn-}\beta \) transition occurs. On the other hand, the presence of \( \delta \)-ferrite in the diffraction patterns indicates a FA (ferritic-austenitic) solidification mode. According to Lee et al. [10] the FA mode (\( L \rightarrow L + \delta \rightarrow L + \delta + \gamma \rightarrow \delta + \gamma \)) can be modified depending on the Al content in the alloy, resulting in a fully austenitic mode at lower Al content. The high content of Al also promotes the formation of ferrite. Joeng et al. [26] detected small amounts of ferrite in steels containing 10 and 11 wt.% Al by X-ray diffraction and transmission electron microscopy (TEM) studies. Chou and Lee [27] found that when the alloy has a residual ferrite its resistance to hot cracking increases, and on the contrary, when the alloy is fully austenitic lower hot cracking resistance is noticed, which is associated with its lower thermal conductivity and a higher thermal expansion.

3.4 Hardness profiles by Vickers microhardness (HV) technique.

Figure 12 shows the surface and transverse hardness profiles under the two welding conditions for the studied LD steels. Figures 12a and b show the LD-NM steel at the 20 and 25 A welding conditions, in which a similar behavior is observed, that is, a stable hardness of 225 HV in the FZ, after a slight increase to 250 HV in the HAZ 1, followed by a strong hardening in the HAZ 2 which was attributed to the kappa phase precipitation. In turn, the transverse profiles indicate the same trend even though the hardness values are slightly different. The hardness in the HAZ 1 is dominated by its fine grain size, which generates higher hardness values compared to the FZ. The sharp increase in hardness of the HAZ 2 is also associated to the precipitation of kappa carbides, as shown in Figure 8.
Figure 12. Surface and transverse microhardness profiles of the welding nuggets for the studied steel. a) LD-NM steel 20 A, b) LD-NM steel 25 A, c) LD-REM steel 20 A, d) LD-REM steel 25 A, e) LD-Ti/B steel 20 A, f) LD-Ti/B steel 25 A.

The kappa phase was found dispersed throughout the HAZ 2 and in lower amount in HAZ 1. Figures 12c and d show the same comparison between hardness profiles for the LD-REM steel. It shows the same behavior in the three profiles, that is, low hardness in the FZ associated to the dendritic structure, then, a slight increase at the HAZ 1, dominated by a fine grain size and the beginning of kappa phase precipitation, and an important hardening at the HAZ 2 due to kappa.
phase precipitation. Figures 12e and f show the hardness behavior of the three hardness profiles of the LD-Ti/B steel. It is also observed that, in the FZ, the hardness values are similar in the three profiles because the FZ presents the same dendritic structure in the molten zone. It is noteworthy that differences in grain size were observed in the HAZ 1 both in the superficial and transverse sections.

As a general trend the highest hardness values are presented in the HAZ 2 at 25 A condition on the surface, which is attributed to preferential energy conditions (temperature and time) to promote precipitation of kappa carbides in this region. On the other hand, the lower hardness obtained from the second cross-sectional profile in both 20 and 25 A welding conditions is also evident, which indicates less thermal affectation in the sample as depth below the surface increases. A non-uniform heat distribution is observed according to the variations in grain size and hardness profiles.

Kim et al. [28] found a higher hardening in the HAZ than in the FZ and MB in austenitic LD steels due to the temperature ranges of kappa carbides (500-900 °C) precipitation. Those areas far from the FZ reached hardness peaks of 330 HV. This is attributed to the short holding time in the ranges of formation of kappa carbides (fast heating and cooling). Kim et al. [28] simulated the welding thermal cycles in austenitic LD steel according to the Rosenthal [29] heat equation, and concluded that the holding time in the formation range is crucial for the formation of kappa carbides.

On the other hand, Moon et al. [30] reported that LD steels without microalloying elements such as Nb and V, which have similar effects in grain refining such as Ti, exhibited greater precipitation of kappa carbides than microalloyed steels due to the partition of C to the precipitation of metallic and kappa carbides, which explains the higher hardness values observed in the HAZ 2 both in 20 and 25 A conditions. Jeong et al. [26] carried out hardness tests in the HAZ in Fe-31.5Mn-8.73Al-0.8C steel. They found an important evolution of hardness as the Al content increases, directly related to the formation of kappa carbides. Jeong et al. [31] pointed out the importance of the permanence time at high temperatures of the kappa phase. They found that if kappa phase is already present and it is subjected to fast heating as a result of a welding process, then the HAZ could undergo the greatest hardening as it remains at high temperatures (in the range of formation of additional kappa carbides). This phenomenon has more influence than in the FZ, where higher peak temperatures are reached but where cooling is very fast, avoiding the formation of carbides. Additionally, the previously existing carbides are able to dissolve due to the high temperature.

Although an aging heat treatment was not directly performed, the focused temperatures reached in any welding process are very high and considering the air cooling, the weld nuggets can easily fall into the kappa carbide forming temperature range, with a maximum precipitation reached between 500 and 600 °C. The temperatures reached during the welding process are enough to promote the precipitation of kappa carbides in the entire sample. However, the HAZ 1 is a region where cooling
rates are faster, hence, a lower precipitation of kappa carbide is reached than in the HAZ 2. James [32] reported that with increasing C and Al content in a Fe-Al system, a hardening effect is promoted, which produced the highest hardness in the range from 500 to 550 °C. Bentley et al. [33] demonstrated that kappa carbide precipitation in austenitic Fe-32Mn-11Al-0.8C alloy, was the result of a decomposition of the austenitic phase through fluctuation of solute. They discovered that the mechanism is a spinodal decomposition and rapid ordering during cooling. In this way, two types of kappa carbides have been reported in austenitic Fe-Mn-Al-C steels aged between temperatures of 500 and 900 °C: intragranular kappa’ carbides and intergranular kappa* carbides. Intragranular kappa’ carbides are fine and cause age hardening and this significantly increases the yield strength [34], while intergranular kappa* carbides are thicker and cause a serious loss of ductility [35].

Conclusions
1. Weld nuggets of LD steels showed three well-defined zones generated by thermal cycling during the welding process: i) a FZ of dendritic structure, ii) a HAZ of fine equiaxed grain, and iii) a second HAZ of coarse equiaxed grain with high precipitation of kappa carbides.
2. Weld nuggets of the LD-NM steel exhibited the highest level of hot cracking, which is attributed to higher N and C contents. On the other hand, the microalloyed LD steels show a considerable decrease in hot cracking, particularly the LD-Ti/B steel.
3. Weld nuggets presented an important presence of AlN and MnS particles, which act as nuclei of precipitated particles of microalloying elements. The LD-Ti/B steel shows nanometric particles of TiC and B. The LD-REM steel exhibits complex particles of (Ce, La)O combined with AlN and MnS.
4. The HAZ 2 farthest from the center of the weld nugget produced a large amount of kappa phase with a modulated structure, which strongly increases the hardness.
5. X-ray diffraction studies demonstrated that γ-austenite is the main phase in the weld nuggets, although also other minor phases were detected such as δ-ferrite, kappa carbides and undesirable β-Mn.
6. Weld nuggets exhibited significant variations in hardness. The FZ exhibited the lowest hardness values (≈ 225 HV), which is associated with a dendritic structure. The HAZ 1 showed a generalized increase in hardness (250-300 HV) due to grain refinement and the beginning of kappa phase formation. Finally, the HAZ 2 displayed the highest hardness values (up to 450 HV) due to strong precipitation of kappa carbide with a modulated structure.

Acknowledgments:
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Authors would like to thank the National Council on Science and Technology (CONACyT-México) for the support during the project CB-2012-01-0177572. The present research project was also supported by the Coordinación de la Investigación Científica-UMSNH (México) (CIC-1.8). C. Coronado’s studies were sponsored by the National Council on Science and Technology (CONACyT-México), N.B. 582985. J.M. Cabrera thanks the financial support by CONACyT which made possible his sabbatical leave at UMSNH.

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Herein, the metallographic/structural/mechanical changes of weld nuggets in Fe-Mn-Al-C low-density steels microalloyed with Ti/B and Ce/La by GTAW process are studied. Weld nuggets show three well-defined zones: FZ, HAZ-1 and HAZ-2, where γ-austenite is the main phase. Microalloyed low-density steels show a significant decrease in hot-cracking. HAZ-2 exhibits the highest hardness values (≈ 450 HV) due to precipitation of κ-carbide.