

# THE EFFECTS OF NIOBIUM MICROALLOYING IN SECOND GENERATION ADVANCED HIGH STRENGTH STEELS

By

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## ABSTRACT

Advanced high strength steels have found increasing use in car body engineering since the late 1990's. The use of such steel grades allowed to seriously increase the crash resistance and to significantly reduce the weight of the body-in-white. However, several manufacturing issues in the press shop and assembly plant indicated potential to improve the first generation of advanced high strength steels.

For the second generation of advanced high strength steels thus a number of development targets were defined as to improve the manufacturing behavior. As such the carbon content had to be reduced to facilitate welding and the balance of elongation and hole expansion ratio had to be optimized with respect to forming. One of the most critical challenges is that of avoiding delayed cracking typically occurring in steels of more than 1000 MPa strength.

Niobium microalloying was found to be a very effective means of achieving the targets set for the second generation advanced high strength steels. The beneficial impact of Niobium can be due to three basic effects: grain size control, transformation control and precipitation hardening. These three effects can be used individually or in combination. The paper demonstrates how to utilize these effects and indicates their compatibility with the typical processing routes of automotive sheet production. Furthermore concepts for new high strength steel types will be indicated.

**Keywords:** Niobium microalloying, microstructure, grain refinement, precipitation hardening, transformation hardening, hydrogen trapping

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## INTRODUCTION

In recent passenger vehicles a broad variety of high strength steel grades has been introduced replacing mild steel grades. Often the share of mild steel in European body designs is nowadays below 50% of the total body weight and in particular cases even below 30%. Accordingly, the share of high strength steel has significantly increased. The focus has been much on multiphase steels (DP, CP, TRIP) during recent years, which typically account for around 20% of the body weight in current vehicles. The remaining part spectrum is mainly made from microalloyed (HSLA), high strength interstitial free (IF-HSS), bake hardening (BH) and conventional CMn steel grades. Figure 1 ranks these steel grades according to yield strength and elongation. It is the aim of developing steels with good formability at high strength. Taking elongation as a criterion, DP and particularly TRIP steel appear to have the highest potential to reach this aim.

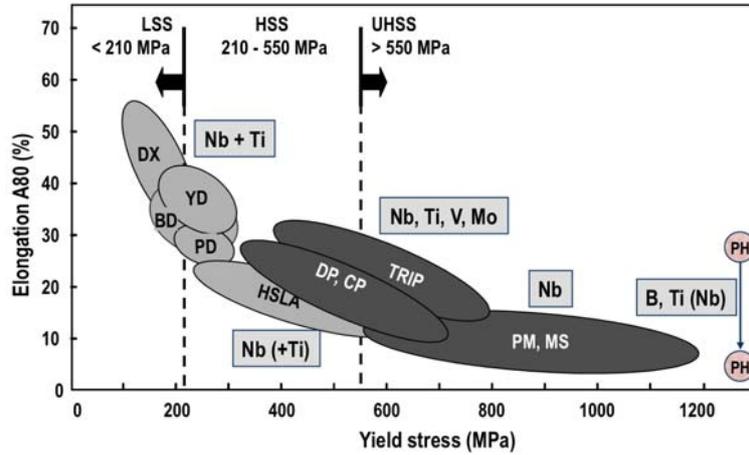
Automotive forming methods often consist of a sequence of individual forming operations. With regard to these specific forming conditions the mere contemplation of the strength–elongation diagram (Figure 1) is not sufficient to select the optimum material. Each forming mode has additional demands with regard to specific mechanical parameters like the Lankford parameter ( $r$ -value), work hardening coefficient ( $n$ -value) and hole expansion ratio ( $\lambda$ -value). These parameters are strongly related to microstructural features of the material and these can be influenced by niobium microalloying in combination with a suitable processing strategy.

Figure 2 relates the hole expansion ratio ( $\lambda$ -value) to the strength. It is clear that for the same strength a very different level of performance can be achieved, depending largely on the microstructure of the steel. Accordingly, bainitic steel grades are performing best especially at high strength whereas DP and TRIP steel are relatively weak performers in that respect due to the inherent inhomogeneity of their microstructure. As a consequence sharp bending and severe stretch flanging are causing unexpected failure in these steel grades. Nevertheless, this relatively poor performance of multiphase steels can be still improved, particularly by refining the microstructure using Nb microalloying.

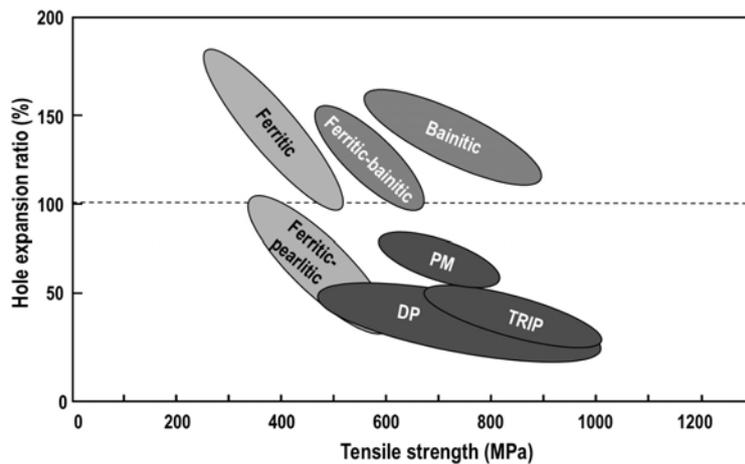
Automotive welding processes typically rely on low heat input methods such as laser and resistance spot welding as well as to a fewer extent on arc welding techniques such as MIG/MAG or TIG welding. The cooling speed in the fusion zone after welding is usually so high that martensite is formed. The hardness of that martensite solely depends on the carbon content of the base material [1]. On the contrary, for those steels containing martensite as a majority phase already in the base microstructure, softening in the heat affected zone (HAZ) is experienced under any welding process. This leads to a severe loss of strength in the HAZ and thus to unpredictable failure. The performance of the various steel grades with respect to welding is ranked in Figure 3.

The first generation of multiphase steels mainly focused on achieving the baseline properties in terms of the stress-strain curve and the total elongation versus tensile strength. Many efforts are being done to develop 2nd-generation multiphase steels, which add a number of improvements regarding particular forming operations and weldability. As such, low carbon equivalent (LCE) variants of DP and TRIP steels have been designed. DP steels up to 1000 MPa tensile strength with the carbon content not exceeding 0.1% are already in use. For TRIP steel there are developments aiming at carbon levels of 0.15% or even below. The loss of strength due to the reduction of carbon has to be recovered by other strengthening methods such as grain refinement and precipitation hardening due to Nb microalloying. Particularly in TRIP steel alternative mechanisms to stabilize a sufficient amount of retained

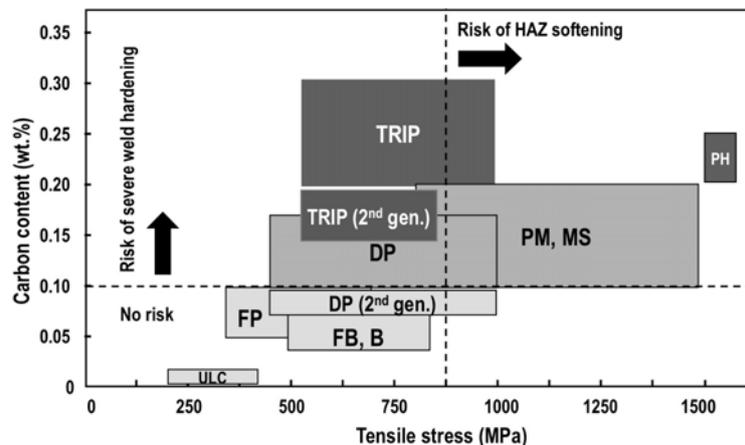
austenite have to be enabled. Other second generation variants are high yield strength as well as high hole expansion DP steels. In the latter a general refinement of the microstructure, avoidance of very high hardness gradients between the phase constituents and a strengthening of the ferrite phase are typically employed to achieve the property improvement.



**Figure 1** Strength and elongation diagram of cold rolled steel grades and potential application of microalloying elements.



**Figure 2** Dependence of hole expansion ratio on strength and microstructure.



**Figure 3** Ranking of the weldability for different automotive steel types

## PRINCIPAL EFFECTS OF NIOBIUM

Niobium is known as an alloying element by which austenitization, recrystallization, grain growth, phase transformation, and precipitation behavior can be controlled in a very efficient way and which can vary the mechanical properties in a wide range. With regard to multiphase steels niobium affects among other things the transformation of austenite to ferrite and to bainite and thus volume fraction and stability of the retained austenite which is the key to the outstanding mechanical behavior of TRIP steels. Thus, niobium can be utilized as a metallurgical tool to adjust microstructure and properties.

### Hot-rolling

The principal effect of Nb is a refinement of the microstructure, which relates to the retardation of recrystallization by solute Nb and by Nb(C,N) precipitates finish hot rolling [2]. The pan caked austenitic microstructure translates into a fine-grained ferrite after hot rolling. In case not all Nb has been precipitated at the end of hot rolling, it can precipitate during coiling leading to a further increase of strength. This is particularly effective when the coiling occurs at temperatures of around 600°C. Nb being in solid solution at the end of hot rolling also has the effect of retarding the austenite-to-ferrite phase transformation. This effect in combination with a reduced coiling temperature of around 500°C can result in a particularly fine-grained ferritic-bainitic microstructure, preserving some Nb in solid solution.

### Cold-rolling and annealing

Cold rolled strip is subsequently annealed and galvanized to produce the majority of today's automotive sheet. The microstructural features of the cold rolled band necessarily relate to the prior hot rolled microstructure. However, particular features can be achieved depending on the annealing strategy. For multiphase steels continuous annealing either in a CAL or CGL is used.

For cold rolled multiphase steels two different hot rolling approaches prior to cold rolling are conceivable [3]. One of them is leading to a soft material with a microstructure of ferrite and pearlite. This microstructure requires the application of high coiling temperatures of around 700°C. The material is well suited to subsequent cold rolling. Alternatively, a hot rolling cycle making use of a lower coiling temperature in the range of bainite formation can be applied. Since bainite forms at about 500°C and represents a relatively hard microstructural component, distinctly higher rolling forces must be applied when cold rolling the material. However, it is expected that this process route is leading to a more homogeneous and fine-grained microstructure and superior properties of the as annealed product. The effects of Nb in the subsequent annealing cycle can be the following:

- Nb refines the hot-rolled grain structure and thus the final microstructure improving the strength as well as other aspects of performance.
- The fine-grained microstructure results in a quicker austenitization during intercritical annealing.
- Nb provides additional precipitation hardening contributing to strengthening of the product.
- Nb increases the stability of austenite by impairing the martensite nucleation in TRIP steel.
- Nb enhances the bainite reaction, particularly at temperatures of galvannealing

enriching the remaining austenite with carbon and stabilizing it.

By making dedicated use of these Nb induced effects, steel properties can be optimized and the processing window can be enhanced leading also to lower property scattering.

## **IMPROVING MULTIPHASE STEELS BY NIOBIUM MICROALLOYING**

After having gained initial experience with multiphase steels in automotive production, several improvement actions appear to be desirable to optimize their properties for the application in future vehicle generations. These issues relate to the forming as well as the welding behavior. More precisely the following trends are in progress:

- Reduction of scattering in mechanical values to better control spring-back.
- Improve bending and stretch flanging behavior.
- Reduce carbon content for better weldability.
- Avoid microstructural inhomogeneities (particularly martensite bands in the sheet center).
- Further increase of strength level.

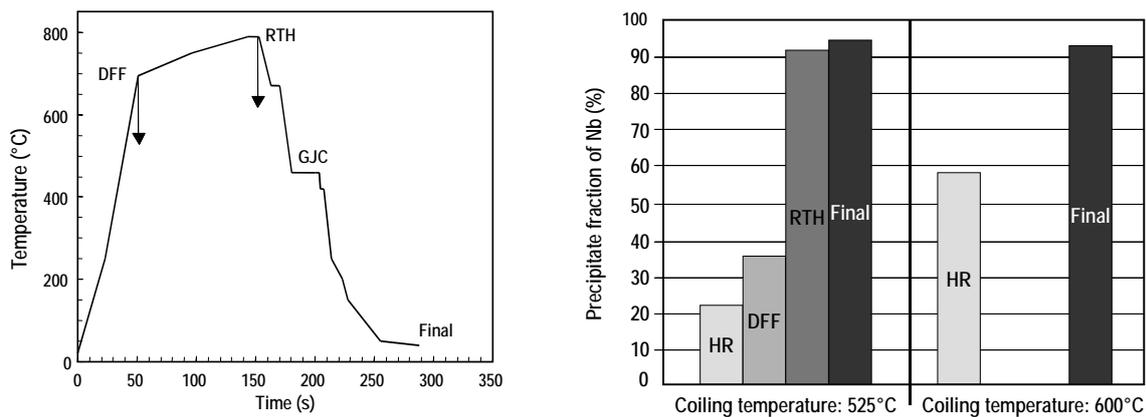
### **Dual Phase Steel**

The strength level of DP grades is mainly determined by the fraction and strength of ferrite and martensite. The strength level of ferrite can be controlled by solid solution hardening, precipitation hardening and the grain size. As the addition of the most efficient solid solution hardening elements Si and P is restricted due to surface defects and weldability aspects, respectively, the application of microalloying elements such as Nb resulting in precipitation hardening and grain refinement forms an interesting alternative for controlling the ferrite strength.

Several projects have been undertaken to investigate this potential of Nb in more detail [4,5]. As mentioned before, Nb takes effect already by refining the hot band structure. Depending on the coiling temperature more or less Nb will remain in solid solution (Figure 5). Nb being in solid solution after hot rolling has then the potential of precipitating during the annealing stage as shown for a hot dip galvanizing cycle in Figure 4. During the soaking period of about 60 seconds, most of the Nb is precipitated as very fine particles rendering the basis for a significant strength increase. For the higher coiling temperature the effect is less pronounced since the amount of Nb that can precipitate in the HDG cycle is smaller.

A second strength increasing effect originates from the significant grain refinement that is achieved upon Nb microalloying. This not only leads to finer ferrite and martensite grains as such, but also the distribution of these phases is typically found to be more homogeneous (Figure 5). Specifically martensite band formation in the centerline of the sheet can be avoided resulting in an improvement of the forming behavior. Furthermore, the transformation kinetics after quenching from the intercritical annealing temperature is influenced by Nb microalloying. It has been observed that Nb microalloyed DP steel has a significantly enhanced ferrite formation, particularly at high cooling rate [4]. This results consequently in a reduced amount of martensite, which in turn is richer in carbon and

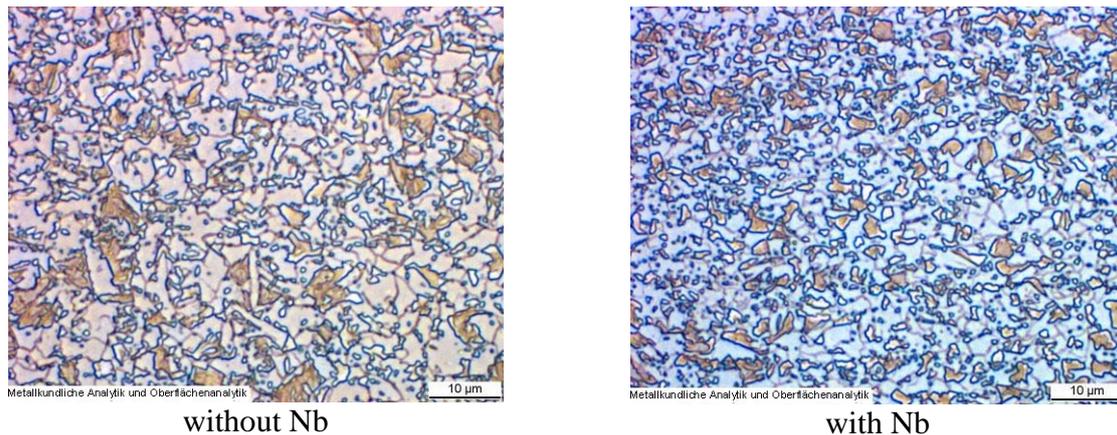
accordingly has a higher strength. On the other hand, the refined cold-rolled microstructure should easier nucleates austenite during the annealing cycle, resulting in a more homogeneous distribution of martensite later after quenching. Thus, it is possible to produce a DP grade of a given strength with less brittle martensite phase and more ductile ferrite phase [5]. Actually one can expect two types of ferrite in such a Nb microalloyed DP steel. The old ferrite, originating from the hot band structure, can be assumed to contain Nb precipitates. Depending on the intercritical annealing temperature, this ferrite may not even be fully recrystallized due to the retarding effect by the Nb precipitates. This is in contrast to the new ferrite originating from the decomposition of the intercritical austenite. Thus these two ferrite types must have different strength levels.



**Figure 4** Precipitation behavior in a DP800 steel during a HDG cycle (0.1% C, 1.7% Mn, 0.25% Si, 0.55% Cr, 0.03% Nb) [6]

As a consequence of these microstructural features of Nb microalloyed DP steel, a number of advantages with respect to automotive manufacturing can be identified:

- Grain refined DP steels were shown to have a higher resistance against splitting in forming operations where highly localized strain is imposed, such as bending, stretch-flanging and also clinching. This is particularly helpful as sharp bending radii are an effective means to reduce spring-back in forming operations.
- The strength gain by grain refinement and Nb precipitation hardening allows to reduce the carbon content to below 0.1 percent especially in DP grades of 780 MPa or higher. This also avoids the traditional peritectic composition (0.10–0.15% C) of such steels and thus the typical inhomogeneous microstructures due to segregation. Steels with less carbon develop a lower hardness in automotive welding and render a more ductile weld seam.
- DP steels of increased strength typically have martensite as a majority phase. Such a microstructure shows pronounced heat affected zone softening due to tempering of the martensite. Nb microalloyed DP steel of the same strength contains less martensite and thus less risk of HAZ softening.
- The addition of Nb to DP steels was found to increase the operating window during the annealing cycle in the HDG line resulting in more stable properties of the steel.



**Figure 5** Influence of Nb microalloying on the microstructure of a DP600 steel grade [4]

### TRIP Steel

The mechanical properties of TRIP grades are determined by the fraction and strength of ferrite, bainite and austenite and in particular, on the stability of the retained austenite against strain induced martensitic transformation. Like in DP steels an overall control of the microstructure and the size of the different phases seem to be key factors for optimizing the final mechanical properties. Nb microalloying accordingly strengthens the ferritic matrix by means of grain refinement and precipitation hardening. When producing cold-rolled TRIP steel, the combination of a low coiling temperature ( $\sim 500^{\circ}\text{C}$ ) after hot rolling and Nb microalloying was found to cause a remarkable decrease of the martensite start temperature [7]. Nb retained in solution precipitates as very small particles during intercritical annealing controlling the grain size and ensuring a homogeneous microstructure.

According to several authors [8,9] the coiling temperature determines the precipitation state of Nb when alloying TRIP-steels with Nb. At low coiling temperatures (around  $500^{\circ}\text{C}$ ), Nb stays partly in solid solution whereas at high coiling temperatures ( $700^{\circ}\text{C}$ ) coarse Nb precipitates are formed and the influence of Nb on the mechanical properties diminishes. Using coiling temperatures between  $600$  and  $650^{\circ}\text{C}$ , small precipitates are formed being very effective in refining the microstructure by particle pinning and consequently, the highest tensile strength levels can be reached.

It has also been indicated that the fine-grained microstructure is responsible for a delayed bainite formation in Nb-alloyed TRIP-steels. The retarded bainite formation is attributed to an enhanced ferrite formation during cooling due to efficient nucleation in the fine-grained microstructure. The enhanced ferrite formation in turn results in a more effective carbon enrichment of the remaining austenite, stabilizing it and delaying transformation to bainite [8]. In addition to the enhanced ferrite formation, a deactivation of nucleation sites for bainite by the very fine dispersed carbonitrides could be made responsible for the delayed bainite formation kinetics [8]. Small amounts of Nb staying in solid solution are reported to retard the bainite formation kinetics as well [10].

The retardation of bainite formation is beneficial during overaging or when producing galvanized TRIP steel. Nb microalloyed TRIP steel shows already a high yield strength without overaging and the strength increase during overaging is rather marginal. Likewise the tensile strength is only slightly reduced whereas a non-Nb microalloyed TRIP steel suffers a significant loss in tensile strength during overaging (Figure 6) [4].

To obtain sufficient adhesion of the different ZnFe phases higher galvannealing temperatures are often required. However, an increased galvannealing temperature bears the

risk of decomposition of retained austenite. The austenite decomposition during galvannealing can be significantly reduced by Nb microalloying as shown in Figure 7. In a horizontal line, the retained austenite content appears to remain stable in Nb-microalloyed TRIP steel regardless of the galvannealing temperature. However, from its mechanical characteristics, the latter steel is rather a TRIP-aided DP steel of very high strength whereas the same alloying concept results in a TRIP 800 grade using the vertical line concept [5].

Like in DP steels, the microstructural refinement of Nb microalloyed TRIP steel results in a better performance under forming methods involving highly localized stress (bending, stretch-flanging). Besides the formation of detrimental martensite bands in the sheet center especially for heavier gages with accordingly lower cold reduction degree are avoided by Nb microalloying [11].

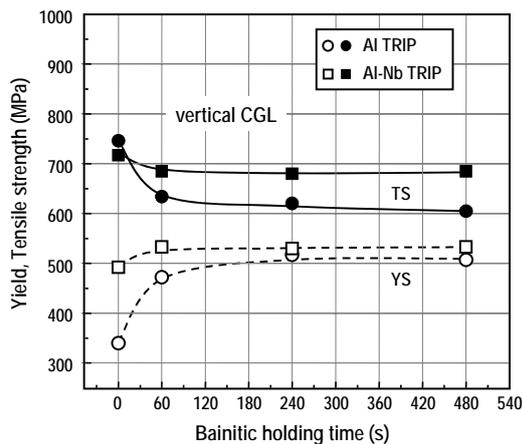


Figure 6 Influence of the bainitic holding time at 400°C on strength properties [4]

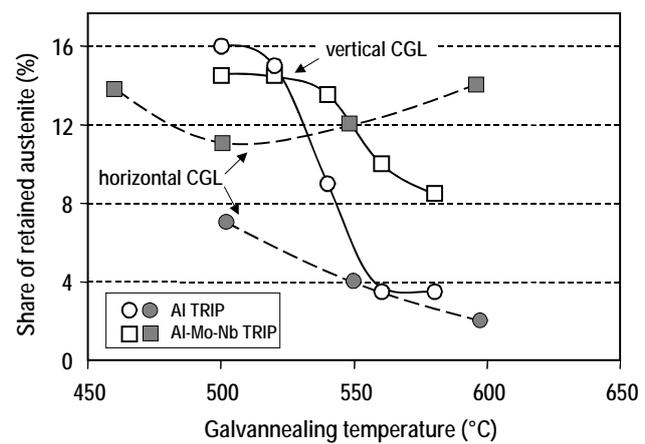
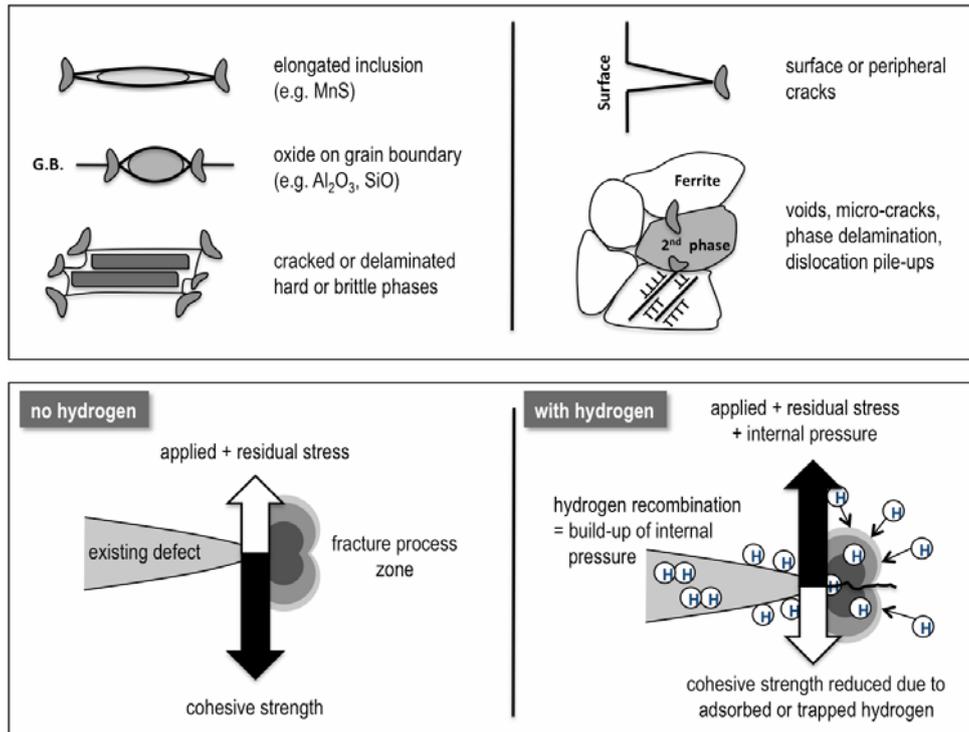


Figure 7 Influence of galvannealing temperature on retained austenite share [5]

### STRATEGY FOR DELAYED FRACTURE AVOIDANCE IN AHSS

Recently, multiphase steels of ultrahigh strength have been looked at concerning the risk of hydrogen induced delayed cracking. Delayed fracture can occur if a steel component containing previously generated defects and being subjected to a sub-critical stress collective is charged with hydrogen. Two effects may be responsible for the onset of fracture as indicated in Figure 8. Hydrogen diffuses into the fracture process zone at the tip of an existing defect. Localized concentration of hydrogen reduces the cohesive lattice strength in the vicinity of the defect. These changes can manifest themselves as an effect on a local stress criterion or a local strain criterion for fracture. In the former case, local brittle fracture modes such as cleavage or intergranular fracture are expected, while for the latter, it will be a more ductile fracture, requiring large strains and manifested most likely by micro-void coalescence. There are expected to be many common elements of behavior between the two, and further, it is by no means necessary that the macroscopic fracture mode closely follow in appearance these local events. The second effect is the accumulation and recombination of hydrogen in the cavity of the defect. The hydrogen molecule has a low diffusivity and cannot escape from the cavity anymore. Thus internal pressure is building up adding to the existing stress collective by applied and residual stresses. By either one or a combination of these hydrogen-induced effects an initial sub-critical stress collective can thus turn super-critical triggering crack growth. It then depends much on the toughness of the material if the expanding crack can be arrested.



**Figure 8** Initial microstructural defects and mechanism leading to hydrogen induced delayed cracking

As cornerstones of a strategy for reducing the delayed fracture risk in advanced high strength steels the following items should be considered:

- Steel cleanliness and avoidance of inclusions.
- Homogenization of the microstructure and balancing of the phase properties.
- Minimization of preexisting damage (voids, micro-cracks, phase delamination).
- Introduction of precipitates as hydrogen traps.

Steel cleanliness and avoidance of inclusions is a well know issue and particularly relevant to the production of HIC resistant line pipe steels. A respective steel treatment procedure was outlined in detail by Lachmund and Bruckhaus [12] and shall not be further discussed here.

### TRIP steel

The presence of banded microstructure, consisting of martensitic bands in the strip is typical for some higher alloyed advanced high strength steels. Severe plastic deformation may lead to the introduction of local damage in these hard bands. Since the small damage zones in the martensitic bands can trap hydrogen and enable hydrogen recombination, the reduction of banded structure in the case of such steels remains an important tasks to be solved. The metallurgical reason for the formation of banded structures is segregation during continuous casting. One principal solution of avoiding a banded structure lies in the optimization of process in the steel plant by using of soft reduction during continuous casting [13]. A second step is to increase the overall degree of deformation during hot and cold rolling. Finally, the global refinement of microstructure mainly by micro-alloying and phase control by Nb

microalloying offers an interesting potential for improvement. Pichler et al. [14] have clearly demonstrated that Nb microalloying to dual phase as well as TRIP steel brings about a remarkable refinement of the ferrite as well as the embedded second phases. Secondly, the refined hot band structure promotes ferrite formation upon intercritical annealing. By that the amount of second phase is somewhat reduced but contains in the average more carbon, i.e., retained austenite becomes more stable. The enhanced ferrite formation constitutes an additional protection against clustering of hard phases. Statistically the probability of nucleation of two hard phases next to each other is reduced. Recent observations by Imlau et al. [15] related the risk of void formation after straining of TRIP steel to the stability of the retained austenite. Less stable austenite transforms at lower strain and more completely into martensite rendering a higher risk of void formation and coalescence. In this respect the microstructural refinement by Nb microalloying is beneficial as well, since the smaller, higher carbon enriched austenite grains transform less easily into martensite [16].

### Dual phase steel

In dual phase steel a similar effect of Nb microalloying is observed as both the ferrite matrix and the martensite island sizes are reduced [14, 17]. This is a direct consequence of the finer grain size of the former hot strip. Hashimoto [18] pointed out that ferrite and martensite grain refinement by Nb addition results in higher strength, uniform elongation, n-value, BH and stretch flangeability. Particularly the refinement of the ferrite phase in combination with NbC precipitation increases its hardness. Hence the hardness difference between ferrite and martensite is diminished. Hebesberger et al. also demonstrated the superior bendability and stretch-flangeability of Nb microalloyed DP780 [19]. For a given press part geometry the risk of microscopic damage in bending areas or at strained edges is thus significantly reduced and thus the possibility of having micro damage sites allowing hydrogen recombination.

### Hydrogen deep trapping by precipitates

Besides microstructural refinement the dedicated introduction of precipitates acting as hydrogen traps is a crucial step in achieving anti-delayed fracture properties. The hot rolling and annealing practice control the state of precipitates. By selecting the rolling temperature avoids strain-induced precipitation can be promoted or avoided. The coiling conditions can trigger or suppress precipitation. The annealing conditions after cold rolling can either lead to precipitation or dissolution of particles as discussed above.

*Table 1 Hydrogen trapping energy of various nitrides and carbides in an iron matrix [20]*

Alloy system	Fe-Zr-N	Fe-Ti-N	Fe-Nb-N	Fe-V-N	Fe-Mo-N
Flat traps (kJ/mol H)	-20.4	-20.7	-18.2	-18.9	-19.3
Deep traps (kJ/mol H)	-56.1	-60.5	-54.9	-56.0	-56.0
Alloy system	Fe-Zr-C	Fe-Ti-C	Fe-Nb-C	Fe-V-C	Fe-Mo-C
Flat traps (kJ/mol H)	-19.9	-20.6	-18.3	-17.2	-13.9
Deep traps (kJ/mol H)	-58.5	-58.5	-56.0	-57.0	-56.5

Grabke et al. [20] in detail investigated the binding energy of various typical carbide and nitride formers as flat traps and deep traps. The influence of the various carbide and nitride formers on the binding energy is of secondary importance as can be seen from the data

shown in Table 1. Obviously there are a large number of possible choices when producing automotive strip, but the question is whether the microalloying element to be precipitated is compatible to the rolling/annealing strategy. Ti always tends to form bigger TiN particles at very high temperature, i.e. during continuous casting, which are detrimental with regard to toughness and HIC. On the other hand, V barely precipitates as strain induced particles during hot rolling. Furthermore, vanadium precipitates that have been generated by a suitable coiling condition have the tendency to re-dissolve during annealing or autenitizing treatments. Therefore, from a practical point of view, only Nb offers the potential of thorough precipitation of incoherent particles that are not too large and have still a strong hydrogen trapping capability. In addition the Nb precipitates are effective in controlling the microstructure during rolling and annealing treatments leading to a refined and homogeneous phase distribution as outlined before.

## CONCLUSION

Although Nb microalloying was not readily applied in first generation multiphase steels, intensive research meanwhile has indicated a very interesting potential of assets with regard to safe production of such steels and also to superior application properties and the end customer. These assets are based on grain refinement, precipitation hardening, and transformation control caused by Nb microalloying and appropriate processing in the hot rolling, cold rolling and galvanizing mill.

The combination of microstructural homogeneity, avoidance of pre-damage and dispersion of hydrogen trapping particles is suggested as an approach of avoiding or minimizing the risk of hydrogen induced delayed fracture in advanced high strength steels. Although the hydrogen trapping energy is little influenced by the actual carbide or nitride forming alloying element, Nb microalloying appears to be the best solution. Due to its solubility and precipitation kinetics Nb offers the best compatibility in terms of precipitation behavior with the typical thermo-mechanical treatment of automotive high strength steel sheet. Compared to vanadium and titanium these benefits can be more efficiently achieved with a smaller alloying addition of niobium making it interesting also from an economical point of view.

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