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Dependence of martensite start temperature on fine austenite grain size

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It has been broadly reported that determination of the martensite start temperature in steels, M_s , requires a complete description of their chemical composition. Recently, several neural networks models considering both chemical composition and austenite grain size (AGS) have been developed. Such models predict a moderate dependence of M_s with AGS. The present work examines the validity of existing neural network models, but focusing on fine AGS (below 5 μ m). © 2007 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

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The temperature at which austenite transforms to martensite during cooling of steels, $M_{\rm s}$, and the factors that affects its value have been broadly investigated. It is well known that $M_{\rm s}$ is strongly dependent on the austenite chemical composition. Over a period of decades the effect on $M_{\rm s}$ of different alloying elements in solid solution, the additive effect of individual elements, and the negligible effect of impurities and small amounts of elements have all been determined [1–6]. Thus, different empirical equations have been reported. All these investigations have shown that carbon is the alloying element with the strongest influence on decreasing of the $M_{\rm s}$ values. However, the effect of austenite grain size (AGS) has not generally been included in such equations.

On the other hand, artificial neural network models have been developed during the last years to predict the M_s from chemical composition alone [7], and from chemical composition and AGS [8]. The AGS values considered in those models range from 5 to 340 μ m in average diameter. The predicted results showed a monotonous and smooth decrease of M_s with AGS, even for AGS values smaller than 5 μ m. It is the aim of this paper to analyze the behavior of M_s with small AGS values in a commercial dual phase steel.

The steel used in this investigation was vacuum melted, cast into a 60 kg ingot and hot rolled. Semi-

rolled slabs, 30 mm thick, were soaked at 1200 °C for 45 min and hot rolled to about 3 mm in several passes, finishing at 900 °C. Two different cooling rates (CR), 7 and 60 °C s⁻¹, and two different coiling temperatures (CT), 500 and 650 °C, were tested in the pilot hot-rolling mill (Table 1). Hot-rolled samples were then cold-rolled to sheets 0.9 mm thick. The steel composition, in wt.%, is Fe–0.15C–1.9Mn–0.2Si–02Cr–0.03Al.

Because of the different combinations of CR and CT during the hot-rolling schedule, four different as-coldrolled microstructures with different amounts of ferrite, pearlite and bainite/martensite were obtained. A set of cold-rolled $12 \text{ mm} \times 2 \text{ mm} \times 0.9 \text{ mm}$ samples were machined parallel to the rolling direction and austenitized at a rate of 5 °C s⁻¹ to different temperatures and times. Some of the austenitization cycles were performed at temperatures very close to Ac_3 (≈ 850 °C for treated samples) in order to reach a very fine AGS. Austenite transforms to martensite during subsequent quenching. For this purpose, the heating and cooling devices of a high-resolution dilatometer were used [9]. The experimental values of M_s were determined from the dilatometric curves registered during quenching. Previous austenite grain boundaries were revealed in those samples by means of a saturated aqueous picric acid plus a wetting agent [10], then, using an image analyzer on acquired optical micrographs, austenite grain size was measured as the mean value as the equivalent circle diameter. Figure 1 shows some examples of those micrographs.

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Table 1. Hot-rolled conditions of tested samples

Sample	CR (°C s ⁻¹)	CT (°C)
S1	7	500
S2	60	500
S3	7	650
S4	60	650

CR, cooling rate; CT, coiling temperature.

Table 2 summarizes the austenitizing treatments, the AGS measured and the experimental M_s values obtained for samples austenitized at temperature close to Ac_3 temperature, where the AGS varies between 2.6 and 4.7 µm. Results suggest that the M_s is very sensitive to small variations in AGS, i.e. small increases in AGS raise M_s significantly. On the other hand, Table 3 reveals that the influence of coarse AGS on M_s is almost negligible. Figure 2, showing the evolution of M_s with fine and coarse AGS, clearly reveals that a slight increase in AGS, of less than 5 µm, produces a substantial increase in M_s , while coarser AGS values have an almost negligible influence on M_s . It is the goal of this paper to explain such behavior.

As was very well documented by Nishiyama [11], the initiation temperature and progress of martensitic transformation are controlled by the chemical and non-chemical free energies of the system. The chemical free energy difference is the transformation driving force and is converted to non-chemical free energy. The latter partly goes into the energy of the inevitable lattice imperfections produced upon transformation.

Several studies [11–15] have reported that in ferrous systems the AGS effect on M_s is likely caused by the reduction of the energy needed for the complementary shear during transformation, which originates in the elimination of lattice imperfections due to the higher austenitization temperature [11]. Likewise, the nucleation of martensite may be boosted by an increase in frozen-in vacancies in the austenite grain due to higher quenching temperatures, which means that the increase in vacancies makes austenite less stable by increasing martensite nucleation sites [11]. Therefore, M_s should increase as austenitization temperature increases.

On the other hand, Machlin and Cohen [12] concluded from studies of Fe–29Ni that martensite transformation is independent of austenitizing temperature with a constant grain size. Thus, the variation in $M_{\rm s}$ is

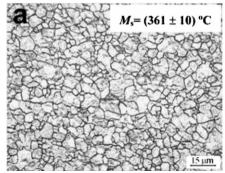
Table 2. M_s evolution with fine AGS

Sample	Soaking temperature (°C)	Soaking time (s)	$M_{\rm s}$ (°C)	AGS (µm)
S1	850 850 850	1 20 100	361 ± 10 364 ± 9 369 ± 10	3.0 ± 1.1 3.6 ± 1.3 3.8 ± 1.5
S2	850 850 850	1 20 100	362 ± 10 366 ± 11 368 ± 10	2.6 ± 1.2 3.3 ± 1.3 3.2 ± 1.3
S 3	850 850	20 100	$\begin{array}{c} 374 \pm 8 \\ 375 \pm 10 \end{array}$	$4.2 \pm 1.7 \\ 4.7 \pm 1.8$
S4	850 850	20 100	$\begin{array}{c} 370 \pm 9 \\ 378 \pm 10 \end{array}$	$3.6 \pm 1.4 \\ 4.7 \pm 1.6$

Table 3. M_s evolution with coarse AGS

Sample	Soaking temperature (°C)	Soaking time (s)	<i>M</i> _s (°C)	AGS (µm)
S1	950	20	380 ± 9	4 ± 1
	1050	20	379 ± 10	7 ± 1
	1150	20	385 ± 11	21 ± 3
	1200	20	390 ± 8	29 ± 4
S2	950	20	372 ± 8	4 ± 1
	1050	20	375 ± 11	6 ± 1
	1150	20	385 ± 12	18 ± 2
	1200	20	392 ± 9	25 ± 3
S3	950	20	382 ± 9	5 ± 1
	1050	20	390 ± 10	7 ± 1
	1150	20	373 ± 11	18 ± 3
	1200	20	388 ± 10	26 ± 3
S4	950	20	384 ± 9	6 ± 1
	1050	20	386 ± 8	10 ± 1
	1150	20	390 ± 11	26 ± 3
	1200	20	393 ± 10	24 ± 3

due to grain size variability. This is consistent with the results presented by Kitahara et al. [16], who showed that the M_s decreases as austenite grain size is reduced. For that purpose, the authors produced a sub-micron austenitic microstructure increasing the number of the accumulative roll bonding (ARB) cycles in an Fe–28Ni alloy. The tendency to decrease M_s as AGS does is in line with our results. However, it should be noted that the reported ultrafine grain structures were produced



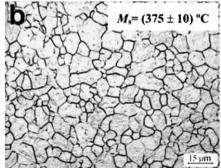


Figure 1. Optical micrographs of: (a) Sample S1, AGS = $(3.0 \pm 1.1) \, \mu m$, (b) Sample S3, AGS = $(4.7 \pm 1.8) \, \mu m$. Etching with saturated aqueous picric acid [10].

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