JOURNAL OF MATERIALS SCIENCE 31 (1996) 1303–1309

Abnormal grain growth in a medium-carbon microalloyed steel

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An experimental study on the grain growth of a medium-carbon V–Ti microalloyed steel with two levels of AlN has been carried out. A system to study grain-size distributions in order to detect the abnormal grain growth has been proposed. Log-normal distributions were verified and then properties of normal distributions were applied to distinguish normal and abnormal grains. The benefits of working with the relative difference (RD) of grain size in order to compare the grain-growth behaviour have been discussed. It was experimentally concluded that abnormal growth appears when RD is larger than 2.5. From the results a map of abnormal grain growth against time and temperature could be plotted. It was concluded that abnormal grain growth is due to the AlN dissolution when the above maps are correlated with the theoretical volume fraction of precipitates. The importance and effect of heating rate have also been shown: high heating rates can produce abnormal growth at higher temperatures than those of the equilibrium dissolution.

1. Introduction

The application of microalloyed steels in hot forming is steadily increasing in industrial importance [1, 2]. Mechanical properties of these steels are obtained at the end of the forming process from unnecessary post-forming heat treatments, so an important economical saving can be derived.

To obtain a good combination of toughness and strength, a fine grain structure is required. Grain size depends on (a) the starting grain size at the austenizing temperature, and (b) the hot-forming conditions and subsequent cooling. In the present work, attention was focused on the first point.

One of the most important ways to control the grain size is by addition of a dispersion of second-phase particles. This is one role of the microalloying elements in this kind of steel. The inhibition of grain growth is usually accompanied by an undesirable abnormal grain growth. Theories indicate that grain growth is controlled up to a critical grain radius, R_{crit}, [3–6], which depends on the volume fraction of precipitates, f, and their size, r, according to

$$R_{\text{crit}} = K\left(\frac{r}{f}\right) \qquad (1)$$

Theories differ on the value of the constant K and some of them introduce a matrix heterogeneity factor in this constant. This heterogeneity seems to be the controlling parameter of the abnormal grain-growth appearance [4–6].

The aim of this work was to study how the precipitates of AlN and complex carbonitrides of V–Ti influence normal and abnormal grain growth on a microalloyed steel.

2. Experimental procedure

A commercial medium-carbon microalloyed steel with two different amounts of aluminium was chosen for the present work (see compositions in Table I). Specimens were obtained from the supplied steels (round rolled at 100 mm diameter from an ingot of 4000 kg, heating temperature 1250 °C, finish rolling temperature 900 °C) and austenized at different temperatures (900, 950, 1000, 1050, 1100, 1150 and 1250 °C) for various times (5, 10, 15, 30, 60, 90 and 180 min) in a tubular furnace with an argon protective atmosphere to avoid decarburization. After the holding time, specimens were directly quenched in to water at room temperature.

Following the polishing step, samples were chemically hot etched in a saturated picric acid solution to reveal the previous austenite grain size. The area of each grain and the equivalent grain diameter was determined using a computer-based image analyser. An average of 300 grains was used for every sample.

3. Results

The main problem when studying abnormal grain growth is to determine from the size distribution if a given grain belongs to the normal or abnormal population. Usually the grain-size histogram follows a log-normal distribution [7] and abnormal grains often remain integrated at the right tail of the distribution which makes their separation difficult. Some authors [8] have provided evidence of abnormal grains by multiplying the frequency by the size, but in fact this is not always a clear criterion to separate both

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TABLE I The compositions (wt%) of steels A and B

	C	Mn	Si	P	S	V	Al	Ti	N (p.p.m.)
Steel A	0.34	1.52	0.72	0.025	0.025	0.083	0.0145	0.0181	114
Steel B	0.36	1.35	0.71	0.018	0.011	0.09	0.0224	0.0171	139

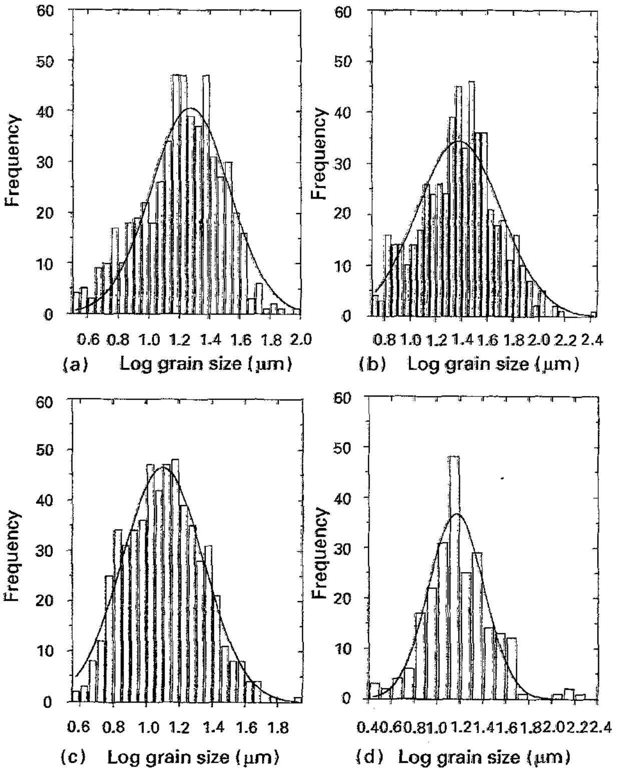

Figure 1 Histograms of the logarithm of grain size for: (a) steel A, 950 °C, 60 min; (b) steel A, 1050 °C, 10 min; (c) steel B, 1000 °C, 15 min; (d) steel B, 1050 °C, 90 min.

distributions, and it seems necessary to study the distribution tails in more detail.

In Fig. 1, grain-size distributions for representative cases of this study are shown. A good agreement with log-normal distributions is observed, as is usually reported. In order to study the right tail of the distribution, in this work, initially those grains with log-sizes larger than the average plus two standard deviations were taken to be abnormal. It must be kept in mind that average and standard deviation are those of the corresponding log-normal grain-size distributions. If, instead of the average and standard deviation of the *sample*, those of the *population* were known, it could be said that grains in that situation do not belong to the normal population with a confidence interval of 97.7% [9]. The implicit hypothesis is that the average and standard deviation of the *sample* are good estimators of those of the *population*. This value (average + 2 standard deviation) will initially be used to try to separate both grain families. Every family will then be characterized by its own average: AGS_{normal} will be the average grain size of all the grains without considering the abnormal candidates, and $AGS_{abnormal}$ will represent the average of the abnormal candidates, that is, the average of the right tail (separated according to the above criterion) of the grain-size distribution. In other words, the distribution of logarithms of grain sizes will be used only as a separation criterion and then the resulting grain-size distributions will be used in a conventional way.

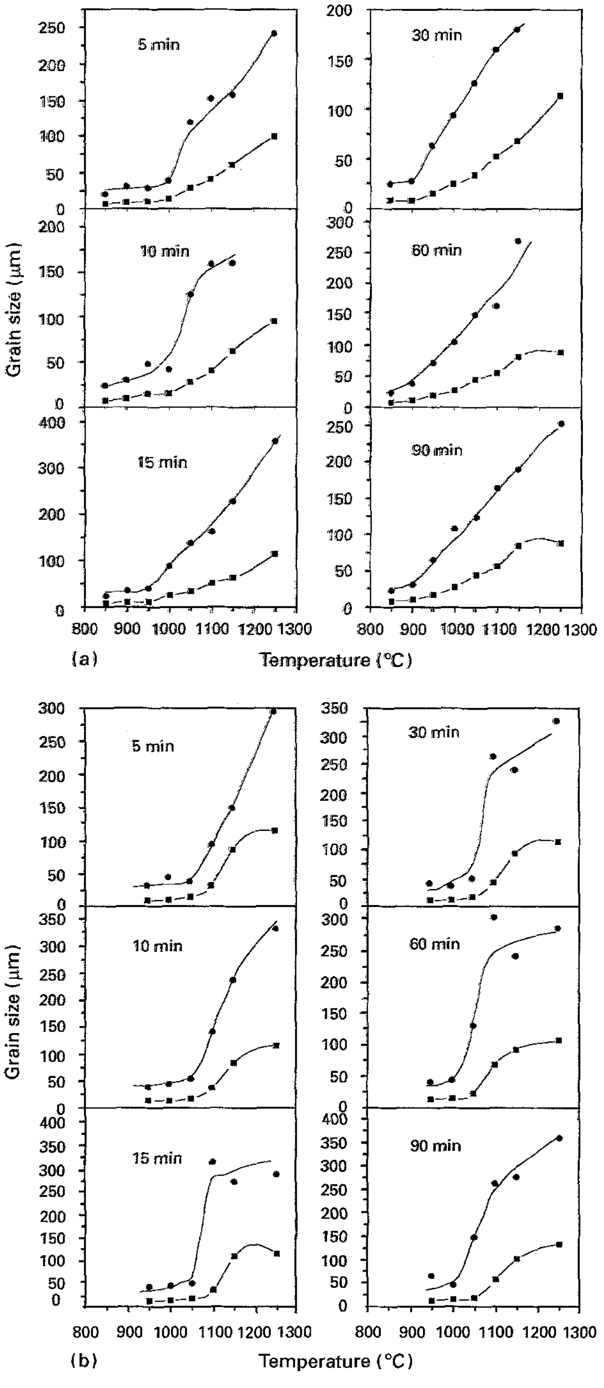

Figure 2 Evolution with temperature and time of (■) AGS_{normal} and (●) $AGS_{abnormal}$ (a) for steel A and (b) for steel B. (For explanation of these values see text.)

The evolution with temperature and holding time of AGS_{normal} and $AGS_{abnormal}$ is shown in Fig. 2 for the studied steels. It can be observed that both curves are practically parallel up to a temperature where $AGS_{abnormal}$ deviates clearly from AGS_{normal}. At this point, the difference between both averages increases, and at higher temperatures it again tends to remain constant. It is also shown in Fig. 2 that the deviation temperature clearly depends on temperature. However, in order to get a clearer picture of what is happening during the growth of both grain families, it is better to study the variation with temperature of the relative difference, which will be defined as follows: $RD = (AGS_{abnormal} - AGS_{normal}) / (AGS_{normal})$. Absolute differences are not appropriate because they depend on the instantaneous grain-size magnitude. The value RD can also be seen as a quantification of the heterogeneity of the grain-size distributions. This parameter is similar, but not identical, to those used by other authors [4, 6]. Fig. 3 shows the variation of RD with temperature and, in all cases, it can be observed that RD is roughly constant except in a narrow temperature interval where it reaches a maximum. This suggests that a change in the grain growth mechanism has taken place in that temperature interval. Microstructural observations (Fig. 4) indicate that abnormal grain growth occurs in coincidence with this peak interval, when RD is greater than 2.5–3. Another interesting observation is that the value of RD outside the peak zone is practically constant and close to 2. This value agrees with that reported by Gladman [4, 10] for the size advantage, Z. The parameter Z (grain-size ratio between matrix and growing grains), is very similar to the parameter RD defined here. After Gladman, a grain-size distribution with a size advantage of 2 can be effective in causing abnormal grain growth. Accordingly, the grain-size distributions of this study are very near to promoting abnormal grain growth. Nevertheless, a change in the pinning force on grain boundaries is required to surpass this equilibrium state (RD = constant), such as a variation in the volume fraction of precipitates, as considered below. Representing the RD values versus time and temperature, a map of the heterogeneity of the grain-size distributions can be obtained (see Fig. 5). Zones with isolines of $RD > 2.5$ are abnormal grain-growth regions, as discussed above.

In order to be able to relate maps of grain-size heterogeneity to the existence of different types of precipitates, a theoretical study of the equilibrium precipitation has been carried out. The precipitation model of Hillert and Staffanson [11–13] for a multicomponent system was applied to derive the evolution in equilibrium of the volume fraction of precipitates with temperature in the steels studied here (without taking into account the interaction parameters). This model assumes a regular solution of a single precipitate of vanadium and titanium to form a complex V–Ti carbonitride and the AlN precipitate was added for this case (see Equation 2). The same carbonitride solubility products used elsewhere [12] were used for the steels studied here. For AlN, the usual value was taken [14]

$$V_aTi_{1-a}C_bN_{1-b} + AlN \qquad (2)$$

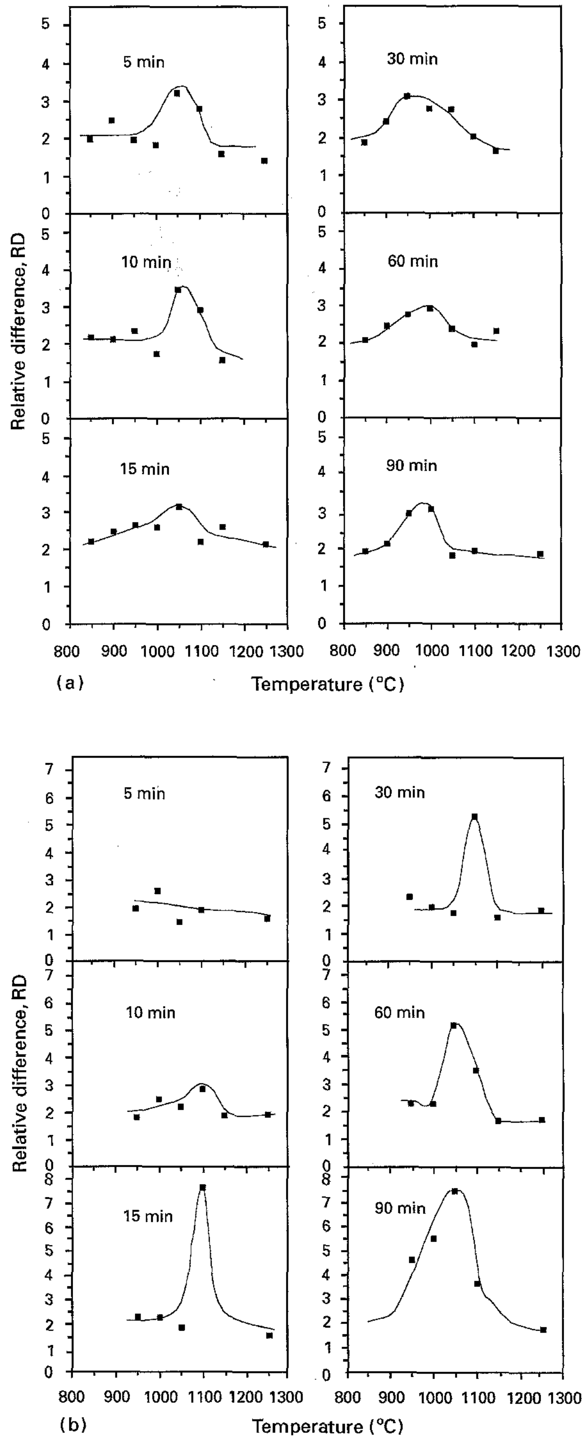

Figure 3 Dependence of the relative difference (RD) in grain size with time and temperature for (a) steel A, and (b) steel B.

The results are shown in Fig. 6a, and it must be remarked that both steels show the same amount of V–Ti precipitates; the main difference lies in the different concentration of AlN precipitates. The AlN dissolution takes place in a narrow range of temperatures, while the carbonitride dissolution occurs progressively. The high stability of titanium nitrides at high temperatures and vanadium carbides at low temperatures (Fig. 6b) should also be noted. Results of dissolution temperatures for the AlN are included in Fig. 5 for comparison purposes (shaded areas).

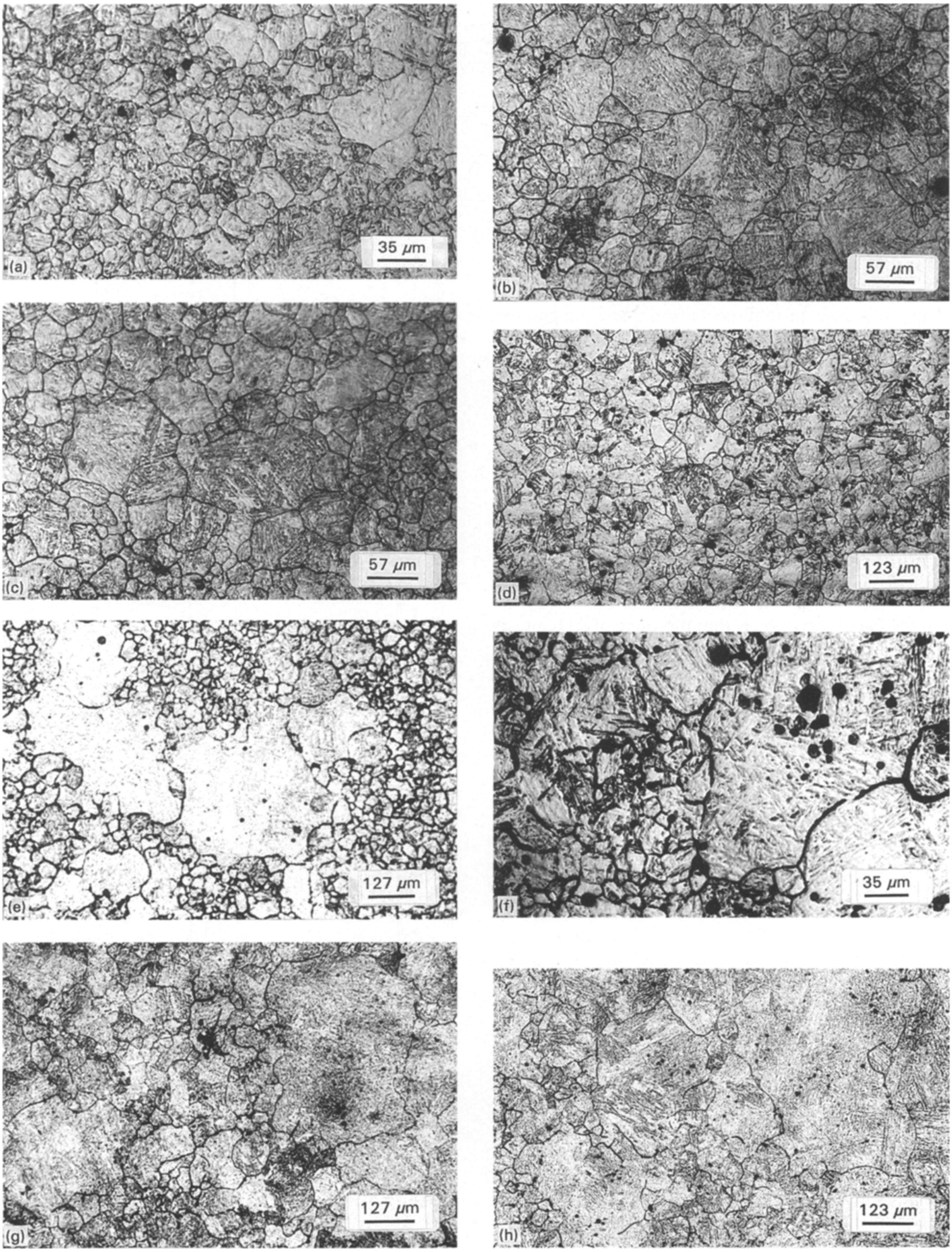

Figure 4 Micrographs of different samples showing the distinct grain-growth modes observed. Steel A presents a moderate abnormal grain growth while steel B shows a severely abnormal growing. Steel A: (a) austenized at 900 °C, for 180 min. (b) 1000 °C, 30 min. (c) 1000 °C, 60 min. (d) 1050 °C, 60 min. Steel B: (e) 950 °C, 90 min. (f) 1050 °C, 180 min. (g) 1100 °C, 15 min. (h) 1100 °C, 30 min.

4. Discussion

It is well documented that a dispersion of second-phase particles can produce abnormal grain growth when their dissolution takes place. Commonly, theories and experimental results only take into account a single kind of precipitate and according to Equation 1, grain size follows the critical grain size determined by the volume fraction and size of the present precipitate. The progress of abnormal grain growth is explained by an increase of the heterogeneity of

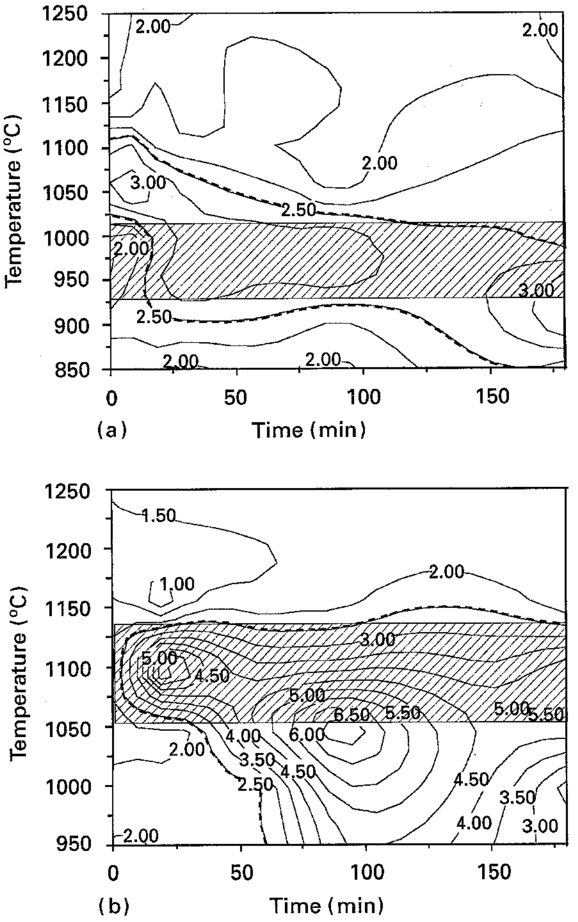

Figure 5 Maps of relative differences (RD) in the grain-size distribution for (a) steel A, and (b) steel B. Isolines in the chart correspond to isovalues of RD. Abnormal grain growth is observed for RD > 2.5 (---). Shaded areas on each map indicate the range of dissolution temperatures in equilibrium of the AlN precipitates after Fig. 6.

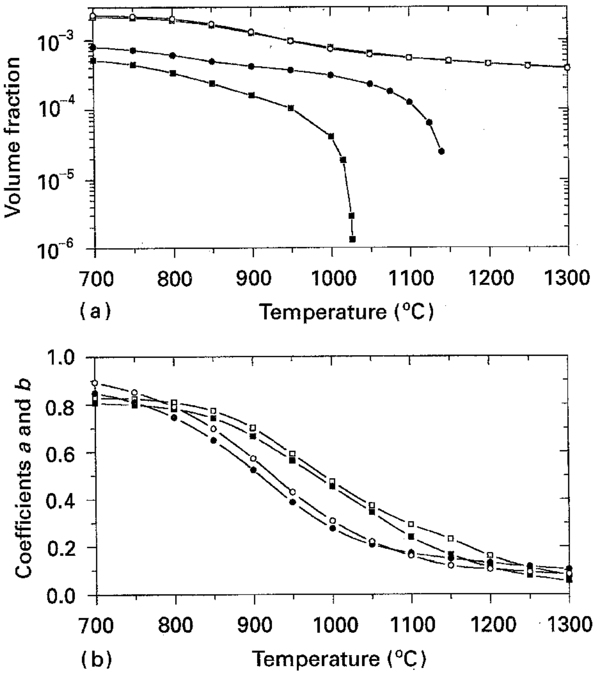

Figure 6 (a) Evolution in equilibrium of the volume fraction of the precipitates present in both steels. These results were obtained by applying the model of [11–13]. (□, ○) Carbonitride of V–Ti, and (■, ●) nitride of Al, for (□, ■) steel A, and (○, ●) steel B. (b) The character of the complex carbonitride ($V_aTi_{1-a}C_bN_{1-b}$) as a function of temperature: at high temperatures it is almost exclusively TiN and at low temperatures it is practically VC (see Equation 2). (■, □) Coefficient *a*, and (●, ○) coefficient *b* for (■, ●) steel A, and (□, ○) steel B.

the matrix grain-size distribution. The progressive unpinning, which occurs when the precipitate dissolves, favours the growth of certain grains, raising the heterogeneity of the distribution, so abnormal grain growth can progress. However, limited efforts have been addressed to the study of grain-growth behaviour in the presence of more than one family of precipitates, especially when one of them is quite stable (V–Ti carbonitrides in this study) and the other one presents an apparent dissolution temperature (AlN in this case).

Observation of Figs 5 and 6a shows that abnormal grain growth takes place during the temperature interval of dissolution of AlN with two exemptions, which will be discussed later. It seems clear that the different abnormal grain-growth behaviour observed for the studied steels can be attributed to the different amounts, and therefore stabilities, of the AlN precipitates. An obvious question which arises from Fig. 6 is how the AlN can affect the grain size when the volume fractions of V–Ti precipitates are superior to those of AlN. Fig. 2a and b show that the matrix grain size (or AGS_{normal}) for steel B is pinned to higher temperatures than for steel A, in agreement with the higher stability of the AlN. From Equation 1, the volume fraction of precipitates is seen to be not the only factor, and therefore the precipitate size must be also considered. In fact, both precipitates, V–Ti carbonitrides and AlN, probably determine the matrix grain size, that is, the mean grain size follows the critical size specified by both precipitates. However, the AlN precipitates can locally promote the appearance of abnormal grain growth, because their relatively rapid dissolution can eventually favour a local reduction in the pinning force. This diminution allows the movement of certain grain boundaries (probably in the largest grains), increasing the heterogeneity of the matrix, which is already close to promoting abnormal growth (Z or RD ~ 2), so abnormal grain growth can proceed. The results presented here suggest that the disappearance of a second phase when the matrix heterogeneity is near to its critical value, can produce an almost instantaneous abnormal grain growth, even in the presence of other stable precipitate families.

The two exemptions mentioned before occur at short times and temperatures above those determined from the theoretical study, and at relatively long times and temperatures below the theoretical range. The appearance of abnormal growth in the first case must be attributed to the heating-rate effect. Specimens were directly introduced in to the furnace at the austenizing temperature, so high heating rates occurred. Therefore, overheating produced the well-known phenomenon of shifting the theoretical dissolution temperatures of the AlN towards higher values. This feature would not be observed with slower heating rates, because dissolution curves would tend to those of equilibrium. In the second case, abnormal grain growth occurs when the dissolution of precipitates is not expected. Here, according to Equation 1, the coarsening of precipitates must be having the same effect as their dissolution at high temperatures. The presence of abnormal grain growth in the first case is well marked for steel A, while for steel B and for the experimental times tested, is less noticeable, because it takes place at high temperatures where the heating-rate effect is less important. In this case, at very short times, an insufficient period for the dissolution kinetic must not be rejected, so grains still remain completely pinned. The second case is quite apparent for steel B because the complete dissolution of the AlN precipitates takes place at relatively high temperatures. However, at lower temperatures, there is enough thermal activation to assist their ripening. Nonetheless, the coarsening of the V–Ti carbonitride precipitates must not be rejected, and their possible effect on the grain-growth mode should be considered. For the steel A, this second case is only slightly observed at very long times. The reason lies in the relatively low temperature of complete dissolution in this steel. In this case, thermal activation is not able to promote sufficient growth of the AlN precipitates at intermediate times.

An interesting feature also shown in Fig. 5 is the existence of diverse peaks in the heterogeneity value of the grain-size distribution. Steel A shows a clear moderate peak located at 1050 °C and short times, while for steel B the presence of two maxima, one situated at 1100 °C and short times, and the other at 1050 °C and long times, is apparent. The presence of these maxima can be explained in terms of a balanced effect between the appearance of abnormal grains (increasing heterogeneity) and the progressive unpinning of the matrix (decreasing heterogeneity). One can observe that when peaks are surpassed, at constant temperature, RD decreases slightly and maintains an almost constant value. In an alloy with a single precipitate, an increase in the matrix grain size must be expected, once the abnormal grain growth has taken place (i.e. the second phase has disappeared), and finally the abnormal grains must re-enter the global distribution (not by diminution of the abnormal grains, but by increasing the matrix grain size). Therefore, the matrix heterogeneity should decrease after the maximum and abnormal grain-growth stop. This is not observed in this study due to the presence of an additional stable second phase. The grain-growth behaviour after the maxima can be assimilated to that of a free particle matrix, so many grains can now grow (i.e. to decrease the distribution heterogeneity). However, this growth of the matrix can only progress up to the critical grain size determined by the stable V–Ti precipitates. This explains why the heterogeneity does not disappear at the long times tested here and it tends to be constant. In other words, after the heterogeneity peak a certain decrease in heterogeneity must be expected and finally it must rest approximately constant with time.

Another interesting feature is the small value of RD observed for steel A (2.5–3.5) whereas for steel B it oscillates in a wider range (2.5–7). This is due to the temperature of grain-boundary unpinning. Abnormal grain growth in steel B starts at relatively high temperatures and therefore thermal activation favours extensively extraordinarily large grains. In steel A the abnormal growth begins at lower temperatures. This trait justifies the moderate abnormal grain growth observed in steel A. This analysis also provides evidence of how the proposed criterion to study abnormal grain growth can even detect moderately abnormal growth (RD ~2.5).

Finally it must be noted that, at high temperatures, the observed grain growth seems to stop and to be irrespective of the austenitizing temperature and time (see Fig. 2, especially for steel B). This cessation of grain growth when the grain size becomes relatively large has already been reported, and could be due to the stable and coarse precipitates of TiN and coarse non-metallic inclusions [15].

4. Conclusions

1. The proposed criterion to separate normal and abnormal grains is a good tool to characterize abnormal growth. The parameter RD or heterogeneity of the grain-size distribution, permits the presence of abnormal grains to be detected, even when the growth is moderate.
2. Maps of grain-size heterogeneity–time–temperature (i.e. abnormal grain growth–time–temperature) can help to improve the understanding of the behaviour of microalloyed steels at the austenizing stage.
3. The results suggest that the dissolution of a second phase, when the heterogeneity of the grain distribution is close to its critical value, can produce abnormal grain growth, even in the presence of additional precipitated families.
4. The onset of abnormal grain growth is controlled by the r/f ratio of AlN. From high temperatures up to the theoretical dissolution temperatures, the predominant factor is the AlN dissolution, f, whereas at low temperatures and long times, it is the ripening, r, of both precipitates, V–Ti carbonitrides and AlN.

Acknowledgements

One of the authors (J.M.C.) thanks the Comissionat per a Universitats i Recerca de la Generalitat de Catalunya for a researchers formation grant. The authors also acknowledge the financial support received from Comisión Interministerial de Ciencia y Tecnología (CICYT) through the project MAT 94–0433.

References

1. D. J. NAYLOR, *Ironmaking Steelmaking* **16**(4) (1989) 246.
2. K. PLATTS, A. D. VASSILIOU and F. B. PICKERING, *Metall. Mater. Technol.* September (1984) 447.
3. C. ZENER, private communication to C. S. Smith, *Trans AIME* **175** (1948) 47.
4. T. GLADMAN, *Proc. R. Soc. A* **294** (1966) 298.
5. M. HILLERT, *Acta Metall* **13** (1965) 227.
6. P. R. RIOS, *ibid.* **35** (1987) 2805.
7. B. R. RALPH, *Mater. Sci. Technol.* **6** (1990) 1139.

8. R. KASPAR and N. MAHMOUD, *Mater. Sci. Technol.* **7** (1991) 249.
9. G. E. BOX, W. G. HUNTER and J. S. HUNTER, "Statistics for Experimenters", (Wiley, New York, 1978).
10. T. GLADMAN, *Mater. Sci. Forum* **94–96** (1992) 113.
11. M. HILLERT and L. I. STAFFANSON, *Acta Chem. Scand.* **24** (1970) 3618.
12. H. ADRIAN and F. B. PICKERING, *Mater. Sci. Technol.* **7** (1991) 176.
13. P. R. RIOS, *ibid.* **4** (1988) 324.
14. F. G. WILSON and T. GLADMAN, *Int. Mater. Rev.* **33**(5) (1988) 221.
15. T. GLADMAN, in "Inclusions", Institute of Metallurgists Monograph 3, edited by F. B. Pickering (Institute of Metallurgists, London, 1979) p. 172.

Received 1 December 1994
and accepted 8 September 1995