LOW CARBON MICROALLOYED COLD ROLLED, HOT DIP GALVANIZED DUAL PHASE STEEL FOR LARGER CROSS-SECTIONAL AREAS WITH IMPROVED PROPERTIES

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ABSTRACT

Hot-dip galvanized dual phase steels are increasingly gaining relevance within the automotive industry. The material properties of these steels were initially defined through standardization 10 years ago (SEW097 followed by DIN EN 10336, currently DIN EN10346). The practical range of dimensions used by customers was initially limited to a width smaller than 1500 mm and a thickness between 1,0 mm and 2,0 mm. Due to good experiences made by customers with regard to dual phase steel, different types of applications were established accompanied by an extension of the original physical dimensions towards both smaller and larger sizes. This circumstance revealed consequences for the entire process chain from steelmaking, over hot rolling, cold rolling, all the way to hot-dip galvanizing. The influence of microalloying with niobium for the steel grade HCT600D having a carbon content of less than 0.1 % is discussed in the following example. An outlook on a principally analogous production of HCT780X and HCT980X with selected micro-alloy combinations concludes the presentation.

KEYWORDS

Dual phase steel, niobium microalloying, hot rolling conditions, annealing parameters, large cross-section, hot-dip galvanizing, surface conditions

INTRODUCTION

Dual phase (DP) steel is being increasingly applied by carmakers initially covering tensile strength levels between 500 and 600 MPa either as hot rolled or as cold rolled grade. Currently the share of DP steel in a car body can reach in the order of 20% of the total body weight. The recent introduction of DP steel with a tensile strength of 1000 MPa (DP980) allows further reduction the vehicle weight.

The optimum microstructure and hence properties of cold rolled hot-dip galvanized DP steels require an exact adjustment of chemical analysis, hot rolling conditions, cold reduction and annealing conditions. Practical experience revealed that the applicable processing windows can vary depending on the chemical analysis. Already small deviations from the ideal processing route of non-microalloyed DP steels result in inhomogeneous microstructure, banding, varying mechanical properties and severe anisotropy. Consequently, limitations in the manufacturing properties (sometimes directionally dependent) and premature damage are being observed.

The current study investigates if Nb microalloying and simultaneous lowering of the carbon content succeeds in reducing the mentioned critical behaviour. In this regard suitable hot rolling conditions (finish rolling temperature, coiling temperature), cold reduction as well as annealing and cooling conditions during hot dip galvanizing have to be determined.

1. PRINCIPAL EFFECTS OF NB IN DP STEEL PROCESSING

Whether the processing route is adapted to Nb microalloying or not, the effect of Nb will be always noticed when producing DP steel. Therefore it is important to perform a holistic consideration of possible effects of Nb during the entire process chain and then to optimize individual processing steps.

The foremost and best known effect of Nb is recrystallization delay during hot rolling leading to pancaking of the austenite. The pancaked austenite transforms into a fine-grained polygonal ferrite and dispersed pearlite islands under conventional coiling conditions. This condition is usually chosen if the hot strip is destined for further cold rolling followed by intercritical annealing. The refined ferritic-pearlitic microstructure is inherited to the final material exposing a finer grained ferrite matrix embedding smaller martensite islands [1, 2].

When the coiling condition is set to directly produce DP steel from the rolling heat, the effect of the pancaked austenite is to enhance ferrite nucleation due to the larger total austenite grain boundary area [3]. This allows obtaining the desired amount of ferrite in a shorter time and thus helps optimizing the cooling path on the run-out table. Naturally, the ferrite as well as martensite grain sizes are also reduced.

Another option is coiling in the temperature range of 500 to 550°C. This results in a bainitic structure of the hot strip [1]. A significant portion of Nb remains in solid solution under this coiling condition and is available for precipitation during subsequent intercritical annealing [4].

The influence of Nb microalloying noticed during cold rolling is a direct consequence of the microstructure resulting from the coiling condition after hot rolling. Since the addition of Nb leads to grain refinement, the yield strength of conventional ferritic-pearlitic hot strip will be increased [2]. This reflects in higher cold rolling forces necessary to achieve the desired cold reduction. The yield and tensile strength of the cold rolled full hard strip are also increased compared to Nb-free material and depends on the degree of cold reduction. If the hot strip was coiled to bainitic microstructure, the yield strength can become so high that typical cold reduction schedules might exceed the rolling force limits of the mill.

Nb microalloying has multiple effects with regard to the metallurgical mechanisms occurring during the intercritical annealing cycle [2, 3]. Nb precipitates usually exist in the hot rolled strip when coiling at conventional temperatures in the range of 600-650°C. Any Nb remaining in solid solution has the potential to in-situ precipitate during the annealing cycle. The precipitation potential is enhanced when coiling temperature is lowered to 500-550°C as more Nb is retained in solid solution [4]. This coiling condition also results in a very fine-grained bainitic microstructure. Experimets have shown that in either case Nb precipitation is practically complete after reaching the intercritical soaking phase. For the bainitic coiling condition most of the Nb is retained in solid solution, which then precipitates during the heating cycle as very fine particles with a relevant contribution to strengthening. The existing precipitates produced in larger amount after conventional coiling conditions are subjected to some degree of coarsening during the heating phase of the annealing cycle and are hence less strength effective.

Nb delays the recrystallization of cold deformed ferrite either by precipitation or by solute drag of Nb on the grain boundaries. Experience with Nb alloyed DP steel indicated that the recrystallization temperature is typically raised by around 20°C as compared to the same base analysis without Nb addition. The retarded recrystallization also preserves dislocation networks that act as nucleation site for austenite. Hence austenite formation should be accelerated in Nb alloyed DP steel [5]. On the other hand, the grain-refined microstructure of a Nb microalloyed strip additionally provides an increased grain boundary area as nucleation site for austenite when annealing in the intercritical temperature range. Measurements have indeed confirmed that at a given intercritical annealing temperature the amount of austenite in the Nb added alloy is higher compared to the Nb-free base alloy [5]. During the soaking phase, carbon partitioning is accelerated and more homogeneous in

the finer grained microstructure of the Nb alloyed strip due to the shorter diffusion distances in the smaller grains.
By slow cooling to quenching temperature a defined amount of new ferrite is being nucleated from the existing austenite. Again the refined microstructure of Nb microalloyed exhibits a quicker kinetics of this ferrite formation. A consequence of the enhanced ferrite amount is that the remaining austenite phase is further enriching in carbon. This means that the hardenability of the carbon enriched and smaller austenite grain is improved. With regard to mechanical properties Nb microalloyed DP steel should have less but stronger martensite as a second phase when subjected to a given annealing cycle as compared to the Nb-free base alloy.

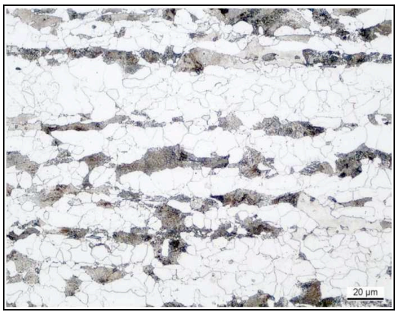
2. LABORATORY ALLOY QUALIFICATION

Three laboratory heats with reduced carbon content (0.1%) have been generated with chemistries shown in Table 1. A reference sample with standard carbon content (0.15%) was also included. The heats were then cast into mini slabs (130x110x30) and hot rolled to gages of 3.33, 2.00 and 1.43 mm, respectively. Thereby the finish rolling temperature was set to either 910°C or 860°C. The coiling temperature was set to either 650°C or 500°C. All hot rolled samples were cold rolled to a final gage of 1.0 mm. According to the hot rolled strip gage, the cold reduction was 30%, 50% and 67%, respectively.

Table 1 Chemical composition of the investigated trial alloys.

	C	Si	Mn	Cr	Nb
Reference	0.15	0.24	1.84	0.40	–
Low-C	0.09	0.26	1.83	0.43	–
Low-C + Nb	0.09	0.24	1.80	0.42	0.03

CCT diagrams of the various trial steels were produced following the SEP 1680/1681 guidelines. Comparing the Nb-free low-carbon variant with the reference steel the ferrite, pearlite and bainite phase fields are shifted to shorter times and the transformation temperatures are raised. Adding Nb to the low-carbon variant shifts the phase fields back towards longer times but leaves the transformation temperatures nearly unchanged. Consequently, Nb microalloying can compensate the carbon reduction with regard to transformation times. However, transformation starts at higher temperatures in the low-carbon variant. The Nb microalloyed variants also expose an elevated recrystallization stop temperature. Depending on the exact chemical composition this increase is in the range of 50-100°C for Nb microalloyed compositions.
Particularly the coiling conditions have as expected a marked influence on the microstructure of the hot strip (Fig. 1). For the high coiling temperature (650°C) the microstructure is ferritic-pearlitic. At low coiling temperature (500°C) the dominating phase is bainite with residuals of ferrite and pearlite. Accordingly, the yield strength of the steels coiled at the lower temperature increases by 100-150 MPa. Niobium additions promote the formation of bainite at low coiling temperature.



FRT: 860°C – CT: 650°C

FRT: 860°C – CT: 500°C

Fig. 1 Microstructure of hot rolled samples (3.33 mm gage) for different finishing conditions.

Intercritical annealing was performed in a MULTIPAS annealing simulator. Different RTF soaking temperatures were adjusted to 780°C, 810°C and 840°C. Slow cooling was performed to 670°C followed by rapid cooling down to 350°C. The simulated annealing cycles are shown in Figure 2. The annealing simulations on the cold rolled specimens resulted in ferritic-martensitic dual phase microstructures (Fig. 3).

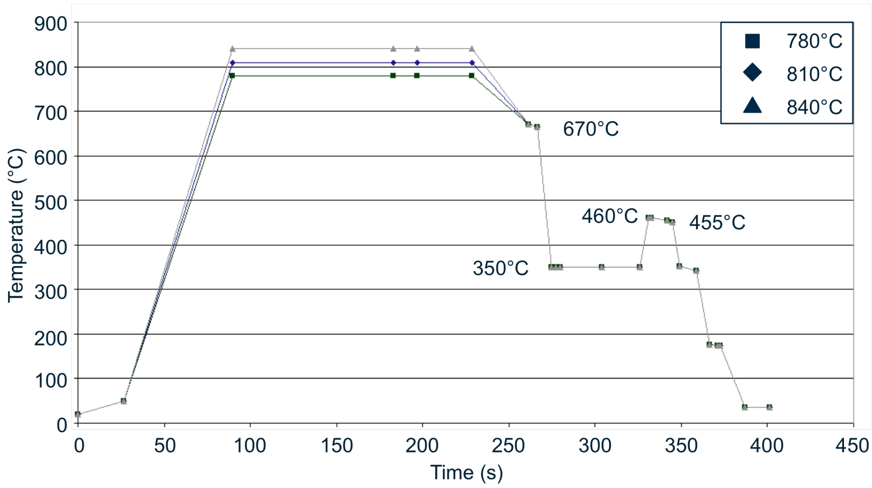
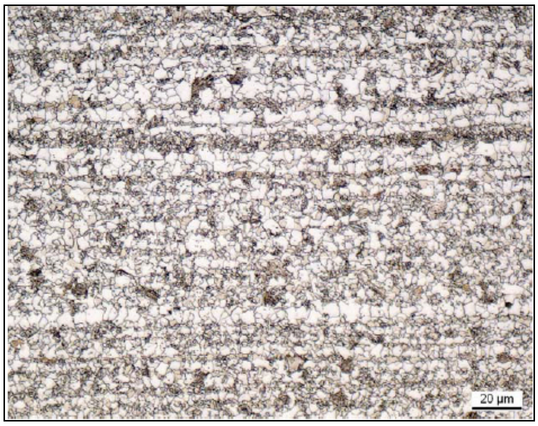


Fig. 2 Simulated MULTIPAS HDG annealing programs.

FRT: 860°C – CT: 650°C (M: 45%, F: 55%)



FRT: 860°C – CT: 500°C (M: 55%, F: 45%)

Fig. 3 Cold rolled (CR: 67%) annealed (810°C) samples of different hot rolling finishing conditions.

The martensite content varied between 40 and 60% depending on the processing conditions. Niobium microalloying additionally provided a refined grain size. The influence of chemical composition, coiling temperature, and annealing temperature is shown in Fig. 4 for a cold reduction of 50%. It is evident that the effect of Nb is most pronounced in combination with a low coiling temperature after hot rolling (Fig. 4a). Under such conditions the strength requirements of a HCT780X (YS: 450-560 MPa, TS: >780 MPa) could be likely met with both, the higher carbon reference grade as well as the carbon-reduced Nb microalloyed grade considering that temper rolling was not performed in the present simulations. The carbon-reduced grade without Nb microalloying does not achieve the HCT780X level under any condition. When the hot rolled pre-material is coiled to ferritic-pearlitic microstructure, only the higher carbon reference grade has potential for reaching the strength level required for HCT780X (Fig. 4b). The carbon-reduced grades in this case make a good HCT600X (YS: 340-420 MPa, TS: >600 MPa). However, both yield and tensile strength are somewhat improved for the Nb microalloyed version.

The strengthening effect of Nb in these carbon-reduced variants becomes more significant when the cold reduction is reduced as shown in Fig. 5. At small reductions Nb microalloying provides higher process robustness in the range of applicable annealing temperatures. Hence, the Nb microalloyed low-C grade appears particularly suitable for production of heavier gauged HCT600X.

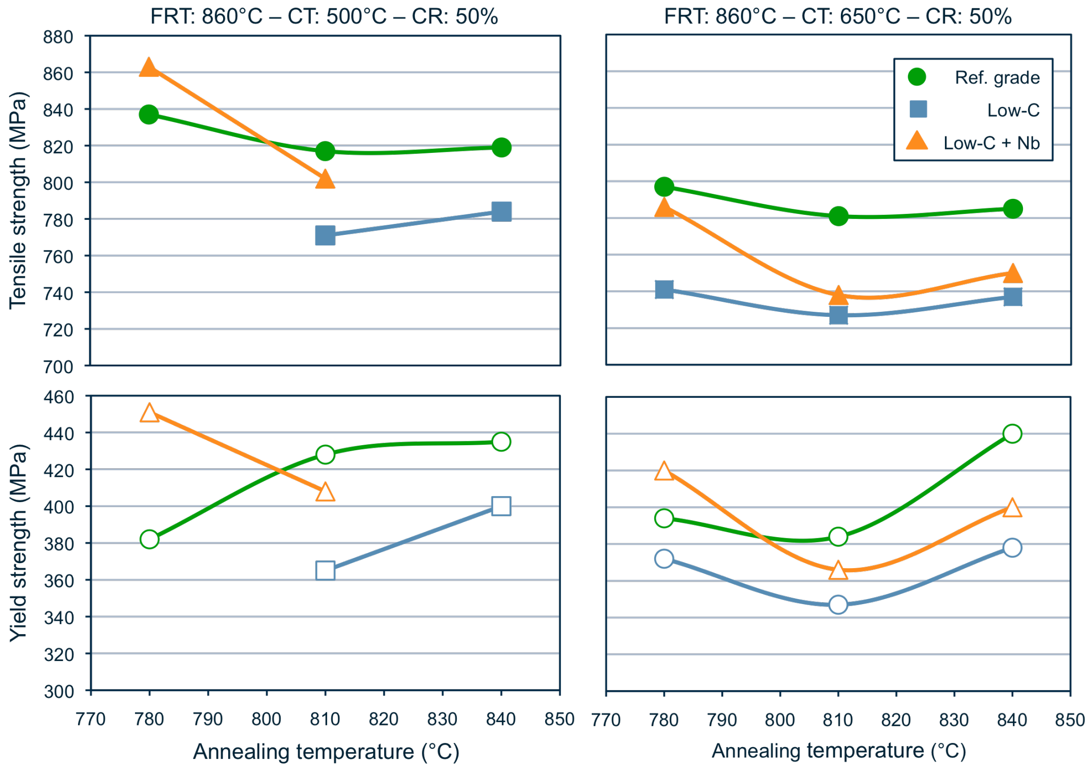


Fig. 4 Effect of annealing temperature on strength properties for different rolling conditions.

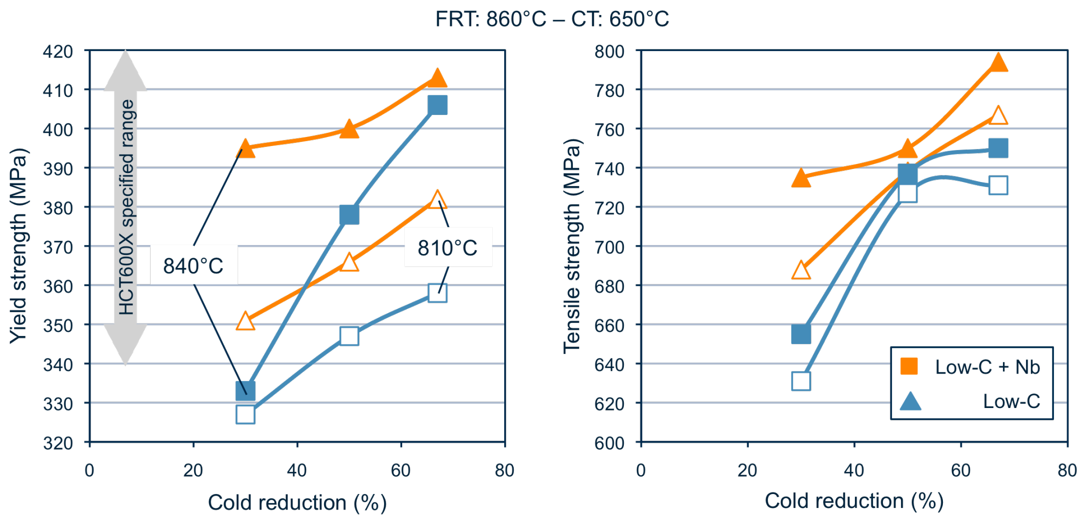


Fig. 5 Effect of cold reduction on strength properties for different annealing temperatures.

3. INDUSTRIAL PRODUCTION OF HCT600X WITH LARGE CROSS SECTION

The developed alloying concept (Table 2) was initially produced on industrial scale for hot dip galvanized strip with a cross section of 2.5x1623 mm and a coating layer of 100 g/m^2. This material was intended to manufacture a longitudinal member for a passenger car. The required large cross section reduces the processing speed in the hot dip galvanizing line at the given annealing power of the furnace. The cooling speed after intercritical annealing depends on the strip gage as indicated by

Fig. 6. The reference grade having a carbon content of 0.14% without Nb microalloying did not show the desired microstructure and thus provided insufficient mechanical properties.

Table 2 Chemical compositions of industrial heats.

	C	P	Mn	Cr	Nb
Reference alloy	0.14	0.02	1.35	0.60	–
New alloy	0.09	–	1.80	0.43	0.01

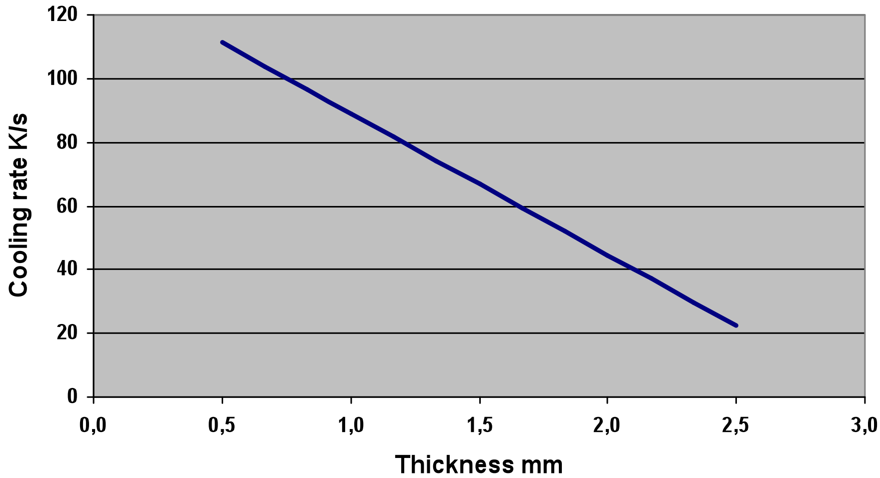


Fig. 6 Gage dependence of the cooling rate in the Salzgitter HDG line.

Industrial heats were cast into slabs of the dimension 250x1500x10.000 mm. Guided by the prior laboratory simulations, these slabs were hot rolled to a gage of 4.4 mm with a ferritic-pearlitic microstructure as shown in Fig. 7. The grain size in the Nb microalloyed variant of ASTM 11-12 was one class finer than in the non-Nb containing reference grade subjected to the same rolling conditions. The formation of bainite in the hot rolled strip was avoided by selecting suitable cooling conditions. A typical time-temperature path is displayed within the CCT diagram of the newly developed steel (Fig. 8).

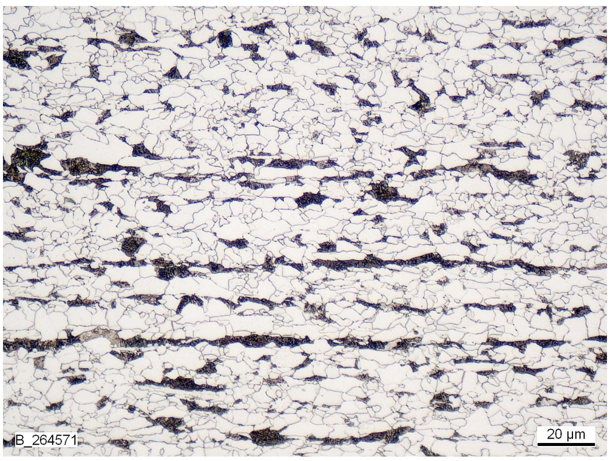


Fig. 7 Microstructure of hot strip for the Nb microalloyed low-C steel.

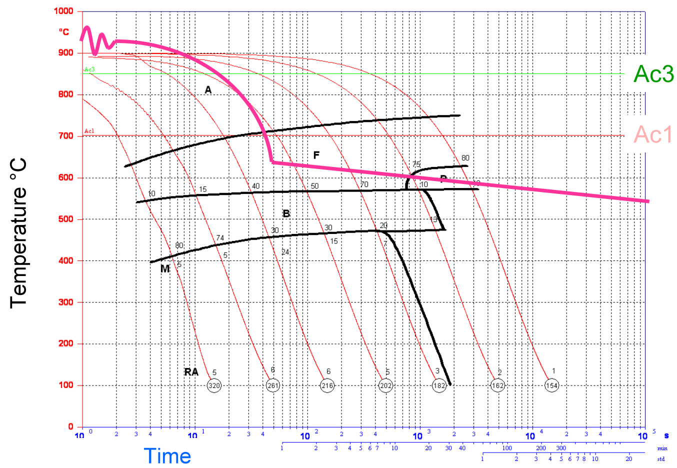


Fig. 8 Cooling curve after finish rolling and CCT diagram of the Nb microalloyed low-C steel.

The further processing of the hot rolled strip was HCl pickling followed by cold rolling to a gage of 2.5 mm. It should be mentioned that the carbon-reduced variant facilitated the coil welding in the continuous pickling line as well as in the HDG line. The time-temperature profile in the industrial HDG line (Fig. 9) is analogous to the profile shown in Fig. 2. The actual line speed is determined by the heating power of the furnace in relation to the strip gage. A characteristic of the line is the fast cooling below zinc pot temperature. The martensite formed after this quenching step is then tempered by a short inductive re-heating cycle to zinc pot temperature. The resulting galvanized strip shows the characteristic dual phase microstructure with a share of about 75% ferrite and 25% transformation product (20% martensite, 5% bainite). The Nb microalloyed variant is particularly fine-grained (Fig. 10). Table 2 reveals the mechanical properties of the reference as well as the newly developed grades. The customer specification required properties independent of the rolling direction. Therefore, mechanical properties were determined in rolling and transverse direction. The values given are average values of 70 produced coils for either variant. The reference grade with increased carbon content does not satisfactorily fulfil the customer specification. On the contrary, the Nb microalloyed variant with reduced carbon content meets the specification reliably. In addition, it is obvious that the standard deviation for the latter variant is smaller indicating reduced scattering of mechanical properties. Furthermore, the newly developed variant offers a better forming potential due to its higher elongation and slightly better work hardening potential. The bake hardening potential is particularly high in both variants.

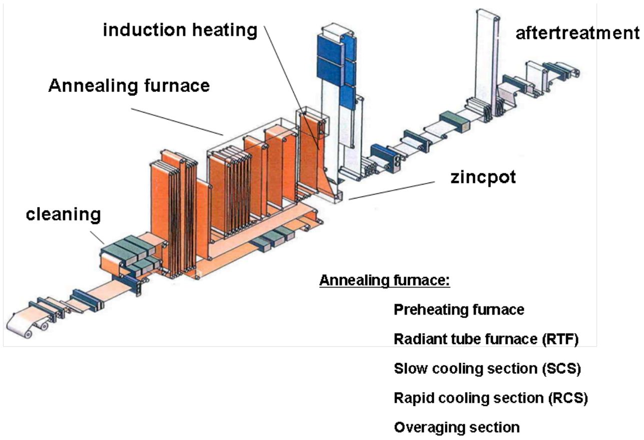


Fig. 9 Layout of the Salzgitter Flachstahl HDG line (max. strip width: 1880 mm).

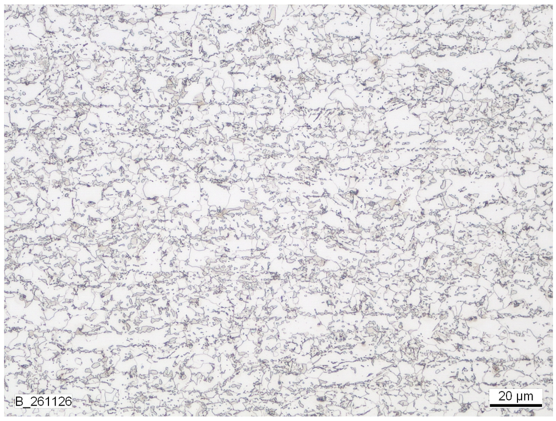


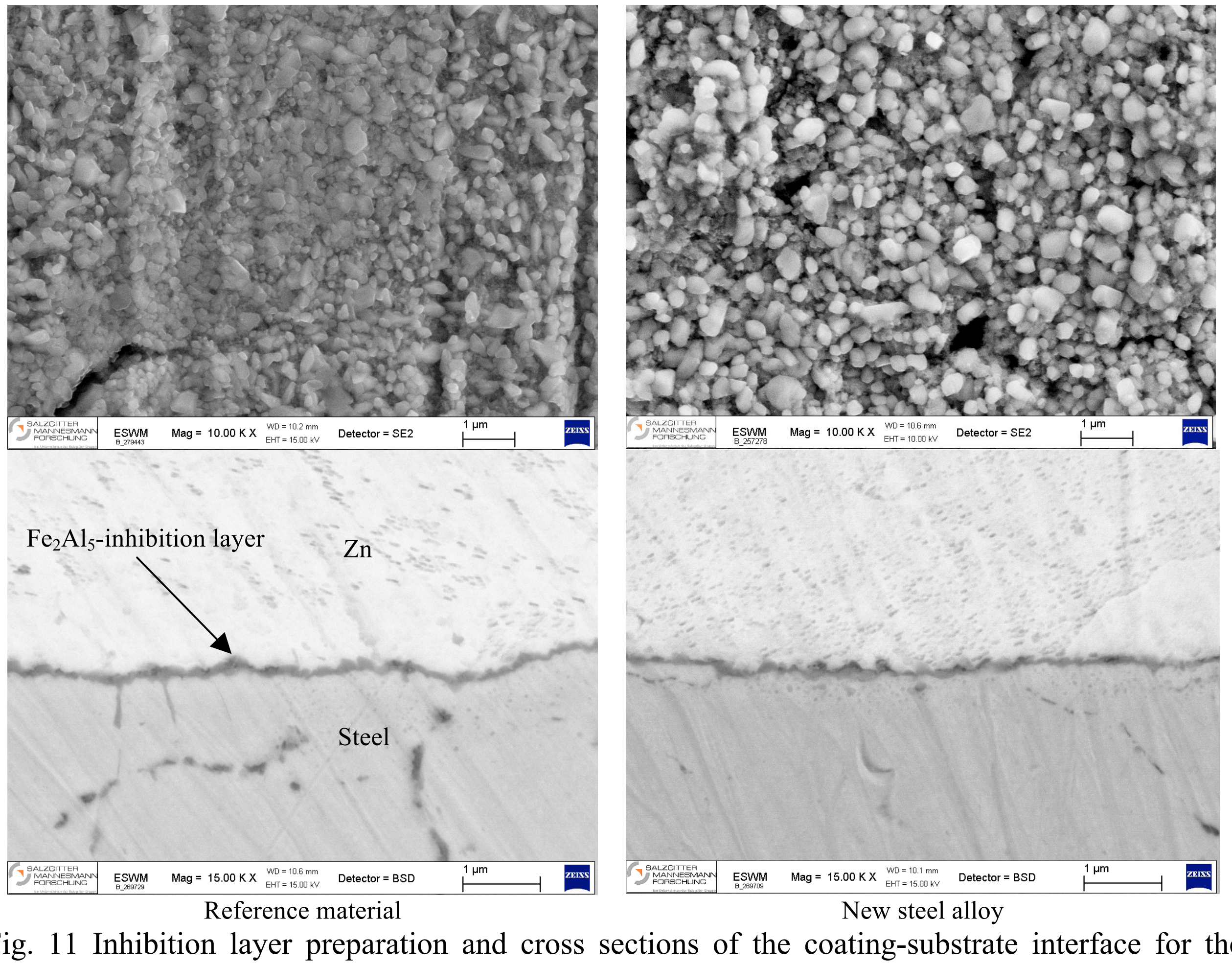
Fig. 10 Final DP microstructure of the Nb microalloyed low-C steel.

Table 3 Mechanical properties (ave.: mean value, std.: standard deviation) of DP steel with the cross-section 2.5x1623 mm.

		Yield strength (MPa)		Tensile strength (MPa)		Elongation A_{80} (%)		n-value		BH_2-value (MPa)	
		ave.	std.	ave.	std.	ave.	std.	ave.	std.	ave.	std.
Reference alloy	long.	372	25	591	35	24	2	0.14	0.01	70	17
	trans.	376	24	604	34	22	2	0.14	0.01	57	6
New alloy	long.	387	17	616	14	25	2	0.15	0.01	67	2
	trans.	403	16	631	19	24	2	0.15	0.01	65	4
Customer specification		*340 – 420*		*600 – 700*		*min. 20*		*min. 0.14*		*min. 0.30*	

A particular aspect when forming DP steels is its resistance against sheared edge cracking. This resistance can be estimated by the hole-expansion ratio (HER). Experience has shown that when the HER is below a value of 30% sheared edge cracking is very likely to occur. If the HER exceeds a value of 60%, the risk of sheared edge cracking becomes negligible. In the intermediate range from 30 to 60% cracks can occur depending on other boundary conditions. The current reference grade revealed HER values of 45% +/-15%. The grain refined Nb microalloyed low-carbon variant however showed with HER values of 70% +/-10% a much better resistance against sheared edge cracking. Actual experience by producing longitudinal members in the press shop using the newly developed DP steel confirmed that the material fulfils all requirements safely.

With regard to galvanization, diffusion of alloying elements like Si, Mn or Cr to the steel surface followed by external oxidation in the annealing furnace of a hot dip galvanising line can disturb the reactivity of the steel surface. At a certain degree of oxidation that depends on the combination of alloying elements, annealing time and annealing temperature wetting of the surface is significantly impeded. Furthermore, this phenomenon may also lead to coating adhesion problems. In the newly developed steel Cr alloying has been reduced whereas the Mn level has been increased (Table 2). Mn and Cr are both known for forming oxides at the steel surface. Nevertheless, the modified alloying concept has the potential of providing optimum surface wetting. Fig. 11 compares the inhibition layer morphology and cross sections of the coating-substrate interface of the newly developed steel with that of the reference steel alloy. The inhibition layer on both steel grades is dense. On the reference steel grade crystal size ranges from less than 50 up to 500 nm while in the newly developed steel the size distribution predominantly ranges between 300 and 500 nm. Interspacings between these larger crystals are filled up by smaller crystals resulting in a fully covered surface. It can thus be concluded that the reactivity of both steel surfaces is comparable. The cross sections show no anomalies at the interface as the zinc layer has been well formed without bare spots. In the reference material, enrichment of the alloying elements at grain boundaries shows more pronouncedly than in the newly developed steel grade. Such enrichment is expected for high strength steel grades, but is not always readily detectable in metallographic cross sections. The standardized adhesion test of the zinc layer (SEP 1931) revealed good results for both grades produced on the Salzgitter galvanizing line.



Reference material New steel alloy

Fig. 11 Inhibition layer preparation and cross sections of the coating-substrate interface for the reference material (left) and the new steel alloy (right).

CONCLUSIONS

Hot dip galvanized dual phase steel with reduced carbon content (0.09%) and Nb microalloying has been successfully developed based on laboratory trials and was then implemented in industrial production.

The laboratory trials revealed that:

- The carbon-reduced steel without Nb microalloying does not reach the strength level required for HCT780X.
- The addition of Nb the carbon-reduced steel allows achieving the strength level of HCT780X under the condition of low finish rolling in combination with a low coiling temperature. The resulting bainitic hot band has however a significantly increased yield strength. This is a challenge with respect to rolling and tension forces in the cold rolling mill.
- Nb microalloying in a carbon-reduced steel offers the potential of producing thick-gaged HCT600X grades.

Industrial production confirmed that the carbon-reduced steel with Nb microalloying:

- Allows safe production of a HCT600X with 2.5 mm gage.
- Shows superior formability properties due to increased elongation and n-value.
- Has a finer and more homogeneous microstructure providing a significantly better hole expansion ratio and thus high safety against sheared edge cracking.
- Provides good hot dip galvanizability resulting in good coating adhesion on the substrate and absence of bare spots.

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