Effect of soaking temperature on the microstructure and mechanical properties of heat-treated Al-Si-Nb TRIP steel

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Abstract. Two-step heat treatment with bainitic hold at 425 °C was applied to low alloyed high strength steel with partial substitution of silicon by aluminium. The steel was further micro-alloyed by niobium. Various soaking temperatures in the range of 750-1250 °C were used to analyse the effect of soaking on mechanical properties and microstructure of the steel. According to calculations in JMatPro, the temperatures below 900 °C should lie in an intercritical (two phase) region, while the temperature of 900 °C should be the first one to ensure full austenitization of this steel. To dissolve different portion of niobium into solid solution, higher soaking temperatures were also used. Tensile strengths of 780-1069 MPa and total elongation of 22-46 % were achieved.

1. Introduction
Ongoing development of materials with increased tensile strength, ductility and formability are fuelled particularly by the demands of an automotive and vehicle industry. Increasing requirements on CO₂ reduction and lower fuel consumption coupled with increasing safety requirements could be satisfied by application of materials with higher strengths which will enable weight reduction without compromising on the safety of the constructions.

Several grades of advanced high strength steels (AHSS) have been developed in recent years, with various combinations of tensile strength and ductility [1-5]. TRIP (transformation induced plasticity) steels belong to the first generation of AHSS, using the principle described by Zackay [6] on a novel lean alloying concept. Modern TRIP steels are low alloyed steels with carbon contents between 0.2-0.4 % [1, 7-11], but most frequent are carbon contents around 0.2 %. The steel is alloyed by 1-2 % of manganese which increases stability of retained austenite and originally, between 1-2 % of silicon was used to prevent carbide precipitation during the treatment. As those high silicon steels possessed low surface quality, which presents problems during galvanising, attempts were made to find suitable substitution [12]. The most promising possibility seems to be partial substitution of silicon by aluminium, which has the same effect on carbide precipitation hindering and solid solution strengthening as silicon [13, 14]. However, the effects of aluminium are weaker than those of silicon for the same contents of both elements. This is the reason why a full substitution of silicon is not convenient, as it results in a decrease of tensile strengths [15-18]. Other minor alloying elements were also investigated in the attempts to improve mechanical or technological properties of TRIP steels. In this work, micro-alloying by niobium was used, as it was shown to have a beneficial effect on sensitivity to pearlite formation [5]. Carbide precipitation and pearlite formation are undesirable in TRIP steels, as they consume carbon which is needed for proper stabilization of retained austenite. TRIP steels contain typically multi-phased microstructures with either ferrite or bainite matrix, always containing bainite, retained austenite and often also small amounts of either martensite or so-called M-A constituent. M-A constituent
are larger bulk islands of remaining austenite that were not stable enough during the final cooling to the room temperature and therefore partially transformed to martensite (mainly at the edges of the islands). Retained austenite in TRIP steels can be of either lath or bulky morphology, the laths are mainly part of the bainitic microstructure, smaller islands of irregular shapes are often part of globular bainite while larger bulky islands are typically found at the edges of bainitic blocks [3, 5, 11]. The volume fraction, morphology and distribution of phases and structural components have a significant effect on mechanical properties of TRIP steels [9]. In this work, the effect of soaking temperature on the final microstructure and mechanical properties of Al-Si-Mn-Nb TRIP steel.

2. Experimental program

2.1. Material

Low carbon low alloyed high strength steel with partial substitution of silicon by aluminium and micro-alloying by niobium was used in this work (table 1).

| Table 1. Chemical composition of used steel in weight %. |
|-----------------|---|---|---|---|---|---|---|---|
| C               | Mn | Si | P  | S  | Cr | Ni | Cu | Al | Nb |
| 0.2             | 1.5| 0.5| 0.008| 0.005| 0.008| 0.072| 0.058| 1.5| 0.06|

TTA (Time–temperature-austenitization) diagram of this steel was calculated using JMatPro software (figure 1 a). According to the diagram, the austenitization of used experimental steel starts around 740 °C and full austenitization can be expected above the temperature of 980 °C.

a) Figure 1. a) TTA diagram. b) Scheme of used two-step heat treatment.

2.2. Heat treatment

Experimental steel was cast in induction vacuum furnace, the ingot was cut into four parts and forged at 1150 °C into bars with the diameter around 25 mm. The bars were cooled in the cold furnace after the forging used for the preparation of samples for subsequent heat treatment.

Heat treatment was carried out at thermo-mechanical simulator, which enables the application of precisely defined and controlled thermal and deformation parameters. High-frequency electrical resistance heating is used to heat the specimen. With a maximum power of 3 kW, the specimens can be heated in a controlled manner at rates in excess of 200 °C/s, depending on the material and shape of the test piece. The active part of the sample is cylindrical with 8 mm diameter and length of 16 mm.

Two-step heat treatment typical for processing of TRIP steels was designed for this work (figure 1 b). Various soaking temperatures in the region of 750-1250 °C were applied to the steel, the other processing parameters were kept the same and chosen based on previous experiments [10,13]. All the samples had the same 100 s hold at the soaking temperature, followed by cooling to the second
600 s hold at 425 °C, where bainite transformation took place. Constant cooling rate of 30 °C/s was used. Lower soaking temperatures of 750-950 °C should be in the two-phase intercritical temperature interval, according to a JMatPro calculation and therefore a varying fraction of polygonal ferrite should remain in the resulting microstructure. The temperature of 1050 °C should already ensure a full austenitization of the prior microstructure and only proeutectoid ferrite should appear in the final microstructure. The highest temperatures of 1230 and 1250 °C were chosen to observe the effect of niobium dissolved in solid solution on the mechanical properties, as niobium particles were reported to start dissolving in these steels at around 1050 °C [19].

2.3. Characterisation
Processed samples were cut along the longitudinal axes for metallographic analysis and the metallographic sections were prepared in a standard way. All samples were etched in 3 % Nital to reveal the microstructure, which was observed by an Olympus BX 61 light microscope and a Zeiss Crossbeam scanning electron microscope. The volume fraction of the retained austenite was determined by X-ray diffraction phase analysis using an AXS Bruker D8 Discover automatic powder diffractometer with a HI-STAR detector and Co lamp (λKα = 0.1790307 nm). The measurement was carried out in the central part of the samples and spectra were taken in the range of 2θ from 25° to 110°. The integrated intensities of (200) ferrite peak and (111), (002) and (022) austenite peaks were used for evaluation. Mechanical properties were determined by tensile tests of sub-size flat samples with a gauge length of 5 mm and a cross-section of 2 x 1.2 mm. Two samples from each treatment were tested and average values are given in table 2.

3. Results and discussion
A significant amount of polygonal ferrite was found in the samples with soaking temperatures of 750 and 770 °C (figure 2 a). The sample soaked at 800 °C possessed a mixture of polygonal ferrite grains and proeutectoid ferrite placed at prior austenite boundaries (figure 2 b). Interestingly, polygonal ferrite was missing in sample soaked at 850 °C despite JMatPro prediction. Only large islands of bainitic ferrite could be observed at light micrographs (figure 2 c).

![Figure 2. Microstructures of samples soaked at a) 770 °C, b) 800 °C, c) 850 °C.](image)

Very fine lines of proeutectoid ferrite could be still seen also in the microstructure obtained by soaking at 900 °C (figure 3 a), while higher soaking temperature resulted in the bainitic microstructures (figure 3 b, c) with various bainite morphologies. Detailed microstructure analysis (figure 4) showed changing bainitic microstructure. For lower soaking temperatures, the bainite was predominantly granular, with just occasional area of lath bainite. Retained austenite in bainite obtained at soaking temperature of 750 °C (figure 4 a) possessed relatively uniform size and by far the largest amount of retained austenite (23%) was measured in this microstructure by an X-ray diffraction phase analysis (table 2). Around 50% of ferrite in the microstructure could have contributed to this extensive retained austenite stabilization, as more carbon was, therefore, present in the remaining austenite leading to more significant chemical stabilization of austenite.
Figure 3. Microstructures of samples soaked at a) 900 °C, b) 1050 °C, c) 1250 °C.

Table 2. Mechanical properties (YS - yield strength, UTS – ultimate tensile strength, EL – total elongation) and retained austenite volume fraction (RA)

| Soaking temperature [°C] | YS [MPa] | UTS [MPa] | EL [%] | RA [%] |
|--------------------------|----------|-----------|--------|--------|
| 750                      | 521±13   | 786±10    | 41±0   | 23     |
| 770                      | 633±6    | 781±8     | 46±0   | 14     |
| 800                      | 561±10   | 789±2     | 45±0   | 12     |
| 850                      | 614±3    | 822±2     | 46±0   | 10     |
| 900                      | 582±1    | 836±1     | 43±1   | 14     |
| 950                      | 658±4    | 843±3     | 43±1   | 12     |
| 1050                     | 897±1    | 1030±5    | 24±0   | 11     |
| 1230                     | 960±5    | 1069±6    | 22±0   | 11     |
| 1250                     | 946±6    | 1054±1    | 22±0   | 12     |

Even this high retained austenite content, however, did not result in an increased elongation in comparison with following samples with 10-14 % of retained austenite. It could be therefore expected that about half of this austenite didn’t contribute to the TRIP effect. Relative thin austenitic phase was detected at grain boundaries of proeutectoid ferrite obtained in the sample with a soaking temperature of 800 °C (figure 4 b).

Figure 4. Scanning electron microscopy of samples obtained by soaking at a) 750 °C, b) 800 °C, c) 900 °C, d) 1050 °C, e) 1230 °C, f) 1250 °C.
The samples with soaking temperature in the two-phase region (750-800 °C) possessed very similar mechanical properties, reaching tensile strength of 780-790 MPa with total elongations above 40% . Bainitic microstructure without polygonal ferrite was obtained in the sample soaked at 900 °C (figure 4 c), consisting of bainitic ferrite plates with sparsely distributed areas of retained austenite. The mixture of granular and lath bainite was found, with granular bainite prevailing. Larger islands of M-A constituent were observed in the microstructure. The best combinations of tensile strengths and total elongations were obtained for samples obtained by soaking at temperatures of 850-950 °C. These samples reached tensile strengths of 820-840 MPa with elongations of 43-46%.

With increasing soaking temperature, the bainite became denser and more of a lamellar type, with thinner laths of retained austenite and bainitic ferrite (figure 4 d-f). These laths are however slightly growing in length with increasing soaking temperature. Fine grains of proeutectoid ferrite can be found occasionally in the microstructure obtained by soaking at 1050 °C (figure 4 d). The mixture of lamellar upper bainite and granular bainite could be found in the microstructures obtained by soaking at the highest temperatures above 1200 °C (figure 4 e, f). The highest soaking temperature of 1250 °C resulted in the thinnest bainitic laths. The microstructures obtained by soaking at 1050-1250 °C have the same volume fraction of retained austenite (11%), regardless of the morphology of the bainite. Similarities in the microstructures are mirrored by their total elongations of 22-24% and high ultimate tensile strengths of 1030-1070 MPa.

It can be assumed that these high strengths were caused by thin bainitic laths, prevailing lath bainite could be also responsible for the drop of elongation. Lower elongation could be also assigned to the very high stability of retained austenite in these samples, which caused by extreme thickness of retained austenite laths (size stabilization). Nearly 9% of retained austenite were determined by X-ray diffraction phase analysis at the neck area of fractured tensile sample processed by soaking at 1250 °C. This suggests that only a very small amount of retained austenite actually transformed during the deformation and TRIP effect, therefore, could not be utilized.

4. Conclusions
Two-step heat treatment with annealing hold at 425 °C was applied to low carbon low alloyed TRIP steel. Nine heat treatments with various soaking temperatures in the region of 750-1250 °C were carried out to determine the effect of soaking temperature on the final microstructure and mechanical properties. Samples with soaking temperature in a two-phase (ferrite-austenite) region possessed the lowest tensile strengths around 785 MPa due to the presence of polygonal ferrite in the final microstructure. Their total elongation was above 40%. The soaking temperature of 850 °C already produced final microstructure without polygonal ferrite despite JMatPro calculations.

Samples with soaking temperatures around 900 °C produced predominantly bainitic microstructures with mainly granular morphology. They reached the best combinations of tensile strengths around 835 MPa with total elongations about 43%.

The last group of samples was processed by the highest soaking temperatures of 1050-1250 °C, where the increasing dissolution of niobium particles could be expected. Lath bainite was becoming the dominant morphology with increasing soaking temperature. These samples obtained the highest strengths around 1050 MPa with the lowest elongations around 22%. It was found out that the retained austenite in these samples was over stabilized and did not transform to martensite during tensile loading, probably due to very thin laths of retained austenite in the bainitic microstructures.

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