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RESEARCH PAPER

Investigation of the effect of isothermal heat treatments on mechanical properties of thermo-mechanically rolled S700MC steel grade

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ABSTRACT

The effect of isothermal heat treatments (1 hour at 200, 400, 600 and 800°C) on mechanical properties of thermo-mechanically rolled S700MC steel has been investigated by extensive mechanical characterizations. Treatments at 600°C increase yield and tensile strength and decrease impact energy. Below 600°C the steel retains its bainitic structure. Simulations of precipitation kinetics suggest that this hardening effect arises from the nucleation of fine (Ti,Nb)C particles, indicating that the bainitic structure is unstable above 600°C due to its high supersaturation with respect to C, Nb and Ti. These results can help to optimize the operating practices for post-weld heat treatments.
Keywords: High Strength Steels, TMCP, thermal treatment, S700MC

INTRODUCTION

In the last years the industrial demand for high strength steels (HSS) with increasing mechanical performance to reduce the overall weight of the components, has stimulated the steel producers to develop and commercialize many steel grades capable of reaching a strength up to 1 GPa and even more [1].
The industrial route for producing these materials makes use of the so-called thermo-mechanical control process (TMCP) which exploits the austenite grain refining properties of Nb as microalloying element, together with a proper design of the hot rolling schedule, to improve the homogeneity of the microstructure and enhancing the mechanical properties in the as-hot rolled state. The corresponding refinement of the final microstructure after phase transformation on the run-out table permits to employ leaner chemical compositions thus reducing the production costs and casting problems. Moreover, less alloyed compositions have a lower carbon equivalent which makes easier all the joining operations to build up the final component, such as conventional arc welding processes as well as hybrid techniques.
Among the most performing high strength grades, S700MC is largely employed since its high tensile properties are associated to a good toughness.
To achieve the grade it is necessary to obtain a bainite + ferrite or a fully bainitic microstructure together with a controlled level of alloying elements.
Ferrite-bainite steels have a high tensile strength and both an adequate elongation and excellent hole expansion performance which ensures good formability. Fully bainitic high strength steels can be employed in the automotive industry to produce structural components for passenger safety such as anti-intrusion frames [2, 3] as well as in applications where wear resistance is required, such as components of industrial vehicles and trucks.
There are two main routes for producing this grade as hot rolled strip. In the first one, the required level of hardenability is achieved by a proper alloying with Mo, Cr and Mn. In the second one the addition of boron is exploited to induce the necessary hardenability to the austenite and permits to use a lower amount of alloying elements.
In some cases additional thermal treatments are employed to correct or improve the mechanical properties of as-rolled strips, especially of large thickness, also in-line with the hot rolling mill [4]. However, only few studies of the stability of bainitic structures and the evolution of the mechanical properties with the treatment temperature are available in the literature for this grade [5, 6].
In this work, the response to different isothermal heat treatments of a fully bainitic 5 mm thin strip of S700MC steel is investigated and analyzed.
Although the behavior of S700MC to welding is generally good due to its relatively low carbon equivalent [7-11], this study provides additional information on joining of this high strength steel, especially regarding the optimization of the operating practices for post-weld heat treatments when they are required to reduce hardness peaks in the heat affected zone caused by the joint geometry or by a not adequate selection of the welding conditions.

MATERIAL AND METHODS

Slabs of the S700MC steel grade have been produced by electric arc furnace (EAF) route. The chemical composition of the steel, reported in Table 1, complies with the DIN EN 10149 2 reference. The fully bainitic microstructure of the as-hot rolled material is obtained without boron additions. After reheating at 1230±20°C, slabs are thermo-mechanically hot rolled from 220 mm to 5 mm thickness with a finish roughing temperature between 1050 and 1100°C, a finish rolling temperature of 860 ±20°C (the calculated Ae_3 temperature is about 825°C) and a coiling temperature of 450 ± 20°C.

Table 1. Chemical composition of the hot rolled strip (mass %) and tensile properties of the S700MC steel grade according to the DIN EN 10149-2 reference standard for a strip thickness between 3 and 8 mm.

C	Mn	Si	P	S	Cr	Mo
0.09	1.9	0.19	0.015	0.003	0.22	0.14
Nb	V	Ti	N	Ni	Cu	Al
0.069	0.04	0.10	0.006	0.11	0.2	0.036

Re_H (MPa)	Rm (MPa)	Elongation (%)
≥ 700	750 - 950	> 12

Some thermodynamic and kinetic calculations, performed by the *JMatPro®* [12] commercial software, are here reported to characterize the behavior of the S700MC steel grade with respect to phase transformations and precipitation of Nb and Ti carbonitrides. In this formulation of the S700MC grade titanium microalloying is used to control the austenite grain size during slab reheating in order to avoid abnormal grain growth and inhomogeneous starting microstructures in the slab that might impair the product quality [13]. The first aspect considered is the precipitation in austenite of carbonitrides of the microalloying elements Nb and Ti as a function of temperature, with particular reference to the interval from 1300 to 850°C where the hot rolling is carried out. Although carbonitrides are crystallographically isomorphous and mutually miscible, two distinct phases have been considered. They have been schematically denoted as (Ti,Nb)(C,N) and (Ti,Nb)C to mean the high- and low-temperature stable phases, the former being rich in Ti and N, the latter in Nb and C. Typically, the first one is representative of the coarse precipitation (about 1 µm average size) produced from casting down to slab reheating. Its calculated composition is $(Ti_{0.95},Nb_{0.05})(C_{0.7},N_{0.3})$. The second one, finer, is typical of the hot deformation hand its calculated composition is $(Ti_{0.55},Nb_{0.45})C$. The evolution of their mole fraction with temperature is shown in Figure 1. (Ti,Nb)(C,N) starts forming at about 1500°C whereas the precipitation start of the Nb-rich phase occurs at a temperature close to the roughing of the slab (about 1100°C) and increases as temperature decreases.

As far as the austenite decomposition is concerned, a continuous cooling transformation (CCT) diagram has been calculated from the finishing rolling temperature (850°C) assuming an austenite grain size of 10 µm. The curves in Figure 2 refer to a transformed fraction of 5%. It can be observed that the critical cooling rate for avoiding the ferrite nose is about 40°C/s in the range 650-550°C and it can be easily achieved on the run-out table of a hot strip mill for a 5 mm thin strip. Coiling at 450°C prevents the formation of martensite, being the Ms temperature for this steel about 425°C and the bainite start Bs about 590°C.

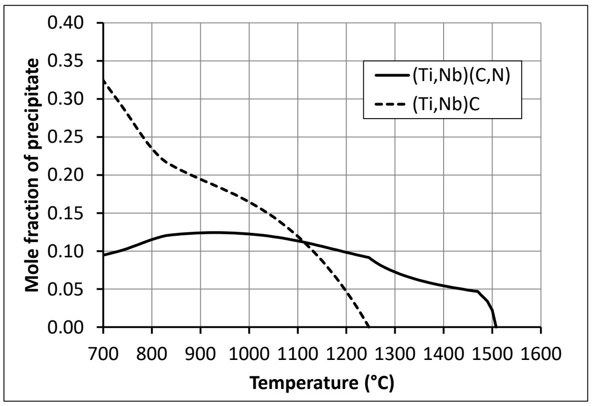


Fig. 1 Calculated equilibrium mole fraction of Nb and Ti carbonitrides in the S700MC steel as a function of temperature.

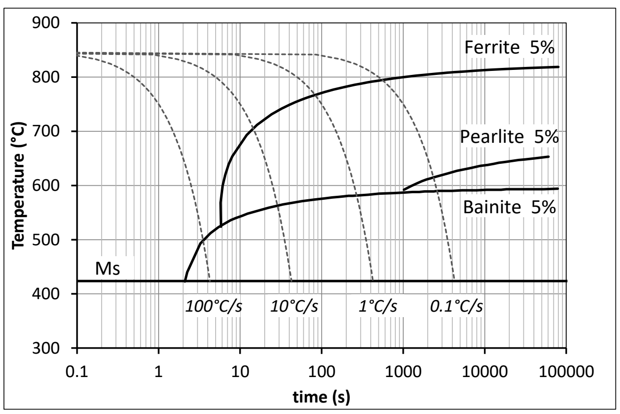


Fig. 2 Calculated CCT diagram of the S700MC steel austenitized at 850°C with an average austenite grain size of 10 µm. Lines refer to 5% transformed volume.

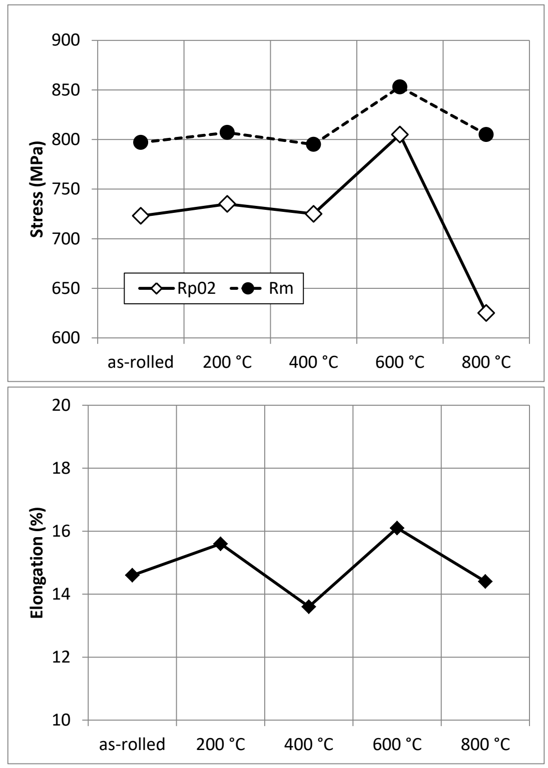
Specimens for tensile, impact and bending tests and for microstructural examinations have been taken from the coil tail. Isothermal heat treatments have been carried out in a laboratory furnace for 1 hour at 200, 400, 600 and 800°C followed by free cooling in air. The as-rolled material has been used as reference. Tensile tests have been carried out on a Zwick Z600 device with 600 kN maximum load capacity. Charpy impact tests have been performed by a Zwick RKP-450 device with 450 J maximum impact force. Bending tests have been performed by a DYZ-200 device.

Microstructural examinations of the samples have been carried out by a Nikon MA200 optical microscope with Clemex Image Analyzer System and by a Field Emission Gun Scanning Electron Microscope (FEG-SEM) Zeiss Sigma 300 equipped with Energy Dispersive X-ray Spectrometry (EDS) for microanalysis.

RESULTS

Tensile tests

Tensile tests have been carried out on flat test specimens according to the ASTM E8 reference standard (gauge length 50 mm). The results of tensile tests in the longitudinal direction are reported in Figure 3a. No relevant changes compared to the as-hot rolled condition are observed up to a holding temperature of 400°C. In this range the material still meets the standard requirements for the S700MC grade. A sharp increase in yield and tensile strength is observed for the holding temperature of 600°C where the highest values are obtained with an increase with respect to the as-rolled condition of about 80 MPa and 55 MPa, respectively. The sample treated at 800°C shows the lowest yield strength and a tensile strength substantially equivalent to the as-rolled reference state. Total elongation appears not affected by the treatments and the observed fluctuations can be considered within the scatter of the experimental determinations (Figure 3b).



a)

b)

Fig. 3 Results of tensile test in longitudinal direction for the S700MC steel in the as-rolled condition and after isothermal heat treatment of 1 hour at different temperatures. a) Rp02 and Rm; b) Total Elongation.

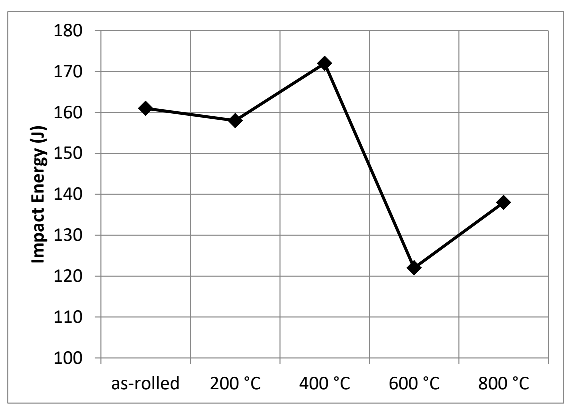


Fig. 4 Impact energy of the S700MC steel in the as-rolled condition and after isothermal heat treatment of 1 hour at different temperatures.

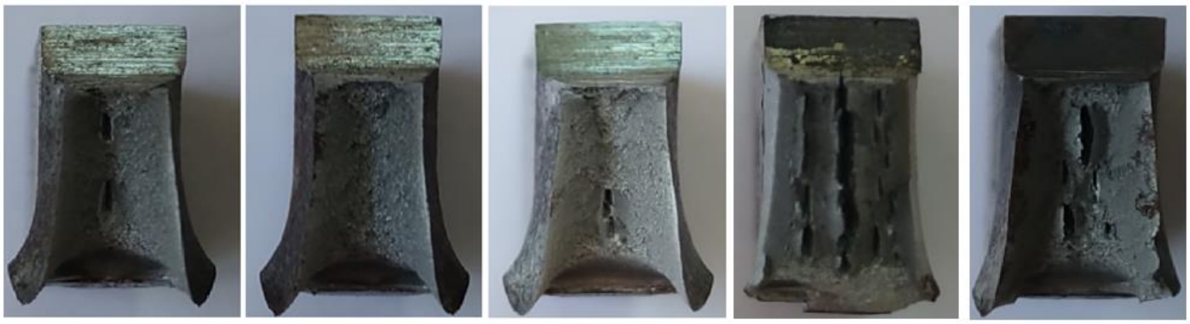
Impact and bending tests

Charpy V-notch impact tests have been carried out at room temperature on samples taken in the longitudinal direction. The test results are shown in

Figure 4. Up to the holding temperature of 400°C the impact energy is in the range of 155–175 J. A drop is observed for the treatment at 600°C. A further increase in the holding temperature to 800°C produces a partial recovery of the toughness level.
Fracture surfaces after the impact test are shown in Figure 5. Ductile fracture is the dominant mechanism for all the samples. Delaminations are particularly evident in the sample treated at 600°C.
Finally, bending tests have been carried out on samples in the transverse direction. The images of bent specimens are shown in Figure 6. In all cases the surfaces are defect-free.

Microstructure

Metallographic sections in transverse direction observed by optical microscope after etching with a Nital 2% solution are reported in Figure 7.
The microstructure of the sample treated at 200°C is fully bainitic and very similar to that of the as-rolled material. For a treatment at 400°C only small changes are observed. Instead, by increasing the holding temperature at 600°C the rolling structure undergoes an apparent rearrangement due to recovery with the partial recrystallization of bainite. In fact, ferrite grains appear slightly coarser and more equiaxed than those in the samples treated at lower temperatures. In addition, the reduction of the carbon supersaturation in ferrite promotes the observed coarsening of grain boundary carbides. Finally, the microstructure of the specimen treated in the intercritical region is reverted to a ferrite-pearlite mixture. Large (Ti,Nb)(C,N) precipitates formed at high temperature with size in the range 3 to 5 μm are evidenced in the pictures.



as-rolled 200°C 400°C 600°C 800°C

Fig. 5 Appearance of the fracture surfaces of S700MC steel grade as-rolled and isothermal heat treated at different temperatures after impact tests.

as-rolled 200°C 400°C 600°C 800°C

Fig. 6 Appearance of the bent surfaces of the S700MC steel grade as-rolled and isothermal heat treated at different temperatures after the bending tests.

SEM and microanalytical EDS investigations have been carried out on all samples. For the sake of clarity, only the results of the specimens in the as-rolled condition and after 1 hour at 600°C are reported and discussed. In-Lens and secondary electron images of the selected samples are shown in Figure 8.
The bainitic structure of the as-rolled material is characterized by a relatively small grain size with a sub-grain structure (with average size of about 1.5 μm) and many inter-lath cementite particles. The material treated at 600°C has a larger and slightly more equiaxed structure of the ferrite grains with less sub-grains and cementite particles located at grain boundaries.
In both cases the smallest precipitates detected have an average size of about 200 nm and have been identified as (Ti,Nb)C. Being already present in the as-hot rolled state, they have been formed during hot rolling of the strip and probably during roughing. Some examples are reported in Figure 9. No precipitates containing Mo or V have been found.
SEM images of the fracture surface of the selected impact test samples are shown in Figure 10. The as-rolled material is characterized by a ductile fracture. Dimples are associated to precipitates and inclusions. The sample treated at 600°C has a mixed fracture mode. Although the fracture surface is generally ductile, locally some crystalline regions can be found.

DISCUSSION

Microstructural changes

From the characterization of the mechanical properties and microstructures of the S700MC steel grade it is apparent that a treatment temperature of 600°C is critical since it induces a reduction of toughness (Figure 4) although it is associated with an increase in YS and, at a lower extent, also of UTS (Figure 3).
The microstructural changes can be explained invoking an intense recovery of the bainite structure with a reduction of the dislocation density, coarsening of inter-lath carbides and a rearrangement of the high-angle grain boundaries with a slight increase of the average ferrite grain size. In addition, the sub-grain structure is almost completely eliminated. These factors support the decrease of the impact energy.
On the other hand, notwithstanding the slightly coarser grain size, tensile properties, and in particular the yield stress, increase. Since from the SEM investigation no apparent new precipitation or coarsening of (Ti,Nb)C particles in the size range below 100 to 200 nm is observed, it has to be argued that nanoprecipitation of (Ti,Nb)C (not detectable by the SEM) is produced in the matrix. As a matter of fact, as indicated by the thermodynamic calculations, in the as-rolled material, some niobium and titanium are still in solid solution together with some carbon due to the low coiling temperature of the hot strip and

to the formation of a bainitic microstructure composed by carbon-supersaturated bainitic ferrite laths.

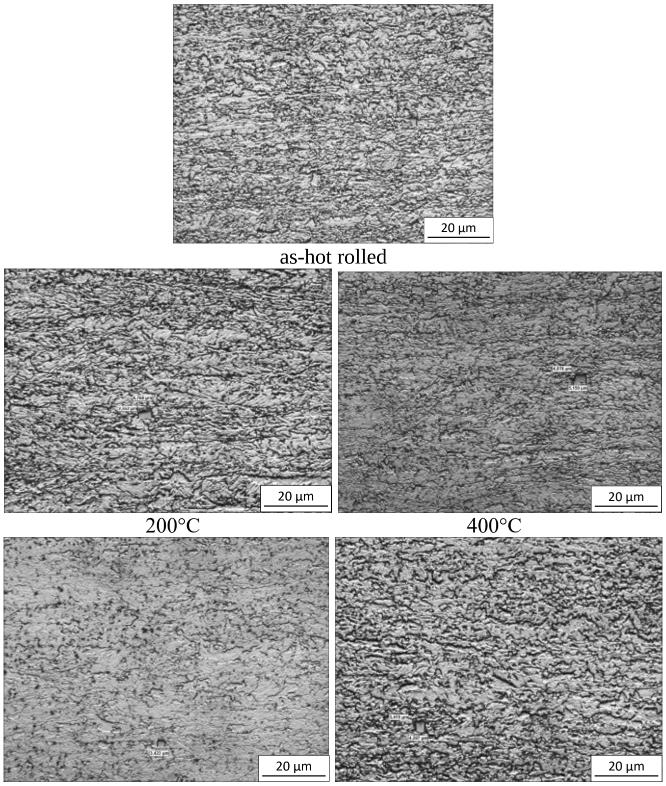


Fig. 7 Microstructure of the as-rolled and isothermal heat-treated S700MC steel (2% Nital etching, transverse direction).

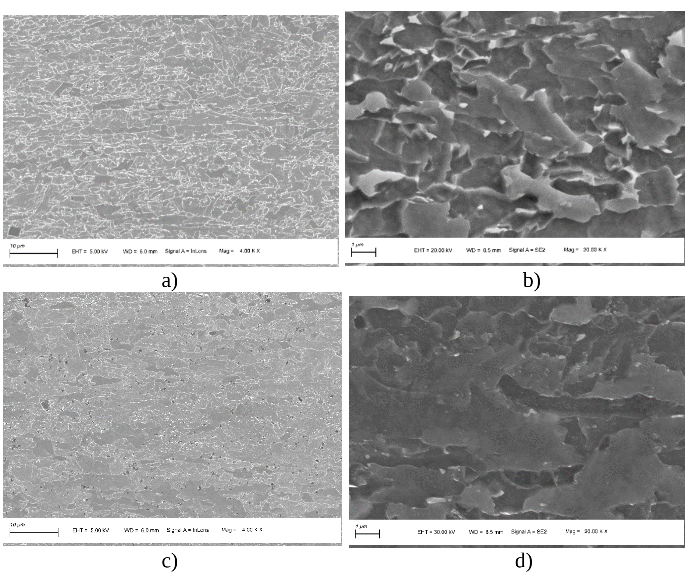


Fig. 8 In-lens SEM images of as hot rolled (a, b) and isothermally heat treated at 600°C for 1 hour (c, d) S700MC steel samples.

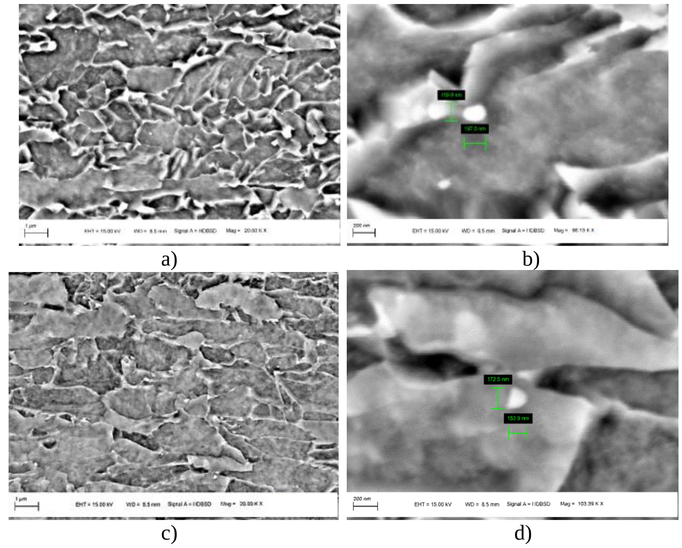


Fig. 9 Microstructure of S700MC steel (backscattered electrons) and details of typical (Ti,Nb)C precipitates in the as hot rolled state (a, b) and after isothermal heat treatment at 600°C for 1 hour (c, d).

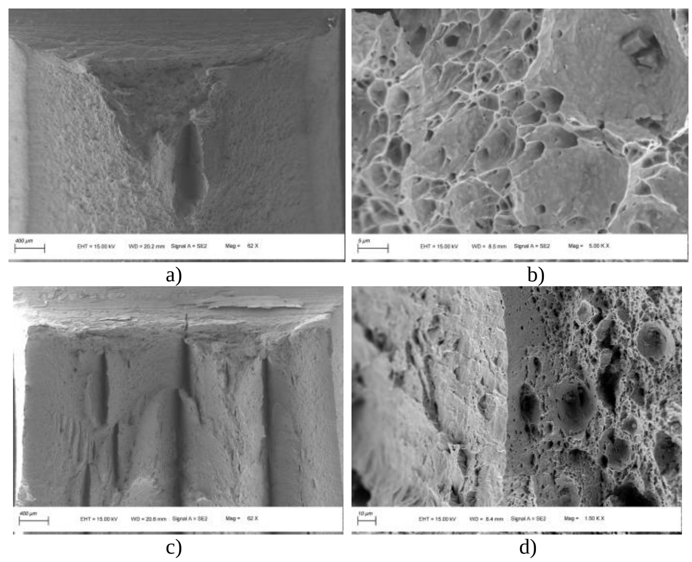


Fig. 10 SEM images of the fracture surface of selected impact test samples of S700MC steel strip: (a, b) as hot rolled; (c, d); isothermally heat treated 1 hour at 600°C.

In order to ascertain if this mechanism can be effective in explaining the above results, some calculations have been carried out using the commercial software *MatCalc* [14-19] which is able to simulate not only the thermodynamic equilibria but also the kinetics of nucleation and growth of precipitates in multicomponent systems. Calculations have been performed with the version 5.42 of the software using the databases mc_fe_v2.017.tdb for thermodynamic data and dmc_fe_v2.001.ddb for diffusion and mobility data.

Isothermal treatments of 1 hour at 400 and 600°C have been simulated considering the nucleation of (Ti,Nb)C particles on the grain boundaries of a bainitic ferrite matrix. In order to estimate the effect of hot rolling and coiling in determining the maximum amount of second phase that can be formed, the thermodynamic equilibrium composition of austenite at the roughing temperature (taken for the sake of simplicity equal to 1100°C) has been assumed as initial condition. At this stage, most of nitrogen is precipitated in coarse (Ti,Nb)(C,N) particles and the corresponding elements involved do not take part any more to

the subsequent precipitation processes. Furthermore, it has been reasonably assumed that a negligible precipitation occurs after roughing due to the short time of the hot rolling finishing treatment and to the low coiling temperature (about 450°C). By assuming an average grain size of the bainitic ferrite plate of 2 μm with a grain elongation factor of 2 and a dislocation density of 10^{12} m^{-2}, the calculation yielded a precipitate volume fraction of about $7{\cdot}10^{-5}$ and an average particle size of about 10 nm. After 1 hour of treatment the amount of Nb in solution is reduced of about 50% with respect to the initial condition and that of Ti of about 30%. Moreover, the calculations confirm that the same treatment at 400°C is not sufficient to activate the nucleation process, as observed in the experiments.
In order to estimate the strengthening effect produced by the fine precipitates after 1 hour at 600°C, the Ashby-Orowan equation [20] has been used

$$\Delta\sigma_{Y\,ppt}\,[MPa] = 10.8 \cdot \frac{\sqrt{f_V}}{d} \cdot ln\left(\frac{d}{6.25 \cdot 10^{-4}}\right) , \quad (1.)$$

where d is the average particle size (μm) and f_V the volume fraction of second phase. The *MatCalc* simulation results at 600°C permit to estimate a precipitation strengthening between 25 and 30 MPa, corresponding to about 50% of that measured at this temperature (Figure 3a). The remaining contributions, not explained by the present simulations, come presumably from precipitation of the supersaturated carbon as epsilon carbide in the bainitic ferrite. Also an ageing mechanism could be invoked which could explain the more pronounced increase of YS compared to UTS.
It has to be noticed that, although vanadium has been included in the kinetic calculations since, being miscible in the Ti-Nb carbonitrides, it can contribute to the precipitate volume fraction, nevertheless it does not enter the particles due to the too short annealing time not permitting a complete precipitation of solutes.
Annealing temperatures greater or equal to 800°C, implying a partial or full transformation in austenite (the calculated Ae_3 temperature for the present steel composition is about 825°C), annihilate the bainitic structure and dissolve all cementite precipitates (Figure 7). Even (Ti,Nb)C particles can be dissolved if temperature is high enough. Thus, after the slow cooling to room temperature, the steel recovers a structure composed of a mixture of ferrite and pearlite which is characterized by a much lower yield stress than the as-rolled material (Figure 3a). It has to be remarked that, unlike the treatments investigated by Sas *et al.* [21], in the present case there is neither hot deformation in the two-phase region nor a fast cooling. Consequently, the mechanical properties are not enhanced.
The above results permit to conclude that, due to the high level of residual supersaturation of the bainitic microstructure of the S700MC steel in the as-rolled state with respect to the precipitation of carbides of iron and microalloying elements, any thermal treatment carried out at a temperature high enough to activate the diffusion of interstitial elements has a relevant impact on the mechanical properties. In particular, a tempering temperature higher or equal to 600°C is particularly unfavorable since it induce a relevant secondary hardening effect and a reduction of toughness.

Welding issues

The present results can be exploited in relation to welding of the S700MC steel grade. For conventional welding techniques such as MAG, the commonly adopted procedures prescribe the use of low heat inputs, generally not exceeding 0.8 kJ/mm and typically around 0.4 kJ/mm [7-11] in order to prevent an excessive hardness in the heat affected zone (HAZ). Some welding simulations have been carried out by the CSMWeld model [22-25], a physical-semiempirical model developed at RINA-CSM which predicts the hardness in the HAZ of multi-pass joints. Starting from the imposed welding conditions (number of passes, heat input and chemical composition of the base material) it makes use of different sub-models for the simulating the thermal evolution at each pass, the austenite grain coarsening in the HAZ and the austenite decomposition during cooling as a function of the distance from the fusion line.
The model has been used to estimate the possible formation of hardness peaks in the HAZ in case of standard and not-standard welding conditions. Calculations have indicated that, with an heat input of 0.8 kJ/mm, the maximum hardness expected in HAZ is about 330±20 HV_{10}, to be compared with a hardness in the base metal of about 280 HV_{10}. An example of a hardness profile calculated for a bead-on-plate weld is reported in Figure 11. Consequently, no specific weldability problems are expected for this steel grade, in accordance with the EN 15614-1 standard which, for this class of materials (steel group 2.2 according to ISO/TR 15608) sets 380 HV_{10} as upper limit of untreated HAZ.
Nevertheless, especially if the joint geometry is complex and in association with higher strip thickness, welding issues can arise. In these specific cases, a post-weld heat treatment might be necessary to reduce the hardness peaks. Consequently, it is important that the thermal treatments are performed below 600°C in order to avoid loss of toughness in the base metal close to the HAZ.

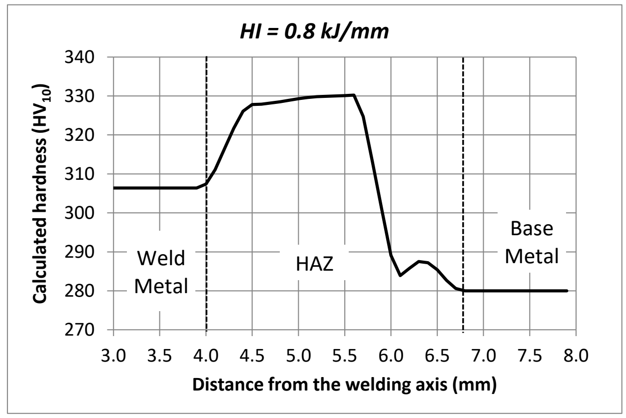


Fig. 11 Calculated hardness profile in HAZ for a bead-on-plate weld of S700MC with a heat input of 0.8 kJ/mm.

CONCLUSION

This study on the behavior of the bainitic HS steel grade S700MC subjected to isothermal treatments of 1 hour at temperatures in the range 200-800°C has shown that temperatures greater or equal to 600°C, although improving the tensile properties of the material, produce a decrease of toughness, thus confirming previous results in the literature. It is here proposed that this hardening effect arises from the additional precipitation of fine particles of (Nb,Ti)C and epsilon carbides and ageing, thus indicating that the bainitic structure is unstable above 600°C due to its inherent high level of supersaturation with respect to C, Nb and Ti. In case of application of post-weld heat treatments of joints involving this steel grade, the above temperature should not be exceeded.

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