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DESIGN OF A MODULAR ALLOYING CONCEPT FOR HDG LOW-CARBON DP STEEL, ITS INDUSTRIAL IMPLEMENTATION AND EXPERIENCES WITH OEM PARTS

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Abstract

The application of higher strength multiphase steels (AHSS) in automotive body construction can significantly contribute to an increased crash safety level and at the same time reduce body weight. However, frequently encountered difficulties during body manufacturing, due to reduced formability, enhanced crack formation at trim edges, increased spring-back or deteriorated fracture behavior of weld joints, are typically related to unfavorable microstructures. The strict limitation of the C content to below 0.1 wt.% across all strength classes ranging from 500 to 1000 MPa allowed the development of hot dip galvanized dual phase steels which avoided the mentioned manufacturing difficulties. Key to the successful development was a well-tuned addition of Nb for grain refinement, in combination with Mn and Cr bulk alloying on the one hand and adjusted cold rolling reductions, as well as annealing and cooling conditions on the other hand. This modular alloying concept not only brings about advantages for the steel user but it also simplifies sequencing during production in the steel mill when producing various strength grades. For instance, the transition between heats of two strength classes during continuous casting can be shortened and also coil welding between continuous processes such as pickling, tandem rolling and hot dip galvanizing becomes simpler and safer.

Extended annealing simulations were utilized to optimize the combination of alloying elements during the design of the modular alloy concept. These were helpful to fine-tune the temperature profile with regard to the transformation behavior, which in turn is influenced by cold reduction and chemical composition. These simulations were performed under boundary conditions reflecting the process parameters in the industrial annealing lines. By that approach, swift industrial implementation became possible. The resulting trial coils rapidly satisfied general customer demands and in particular concerning surface quality with regard to roughness and zinc adhesion. Consequently, a quick production release by OEMs could be achieved.

Introduction

Within the large group of multiphase steels available worldwide, dual phase (DP) steels in the thickness range of 0.8 to 2.5 mm find the widest application in car body construction. DP steels are mostly used as hot dip galvanized (HDG) sheet. In Europe, these steels currently are standardized within EN10346. However, to guarantee a global availability of automotive sheet with identical properties, the OEMs AUDI, BMW, Daimler, Ford, GM and VW have defined characteristic properties of these steels in the new standard VDA239 [1]. The requirements of VDA239 with regard to chemical compositions and mechanical properties are applicable, irrespective of the surface treatment and are explained in detail in [2]. Tables I and II display the mechanical properties to be determined by tensile testing along the sheet rolling direction and the chemical composition of cold rolled DP steel, respectively. In the grade designation, “Y” and “T” refer to the minimum yield and tensile strength, respectively.

Table I. Mechanical Properties of Cold Rolled Dual Phase Steels (Testing along Rolling Direction) According To VDA239 [1]

	CR290Y490T-DP	CR330Y590T-DP	CR440Y780T-DP	CR590Y980T-DP	CR700Y980T-DP
Yield Strength $R_{p0.2}$ MPa	290 - 380	330 - 430	440 - 550	590 - 740	700 - 850
Tensile Strength Rm MPa	490 - 600	590 - 700	780 - 900	980 - 1130	980 - 1130
Elongation A_{50mm} %	min. 26	min. 21	min. 15	min. 11	min. 9
Elongation A_{80mm} %	min. 24	min. 20	min. 14	min. 10	min. 8
n-value n_{4-6}	min. 0.19	min. 0.18	min. 0.15	-	-
n-value $n_{10-20/Ag}$	min. 0.15	min. 0.14	min. 0.11	-	-
Bake hardening index BH_2 MPa	min. 30	min. 30	min. 30	min. 30	min. 30

n_{4-6} strain hardening exponent determined between 4 and 6% plastic strain
$n_{10-20/Ag}$ strain hardening exponent determined between 10 and 20% plastic strain or Ag if Ag<20%

Table II. Chemical Composition of Cold Rolled Dual Phase Steels According To VDA239 [1]

	CR290Y490T-DP	CR330Y590T-DP	CR440Y780T-DP	CR590Y980T-DP	CR700Y980T-DP
max. C %	0.14	0.15	0.18	0.20	0.23
max. Si %	0.50	0.75	0.80	1.00	1.00
max. Mn %	1.80	2.50	2.50	2.90	2.90
max. P %	0.080	0.040	0.080	0.080	0.080
max. S %	0.015	0.015	0.015	0.015	0.015
Al %	0.015-1.00	0.015-1.50	0.015-2.00	0.015-2.00	0.015-2.00
max. (Cr+Mo) %	1.00	1.40	1.40	1.40	1.40
max. (Nb+Ti) %	0.15	0.15	0.15	0.15	0.15
max. B %	0.005	0.005	0.005	0.005	0.005

The well known designations DP600 and DP780 can be used instead of CR330Y590T-DP or CR440Y780T-DP. Accordingly, the specification allows chemical compositions over a wide range of alloying elements as long as the requirements regarding mechanical properties are fulfilled and cold forming properties (including edge stretching or bending in some applications) are not restricted by martensite lines, slag inclusions or segregations. At first sight a steady increase of the C content from 0.05% to around 0.20% within the allowed range of VDA239 appears appropriate for realizing increasing strength levels from 490 MPa to 980 MPa. Other elements such as Mn, Si, Cr and P, which increase strength and hardenability, complete the analysis. Pursuing the strategy of continuously increasing the C content, however, bears a process related risk as casting of melts in the peritectic range causes problems. In order to avoid these problems, alloying strategy-1 excludes the peritectic range, ie. lower strength DP grades are being produced with C content < 0.1% whereas a higher C content is used for higher strength grades (Figure 1). In the case of DP600, two principal alloys are possible. In a low C variant, an increased Mn content has to be selected, whereas the high C variant requires a lower Mn addition. Generally for the higher strength DP grades, the Mn content is raised up to a level of around 2.2%. Practical experience revealed in this case that the applicable processing windows could vary considerably, depending on the chemical analysis. Even small deviations from the ideal processing route of such DP steels result in inhomogeneous microstructure, banding, varying mechanical properties and severe anisotropy. Consequently, stringent limitations in the manufactured properties (sometimes directionally dependent) and premature damage are being observed. The optimum microstructure and hence properties of cold rolled hot-dip galvanized DP steels require an exact adjustment, not only for the chemical analysis, but also for the hot rolling conditions, cold reduction and annealing conditions.

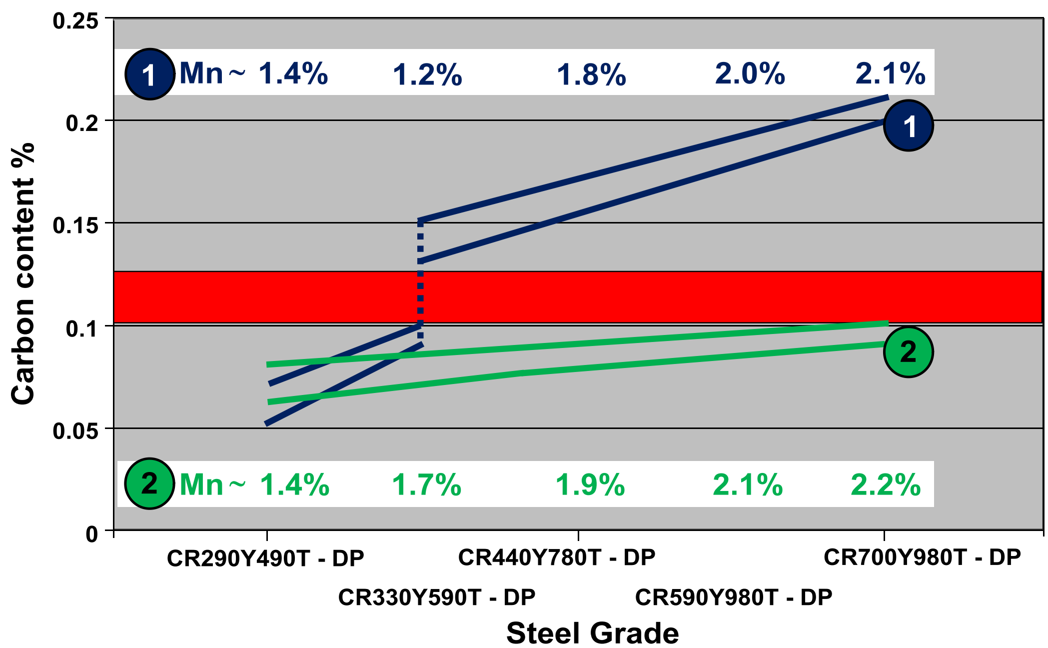

Figure 1. C and Mn alloying strategies for DP grades defined by VDA239
(the peritectic C range preferably to be avoided is indicated in red).

Table III reveals several examples of alloying concepts for DP600 and DP780 grades being commercially supplied by major European steelmakers. It is evident that some DP600 concepts are indeed designed to avoid the peritectic reaction, whereas several others are in the unfavorable peritectic C range (the element C has the strongest influence on the peritectic of steel). The Mn level is typically on the high side for sub-peritectic DP600 concepts. Additional hardenability is achieved by either an increased Cr content (A.1, B.2, D.1, E.2, F.1, F.3) or by Mo alloying (B.1, C.2, E.1, E.3, F.2) in combination with a reduced Cr (and sometimes Mn as in E.3, F.2) content. In one approach, (E.2), B microalloying is used to increase hardenability. This concept implies a small Ti addition to fix interstitial N.

The alloy concepts for DP780 generally apply C additions in the peritectic range and high Mn levels. In the majority of cases Cr is used as a second hardenability element whereas Mo is rarely used for this grade. It is apparent that several DP780 concepts use Nb microalloying and in one case Ti microalloying, while B microalloying is not being applied in these examples.

The considerable variation in alloying concepts, especially for DP600 grades, has in some part arisen historically. On the other hand, in several cases limitations within the production route mandate certain alloying elements. More precisely, the cooling power in the hot-dip galvanizing line is one of the most decisive issues in determining the bulk-alloying concept (Figure 2). Lines having limited cooling power require either rather high alloying contents of Mn and Cr, or alternatively, a relatively smaller Mo addition. Due to the significant amount of HDG DP600 being produced, steelmakers often have to employ several coating lines flexibly, so that usually the line with the weakest capability determines the overall alloy concept.

Table III. Commercialized Alloy Concepts for DP600 and DP780 by European Steel Mills (alloy additions in wt.%)

Producer	Concept	C	Si	Mn	P	Al	Cr	Mo	Ti	Nb	B
		DP600									
A	A.1	0.09	0.13	1.49	0.014	0.05	0.75	-	-	-	-
B	B.1	0.07	0.02	1.85	0.014	0.05	0.21	0.18	-	-	-
	B.2	0.09	0.24	1.78	0.013	0.04	0.56	-	-	-	-
C	C.1	0.09	0.25	1.88	0.019	0.04	0.21	-	-	-	-
	C.2	0.12	0.32	1.38	0.013	0.03	0.25	0.07	-	-	-
D	D.1	0.11	0.19	1.62	0.012	0.05	0.46	-	-	-	-
E	E.1	0.11	0.18	1.39	0.011	0.04	0.15	0.20	-	-	-
	E.2	0.11	0.21	1.51	0.012	0.03	0.46	-	0.03	-	0.007
	E.3	0.10	0.07	1.24	0.014	0.91	0.03	0.20	-	-	-
F	F.1	0.10	0.21	1.78	0.010	0.03	0.44	-	-	-	-
	F.2	0.11	0.11	1.23	0.014	0.88	0.04	0.20	-	-	-
	F.3	0.11	0.23	1.49	0.009	0.03	0.66	-	-	-	-
		DP780									
A	A.2	0.15	0.18	2.08	0.011	0.04	0.26	-	-	0.02	-
C	C.3	0.15	0.21	1.97	0.023	0.03	0.25	-	-	-	-
	C.4	0.15	0.21	1.91	0.016	0.03	0.19	-	0.03	-	-
D	D.2	0.14	0.30	1.75	0.012	0.04	0.52	-	-	0.02	-
F	F.4	0.16	0.17	1.72	0.013	0.03	0.32	0.16	-	-	-
	F.5	0.14	0.30	1.76	0.010	0.03	0.50	-	-	0.02	-

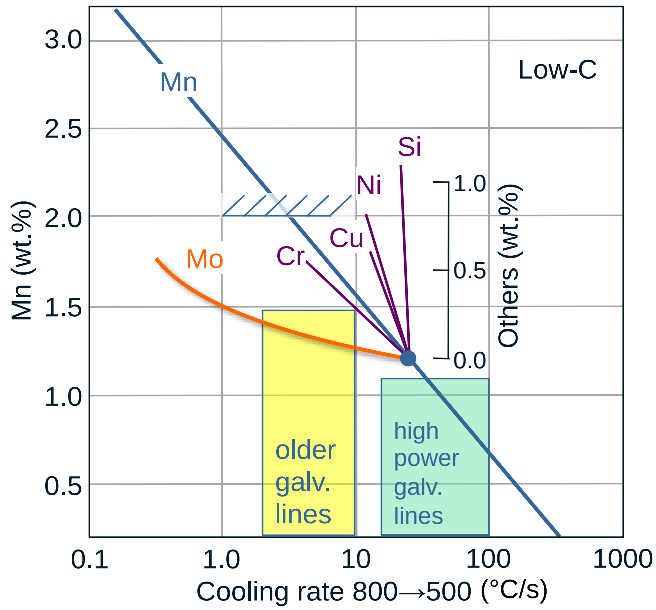

Figure 2. Influence of Mn and other bulk alloying elements on the critical cooling rate for producing low-C dual phase steel on galvanizing lines [3].

A particular advantage of adding Mo to hot dip galvanized DP steels refers to the coating quality. Elevated amounts of Mn, Si or Cr can cause problems of surface wetting and adhesion of the zinc layer. This is related to the fact that these elements can enrich at the surface during intercritical annealing, forming surface oxides. The presence of an oxide layer lowers the wettability of the surface. Mo on the contrary shows no tendency to enrich and form an oxide layer at the surface. Therefore, Mo alloyed concepts such as E-I, E-III and F-II with significantly reduced Mn and Cr additions are superior concepts for hot-dip galvanizing processes.

The bulk alloying concepts of DP780 grades are much more similar throughout the supplier base. This is because the quantities of HDG DP780 demanded by the market thus far have been quite limited. Therefore, steelmakers can run the production exclusively on the more capable galvanizing lines allowing a reduction in the amount of hardenability alloys.

Microalloying with Nb, providing microstructural refinement, has been found to significantly improve bendability, particularly of DP780 steel [3]. This aspect will be detailed in a later section. Basically, it is sufficient to add a small amount of Nb to the otherwise unchanged alloy concept (A.2) to enable the improvement, as is indicated in Figure 3.

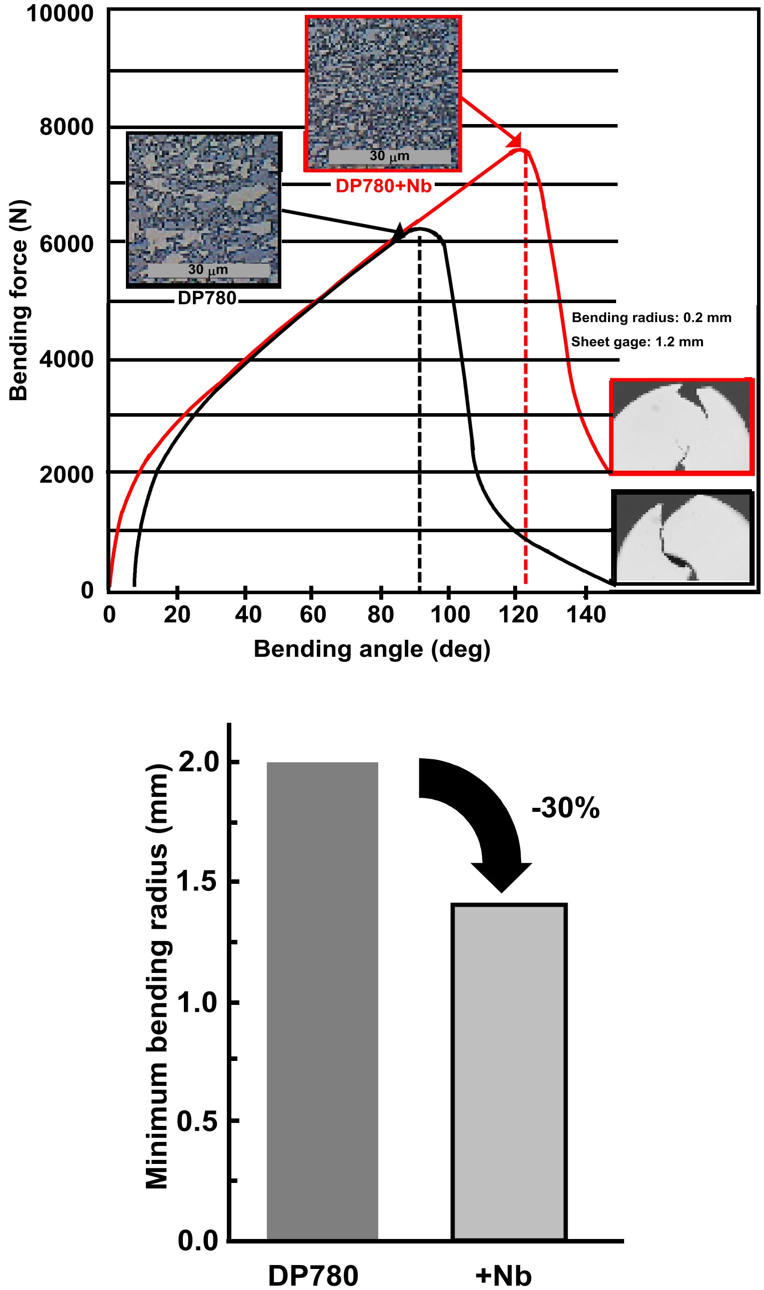

Figure 3. DP780 steel under 3-point bending conditions (acc. to VDA238) and 180°-bending before and after microstructural refinement (corresponds to steel A.2 in Table III) [4].

Cornerstones of a Novel Platform - Alloying Concept for Low-C DP Steels

An alternative to current mainstream alloy concepts for DP steels is indicated by strategy 2 in Figure 1, wherein the C content is principally limited to a maximum of 0.1% for the entire strength range. When pursuing such a low-C approach, it is important to consider the hardenability concept in detail. Reducing the C content brings the bainite nose forward, increasing the risk of forming bainite during holding at the zinc bath temperature, as is schematically demonstrated by Figure 4. It has been discussed already that increased additions of Mn and Cr, as well as a smaller addition of Mo, can delay bainite formation. Furthermore, microstructural refinement by Nb microalloying can assist the rapid formation of ferrite during cooling, thus promoting C enrichment in austenite. A higher austenite C content facilitates transformation into martensite rather than into bainite.

Based on these ideas, it was decided to develop an alloy platform concept that is strictly limited to a maximum of 0.1%C. The hardenability concept to be defined should provide:

- high robustness against process variations, resulting in low scatter of the specified mechanical properties;
- the possibility of producing the grade on galvanizing lines with somewhat different time-temperature characteristics;
- the possibility of producing increased sheet gages up to 2.5 mm;
- good quality of the galvanized surface;
- high resistance against edge cracking (high hole expansion ratio).

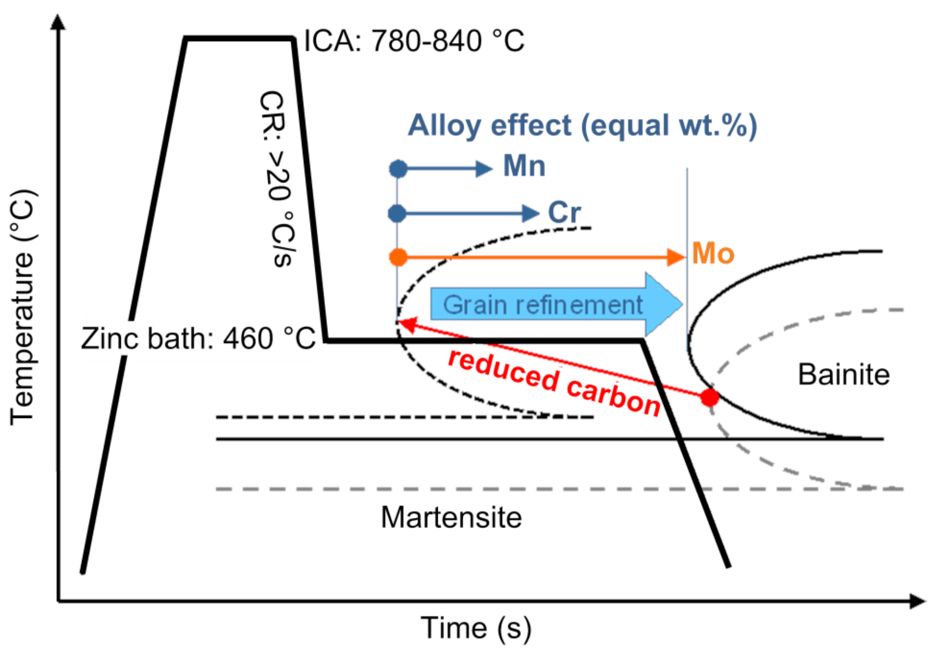

Figure 4. Alloying effects on bainite formation in low-C DP steel during a hot dip galvanizing cycle.

The qualification of respective alloy concepts has been done initially by laboratory simulation for the DP600 and DP780 grades. Thereafter, industrial trials have been initiated to verify the alloy concept under realistic production conditions in the two hot-dip galvanizing lines available at Salzgitter Flachstahl GmbH. Subsequently, the established industrial concept was tested regarding press shop performance by stamping various parts in serial production dies.

Microstructural Refinement in DP Steels and the Essence of Nb Microalloying

Several prior research efforts have indicated that microstructural refinement in DP steels not only enhances strength, but also is beneficial to secondary (ie. non-specified) cold forming properties, such as bending, stretch flanging or hole expansion.

For instance, Calcagnotto et al. [5] demonstrated that severe grain refinement in DP steel could raise yield as well as tensile strength by about 20%. This strength surplus provides a higher safety margin with respect to the minimum specified values on the one hand and on the other hand, the strength surplus provided by grain refinement allows a reduced martensite content in the dual phase microstructure and hence increased elongation.

Grain refinement of the dual phase microstructure was shown to have a considerable positive effect on the bending behavior of cold rolled DP steel. Figure 3 demonstrates that the grain-refined steel (A.2) supports a higher bending force at an increased bending angle. The non-grain-refined steel fails at a bending angle of around 90° so that the production of typically U-shaped profiles is problematic. The grain-refined steel on the contrary offers a sufficient margin for the forming process thus offering clearly improved process robustness. Furthermore, the grain-refined DP steel allows a tighter minimum radius for 180°-bending showing an improvement of around 30% (Figure 3).

Grain refinement of the final dual phase microstructure is for a large part inherited from the hot rolled microstructure. The prior hot rolled strip microstructure can be principally adjusted to be either ferritic-pearlitic or bainitic. The occurrence of either is mainly related to the coiling temperature. Bainitic hot rolled strip will be typically finer grained than ferritic-pearlitic hot rolled strip. Yet Nb microalloying provides microstructural refinement, irrespective of the coiling temperature.

Irrespective of whether or not the processing route for producing DP steel is adapted to Nb microalloying, the effect of Nb is always noticeable. 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 most prominent 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. This refined ferritic-pearlitic microstructure is inherited by the final material producing a finer grained ferrite matrix embedding smaller martensite islands, as shown in Figure 5, where a Nb microalloyed variant is compared to a Nb-free one for a coiling temperature of 680 °C. Comparing these microstructures, one has to state that the effect of Nb is partly overlaid by the effect of Si, the Si content being higher in the non microalloyed steel and thus promoting ferrite formation.

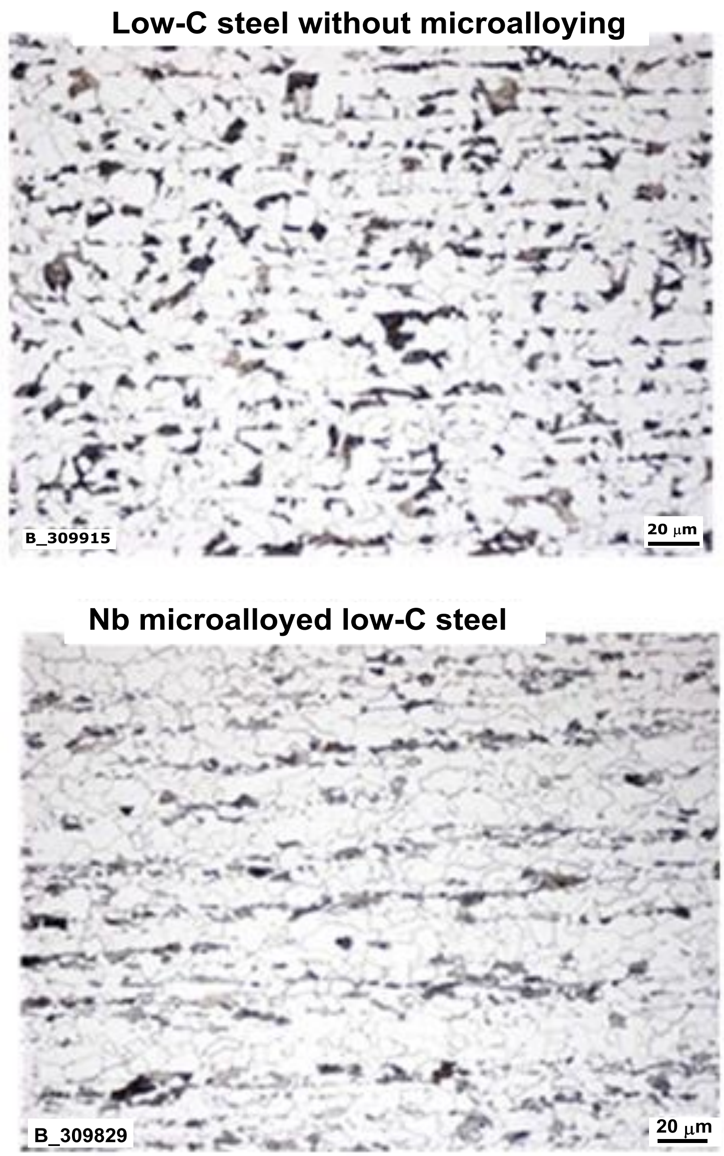

Figure 5. Effect of Nb microalloying on ferritic-pearlitic microstructure of low-C DP steel after coiling at 680 °C.

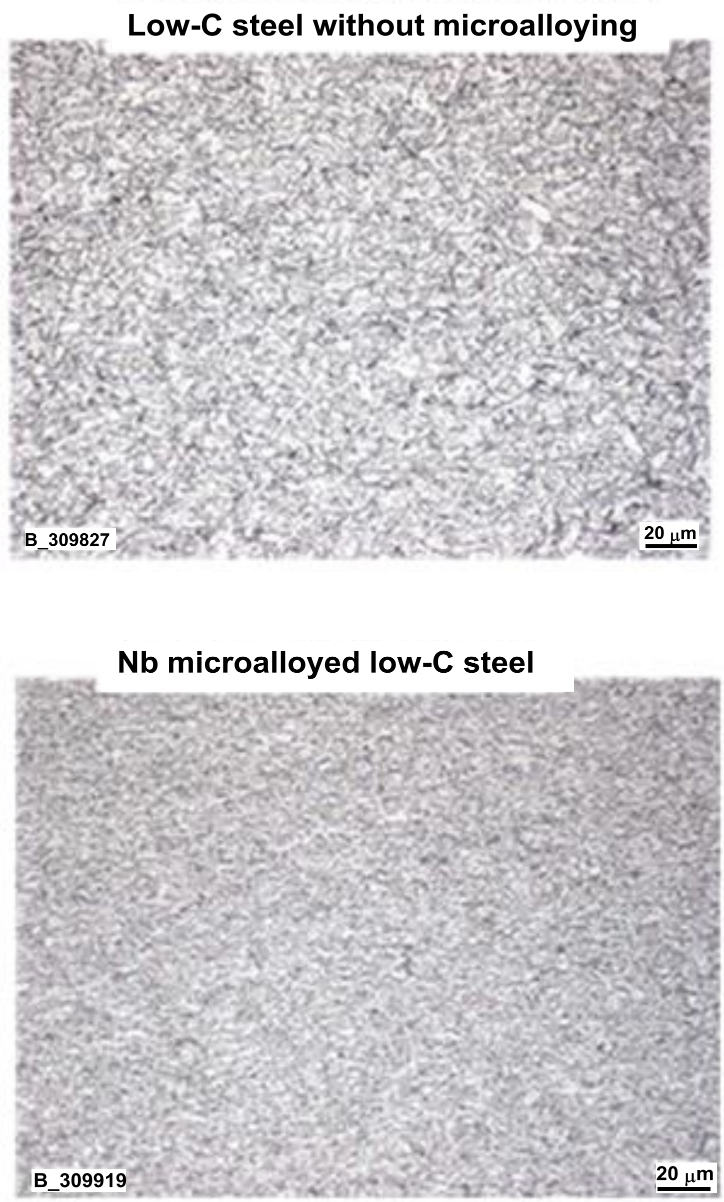

Figure 6. Effect of Nb microalloying on bainitic microstructure of low-C DP steel after coiling at 500 °C.

As mentioned earlier, an option for producing cold rolled DP steel is coiling the hot strip in the temperature range of 500 to 550 °C. This results in a bainitic microstructure of the hot strip, as shown in Figure 6, for the same low-C chemistry. This observation agrees with results published by Pichler et al. [6] and was also reported by [7]. For example, the CCT diagram shown for the Nb microalloyed variant in Figure 7 demonstrates the resulting microstructures for two coiling temperatures. Because a significant portion of Nb remains in solid solution under this coiling condition, it is available for precipitation during subsequent intercritical annealing [8].

When the coiling condition is set directly to produce DP steel from the rolling heat (ie. hot rolled DP steel), the effect of the pancaked austenite is to enhance ferrite nucleation, due to the larger total austenite grain boundary area [9]. 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, ferrite as well as martensite grain sizes are also refined. Such grain refinement has been found to increase the tensile strength of hot rolled DP steel by around 150 MPa and thus provides an efficient possibility for upgrading DP600 towards DP780.

Grain refinement and particularly bainitic transformation lead to a significant increase of yield strength of the hot strip, which has to be taken into consideration with respect to the subsequent cold rolling operation. Since the addition of Nb leads to grain refinement, the yield strength of conventional ferritic-pearlitic hot strip will be increased [6]. The yield and tensile strength of the cold rolled full hard strip are also increased compared to Nb-free material and depends on the cold roll reduction. When the hot strip is coiled for a bainitic microstructure, the yield strength can become so high that typical cold reduction schedules might exceed the rolling force limits of the mill. Therefore, hot strip with bainitic microstructure is not recommended when a high level of cold reduction is targeted. However, such strip could be directly hot dip galvanized, ie. either without any or with a limited cold reduction.

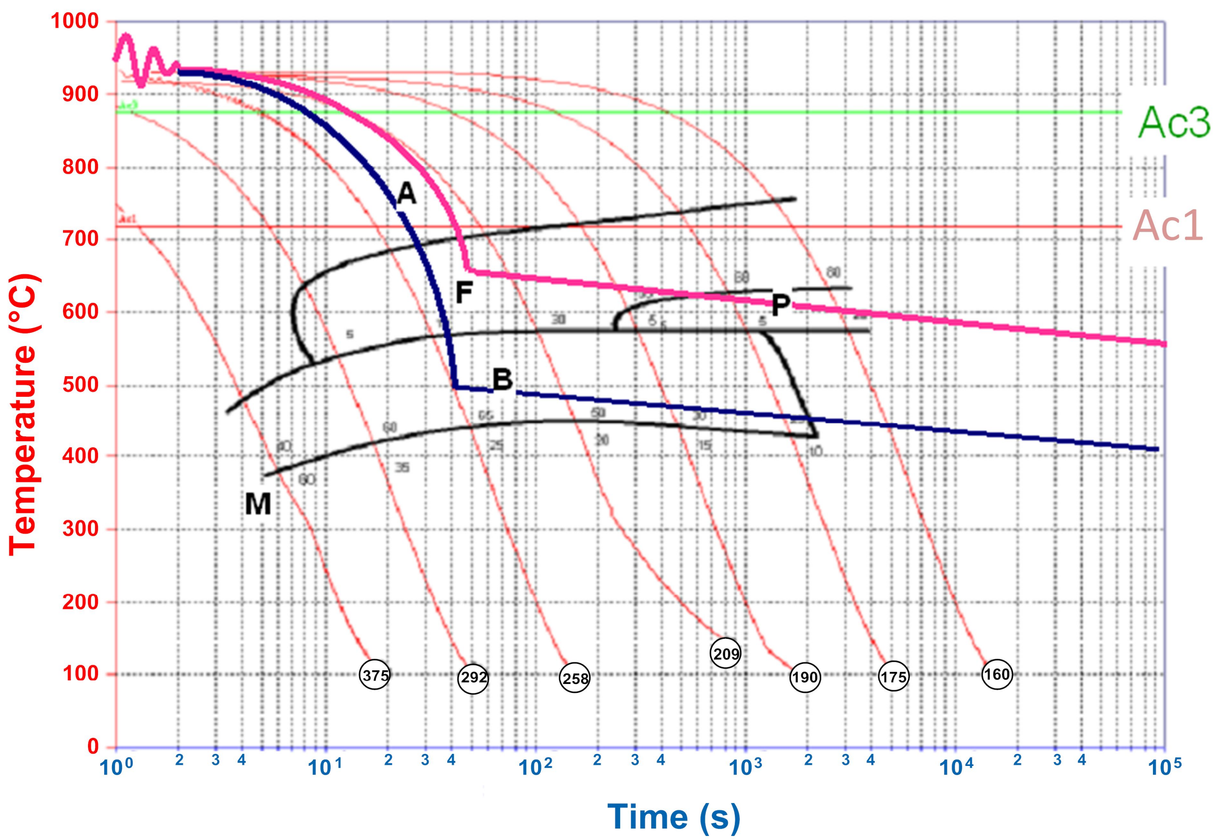

Figure 7. CCT diagram of the Nb microalloyed low-C steel and cooling curves after finishing.

Nb microalloying has multiple effects with regard to the metallurgical mechanisms occurring during the intercritical annealing cycle [6,9]. 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 precipitate *in situ* 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 [8]. This coiling condition also results in a very fine-grained bainitic microstructure. Experiments 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 amounts 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 during reheating 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 sites for austenite. Hence austenite formation should be accelerated in Nb alloyed DP steel [10]. Furthermore, the grain-refined microstructure of an Nb microalloyed strip additionally provides an increased grain boundary area for nucleation sites 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 [10]. During the soaking phase, C partitioning is accelerated and is more homogeneous within the austenite from the finer grained microstructure of the Nb alloyed strip, due to the shorter diffusion distances in the smaller grains.

By slow cooling to the (sub-Ae_3) quenching temperature, a defined amount of new ferrite is being nucleated from the existing austenite. Again, the refined microstructure of Nb microalloyed steels exhibits quicker kinetics for this ferrite formation. A consequence of the enhanced amount of ferrite is that the remaining austenite phase is further enriching in C. This means that the hardenability of the C-enriched and smaller grained austenite 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.

Laboratory Development and Pre-qualification of Low-C DP Platform Concepts

The development stages described here have the purpose of qualifying different alloy concepts which all have low C content (max. 0.1%) in common. The various trial concepts have an increased addition of Nb, partly in combination with other microalloying elements such as Ti and B (Table IV). The content of bulk alloying elements such as Si (0.25%), Mn (1.85%) and Cr (0.4%) were kept constant. Other elements were not employed. The aim was to clarify how these alloy concepts, in combination with adapted hot and cold rolling conditions, as well as annealing parameters, influence the microstructure and processing properties of DP steel.

All five microalloyed concepts, as well as a microalloy-free reference variant, were finish rolled at 920 °C to a hot rolled sheet gage of 3 mm. Subsequently, all samples were coiled at both 500 °C and 680 °C, resulting in a bainitic or ferritic-pearlitic microstructure, respectively. The strength data and microstructural characteristics for all combinations are summarized in Table V. It is evident that increasing the Nb content from 0 to 0.03% results in the expected microstructural refinement, irrespective of the coiling temperature. The consequence is an increase of the flow stress by about 50 MPa (see also Figure 8). Further addition of Ti and/or B to the higher Nb alloyed variant does not lead to significant changes in the hot strip properties. Comparing W-3, W-4 and W-6, one has to consider that the effective B content (B in solid solution) of alloy W-4 is significantly lower than 0.003 due to the absence of Ti, ie. some of the B will be tied up with N.

Table IV. Base Alloy Concept and Microalloying Options for Laboratory Trials and Industrial Pre-qualification (wt.%)

Base alloy	Microalloy option	Nb	Ti	B
<0.1%C 0.25%Si 1.85%Mn 0.40%Cr 50 ppm N 100 ppm P	W-1	0.00	0.00	0.000
	W-2	0.015	0.00	0.000
	W-3	0.030	0.00	0.000
	W-4	0.030	0.00	0.003
	W-5	0.030	0.030	0.000
	W-6	0.030	0.030	0.003

Each of the hot rolled variants was stepwise cold rolled on a laboratory rolling mill to a total cold reduction of 60%. The yield strength was determined by tensile testing for the different reduction stages. The flow curves are shown in Figure 9. Generally, strong work hardening is observed with increasing cold reduction. The group of flow curves originating from the bainitic coiled material (cooling option HR1) is consistently at least 150 MPa higher than the ferritic-pearlitic material (cooling option HR2). Only the flow curves of option HR2 were finally used to calculate cold reduction schedules for industrial trials since the material coiled under option HR1 was too strong for efficient cold rolling.

Table V. Properties and Microstructural Characteristics of Hot Strip (3 mm) Under Two Coiling Conditions

HR1 (FRT: 920 °C CT: 500 °C)	**W-1 (Nb free)**	**W-2 (+Nb)**	**W-3 (++Nb)**	**W-4 (++Nb+B)**	**W-5 (++Nb+Ti)**	**W-6 (++Nb+Ti+B)**
YS (MPa)	480	512	533	564	553	525
TS (MPa)	612	645	679	706	682	741
Grain size (ASTM)	12.5	13.5	13	13	12.5	13.5
Ferrite (%)	25	15	25	15	15	10
Pearlite (%)	15	5	5	5	5	5
Bainite (%)	60	75	65	70	75	70
Martensite (%)	0	5	5	10	5	15
HR2 (FRT: 920 °C CT: 680 °C)	**W-1 (Nb free)**	**W-2 (+Nb)**	**W-3 (++Nb)**	**W-4 (++Nb+B)**	**W-5 (++Nb+Ti)**	**W-6 (++Nb+Ti+B)**
YS (MPa)	310	345	363	348	386	366
TS (MPa)	464	485	495	483	497	496
Grain size (ASTM)	10	11	11	10	11	10
Ferrite (%)	75	85	78	78	78	85
Pearlite (%)	25	15	22	22	22	15
Bainite (%)	0	0	0	0	0	0
Martensite (%)	0	0	0	0	0	0

In a separate development project, a processing route allowing the direct annealing and galvanizing of bainitic coiled (~ 500 °C) hot strip has been designed using a modified analysis of variant W-6. By this route hot dip galvanized DP steels in the gage range of 2 to 3 mm, having minimum yield and tensile strength of 330 MPa and 580 MPa, respectively could be successfully produced. The details of this development will be published elsewhere.

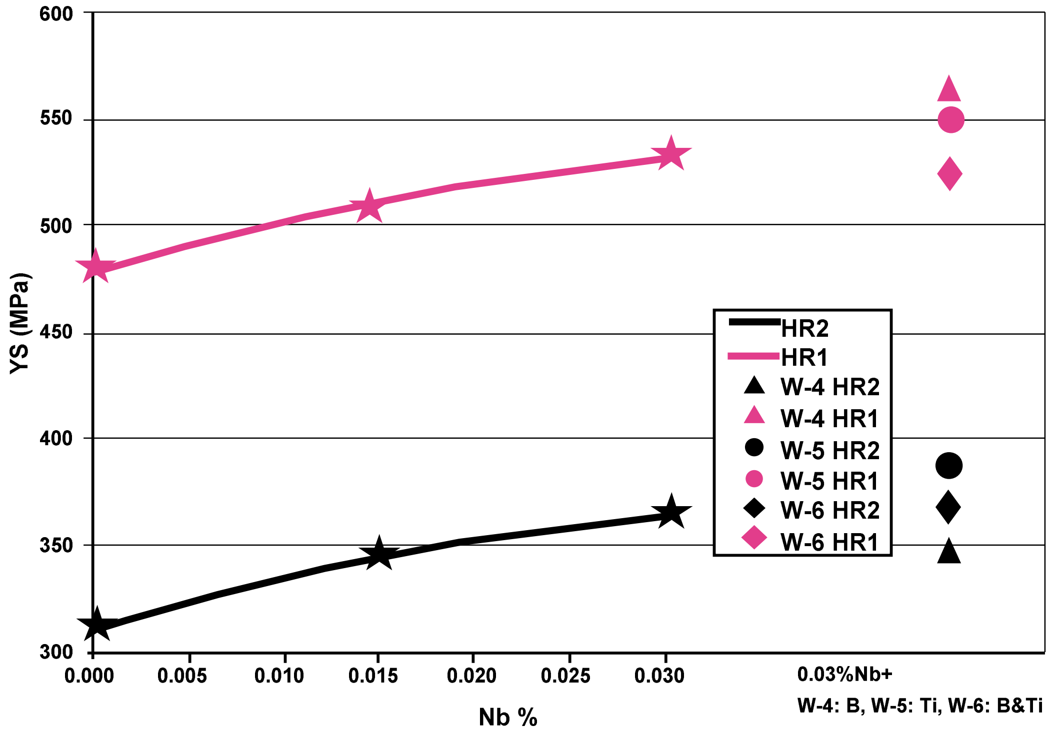

Figure 8. Effect of Nb and other microalloying elements on the yield strength of hot rolled 3 mm strip using different coiling temperatures.

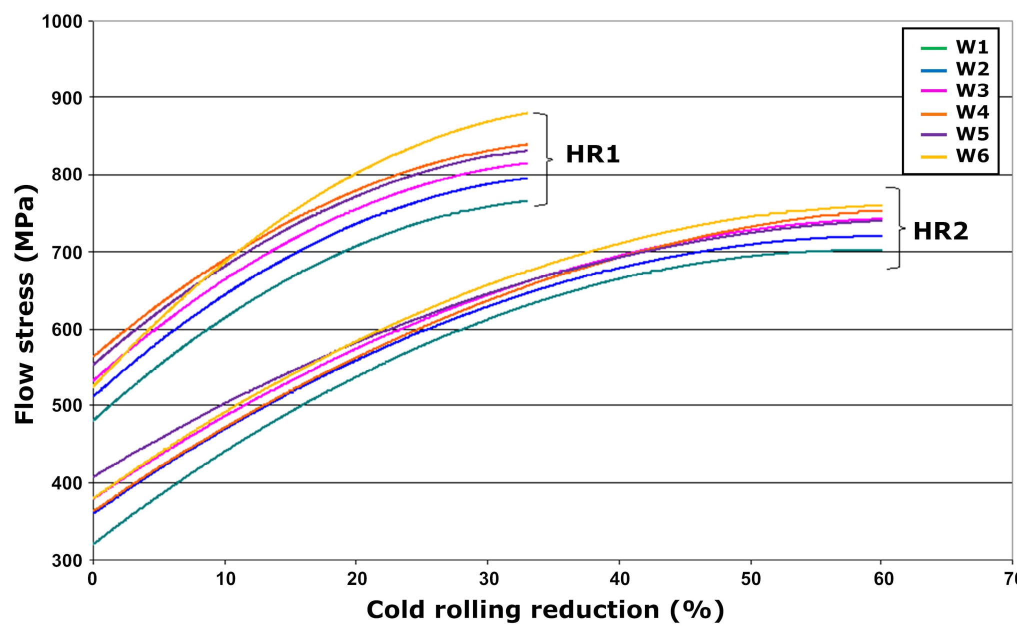

Figure 9. Influence of cold reduction on flow stress for two coiling conditions.

The further development process considered only cooling variant HR2 in combination with the analytical variants W-2 (0.015%Nb) and W-6 (0.030%Nb +Ti+B), as well as the non-microalloyed reference variant W-1. With these it had to be clarified which cold reduction and which annealing temperature program would deliver the best properties. However, one has also to consider that, due to the strong cold working, the tandem mill is exposed to higher loads. Thus, the cold roll reduction has to be kept as small as possible.

Figure 10 shows two CCT diagrams which were calculated using JMatPro® [11]. It demonstrates schematically for the Nb microalloyed variant W-3 that cold reduction (in this case 57%) results in an accelerated formation of ferrite. As an approach to simulate this influence, the grain size was varied from ASTM no. 9 to ASTM no. 14 for the cold-rolled material. In order to reasonably study this effect, samples of 2.0 mm thickness (33% cold reduction) as well as 1.3 mm thickness (57% cold reduction) were annealed by time-temperature cycles that correspond to the characteristics of the available two (horizontal and vertical) galvanizing lines. According to Figure 11 these lines are characterized by:

- High annealing temperature, short holding period and moderate cooling rate (horizontal line, HDG1);
- Lower annealing temperature, long holding time and accelerated cooling (vertical line).

In the vertical line (HDG2) there is the additional possibility of cooling the strip to below 300 °C before the zinc pot. By rapid induction heating, the strip can then be reheated to the required zinc bath temperature.

It turned out that a cold reduction of 57% is sufficient to obtain the desired microstructure as well as mechanical properties when the annealing and cooling strategy is appropriately adjusted. This applies for both time-temperature cycles, ie. those for the vertical as well as horizontal lines.

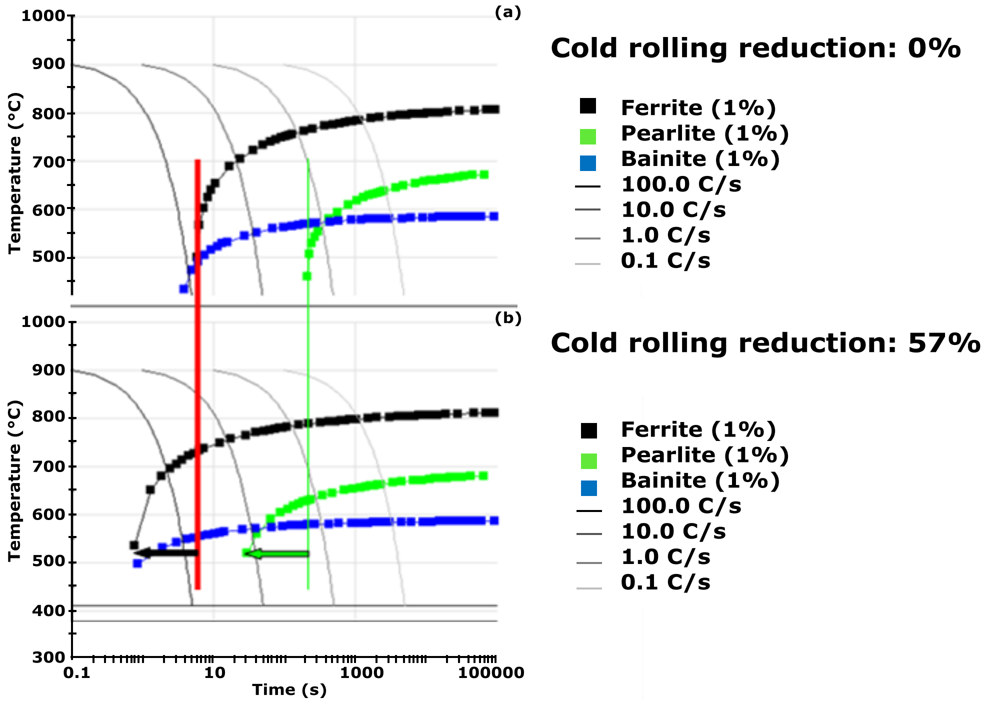

Figure 10. Influence of cold reduction on phase formation in variant W-3, calculated using JMatPro® [11].

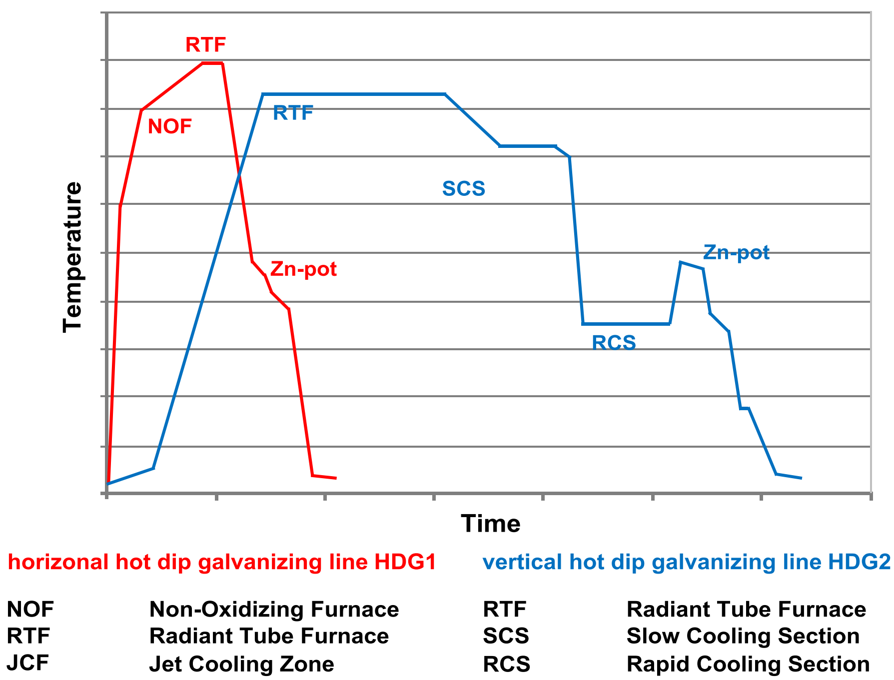

Figure 11. Typical annealing cycles of the two hot dip galvanizing lines used by Salzgitter Flachstahl GmbH for production of DP steel.

Industrial Implementation and Results

Large-scale trials were conducted on both hot dip galvanizing lines using strip gages between 1 and 2 mm and strip widths up to 1500 mm. The hot rolling was according to condition HR2 and the cold reduction varied between 40 and 60%. With regard to the chemical composition, the coils represented concepts W-1, W-2 and W-6. Particularly, the adaption of furnace operation conditions, as well as cooling intensity to the line speed, assured that the simulated optimum parameters could be achieved. The obtained results, with respect to mechanical properties and microstructural characteristics, are summarized in Table VI.

Table VI. Properties and Microstructural Characteristics of Cold Rolled Strip (Cold Reduction 57%) for the Two Different Galvanizing Cycles

	HDG1			**HDG2**		
Mech. properties in Transverse direction	**W-1 (Nb free)**	**W-2 (+Nb)**	**W-6 (++Nb+Ti+B)**	**W-1 (Nb free)**	**W-2 (+Nb)**	**W-6 (++Nb+Ti+B)**
YS (MPa)	336	392	537	317	384	497
TS (MPa)	560	655	856	525	626	783
A80 (%)	26	22	15	28	23	18
n-value	0.17	0.16	0.12	0.17	0.15	0.11
Grain size (ASTM)	13	13	13	11	11	12
Ferrite (%)	70	60	65	80	75	65
Bainite (%)	10	20	0	5	15	0
Martensite (%)	20	20	35	15	10	35

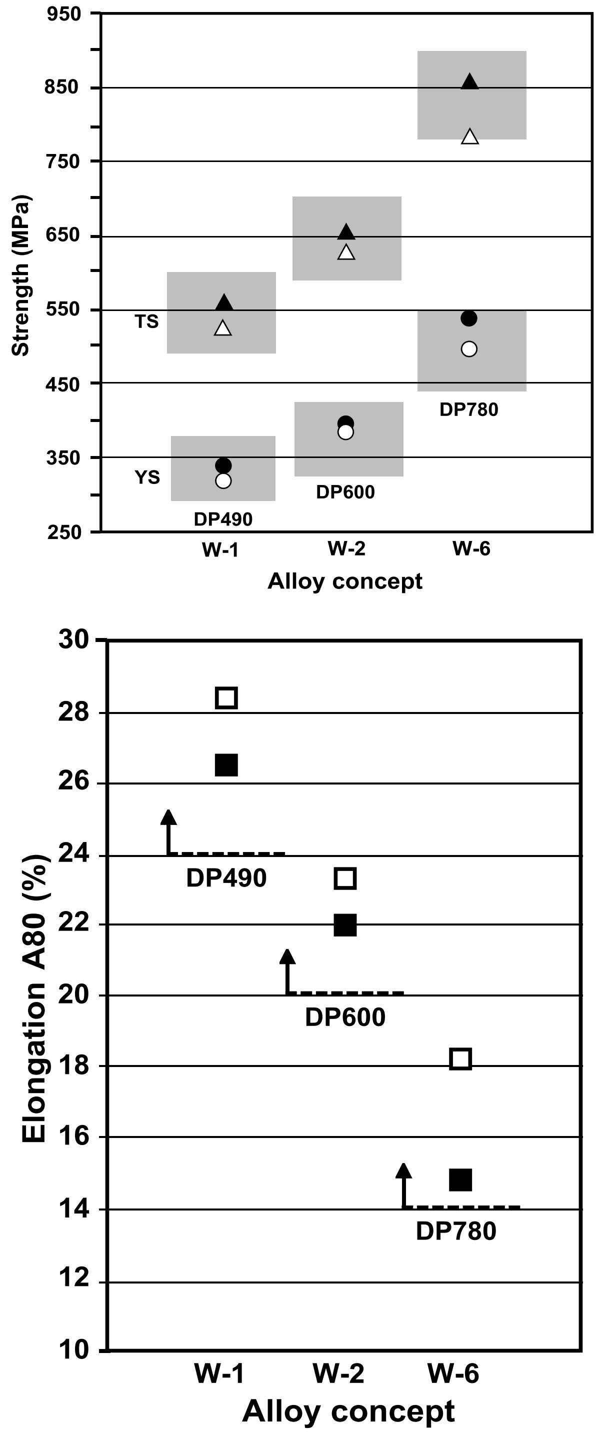

Figure 12. Mechanical properties of produced coil materials in relation to property ranges specified by VDA239 (filled symbols: horizontal HDG1, open symbols: vertical HDG2).

Figure 12 summarizes the obtained strip properties after treatment in the vertical hot dip galvanizing line. The base analysis consisting of 0.09%C, 0.25%Si, 1.85%Mn and 0.4%Cr in combination with stepwise increased addition of Nb, as well as Ti and B (W-6), succeeds in generating the strength classes of 500, 600 and 800 MPa. Using the process provided by the horizontal galvanizing line, a similar gradation of strength is obtained whereby the actual strength is approximately 40 MPa higher in each grade as compared to the vertical line.

Press Shop Performance

The performance of the industrially produced material was verified for several commercial car body parts having gages between 1 and 2 mm, as described in Table VII. The press shop trials were performed using production dies that were not specifically tuned to the new material. Although in regular production, softer HSLA steels with somewhat increased gage are being stamped, the majority of the trial material could be successfully formed into parts. Due to the increased base strength of the trial material being further enhanced by cold working and bake hardening, a weight reduction potential in the range of 10 to 20% is achievable compared to the HSLA material.

Two examples of a successful trial part made from the grade DP600 (option W-2) are shown in Figure 13 and 14. In the formed part the actual strength after drawing, as well as after drawing and baking, was determined. Therefore, small tensile samples with a gage length of 15 mm were taken at appropriate positions taking the original rolling direction into account. Figure 14 indicates this procedure, as well as the sample geometry for another trial part, representing a seat cross member that was manufactured from DP600 in 1 mm gage.

Table VII. Die Trial Schedule for Various Press Parts and Strength Options
(Thickness Reduction Compared to the HSLA Material as Indicated)

Option	Grade acc. to VDA239	Gage (mm)	Press part	OEM	Configuration	Original material (mm)
W-1	CR290Y490T-DP	1.0	Seat cross member	VW Golf PQ35	Double attached	HSLA 1.25
W-2	CR330Y590T-DP	1.0	Seat cross member	VW Golf PQ35	Double attached	HSLA 1.25
W-2	CR330Y590T-DP	2.0	A-pillar lower outside	Audi A5 Cabriolet	Double attached	HSLA 2.25
W-2	CR330Y590T-DP	1.0	Reinforcement D-pillar	Audi A6 Avant	Double attached	HSLA 1.20
W-3	CR440Y780T-DP	2.0	Rail front	N.N.	Single part	HSLA 2.00

Figure 13. A-Pillar lower outside made from 2 mm CR330Y590T-DP (option W-2).

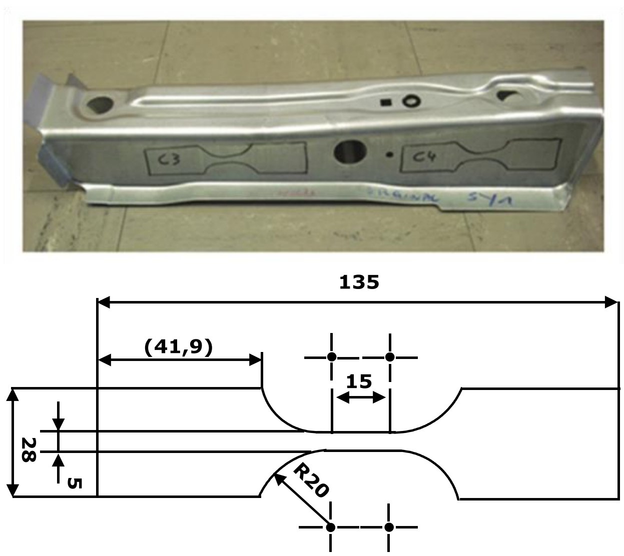

Figure 14. Seat cross member made from 1 mm CR330Y590T-DP with marked samples for tensile tests and schematic tensile test specimen showing dimensions.

Comparison of actual mechanical properties, ie. after forming and baking versus the original properties in the as delivered sheet, is provided in Table VIII for three different trial parts. The following observations can be made:

1. The as-delivered material reveals a very high isotropy for both yield and tensile strength, which is particularly favorable for stretch forming operations;
2. After forming, yield strength is significantly increased by work hardening. The amount of work hardening depends on the severity of deformation and the direction. The magnitude of the work hardening effect can bring an increase of over 200 MPa in yield strength and of around 50 MPa in tensile strength;
3. The bake hardening effect was also found to be rather high as is typical for DP steels. In some cases the yield strength increase by this effect is in the order of 100 MPa while its influence on the tensile strength is rather limited.

Table VIII. Actual Measured Properties in Trial Parts after Stamping (Work Hardening) and Baking Compared to the Original Sheet Properties

Stamped part	**Condition**	**Yield strength (MPa)**		**Tensile strength (MPa)**	
	Orientation to rolling direction	transverse	parallel	transverse	parallel
Seat cross member	As delivered sheet (1 mm)	377	374	660	658
	Pressed part (PP)	570	563	669	731
	PP after baking (180 °C / 20 min)	658	622	687	697
A-pillar lower outside	As delivered sheet (2 mm)	362	363	620	622
	Pressed part (PP)	447	385	626	622
	PP after baking (180 °C / 20 min)	566	560	644	641
Reinforcement D-pillar	As delivered sheet (1 mm)	377	374	660	658
	Pressed part (PP)	605	580	702	645
	PP after baking (180 °C / 20 min)	639	659	678	695

Resistance against Edge Cracking

DP steels generally allow forming of complex shapes due to their high elongation. However, practical experience repeatedly revealed unexpected failure, such as sheared edge splitting during flanging operations, as indicated in Figure 15. Highly localized strain leads to the initiation of micro damage at the ferrite-martensite phase boundary. The induced micro damage subsequently grows into a propagating crack under the applied stress. The larger the size of an initial damage site, the smaller is the critical stress required for crack propagation. A crack typically propagates along the ferrite-martensite interface. Hence, a refined microstructure and non-agglomerated martensite islands, in particular, should characterize optimized DP steel.

Another significant influence on the hole expansion ratio of DP steel originates from the difference in hardness between the soft ferrite phase and the hard martensite phase. Hosoya et al. [12] have demonstrated this effect by performing different low temperature tempering treatments to quenched DP steel in the strength range of 800 to 1000 MPa. Their results suggest a linear relationship between the hardness difference and the hole expansion ratio. Therefore, the volume fraction of martensite in the steel appears to be of less significant impact, provided that its distribution is homogeneous.

The developed low-C microalloyed DP steel concept principally provides all the mentioned prerequisites for high resistance against edge cracking:

- Nb microalloying provides general refinement of ferrite and martensite, resulting in relatively short hard-to-soft interfaces;
- Reduced segregation and pearlite banding due to sub-peritectic alloy design in combination with optimized coiling conditions after hot rolling avoid martensite agglomeration;
- The low overall C content leads to reduced C enrichment in austenite during intercritical annealing (for a given austenite fraction, which in a lower C steel will occur at a higher annealing temperature) and hence softer martensite after quenching resulting in a smaller hardness difference between ferrite and martensite phases.

Benchmarking of the newly developed Nb microalloyed DP600 grade (variant W-2) versus non-optimized reference materials of the same grade using hole expansion testing according to ISO-TS 16630 indeed revealed a clearly superior performance. The hole expansion ratio of the developed low-C steel is above 60%, while that of existing non-optimized DP steels of 590 MPa strength level shows only values of around 40% (Figure 16). Although the hole expansion ratio is currently not specified by VDA239, it is an important criterion in several automotive markets. Accordingly, it can be expected that the hole expansion ratio may be included in VDA239 during a future revision of this specification.

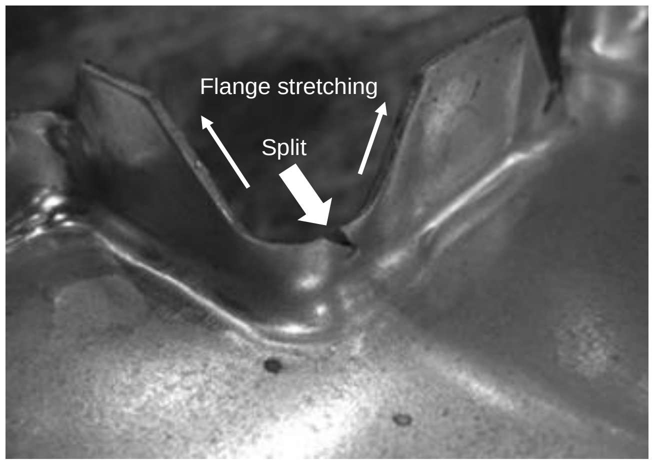

Figure 15. Sheared edge cracking phenomenon in DP steel under stretch flanging conditions [13].

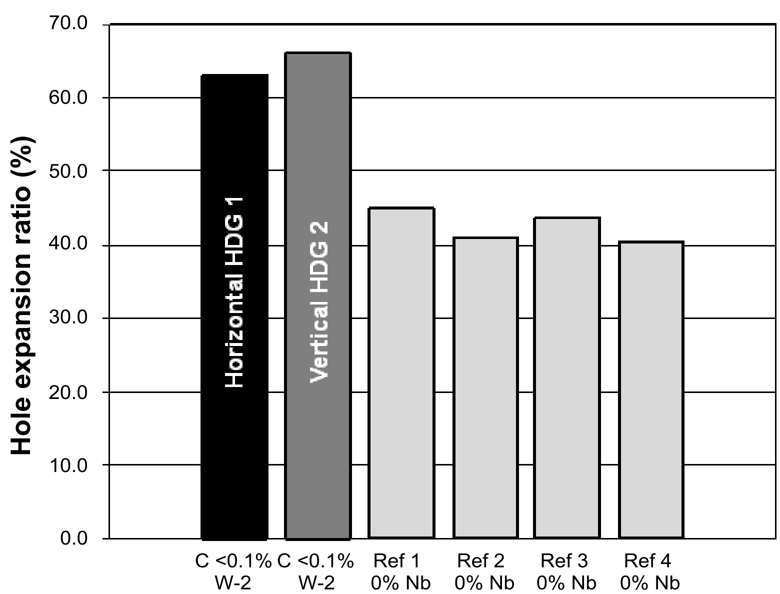

Figure 16. Performance of newly developed low-C Nb microalloyed DP steel in ISO-TS 16630 hole expansion test in comparison to non-optimized reference material.

Conclusions

A low C based modular alloying concept for hot dip galvanized DP steels has been developed and successfully tested on press stamped parts using commercial dies.

Based on laboratory simulations, a common alloy platform based on low C, Si, Mn and Cr was defined. Systematic variation of the Nb microalloy content, as well as selective additional microalloying of Ti and B allowed producing DP steels in the strength range of 500 to 800 MPa.

The concept was shown to be robust under greatly varying processing conditions in the cold rolling and galvanizing operation. This allows running the same alloy concept through two rather different galvanizing lines. It is also possible to produce galvanized DP steel with a large thickness. The mechanical properties of all grades safely meet the requirements of the VDA239 specification. Very good isotropy of properties could be also verified. Cold working and bake hardening were observed to provide a substantial strength increase in finished press stamped parts.

The developed DP steel furthermore exhibited a substantially improved hole expansion behavior. This enhances the resistance against cracking under particular forming operations such as stretch flanging or bending.

References

1. Anonymous, VDA 239-100 Sheet Steel for Cold Forming, Publication "Verband der Autoindustrie VDA," 20.08.2011, www.vda.de.

2. T. Dettinger et al., VDA Werkstoffblatt 239: "An Approach for a Global Sheet Steel Specification," *Proceedings of the 3rd International Conference on Steels in Cars and Trucks,* Salzburg (2011), 273.

3. K. Hashiguchi et al., "Effects of Alloying Elements and Cooling Rate After Annealing on Mechanical Properties of Dual Phase Steel Sheet" (Kawasaki Steel Technical Report No. 1, September 1980), 70.

4. P. Larour et al., "Influence of Post Uniform Tensile and Bending Properties on the Crash Behaviour of AHSS and Press-hardening Steel Grades," *Proceedings of the IDDRG 2010 Biennial Conference*, Graz, Austria, (2010).

5. M. Calcagnotto, "Ultrafine Grained Dual-phase Steels" (Ph.D. thesis, Verlag Shaker, Aachen, 2010).

6. A. Pichler et al., "Advanced High Strength Thin Sheet Grades: Improvement of Properties by Microalloying Assisted Microstructure Control," *Proceedings of the International Symposium on Niobium Microalloyed Sheet Steel for Automotive Applications*, TMS, (2006), 245.

7. Y. Granbom, "Influence of Niobium and Coiling Temperature on the Mechanical Properties of a Cold Rolled Dual Phase Steel," *La Revue de Métallurgie,* 4 (2007), 191.

8. H. Mohrbacher, "Effects of Nb in Galvanized Advanced High Strength Steels for Automotive Applications," *Proceedings of Galvatech '07*, Osaka, (2007), 386.

9. W. Bleck, A. Frehn and J. Ohlert, "Niobium in Dual Phase and TRIP Steels," *Proceedings of the International Symposium Niobium 2001*, TMS, Orlando, (2001), 727.

10. O. Girina, N. Fonstein and D. Bhattacharya, "Effect of Nb on the Phase Transformation and Mechanical Properties of Advanced High Strength Dual Phase Steels," *Proceedings of New Developments on Metallurgy and Applications of High Strength Steels,* Buenos Aires (2008).

11. JMatPro® developed by Sente Software Ltd. http://www.jmatpro.com.

12. K. Hasegawa et al., "Effects of Microstructure on Stretch-Flange-Formability of 980 MPa Grade Cold-rolled Ultra High Strength Steel Sheets," *The Iron and Steel Institute of Japan International*, 44 (3) (2004), 603.

13. T. Hebesberger et al., "Dual-phase and Complex-phase Steels: AHSS Material for a Wide Range of Applications," in: *SCT 2008, Future Trends in Steel Development, Processing Technologies and Applications: Bringing the Automotive, Supplier and Steel Industries, Proceedings of the 2nd International Conference on Steels in Cars and Trucks*, 1st–5th June, 2008, Wiesbaden, ed. by Bernhard Fuchsbauer and Hans J. Wieland, (Düsseldorf: Verlag Stahleisen, 2008), 456-463.