Effects of Titanium Addition on Precipitate and Microstructural Control in C–Mn Microalloyed Steels

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(Received on May 24, 2002; accepted in final form on September 11, 2002)

A study was carried out on the effects of Ti in a Ti-lean and a Ti-modified C–Mn microalloyed steels of otherwise comparable compositions. Analyses were carried out by SEM and TEM on steel microstructure, microalloying element precipitates and on non-metallic inclusions. A theoretical support to the experimental data was obtained by thermodynamic analyses aimed at stating phase stability and composition as a function of temperature for the steels investigated. Experimental results and theoretical predictions were combined to cast light on carbide, nitride and sulphide evolution during thermal cycles associated to fabrication route of the steels.

KEY WORDS: HSLA steels; effects of titanium; TEM microstructure; thermodynamic calculation; precipitate stability.

1. Introduction

The properties of microalloyed steels are controlled by the addition of the carbide and nitride forming elements Nb, V, Ti aimed at refining the austenite grain size and the products formed from austenite decomposition. Additional precipitation strengthening effects can also act, provided the precipitate particles are finely dispersed and very small in size. A proper design of the carbonitride precipitation sequence through control of composition, solidification and cooling rate is therefore of great importance and it was the subject of a large number of research works in the last decades.1–7)

During recent years, particular attention was dedicated to optimisation of solidification conditions and avoidance of transversal and edge cracking, especially during continuous casting. From hot tensile testing and from industrial practice on microalloyed steel, it was established that a loss of ductility is often encountered at temperatures approximately ranging from 750 to 950°C. This ductility through becomes of particular concern when it occurs within the straightening sectors along the continuous casting strand. Loss of hot ductility is always related to intergranular failure of the steel at the γ grain boundaries and can be accounted for by two possible microstructural mechanisms. Cracks are readily formed by grain boundary sliding when the boundaries are decorated with microalloying precipitates or manganese sulphide inclusions. Alternatively, transformation controlled cracking can occur due to formation of thin films of soft ferrite, promoting strain localisation at grain boundaries.8–11)

Microalloying with Ti is a well known method to reduce hot cracking susceptibility and a large number of papers were published on its effects in microalloyed steels. It is now established that Ti, being a powerful nitride former, acts by removing most of the N from solid solution at very high temperatures, thus preventing subsequent grain boundary embrittlement to occur due to formation of nitrides with other microalloying elements.1,5,10,11) A debate was raised in literature on optimal Ti : N ratio as a function of other alloying elements and on the actual mechanisms acting in reducing hot cracking susceptibility.8,9,11,12) It is now widely accepted that optimal Ti : N ratio should lie on the hyperstoichiometric side, at values ranging from 4 to 10, depending on actual steel composition.

When focussing mainly on austenite grain size control during hot working or welding, different optimal Ti : N values were found. Within this context, an interesting research work by Medina and co-workers13,14) allowed to demonstrate that optimal precipitation for grain size control in C-Mn-Al, Ti-bearing steels corresponded to hypostoichiometric compositions at Ti : N values close to 2.

In addition to fabrication properties, Ti can also impart a variety of important advantages on final products such as improved cold formability and toughness isotropy. The above properties are mainly due to the preferred formation of titanium carbosulphide (Ti$_4$C$_2$S$_2$) instead of manganese sulphide (MnS) inclusions. The former being harder than the MnS at elevated temperatures, thus preventing the formation of elongated stringers of inclusions in hot rolled products.15) It was also proposed that coarse Ti$_4$C$_2$S$_2$ precipitation at high temperature would slightly improve hot ductility by removing sulphur atoms from matrix, otherwise available for austenite grain boundary precipitation.11) A recent research work on final properties of Ti-bearing steel products further showed that coarse TiN particles promoted...
by a extremely high Ti:N ratio are detrimental to fracture toughness behaviour.  

In spite of the importance of combined precipitate and inclusion control in Ti-bearing microalloyed steels, little information is currently available in the literature on this issue. In the present paper the effects of Ti addition on precipitation and microstructural behaviour of a microalloyed steel grade was studied with the aim of improving the knowledge on interaction between titanium precipitates and inclusions formed at high temperatures and other phases that precipitate in the austenite matrix at lower temperatures, during cooling and heat treating.

2. Materials and Experimental Procedures

A C–Mn steel grade microalloyed with Nb and V was investigated both in a standard heat, produced according to established practice, and in a modified version, alloyed with 0.14% Ti. The compositions of the heats investigated are given in Table 1.

The two heats were produced by a conventional industrial route, consisting in melting selected scarp in an electric arc furnace, refining the melt in ladle furnace and continuously casting the steel in a curved four-strand machine to produce billets having a diameter of 145 mm. After reheating, hot rolling to form seamless pipes was carried out at temperatures ranging from 1300 to 1100°C. The products were then quenched and tempered to achieve the required design properties.

Samples of the heats for the experimental investigations were taken at the end of the manufacturing process whereas specific data on manufacturing practice (chemical analyses and temperatures during refining and casting, amount of reduction and temperature of rolling during pipe forming, heat treatment temperatures) were collected from manufacturer database to evaluate the thermo-mechanical cycles experienced by the steels.

Specimens for optical and scanning electron microscope (SEM) metallography were prepared by standard grinding and polishing and by etching in 2 vol% Nital reagent or in saturated aqueous picric acid solution. Particular attention was paid to inclusion morphology and composition, as detectable by energy dispersive X-ray spectroscopy (EDS) performed during SEM analyses on polished and unetched samples. The average chemical composition of each inclusion type was estimated by performing point analyses on several distinct inclusions and then averaging the results.

Transmission electron microscopy (TEM) studies were carried out on thin foils perforated by electropolishing using a 10% perchloric acid solution in 2-butoxyethanol. Also in this case, particular attention was given to microalloying precipitates and tiny inclusions that were revealed in their composition and crystal structure by EDS and selected area electron diffraction (SAD), respectively.

Room temperature tensile strength was measured on cylindrical specimens having a diameter of 8 mm and a gauge length of 40 mm, machined longitudinally with respect to pipe axis. The fracture surfaces of the specimens were examined by SEM to reveal fracture topography and to characterise microstructural features promoting fracture.

Finally, theoretical support for discussion of the experimental data was given by estimates of the equilibrium phases to be found in the investigated steel heats as a function of temperature. To this purpose, detailed thermodynamic analyses were carried out using the Thermo-Calc software, selecting the TC–Fe steel database.

3. Results

Representative micrographs of the investigated steels are given in Fig. 1. Both steels featured a small ferrite grain structure with moderate amounts of upper bainite formed during quenching. The Ti-modified steel had a slightly finer structure and a higher fraction of bainite.

TEM images of the precipitates found in the two steels are given in Figs. 2 and 3 together with the corresponding EDS spectra. The standard Nb–V microalloyed steel (Fig. 2) showed an extremely fine dispersion of globular (Nb, V)CN precipitates along with occasional coarse cuboids rich in Ti and N. Nb and V were also present in these coarse precipitates, although in lower amount. The addition of Ti to the above steel led to marked modification of the microprecipitate evolution (Fig. 3). The amount of fine (Nb, V)CN precipitates significantly reduced and the coarse TiN particles, still containing Nb and V, predominated.

Table 1. Chemical compositions of the steels investigated (mass%).

|          | C  | Mn | N  | P  | Si | Ti | Nb | V  | Al | Cr | Cu |
|----------|----|----|----|----|----|----|----|----|----|----|----|
| Standard | 0.11| 1.05| 0.0066| 0.010 | 0.001 | 0.30 | 0.005 | 0.024 | 0.044 | 0.026 | 0.12 |
| Ti-mod.  | 0.11| 1.02| 0.0056| 0.012 | 0.002 | 0.25 | 0.014 | 0.022 | 0.044 | 0.026 | 0.08 | 0.12 |

Fig. 1. SEM micrographs of the investigated steels; (a) standard steel; (b) Ti-modified steel.
Analyses of non-metallic inclusions were carried out on polished and unetched samples by SEM observations and quantitative EDS microprobe analyses. Both steels contained a relatively low amount of small endogenous inclusions, to be considered as residuals of the ladle furnace refining practice. In Tables 2 and 3 a summary of the chemical composition of the inclusions detected in the steels is given. The observations were carried out on several samples by analysing about 25 inclusions for each sample. Consideration of a larger number of inclusions was judged unnecessary due to good repeatability of the results obtained. Globular inclusions having a size ranging from 2 to 5 μm were generally detected in both steels. Irrespective of the steel examined, the inclusions detected by SEM and EDX microprobe analyses, namely those having a size larger than about 2 μm, consisted of calcium aluminates (XCaO · YAl2O3) of different compositions and containing relevant amounts of Mg, combined with (Ca, Mn)S type sulphides, as expected from known steelmaking theory.17,18 Traces of Ti were found also in the above inclusions, in relatively small amounts, in the Ti-modified steel. It is worth noting that light elements such as O, C and N could not be quantitatively detected by the microprobe system adopted for the present investigation. In addition, a fraction of the measured iron content could reasonably be originated by the steel matrix regions surrounding the smallest inclusions analysed.

A rough assessment of the microprecipitate and inclusion effects was obtained by performing tensile tests and fractographic analyses on the broken specimens. Data on the tensile properties of the two investigated steels are given in Table 4. Surprisingly, the tensile results showed that the standard Nb–V microalloyed steel had a slightly higher tensile and yield strength but lower ductility than the Ti-bearing steel. In Fig. 4 the typical aspect of the fracture surface of broken tensile samples is depicted. As expected, both materials fractured in a ductile mode, the larger dimples being generated from the coarser calcium aluminate inclusions previously mentioned. No significant difference in the fracture appearance was noted between the two steels.
To support the experimental data obtained on precipitate formation in the steels, theoretical calculations were performed by using the Thermo-Calc database. By this software, predictions were made on equilibrium phase stability and composition as a function of temperature and alloy composition. Particular emphasis was given to the evolution of precipitates related to titanium and microalloying element effects. Therefore, the temperature range corresponding to solid state transformation (700–1 400°C) was specifically considered. Data on generation of non-metallic inclusions within the liquid phase and on the effect of residual oxygen in the melt will not be described in this investigation.

**Table 2.** Chemical composition (mass%) of the inclusions detected in the investigated standard microalloyed steel.

|       | Al  | Mg  | Ca  | S   | Mn  | Si  | Fe  |
|-------|-----|-----|-----|-----|-----|-----|-----|
| Average | 27.4 | 8.1 | 9.8 | 4.6 | 0.4 | 0.3 | 14.5 |
| Std. dev. | 8.3 | 3.8 | 6.8 | 3.4 | 0.2 | 0.5 | 9.3 |

**Table 3.** Chemical composition (mass%) of the inclusions detected in the investigated Ti-modified microalloyed steel.

|       | Al  | Mg  | Ca  | S   | Mn  | Si  | Fe  | Ti  |
|-------|-----|-----|-----|-----|-----|-----|-----|-----|
| Average | 16.5 | 2.7 | 4.2 | 2.9 | 0.6 | 0.1 | 8.8 | 0.3 |
| Std. dev. | 5.1 | 1.1 | 1.2 | 1.9 | 1.3 | 0.1 | 6.5 | 0.3 |

**Table 4.** Average tensile properties of the steel investigated.

|       | UTS (N/mm²) | YS (N/mm²) | Fracture elong. (%) |
|-------|-------------|-------------|---------------------|
| Standard | 572         | 471         | 25.1                |
| Ti-modified | 566        | 462         | 27.7                |

In Figs. 5 and 6 the amount of the different equilibrium phases to be found in the steels investigated is plotted as a function of temperature. In the graphs, FCC_A1 refers to the γ-iron phase, FCC_A1#2 refers to a carbonitride phase and BCC_A2 refers to the α-iron phase. By comparing the two set of curves, it can be inferred that an increase of the Ti content in the considered microalloyed steel leads to an anticipated formation of the FCC_A1#2 carbonitride phase during cooling (phase stability is encountered during cooling at 1 480°C and at 1 405°C for the Ti-modified and the standard steel, respectively) and to retarded precipitation of AlN with respect to the standard microalloyed steel (840°C versus 1 045°C). In addition, a limited field of Ti₅C₆S₂ carboxsulphide stability is also predicted for the Ti-bearing grade at 1 450–1 500°C.

In Tables 5 and 6, further data are given in terms of com-
positions of the equilibrium phases at 1300, 1000 and 700°C for the standard Nb–V microalloyed steel and the Ti-modified steel, respectively. The phase identification codes match those used in Figs. 5 and 6. It is found that the chemistry of the AlN, MnS, \(\alpha\)-iron and \(\gamma\)-iron phases generally remains unchanged as a function of temperature and steel composition, at least within the ranges here examined. As expected, the only modification predicted in the \(\gamma\)-phase at 700°C accounts for the occurrence of the \(\gamma\) to \(\alpha\) transformation and therefore to the greater concentration of gamma stabilising elements (C, Mn) in the remaining austenite. On the contrary, the carbonitride phase (FCC\_A1\#2) undergoes notable changes in composition during cooling. In both steels, at 1300°C this phase actually represents a titanium nitride containing limited amounts of Nb, V and C. By lowering the temperature, a depletion in Ti and N and an enrichment in Nb, V and C continuously occurs. Eventually, at 700°C, this phase becomes a carbide particularly rich in V and Nb. The above compositional variation predicted by the thermodynamic analysis is a consequence of the continuous mutual solubility of the carbides and nitrides formed by Ti, Nb and V and of the decreasing solubility of the different carbides and nitrides with temperature in the \(\gamma\)-iron.1–5)

4. Discussion

Samples of Ti-lean and Ti-containing microalloyed steels of otherwise comparable compositions taken from standard and experimental production were investigated in this study. Combined experimental and theoretical analyses were carried out to better elucidate the effects of Ti on concurrent precipitation of carbonitrides and possible carbosulphides or other inclusions in final products. When alloying with Ti, a composition corresponding to a Ti : N ratio of 2.5 was selected in order to improve welding performance and final product properties. Although it is not possible to state an ideal Ti and N composition irrespective of manufacturing process and steel composition, in many cases it is reported that a hypo-stoichiometric Ti : N ratio ranging from 1 to 3 provided good results.8,13,14)

Apart from the expected differences in austenite grain growth behaviour during hot forming and welding, readily

Table 5. Composition of the equilibrium phases (mass%) at the indicated temperatures for the standard microalloyed steel.

| Phase composition at 1300°C |
|-----------------------------|
| Al | N  | Cr | Fe | Mn | S  | Nb | C | Ti | V  |
| FCC\_A1 | 0.03 | 0.12 | 98.32 | 1.05 | - | 0.02 | 0.11 | - | 0.04 |
| FCC\_A1\#2 | - | 18.99 | 0.01 | - | - | 8.89 | 2.34 | 67.96 | 1.81 |

Table 6. Composition of the equilibrium phases (mass%) at the indicated temperatures for the Ti-modified microalloyed steel.

| Phase composition at 1300°C |
|-----------------------------|
| Al | N  | Cr | Fe | Mn | S  | Nb | C | Ti | V  |
| FCC\_A1 | 0.03 | 0.08 | 98.44 | 1.02 | - | 0.02 | 0.11 | - | 0.04 |
| FCC\_A1\#2 | - | 18.49 | - | - | 5.17 | 3.16 | 72.20 | 0.97 |
| MnS | - | - | - | - | 0.08 | 62.33 | 36.85 | - | - |

| Phase composition at 1000°C |
|-----------------------------|
| Al | N  | Cr | Fe | Mn | S  | Nb | C | Ti | V  |
| FCC\_A1 | 0.03 | 0.08 | 98.46 | 1.02 | - | 0.02 | 0.09 | - | 0.04 |
| FCC\_A1\#2 | - | 14.52 | - | - | - | 32.42 | 3.99 | 41.81 | 7.29 |
| MnS | - | - | - | - | 0.09 | 63.05 | 36.86 | - | - |

| Phase composition at 700°C |
|-----------------------------|
| Al | N  | Cr | Fe | Mn | S  | Nb | C | Ti | V  |
| FCC\_A1 | 0.02 | 0.06 | 98.75 | 0.90 | - | 0.02 | - | - | 0.01 |
| CEMENTITE | - | 1.70 | 82.59 | 8.59 | - | 6.71 | - | - | 0.41 |
| FCC\_A1 | - | 0.12 | 95.66 | 3.40 | - | - | 0.63 | - | - |
| FCC\_A1\#2 | - | 1.62 | 0.35 | 0.05 | 0.13 | 26.47 | 15.35 | 16.85 | 39.17 |
| MnS | - | - | - | - | 0.09 | 63.14 | 36.86 | - | - |
affected by titanium alloying practice, the two steels showed limited differences in terms of resulting grain structure and mechanical properties at room temperature. In the Ti-modified steel a slightly refined ferritic microstructure containing a small fraction of upper bainite was achieved (Fig. 1). However, the corresponding tensile properties were not consistent with this improved structure over the standard microalloyed steels (Table 4). It is supposed that the slight difference found in the mechanical behaviour of the two steels reflects the modifications induced by Ti addition on microalloying precipitate formation.

As stated in the introduction of the paper, the sequence of carbonate precipitation in microalloyed steels is a widely studied issue. Although the subject is extremely complex and large variations do exist owing to differences in steel composition and thermomechanical treatments, qualitative support for microstructure interpretation is readily supplied by the existing literature. In the present case, the expected formation of high-temperature coarse TiN rich precipitates2-9 was experimentally verified in both steels (Figs. 2 and 3) since residuals of Ti were also present in the standard Nb–V microalloyed steel. It was also experimentally confirmed that when a relatively large amount of TiN rich particles formed at high temperatures, a reduced fraction of (Nb, V)CN could form at lower temperatures, as was the case for the Ti-modified steel.

The present investigation was also extended to the evaluation of inclusion morphology and composition. Little information is available on interaction of Ti with combined oxides and sulphides, typically found as non-metallic inclusions in steels. From studies on steels deoxidised with titanium9 and on Ti-bearing microalloyed steels with higher Ti contents than those of the present research,11,15 it was found that Ti-containing oxides and sulphides can form before solidification and within the solid steel. These can in turn affect the subsequent or concurrent microalloying precipitate evolution. From SEM observations and microprobe analyses, it was found that in the present steels the predominant inclusions were globular calcium aluminates combined with mixed sulphides. Ti weakly interacted with the above inclusions, being occasionally found in small concentrations only in several inclusions. The large relative scatter of Ti concentration within these inclusions does not allow to draw any experimental conclusion on the specific phase formed. Based on Thermo-Calc predictions (Fig. 6), the Ti₃CₓSᵧ carbo-sulphide can potentially form at high temperatures in a small temperature range. From literature it is reported that the preferential precipitation of this phase rather than MnS would slightly improve ductility by removing sulphur from matrix and grain boundaries.11 However, it is the authors opinion that the limited amount of Ti and S allowable in the present steel would not generate appreciable changes in steel behaviour even if the Ti₃CₓSᵧ carbo-sulphide precipitation could be promoted.

The analysis was then focussed on the interpretation of the resulting microprecipitate formation on the basis of thermodynamic calculations. Phase stability was judged an important parameter necessary to evaluate the transformations that the steel underwent during fabrication stages, namely solidification, hot forming, heat treating. From Figs. 5 and 6 it can be inferred that during reheating before hot rolling (1300–1000°C) only the coarse TiN rich precipitates remain undissolved. Formation of the new precipitates on cooling can theoretically occur at the above pre-existing high-temperature nitrides or within the matrix, often by deformation induced nucleation mechanisms. The former is a well documented mechanism and possible evidences of this tendency were also noted in the present investigation. Amongst other features, Fig. 3(b) showed a heterogeneous layer at the edges of the coarse TiN particle suggesting the occurrence of a different phase precipitation.

Contrarily to the purely theoretical predictions obtained from thermodynamic calculations, the TEM micrographs showed that distinct finer carbonitrides could form, thus suggesting that nucleation within the steel matrix could also take place, especially in the standard microalloyed steel where more N was left available for precipitation. In addition, the limited amount of MnS phase theoretically expected in both steels starting from 1200–1300°C, is supposed to nucleate mainly at globular inclusions, according to the data detected from inclusion composition. From previous studies it is known that deleterious precipitation of fine MnS at austenite grain boundary can also occur.11

AIN, which is supposed to precipitate out starting from 1045 and 840°C in the standard and Ti-alloyed steel, respectively, was not observed. Although it cannot be excluded that fine precipitates could have been present in the steels and not observed by the used experimental techniques, it is also known that AIN precipitation is generally very sluggish and appearance of such phase does not occurs until the product [Al]×[N] reaches about 2.5·10⁻⁴. With typical N and Al contents such as those of the steels investigated, AIN is not likely to precipitate.3,9,10

Finally, the thermal treatment stage performed by austenitising at 920°C, quenching and tempering only affects the less stable carbides and nitrides. Here, factors concerned with phase coalescence and non-equilibrium generation of new constituents assume greater importance but their study is beyond the scope of the present investigation.

5. Conclusions

A study was presented on the effects of Ti addition on precipitate and inclusion features in C–Mn steels microalloyed with Nb and V. From samples of Ti-lean and Ti-alloyed steels of otherwise comparable compositions, the following conclusions could be drawn.

1) Based on SEM microscopy, after thermal treatment, the two steels had a predominant ferritic grain structure with limited amounts of upper bainite. The Ti modified steels featured a slightly finer grain structure and a higher amount of bainite.

2) Subsequent TEM analyses focussed on precipitate properties showed that a bimodal distribution of particles was present in the steel structure. The standard Nb–V microalloyed steel showed a fine dispersion of globular Nb(C, N) precipitates along with occasional coarse cuboids based on the TiN phase. In the Ti-modified steel, the amount of Nb(C, N) significantly reduced and coarse TiN particles containing small amounts of Nb and V predominated.

3) Analyses on inclusion structure and composition al-
allowed to detect in both steels globular inclusions of calcium aluminates \((\text{XCaO} \cdot \text{YAl}_2\text{O}_3)\) of different compositions and containing relevant amounts of Mg, combined with \((\text{Ca, Mn})\text{S}\) type sulphides. In the Ti-modified steel, the limited amounts of Ti and S allowable did not generate appreciable amounts of the Ti\(_4\)C\(_2\)S\(_2\) carbo-sulphide, as would be expected from literature.

(4) By using a thermodynamic database, stability of equilibrium phases formed in the two steels was evaluated. It was shown that a increase of the Ti content in the considered microalloyed steel leads to an anticipated formation of a carbonitride phase initially rich in Ti and N. On the contrary, precipitation of the AlN phase is delayed with respect to the standard microalloyed steel.

(5) Theoretical calculations also confirmed that the average composition of the carbonitrides to be formed is strongly affected by precipitation temperature. In both steels, at 1 300°C, the carbonitride phase actually represents a titanium nitride containing reduced amounts of Nb, V and C. By lowering the temperature, a depletion in Ti and N and an enrichment in Nb, V and C progressively occurs. Eventually, at 700°C, this phase becomes a carbide rich in V and Nb.

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