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Austenitic grain size evolution and continuous cooling transformation diagrams in vanadium and titanium microalloyed steels

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The evolution of the austenitic grain size in medium carbon steels microalloyed with vanadium and titanium was studied as a function of reheating temperature, heating rate, and titanium content. High resolution dilatometric techniques were used to determine the continuous cooling transformation (CCT) diagrams for two different austenitization temperatures. The microstructure and hardness were determined for different cooling rates. The results revealed a significant effect of titanium concentration on the austenitic grain growth control. The smallest grain size was found in the steel with a Ti concentration = 0.019 wt%. Low heating rates produced smaller grain sizes than high heating rates although an abnormal grain growth took place. In these steels, at temperatures above 1050 °C the influence of the reheating temperature on their hardness for cooling rates around 2 $°C \cdot s^{-1}$ was negligible. The higher reheating temperatures caused a slight increase in their hardenability. Finally, it was found that the greater the titanium content, the greater the hardness of these steels, but only when the titanium percentages were higher than 0.020 wt%.

1. Introduction

This paper describes part of a study [1–6] on medium carbon steels microalloyed with vanadium and titanium (titanium varies from 0.003–0.039 % wt) which are of interest to the automotive industry. Amongst the properties of the V and Ti microalloyed steels studied are included the solid state phase transformations, the hot and warm ductility, the grain growth and the static and dynamic recrystallization. We also discuss the influence of both hot and warm forging parameters on the structures and properties of forged critical automotive components. Titanium was selected as a grain growth control element in the steel. Vanadium was added because of its precipitation hardening capability, with a view to improving the toughness properties. The vanadium and titanium–vanadium precipitates formed would control the austenitic grain growth and the recrystallized austenitic grain size [1]. Thereby, these precipitates (vanadium and titanium carbonitrides and nitrides) reduce the ferrite–pearlite grain size obtained by decomposition of the austenite during cooling at rates close to air-cooling.

This paper is focused on the study of the nonequilibrium transformations caused in these V and Ti microalloyed steels by nonisothermal cycles at two different austenitization temperatures, and the influence of the titanium content on the austenitic grain growth for two different heating rates. Finally, a study was conducted on the influence of the cooling rates on the microstructure and hardness of these steels. All these data are necessary to predict the effects produced by the forging processes.

2. Experimental procedure

The experiments performed to study the solid state transformations of these steels were carried out with the aid of dilatometry techniques [3, 4].

2.1. Materials

Three medium carbon microalloyed steels were used in the present study and their compositions in weight percentage (except the gases, in parts per million) are shown in Table I. The vanadium content is similar in the three steels, whereas the titanium content is variable. The nitrogen content varied to such an extent that its influence on the austenitic grain size was minimal, although relatively important in regard to strength properties [7]. Steel manufacture methods are described elsewhere [4].

2.2. Austenitic grain growth

Longitudinally oriented dilatometry test pieces of 2 mm diameter and 12 mm length were machined from the 38.5 mm square billets. Tests were carried out by means of the heating–cooling system of a dilatometer DT 1000 Adamel-Lhomargy described elsewhere [1]. Specimens were heated to the several

TABLE I Chemical composition (wt %)

	% C	% Mn	% Si	% S	% P	% Cr	% Ni	% Mo	% V	% Ti	% Cu	% Sn	% Al	N_2*	O_2*	N Free*
Steel 1	0.29	1.34	0.41	0.026	0.021	0.09	0.10	0.02	0.10	0.003	0.244	0.020	0.029	167	30	18
Steel 2	0.29	1.28	0.34	0.028	0.017	0.13	0.08	0.01	0.09	0.019	0.134	0.015	0.036	106	45	10
Steel 3	0.32	1.39	0.33	0.021	0.015	0.13	0.14	0.03	0.129	0.039	0.129	0.017	0.049	148	57	14
38Cr2	0.39	0.65	0.22	0.034	0.025	0.50	–	–	0.005	0.002	0.23	0.016	0.034	–	–	–

* p.p.m.

testing temperatures between 900–1250 °C at two preset rates: 5 °C . s^{-1} and 0.6 °C . s^{-1} corresponding to induction furnace and conventional furnace heating rates respectively. The holding time was three minutes at each austenitizing temperature. In order to freeze the microstructure, quenching was conducted under a helium gas flow at a cooling rate of 200 °C . s^{-1}. The austenitic grain size was determined by the linear intercept techniques with the aid of an image analyser (Quantimet 520–Cambridge Instruments) described in [1].

2.3. Continuous cooling transformation diagrams (CCT)

The high resolution dilatometer mentioned-above was also used for determining CCT diagrams. The heating rate, 5 °C . s^{-1}, was the same in all the tests performed. Two CCT diagrams, one with an austenitizing temperature of 1050 °C and another with the temperature at 1250 °C, were obtained for each steel. By means of continuous heating at 1050 °C we reproduced the finest recrystallized austenitic grain size which can be obtained in each steel at the end of a hot forging (1). Therefore, the CCT diagrams obtained from this temperature will predict the different transformations during the post-forging cooling of these steels. The second temperature is widely used to forge pieces with medium and high degrees of difficulty and was 1250 °C. This austenitizing temperature determines if dissolution of titanium compounds, and therefore the precipitation in the subsequent cooling, would have a significant influence on the CCT diagrams. Once these parameters were fixed, thermal cycles were carried out for several cooling rates and its corresponding dilatometric curves were obtained. Finally, CCT diagrams were drawn with the aid of the different dilatometric results and of the microstructural analysis of each dilatometric specimen tested. The microstructural composition (area percentages) were determined by means of optical microscopy with the image analyser [1]. Vickers hardness tests were also performed in all the dilatometric specimens.

3. Results and discussion

3.1. Austenitic grain size evolution, the effect of titanium.

The evolution of the austenitic grain size at a heating rate of 5 °C . s^{-1} for the three steels are shown in Fig. 1. Steel 2 has the smallest grain size in all the temperatures tested. Grain coarsening temperature (GCT), the temperature at which an abnormal grain growth (bimodal distribution) starts [8], is close to 1250 °C for steel 2. Steel 1 has a GCT between 1000–1050 °C, whereas GCT of the steel 3 is between 1050–1100 °C. Optical micrographs of grain sizes corresponding to various temperatures of the three steels studied are shown in Fig. 1. The influence of the heating rate on the austenitic grain size evolution for steel 1 is shown in Fig. 2. The bimodal distribution of the grain size is indicated in Fig. 2 by the symbol (↑↑). For slow heating rates – 0.6 °C . s^{-1}-bimodal grain size occurs throughout the wide range of heating temperatures tested. For fast heating conditions – 5 °C . s^{-1}, at heating temperatures of less than 1100 °C, the distribution of the grain size appears bimodal, similar to what occurs for slow heating rates. However, at heating temperatures between 1100–1250 °C a more uniform austenitic grain growth is obtained. The same tendency has been reported by A. Rossi *et al.* [8] on Nb–Ti–N microalloy steels.

The effect of Ti on the austenitic grain size variation for the different temperatures tested is summarized in Fig. 3. In this figure the grain size evolution of a steel without vanadium and titanium is included. This steel (38Cr2), whose chemical composition is also shown in Table I, is a carbon steel with 0.50 wt % of chromium. It was manufactured in an electric arc melting furnace and a vacuum laddle furnace and, finally, rolled to a 36 mm diameter round billet. This steel was formerly used in the forging of automotive components [3], that are now forged in a microalloyed steel. Fig. 3 shows that the influence of the titanium content on the austenitic grain size decreases at low temperature. The finest grain size is found for a titanium percentage of 0.019 wt %. The grain size values indicated in Fig. 3 when bimodal grains appear, are average values. The fact that the grain size decreases as the titanium content goes up to 0.019 wt % (steel 2) is due to the pinning of the grain boundaries produced by fine Ti and (V, Ti) precipitates. From a previously, published paper [5], in which the main precipitate parameters of the three microalloyed steels were studied it can be deduced that the increase of the grain size when the titanium content increased from 0.019 to 0.039 wt % is due to both the fact that the number of titanium precipitates decrease and that they are bigger than in steel 2 in the segregation areas. These coarse precipitates exceed the critical particle size defined by Gladman's equation [9], above which unpinning occurs and the grain grows sharply. Other authors [10, 11] have confirmed that the optimal titanium quantity for grain growth control is between

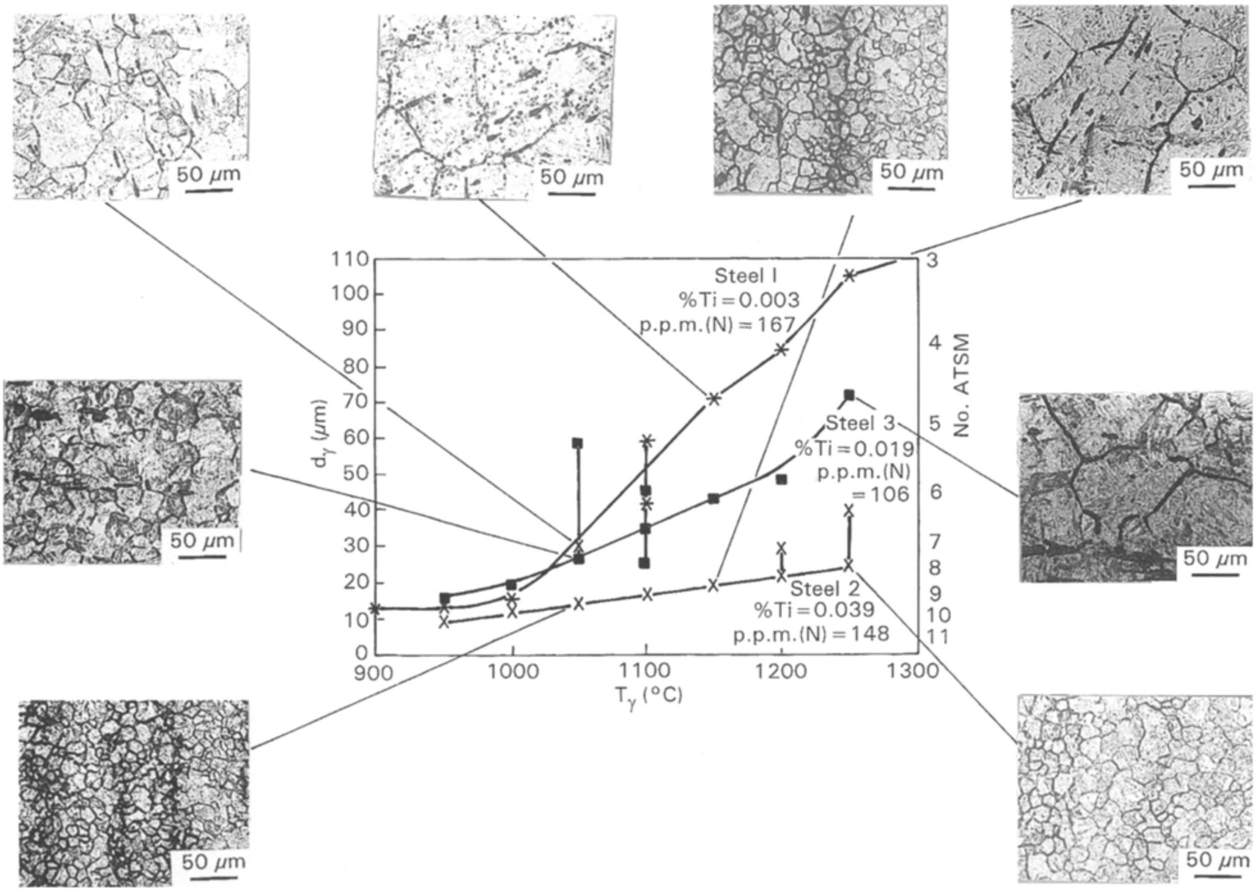

Figure 1 Evolution curves of the austenitic grain size with temperature.

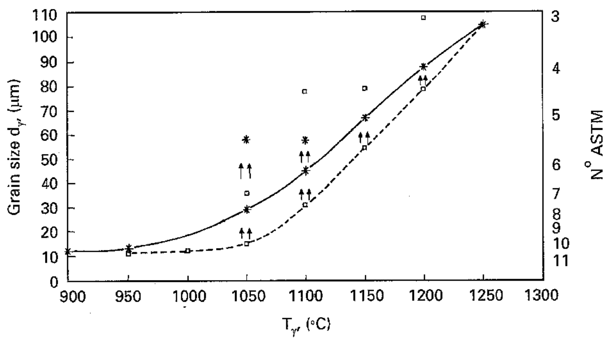

Figure 2 Evolution curves of the austenitic grain size in steel 1 with temperature for two different heating rates (∗) 5 °C . s^{-1} and (□) 0.6 °C . s^{-1}. The symbol ↑↑ denotes bimodal grain growth.

0.010–0.020 wt % and they have reported that if too much titanium was added, at very high temperatures, there was a formation of particles of TiN which were too large to pin the austenite boundaries. Vanadium precipitates do not have any influence on the austenitic grain growth, because these precipitates dissolve at temperatures too low for this to occur. Nevertheless, the vanadium precipitates become fundamental in the precipitation strengthening [10, 11]. The nitrogen content of the three steels (Table I) varies so little that its influence on the austenitic grain size can be considered negligible [7].

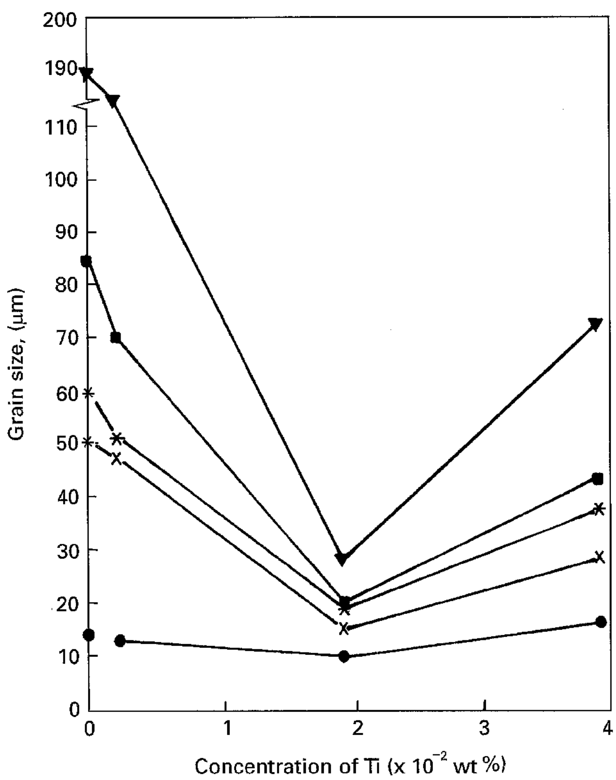

Figure 3 Variation curves of the austenitic grain size with the Ti wt % for the austenitizing temperatures; (●) 950 °C, (×) 1050 °C, (∗) 1100 °C, (□) 1150 °C and (▽) 1250 °C. Note that the d_γ axis values at a 0 % wt % addition of Ti correspond to the steel 38 Cr 2.

3.2. Continuous cooling transformation diagrams (CCT)

The CCT diagrams of the three steels for the 1050 °C and 1250 °C austenitizing temperatures studied are shown in Figs. 4, 5 and 6. Critical temperatures Ac_1 and Ac_3 are plotted in these diagrams by two parallel lines to the time axis. Micrographs of the microstructures at the same cooling rates for both austenitizing temperatures, along with the area percentages of phases and/or microconstituents, are also shown in these figures. CCT diagrams show the transformation of the austenite during cooling at different rates. There is a displacement of the transformation fronts to the right along the time axis as the titanium content

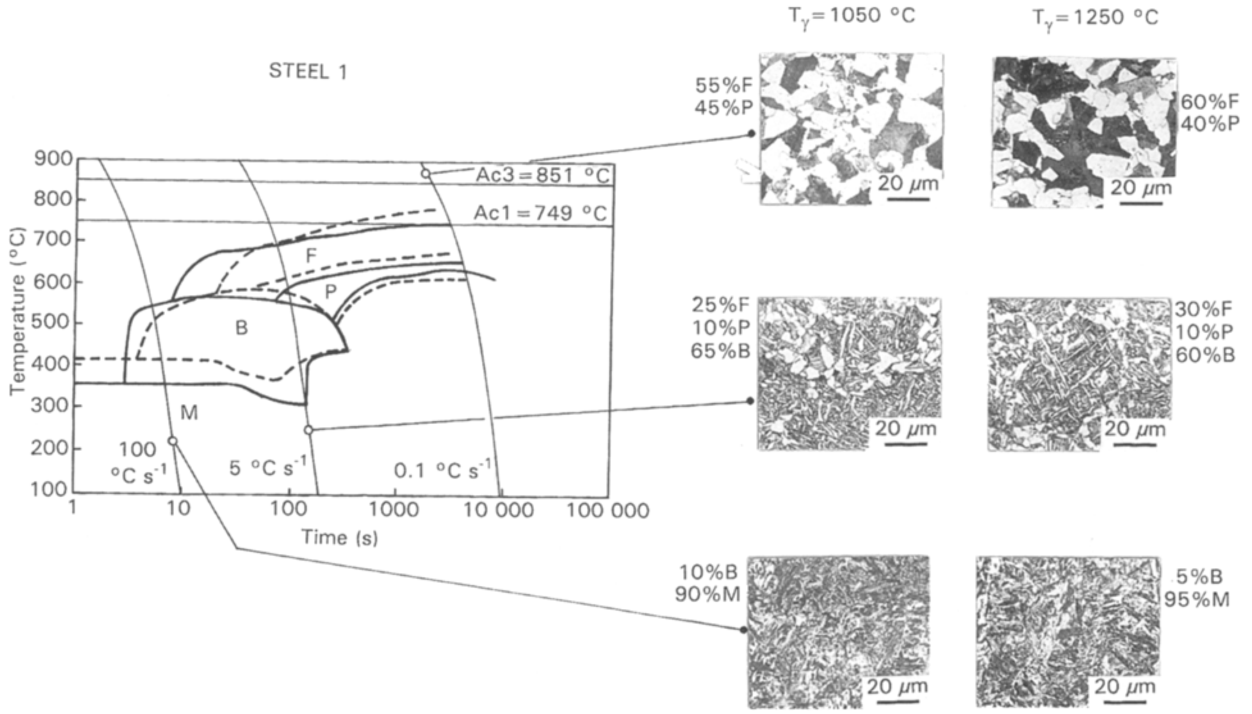

Figure 4 CCT diagram for steel 1. The unbroken line (—) represents the conditions $T_\gamma = 1050\,°C$; $T_G = 30$–$58\,\mu m$ (5–7ASTM) and the dashed line (- - -) represents the conditions, $T_\gamma = 1250\,°C$, $T_G = 105\,\mu m$ (3–4ASTM).

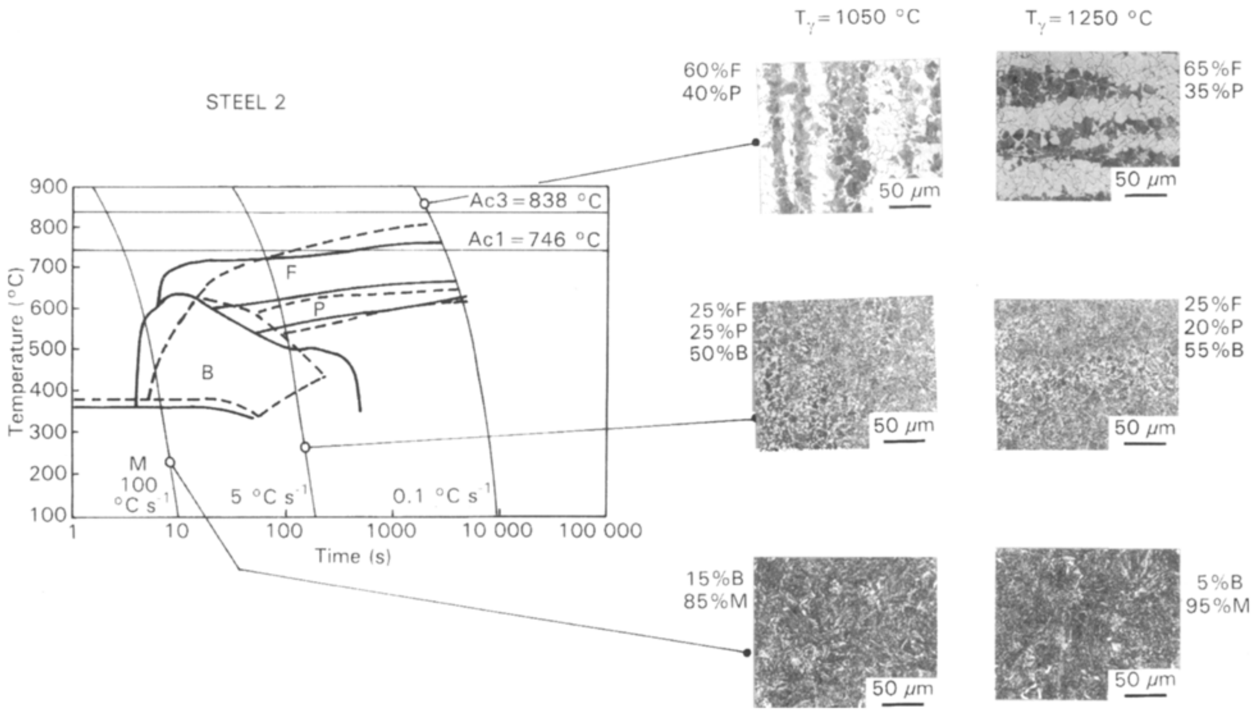

Figure 5 CCT diagram for steel 2. The unbroken line (—) represents the conditions $T_\gamma = 1050\,°C$; $T_G = 13\,\mu m$ (9–10ASTM) and the dashed line (- - -) represents the conditions, $T_\gamma = 1250\,°C$; $T_G = 26\,\mu m$ (7ASTM).

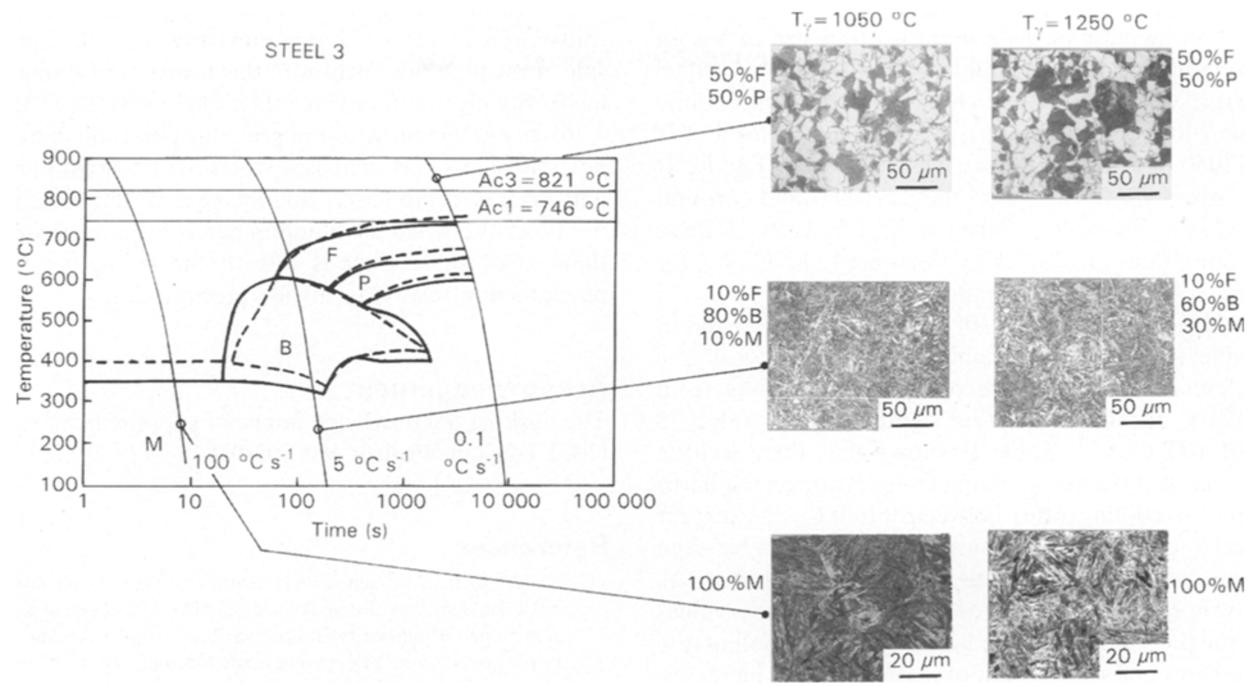

Figure 6 CCT diagram for steel 3. The unbroken line (–) represents the conditions $T_\gamma = 1050\,°C$, $T_G = 28\,\mu m$ (7–8ASTM) and the dashed line (- - -) represents the conditions, $T_\gamma = 1250\,°C$; $T_G = 72\,\mu m$ (4–5ASTM).

TABLE II Hardness of different microstructures

Cooling rates $°C.s^{-1}$	Heating Temp. °C	Steel 1		Steel 2		Steel 3	
		Hardness (HV10)	Structure (%)	Hardness (HV10)	Structure (%)	Hardness (HV10)	Structure (%)
0.1	1250	210	F-60 P-40	188	F-65 P-35	220	F-50 P-50
	1050	215	F-55 P-45	197	F-60 P-40	222	F-50 P-50
5	1250	286	F-30 P-10 B-60	282	F-25 P-20 B-55	370	F-10 B-60 M-30
	1050	290	F-25 P-10 B-65	280	F-25 P-25 B-50	334	F-10 B-80 M-10
25	1250	440	B-25 M-75	400	F-5 B-30 M-65	476	B-10 M-90
	1050	360	F-20 B-50 M-30	330	F-20 P-5 B-55 M-20	536	B-5 M-95
100	1250	523	B-5 M-95	485	B-5 M-95	550	M-100
	1050	485	B-10 M-90	405	B-15 M-85	566	M-100

increases for both austenitizing temperatures tested as has been reported by many authors [12–15]. Nevertheless, in these steels this displacement is almost negligible up to 0.020 wt % Ti. This can be explained by a decrease of the carbon and alloying element concentration in the austenite, owing to an excess of carbides and carbonitride precipitates present in the steel 2 [13]. For an austenitizing temperature (AT) of 1050 °C, the critical martensitic cooling rate, the minimum cooling rate which produces a completely martensitic structure, decreases (while hardenability increases) with increasing titanium content. Critical martensitic cooling rates are over $100\,°C\,.\,s^{-1}$ for steels 1 and 2 and the rate is $35\,°C\,.\,s^{-1}$ for steel 3. As is indicated by the dashed line in Figs. 4, 5 and 6, when the AT is 1250 °C, the critical martensitic cooling rates in the three steels are slower. This is due to the fact that an increase in the austenitic grain size causes an increase in the hardenability of the steels. The critical ferritic cooling rate, which is the minimum cooling rate which produces a ferrite free structure, for an AT of 1050 °C is almost the same for both steels 1 and 2. It is over 75 °C . s^{-1} for these steels and around 8 °C . s^{-1} for steel 3. When the AT is 1250 °C, these cooling rates are 20 °C . s^{-1} for steel 1, 30 °C . s^{-1} for steel 2 and 8 °C . s^{-1} for steel 3.

Hardness values (HV10) have been summarized in Table II, in order to compare the hardness of the different microstructures obtained by cooling from 1050 °C and 1250 °C at four significant rates: 0.1, 5, 25 and 100 °C . s^{-1}. Table II shows that there is little influence of the austenitizing temperature on the hardness for cooling rates between 0.1–5 °C . s^{-1} (except steel 3 for 5 °C . s^{-1} because of the difference between the amount of martensite of the two AT's studied). However, the differences amongst the hardness values of the three steels become higher when the cooling rate increases and the amount of martensite also increases. From these results, it can be concluded that there is a minimal difference if one or other austenitizing temperature is used for cooling rates corresponding to normal post-forging air cooling rates (approx. 2 °C . s^{-1}) [3]. For steel 2 (Ti = 0.019 wt %) the hardness is lower than for steel 1 (Ti = 0.003 wt %) in apparent contradiction to the fact that the increase of hardenability is produced by an increase in titanium content [12–15]. This result can be explained by nitride precipitation. In steel 2, an increase in the hardness by precipitation is not observed because only a part of the nitrogen is combined with titanium. The remaining nitrogen contributes to precipitation strengthening by combining with vanadium. This strengthening, however, is less than in V–N steel with low Ti content. Part of the vanadium bonds with the titanium to form (Ti, V) N (rather than TiN) particles [12, 16]. In addition, the preexisting (Ti, V) N precipitates act as nuclei for equiaxial deposition of VN in austenite. As a consequence of these two reactions, less vanadium is available to precipitate as VN in ferrite [17]. A lower nitrogen content in steel 2 also contributes to the reduction of the VN precipitation [17, 18].

4. Conclusions

(1) A titanium and vanadium microalloy is capable of exerting a significant control on austenitic grain growth of medium carbon steel at very high forging temperatures.

(2) The maximum grain growth control on V–Ti steels with V concentration = 0.10 wt % is achieved when the amount of titanium added to these steels is 0.019 wt %.

(3) In these steels, the higher the heating rate, the slightly higher the austenitic grain size.

(4) For cooling rates around 2 °C . s^{-1}, the influence of the variation of austenitizing temperature between 1050–1250 °C on the hardness of these steels is negligible.

(5) The increasing of the Ti percentage produces the translation of the CCT diagrams towards the right side of the time axis. However, this translation is small in steels with titanium percentages below 0.020 wt %.

(6) In agreement with the previous point, the hardness of these steels increases when the titanium content increases. However, this increase in hardness is not produced when the titanium percentages are lower than 0.020 wt %. That is due to the strengthening phenomenon related to nitride precipitation.

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