

STABILIZATION OF RETAINED AUSTENITE IN HIGH-STRENGTH MARTENSITIC STEELS WITH REDUCED MS TEMPERATURE

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Abstract

Today's advanced steels are required to possess high strength and ductility. One way of achieving a favourable ductility level and a very high ultimate strength is to stabilize retained austenite in martensitic matrix. Among heat treatment processes capable of producing such microstructure, there is the Q&P process (Quenching and Partitioning). It can produce microstructures consisting of martensite and a certain amount of retained austenite, which exhibit strengths above 2000 MPa and elongation levels of 10-15 %. For some processes, it is appropriate to depress the Ms and Mf temperatures and modify the ferritic and pearlitic transformations to occur at lower cooling rates. By choosing an appropriate steel chemistry, the retained austenite can be stabilized in martensitic matrix at lower temperatures than in the ordinary Q&P process. Four steels were selected for the present experimental programme. Several heat treatment sequences were tried with these steels and multiple parameters were varied: austenitizing temperature, cooling rate, quenching temperature and the carbon partitioning temperature.

Final strengths of the steels were in the range of 1750-2400 MPa and their A5 mm elongation level was up to 10 %. The morphology and distribution of retained austenite dictate the resulting mechanical properties. The microstructures were studied using transmission electron microscopy. The distribution and morphology of retained austenite were examined using diffraction analysis (SAED) and bright and dark-field illumination. X-ray diffraction was employed to measure the volume fraction of retained austenite. The values were between 9 and 15 %.

Keywords: Q-P process, retained austenite, AHSS, UHSS, transmission electron microscopy, XRD

1. INTRODUCTION

Current trends in the processing of high-strength low-alloy steels consist in an effort to achieve the best possible toughness while maintaining high strength of the processed material. This can be accomplished by several methods. The methods include long-time low-temperature austempering, TRIP processing and Q&P processing. Long-time low-temperature austempering can produce tensile strengths of more than 1500 MPa and hardness levels of 420-480 HV10. The resulting microstructure can show elongations between 15 and 20 % [1]. Long-time low-temperature austempering is characterized by long holding times of several tens of hours, by low temperatures and by the use of various media, typically molten salt baths. The resulting microstructure consists of very fine bainitic ferrite [2]. The weakness of this procedure is its long annealing times. It is also the reason why it has not found use in industry. In TRIP steels, the desired properties are achieved thanks to their microstructure containing bainite, ferrite and retained austenite (RA). During deformation, RA transforms to deformation-induced martensite. These steels can have strengths up to 1200 MPa [3]. The third method is the Quenching and Partitioning (Q&P) process, which allows strengths of more than 2000 MPa to be obtained, together with an elongation of about 10 %. An important aspect of this process is the stabilisation of austenite in the martensitic matrix. One of the ways of obtaining martensitic structure with the desired fraction of retained austenite is a special heat treatment procedure described below. It is characterized by rapid cooling from the austenite region to a temperature between the Ms and Mf. During such cooling, martensite forms, while a portion of austenite remains untransformed. During subsequent

isothermal holding, the retained austenite becomes stabilized thanks to carbon which migrates from the super-saturated martensite to austenite. According to current knowledge, this austenite exists primarily in the form of thin films between martensite laths or plates [4-7]. In this respect, it differs from the granular austenite in TRIP steels. An important factor in stabilizing the retained austenite by this heat treating sequence is the chemistry of the material.

2. EXPERIMENTAL PROGRAMME

The use of the Q&P process in real-world processing depends on the ability to interrupt quenching between the Ms and Mf temperatures. With respect to this issue, four new experimental steels have been proposed. Their special chemistries were designed to depress the Ms and Mf temperatures (**Table 1**). The aim of this effort was to explore whether the process can be simplified and other quenching media or procedures can be used. In all of these experimental steels, the Ms and Mf temperatures were depressed predominantly through additions of manganese, silicon and chromium. Silicon was added in order to prevent carbide formation and facilitate the super-saturation of martensite with carbon. Molybdenum was employed to reduce the Ms and Mf temperatures and to shift the start of ferritic and pearlitic transformations towards lower cooling rates. Nickel was added in small amounts to stabilize 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 have been developed with the aid of the JMatPro software which was used for calculating the approximate transformation temperatures. In the AHSS-1 steel, the manganese level was 2.5 % and the silicon level was 2 %. The calculated Ms and Mf 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 chosen as 0.15 %. This molybdenum content has not altered the Ms and Mf temperatures in any substantial way. The Ms temperature was 214 °C and the Mf was 83 °C. In AHSS-3, the nickel level was set at 0.5 % to achieve the desired hardenability and to depress the martensitic transformation temperatures. The Ms and Mf temperatures were 209 °C and 78 °C, respectively. In AHSS-4, the nickel and molybdenum levels were identical to the previous two steels. The resulting chemistry led to the lowest transformation temperature Ms of 204 °C and the Mf of 73 °C.

The maximum calculated difference between the Mf values was a mere 15 °C. Yet, even this small variation may play a role in the process and in the choice of quenchants.

Table 1 Chemical composition of the experimental steels AHSS-1 - AHSS-4 [wt. %]

	C	Mn	Si	P	S	Cu	Cr	Ni	Al	Mo	Nb	Ms	Mf
AHSS-1	0.430	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

To find whether this heat treating sequence is viable, several important parameters of the Q&P process had to be trialled and optimised for the experimental steels which had been designed on the basis of calculations. These parameters included austenitising temperature, soaking time at austenitizing temperature, cooling rate, quenching temperature and partitioning temperature and time. A number of heat treatment schedules (**Table 2**) were tried with the goal of appropriate stabilization of austenite. The cooling from the austenitising temperature (TA) to the quenching temperature (QT) of 150-100 °C took place at the rates of 1 °C / s, 5 °C / s, 10 °C / s and 16 °C / s. The partitioning temperature (PT), at which carbon diffuses from super-saturated martensite,

was set at 150-200 °C. Specimens treated in this way were then used for metallographic examination and mechanical testing.

The retained austenite fraction was measured using X-ray diffraction. The distribution and morphology of retained austenite in martensitic matrix were studied in selected specimens using selected area electron diffraction (SAED) and bright field and dark field illumination in a transmission electron microscope.

Table 2 Heat treatment schedules

Schedule number	TA [°C]/tA [s]	Cooling rate [°C/s]	QT [°C]	PT [°C] /tPT [s]
1	850/100	1	100	150/600
2	850/100	5	100	150/600
3	850/100	10	150	200/1200
4	850/100	16	150	200/600

3. DISCUSSION OF RESULTS

In all steels, the resulting microstructures consisted of a majority of martensite, a small amount of bainite and a fraction of retained austenite (**Figs. 1-4**). The ultimate strength was between 1750 and 2400 MPa and the elongation reached approx. 10 %. The retained austenite volume fraction in the martensitic matrix was up to 15 vol. %, which corresponds to the lower limit of the ultimate strength achieved. This material contained the largest fraction of retained austenite (**Table 3**). As the best values have been achieved in the AHSS-3 steel (UTS of 2400 MPa, elongation of 10 % and RA of 15 %), its specimen was subsequently studied using transmission electron microscopy (TEM). TEM allows the microstructure and the presence of retained austenite to be explored in greater detail.

Table 3 Retained austenite fractions in individual Q&P-processed AHSS steels

Material	Fraction of γ -Fe [vol. %]
AHSS-1	9 %
AHSS-2	14 %
AHSS-3	15 %
AHSS-4	15 %

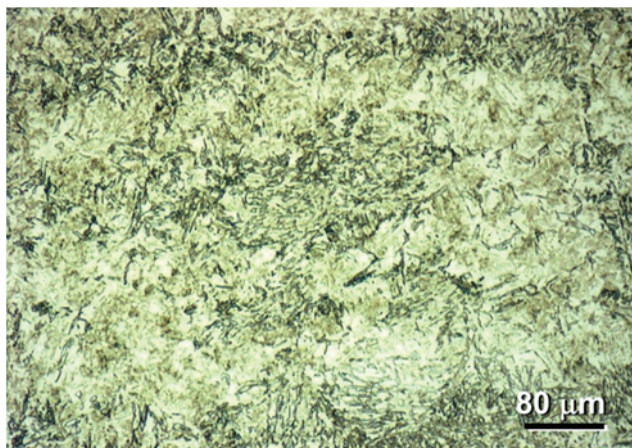


Fig. 1 AHSS-2 - cooling rate: 16 °C / s

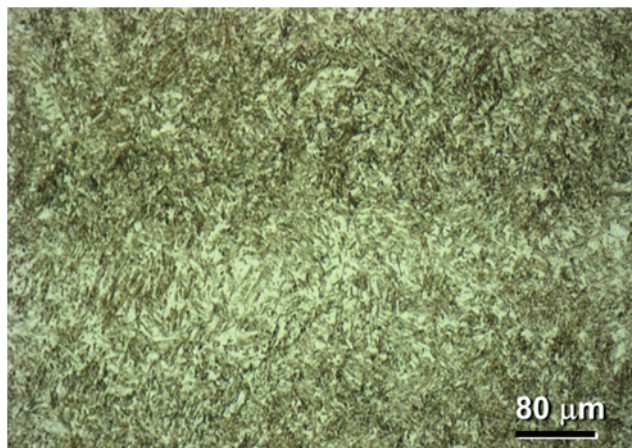


Fig. 3 AHSS-1 - cooling rate: 1 °C / s

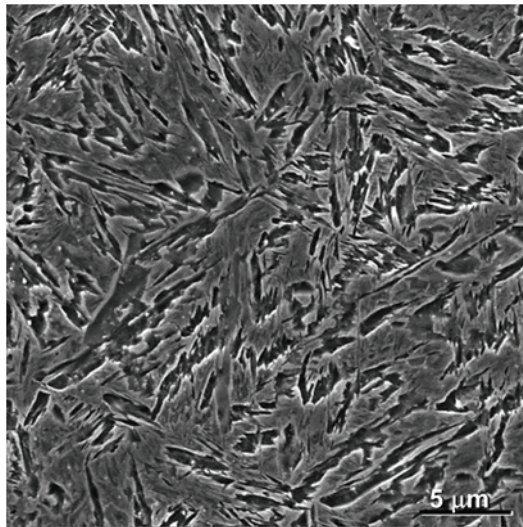


Fig. 2 AHSS-2 - cooling rate: 16 °C / s

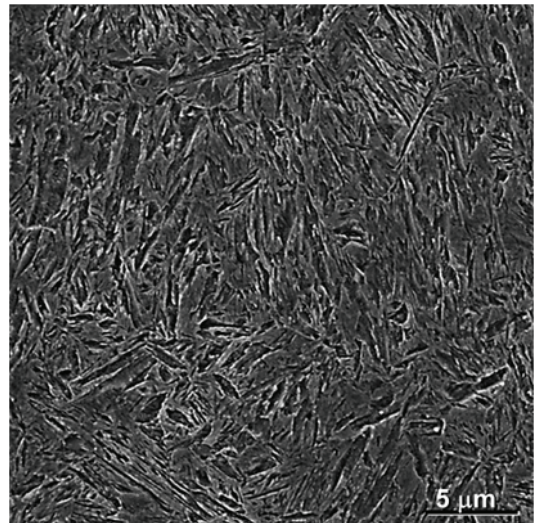


Fig. 4 AHSS-1 - cooling rate: 1 °C / s

First, the material was observed under bright-field illumination. Individual martensite laths were found which had grown from the prior austenite grain boundaries (**Fig. 5**). The martensitic matrix was confirmed using SAED. Retained austenite in the martensitic matrix was identified using dark-field illumination and selected diffraction spots. It was found along prior austenite grain boundaries, sub-grain boundaries and boundaries of martensite laths (**Figs. 6-11**).



Fig. 5 Prior austenite grain in martensitic matrix, AHSS-3

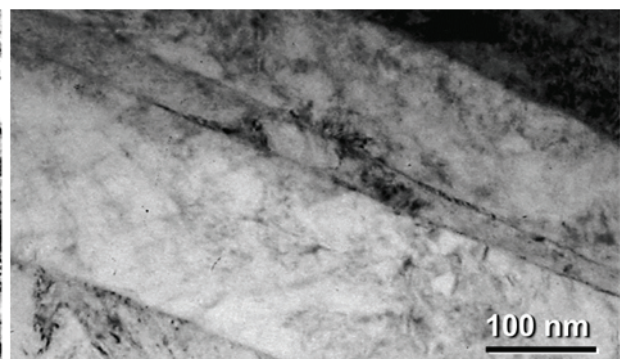


Fig. 6 Martensitic matrix under bright-field illumination, AHSS-3

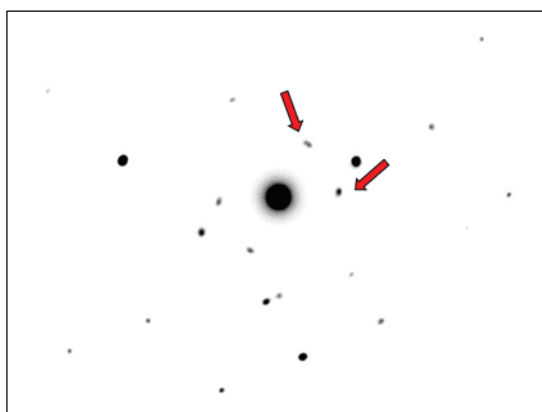


Fig. 7 Diffractogram of the martensitic matrix with selected austenite diffraction spots under dark field illumination. Zone axis of diffraction: $z = [1\ 1\ 1]$, AHSS-3

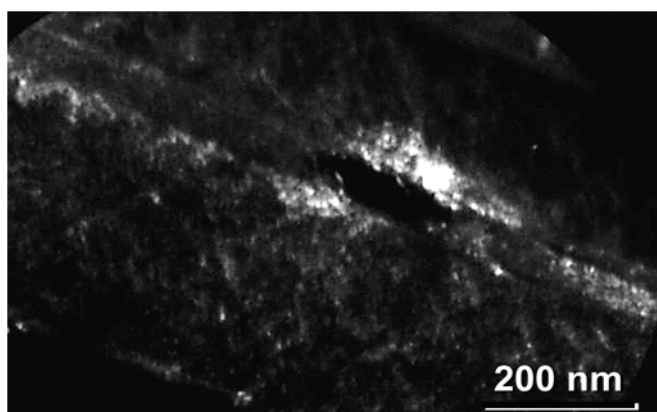


Fig. 8 Distribution of retained austenite under dark field illumination, AHSS-3

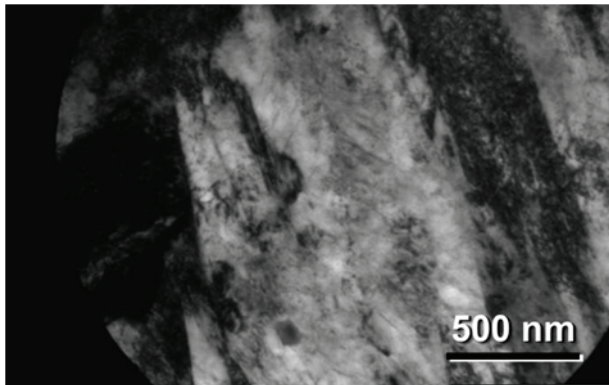


Fig. 9 Martensitic matrix under bright-field illumination, AHSS-3

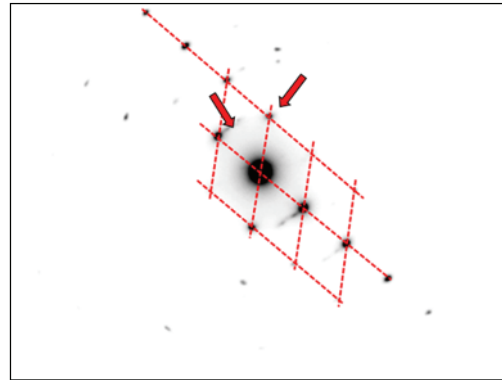


Fig. 10 Diffractogram of the martensitic matrix with marked spots taken to verify the presence of austenite. Zone axis of diffraction: $z = [3\ 5\ 6]$, AHSS-3

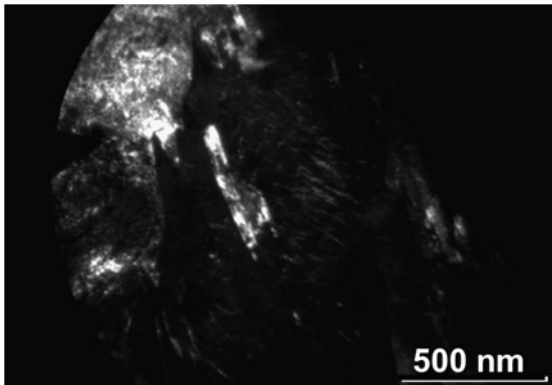


Fig. 11 Distribution of retained austenite under dark field illumination, AHSS-3

4. CONCLUSION

A novel heat treatment procedure based on the Q&P process has been tested on newly-developed AHSS-type low-alloy steels alloyed with manganese, silicon, chromium, molybdenum and nickel. The procedure involved low-temperature treatment, in which quenchants other than the commonly used ones can be applied in the future. The purpose is to replace molten salt baths with quenchants based on water, steam, polymers or mist. Quenching oils are considered to be another alternative. The use of various cooling rates during the quenching of these experimental steels proved that within the broad range between 5 °C / s and 16 °C / s, martensite-based hardening microstructures with a fraction of retained austenite of up to 15 % can be obtained and strengths between 1750 and 2400 MPa can be achieved. The highest content of retained austenite showed AHSS-1 and AHSS-3 steels with increased content of Ni (0.56 %). Transmission electron microscopy observation of the steel AHSS-3 which showed the best results revealed the distribution of retained austenite along prior austenite grain boundaries and subgrain boundaries.

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