Design of thermomechanical treatment of high strength steels

J Vrtáček1, M Peković2, H Jirková3, D Bubliková4 and L Kučerová5
UWB in Pilsen, Regional Technological Institute, Univerzitní 22, 301 00 Pilsen, Czech Republic

Abstract. Reducing the level of COx and NOx emissions from combustion engines is an important goal in the automotive industry. One way to reduce the emissions is to use lightweight parts in the car construction. High-strength steels are one of the successful choices for body parts. Their strengthening mechanisms provide them with better mechanical properties, which translates into thinner sheet metal blanks and ultimately reduced weight of parts. The main strengthening mechanisms include TRIP (TRansformation Induced Plasticity) and TWIP (TWinning induced plasticity). This paper outlines processing of high-strength steels, from forging to round bars and heat treatment to determination of phase transformations and temperature profiles for thermomechanical processing in an innovative tool. The experimental materials contained manganese, silicon and different aluminium levels. Their compositions were selected with a goal to observe the properties of advanced multiphase high strength steels during processing.

1 Introduction
Modern high-strength steels have an important role in enhancing passenger safety. The purpose of vehicle parts made from these materials is to absorb the largest possible portion of crash energy while minimizing plastic deformation in the deformation-absorbing parts of the vehicle body or frame. These advanced high-strength steels rely on a combination of several strengthening mechanisms, including the TRIP (TRansformation Induced Plasticity) and TWIP (TWinning induced plasticity) effects [1, 2]. These mechanisms require retained austenite to be present and rely on lattice transformation induced by plastic deformation [1, 10]. TRIP effect consists in the transformation of retained austenite (RA) to martensite, whereas the TWIP mechanism produces twins in austenite. Once the mechanical properties of the material are improved, the designers can reduce the thickness of sheet blanks. Along with thickness, the final weight of the body or frame of the vehicle as well as the deformation-absorbing parts can be reduced, which leads to less emissions. Recently, however, the sheets already have thicknesses which are close to the acceptable limit (0.8–1.2 mm) which impacts the part’s performance. Where further reduction is required, it is achieved by alloying the material with light metals, such as aluminium [3]. Aluminium reduces the density of the matrix, has favourable effects on grain size obtained by heat treatment and can substitute silicon to some extent [4]. The past decade has seen the development of high-strength multiphase steels of a new generation which combine the benefits found in the previous generations. They contain manganese and aluminium. It is the manganese-aluminium ratio plays a key role in obtaining the desired mechanical properties and the forming behaviour. This study therefore focuses on two advanced high-strength steels with different aluminium levels in an attempt to characterize the differences between their behaviour during forming and heat treatment.
2 Materials and methods

2.1 Experimental materials
Two steel grades were studied: C3Mn2Al and C3Mn1.4Al. The C3Mn1.4Al grade contained 0.22% C, 3.06% Mn, 0.59% Si, 1.45% Al, 0.008% P and 0.003 S. For the sake of comparison, the other material had a higher level of aluminium, which was 2%. Manganese [12] is added as an austenite-forming element which does not tend to form carbides, i.e. particles which are undesirable for the final thermomechanical treatment to make the finished product [6]. Manganese also improves elasticity of the material, as it dissolves in ferrite. Its atoms cause distortion of the lattice and prevent dislocation movement [5,11]. The role of aluminium is to act as a ferrite-former and to provide deoxidation [5,6].

In order to collect extensive data, the measurement of mechanical properties (tensile and hardness testing) was complemented with diffraction phase analysis by which the fraction of retained austenite was determined. The measurement was carried out using the AXS Bruker D8 Discover automatic powder diffractometer with a HI-STAR position-sensitive area detector and a cobalt X-ray source ($\lambda_{K\alpha} = 0.1790307$ nm). Measurements were taken at the centres of metallographic sections at diffraction angles in the interval of $25 \div 105^\circ$.

2.2 Determination of phase transformation temperatures
Prior to hot rolling were the top and bottom of the ingots removed. The ingots were placed into a pre-heated furnace at 1050 °C for 2 hours. Then they were transferred from the furnace to the rolling mill. The rolling sequence consisted of nine successive reductions with no reheating operation. The initial diameter was 97 mm and the final thickness of the strip was 13 mm. The temperature at the entry was about 1035 °C and temperature did not dropped below 950 °C during all reductions. With each reduction, the rolling force increased: from the initial 680 kN to 2000 kN in the last reduction. No excessive buildup of force, irregular thickness or waviness were experienced in the rolling process. Immediately after finish rolling to 13 mm, the rolled part was cut into three identical segments 800 mm in length and reheated in a furnace for 1 hour.

To design the heat treatment sequences for this experiment, one had to determine phase transformation temperatures for both materials. They were calculated from their chemical composition using JMatPro software [7]. In an alternative procedure, Andrews equations for calculating phase transformation temperatures were employed (1 through 3) [8]. There was a good agreement between the calculated values and the data from the dilatometer (table 1). Yet, there were differences in $A_{c3}$ temperature, up to 200°C. It was because the calculation did not account for all alloying elements in the material. The effect of aluminium as a ferrite-former was manifested in the $A_{c3}$ temperature which was higher in C3Mn2Al.

$$Ms(\degree C) = 539 - 423C - 30,4Mn - 17,7Ni - 12,1Cr - 7,5Mo$$ (1)

$$A_{c3}(\degree C) = 910 - \sqrt{203C} + 44,7Si - 15,2Ni + 31,5Mo + 104,4V + 13,1W$$ (2)

$$A_{c1}(\degree C) = 723 - 10,7Mn - 16,6Ni + 29,1Si + 16,9Cr + 290As + 6,38W$$ (3)
Table 1. Phase transformation temperatures obtained from equations, JMatPro software and dilatometric measurement.

|             | C3Mn1.4Al | C3Mn2Al |
|-------------|-----------|---------|
|             | JMatPro   | Andrews | Dilatometer | JMatPro | Andrews | Dilatometer |
| Ms [°C]     | 351       | 349     | 342         | 355     | 346     | 328         |
| Ac3 [°C]    | 815       | 841     | 961         | 981     | 841     | 1025        |
| Ac1 [°C]    | 709       | 691     | 728         | 714     | 691     | 739         |

2.3 Heat treatment design
After breaking down and forging the C3Mn1.4Al and C3Mn2Al ingots into the required feedstock, an appropriate austenitizing temperature had to be found for the heat treatment. Specimens of the steels were brought to different temperatures between 900 and 1200°C set at 50°C intervals. For each temperature, the holding time in the furnace was 30 minutes.

Austempering trials were the next step. The specimens were brought to 1000°C and held for 20 minutes in a furnace, after which they were quenched in a salt bath at 400°C. Once they reached 400°C, they were removed from the bath. The temperature was measured with a K-type thermocouple which was inserted in a hole in the centre of the specimen. After removal from the bath, the specimens were held in a furnace at 400, 425 or 450°C. Two different holding times were used: 15 and 60 minutes. For both steels, the annealing temperatures were in the bainitic transformation region and above the Ms. In the salt bath, the cooling rate was 35°C/s, as determined from the thermocouple data.

2 Results and discussion

3.1 Determination of suitable austenitizing temperature
The resulting microstructures were observed using light microscopy. In both materials, austenitizing at the lowest temperature, 900°C, led to a martensitic-bainitic microstructure with a small amount of ferrite (figure 1 a,b). C3Mn1.4Al had a hardness of 513 HV10. In C3Mn2Al, it was 476 HV10. Austenitizing at the next higher temperature, 950°C, led to different microstructures. Martensite was the prevalent phase (figure 1 c,d), which led to a higher hardness of 535 HV10 in C3Mn1.4Al and 505 HV10 in the C3Mn2Al grade. After austenitizing at still higher temperatures, the martensitic structure became coarser. Nevertheless, its hardness was not any higher (figure 2). The difference in hardness of the experimental materials is due to the different aluminium levels, since this element affects the transformation behaviour and mechanical properties.
Figure 1. Microstructures of the materials after austenitizing trials: 900°C (a,b), 950°C (c,d) and 1200°C (e,f).

Figure 2. Hardness of the experimental materials C3Mn1.4Al and C3Mn2Al after austenitizing trials.

3.2 Austempering

The purpose of the austempering trials was to determine the effect of the aluminium level, temperature and holding time at a temperature in the bainitic transformation region.

Austempering at 400°C produced a bainitic-martensitic microstructure in C3Mn1.4Al (figure 3) with an ultimate strength of 1220 MPa, elongation of 25% (table 2) and a hardness of 387 HV10 (figure 6). The retained austenite volume fraction was 15% (figure 7). The microstructure of C3Mn2Al was similar to the preceding one: bainite-martensite, with some bainitic ferrite and visible prior austenite grain boundaries. Its strength was 1390 MPa, elongation reached 17% (table 2), hardness was 424 HV10 (figure 6) and the volume fraction of retained austenite was 9% (figure 7). When the specimens were held for 60 minutes instead of 15 minutes, the final bainite fraction was higher. This led to a lower ultimate strength of 1220 MPa (whereas at 15 minutes, it was 1260 MPa in the C3Mn1.4Al grade). The resulting hardness of 376 HV10 was lower than in the previous specimen (387 HV10). However, elongation $A_{5mm}$ [14] was higher: 22%, as opposed to 17%. The
volume fraction of retained austenite decreased to 16%. With the other material, C3Mn2Al, the trend was the same.

![Microstructures](image)

**Figure 3.** Microstructures of the materials after austempering at 400°C with 15 minute (left) and 60 minute (right) holding times.

**Table 2.** Mechanical properties of the materials after austempering at 400°C.

| Experimental material | Treatment route | Offset yield strength ($R_{\text{p0.2}}$) [MPa] | $R_{\text{p0.2}}$ standard deviation | UTS ($R_m$) [MPa] | $R_m$ standard deviation | $A_{5\text{mm}}$ [%] | $A_{5\text{mm}}$ standard deviation |
|-----------------------|-----------------|---------------------------------------------|----------------------------------|------------------|--------------------------|-----------------|----------------------------------|
| C3Mn1.4Al             | 400°C - 15 min. | 683                                         | 7.1                              | 1257             | 7                        | 19.3            | 0.008                             |
|                       | 400°C - 60 min. | 673                                         | 6.8                              | 1223             | 2                        | 25.3            | 3.1                              |
| C3Mn2Al               | 400°C - 15 min. | 824                                         | 19                               | 1386             | 8                        | 16.6            | 1.0                              |
|                       | 400°C - 60 min. | 631                                         | 4                                | 1220             | 5                        | 21.1            | 0.7                              |

After austempering at the higher temperature of 425°C, the C3Mn1.4Al grade developed a bainitic-martensitic microstructure with a larger amount of martensite and less bainitic ferrite (figure 4) than after the previous treatment at 400°C. The strength after the sequence with a holding time of 15 minutes was higher: 1380 MPa. Elongation reached 15% (table 3) and hardness was 428 HV10 (figure 6). The amount of retained austenite was 8% (figure 7). After the 60-minute route, the ultimate strength was higher, 1360 MPa, and elongation and hardness were 15% and 400 HV10, respectively, whereas the amount of RA was less: 8%. The C3Mn2Al grade had a bainitic-martensitic microstructure with some bainitic ferrite and 9% retained austenite. The ultimate strength was between 1360 and 1375 MPa, the elongation was 15 % (table 3) and hardness reached 408 HV (figure 6).

After intercritical annealing at 425°C, mechanical properties were higher but elongation remained unchanged in both materials. The retained austenite fraction decreased to 8–11% (figure 7). The final microstructure contained bainite and martensite at a well-balanced ratio (figure 4). After this treatment, the material attained the properties required in final products for safety-relevant parts of the car body.
Figure 4. Microstructures of the materials after austempering at 425 °C with 15 minute (left) and 60 minute (right) holding times.

Table 3. Mechanical properties of the materials after holding at 425°C.

| Experimental material | Treatment route | Offset yield strength ($R_{p0.2}$) [MPa] | $R_{p0.2}$ standard deviation | UTS ($R_m$) [MPa] | $R_m$ standard deviation | $A_{5mm}$ [%] | $A_{5mm}$ standard deviation |
|-----------------------|-----------------|------------------------------------------|-----------------------------|-------------------|--------------------------|-------------|--------------------------|
| C3Mn1.4Al             | 425°C - 15 min. | 893                                      | 69                          | 1379              | 14                       | 15.3        | 0.04                     |
|                       | 425°C - 60 min. | 727                                      | 43                          | 1238              | 100                      | 15.0        | 2.3                      |
| C3Mn2Al               | 425°C - 15 min. | 765                                      | 9                           | 1364              | 4                        | 14.9        | 2.0                      |
|                       | 425°C - 60 min. | 730                                      | 1                           | 1361              | 6                        | 13.5        | 1.0                      |

After holding at 450°C, the amount of martensite in the martensitic-bainitic structure C3Mn1.4Al was higher still (figure 5). The ultimate strength increased to 1400 MPa, elongation was 14–18% (table 4) and hardness was close to 450 HV10. The retained austenite fraction was 6% after the route with the shorter holding time and 9% after the 60-minute hold. In C3Mn2Al, the microstructure consisted of martensite and bainite as well, with a higher proportion of martensite and some free ferrite (figure 5). The ultimate tensile strength and hardness were 1400 MPa and 431 HV10, respectively, after holding for 15 minutes. After the 60-minute hold, these values were 1424 MPa and 438 HV10. Elongation was approximately 14%. Treatments at higher holding temperatures led to higher mechanical properties and less retained austenite.
Figure 5. Microstructures of the materials after austempering at 450°C with 15-minute (left) and 60-minute (right) holding times.

Table 4. Mechanical properties of the materials after holding at 450°C.

| Experimental material | Treatment route | Offset yield strength (R_p0.2) [MPa] | R_p0.2 standard deviation | UTS (R_m) [MPa] | R_m standard deviation | A5mm [%] | A5mm standard deviation |
|-----------------------|----------------|------------------------------------|---------------------------|----------------|------------------------|---------|------------------------|
| C3Mn1.4Al             | 450°C - 15 min. | 860                                | 10                        | 1370           | 13                     | 14.6    | 0.9                    |
|                       | 450°C - 60 min. | 888                                | 13                        | 1394           | 1                      | 13.7    | 0.4                    |
| C3Mn2Al               | 450°C - 15 min. | 811                                | 5                         | 1402           | 8                      | 13.8    | 0.1                    |
|                       | 450°C - 60 min. | 851                                | 11                        | 1424           | 2                      | 13.2    | 0.5                    |

Figure 6. Effects of heat treatment on hardness in C3Mn1.4Al and C3Mn2Al.
4 Conclusion

Intercritical annealing routes were designed for C3Mn1.4Al and C3Mn2Al grades. Breaking down and forging of ingots into round bars was trouble-free for C3Mn2Al. However, the other material suffered irreparable damage during heating to the forging temperature of 1150°C. Forging at lower temperatures was successful eventually.

The outcomes of the austenitizing trials were similar for both materials, leading to a temperature of 950°C and a soaking time of 30 minutes which led to fully martensitic microstructure after quenching. Finally, 425°C was chosen as the intercritical annealing temperature – on the basis of the mechanical properties and microstructures obtained in the trials.

The findings from the manufacture of the stock and from the austenitizing and austempering trials will be important for future thermomechanical treatment of advanced multiphase high-strength aluminium-alloyed steels. The ultimate goal is to apply this knowledge to a general design of thermomechanical treatment of third-generation advanced high-strength steels in the form of an upgraded press-hardening process.

Acknowledgement

This paper presents results of Student Grant Competition at the University of West Bohemia, SGS 2019-019 – Materials and technological research into advanced high-strength steels, supported from specific-purpose funds of the state budget for research and development administered by the Ministry of Education of the Czech Republic.

References

[1] Karbasian H Tekkaya A E 2010 A review on hot stamping, Journal of Materials Processing Technology 210 2103-18.

[2] Kučerová L, Jirková H and Mašek B 2014 The Effect of Alloying on Mechanical Properties of Advanced High Strength Steels Archives of Metallurgy and Materials 59 1189-92.

[3] Tamarelli C M 2011 AHSS 101: The Evolving Use of Advanced High-Strength Steels for Automotive Applications, Southfield, Michigan.

[4] Automotive Steels: Design, Metallurgy, Processing and Applications, Woodhead Publishing Series in Metals and Surface Engineering, 2017 ISBN: 978-0-08-100638-2.

[5] Totten G E 2007 Steel heat treatment: metallurgy and technologies, 2nd edition, ISBN-13: 978-0-8493-8455-4.
[6] Bhadeshia H K D H 2001 Bainite in steels, 2nd edition, London: Institute of Materials ISBN 1-86125-112-2.

[7] SENTE SOFTWARE LTD 2017, JMatPro the Materials Property Simulation Package Version 9.0 United Kingdom.

[8] Kim H Inoue J Okada M Nagat K 2017 Prediction of Ac3 and Martensite start temperatures by a data-driven model selection approach, ISIJ International 57 No. 12 2229-36.

[9] Cooman B C 2004 Structure-properties relationship in TRIP steels containing carbide-free bainite, Current Opinion in Solid State and Materials Science 8 pp. 285-303.

[10] Jirková H Opatová K Jeniček Š, Vrtáček J Kučerová, L Kurka P 2019. Use of multi-phase trip steel for press-hardening technology. Acta Metallurgica Slovaca, 25(2), 101-106. doi:10.12776/ams.v25i2.1267

[11] Kučerová L., Bystrianský M., Jeniček Š., Francisko, P 2017. Effect of deformation conditions on microstructure and mechanical properties of low alloyed steel. Manufacturing Technology, 17(5), 752-756.

[12] Jeniček Š, Bublíková D, Jirková H., Kána J. 2017. Stability of retained austenite in high-strength martensitic steels during cold deformation. Paper presented at the Annals of DAAAM and Proceedings of the International DAAAM Symposium, 289-294.

[13] Kotěšovec V, Vorel I., Jeniček Š., Káňa, J brahim, K. 2017. Impact of quenching temperature and isothermal holding time during austempering on bainite content in high-silicon steel. Paper presented at the IOP Conference Series: Materials Science and Engineering, 179(1) doi:10.1088/1757-899X/1161/1/012006

[14] Kučerová L., Jeniček Š., Mach J. 2018. Effect of sample geometry on tensile properties of chromium alloyed middle carbon steel. Paper presented at the METAL 2018 - 27th International Conference on Metallurgy and Materials, Conference Proceedings, 761-765.