THE INFLUENCE OF NIOBIUM ON THE RESULTING STRUCTURE OF TRIP STEELS DURING THE CONTINUOUS COOLING

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Abstract

High strength multiphase TRIP steels are very often used as structural materials, especially in the automotive industry. Due to their ability to absorb energy during the impact, they are used for safety components of the car body. These parts can be produced by a hot stamping process, which has the advantage of using lower forming forces and a smaller spring back effect. The problem during heat treatment of TRIP steels is the holding time in the region of bainitic transformation, which facilitates bainite formation and stabilization of retained austenite.

In the present study, two low-alloy TRIP steels with and without niobium were chosen. Physical simulation was employed to test an alternative route without an isothermal hold during cooling after hot stamping. Several continuous cooling profiles from the tool temperature were applied. The results were compared with isothermal processing in the region of bainitic transformation. Mixed structures consisting of bainite, martensite, ferrite and retained austenite were obtained for both steels. The ultimate strength reached more than 900 MPa with the elongation A_{20mm} over 15 %.

Keywords: TRIP steel, niobium, continuous cooling, retained austenite

1. INTRODUCTION

High-strength steels are widely used in the automotive industry because of their properties, affordability and their contribution to reduction in body-in-white weight, which results in fuel savings [1]. Their mechanical properties result from their composition and from appropriate heat treatment and thermomechanical processing [2]. This class of steels also includes multiphase TRIP steels (the acronym stands for transformation-induced plasticity). The microstructure of these steels consists of ferrite, carbide-free bainite and retained austenite (RA) [3-5]. Upon cold deformation, retained austenite transforms into high-carbon martensite which substantially contributes to work hardening [5]. Work hardening is further enhanced by dislocations and internal stresses which occur within adjacent phases [3].

Steels of this type typically contain 0.2 - 0.25 % carbon, manganese and silicon, and perhaps aluminium. Manganese and silicon play important roles in controlling phase transformations, stabilizing retained austenite and solid solution strengthening, which contributes to overall strength [4, 6]. Niobium is one of the most frequently-used microalloying elements. Whether present in precipitates or in solid solution, it has a powerful influence on microstructure and mechanical properties. Niobium impacts on austenitization, recrystallization, grain size evolution, phase transformations, the enrichment of austenite with carbon and nucleation of martensite, which improves mechanical properties [7-9]. In addition, niobium has effect on retained austenite morphology and promotes formation of lath and foil-like retained austenite [10].

Heat treatment of TRIP steels involves intercritical annealing i.e. isothermal holding in the bainitic transformation region [2, 4]. During this operation, bainite forms and retained austenite is stabilized thanks to diffusion of and enrichment with carbon. The hold poses difficulties from the processing technology viewpoint. Alternative routes are thus sought, such as continuous cooling at appropriate controlled rates [11]. Hot stamping is another alternative, being well-suited for making sheet parts of TRIP steels [12]. It is in fact used

in the automotive industry [13]. Its benefits include reduced forming force and reduced spring-back effect [13, 14]. High-strength steels are used for components which improve crash safety [14]. Hence, hot stamping of TRIP steels and effects of process parameters and alloying elements needs to be explored.

2. EXPERIMENTAL PROGRAMME

In these experiments, hot stamping followed by continuous cooling was tested on two low-alloy multiphase TRIP steels. Physical simulation was performed in a thermomechanical simulator in order to vary relevant process parameters. High-frequency resistive heating enables close control of the thermal profile. It offers heating rates of up to 200 °C / s. The cooling can be effected with water, water spray and air.

2.1. Experimental materials

Two low-alloy steels with 0.2 % carbon alloyed with manganese and silicon were chosen for these experiments (**Table 1**). One of them was micro-alloyed with niobium which, both in precipitates and solid solution, has strong effect microstructure and mechanical properties.

Both experimental materials were specially manufactured and cast as 50 kg ingots of 110 mm diameter. The top and bottom of each ingot was removed, the ingots were cleaned, cut into halves along their axis and homogenized in protective argon gas atmosphere at 1100 °C for six hours. The ingot halves were then rolled at 1150 °C from their initial height of 55 mm to 5.5 mm using single-pass reductions of 10 %. The final thickness of approx. 1.8 mm was obtained by cold rolling. After final annealing at 900 °C, specimens for physical simulation were made from the sheet. They were then ground to a thickness of 1.6 mm.

Table 1 Chemical compositions of experimental steels (wt. %)

Steel	C	Si	Mn	P	S	Cr	Nb	M_s (°C)	M_f (°C)	HV10 [-]	$R_{p0.2}$ (MPa)	R_m (MPa)	A_{20mm} (%)
CMnSi	0.21	1.797	1.449	0.008	0.005	0.008	0.059	360	246	190	422	635	27
CMnSiNb	0.21	1.797	1.449	0.008	0.005	0.008	0.03	370	257	180	427	627	26

In this initial condition, both materials exhibited ferritic-pearlitic microstructure and hardness levels of 190 HV10 and 180 HV10 (**Table 1**). Tensile strengths of the CMnSi and CMnSiNb steels were found to be 635 MPa and 627 MPa, respectively. Their elongation (A_{20mm}) values were near identical, 26 % and 27 %. Their M_s and M_f temperatures [15] were calculated using the JMatPro program. The M_s of the CMnSi steel was 360 °C. For CMnSiNb the M_s was higher: 370 °C (**Table 1**).

2.2. Hot stamping and continuous cooling

The input data for physical simulation was gathered by measurement in a real-world hot stamping process. By this means, thermal profiles for hot stamping process with tools at various temperatures were obtained. Using these data, physical simulation sequences were designed and tested on sheet specimens in a thermomechanical simulator.

The first step of the physical simulation sequence involved soaking at 937 °C for 100 seconds. The transfer of an actual blank from a furnace to the tool was modelled as a 10-second air-cooling step. It was followed by cooling at the rate at which the blank cools in the tool at a defined temperature. The sets of these temperatures were identical for both steels: room temperature, 350 °C, 400 °C, 425 °C and 500 °C (**Table 2, Table 3**). Rapid temperature equalization was followed by final cooling at the cooling rate of the blank in still air. The optimal isothermal holding temperature for both steels is 425 °C. For this reason and for the sake of comparison, an additional sequence was carried out which involved a 600-second hold at this temperature. Another sequence

was added for the CMnSi steel, for which the tool temperature was 300 °C. For the CMnSiNb steel, one additional sequence was carried out with a tool temperature of 450 °C.

Table 2 Heat treatment sequences and mechanical properties of CMnSi TRIP steel

Treatment	Tool temp. (°C)	Holding time (s)	Cooling rate in the tool (°C / s)	HV10 (-)	$R_{p0.2}$ (MPa)	R_m (MPa)	A_{20mm} (%)	RA (%)
TRIP-01	RT	-	100	471	900	1340	7.2	3
TRIP-02	300	-	92	407	875	1130	5.6	7
TRIP-03	350	-	90	412	790	1140	7.8	-
TRIP-04	400	-	84	393	709	1020	10.5	-
TRIP-05	425	-	82	323	568	977	15.3	10
TRIP-06	425	600	82	316	603	850	12.7	10
TRIP-07	500	-	70	291	525	920	18	12

Table 3 Heat treatment sequences and mechanical properties of CMnSiNb TRIP steel

Treatment	Tool temp. (°C)	Holding time (s)	Cooling rate in the tool (°C / s)	HV10 (-)	$R_{p0.2}$ (MPa)	R_m (MPa)	A_{20mm} (%)	RA (%)
TRIPNb-01	RT	-	100	451	731	1210	5.7	3
TRIPNb-02	350	-	90	393	557	1025	8	6
TRIPNb-03	400	-	84	368	614	1050	11.4	9
TRIPNb-04	425	-	82	336	572	976	12.3	10
TRIPNb-05	425	600	82	302	478	818	14.8	12
TRIPNb-06	450	-	80	313	501	930	19.5	12
TRIPNb-07	500	-	70	301	478	933	17.1	10

The instruments used for metallographic observation included a light microscope (LM) and scanning electron microscopes (SEM) Tescan VEGA 3 SEM and Zeiss EVO MA 25. Retained austenite distribution was examined after two-stage etching (stage 1: Nital, stage 2: 10 % aqueous solution of $Na_2S_2O_5$). The amount of retained austenite was measured by XRD phase analysis in the automatic powder diffractometer AXS Bruker D8 Discover with a position-sensitive area HI-STAR detector and a cobalt X-ray source ($\lambda K\alpha$ = 0.1790307 nm). Measurements were taken at the centres of metallographic sections using the diffraction angle interval of 25 ÷ 110°. Mechanical properties were determined through HV10 hardness testing and tensile testing.

3. RESULTS AND DISCUSION

3.1. CMnSi steel

The sequence with the average cooling rate of 100 °C / s and the tool at room temperature produced a martensitic microstructure with a small amount of free ferrite and a hardness of 471 HV10 (**Figure 1a**). The ultimate strength reached 1340 MPa and the A_{20mm} elongation was 7 % (**Table 2**). Upon higher tool temperatures, i.e. 300 °C and 350 °C, small amounts of proeutectoid ferrite formed as well. That led to slightly lower ultimate strengths, 1130 MPa and 1140 MPa, but no significant changes in elongation. After the sequence with a tool at 400 °C, bainite was found in the final microstructure, in addition to a higher amount of ferrite. This resulted in higher elongation, 10 %, and strength of 1020 MPa. A tool temperature of 425 °C led to a microstructure in which bainite sheaves dominated over free ferrite and small fractions of martensite and retained austenite (**Figure 1b**). Two-stage etching revealed that retained austenite was present in both

morphologies: globular grains under 1 μm and foils between bainite needles (**Figure 1c**). The fraction of retained austenite was 10 %. Elongation rose to 15 %, whereas strength decreased to 977 MPa. Holding at 425 °C removed almost all martensite and produced a larger amount of ferrite but failed to increase elongation (**Figure 1d**). After this sequence, the strength was a mere 850 MPa.

The sequence with an even higher tool temperature of 500 °C resulted in as yet highest elongation of 18 % and strength of 920 MPa. The cooling rate during hot stamping in a tool at this temperature was lower than in previous cases: 70 °C / s. The final mixed microstructure contained a majority of bainite, free ferrite and small martensite islands. Those had formed in regions of insufficiently-stabilized retained austenite during cooling to room temperature (**Figure 1e**). The presence of RA was confirmed by both X-ray diffraction analysis (12 %) and two-stage etching. RA was found mostly between bainite needles (**Figure 1f**). The M-A constituent was found in some of these intermediate spaces.

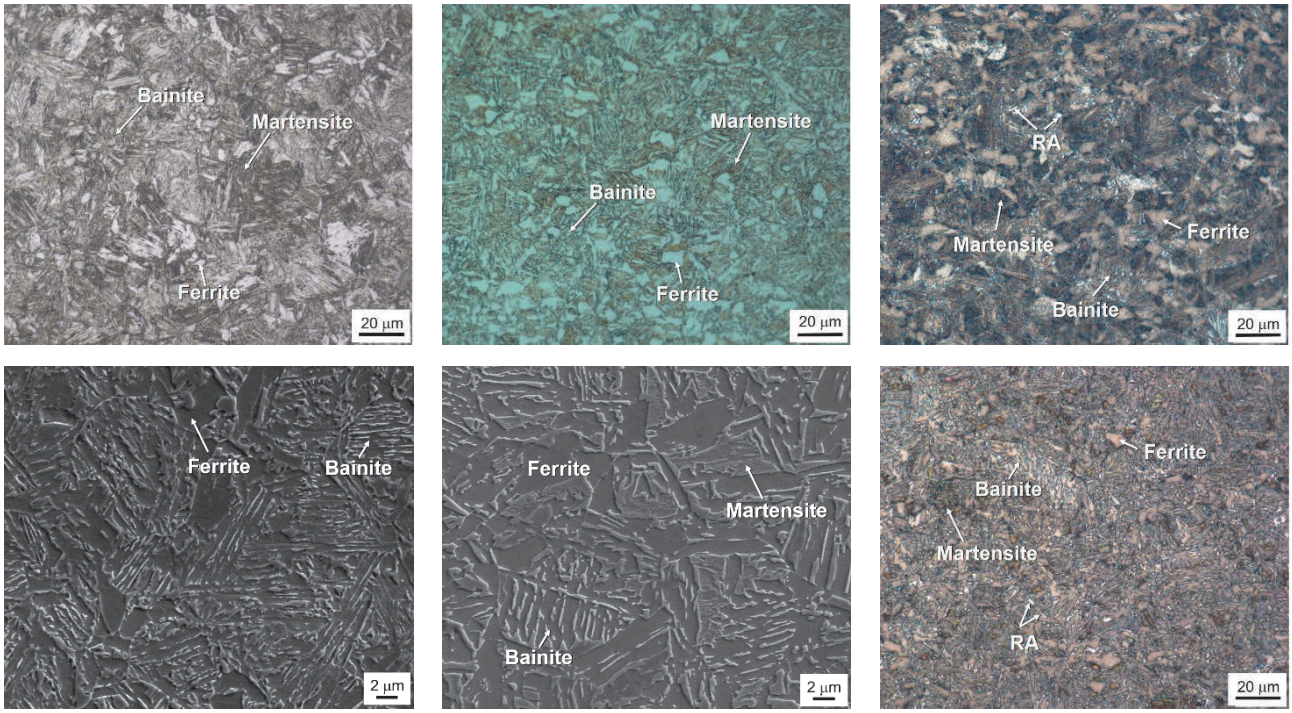

Figure 1 Micrographs of CMnSi steel after hot stamping in tools at: a) room temperature - light micrograph (LM), b) 425 °C - LM, c) 425 °C - two-stage etch for highlighting retained austenite, d) 425 °C / 600 s - scanning electron micrograph, e) 500 °C - scanning electron micrograph, f) 500 °C - two-stage etch - LM

3.2. CMnSiNb steel

Hot stamping of the niobium-microalloyed steel in a tool at room temperature led to a martensitic-ferritic microstructure and a hardness of 451 HV10 (**Figure 2a**). The ultimate strength was high, 1210 MPa, and elongation was 6 %. The sequences with tool temperatures of 350 °C and 400 °C produced larger amounts of bainite and less martensite (**Figure 2b**). The fraction of retained austenite increased from 6 to 9 %. As a result, ultimate strengths decreased to 1025 MPa and 1050 MPa, respectively, whereas elongation rose slightly to 11 %. A tool temperature of 425 °C led to a majority of bainite, free ferrite and small amounts of martensite and 10 % of retained austenite (**Figure 2c**). The ultimate strength and elongation were 976 MPa and 12 %, respectively. The sequence which involved holding at 425 °C for 600 seconds, to promote bainite formation and stabilize retained austenite produced a microstructure of ferrite, bainite and retained austenite (**Figure 2d**). The fraction of retained austenite increased up to 12 %. Using colour etching, retained austenite was detected in both globular and needle-like forms (**Figure 2e**). Some globular retained austenite particles had partially transformed to martensite, forming the M-A constituent. Although the A_{20mm} elongation was higher

(15 %) than in preceding sequences, the ultimate strength decreased to 818 MPa, owing to a large ferrite fraction.

After the sequence with a higher tool temperature of 450 °C, the resultant microstructure was found to contain not only bainite but also martensite. Martensite formed in the regions of insufficiently stabilized retained austenite. The sequence in which the tool temperature was 500 °C produced a higher martensite fraction, resulting in a mixture of ferrite, martensite and bainite (**Figure 2f**). The fraction of retained austenite was 10 % and RA had, again, both morphologies. Although cooling was slower than in preceding sequences, i.e. 70 °C / s instead of 100 °C / s, and the tool temperature was high, no pearlite formed. The ultimate strength was 933 MPa and elongation reached 17 %.

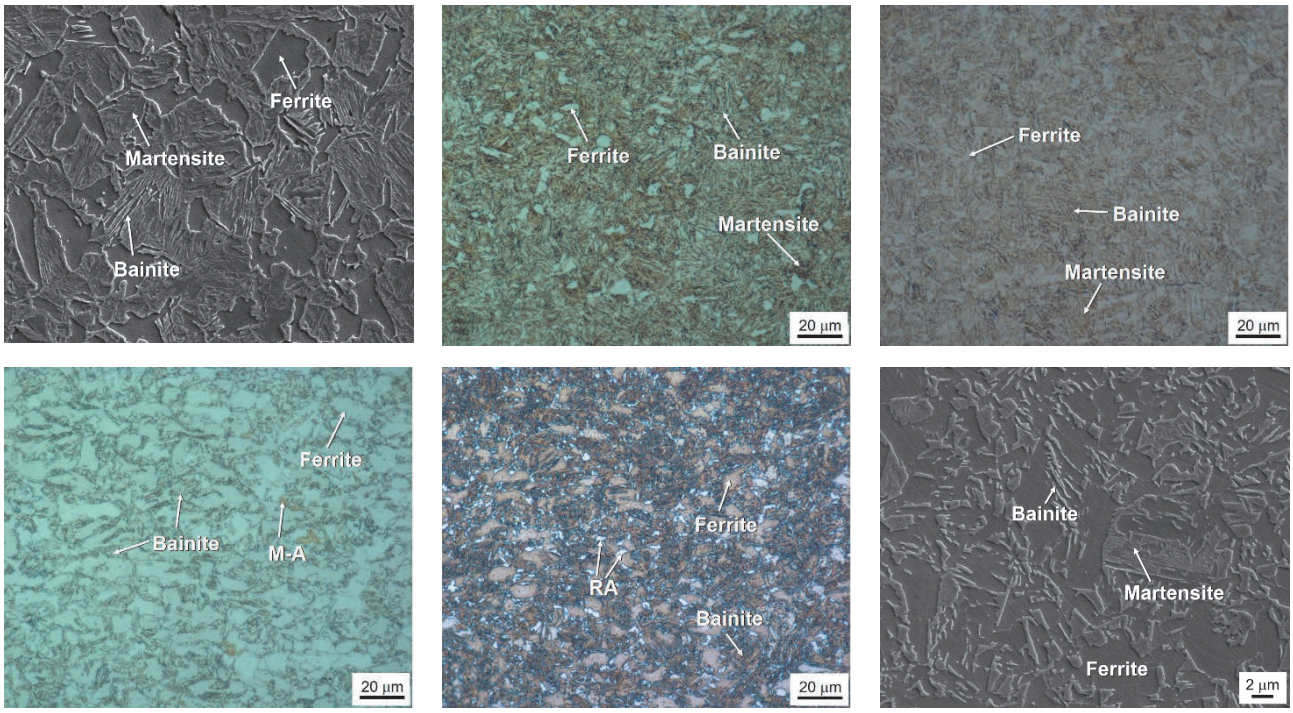

Figure 2 Micrographs of CMnSiNb steel after hot stamping in tools at the following temperatures: a) room temperature - light micrograph (LM), b) 400 °C - LM, c) 425 °C - LM, d) 425 °C / 600 s - LM, e) 425 °C / 600 s - two-stage-etch - LM, f) 500 °C - scanning electron micrograph

4. CONCLUSION

Hot stamping followed by cooling without an isothermal hold was physically simulated on two low-alloy steels, CMnSi and CMnSiNb. The input data for physical simulation were collected in a real-world process. In order to test several cooling rates, the physical simulation sequences involved various tool temperatures, from room temperature to 500 °C.

Higher tool temperatures led to more intensive bainite formation in the 0.2 %-carbon CMnSi steel. Sequences with tool temperatures of 425 °C and 500 °C produced mixed microstructures of bainite, martensite, free ferrite and retained austenite and elongations in excess of 10 %. In spite of continuous cooling, a sufficient amount of RA was stabilized and elongation and strength were up to 18 % and above 920 MPa, respectively.

In the niobium-microalloyed CMnSiNb steel, no effect of niobium on solid solution strengthening was detected. The material has not reached higher mechanical properties than CMnSi. The possible reason is the soaking temperature which was lower than the temperature necessary for dissolving niobium carbides. In this case, too, elongation was almost 20 % and ultimate strength reached 930 MPa.

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REFERENCES

[1] ASGARI, S. A., PEREIRA, M., ROLFE, B. F., DINGLE, M., HODGSON, P. D. Statistical analysis of finite element modelling in sheet metal forming and springback analysis. *Journal of Materials Processing Technology,* 2008, vol. 203, pp. 129-136.

[2] ZHAO, J. and JIANG, Z. Thermomechanical processing of advanced high strength steels. *Progress in Materials Science,* 2018, vol. 94, pp. 174-242.

[3] CHIANG, J., BOYD, J. D. and PILKEY, A. K. Effect of microstructure on retained austenite stability and tensile behaviour in an aluminum-alloyed TRIP steel. *Materials Science and Engineering,* 2015, vol. 638A, pp. 132-142.

[4] BLECK, W. Using the TRIP effect - the dawn of a promising group of cold formable steels. In *International Conference on TRIP-Aided High Strength Ferrous Alloys.* Mainz: GRIPS´sparkling world of steel, 2002, pp. 13-23.

[5] BLECK, W., GUO, X. and MA, Y. The TRIP Effect and Its Application in Cold Formable Sheet Steels. *Steel Research International,* 2017, vol. 88, no. 10, pp. 1-21.

[6] PERELOMA, E. V., TIMOKHINA, I. B. and HODGSON, P. D. Transformation behaviour in thermomechanical processed C-Mn-Si steel with and without Nb. *Materials Science and Engineering,* 1999, vol. 273 - 275A, pp. 448 - 452.

[7] BAIK, S. CH., KIM, S., JIN, S. and KWON, O. Effects of alloying elements on mechanical properties and phase transformation of cold rolled TRIP steel sheets. *ISIJ International,* 2001, vol. 41, no. 3, pp. 290-297.

[8] OHLERT, J., BLECK, W. and HULKA, K. Control of microstructure in TRIP steels by niobium. In *International Conference on TRIP-Aided High Strength Ferrous Alloys.* Mainz: GRIPS´sparkling world of steel, 2002, 199-206.

[9] HONG, S. CH. et al. Effects of Nb on strain induced ferrite transformation in C-Mn steel. *Materials Science and Engineering,* 2003, vol. *355A*, pp. 241 - 248.

[10] PARK, S. H., CHOO, W. Y., KIM, N. J. and KO, J. H. Effects of hot rolling conditions on the microstructure and tensile properties of Nb-bearing TRIP steels. In *International Symposium on Hot Workability and Light Alloys Composites.* Montreal: TMS of CIM, 1996, pp. 493- 498.

[11] KUČEROVÁ, L., JIRKOVÁ, H. and MAŠEK, B. Influence of Nb micro-alloying on TRIP steels treated by continuous cooling process. *Manufacturing Technology,* 2016, vol. 16, no. 1, pp. 145-149.

[12] JIRKOVÁ H., OPATOVÁ K., WAGNER M.F.X. and MAŠEK B. Physical Simulation of Press Hardening of TRIP Steel. In *ICOMAT 2017: Proceedings of the International Conference on Martensitic Transformations*. Cham: Springer International Publishing, 2018, pp. 135-141.

[13] KARBASIAN, H. and TEKKAYA, A. E. A review on hot stamping. *Journal of Materials Processing Technology,* 2010, vol. 210, pp. 2103-2118.

[14] MORI, K. et al. Hot stamping of ultra-high strength steel parts. *CIRP Annals - Manufacturing Technology,* 2017, vol. 66, pp. 755-777.

[15] JMatPro, Release 9.0, Sente Software Ltd., 2016.

[16] JIRKOVA, H., KUČEROVÁ, L., PRUCHA, V. AND MASEK, B. Effect of advanced thermomechanical treatment on mechanical properties of low allowed high strength steels. In *Metal 2012: 21st International conference on metallurgy and materials.* Ostrava: TANGER, 2012, pp. 532-538.