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Manganese partitioning and dual-phase characteristics in a microalloyed steel

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A two-stage intercritical annealing treatment has been attempted to develop dual-phase structure and properties in a commercial grade microalloyed steel containing Nb and V. Manganese concentration in austenite enhanced the hardenability which attributes to martensite formation on air cooling after annealing. The volume fraction of martensite and the manganese distribution were found to depend on both annealing time and temperature. Different morphologies of martensite were identified for various intercritical annealing conditions, e.g. dispersed type, ring type, acicular type. The tensile data and behaviour confirmed the dual-phase characteristics in the microalloyed steel.

1. Introduction

Dual-phase steels exhibiting a duplex structure of ferrite and martensite are presently manufactured as thin sheets. Increased hardenability of the steel by alloying can help to obtain dual-phase structure in thick plates. However, this would increase production cost, enhance strength and lower the ductility, affecting ultimately the formability characteristics of the steel. An alternative approach would be to modify the intercritical annealing (IA) process. A number of interesting studies have revealed that significant partitioning of manganese between austenite and ferrite phase occurs during the annealing process [1–6]. Manganese concentration in γ essentially contributes to the hardenability of steel and the carbon content only affects the volume fraction and the nature of martensite formed when γ is quenched to martensite. Austenite formation in low-carbon manganese steel is reported to occur by nucleation and growth of the Mn-enriched phase accompanied by the concurrent dissolution of the Mn-lean high carbon γ transformed from pearlite [5]. Speich *et al.* [1] demostrated that the Mn-enrichment of γ may increase the hardenability near the γ–α interface. A better understanding of the practical intercritical annealing process, particularly the role of the grain boundary on nucleation and growth behaviour of γ during annealing, has been demonstrated by Yi *et al.* [7].

The present investigation aimed to study the effects of a two-stage intercritical annealing process on the commercial grade microalloyed steel containing a slightly higher percentage of carbon. The effectiveness of the IA was studied with respect to the manganese distribution between austenite and ferrite phases and the dual-phase characteristics of the steel. The morphology of martensite formed by IA and air cooling was also investigated.

2. Experimental procedure

Commercially cast and hot-rolled microalloyed steel plate of composition, C 0.18%, Mn 1.48%, S 0.033%, P 0.29%, Nb 0.03% and V 0.037% was used in the present study. Round tensile samples of gauge length 25 mm and the metallographic samples were machined from the steel plate before heat treatment. The tensile testing was conducted in an Instron machine at a cross-head speed of 0.1 cm min^{-1}. Two-stage annealing was given to all the samples tested. The first stage of annealing was given at 765 °C for 2 h to all samples, followed by second stage of annealing at three different temperatures (725, 700 and 680 °C) and holding time (2, 5 and 12 h). The second stage of annealing was followed by air cooling. Microstructural investigation was carried out in both optical and scanning electron microscopes. A few samples were etched in Lepera's reagent [8] in addition to nital etching to confirm the presence of martensite under the optical microscope. TEM work was also carried out on a few samples by using thin foils. Manganese partitioning was studied using quantitative energy dispersive microanalysis at different spots on the microstructure under the SEM. An average of six readings is reported.

3. Results and discussion

3.1. Microstructure

The microstructure of the hot-rolled plate essentially consists of ferrite and pearlite in banded form in the longitudinal direction. The first stage of IA at 765 °C for 2 h followed by air cooling produced martensite in the form of islands, as may be seen in Fig. 1. The martensite islands, although very small in amount, are predominantly situated at ferrite grain boundaries.

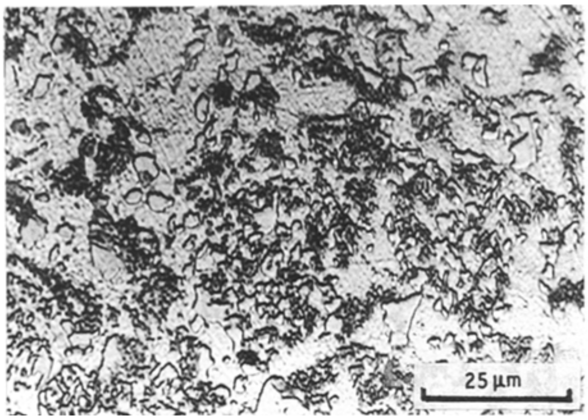

Figure 1 Optical micrograph after single stage intercritical annealing at 765 °C for 2 h followed by air cooling.

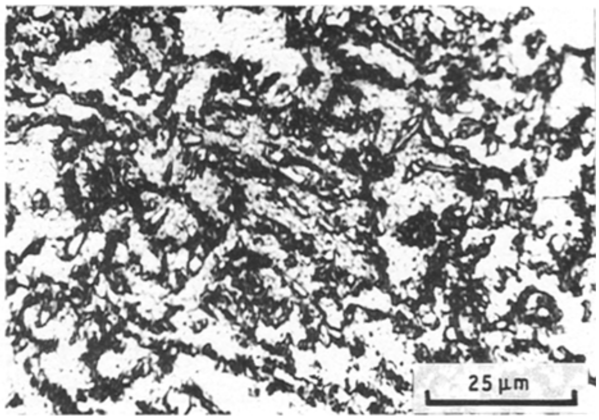

Figure 3 Optical micrograph after second stage of IA at 680 °C for 12h.

The pearlite region is also observed in the microstructure in addition to ferrite and martensite.

The microstructure after a second stage of IA and air cooling is shown in Figs 2–4. In general, a uniform distribution of martensite islands and considerable ferrite grain coarsening may be observed after the second stage of annealing. The pearlite region may also be seen depending on the second stage IA temperature. The martensite islands are mostly present on ferrite grain boundaries. Fig. 5 confirms the presence of martensite islands (white) in a matrix of ferrite when Lepera's reagent was used [8]. Holding time at the second IA temperature seems to have a marginal effect on volume fraction of martensite, but a very significant effect on ferrite grain coarsening. The dark shades, as may be seen in the micrographs (Figs 2–4), are presumably the regions enriched with manganese within the ferrite grains.

The SEM study revealed various morphologies of martensite formed at different IA temperature and time. Three types of morphology could be identified: island or ring type, acicular type and homogeneously distributed dotted type as may be seen in Fig. 6. At relatively lower temperature (~ 680 °C) the islands of martensite formed preferentially on grain boundaries which appear as rings. However, at higher IA temperature the martensite formed appear to be dotted type. The dot-type martensite is found to be distributed on grain boundaries as well as within the grains (~ 725 °C). At intermediate temperature, an acicular type of martensite could be noticed. Morphological differences of martensite have been reported by several investigators depending on the annealing temperature, time and cooling rate. The transformed ferrite growth has been observed to occur by a nucleation and growth process, and not epitaxially [9, 10]. TEM study confirmed the presence of fine martensite in ferrite grain boundaries at high magnification, as shown in Fig. 7 at two magnifications.

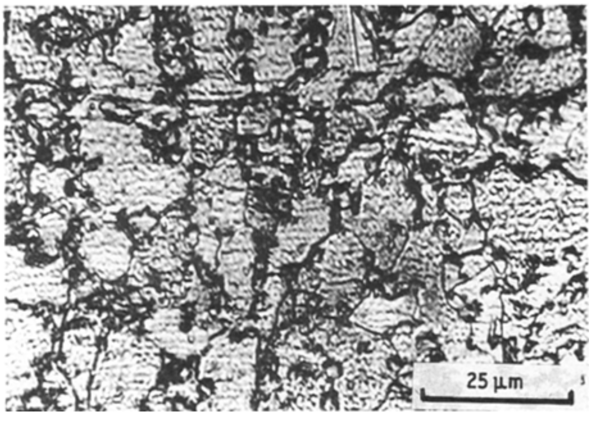

Figure 2 Optical micrograph after second stage of IA at 700 °C for 5 h.

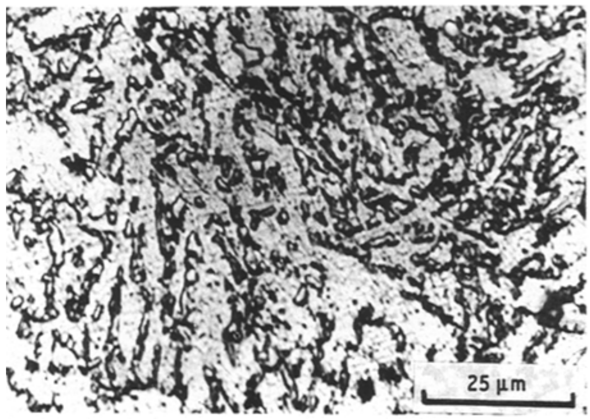

Figure 4 Optical micrograph after second stage of IA at 725 °C for 2 h.

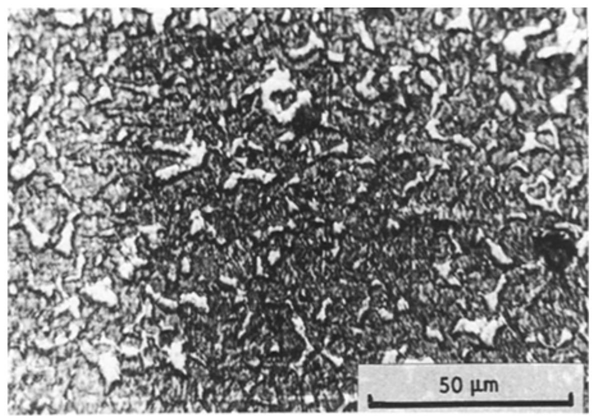

Figure 5 Optical micrograph after etching with Lepera's reagent for IA at 700 °C for12 h.

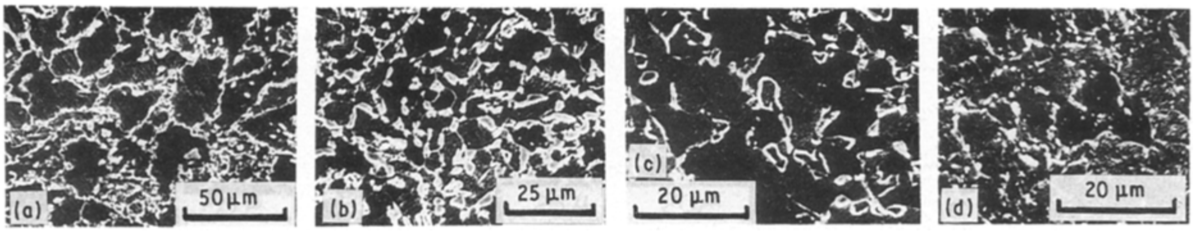

Figure 6 Scanning electron micrographs for IA at: (a) 680 °C, 5 h; (b) 680 °C, 12 h; (c) 700 °C, 5 h; (d) 725 °C, 12 h.

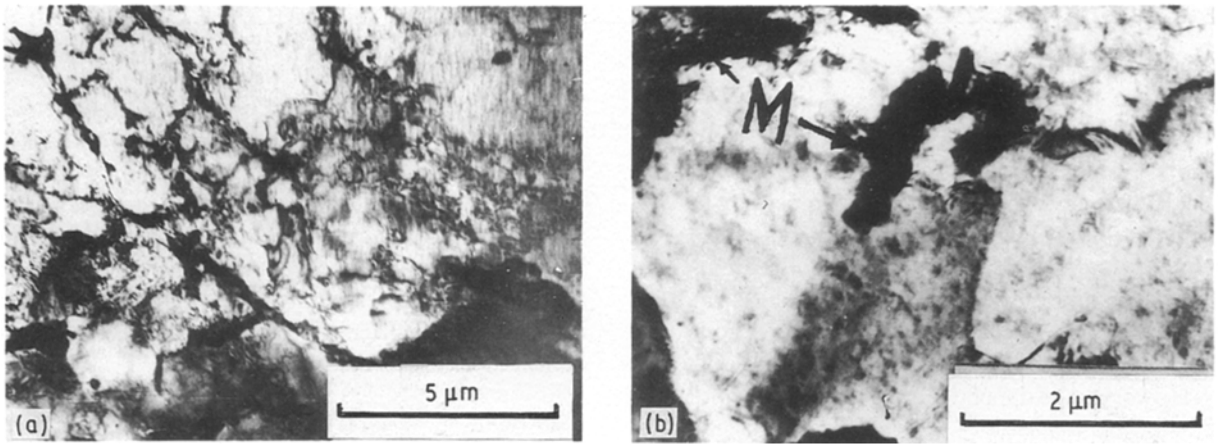

Figure 7 Transmission electron micrograph for IA at 700 °C for 5 h at two magnifications.

3.2. Tensile test

The load–elongation diagram of the as-received material obtained during tensile testing showed discontinuous yielding. In contrast, the intercritically annealed specimens exhibited continuous yielding behaviour with enhanced ductility. This behaviour is similar to that exhibited by dual-phase steels.

Table I shows the typical tensile data for intercritically annealed samples. The per cent elongation values are appreciably improved after intercritical annealing and air cooling in comparison to batch-annealed and quenched material of the same steel, as reported earlier [11]. Increased holding time for annealing decreases the strength values, while the per cent elongation is affected marginally, as may be seen for 700 °C annealing temperature in Table I. The yield ratio values which reflect the extent of strain hardening in the alloys are found to be in the range 0.66–0.75 (Table I). The tensile results obtained from the microalloyed steel are comparable to those exhibited by dual-phase steels after the inter/subcritical annealing treatments.

3.3. Manganese partitioning

Microanalytical measurements using energy dispersive spectroscopy produced the extent of Mn partitioning in ferrite and martensite phases. A few typical results of Mn distribution in ferrite and martensite are reported in Table II. The average Mn concentration in martensite and ferrite is found to be dependent on both annealing temperature and holding time [9]. As may be seen from Table II, the Mn concentration in martensite varies from 1.5%–2.3%, while in the ferrite phase it varies from 0.7%–1.2%. An earlier study confirmed from a thermodynamic point of view the composition partitioning between ferrite and austenite phase during the IA process [3, 7]. Air cooling after IA of the steel under investigation produced martensite due to increased hardenability which may be attributed to the manganese enrichment in austenite during two stages of the IA process. Navara and Harrysson demonstrated that a high hardenability of steels may be achieved even when the IA temperature is lower than A_1 [5]. Manganese redistribution and austenitic growth have been found to occur in three separate stages as demonstrated

TABLE I Typical tensile properties

IA conditions		Yield	Tensile	% El	Yield
Temp. (°C)	Time (h)	strength (MPa)	strength (MPa)		ratio
725	2	430	650	35.0	0.66
700	6	550	837	34.9	0.66
700	12	520	734	33.0	0.71
680	5	418	607	34.2	0.69

TABLE II Martensite volume fraction and Mn distribution in ferrite and martensite phases

IA conditions		Martensite	Mn in ferrite	Mn in austenite
		(Vol %)	(wt %)	(wt %)
Temp. (°C)	Time (h)			
725	2	17.8	1.19	2.04
700	2	15.75	1.10	1.53
700	5	17.2	1.23	1.64
700	12	19.0	0.71	2.33
680	2	10.3	0.70	1.52
680	5	13.0	1.234	1.72
680	12	14.5	1.07	2.2

by Wycliffe *et al.* [4] and Speich *et al.* [1]. The microstructural investigation, as reported above, confirmed the dual-phase characteristics of the steel. The morphological differences in martensite after air cooling may be attributed to the nucleation mechanism and nucleation sites in Mn-enriched austenite phase. Different morphologies of martensite can be formed from the austenite phase after IA because ferrite growth occurs by a nucleation and growth mechanism, and not epitaxially [9]. However, the dependency of martensitic morphology on the nucleation site is still not clear. It is presumed that at relatively higher holding temperature the diffusivity of manganese might have resulted in martensite in the dispersed form, and at a lower annealing temperature (680/700 °C) ring or island-type morphology is favoured. The dispersed martensite obtained at higher temperature may be seen to be distributed both at grain boundaries as well as inside the grains (Fig. 6). At intermediate annealing temperature the acicular type of martensite could be observed. TEM study revealed the presence of fine martensite present at ferrite grain boundaries. Precipitation of any carbide or the presence of a pearlite region could not be observed. Table II also reports the volume fraction of resulting martensite (V_P) obtained by X-ray diffraction studies. Both V_P and the Mn content in martensite may be seen to increase with annealing time at the same temperature. However, a decrease in annealing temperature tends to decrease the Mn content in martensite (Table II), presumably due to the slower diffusion rate. As reported earlier, Mn can diffuse to the centre of austenite grain at a reasonably faster rate at around 695 °C [3, 4].

4. Conclusions

1. A two-stage intercritical annealing treatment followed by air-cooling produced dual-phase characteristics in increased thickness of Nb–V containing microalloyed steel. Manganese partitioning into ferrite and austenite, as revealed by energy dispersive analysis, enhanced hardenability and thus formed martensite. The volume fraction of martensite and the manganese redistribution were found to be dependent on both IA temperature and holding time.

2. Morphological differences of martensite may be attributed to the nucleation mechanism and nucleation sites. Dispersed martensite was obtained at higher annealing temperatures in the second stage, while ring or island-type of martensite was seen at relatively lower annealing temperatures.

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