New heat treatment process for advanced high-strength steels

D Bublíková, Š Jeníček, I Vorel, B Mašek

University of West Bohemia, RTI- Regional Technological Institute, Univerzitní 22, CZ – 306 14 Pilsen, Czech Republic

E-mail: dagmar.bublikova@seznam.cz

Abstract

Today’s advanced steels are required to possess high strength and ductility. It can be achieved by choosing an appropriate steel chemistry which has a substantial effect on the properties obtained by heat treatment. Mechanical properties influenced the presence of retained austenite in the final structure. Steels of this group typically require complicated heat treatment which places great demands on the equipment used. The present paper introduces new procedures aimed at simplifying the heat treatment of high-strength steels with the use of material-technological modelling.

Four experimental steels were made and cast, whose main alloying additions were manganese, silicon, chromium, molybdenum and nickel. The steels were treated using the Q-P process with subsequent interrupted quenching. The resulting structure was a mixture of martensite and retained austenite. Strength levels of more than 2000 MPa combined with 10-15 % elongation were obtained. These properties thus offer potential for the manufacture of intricate closed-die forgings with a reduced weight. Intercritical annealing was obtained structure not only on the basis of martensite, but also with certain proportion of bainitic ferrite and retained austenite.

Keywords: Q&P process, retained austenite, AHSS, intercritical annealing

1 Introduction

Today, there is a strong demand for simplified heat treatment of high-strength low-alloy steels. Modern heat treatment routes include processes that lead to TRIP-type microstructure, and Q&P processing. The concept of TRIP steels relies on a mixture of bainite, ferrite and retained austenite [Fig. 1] which is produced by intercritical annealing followed by controlled cooling and isothermal holding at the bainitic transformation temperature [1]. This route can provide ultimate strengths up to 1500 MPa and elongation levels of about 25 %. The other method, the quenching and partitioning (Q&P) process, allows strengths of more than 2000 MPa to be achieved, together with an elongation level of about 10 % [2]. Neither of them can be developed under real-world conditions. The reason is that both of them involve complex processes, in which a number of parameters must be optimized. In such case, material-technological modelling carried out in a thermomechanical simulator becomes a useful tool [Fig. 3]. With this method, small amounts of material can processed under laboratory conditions approaching the real-world process.

Stabilisation of retained austenite is an important factor in both of these heat treatment routes [Fig. 2]. Q&P processing can lead to high fractions of retained austenite. It is characterized by rapid cooling from austenite region to a temperature between the $M_f$ and $M_s$ temperatures, during which martensite forms, while a portion of austenite remains untransformed. During subsequent isothermal holding, the retained austenite becomes stabilised thanks to carbon which migrates from the super-saturated martensite to austenite. According to current knowledge, this austenite, which forms during the Q&P process, exists primarily in the form of thin films between martensite laths.
or plates [3]. It differs in this respect from the granular austenite that exists in steels which were heat treated to obtain the TRIP microstructure [4]. In high-strength steels, final microstructure and mechanical properties upon heat treatment are dictated mainly by their alloying concept. In order to stabilise retained austenite, it is important to increase the nickel content. To shift the $M_s$ and $M_f$ temperatures toward lower values, high-strength steels are alloyed with molybdenum and manganese. In addition, the levels of chromium and silicon are important for achieving sufficient mechanical properties.

2 Experimental

For this experiment, four new experimental steels were created whose special chemistries lower the $M_s$ and $M_f$ temperatures (Tab. 1). The aim was to carry out Q&P processing and heat treatment for TRIP microstructure on these steels to ascertain whether these chemistries can lead to different final microstructures, and to acceptable amounts of stabilised retained austenite.

In all these steels, the $M_s$ and $M_f$ temperatures were depressed predominantly through additions of manganese, silicon, and chromium. Silicon was chosen in order to prevent carbide formation and thus to provide adequate super-saturation of martensite with carbon. Molybdenum was employed to reduce the $M_s$ and $M_f$ temperatures and to shift the start of ferritic and pearlitic transformations towards lower cooling rates. Nickel was added in small amounts to stabilise austenite during cooling, to enhance hardenability, and to provide solid solution strengthening. The carbon content was the same in all steels: between 0.42 and 0.43 %.

These chemistries were developed with the aid of the JMatPro software which had been used for calculating the approximate transformation temperatures. In the AHSS-1 steel, the manganese level was 2.5 %, and the silicon level was 2.03%. The calculated $M_s$ and $M_f$ temperatures were 218 °C...
and 88 °C, respectively. In order to find whether molybdenum affects mechanical properties and transformation temperatures, its content in the AHSS-2 steel was set to 0.16%. However, this molybdenum content has not altered the $M_s$ and $M_f$ temperatures in any substantial way. The $M_s$ and $M_f$ temperatures were 214°C and 83°C, respectively. In AHSS-3, nickel level was 0.56 % to provide the desired hardenability and to depress martensitic transformation temperatures. The $M_s$ and $M_f$ temperatures were 209 °C and 78 °C, respectively. In AHSS-4, the nickel and molybdenum levels were identical to the previous two steels. This led to the lowest transformation temperature $M_s$ of 204 °C, and the $M_f$ of 73 °C. The largest difference between the $M_f$ temperatures found by calculation was a mere 15 °C. Nevertheless, it can still affect the treatment and the choice of quenchants.

Table 1. Chemical compositions of experimental steels AHSS-1–4 [wt. %.]

|       | C  | Mn | Si  | P     | S     | Cu  | Cr  | Ni  | Al  | Mo  | Nb  | $M_s$ | $M_f$ |
|-------|----|----|-----|-------|-------|-----|-----|-----|-----|-----|-----|-------|-------|
| AHSS-1| 0.43| 2.5| 2.03| 0.005 | 0.003 | 0.07| 1.33| 0.07| 0.008| 0.03| 0.03| 218   | 88    |
| AHSS-2| 0.428| 2.48| 2.03| 0.005 | 0.003 | 0.07| 1.46| 0.08| 0.004| 0.16| 0.03| 214   | 83    |
| AHSS-3| 0.419| 2.45| 2.09| 0.005 | 0.002 | 0.06| 1.34| 0.56| 0.005| 0.04| 0.03| 209   | 78    |
| AHSS-4| 0.426| 2.46| 1.99| 0.005 | 0.002 | 0.06| 1.33| 0.56| 0.005| 0.15| 0.03| 204   | 73    |

2.1 Q&P Process

Several heat treatment routes were designed (Tab. 2). The appropriate austenitizing temperature ($T_A$), quenching temperature (QT), and partitioning temperature (PT) were chosen. For this treatment method, the quenching temperature is normally between the $M_s$ and $M_f$. The partitioning temperature, at which carbon migrates from the super-saturated martensite to austenite, is of importance as well.

Table 2. Heat treatment parameters and resulting mechanical properties.

| Route number | $T_A$ [°C]/$t_A$ [s] | Cooling rate [°C/s] | QT [°C] | PT $[°C]$ /$t_{PT}$ [s] | HV [1] | $R_m$ (UTS) [MPa] | $A_{5mm}$ [%] | RA [%] |
|--------------|----------------------|---------------------|----------|-------------------------|-------|------------------|-------------|-------|
| 1            | 850/100              | 1                   | 100      | 150/600                 | 573   | 2401             | 7           | 9     |
| 2            | 850/100              | 1                   | 150      | 200/600                 | 679   | 2308             | 10          | 12    |
| 3            | 850/100              | 16                  | 150      | 200/600                 | 693   | 2354             | 7           | 9     |
2 Intercritical Annealing

The other heat treatment method which was tested on the two experimental steels was intercritical annealing for bainite (Tab. 3). Using the JMatPro software, three routes were designed with various austenitizing temperatures ($T_A$), rates of cooling to the holding temperature for bainitic transformation ($T_B$), the rate of cooling to a temperature above $M_S$ and the rate of cooling to room temperature. The heating temperature was between the critical temperatures $A_1$ and $A_3$. Holding at the bainitic transformation temperature was important because at that stage austenite becomes stabilised and the strength of the material increases. The resulting fractions of phases are also influenced by the holding time. The rate of cooling to room temperature affects the final amount of retained austenite.

Table 3. Heat treatment parameters and amounts of retained austenite.

| Route number | $T_A$ [°C]/$t_A$ [s] | Cooling rate to $T_B$ [°C/s] | $T_B$ [°C]/$t_B$ [s] | Cooling rate to 250 °C [°C/s] | Cooling rate to RT [°C/s] | HV [1] | RA [%] |
|--------------|---------------------|-------------------------------|---------------------|-------------------------------|--------------------------|-------|-------|
| 01           | 850/100             | 14                            | 400/240             | 0.1                           | 14                       | 475   | 10    |
| 02           | 850/100             | 14                            | 400/60              | 0.1                           | 14                       | 688   | 9     |
| 03           | 850/100             | 14                            | 400/60              | 1                             | 14                       | 682   | 7     |

After both heat treatment routes, the amounts of retained austenite in selected specimens were measured using X-ray diffraction (Tab. 2., Tab. 3.).

3 Discussion of Results

In all steels, Q&P processing led to predominantly martensitic microstructures with some amounts of bainite and retained austenite. The highest hardness was 693 HV10 (Figs. 1–4). Retained austenite was probably located along the boundaries between martensite needles. The largest amount of retained austenite in the martensitic matrix, almost 12 %, was found in the AHSS-3 steel which had a higher nickel level (Tab. 2). Using transmission electron microscope, the morphology of this retained austenite was studied. In selected diffraction spots under dark field illumination, retained austenite was identified along the boundaries of prior austenite grains, subgrains, and martensite laths (Figs. 5 and 6).
Intercritical annealing for bainite in route 01 resulted in a tempered bainitic microstructure with martensite islands along prior austenite grains and a hardness of 475 HV10 (Figs. 7 and 8). Route 02 had shorter holding time at the bainitic transformation temperature than route 1. This led to martensitic-bainitic microstructure with some islands of bainitic ferrite (Figs. 9 and 10). The amount of martensite was thus larger and hardness was higher: 688 HV10. Cooling rate was increased in route 03 (Tab. 3), which led to martensitic-bainitic microstructure. As in previous cases, the largest fraction of retained austenite was found in the AHSS-3 steel (which had higher nickel level). Route 01 led to 10 % retained austenite. The specimen produced by route 01 was examined by transmission electron microscopy as well. Using dark field illumination, retained austenite was identified mainly along the boundaries between martensite laths.

![Figure 1. Route 2: AHSS-3, cooling rate: 1 °C/s.](image1)

![Figure 2. Route 2: AHSS-3, martensitic-bainitic structure, detail scanning electron micrograph.](image2)

![Figure 3. Route 2: AHSS-2, cooling rate: 1 °C/s.](image3)

![Figure 4. Route 2: AHSS-2, martensitic-bainitic structure, detail scanning electron micrograph.](image4)
Figure 5. Route 2, AHSS-3, dark-field micrograph of retained austenite distribution.

Figure 6. Route 2: AHSS-3, diffractogram of martensitic matrix with indicated austenite diffraction spots under dark field illumination. Zone axis of diffraction: \( z = [1\ 1\ 1] \).

Figure 7. Route 01: rate of cooling to \( T_B \): 14 °C/s, holding time: 240 s.

Figure 9. Route 02: rate of cooling to \( T_B \): 14 °C/s, holding time: 60 s.
Figure 8: Route 01, bainitic structure with martensite islands along prior austenite grain boundaries – detail scanning electron micrograph.

Figure 10: Route 02: martensitic-bainitic structure, scattered areas of bainitic ferrite – scanning electron micrograph.

Figure 11: Route 01, AHSS-3, dark-field micrograph of retained austenite distribution.

Figure 12: Route 01: AHSS-3, diffractogram of martensitic matrix with indicated austenite diffraction spots under dark field illumination and the indicated spot (004)γ+(022)α.

4 Conclusion
Two heat treatment methods, Q&P process and intercritical annealing for bainite, were tested on newly-developed AHSS-type low-alloy steels containing manganese, silicon, chromium, molybdenum and nickel. This experiment with several different heat treatment routes has shown that the microstructure that forms in these high-strength steels is not exclusively martensite-based hardening microstructure. As expected, intercritical annealing produced bainitic ferrite. Mechanical properties
and findings obtained by transmission electron microscopy suggest that Q&P process is a better alternative for these steels. In the material whose nickel content was increased to 0.56%, Q&P processing produced almost 12% retained austenite in the martensitic matrix. Retained austenite was found on boundaries of prior austenite grains and subgrains and along the boundaries of martensite laths. In this material, intercritical annealing for bainite produced approximately 10% of retained austenite. It was found mainly along the boundaries between martensite needles.

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