Influence of Al and Nb on Optimum Ti/N Ratio in Controlling Austenite Grain Growth at Reheating Temperatures

M. CHAPA, S. F. MEDINA, V. LÓPEZ and B. FERNÁNDEZ

National Center for Metallurgical Research (CENIM-CSIC), Av. Gregorio del Amo 8; 28040-Madrid, Spain.
E-mail: smedina@cenim.csic.es

On the basis of steel 38MnSiVS5 a high number of castings were manufactured varying only the Ti and Al contents and obtaining ingots in which the Ti/Al ratio was different in each case. The specimens will be heated in air furnace to different austenitization temperatures, between 900°C and 1200°C, and subsequently quenched in water. The most important achievement would be that Al plays a harmful role as an austenite grain controller at high temperatures (>1050°C), as a consequence of the quick dissolution of AlN particles and therefore the reduction in inhibition forces at the grain boundaries which are intercepted by them. The best austenite grain control was shown by steel with contents of Ti=0.044, Al=0.009, N=0.0131 (mass%) and a Ti/N ratio of 3.36. But the interpolation of the results allows it to be concluded that the best Ti/N ratio would be close to 2.5. It was also found that the control of austenite grain size improved with an addition of Nb. In paralell, a study of precipitate sizes was carried out using transmission electron microscopy (TEM) and scanning electron microscopy (SEM).

KEY WORDS: reheating temperature; austenite grain size; precipitate size distribution; Al and Nb effect.

1. Introduction

Much work[1–6] has been carried out on austenite grain control by TiN particles, and though not all the results are in agreement, it can be said that in most steels which contain Ti the percentage of this element is relatively low, of the order of 0.020 (mass%), with N content which makes the Ti/N ratio lower than the stoichiometric ratio of nitrides (3.42).

The low solubility of TiN in austenite makes Ti and N the ideal elements for controlling the austenite grain in processes where the austenite undergoes reheating at high temperatures.[7–12] Nevertheless, the interaction Ti–Al, both elements controlling the austenite grain size, has been rarely studied and the results found in this work are something different to others reported.[13]

The requirements for a good grain refinement additive can be deduced from Zener,[14] Gladman[15] and Wagner[16] expressions, respectively, and are: (a) The particles should show low solubility in order to maintain high volume fractions at relevant high temperatures; (b) Matrix solute content in equilibrium with the particle should be low in order to limit the diffusion flow of solute to growing particles.

Hence, either fine particles or a large volume fraction of particles is required to achieve a fine austenite grain size. Nevertheless, it is difficult, if not impossible, to achieve a large volume fraction of finely dispersed particles (nitrides, carbides). On the other hand, high levels of added titanium would cause the precipitation of coarse TiN particles in the liquid, and would also result in relatively high matrix levels

in the austenite if virtually all the nitrogen were fixed as TiN.

An aluminium addition of 0.015–0.030 (%, mass) was found to be necessary to obtain effective grain refinement in the titanium treated steels. With low aluminium levels (<0.005) abnormal grain growth occurred, associated with a sparse distribution of relatively coarse TiN particles. A possible explanation for this is that at low aluminium levels titanium combines with oxygen, with less titanium being available for precipitation as TiN.[13]

The steels with mixed additions of titanium, vanadium and niobium show some inconsistencies in grain coarsening behaviour. The results could be explained in part by the effect of the aluminium level. At low aluminium levels very coarse grain sizes were observed, associated with abnormal grain growth. Other steels show unexpected behaviour, with an almost constant mean grain size in the temperature range studied, associated with a form of abnormal grain growth. Examination of the mean TiN particle sizes in fine and coarse grained regions of these steels after austenitising at 1100°C showed similar particle sizes, and no explanation has been found for the behaviour of this steel.[13]

For others authors, the addition of niobium or vanadium to a titanium steel results in the formation of complex titanium–niobium or titanium–vanadium carbonitrides,[17] the compositions of which will vary significantly with temperature. Not all microalloys form such complexes, e.g. aluminium nitride with its hcp structure forms separate particles when vanadium nitride particles (fcc structures) are present. However, Nb, Ti and V carbonitrides appear to

Table 1. Chemical composition (mass%) of the steels manufacturedb and ratio Ti/N.

Steel	C	Si	Mn	V	Ti	Al	N	Nb	Ti/N
TA1	0.33	0.82	1.53	0.115	0.07	0.011	0.0144	-	4.86
TA2	0.32	0.79	1.40	0.097	0.03	0.013	0.0143	-	2.10
TA3	0.34	0.6	1.35	0.092	0.044	0.009	0.0131	-	3.36
TA4	0.32	0.83	1.38	0.100	0.01	0.037	0.0133	-	0.75
TA5	0.35	0.79	1.40	0.100	0.013	0.031	0.0135	-	0.96
TA6	0.33	0.78	1.43	0.100	0.033	0.036	0.0143	-	2.31
TA7	0.33	0.81	1.40	0.100	0.044	0.029	0.0145	-	3.03
TA8	0.34	0.75	1.38	0.098	0.024	0.028	0.0140	-	1.71
TA9	0.34	0.78	1.33	0.098	0.055	0.02	0.0146	-	3.77
TA10	0.35	0.62	1.36	0.092	0.028	0.007	0.0150	0.020	1.87
TA11	0.35	0.76	1.35	0.100	0.034	0.006	0.0150	0.019	2.27

show extended mutually solubilities which result in the complex carbonitrides. An important feature of particles of these complexes is their ability to coarsen at a much faster rate than is observed for titanium nitride particles.

The main aim of this work has been to study the influence of Ti and Al on austenite grain control at high temperatures, especially at the temperatures of thermomechanical treatments are carried out and finally to find an optimum Ti/N/Al ratio which improves the Ti/N above mentioned. As will be seen below in the analysis of results, some concepts hitherto taken as "principles", especially in relation with the role played by Al, have been dismantled, finding that better austenite grain control is obtained using a strategy different from that recommended by current literature.

2. Experimental Procedure

The steel selected to study the control of the austenite grain was a steel 38MnSiVS5. This steel is known for its use in the manufacturing of automobile components, especially crankshafts. With this composition, manufacturers and users consider that the average austenite grain size may be controlled at least to an acceptable degree. However, abnormal growth of the austenite grain size is frequently seen, even when the austenitization temperature is relatively low.

In order to study in greater depth the control of the average grain size and especially abnormal grain growth, a series of chemical compositions were designed with regard to the elements known as grain controllers: Al, Ti. **Table 1** shows the chemical composition of the steels manufactured, it being seen that Al and Ti vary in each one. The steels have been manufactured by Electroslag Remelting (ESR). The ingots was forged and subsequently given a normalised treatment to reduce the band structure produced by deformation and to homogenize the microstructure. The advantages of materials manufactured by ESR are well known, and need not be mentioned, and in this case provide clear advantages for the purposes of the proposed work.[18]

Bearing in mind the results found in this work, which will be below seen, two new steels were manufactured with low Al contents (<0.010%, mass), Ti contents between 0.025 and 0.035 (%, mass), and Nb contents of approximately 0.020 (%, mass), as possible austenite grain control reinforcers (Table 1).

The cubic specimens used of $1.5 \times 1.5 \times 1.5$ cm will be heated in air furnace, and were protected from oxidation by an argon stream, by to different austenitization temperatures, between 900°C and 1 200°C, with holding time of

75 min, and subsequently quenched in water.

The specimens were prepared for metallographic observation. After polishing they were submerged in a saturated aqueous solution of picric acid with some drops of hydrochloric acid and teepol, the latter as humectant. Analysis of the microstructure will make it possible to study the evolution of the austenite grain (average size and abnormal growth) as a function of the temperature. The study of precipitates (TiN) was carried out using SEM and TEM.

3. Results and Discussion

3.1. Evolution of the Austenite Grain Size against Temperature

All the steels showed a duplex microstructure after a certain temperature, except for steel TA1, which for the majority of the steels was 1 000°C. The presence of the duplex microstructure makes it necessary to simultaneously estimate the finer grain and coarser grain fractions, respectively. This is the reason why the austenite grain size has been evaluated taking into account the different grain fractions. However, in order to facilitate the comparison between the different steels, the weighted mean size has been determined.

It should be noted that the weighted mean size reflects the percentage and size of both fine and coarse grains, and therefore presents the advantage of being a single magnitude which is representative of the microstructure. However, if we were to use only the mean size of coarse grains, while being an important measure, this would not faithfully represent the microstructure, since it ignores the percentage of coarse grains and the contribution, though less important, of the size and fraction of fine grains.

Only in this way is it possible to evaluate the real microstructure of these steels at different temperatures and be able to finally estimate how it is influenced by the Ti and Al contents. As example, **Figs. 1–4** show the evolution of the austenite grain size versus temperature for some steels studied.

The final comparison between the weighted mean values for non-Nb steels is shown in **Fig. 5**, it being seen that steel TA3 presents the best behaviour, followed by steel TA2.

Figures 6 and **7** display some examples of micrographs corresponding to the steels used, with an indication at the foot of each figure of the corresponding temperature. Occasionally several micrographs were produced for one sin-
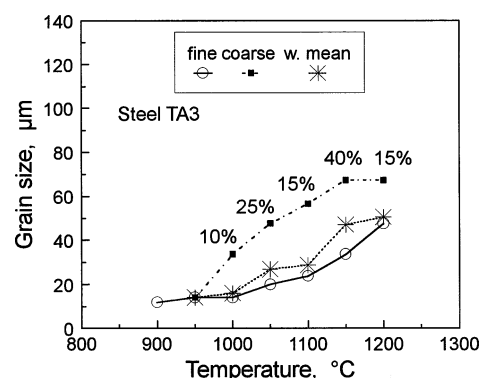

Fig. 1. Experimental data for austenite grain coarsening in steel TA3 against temperature.
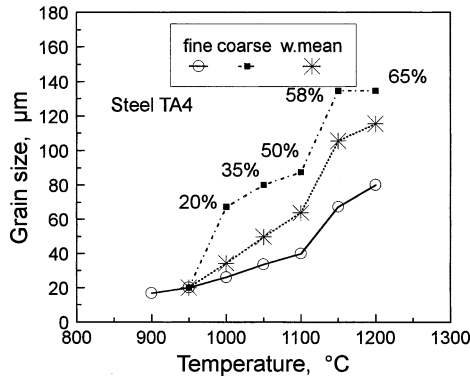
Fig. 2. Experimental data for austenite grain coarsening in steel TA4 against temperature.
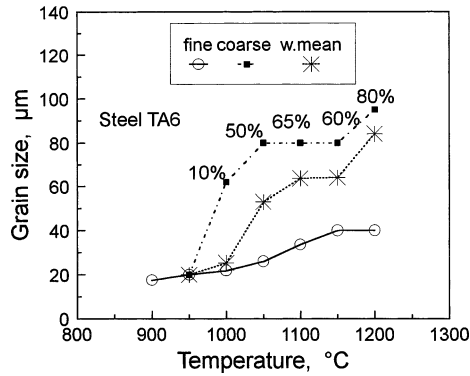

Fig. 3. Experimental data for austenite grain coarsening in steel TA6 against temperature.
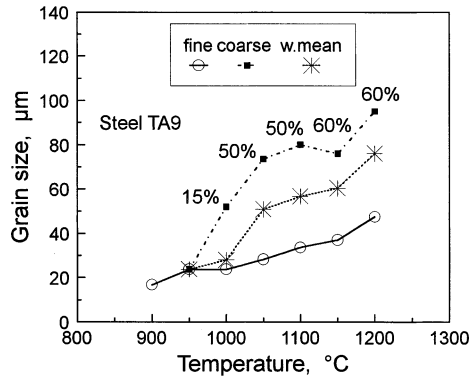

Fig. 4. Experimental data for austenite grain coarsening in steel TA9 against temperature.
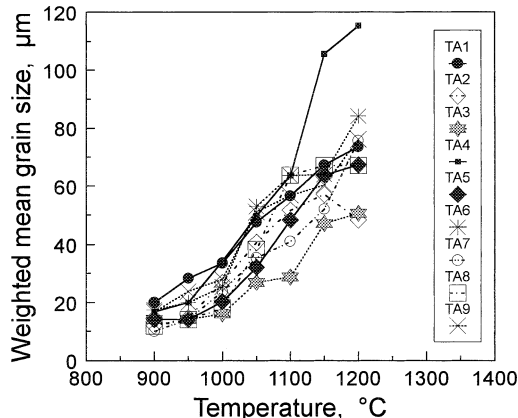

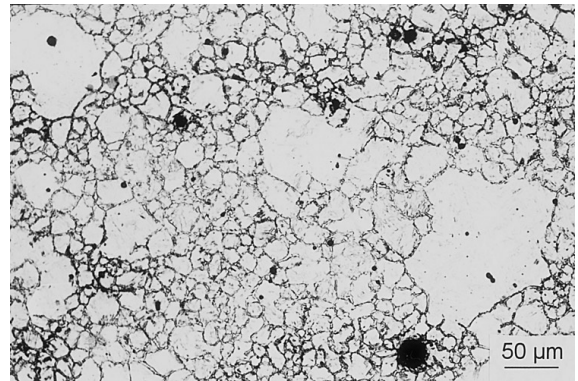Fig. 5. Mean austenite grain size against temperature for steels TA1–TA9.

Fig. 6. Optical micrograph of steel TA4 showing abnormal grain growing. Austenitising temperature: 1 000°C for 75 min.
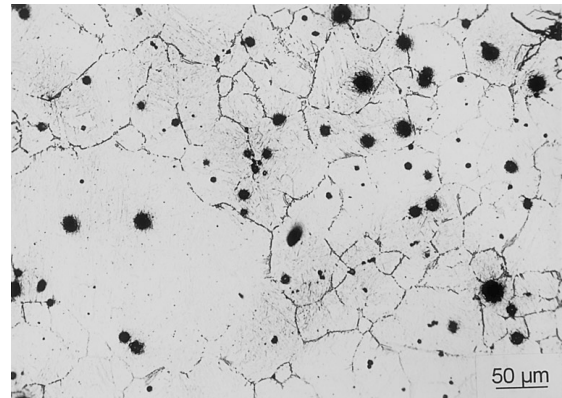

Fig. 7. Optical micrograph of steel TA8 showing abnormal grain growing. Austenitising temperature: 1 150°C for 75 min.

gle specimen when the intention was to emphasize a certain special characteristic, as in the case of grains with abnormal growth at low austenitization temperatures.

3.2. Austenite Grain Size *vs.* Time

With the intention of checking the "solidity" (or consistency) of the austenite grain, the grain size versus time was determined for steels TA3, TA5 and TA7 at the temperatures of 900°C and 1 100°C, respectively. These three steels were selected as the best representatives of three compositional variants: TA3 as a high Ti content and low Al content steel; TA5 as a low Ti content and high Al content steel; TA7 as a high Ti content and high Al content steel. In general terms, steel TA3 is once again seen to be the steel with the finest structure (**Fig. 8**).

3.3. Optimun Ti/Al and Ti/N Ratios

Graphic representation of the average austenite grain size versus Ti/N ratio (**Fig. 9**) of steels used, shows that steels TA2 and TA3 gave the best results, *i.e.* the austenite grain grew less in these two steels than in the others, whatever the austenitization temperature. The two steels have in common a low Al content, 0.013 and 0.009 (%, mass) for TA2 and TA3, respectively. However, their Ti contents, as well as their Ti/N ratios, vary considerably. This ratio was 2.09 and 3.36 for steels TA2 and TA3, respectively; both being below the stoichiometric ratio (3.42). Steels TA6 and TA7 have ratios between these values, but their austenite grain sizes were larger than in the above cases. Steels TA6 and
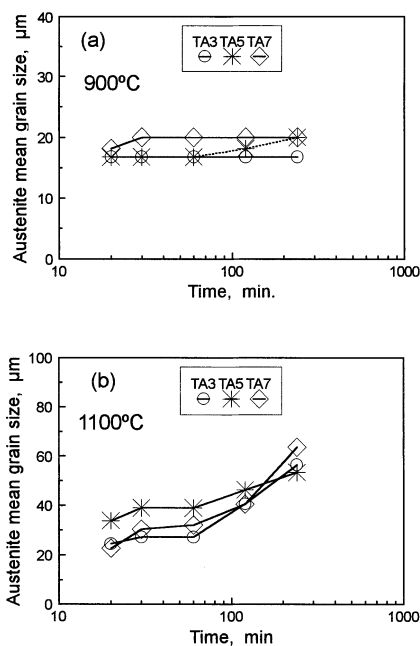
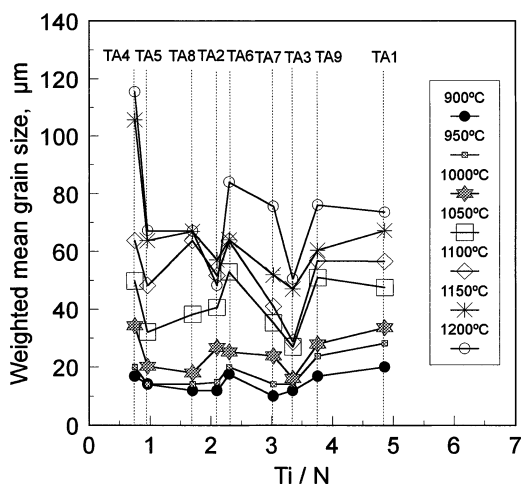**Fig. 8.** Austenite mean grain size against time for steels TA3, TA5 and TA7; (a) 900°C; (b) 1 100°C.

Fig. 9. Austenite mean grain size against Ti/N for steels TA1–TA9.

Fig. 10. Prediction of precipitates in steel TA5 according to Hillert's model.
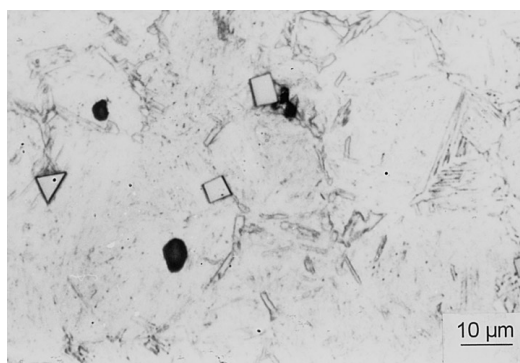
Fig. 11. Optical micrograph showing TiN particles on boundary grain. Steel TA7 at 900°C.

TA7 had Al contents of 0.036 and 0.029, respectively. On the other hand, when the Ti/N ratio was high, as in the case of steel TA1, whose value was 4.86, a reduction in the grain size was observed compared with its immediate predecessor in the order of Ti/N ratios, steel TA9 (Ti/N=3.77). While steel TA9 had an Al content of 0.020 (%, mass), the value corresponding to steel TA1 was 0.011 (%, mass).

In other words, at low temperatures (≤ 950°C) the grain size can be considered to be independent of the Ti/N ratio and also of the Al content. When the temperature of 1 000°C is reached the influence of the Ti/N ratio starts to be noted, as does the influence of Al, it being observed that steels TA4 and TA1, which have the lowest and highest Ti/N ratios, respectively, below and above the stoichiometric ratio, give the largest grain sizes. Between these two steels, and observing the line corresponding to 1 000°C, it is also possible to start seeing the formation of peaks, or relative maximums, that correspond to steels with high Al
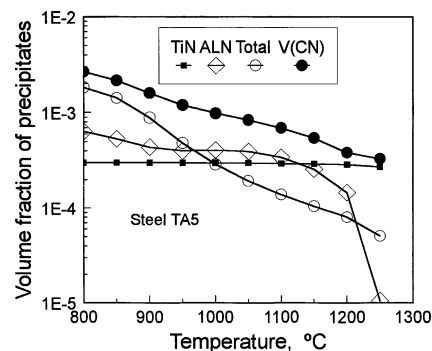
contents (TA6, TA7). As the temperature rises, the formation of peaks becomes accentuated, corresponding to the same steels.

Between steels TA4 and TA1 the formation of relative minimums is seen, corresponding to steels TA2 and TA3, which are precisely those with the lowest Al contents.

These results indicate that Al has been a harmful element in austenite grain control at high temperatures.

3.4. Precipitates, Pinning and Drive Forces

Hillert and Staffanson's model[19] permits prediction of the formation of simple precipitates (nitrides and carbides) and more complex precipitates (carbonitrides) and the results can be expressed as fraction of volume precipitated versus temperature. **Figure 10** shows an example corresponding to steel TA5, it being seen that the model predicts the formation, in addition to TiN, of VCN and AlN, also noting the temperatures at which the latter start to dissolve.

Application of the model to the steels in Table 1 showed that the formation of AlN is foreseeable in steels TA2, TA6 and TA7, while in steels TA4, TA5 and TA8 the formation of AlN could occur even at temperatures at which TiN particles are simultaneously precipitating. Only in steels TA1, TA3 and TA9 does the model predict that the formation of AlN is not possible.

Regarding to coarse precipitates, large TiN precipitates were first seen by light microscopy (**Fig. 11**). It has been reported[6] that the precipitated volume and the mean size of fine and coarse precipitates, respectively, depend to a large extent on the content of Ti solution in equilibrium with the TiN particle, according to Lifshitz, Slyozov and Wagner's expression.[16,20]
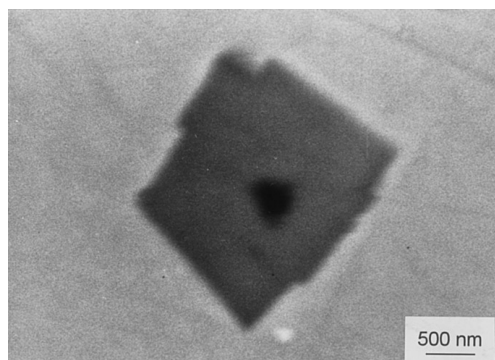
Fig. 12. SEM image of a typical TiN particle with a cuboidal morphology with a core containing Al. Steel TA7 at 900°C.
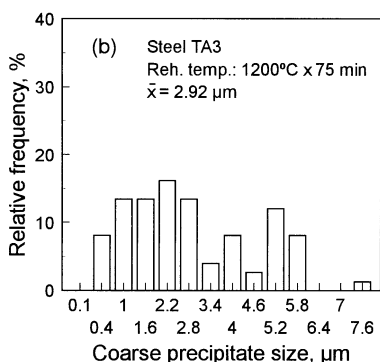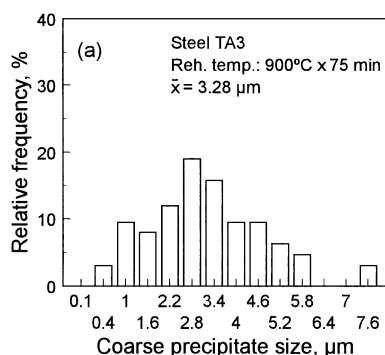

Fig. 13. Relative frequency of TiN coarse precipitates for steel TA3; (a) 900°C; (b) 1 200°C.

Fig. 14. Relative frequency of TiN coarse precipitates for steel TA5; (a) 900°C; (b) 1 200°C.

Fig. 15. TEM image of fine TiN (cuboid form) and coarse AlN (polygonal form) for steel TA7 at 900°C.

A second study of precipitates was carried out by SEM, determining compositional and stoichiometric spectra for the precipitates. The precipitates observed were always of TiN with a stoichiometric ratio close to 1/1. In high Al content steels, such as steel TA7, some precipitates showed a core containing Al and occasionally also Ca, which leads us to suspect that these precipitates nucleated on particles of alumina or calcium–aluminates (**Fig. 12**). The distribution of coarse precipitate size at 900°C (a) and 1 200°C (b) for the steels TA3 and TA5, are showed in **Figs. 13** and **14**, respectively. It can be seen that the average precipitate size was greater in steel TA3 than in steel TA5. However, steel TA3 gave a smaller grain size than steel TA5, but it is known that coarse precipitates do not exert any control on the austenite grain. On the other hand, the precipitated volume in steel TA5 is very small compared with that corresponding to steel TA3, easily calculable on the basis of the Ti and N cont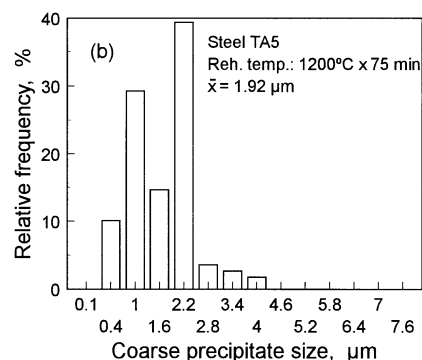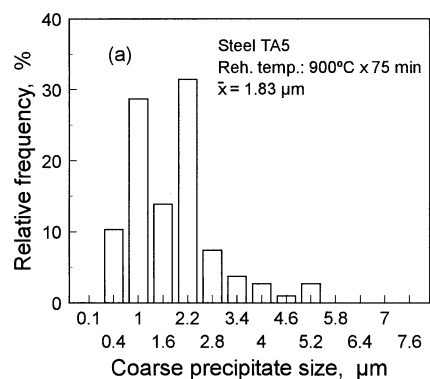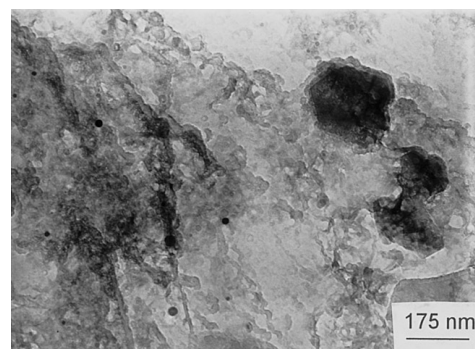ents of each one. A comparison between the distribution of precipitate size at both temperatures for steel TA3, shows that the mean size has not practically grown from 900°C to 1 200°C.

By means TEM the fine precipitates of steels TA3, TA5, TA7 and TA8 were analysed. Fine precipitates were considered to be those with a precipitates size of less than 100 nm. Steels TA5, TA7 and TA8 showed AlN precipitates at 900°C, especially in the cases of steels TA5 and TA8. AlN precipitates were normally larger than TiN precipitates (**Fig. 15**).

In above steels the count of precipitates and the measurement of their sizes was carried out on the TiN precipitates present. At 900°C, the results were favourable for steel TA3, though at the temperature of 1 200°C the sizes were similar. **Figures 16–19** show the distribution of fine precipitates at 900°C and 1 200°C for the aforementioned steels, and **Table 2** indicates the weighted mean sizes of the TiN precipitates and precipitate volumes, both at 900°C and at
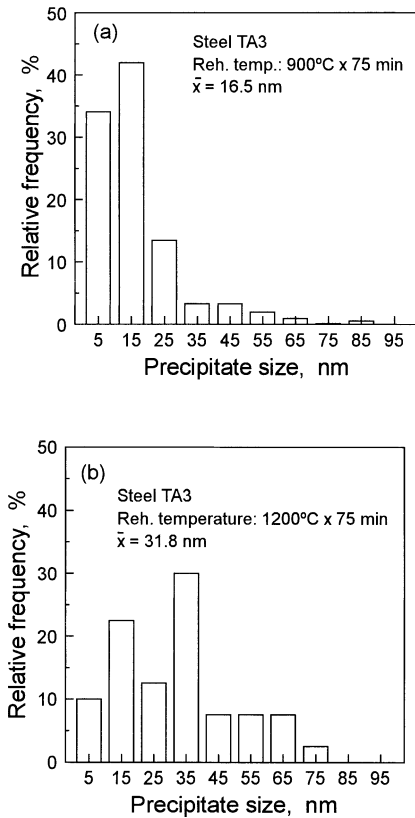
Fig. 16. Relative frequency of TiN fine precipitate sizes for steel TA3; (a) 900°C; (b) 1 200°C.
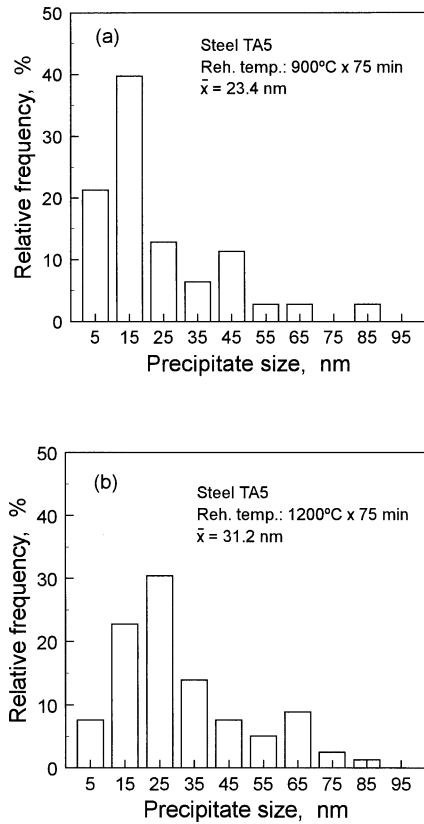

Fig. 17. Relative frequency of TiN fine precipitate sizes for steel TA5; (a) 900°C; (b) 1 200°C.

Fig. 18. Relative frequency of TiN fine precipitate sizes for steel TA7; (a) 900°C; (b) 1 200°C.
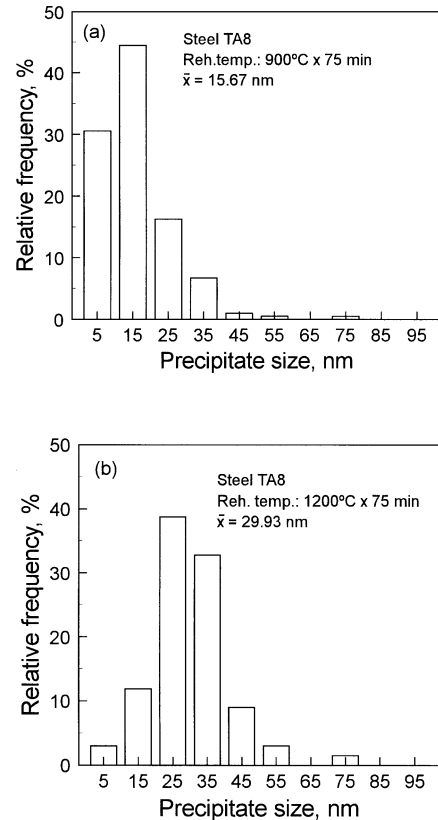
Fig. 19. Relative frequency of TiN fine precipitate sizes for steel TA8; (a) 900°C; (b) 1 200°C.

1 200°C, respectively. At 900°C steel TA5 showed the precipitates of largest size due to the fact that many of the precipitates measured were in fact TiAlN. The o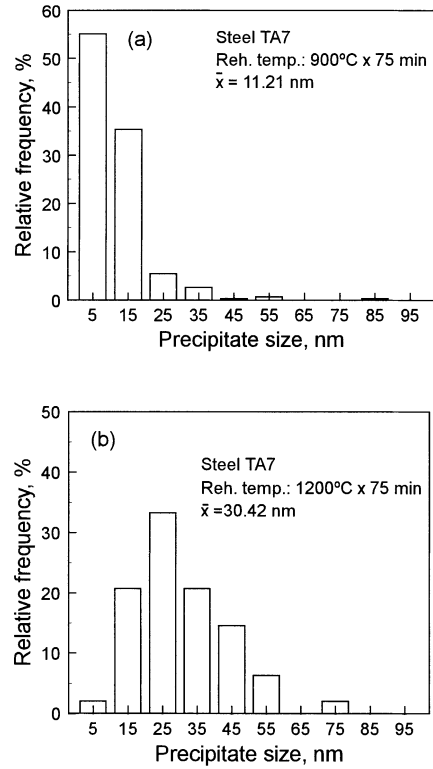ther steels showed precipitates of very similar sizes at both 900°C and at 1 200°C. Specifically, at 1 200°C all the steels showed a weighted mean precipitate size of around 30 nm, which supposes an important finding that facilitates interpretation

Table 2. Values of weighted mean size of precipitates (TiN) for steels TA3, TA5, TA7, TA8 and precipitate volume calculated according to Hillert and Staffanson's model.[19]

Steel	TA3		TA5		TA7		TA8	
Temperature (°C)	900	1200	900	1200	900	1200	900	1200
Fine (nm)	16.5	31.8	23.4	31.2	11.2	30.4	15.7	29.9
Coarse (μm)	3.3	2.9	1.8	1.9	2.6	2.6	2.9	2.8
Precipitate volume	$1.02 \cdot 10^{-3}$	$9.68 \cdot 10^{-4}$	$3.01 \cdot 10^{-4}$	$2.88 \cdot 10^{-4}$	$1.01 \cdot 10^{-3}$	$9.23 \cdot 10^{-4}$	$5.56 \cdot 10^{-4}$	$5.31 \cdot 10^{-4}$

Table 3. Estimated pinning (F_p) and driving (F_d) forces at 900°C and 1 200°C for the most representative steels.

Steel	Temp., (°C)	$\bar{r}$, (nm)	Zener: $F_p = 3f\gamma/2r$ (Mpa)	RBM: $F_p = 6f\gamma/\pi r$ (Mpa)	FBM: $F_p = 3f^{2/3}\gamma/\pi r$ (Mpa)	Zener: $F_d = 2\gamma/R$ (Mpa)	Gladman: $F_d = \left(\frac{3}{2} - \frac{2}{Z}\right)\frac{\gamma}{R}$ (Mpa)
TA3	900	9.3	0.065	0.084	0.524	0.134	0.011
TA5	900	13.2	0.014	0.017	0.164	0.113	0.009
TA7	900	6.3	0.096	0.122	0.767	0.160	0.013
TA8	900	8.9	0.037	0.048	0.367	0.134	0.011
TA3	1200	17.9	0.032	0.041	0.262	0.034	0.008
TA5	1200	17.6	0.010	0.012	0.119	0.024	0.006
TA7	1200	17.2	0.032	0.041	0.266	0.024	0.006
TA8	1200	16.9	0.019	0.024	0.187	0.028	0.007

of the Ti/N ratio and of the role of AlN particles in austenite grain control.

The above demonstrates that there is not an optimum Ti/N ratio but an interval, possibly between 2 and 3.4, which would give a very similar average precipitous size, at least up to 1 200°C. After 1 200°C it is to be expected that the lower limit of this optimum interval would shift to a higher value due to the fact that the volume precipitated starts to drop below the minimum amount necessary to continue controlling the austenite grain.

The abnormal austenite grain growth in these steels, and in general in all microalloyed steels, is due to the progressive dissolution of the precipitates with temperature and also to the coarsening experienced by the precipitates during the holding time at these temperatures, through the Ostwald ripening process.

When the driving forces for grain growth exceed the pinning forces exerted by the particles on the grain boundaries, then abnormal grain growth will take place. Many expressions have been proposed in the literature for calculating pinning forces, practically all of which are modifications of Zener's original expression, since the hypotheses upon which this equation was deduced are not fulfilled in many cases.[14] By way of guidance, the pinning and driving forces have been calculated using the expressions shown in **Table 3**. Given that the TiN precipitates observed on the carbon replica have a practically square shape, precipitate size (Table 2) was assessed as the side of the square. In those cases where the shape was notably rectangular, the size was expressed as the square root of the product of the sides. However, in Zener's equation precipitate size is expressed by the radius, since the precipitates are accepted to be approximately spherical, but this is not the case of titanium nitrides, whose shape is typically cuboid. Thus in order to apply Zener's equation to the case in hand it is necessary to convert the size to a hypothetical circle radius by means of a simple equivalence between the area of a circle and that of a square,[6] given by $\bar{r} = \bar{l}/\sqrt{\pi}$. The equivalence could also be made between sphere and cube volumes, but as the observations and measurements of the precipitates are made for their area it seems more correct to make the equivalence

between circle and square, since precipitate thickness cannot be measured by the carbon extraction replica technique.

The calculations made on driving forces and pinning forces are shown in Table 3. In order to calculate driving forces, use has been made of Zener and Gladman's expressions, respectively.[22] In the case of pinning forces, the three expressions noted in the table have been used: Zener's expression; the rigid boundary model (RBM); and the flexible boundary model (FBM).[23] In order to calculate pinning forces it has been supposed that the volume of fine precipitates (<100 nm), those truly responsible for pinning forces, is approximately 50% of the total precipitated volume.[24] On the other hand, the value given to interfacial energy per unit of area (γ) was $0.8 \, \text{J/m}^2$.

With regard to driving forces, the value of Z in Gladman's equation, the ratio of the radii of growing grains to matrix grains, known as Gladman's heterogeneity factor, was valued at 1.5 for 900°C and 2 for 1 200°C, as the most approximate values.

The results in Table 3 demonstrate that the pinning forces predicted from FBM are greater than those estimated from RBM and Zener's expression. This was expected because more particles are assumed to interact with moving boundaries in the FBM.[23] Moreover, the driving force values predicted by Gladman's expression are one order of magnitude lower than those predicted by Zener's expression.

The reading of the values in Table 3 may lead to different interpretations. On the one hand, if we compare the values of F_p predicted by Zener and RBM with those of F_d predicted by Zener, the conclusion is reached that the driving forces are greater than the pinning forces in all cases, but as both values are very close it would seem that the equilibrium has been reached, with which there would be a stable situation between the coarsening of some grains, or in inverse terms the shrinkage of others, and the state of precipitation, represented by the average size and the precipitated volume. However, if we compare the previous value of F_p with the values of F_d predicted by Gladman, the pinning forces are now always greater, which means that the grain of the matrix is controlled and its growth would be impossible. Finally, if we compare the values of F_p predicted by

FBM with those of F_d predicted by both Zener and Gladman, respectively, the same conclusion is reached, since the former are always greater.

Therefore, if we accept that the pinning forces are always greater than or equal to the driving forces, the conclusion of all this is that abnormal growth in these steels will occur not only due to the coarsening of the precipitates with temperature and holding time, but especially due to the progressive dissolution as the temperature increases.

Finally, if we compare the pinning forces of steels TA3 and TA7, that corresponding to steel TA7 is greater, and therefore it could be expected that the average austenite grain size would be smaller for the latter, or that the coarse grain fraction would also be smaller. These results lead to the conclusion that AlN particles are not only harmful due to their greater size than TiN particles, but especially due to fact that their quick dissolution at temperatures approximately below 1 100°C, in accordance with their solubility temperature,[10] causes a notable drop in pinning forces,[25] leading to a considerable expansion of the grain boundaries where these precipitates are found".

3.5. Effect of Nb on the Austenite Grain Control

Table 1 indicates the chemical composition of the new steels manufactures. The austenite grain size was measured in the new steels manufactured TA10 and TA11, both with a Nb content of close to 0.020 (%, mass), without Al and with a Ti/N ratio below the stoichiometric ratio of titanium nitrides. The results show that austenite grain control was much better in steel TA11, which has a Ti/N ratio of 2.27
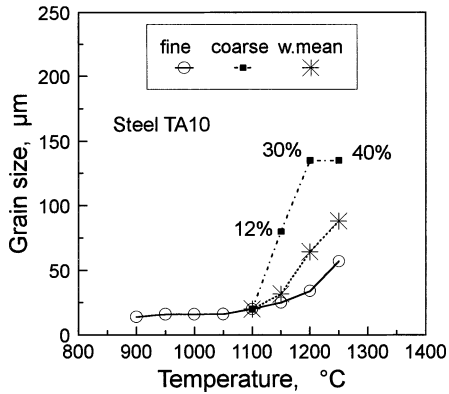
(**Figs. 20** and **21**).

Graphic representation of the average austenite grain size versus temperature for steels giving the better improvement (TA2, TA3, TA5, TA7, TA10, TA11) shows (**Fig. 22**) that the steel TA11 gave the best results, *i.e.* the austenite grain grew less in this steel than in the others, whatever the austenitization temperature.

In the **Fig. 23**, a graphic representation of the average austenite grain size versus Ti/N ratio for the better TA2, TA3, TA10 and TA11 where the improvement of the steels showing the better austenite grain control is now well appreciated. The fourth steels have in common a low Al content, 0.013, 0.009, 0.007 and 0.006 (%, mass) for TA2, TA3 and TA10 and TA11, respectively. However, their Ti contents, as well as their Ti/N ratios, vary considerably. This ratio was 2.10, 3.36, 1.87 and 2.27 for above steels, respectively; all being below the stoichiometric ratio (3.42).

Observing Fig. 23, it can he seen that the addition of Nb to the steel with a Ti/N ratio of approximately 2.27, as is the case of steel TA11, has resulted in a considerable improvement in austenite grain control compared with steel TA3. However, this result must he taken with caution, as with steel TA10, which has a Ti/N ratio of approximately 1.87, very similar as can be seen to steel TA2, and with a Nb content similar to that of steel TA11, no improvement was achieved in austenite grain control compared with steel TA3. The explanation obviously lies in the volume, distrib-

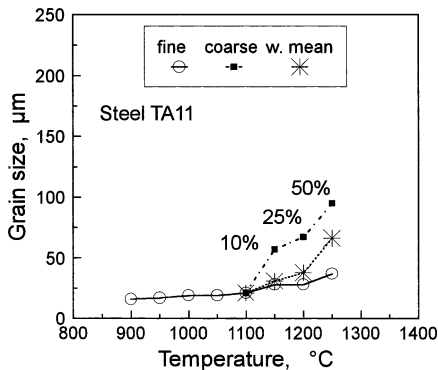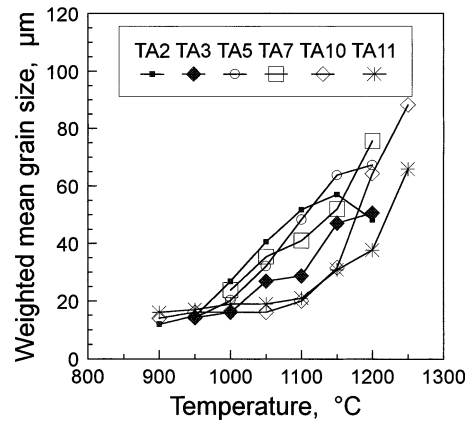**Fig. 20.** Experimental data for austenite grain coarsening in steel TA10 against temperature.

Fig. 21. Experimental data for austenite grain coarsening in steel TA11 against temperature.

Fig. 22. Comparative representation of austenite mean grain size against temperature for steels TA2, TA3, TA5, TA7, TA10 and TA11.
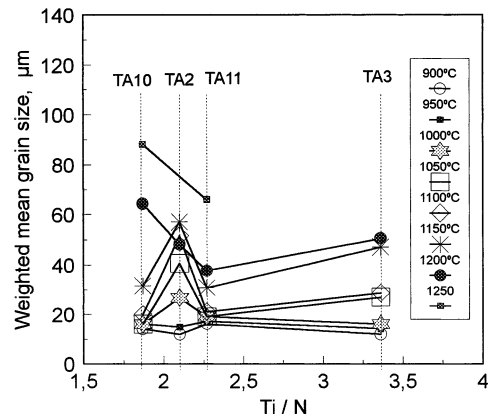

Fig. 23. Austenite mean grain size against Ti/N ratio for steels TA2, TA3, TA10 and TA11.

ution and sizes of the precipitates. However, a preliminary comparison between steels TA2 and TA3 showed that the Ti/N ratio was probably small in the former, and at high temperatures the density of precipitates was insufficient to exercise better control of the austenite grain. Bearing in mind that the Ti/N ratio in steel TA10 is somewhat lower than that of steel TA2, the explanation why worse austenite grain control was obtained with steel TA10 than with steel TA11 is probably that the volume precipitated at high temperatures ($>1\,100°C$) was insufficient. In addition, the Al content of steel TA10 was higher than steel TA11.

4. Conclusions

The aims of this work have been accomplished and in this sense some of the main conclusions are as follows:

(1) The most important achievement would be that Al plays a harmful role as an austenite grain controller at high temperatures ($>1\,050°C$), as a consequence of the quick dissolution of AlN particles and therefore the reduction in inhibition forces at the grain boundaries which are intercepted by them.

(2) Though the best austenite grain control in steels without Nb was shown by steel TA3, with contents of Ti$=0.044$; Al$=0.009$; N$=0.0131$ (%, mass) and a Ti/N ratio of 3.36, this does not mean that this composition, or even this ratio, is the optimum, as steels TA2, TA6, TA7 and TA8 could have given better results if their Al contents had been lower, especially in the case of steel TA2.

(3) The average size of TiN fine precipitates in steels with or without Al, and with Ti/N ratios of between 2 and 3.4, was similar, which serves to scientifically demonstrate that the differences in austenite grain size found between the steels studied is due especially to the presence of AlN precipitates.

(4) Interpolation of the results obtained with regard to the austenite grain size allows it to be concluded that the best Ti/N ratio would be close to 2.5, *i.e.* somewhat higher that that of steel TA2 and somewhat lower than that of steel TA3, though it would be more correct to state that the optimum ratio is found in the interval between 2 and 3.4.

(5) A good Ti/N ratio, *e.g.* between 2 and 3.4 as mentioned above, does not in itself guarantee good austenite grain control, as it is necessary to have a sufficient precipitated volume, whatever the temperature. In this sense, a Ti/N ratio of less than or close to 2 can be insufficient at temperatures close to $1\,200°C$ or higher.

(6) The addition of Nb, maintaining the Al content very low and a Ti/N ratio no lower than 2.27, as was the case of steel TA11, improved austenite grain control compared with the best behaviour shown by steel TA3.

(7) In relation with the above conclusion, when the Ti/N ratio was lower than 2, such as in the case of steel TA10, the presence of Nb did not contribute to improving austenite grain control. In short, the advisability of adding Nb as microalloying element to the steel studied is not thoroughly confirmed and further checks would be necessary.

REFERENCES

1) S. F. Medina and J. E. Mancilla: *Scr. Metall. Mater.*, **30** (1994), 73.
2) H. Baumgart and C. Strassburger: *Steel Res.*, **56** (1985), 645.
3) R. I. Bodnar: *Iron Steelmaker*, **21** (1994), 19.
4) J. R. Paules: *Iron Steelmaker*, **18** (1991), 41.
5) F. B. Pickering: Proc. of the 8th Conf. on Titanium Technology in Microalloyed Steels, The Institute of Materials, London , (1997), 10.
6) S. F. Medina, M. Chapa, P. Valles, A. Quispe and M. I. Vega: *ISIJ Int.*, **39** (1999), 930.
7) J. Kunze: *Met. Sci.*, **16** (1982), 217.
8) J. Kunze, B. Beyer, S. Oswald and W. Gruner: *Steel Res.*, **66** (1995) 161.
9) K. Narita: *Trans. Iron Steel Inst. Jpn.*, **15** (1975), 145.
10) E. T. Turkdogan: *Iron Steelmaker*, **3** (1989), 61.
11) K. Inque, I. Onhuma, H. Ohtani, K. Ishida and T. Nishizawa: *ISIJ Int.*, **38** (1998), 991.
12) H. Wada and R. D. Pehilke: *Metall. Trans. B*, **16B** (1985), 815.
13) P. Reynolds: *Ironmaking Steelmaking*, **18** (1991), 52.
14) P. A. Manohar, M. Ferry and T. Chandra: *ISIJ Int.*, **38** (1998), 913.
15) T. Gladman: *Ironmaking Steelmaking*, **16** (1989), 241.
16) C. Wagner: *Z. Elektrochem.*, **65** (1961), 581.
17) P. R. Rios: *Mater. Sci. Technol.*, **4** (1988), 324.
18) S. F. Medina and F. Reyes: *Rev. Met. Madrid*, **27** (1991), 95.
19) M. Hillert and L. I. Staffanson: *Acta Chem. Scand.*, **24** (1970), 3618.
20) Lifshitz and V. V. Slyozov: *J. Phys. Chem. Solids*, **19** (1961), 35.
21) T. Gladman: The Physical Metallurgy of Microalloyed Steels, The Institute of Materials, London, (1997), 177.
22) T. Gladman and D. J. Senogles: Proc. of the 8th Conf. on Titanium Technology in Microalloyed Steels, The Institute of Materials, London , (1997), 83.
23) O. Kwon and A. J. DeArdo: *Acta Metall. Mater.*, **39** (1991), 529.
24) N. Gao and T. N. Baker: *ISIJ Int.*, **38** (1998), 744.
25) M. Militzer: Proc. of the 1st Joint Int. Conf. on Recrystallization and Grain Growth, Vol. 1, ed. by G. Gottstein and D. A. Molodov, RWTH Aachen, (2001), 361.