Metallurgy and continuous galvanizing line processing of high-strength dual-phase steels microalloyed with Niobium and Vanadium

C. I. Garcia, M. Hua, K. Cho, K. Redkin, A. J. DeArdo

It is well-known that the automobile industry continues to search for stronger, more cost-effective steels to lower the mass of the vehicle for better fuel consumption and to provide better crash worthiness for safety. This movement to higher UTS strength requirements, from the 590-780 range to over 980 MPa, has led to more complex alloy design. In the processing of these steels on continuous, hot-dipped, galvanizing lines (CGL), two major changes in composition have been the addition of hardenability elements and microalloying. For example, very-high strength DP steels, containing high Mn, Cr and Mo along with Nb and V have shown UTS levels in excess of 1100MPa. This paper will present recent research conducted on four experimental steels containing these additions. It will be shown that the choice of intercritical annealing temperature is important when processing microalloyed DP steels, as are the rates of cooling throughout CGL processing. The physical metallurgy of producing ultra-high strength DP steels on CG lines will be presented and discussed.

Keywords:
galvanizing, automotive industry, physical metallurgy, high strength dual-phase steel

INTRODUCTION

It is well-known that the design of engineering components must incorporate materials with a high resistance for both elastic deflection and yielding. For automobiles, the final parts may also need to be aerodynamic and corrosion resistant. In the continuing efforts to reduce the mass of the automobiles for fuel efficiency and increase the safety of the passengers, steels of higher strength continue to be attractive. Since most forming operations in autos involve plane strain bending or stretch forming, the ideal steels must have moderate yield strength to improve die life, and good work hardening, as measured by the YS/UTS ratio or instantaneous "n values". In addition, candidate steels must also have good sheared edge ductility and an acceptable bake hardening response, if painted. Finally, the steel must be available to the fabricators or assemblers from multiple sources and at a competitive price.

The modern dual-phase steels (DP) satisfy these requirements fairly well. Today, DP590 and DP780 are commercial realities, while DP980 and DP1180 are still being investigated. The goal of this paper is to describe research that has been conducted on DP steels at the 980 and 1180 MPa UTS level that have been microalloyed with Nb and V and processed using a CGL simulation on a Gleeble 3500.

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The strength of DP steels at room temperature after complete processing on CG lines is normally assumed to be controlled by two main factors: the amount of martensite or lower bainite, and the hardness or strength of the ferrite (1,2). The ductility is also controlled by several factors, chief among them being the state of recovery or recrystallization of the cold rolled ferrite that occurs during the anneal. Clearly, the final microstructure and properties can be influenced by several factors: the bulk composition, the hot band microstructure, the annealing temperature, the cooling rate to the zinc-pot temperature, the time at 460°C and the final cooling rate to room temperature. Since these steels contain high levels of Mn and other additions such as Cr and Mo, for hardenability purposes, the microstructural banding of the martensite presents challenges to optimization of the system (3). Finally, since the surface quality and spot weldability are important features, both the Si and C levels must be kept to a minimum (4).

This paper describes research that has been conducted on low carbon steels containing high Mn, Cr, Mo and Al. In addition, the steels were microalloyed with Nb, Nb+V or Nb+V+N. After CGL processing, these steels exhibited UTS values between 820 and 1300 MPa, and total elongations (corrected for sub-sized gauge length) between 22 and 13%. The microstructural observations and processing-microstructure-property relations will be presented and described below.

EXPERIMENTAL PROCEDURE

The compositions of the 45Kg vacuum-melted ingots are shown in Table 1. Using these bulk composition and using annealing temperatures near 780°C, and assuming equilibrium at the Ac_3, the transformation temperatures for the intercritally formed austenite are shown in Table 2. These data were calculated using the thermokintetic software JMat Pro (5).

These ingots were reheated to 1200°C and rolled to 3mm in several passes, finishing at 920°C. They were then water sprayed

Element/Steel	DPTR0(Base)	DPTR1	DPTR2	DPTR3
C	0.15	0.144	0.145	0.156
Mn	1.76	1.445	1.751	1.726
Si	0.42	0.398	0.403	0.398
P	0.01	0.008	0.007	0.008
Al	0.064	0.024	0.021	0.15
V	-	0.06	0.062	0.101
Nb	0.024	0.02	0.019	0.019
Cr	0.52	0.499	0.502	0.498
Mo	0.31	0.301	0.303	0.303
N	0.006	0.0063	0.0142	0.0133
Si	0.0067	0.0036	0.004	0.0036

TAB. 1
Chemical Composition of V-DPTR Steels.
Composizione chimica degli acciai V-DPTR.

ID	IAT, °C	V.F of γ, %	Ms, °C	M_{50}, °C	Bs, °C
DPTR1	780	50.6	334	298	547
DPTR2	770	51.7	317	281	527
DPTR3	780	55	325	289	537
Base	770	53.6	311	276	529

TAB. 2
JMat Pro Predictions of Phase Volume Fractions and Transformation Parameters at the IAT.
Previsioni con software JMat Pro delle frazioni di fase in volume e dei parametri di trasformazione al IAT.

cooled at 10-15°C/sec to the coiling temperature of 550°C. After coiling and cleaning, the hot band was cold rolled about 60% to 1.2mm sheet. Following cold rolling, the sheets were sectioned for the Gleeble simulation of the CGL GI processing. They were then heated at 10-60°C/sec to the soak temperature that varied from 740 to 790°C, after which they were cooled at rates 10-60°C /sec to the zinc-pot temperature of 460°C, held 15 sec, then air cooled to room temperature. Other specimens were immediately up-quenched to 520°C from 460°C to replicate the GA process. Still other specimens were cooled at 40°C /sec from 460°C or 520°C to room temperature.
The microstructural analysis of all the intercritically annealed specimens was conducted using standard optical and electron optical techniques (OM, SEM and TEM). The samples for TEM analysis were cut in cross-section at an inclined angle to the rolling surface using the diamond precision saw. The cross-section specimens were polished using grinding papers to make both surfaces of the specimens parallel to each other, and then chemically thinned to 100 m. Finally, thin foil specimens for TEM and STEM analysis were prepared via conventional twin-jet polishing.

MECHANICAL TESTING

The standard tensile properties of the specimens resulting from the CGL simulation treatments were evaluated using a computer-controlled servo-hydraulic machine. Two ASTM sub-size tensile specimens (25.4 mm nominal gage and 6.4 mm nominal width) per condition were tested at a cross-head speed of 2 mm/min. The bulk hardness of the specimens treated by CGL simulation was evaluated using a load of 300g. The total elongations were corrected to a 50mm gauge length.

FIG. 1
Microstructure of Hot-Band DPTR0 Steels.
Microstruttura di nastri di acciaio DPTR0 laminato a caldo.

RESULTS AND DISCUSSION

Metallography

The starting HRB microstructure is shown in Fig. 1 for DPTR0 Steel. The microstructure is a mixture of bainitic ferrite and MA or granular bainite.
After cold rolling and full GI processing, the resulting microstructiures are shown in Figure 2 for the base steel DPTR0 (Nb) and steel DPTR2 (Nb+V+N). The combination of the V plus N and the Nb has resulted in a remarkable refinement in microstructure. It should be noted that this intense refinement was not observed in the absence of higher nitrogen.

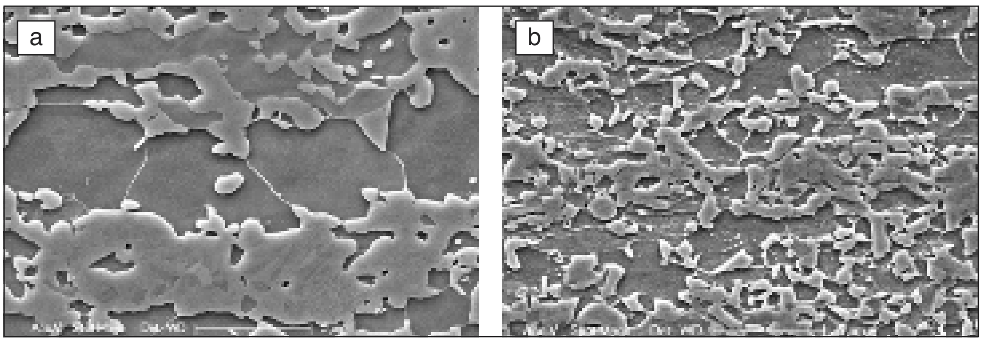


FIG. 2
SEM Micrograph of (a) Base and (b) DPTR 2 Steels As-Water Quenched Condition.
Micrografia SEM di acciai (a) di base e (b) DPTR 2 nella condizione di temprato in acqua.

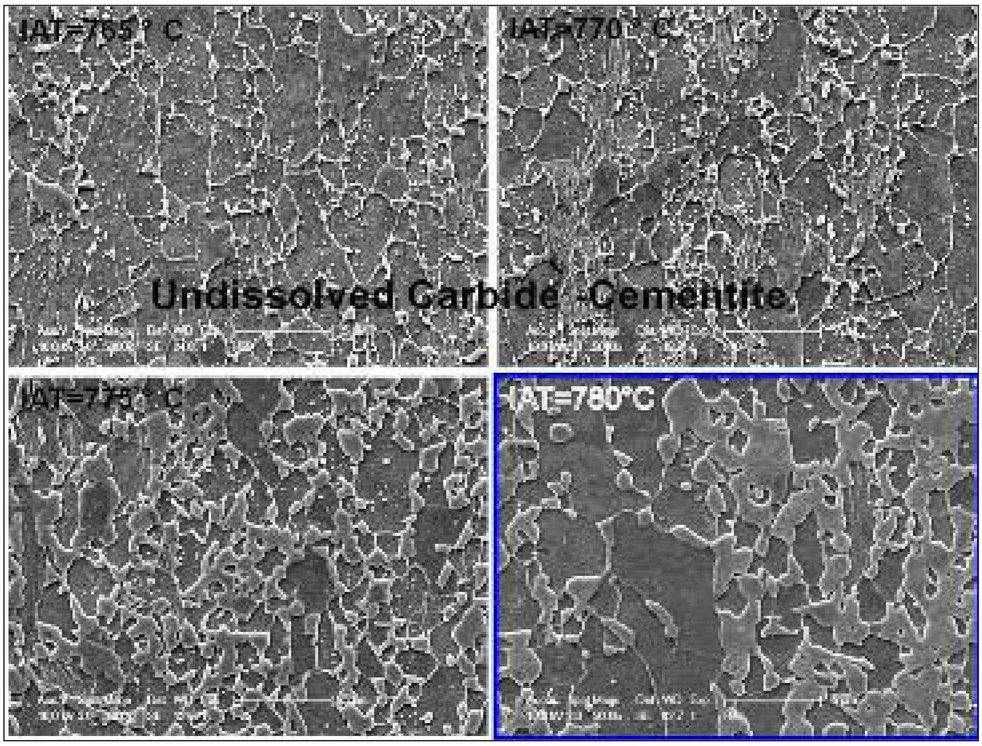


FIG. 3
SEM Micrograph of DPTR1 Annealed at Different Intercritical Annealing Temperatures.
Micrografia SEM di acciaio DPTR 1 ricotto a diverse temperature intercritiche di ricottura.

Tq	460°C	
IAT	740°C	770°C
UTS, MPa	909.5	1298
Elong.%	16.4	12.3

TAB. 3 ***Comparison of the Mechanical Properties of DPTR0 (Base) Steel Annealed at 740°C and 770°C.***
Confronto fra le caratteristiche meccaniche dell'acciaio DPTR0 (Base) ricotto a 740°C e 770°C.

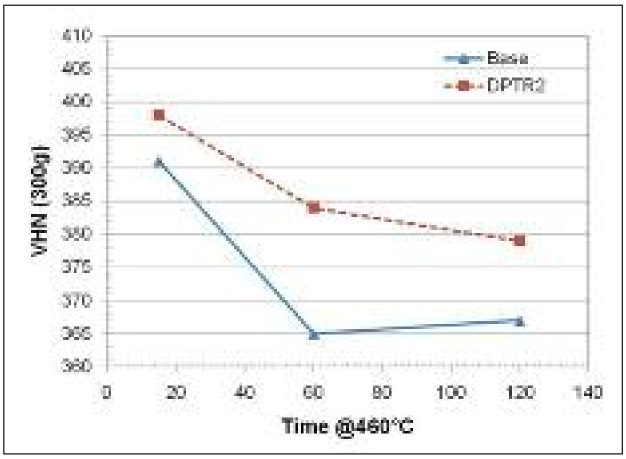


FIG. 4 ***Hardness vs. Isothermal time @ 460°C.***
Durezza vs. tempo di trattamento isotermico a 460°C.

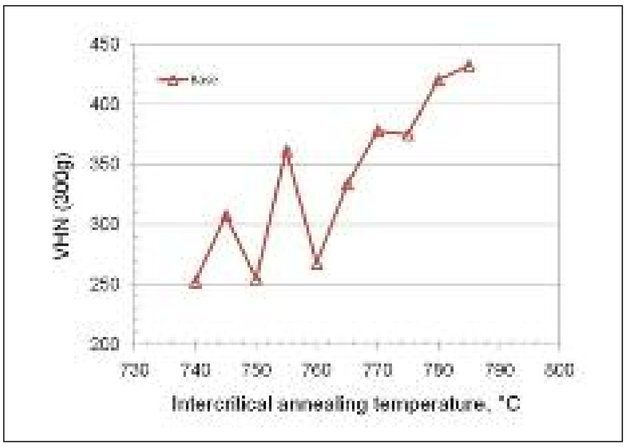


FIG. 5 ***Bulk Hardness vs. IAT for DPTR0 (Base) Steel.***
Durezza vs. IAT per l'acciaio base DPTR0.

Earlier studies have shown a marked effect of annealing temperature on microstructure and properties (6). In this study, there was a pronounced increase in the amount of martensite found at room temperature in annealed and quenched specimens of simple C-Mn DP steels as the annealing temperature was increased from 750 to 775°C austenite. This effect was attributed to the dissolution of Fe_3C during the anneal (6). To investigate the possibility of a similar effect in microalloyed DP steels, steel DPTR1 was annealed at different temperatures and quenched to room temperature at 40°C /sec, immediately after the anneal. After intercritical annealing steel DPTR1 in the range 760-775°C, the microstructure consisted of ferrite, bainite, martensite and undissolved Fe_3C. Only when the steel is soaked at 780°C, did most of the modified Fe3C go into solution. This is exhibited in Fig. 3 in the case of steel DPTR1. Clearly 780°C is the dissolution temperature of the Fe_3C modified by the alloying, especially, the Nb and the V in this steel (7). Annealing below this dissolution temperature causes much lower bulk hardness and strength, Table 3. It is interesting to observe the increase in the volume fraction of austenite at the annealing temperature as the carbides go into solution with increasing temperature. However, it is important to note that once the carbide is dissolved, grain coarsening of both the ferrite and the austenite occurs, as shown for 780°C, Figure 3.
Since the B_S temperatures of the pools of intercritically formed austenite in the steels are above 500°C, Table 2, quenching these

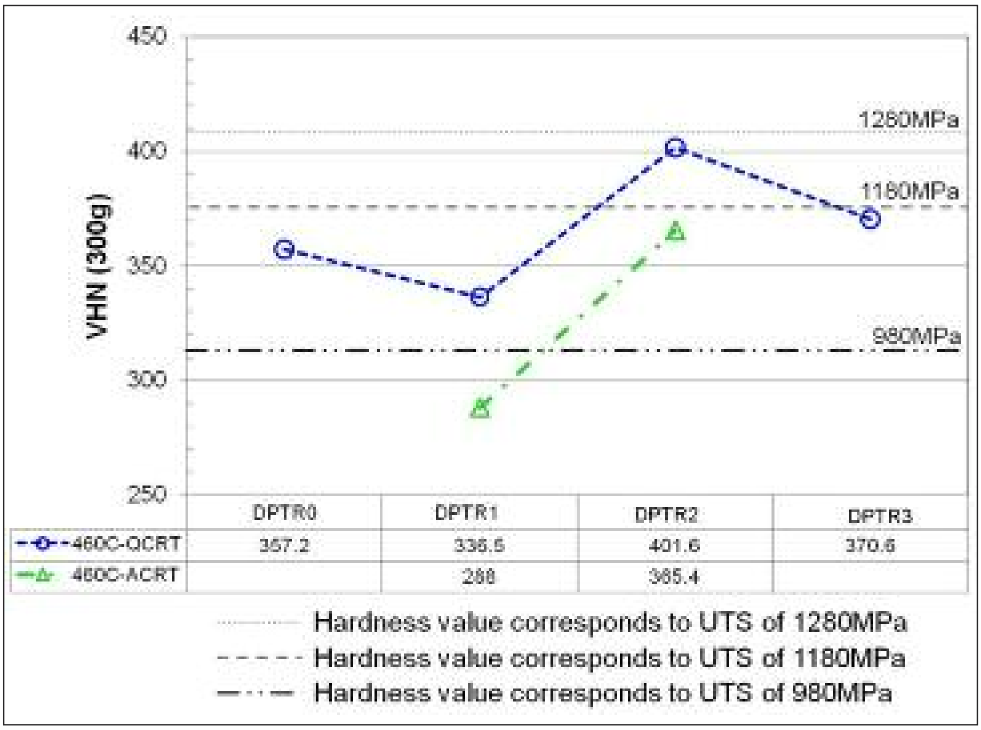


FIG. 6
Effect of cooling rate from isothermal hold at 460°C to RT" QCRT = 40°C/sec, ACRT = 5°C/sec.

Effetto delle velocità di raffreddamento fino a RT", dopo mantenimento isotermico a 460°C, QCRT = 40°C/sec, ACRT = 5°C/sec.

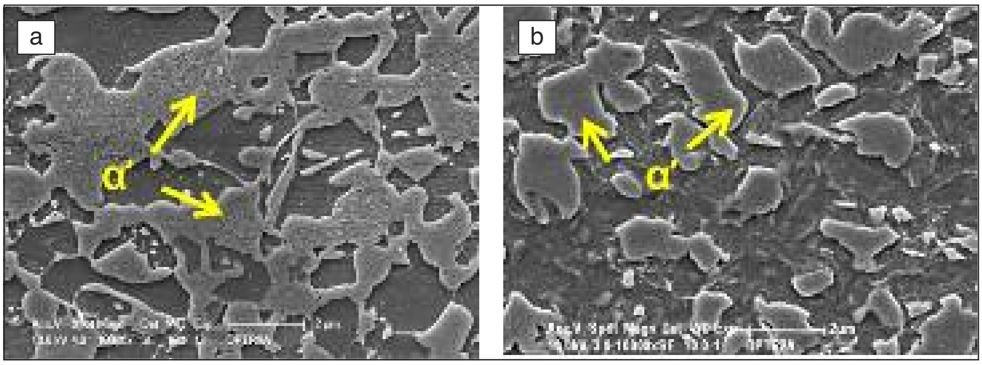


FIG. 7
SEM Micrograph of (a) Base and (b) DPTR2 (770°Cx60sà460°Cx15s–QCRT).

Micrografia SEM di acciaio (a) Base e (b) DPTR 2 (770°Cx60sà460°Cx15s–QCRT).

steels from 780°C to 460°C should result in the formation of bainite. Upon holding at 460°C, the steels were observed to soften with holding time, Fig. 4. However, the addition of the V and N to the Nb steel considerably slowed this softening.
The influence of annealing temperature on the bulk hardness of the fully processed DPTR0 steel is shown in Fig. 5. The effect of composition on the bulk hardness of the DP steels after annealing , cooling to and holding at 460°C for 15 sec, then either quenching (40°C/s) or air cooling (5°C/s) to room temperature is shown in Fig. 6. The much higher VHN values for the quenched specimens (ΔVHN ~50-80), clearly indicates that a large amount of untransformed austenite remains after the 15 second hold at 460°C. It should be noted that the amount of martensite found in the conditions of Figure 7 cannot explain the differences in VHN levels shown in Fig.6. This martensite is presented in Figs. 7 and 8.
The microstructure of the base steel DPTR0 (Nb only) shows a mixture of annealed ferrite, fresh martensite, probably formed during the quench, and lightly tempered bainite, Fig. 7a. Fig. 7b shows the microstructure of steel DPTR2 (Nb-V-N), and exhibits phases and micro-constituents similar to DPTR0. It should be noted, however, that the martensite island size in DPTR2 is much finer than that in DPTR0. In these steels, nano-hardness readings revealed that the annealed ferrite had a hardness of ~ 4.3

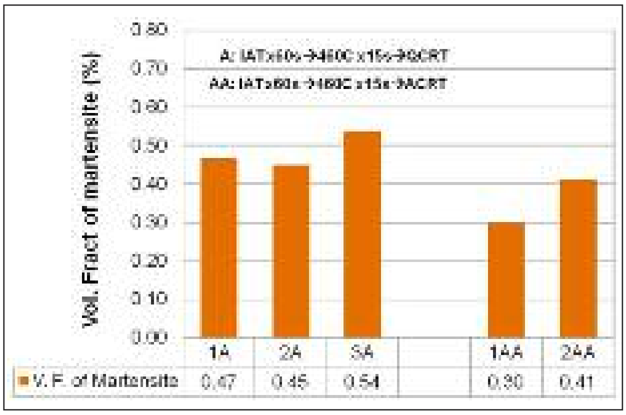
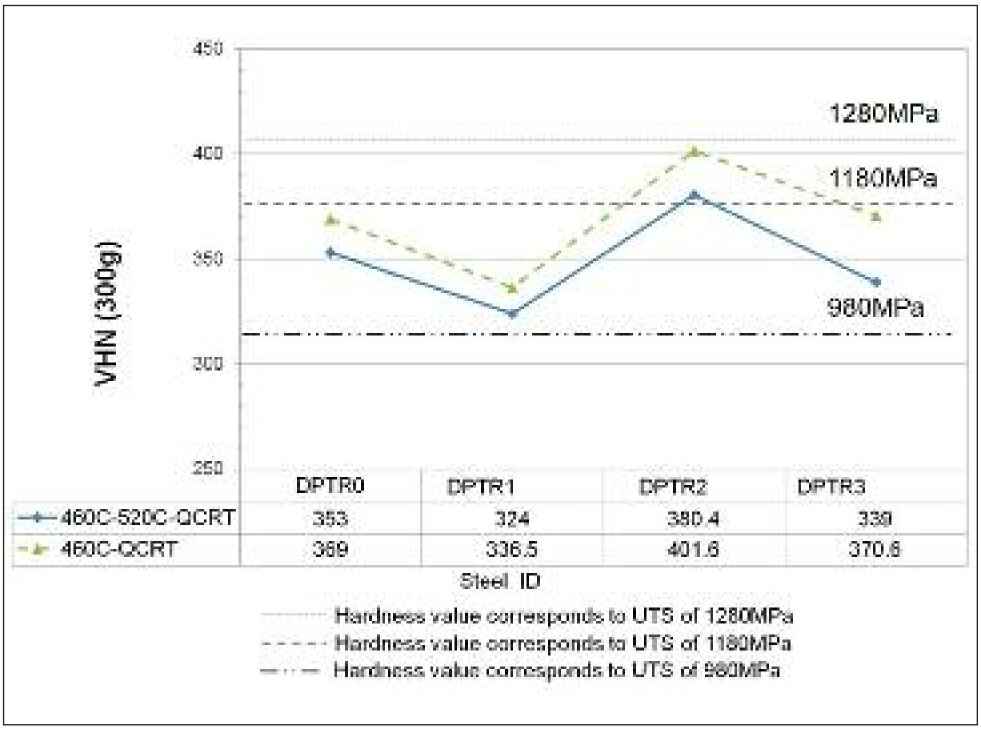


FIG. 8 ***Second Phase Volume Fraction of DPTR Steels Treated by Gleeble CGL Simulation.***

Frazione in volume della seconda fase di acciai DPTR trattati mediante simulazione di CGL Gleeble.

GPa, bainite ~ 9.4 and martensite ~ 12.1. Using similar specimens, no evidence of retained austenite could be found from X-ray diffraction data.
The influence of cooling rate from 460°C, after the GI treatment,

FIG. 9
Influence of Cooling Rate from 460 or 520°C to room temperature. The measured amounts of martensite found in these specimens are shown in Figure 8.
Influenza della velocità di raffreddamento da 460 a 520°C a temperatura ambiente. Le quantità di martensite trovate nei provini sono mostrate in Fig. 8.



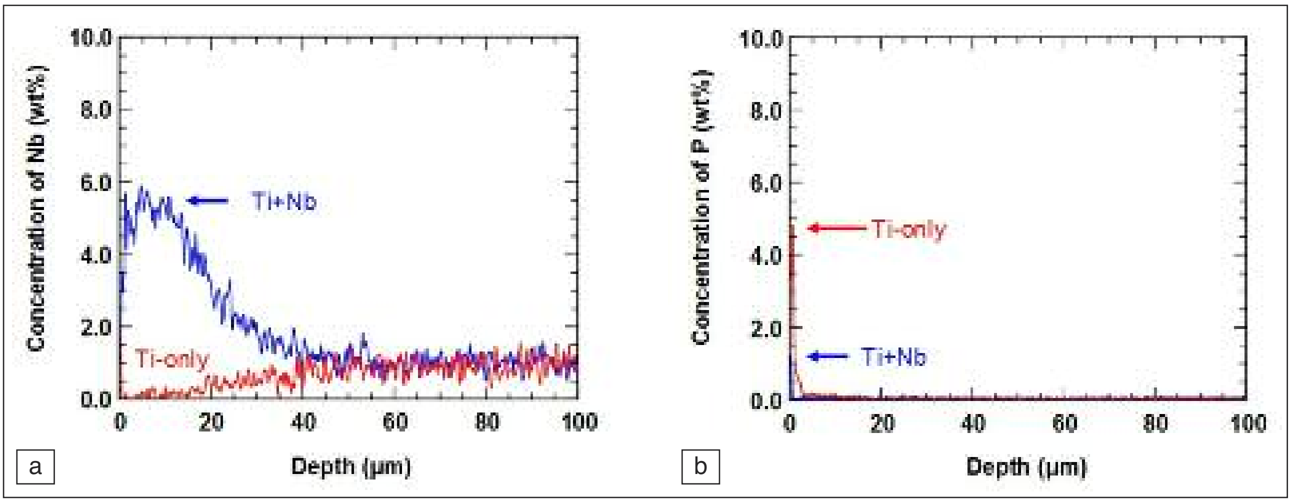


FIG. 10 ***Glow Discharge-Optical Emission Spectroscopy (GD-OES) showing surface enrichment of a) Nb in the Ti+Nb IF steel and b) P in the Ti-only IF steel.***
Spettroscopia ad emissione ottica e scarica luminescente (GD-OES) che mostra l'arricchimento superficiale a) in Nb nell' acciaio Ti+Nb IF e b) in P nell'acciaio IF con solo Ti

to room temperature was also studied. The amount of martensite in the microstructure at room temperature after air cooling or quenching from 460°C is shown in Fig. 8. Two obvious conclusions can be drawn from Fig 8. First, that there remains a substantial amount of untransformed austenite after the 15 second hold at 460°C. And second, the presence of this austenite causes a cooling rate effect on hardness upon cooling from 460°C to room temperature.
To simulate GA processing, the GI specimens were immediately up-quenched from 460 to 520°C and held 15 sec, then quenched to room temperature. The four experimental steels were subjected to this processing and the bulk VHN data are shown in Fig. 9. The data of Fig. 9 indicate that there is a softening of about 15-30 VHN in going from GI to GA processing in this study, and that the steel containing Nb + V+N (DPTR2) showed the highest hardness in both conditions. According to Fig. 9, DPTR2 can be expected to reach the DP1180 strength range.
There are additional benefits to the addition of MAE to DP steels, especially for CGL processing. Elements such as Nb and V, when present as solute in ferrite, are surface active, and segregate to boundaries and free surfaces (8). An example of solute Nb in an interstitial-free steel after CGL processing is shown in Fig. 10 (8). These data, generated using the GDOES technique, show that not only is the Nb heavily segregated to the free surface, but also that it has strongly lowered the sulfur and phosphorous contents at the same surface. This segregation effect is the core of the good adherence and anti-powdering behavior of Nb treated IF steels (9). As long as the Nb is in solution, we

might also expect similar behavior in DP steels. Furthermore, steels containing V in solution might also be expected to exhibit similar behavior.

SUMMARY AND CONCLUSIONS

1. The intercritical annealing temperature (IAT) influences the strength of DP steels because undissolved carbides decrease both the amount and carbon content of the austenite. The minimum intercritical annealing temperature should be equal to or higher than the carbide dissolution temperature.
2. The presence of V and N increases the hardenability of the austenite formed in DP steels annealed at IAT.
3. DPTR2 steel, which contains 0.019Nb-0.06V-0.0142N, has the highest hardness compared to other DPTR steels for the same CGL process.
4. Based on hardness results, it is predicted that Nb-V-N DP steels can reach the strength of 980 MPa by quenching to 460°C in either the GI or the GA process.
5. The decrease of hardness in Nb-V-N DP steel after up quench or tempering is less than that of Nb – DP steel.
6. V additions alone do not seem to increase resistance to softening or tempering, whereas combined V-N additions are more effective.

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Abstract

Metallurgia e trattamento su linea continua di zincatura di acciai bifasici ad alta resistenza microalligati con Niobio e Vanadio

Parole Chiave: trattamenti superficiali, automotive, metallurgia fisica, acciaio

È ben noto che l'industria automobilistica continua a cercare acciai sempre più resistenti, e meno cari, per abbassare la massa dei veicoli al fine di limitare il consumo di carburante e di fornire migliori garanzie di sicurezza in caso di incidente. Questa tendenza a innalzare i requisiti di resistenza UTS, da 590-780 a oltre 980 MPa, ha portato a una più complessa progettazione delle leghe. Nella messa a punto di questi acciai per linee di zincatura a caldo continue (CGL), due modifiche importanti nella composizione sono costituite dall'aggiunta di elementi indurenti e dalla microalligazione. Per esempio, molti acciai DP ad alta resistenza, contenenti alti tenori di Mn, Cr e Mo insieme a Nb e V hanno esibito livelli di UTS oltre i 1100MPa. Questo documento presenta una recente ricerca condotta su quattro acciai sperimentali che contengono questi additivi. Viene dimostrato che la scelta di temperatura intercritica di trattamento è importante durante la manipolazione degli acciai microlegati DP, come nel caso della velocità di raffreddamento durante il processo CGL. Sono presentati e discussi i principi di metallurgia fisica per la produzione di acciai DP ad ultra- alta resistenza per linee CGL.