

# Chapter 7

## Martensitic Sheet Steels

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### 7.1 Introduction

Martensitic steels are the hardest type of steels featured simultaneously by high tensile strength and very high YS/TS ratio that is very important for the safety parts of cars that are intended to protect the driver and the passengers from intrusions during collisions. Because of the importance of this function, the production of martensitic grades of sheet steels historically started far earlier than that of AHSS, in particular, in the early 80s in the USA.

Microstructure of low-carbon martensitic steels is mainly composed of lath martensite as a result of austenite transformation during quenching after hot rolling or annealing. These steels are often subjected to post-quench tempering that improves ductility and toughness, as well as provides good formability even at very high yield strength.

Strength of martensitic grades is practically controlled by the carbon content; however, the alloying elements are added to achieve the necessary hardenability during processing and to affect other properties such as ductility, bendability, and delayed fracture resistance.

## 7.2 Martensitic Grades for Roll Forming

Until recently, the majority of parts in modern cars with martensitic structure were produced using annealed martensitic steels. The existing group of martensitic grades includes steels with TS = 900, 1100, 1300, and 1500 MPa. With high YS/TS ratio, typically above 0.85, these materials are used preferably for roll forming that has proved to be very effective method to manufacture numerous car parts critical for crash safety. In contrast to steels intended for forming using other methods, steels for roll forming should have high yield to tensile strength ratio to minimize strength gradient and confine the plastic strain to bent corners. High YS/TS ratio also imparts high initial and therefore high retained strength of nondeformed portions of the profile.

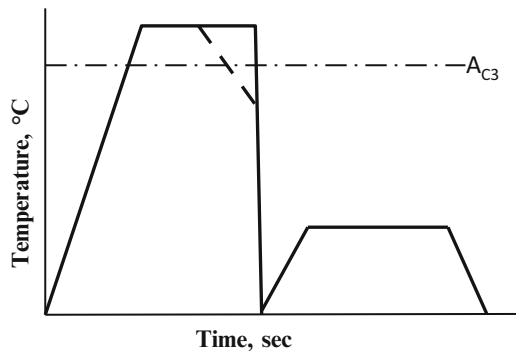
### 7.2.1 Processing and Compositions of Annealed Martensitic Grades

In fact, any continuous annealing line with water quenching capability can produce martensitic grade.

Composition of annealed martensitic grades depends on the design of cooling/water quenching sections. As shown in Fig. 7.1, continuous annealing lines differ in capability to use water quenching directly from soaking at temperatures of full austenitization (solid line) and those that have unavoidable temperature drop in the pre-quenching cooling section (dashed line) with some restrictions for maximum annealing temperatures.

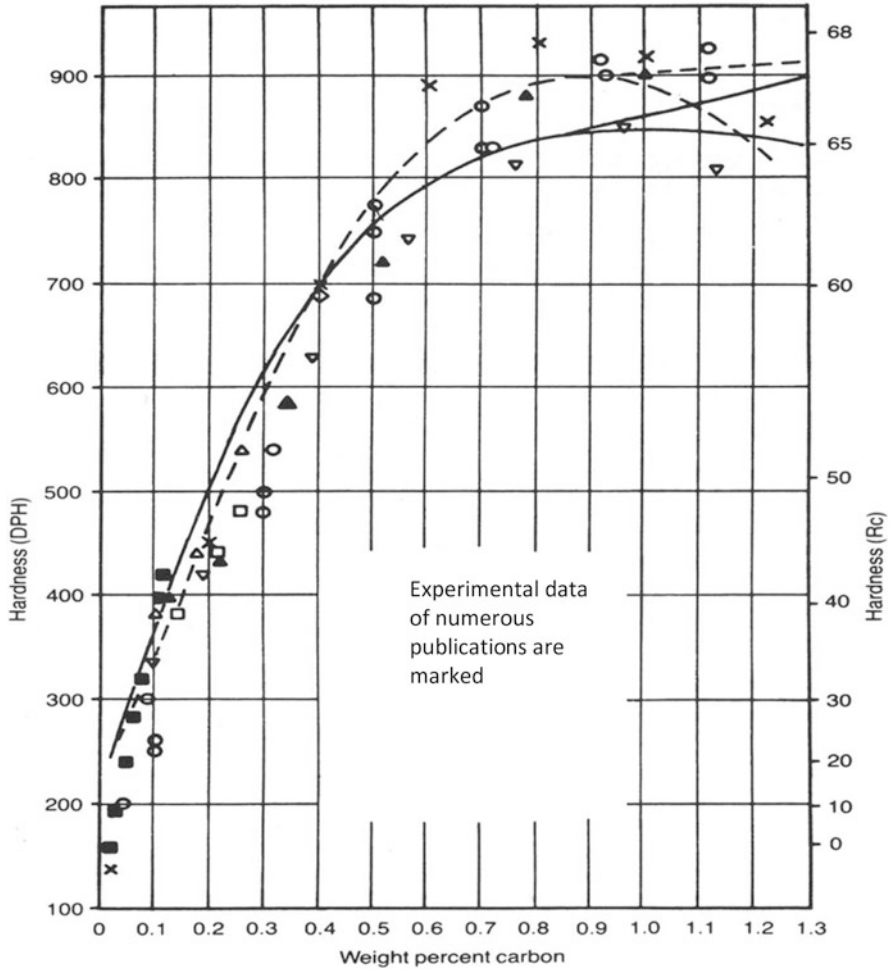
Therefore, in the first case, the complete austenite-to-martensite transformation is possible with minimum alloying, Table 7.1. As shown in Fig. 7.2, the strength of martensite is mostly controlled by carbon content. With up to 0.5 % C, the effect of carbon on steel hardness is practically linear (Krauss 2005). Consequently,

**Fig. 7.1** Typical thermal cycles for CAL equipped with water quenching



**Table 7.1** Chemical compositions of lean martensite grades (ArcelorMittal 2014)

	C	Mn	P	S	Other	Form
M900	0.08	0.45	0.01	0.015	B, Ti	CR, EG
M1100	0.12	0.45	0.01	0.015	B, Ti	CR, EG
M1300	0.19	0.45	0.01	0.015	B, Ti	CR, EG
M1500	0.25	0.45	0.01	0.015	B, Ti	CR, EG
M1700	0.30	0.45	0.01	0.015	B, Ti	CR



**Fig. 7.2** Effect of carbon on strength of martensite (Krauss 2005)

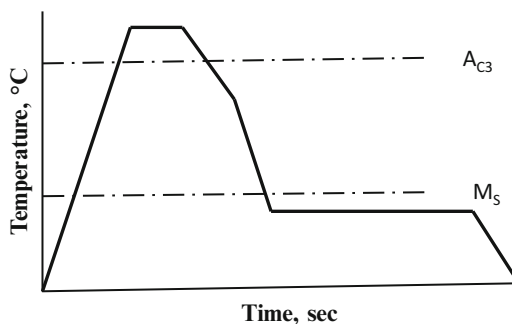
martensite grades with TS = 900, 1100, 1300, and 1500 MPa differ only in carbon content with very lean alloy composition of 0.45 % Mn and small additions of boron protected from interaction with nitrogen by microalloying with Ti. The mechanical properties of such steels are presented in Table 7.2.

**Table 7.2** Mechanical properties of lean martensite grades (ArcelorMittal 2014)

	Test—direction	Yield strength (MPa)	Ultimate tensile strength (MPa)	Total elongation	Recommended bend ratio
M900	ASTM-L	877	1015	6	4T
M1100	ASTM-L	1018	1179	6	4T
M1300	ASTM-L	1212	1423	6	4T
M1500	ASTM-L	1370	1629	6	4T
M1700	ASTM-L	1520	1820	5	4T

**Table 7.3** Chemical composition of DOCOL grades (SSAB Catalog 2015)

Steel grade	C (%)	Si (%)	Mn (%)	P (%)	S (%)	Al <sub>tot</sub> (%)	Nb (%)
Docol 130 M	0.05	0.20	2.00	0.010	0.002	0.040	0.015
Docol 175 M	0.11	0.20	1.60	0.010	0.002	0.040	0.015
Docol 190 M	0.14	0.20	1.50	0.010	0.002	0.040	0.015
Docol 205 M	0.18	0.20	1.20	0.010	0.002	0.040	0.015
Docol 220 M	0.20	0.20	1.00	0.010	0.002	0.040	0.015

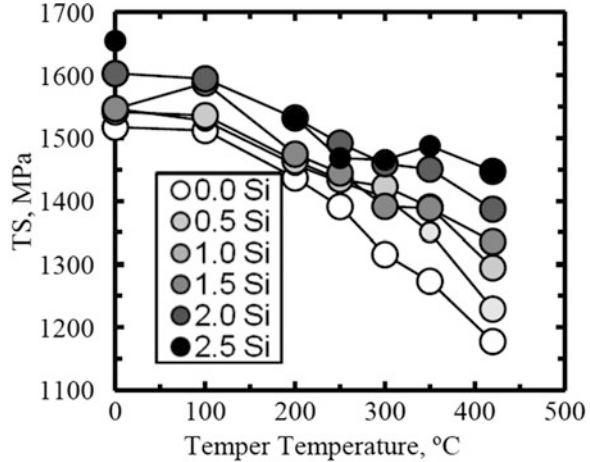
**Fig. 7.3** Thermal cycle to produce martensitic grades based on direct transfer from cooling to overaging (tempering) section

In the case of initial temperature drop (dashed line in Fig. 7.1), higher alloying is necessary to prevent the formation of ferrite and pearlite during initial slow cooling. Consequently, the corresponding martensitic grades produced, in particular, by SSAB (Olsson and Sperle 2006), contain additions of 1–2 % Mn (the higher the lower carbon content) to ensure sufficient hardenability of austenite, Table 7.3. Increased Mn content also helps to decrease the  $A_{c3}$  and therefore annealing temperatures of martensitic grades.

In the case of equipment with direct transfer to overaging zone shown in Fig. 7.3, alloying of steel should be even higher due to slower cooling rate (typically not higher than 70–80 °C/s) from soaking temperatures down to the overaging/tempering section. Depending on the target strength, the martensitic grades should have, besides boron additions, higher amount of Mn, Cr, and sometimes Mo.

Strong demand to further reduce the weight of automobiles and simultaneously achieve higher reliability of safety parts motivated steel producers to develop

**Fig. 7.4** Effect of Si additions and tempering temperatures (150 s holding time) on tensile strength of martensite (Johnson et al. 2013)



martensite grades with TS > 1700 MPa and >2000 MPa. There is a possibility to achieve that strength by the increase in carbon content with some microalloying aiming to ensure necessary resistance to delayed fraction. Other approaches include consideration of strengthening with alloying. In particular, in the study by Arlazarov et al., synergetic effect of carbon and manganese on the martensite strength and strain hardening was detected and was then taken into account. As a result, YS = 1439 MPa, TS = 2150 MPa, and TE = 6.3 % were obtained for 0.38C–1.22Mn–0.23Si–0.10Cr–0.04Ti steel. On the other hand, the authors observed that due to essential increase in strain hardening, a low-carbon 0.15C–5Mn steel could achieve TS > 1700 MPa (Arlazarov et al. 2013).

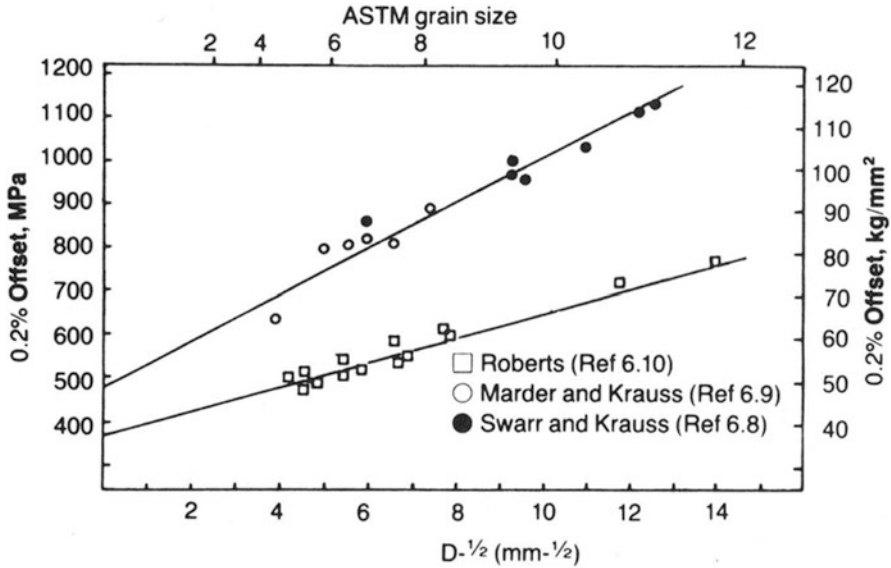
Figure 7.4 shows that increase in Si content in 0.15C–1.8Mn–0.02Nb–0.15Mo steel results in increase in tensile strength of as-quenched and especially tempered martensite due to retardation of softening martensite at tempering (Johnson et al. 2013).

It is also known that grain refinement is the major remedy in improving the mechanical properties of martensitic steel. The size of martensite packets is directly related to the austenite grain size (Krauss 2005), so the data presented in Fig. 7.5 related to packet size, D, emphasize the essential impact of austenite grain refinement.

## 7.2.2 As-Rolled Martensite

Several producers supply hot-rolled sheet martensitic grades.

Processing of martensitic grades in hot-rolling mills requires fast cooling from the finishing rolling temperature immediately upon the exit from the mill down to temperatures below  $M_S$  to prevent or at least to minimize non-martensitic products of austenite transformation. The steels should be alloyed high enough to ensure



**Fig. 7.5** Correlation between strength of lath martensite and packet size,  $D$ . The *upper curve* is for 0.2C martensite and the *lower curve* is for Fe–Mn carbon-free martensite (Swarr and Krauss 1976; Marder and Krauss 1970; Roberts 1970; Krauss 2005)

sufficiently high hardenability to prevent ferrite/pearlite transformation under cooling with real available rate (20–40 °C/s).

In particular, Salzgitter produces SZMS 1200 grade with TS above 1200 MPa, min YS = 900 MPa in thickness from 2 to 3 mm. For available cooling rate of relatively thick strips, the 0.18C–2.0Mn–0.6Cr–0.15Si composition is used to assure the necessary hardenability of steel (Saltzgitter Flachstahl GmbH catalog).

In general, chemical composition of as-hot-rolled martensitic grades depends on both cooling capability of hot-rolling mills and thickness of produced sheets. For example, ThyssenKrupp Steel produces as-hot-rolled martensite MS-W 1200 grade with TS > 1200 MPa and YS > 900 MPa using slightly higher alloying: 0.18 C with up to 2.0 Mn, 1.0 Cr, and 0.8 Si and various combinations of microalloying with Ti, B, and Nb.

### 7.2.3 Effect of Martensite Tempering

Tempering is a common practice to improve ductility or toughness of as-quenched martensite. During tempering or auto-tempering, a number of metallurgical effects take place: decrease of lattice distortion, release of residual stresses, and carbide precipitation. To some extent, these effects take place at tempering temperatures in the range of 150–200 °C but without any significant impact on tensile properties.

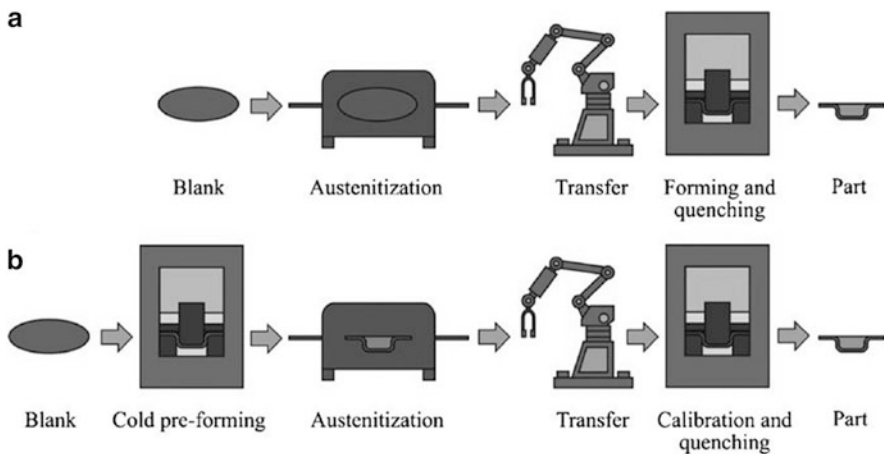
Since the tempering above 200 °C leads to a gradual decrease in strength, the martensitic grades are not usually tempered at temperatures higher than 180–200 °C.

### 7.3 Martensite Produced by Press Hardening

Press-hardening technology solves the long existing conflict of forming extremely high strength steels into complex shape without problems with cracking, excessive press forces, or spring back. Hot stamping as a process was developed in 1977, and with implementation of boron-containing (Al–Si coated) steels in 2000, more hot-stamped parts have been used in cars, and the number of annually produced parts has gone up to several hundred million parts with significant increase every year.

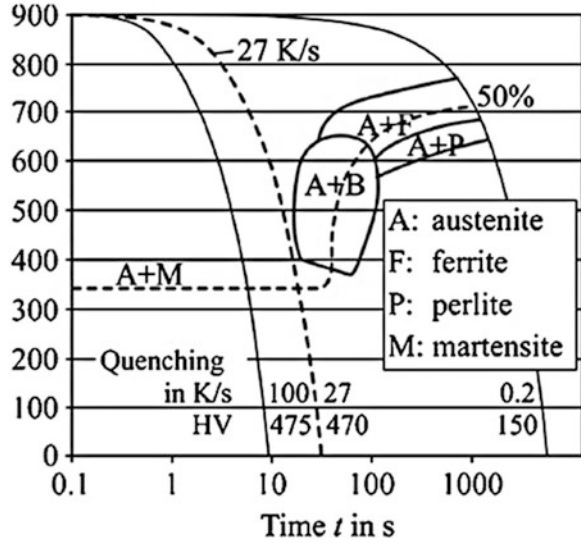
#### 7.3.1 Basic Principles of Obtaining Martensite After Hot Stamping

The hot-stamping process currently exists in two main variants: direct and indirect hot stamping. In the direct hot-stamping process, a blank is heated up in the furnace, transferred to the press, and subsequently formed and quenched in the water-cooled closed tool as depicted in Fig. 7.6a. In the indirect hot-stamping process, a near net shape cold preformed part is subjected only to austenitization followed by



**Fig. 7.6** Basic hot-stamping processes: (a) direct hot stamping. (b) Indirect hot stamping (Karbasiyan and Tekkaya 2010)

**Fig. 7.7** Schematic CCT diagram of steel for hot stamping



calibration operation and quenching in the press (Fig. 7.6b) (Karbasian and Tekkaya 2010).

22MnB5 steel is the most commonly used grade for hot stamping.

Supplied material (typically cold rolled and annealed or coated) has a ferrite–pearlite microstructure with a tensile strength of about 600 MPa. After the hot-stamping process, with complete martensite transformation taking place due to quenching in the water-cooled die, the component must have martensitic microstructure with strength of about 1500 MPa. To achieve such a microstructure, the blank is austenitized in the furnace at temperature above  $A_{c3}$ .

As shown in the CCT diagram in Fig. 7.7, the martensite start temperature of 22MnB5 steel is close to 375–400 °C (the martensite finish temperature is about 280 °C). The critical cooling rate to avoid the formation of softer phases (bainite, ferrite, and pearlite) is about 25 °C/s. Thus, the cooling rate in the die should exceed this critical rate to ensure full martensite transformation and the required final properties.

To make press-hardening technology a reliable process, numerous careful studies were performed, for example, by Naderi et al., focusing on calculations of the effects of variability of blank thickness and cooling conditions (cooling media) on critical cooling rate for martensite transformation (Naderi et al. 2008).

### 7.3.2 Development of Ultrahigh Strength Martensite for Press Hardening

As mentioned above, commercial martensitic grades for press hardening with TS ~1500 MPa are currently available. However, higher strength steels with TS > 1800



and 2000 MPa are required to lighten the parts to desirable levels and/or to increase crush resistance.

To increase the strength of martensitic steels for press hardening, various strategies are considered. They aim not only at increasing strength but also at ensuring robust manufacturability in steel mills and during press-hardening processing by the customers, as well as at better toughness and improved resistance to delayed fracture.

To reach  $TS \geq 2000$  MPa, which is significantly higher than for 22MnB5 grade, the carbon should be increased. To keep the necessary level of weldability, it is necessary to compensate higher carbon content by decrease in Mn content which, in particular, typically negatively affects hydrogen resistance. The presence of Si is considered as useful to improve the resistance to hydrogen embrittlement (Matsumoto et al. 2013). The beneficial effect of microstructural refinement on the strength and toughness of martensitic steels has been verified by many studies (Kubota et al. 2010). Nb should be considered as useful in this concept because grain refinement should be favorable for the resistance to delayed fracture.

Bain and Mohbacher are building their strategy on optimization of conventional alloy design based on 22MnB5 to reach  $TS > 1800$  MPa. They slightly increase carbon content to 0.25 % keeping the same 1.4 % Mn and 0.4 % Si, adding over 0.05 % Nb and 0.15 % Mo, and removing B + Ti.

Essential feature of the proposed improved press-hardenable steel is its fine grain microstructure. Additional useful role of microalloying by Nb can be related to prevention of grain growth during high temperature blank reheating, usually 950 °C, to compensate for temperature loss during transfer of the blank from the furnace to the die.

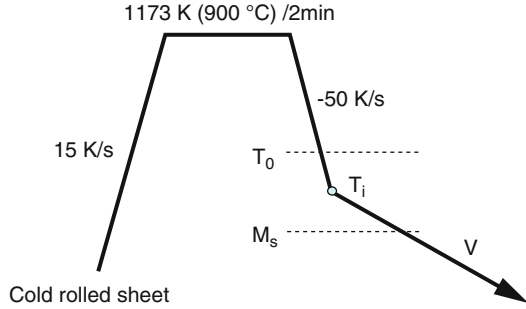
Since in press hardening the steel gauges are quite thin (usually <3 mm), in authors' opinion, cooling rates during die quenching are rather high, especially when the advanced press-hardening technology with increased die pressure is employed. This should allow for avoiding the formation of ferrite even at the absence of boron. Therefore, additions of Ti can be eliminated too. The bainite nose can be sufficiently delayed by adding more Mn, Mo, or Cr if lower cooling rates need to be tolerated.

The important component of the proposed strategy is the strengthening of austenite using additions of Mo. While boron blocks the nucleation of ferrite by segregating to austenite grain boundaries, Mo lowers the activity of carbon and slows its diffusion and consequently significantly delays ferrite, pearlite, and bainite transformations (Bian and Mohrbacher 2013).

### ***7.3.3 Modification of Press-Hardening Technology***

Several studies proposed to modify hot-stamping technology so as to reach the final mechanical properties of stamped parts close to the properties of the third

**Fig. 7.8** Thermal cycle proposed for press-forming process to obtain carbide-free microstructure with retained austenite (Chen et al. 2014)



Generation steels, i.e., to combine ultrahigh strength of martensitic press-hardened parts with high ductility and toughness.

In particular, it was shown that medium Mn (4–5 % Mn) steels are not sensitive to cooling rate and can be fully transformed to martensite at air-cooling. This allows for hot stamping at lower temperatures because of substantially lower  $A_{c3}$  temperatures, and dies should be cooled only for productivity purposes.

Another quite novel modification to hot stamping is the attempt to combine it with the Q&P processing. Full austenitization and hot deformation to simulate hot stamping were performed with subsequent quenching to the temperature below  $M_s$  followed by isothermal holding at the same temperature. The resultant microstructure was refined and contained significant amounts of retained austenite that allowed for appreciable increase in elongation.

For example, Liu et al. proposed special 0.22C–1.58Mn–0.81Si–0.022Ti–0.0024B composition for such processing. After full austenitization for 5 min and hot deformation, the steel was quenched to 280, 300, and 320 °C, which are the temperature in the ( $M_s$ – $M_F$ ) range. This way, one-step Q&P process was realized. The best combinations of properties achieved with partitioning at 320 and 280 °C for 30 s were TS = 1510 MPa at TE = 14.8 % and TS = 1601 MPa at TE = 10.3 %, respectively (Liu et al. 2011).

Chen et al. suggested the so-called interrupted cooling that combined rapid cooling to temperature slightly above the  $M_s$  temperature (305–360 °C) with subsequent slow cooling to room temperature (Fig. 7.8). Using ~0.30C–1.6Si–2.3Mn–0.25Mo steel with up to 0.5Cr, the authors tried to obtain microstructure of TRIP type with bainitic ferrite matrix. By adjusting the composition and cooling interruption temperature, the authors managed to obtain TS > 1400 MPa at TE > 10 % (Chen et al. 2014)

One of the main concerns in hot stamping aimed at fully martensitic microstructure of the deformed part is to avoid austenite decomposition during cold plastic deformation. Therefore, steels considered for this application should have sufficiently stable austenite that could be deformed with minimum of deleterious decomposition products.

## 7.4 Susceptibility of Martensitic Grades to Delayed Fracture

The share of ultrahigh strength steel including martensitic grades is growing. However, there are some justified concerns that the susceptibility to delayed fracture (DF) controlled by hydrogen embrittlement (HE) increases with higher strength of steel. Generally, it is believed that the problem emerges for high-strength steel with a tensile strength of above 1000 MPa.

Since the strength of steel depends on both alloy composition and microstructure, it is difficult to independently distinguish the effects of strength itself from the effects of alloying and microstructure. In addition, different researchers have been using different test methods to evaluate the resistance to HE/DF, as well as high hydrogen content charged, often significantly higher than steel parts can experience after processing or in service.

In fact, strength has never showed a direct correlation with material behavior under hydrogen impact. Steels with the same strength level demonstrate different resistance to hydrogen embrittlement due to different combinations of microstructure and chemical composition that are believed to play the key role (Thiessen et al. 2011).

In particular, after three decades of producing lean chemistry martensite with strength of up to 1500 MPa, Inland Steel/Arcelor Mittal has received zero claims related to delayed fracture. The developed M1700 grade based on the same lean 0.45 % Mn matrix with only higher carbon content also passed very severe test of 600 h immersing the bent sample stressed to 85 % of TS into an acidic environment (0.1 N HCl) without crack appearance.

On the other hand, the study of martensite grades M1200 and M1400 containing 1.5 % Mn, 0.2–0.3 % Si, and 0.2–0.3 % Cr found them susceptible to hydrogen embrittlement, but it started from 4 and 1 weight ppm, respectively, that is in any way much higher than what could be expected (Lovicu et al. 2012).

The development of martensitic products with TS ~2000 MPa requires careful consideration of separate roles of chemical composition and microstructure on the resistance to DF/HE.

In accordance with data related mostly to HSLA, increased content of Mn in steels has a negative influence of susceptibility to hydrogen embrittlement (Hejazi et al. 2012)

Since HE involves intergranular fracture, boron should be considered useful in UHSS not only from the point of view of hardenability but probably for grain boundary strengthening. As shown by Nie et al. (2007), the increase in boron content from 0.0005 to 0.0016 % in medium carbon spring steel was accompanied by increase in delayed fracture strength.

According to Shiraga (1994), the positive effect of Ni is related to its increased concentration on the exterior surface that suppresses the permeation of diffusible hydrogen from aggressive solutions.

Effect of Cu additions was carefully studied by Toyoda et al. (Toyoda et al. 2008; Toyoda 2011). According to their data, steel with additions of Cu in

the amount of 0.1–0.15 % demonstrated the same excellent DF resistance in both 1 N hydrochloric acid and saltwater as steel containing 0.5 % Cu.

It is known that additions of Al (1–1.5 %) solved the problem of DF of fully austenitic TWIP steels, although the mechanism of its effect is not fully understood.

The data related to effects of microalloying are rather inconsistent.

Effects of Nb and V additions are considered in the publication by Zhang et al. (2011). These authors showed that microalloying with Nb or Nb + V reduces the apparent diffusion coefficient of hydrogen in 0.6C–1.6Si–0.75Mn–0.35Cr (wt%) steel that results not only in smaller percentage of strength loss during testing of pre-charged specimens but also in some changes in DF character in favor of some portion of trans-granular and quasi-cleavage rupture instead of intergranular.

Meanwhile, the literature review shows that titanium or vanadium carbides can enhance or suppress DF depending on other conditions and primarily on microstructure. For example, some publications demonstrate that VC increases the critical hydrogen content (threshold for DF), whereas other data show that it also increases the amount of absorbed hydrogen.

For example, Gladshstein et al. (1988) noted that additions of Ti (0.029–0.078 %), Nb (0.025–0.049 %), and boron (0.015–0.0044 %) to tempered 0.2C–2Cr–Ni–Mo steel with microstructure of lath martensite and bainite enhanced the resistance to crack initiation and propagation under hydrogenation. In the author's opinion, the positive role of Ti and Nb is the trapping capacity of their coarse particles, which decrease hydrogen saturation near the crack tip, whereas boron retards initiation and propagation of intergranular cracks.

In some cases, all microalloying elements (Ti, Nb, and V) are added as useful remedies against delayed fracture without distinguishing the role of Nb, as was done in case of steels for fasteners with TS above 1800 MPa (Kubota et al. 2010).

Significant improvement in DF resistance of low tempered (250 °C) martensitic 0.22C–0.5Mn–0.2Si–0.5Mo–1.8Cr (wt%) steel microalloyed with Nb, Ti, and B was noted by Glazkova et al. (1976). The authors suggested that microalloying elements play the dominating role due to both grain refinement and enhancement of trapping capacity by carbonitrides of Ti and Nb that significantly decreased the diffusivity of hydrogen.

Higher content of Si in steel can improve delayed fracture resistance because, on the one hand, Si inhibits/reduces carbide formation during tempering of martensite and, on the other hand, it reduces hydrogen diffusivity (Matsumoto et al. 2013).

Careful comparative study of the effects of 0.05 % V, 0.04 % Ti and, 0.03 % Nb on DF was conducted by Sergeeva et al. The authors evaluated the time before fracture of in situ hydrogen charged samples under stress, the content of occluded hydrogen after 1 h charging, and the desorption rate as an indicator of hydrogen trapping efficiency. It was found that 0.03 % Nb had the maximum effect on increasing time before fracture by two orders of magnitude of 0.31C–0.5Mn–0.25Si–0.15Mo–0.8Cr–1.5Ni (wt%) steel. This was explained by both increased toughness of steel and lower hydrogen consumption. Essential increase in time before fracture demonstrated by Ti additions was considered to be due to favorable compensating trapping role of Ti(C, N), as the total occlusion of H grew in the presence of Ti (Sergeeva et al. 1994).

Using Auger spectroscopy and investigating segregation at free surface that can simulate grain boundary segregation, the same authors established that positive effect of microalloying on delayed fracture was related also to the suppression of grain boundary segregations of P, Sb, C, and S. In terms of increasing the efficiency of this suppression and growing strength, the microalloying elements were ranked as V, Ti, and Nb (Drobyshevskaya et al. 1995).

Since the intergranular fracture between prior austenite grains is the dominant mechanism of delayed fracture, refining of prior austenite grains is important in improving grain boundary strength that was demonstrated, in particular, by Fuchigami et al. (2006).

Appropriateness of the above statements regarding the effects of alloying and microalloying was verified by lab research performed by Song et al. (2015), when Si and Nb were sequentially added to low-Mn-B-Ti steel with TS ~ 2000 MPa. The results of delayed fracture tests as well as sizes of prior austenite grains, defined using EBSD analysis, are presented in Table 7.4.

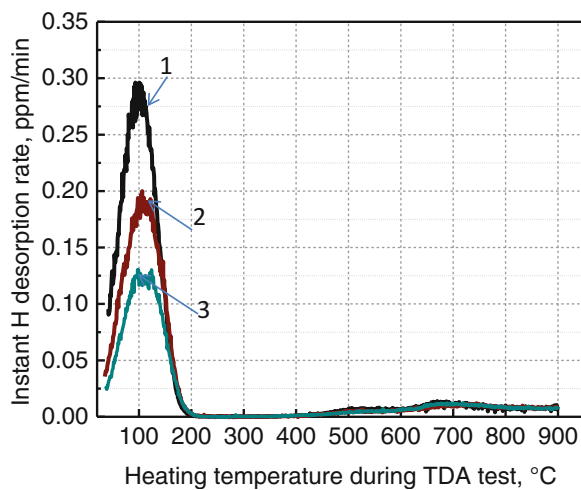
As shown in Fig. 7.9, the growing efficiency of alloying and microalloying additions was confirmed by measurements of diffusible hydrogen that decreased in the same order as the increase in time before delayed fracture.

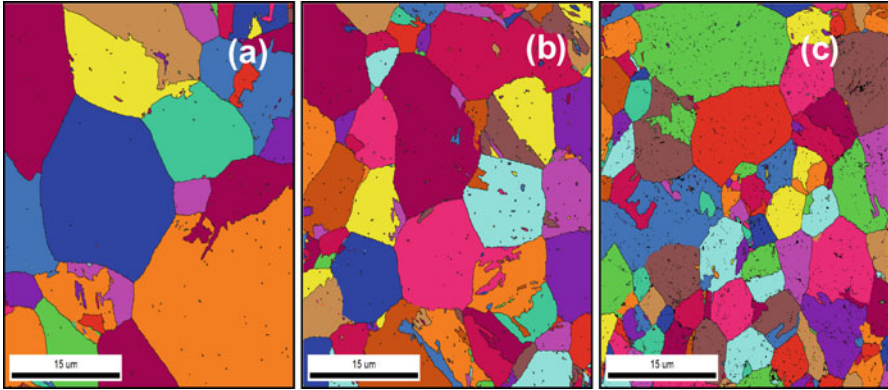
**Table 7.4** Basic mechanical properties and results of delayed fracture tests

Steel—ASTM-L	Time before cracking in hours during U-bend test. 0.1 N HCl, 85 % TS	Diffusible H (<300 °C) (ppm)	PAGS (μm)
Ti-B	<6 h	1.22	18.3
Ti-B-Si	137 h	0.89	10.3
Ti-B-Si-Nb	>600 h	0.60	6.4

**Fig. 7.9** Effect of alloying/microalloying elements on hydrogen desorption:

1-base Ti-B composition, 2-base plus Si, and 3-base plus Si plus Nb (original)





**Fig. 7.10** EBSD analysis of PAGS: (a) base Ti–B composition, (b) base plus Si, (c) base plus Si plus Nb (original)

As was established by EBSD analysis, the added elements, besides their influence on trapping, also produce gradual refinement of prior austenite grain size (PAGS) (Fig. 7.10) with the smallest grain size detected for Nb microalloyed steel.

## 7.5 Summary

The use of martensitic steel with tensile strength of up to 2000 MPa and high YS/TS ratio has become extremely relevant to car body engineering, especially with respect to avoiding the intrusion by high speed impact loading during a crash.

Various types of manufacturing of martensitic steels by water quenching after annealing, by hot rolling, and by press hardening are available facilitating application of ultrahigh strength steels for safety critical car parts.

It was demonstrated that microstructure refinement being mainly related to austenite grain size prior to quenching is the key approach to improve combination of strength and resistance to delayed fracture.

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