INVESTIGATION OF ACICULAR FERRITE STRUCTURE AND PROPERTIES OF C-Mn-Al-Ti-N STEELS

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Abstract
Low carbon-manganese wrought steels with addition of Ti-Al-N have been treated to obtain acicular ferrite structure. The microstructure of fine acicular ferrite nucleated intragranularly at Ti(C,N)+AlN and Ti(C,N)+AlN+MeS inclusions have showed higher strength and good toughness at low temperatures.

1. Introduction
As it is well known, highly organized microstructures can often be found in steels; for example, ferrite plates frequently grow in the form of packets (bainite or martensite) containing parallel plates, which are in the same crystallographic orientation. This can be harmful to mechanical properties because cleavage cracks can propagate readily across the packets. \[1\]

Acicular ferrite (intragranular bainite) structure is far from being organized and can be better described as chaotic. The plates of acicular ferrite nucleate heterogeneously on uniformly dispersed fine non-metallic inclusions and radiate in many different directions from these point nucleation sites. Propagating cracks are then deflected on each encounter with a differently oriented acicular ferrite plate. This gives rise to superior mechanical properties, especially toughness. Inclusions promote intragranular nucleation of acicular ferrite plates and hence improve toughness without comprising strength. But non-metallic inclusions also are responsible for the nucleation of voids during ductile fracture or cleavage cracks during brittle fracture. The inclusions microstructure is particularly important in this respect. Most of investigators conclude that refractory oxides (especially Ti\textsubscript{x}O\textsubscript{y}) are most effective nucleating sites during intragranular inoculation. At the same time for nitrides (AlN, TiN) and carbonitrides (Ti(C,N)) controversial data exists. Some of investigators \[2; 3; 4\] have not found any significant influence of nitrogen additions on bainite transformation. At the same time other investigators \[5; 6\] have concluded that nitrogen addition to liquid steel have significant influence on bainite transformation due to nitrides and carbonitrides formation in steel.

As it is known firstly acicular ferrite was observed at arc-weld seam and heat-affected zone (HAZ). Typically bainite transformation at arc-weld deposit occurred at quasi-isothermal conditions. Most of investigators use isothermal treatment to obtain acicular ferrite structure \[7\]. This approach is difficultly realized at industry practice. Moreover allotriomorphic ferrite net along the austenite grain surfaces can be harmful for mechanical properties.

Most of works, devoted to acicular ferrite properties investigations, have showed increase both strength and toughness due to interlocking nature of acicular ferrite structure. But some investigators have not found significant increasing of toughness in compare with packet bainite structure \[8\].

The purpose of the present work is to characterize acicular ferrite nucleation ability of Al-Ti-N alloying system and compare properties of acicular ferrite obtained in a C-Mn-Al-Ti-N steels with properties of other steels with similar alloying system.
2. Experimental Technique
Two 20 tons ingots of low carbon-manganese steel inoculated by Al-Ti-N were melted in electric furnace. Chemical composition of investigated steels is presented in Table 1.

Table 1. Chemical composition of the investigated steels

<table>
<thead>
<tr>
<th>Steels</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>P</th>
<th>S</th>
<th>Cr</th>
<th>Ni</th>
<th>Cu</th>
<th>Al</th>
<th>Ti</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td>Steel 1</td>
<td>0.19</td>
<td>0.63</td>
<td>0.28</td>
<td>0.015</td>
<td>0.014</td>
<td>0.18</td>
<td>0.13</td>
<td>0.20</td>
<td>0.029</td>
<td>0.003</td>
<td>0.019</td>
</tr>
<tr>
<td>Steel 2</td>
<td>0.20</td>
<td>0.71</td>
<td>0.3</td>
<td>0.016</td>
<td>0.010</td>
<td>0.22</td>
<td>0.13</td>
<td>0.18</td>
<td>0.028</td>
<td>0.016</td>
<td>0.018</td>
</tr>
</tbody>
</table>

Ingots were rolled into 60 mm square semi-finished product and then specimens with thickness up to 12 mm were cut off. Specimens were cut as along direction of rolling as across direction of rolling. Specimens were subjected to austenitizing at 1150°C for 30 minutes, and then cooled to room temperature in oil. The impact tests with U-notch specimens machined in accordance with GOST (ASTM) were made over a temperature range from +20°C to –80°C in order to determine the impact toughness of the steels with acicular ferrite structure. Specimens of 5 mm in gauge diameter and 25 mm in gauge length were used to determine the tensile properties. The microstructures of the specimens were examined by optical microscopy after polished and etched in 4% Nital. For TEM investigations, we used extraction replicas for examination of inclusions morphology. Those have been made using two 200 kV transmission electron microscopes: Jeol 200CX for conventional imaging and Jeol 2010F with field emission gun for high-resolution imaging and nanoanalysis. Last one was equipped with an Energy Dispersive X-ray (EDX) Oxford Instruments device. SEM investigations have been made using Jeol 840A LGS scanning electron microscope.

3. Results and Discussion

3.1. Microstructure
In most of works devoted to acicular ferrite structure, it has been obtained with isothermal treatments. Sometimes short intermediate isothermal treatments in order to make inert austenite grain boundary nucleating sites have been used. It leads to formation of thin layer of inert allotriomorphic ferrite at the austenite grain surfaces. In this case morphology of bainite changes from granular to intragranular (i.e. acicular ferrite structure forms). Formation of thin layer of inert allotriomorphic ferrite at the austenite grain surfaces leads to decreasing of mechanical properties since cracks can propagate easily through allotriomorphic ferrite net. Moreover short isothermal treatments (up to 1 minute) are stubborn task for industry.

In this work continuous cooling on purpose to obtain predominantly acicular ferrite structure have been used. Cooling rate has been adjusted to provide bainitic transformation during continuous cooling. Microstructure of investigated steels after bainitic transformation is showed at Fig.1.

Fig. 1. Microstructure of investigated steels after continuous cooling (mixture of acicular ferrite and packet bainite), light microscopy.
Microstructure consists of intragranular bainite (i.e. acicular ferrite) nucleated at non-metallic inclusions inside austenite grains and bainite nucleated at austenite grain boundaries. Apparently microstructure contains mixture of intragranularly nucleated bainite (i.e. acicular ferrite) and packet bainite. It allows expecting higher strength and ductility compares to steels with mixture of acicular ferrite and allotriomorphic ferrite. Obviously, a continuous cooling tends to leave bulky untransformed austenite into steel 1 and 2. This residual austenite could transform further into martensite or could become retained. Martensite is thought to contribute to the increase in strength of steel 1 and 2, although it can be detrimental to toughness.

In steels 1 and 2 (with addition of Al,Ti and N), Ti(C,N)$_{FCC}$ and AlN$_{HCP}$ inclusions have been most commonly observed (Fig. 2 and 3).

![Fig. 2 Titanium carbonitride Ti(C,N)$_{FCC}$ in steel 1 (2a) and EDX spectra on this particle (2b)](image)

![Fig. 3 Aluminium nitride AlN$_{HCP}$ in steel 1 observed on TEM extraction replica (3a) and EDX spectra of this particle (3b)](image)

Also complex particles like AlN+Ti(C,N) (Fig. 4) and AlN+Ti(C,N)+MnS (Fig 5) have been frequently observed. The presence of MnS is frequently observed as the MeS phase, and less frequently we notice CuS.
Fig. 4: Complex particle AlN$_{HCP}$+Ti(C,N)$_{FCC}$ TEM extraction replica (4a), showing the two structures joined by a dense plane, which one is the $\{111\}$ plane in Ti(C,N) and $\{0002\}$ one in AlN. The zone axis is the $<010>$ in the AlN structure. The composition of this complex particle is checked with EDS spot analysis on the Ti(C,N)$_{FCC}$ part (4b) and the AlN$_{HCP}$ part (4c).

Fig. 5: Complex particle Ti(C,N)+AlN+MnS(CuS) TEM extraction replica (5a); Ti(C,N) EDX spectrum (5b); AlN EDX spectra (5c and 5d); MnS(CuS) EDX spectrum (5e); EDX spectrum acquired on the replica film (5f).
Effectiveness of different inclusions as nucleation sites for acicular ferrite plates have been investigated. It has been established that complex inclusions of Ti(C,N)+AlN from 500 nm up to 1.5 µm sizes are most effective nucleation sites for acicular ferrite nucleation in investigated steels. One of such complex inclusions is showed in Fig. 6 (b).

![Complex Ti(C,N)+AlN inclusion as nucleation site for several acicular ferrite plates in steel, SEM image in SE mode (6a); EDX spectrum of the concerned particle (6b).](image)

Fig. 6. Complex Ti(C,N)+AlN inclusion as nucleation site for several acicular ferrite plates in steel, SEM image in SE mode (6a); EDX spectrum of the concerned particle (6b).

It should be mentioned that some of complex inclusions are encapsulated by sulphide phase and analysis of core phase is complicated. Obviously as core phases act refractory titanium carbonitrides and aluminium nitrides. It have been proved by other investigators [9].

### 3.2. Mechanical properties

The results of the tensile tests are given in Table 2. Investigated steels 1 and 2 show higher yield strength compared with C-Mn-Ti-V steel with acicular ferrite structure [10].

It should be mentioned that in work [10] steels with much higher carbon (0.35-0.37 wt. %) and manganese (1.45-1.56 wt. %) contents have been investigated (cf. Table 1). Such high strength can be explained by fine acicular ferrite structure and martensite laths presence in microstructure. Though the volume fractions of martensite and retained austenite could not be measured due to very low level. Carbides within the ferrite plates were hardly observed.
Elongation is rather low in case of specimens cut across direction of rolling. Obviously it is due to nitrides and carbonitrides presence in investigated steels.

Table 2, Mechanical properties of steels with acicular ferrite structure

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Microstructure</th>
<th>Yield Strength, MPa</th>
<th>UTS, MPa</th>
<th>Elongation, %</th>
<th>Contraction, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti-V [10]</td>
<td>Acicular ferrite</td>
<td>560-666</td>
<td>N/a</td>
<td>N/a</td>
<td>N/a</td>
</tr>
<tr>
<td>Ti [10]</td>
<td>Acicular ferrite</td>
<td>519</td>
<td>N/a</td>
<td>N/a</td>
<td>N/a</td>
</tr>
</tbody>
</table>

| Al-Ti-N Steel 1 | Acicular ferrite and packet bainite | 589 | 807 | 21,7 | 42,7 |
| Al-Ti-N Steel 2 | Acicular ferrite and packet bainite | 628 | 873 | 21,8 | 38,9 |

| Al-Ti-N Steel 1 | Acicular ferrite and packet bainite | 555 | 764 | 13,5 | 22,8 |
| Al-Ti-N Steel 2 | Acicular ferrite and packet bainite | 665 | 893 | 14   | 28,6 |

The results of the impact tests are given in Fig. 7. Therefore these results seem to confirm that interlocking microstructure is superior to the tempered martensite structure in toughness. But large inclusions and hard microphases could affect the toughness [1].

Fig. 7 Variations in impact toughness of steels 1, 2 and C-Mn-V steel [11] with temperature.
Nevertheless impact toughness of steels with acicular ferrite structure and AlN+Ti(C,N) inclusions is superior to impact toughness of steel with V(C,N) inclusions and tempered martensite structure (Fig.7). It can be explained by interlocking nature of acicular ferrite structure. Impact toughness of steels with acicular ferrite structure can be improved by careful control of inclusions size and contribution.

4. Conclusions
The microstructure of interlocking acicular ferrite nucleated intragranularly on AlN+Ti(C,N) and AlN+Ti(C,N)+MeS inclusions shows high strength and high impact toughness at low temperatures.

Bibliography