

## **STRUCTURE AND MECHANICAL PROPERTIES OF HIGH-Mn TWIP STEEL AFTER THEIR THERMO-MECHANICAL AND HEAT TREATMENTS**

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### **Abstract**

The aim of this paper is to determine the impact of heat treatment of steel pre-treated thermo-mechanically using Gleeble 3800 simulator on the structure and mechanical properties of investigated high-manganese austenite Fe-Mn-(Al, Si) steel, containing 25% of Mn, 3% of Si, and 3% of Al. Applying thermo-mechanical treatment using Gleeble 3800 simulator allow to obtain the conditions for a gradual grain refinement controlled mainly by dynamic recrystallization and also by a dynamic recovery in a whole temperature deformation range. The high-manganese TWIP steel after hot-deformation is characterized by a mixture of fine, recrystallized grains and some fraction of dynamically recovered grains with a mean diameter of about 5-7µm. Solution heat treatment of the specimens from a temperature 850°C cause that steel possesses fine-grained microstructure of austenite with grain sizes about 10µm. Increasing solution temperature from 850 to 1000°C results in a rapid grain growth up to about 17µm. Specimens were annealing at time 900s, increasing solution time also cause a growth of austenite mean grain size in high-manganese Fe-25Mn-3Si-3Al TWIP steel. The new developed high-manganese TWIP steels provide an extensive potential for automotive industries through exhibiting the twinning induced plasticity (TWIP) and transformation induced plasticity (TRIP) mechanisms. TWIP steels not only show excellent strength, but also have excellent formability due to twinning, thereby leading to unique combination of strength, ductility, and formability over conventional dual phase steels or transformation induced plasticity TRIP steels.

**Keywords:** high manganese steel, TWIP mechanism, Gleeble simulations, mechanical properties, structure.

### **1. INTRODUCTION**

The dynamic development of technologies, observed in recent years, creates an increased demand for innovative engineering materials with very high functional properties, which will be attractive both economically and ecologically. Steel and iron alloys meet these criteria very well, but an increasing development direction is also revealed in the case of light metal alloys. While recently relatively high interest was associated with aluminum alloys, now more and more hopes are connected to the usage of thin steel plates and other structural elements of small thickness with very high properties. The growing demands of car manufacturers concerning the currently used steels, affect continuing efforts on development of new steel grades that will combine high strength with excellent ductility and thus the steels will be characterized by a high energy absorption capacity in the case of a collision of road vehicles. One of the latest group of steel used in the automotive industry, created with the aim of meet these requirements, it is the high-manganese steel of the austenitic type TWIP (Twinning Induced Plasticity). The TWIP steel due to its homogeneous structure, without inclusions, which creates possible sources of cracking, has a very high of strength to ductility ratio.

Extensive researches over this group of high manganese austenitic steels have been pursued in the recent years for this reason. Such steels contain between 20 to 30% of manganese, and 1-3% of aluminium and 1-

3% of silicone, introduced primarily to lower the density of about 7.3g/cm<sup>3</sup> for this group of steels. A profitable array of mechanical properties achieved by such steels, i.e.  $R_m \sim 800-1100$  MPa,  $R_{p0.2} = 250-550$  MPa,  $\varepsilon_{un} = 35-90\%$  is completely dependent on chemical composition, and mainly a concentration of Mn. The purpose of Si and Al consists of solid solution hardening of steel while carbon is an element stabilising austenite [1-5]. The thermo-mechanical treatment applied to refine the microstructure of austenitic TWIP type steels used by car companies has to be done in controlled conditions. Applying high deformation, or very long isothermal holding times of specimens after the last strain can results in excessive grain refinement of austenite up to about 2  $\mu$ m. It has an influence on increasing strength properties in special increasing the  $R_{p0.2}$  by about 150-200 MPa and also tensile strength increase to 1100 MPa [2-14]. Whereas too large value for the average grain diameter of about 70 nm can improve plastic properties, especially elongations that can achieve value about 80-90% at relatively low strength properties. Therefore, the main objective of the application of thermoplastic deformation is the determination of parameters of the process to achieve optimal value for the average grain diameter [1-7, 13-19].

It is also possible to apply heat treatment prior to the thermo-plastic treatment consisting of isothermal holding of the TWIP steels at temperatures above the recrystallization temperature for this group of steels for a suitable time and therefore a controlled growth of the austenite grain size. In this study were performed investigations for the reason to determine the effect of temperature (1000°C, 950°C, 900°C and 850°C) and heat treatment time on the austenite grain size of the TWIP steels, previously thermo-mechanical treated using a Gleeble 3800 simulator. It was also examined the impact of particle size on the mechanical properties of the investigated steel after the thermo-mechanical treatment as well as after the heat treatment with various process parameters [15-25].

## 2. MATERIALS AND EXPERIMENTAL PROCEDURE

Tests were carried out on new developed high-manganese steel TWIP type steel - X13MnSiAlNbTi25-3-3. The chemical compositions of steel were given in Table 1. For the investigated melt, Nb and Ti microadditions were added in order to refine the structure and achieve precipitation hardening. Investigated steel is characterized by high metallurgical purity, associated with low concentrations of S and P contaminants and gases. Melt was modified with rare earth elements.

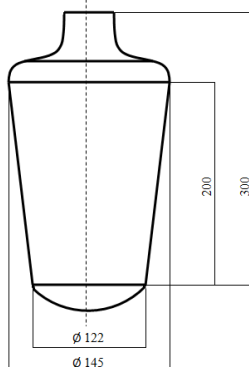
**Table 1** Chemical composition of new developed high-manganese TWIP-type steel, mass fraction

Steel designation	Chemical composition, mass fraction										
	C	Mn	Si	Al	Nb	Ti	P <sub>max</sub>	S <sub>max</sub>	Ce	La	Nd
X13MnSiAlNbTi25-3-3	0.13	25.1	3.5	3.3	0.050	0.018	<0.002	0.003	0.013	0.003	0.005

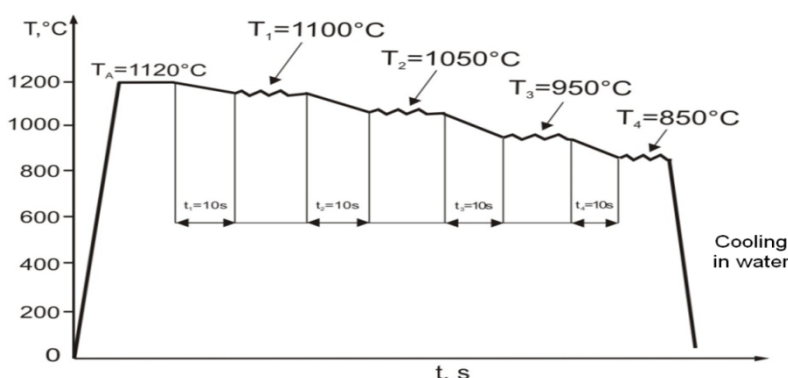
Figure 1 shows a view of the ingot with marked areas of the ingot top and bottom. After cutting out of the ingot top on the high of the shrinkage cavity pipe as well as the ingot basis on a height of about 3 cm from the bottom, the remainder part of the ingot was subjected to free forging at high speed Kawazoe manual hydraulic press with a pressure of 300 tonnes. Forging was carried out in a temperature range of 1200-900 °C. The ingot was forged to the flat dimensions of about 20x220 mm, from which in the next step the test samples were prepared in the form of parallelepipeds with dimensions of 20x15x35 mm. In order to obtain fine-grained structure the samples were subjected to hot plastic deformation using an universal thermo-mechanical Gleeble 3800 simulator as equipment of the Institute of Engineering Materials and Biomaterials, Silesian University of Technology, in Gliwice, Poland.

Thermo-mechanical treatment involves heating of the sample to a temperature of 1120 °C at a heating rate of 3 °C/s. At this point the sample was hold for 30 seconds, in order to homogenise the temperature in the entire volume of the sample, and then the temperature was lowered to 1100 °C and the first deformation of the sample was carried out. Subsequent deformation have followed at temperatures 1050, 950 and 850 °C. At each deformation, the specimens were deformed with a deformation degree of 30%. Scheme of the thermo-mechanical treatment, which is a projection of the hot rolling process, and scheme of the sample

shape in subsequent stages of the process are shown in Figures 2 and 3. Before performing the heat treatment, the sample were cut into smaller parts having a thickness of about 2 mm (Fig. 3e).

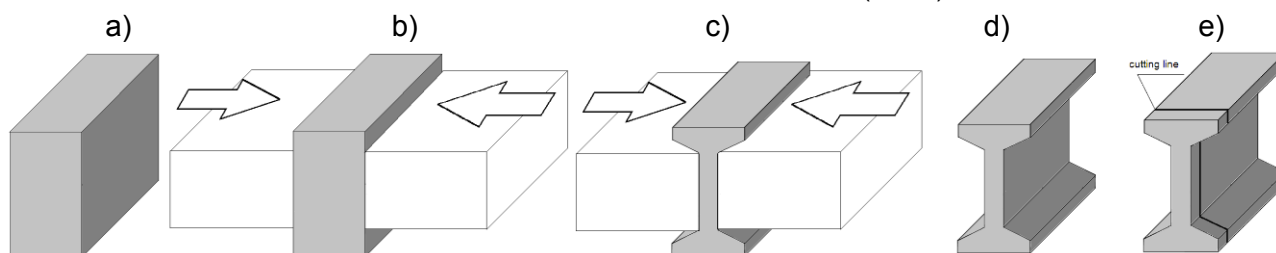


**Fig. 1** Ingot scheme of investigated steel



**Fig. 2** Scheme and parameters of the multi-stage compression test carried out on Gleeble 3800 simulator, as equipment of the Institute of Engineering Materials and Biomaterials, Silesian University of Technology, in Gliwice, Poland

The heat treatment of austenitic high-manganese steel samples was carried out in a Nabrathern company furnace, model HTCT 03/16. The samples were heated to a temperature of 1000 °C, 950 °C, 900 °C and 850 °C and then isothermally annealed at this temperature for 300s and 900s. After removing the samples from the furnace, the samples were quickly immersed in water for cooling at a temperature of about 18 °C. Static tests were performed using tensile testing machine Zwick/Roell Z2020 in order to investigate mechanical properties. Metallographic examinations were carried out using the Axio Observer.Z1m ZEISS light microscope with software AxioVision LE64. Qualitative X-ray analysis of the sample in the as-cast state, after thermo-plastic treatment and after static tensile test was performed using X-ray Philips XPert diffractometer, which was equipped with a copper anode lamp, emitting after filtration, the X-ray line of  $K\alpha_1$  with a wave length of  $\lambda=1,54056$  nm. The resulting diffraction patterns were analyzed based on the data contained in the database of the International Centre for Diffraction Data (ICDD).

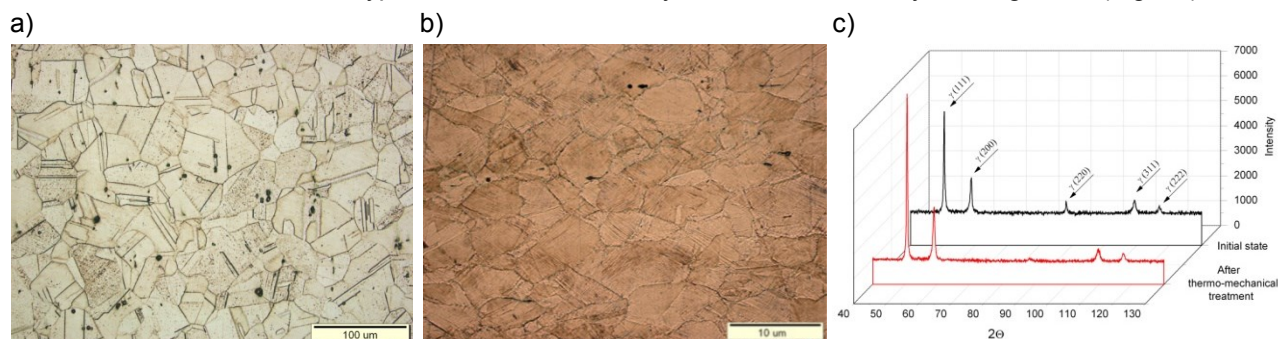


**Fig. 3** Change the sample shape during thermo-mechanical treatment on the simulator Gleeble 3800 simulator: a) the tested sample with dimensions of 20x15x35 mm; b) the shape of the sample prior to application of the loading force; c), d) the shape of the sample after thermo-mechanical treatment according to the scheme shown in Fig. 2; e) hot-deformed sample with a marked cutting line - sample for mechanical properties testing

### 3. RESULTS AND DISCUSSION

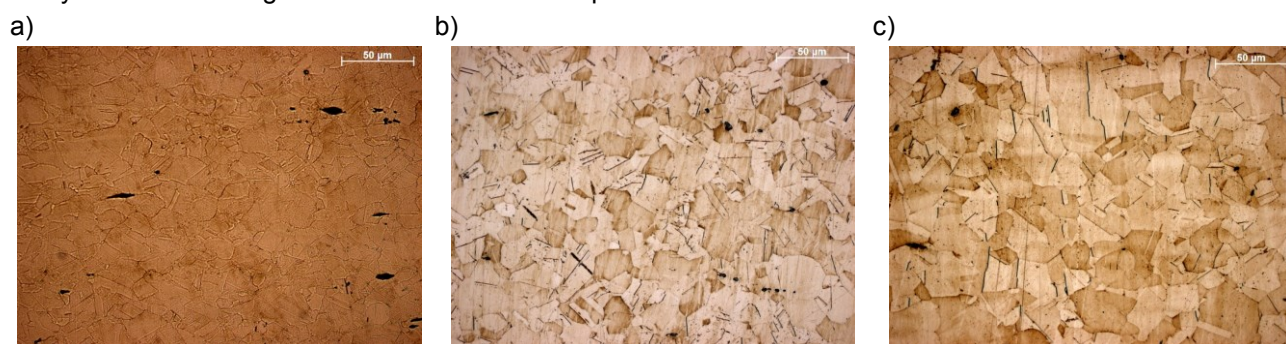
Investigated steel is characterized by the homogeneous microstructure of austenite with a mean grain size in the range from 50 to 60  $\mu\text{m}$ , in which numerous annealing twins can be identified (Fig. 4a). Single-phase microstructure of the steel in the initial state was confirmed by X-ray diffraction pattern (Fig. 4c). Application of thermo-mechanical treatment using Gleeble 3800 simulator allowed to decrease austenite mean grain size up to 5.9  $\mu\text{m}$  (Fig. 4b). True strain equal 0.36 during each compression stage creates excellent conditions for a gradual grain refinement of austenite in investigated steel controlled by the course of

dynamic recrystallization in a whole temperature deformation range. The use of both the heat treatment and thermo-mechanical treatment does not cause a phase change of the investigated high-manganese austenitic X13MnSiAlNbTi25-3-3 TWIP-type steel, as confirmed by the carried out X-ray investigations (Fig. 4c).



**Fig. 4** Austenitic structures of high manganese X13MnSiAlNbTi25-3-3 TWIP-type steel; a) in initial stage b) obtained after four-stage hot- compression test with a true strain equal  $4 \times 0.36$  and cooling in water after final deformation at temperature 850°C; c) X-ray diffraction pattern investigated steel after the initial stage and thermo-mechanical treatment

In the next step, the samples with a thickness of 2 mm were subjected to heat treatment, consisting of annealing at a temperature between 850 and 1000 °C, for 300 and 900 seconds. The application of this process was due to a small but significant for further tests increase of the mean austenite grain size. Figure 5 shows a representative structures of the steel at various stages of annealing. Based on the structure and image analysis it was found that the temperature, as well as the annealing time increase, causes an increase in the average diameter of the austenite grains to a value of 17 µm. Detailed results of the carried out image analysis of the investigated steel structures were presented in Table 2.



**Fig. 5** Austenitic structures of high manganese X13MnSiAlNbTi25-3-3 TWIP-type steel obtained after four-stage hot compression test followed by heat treatment at: a) temperature 850°C for 300s; b) temperature 950°C for 900s; c) temperature 1000°C for 900s

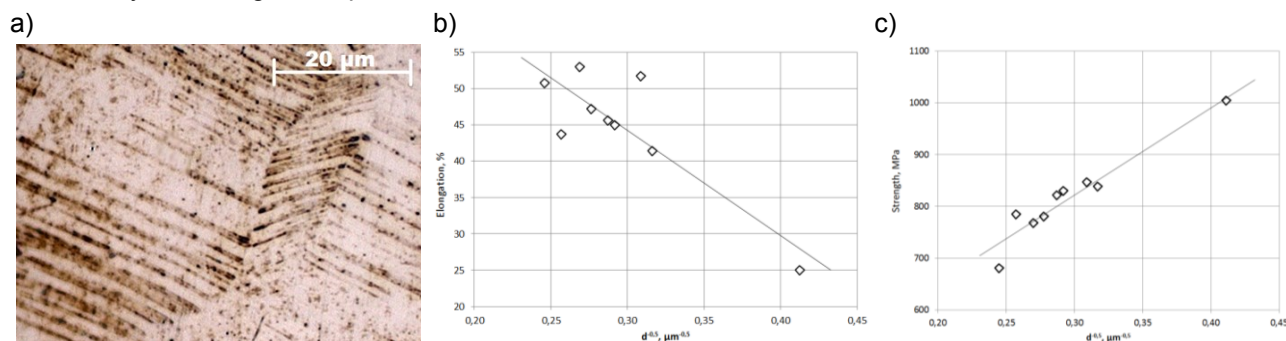
**Table 2** Influence of parameters of heat-treatment on austenite mean grain size of high-manganese X13MnSiAlNbTi25-3-3 TWIP-type steel

State of investigated steel	Austenite mean grain size, µm	
After thermo-mechanical treatment	5,9	
After thermo-mechanical treatment followed by heat treatment:	t = 300s	t = 900s
850°C	9,9	10,5
900°C	11,7	12,1
950°C	12,9	13,7
1000°C	15,1	16,6

In the next step of the investigation, static tensile tests were performed in order to investigate mechanical properties of high-manganese austenitic steels, with various austenitic mean grain size after their thermo-



mechanical and heat treatments. On figure 6a is presented representative austenitic structure with mechanical and micro twins obtained after four-stage hot compression test with a true strain equal  $4 \times 0.36$  followed by annealing at temperature 850 °C for 900s and after static tensile tests.



**Fig. 6** a) Mechanical and micro twins obtained after static tensile tests; b) Uniform Elongation (UE) and c) Ultimate Tensile Strength (UTS) vs. the inverse square root of grain size  $d^{-0.5}$

The dependence of ultimate tensile strength on the mean grain size of samples was examined by plotting the strength at yielding and instability strains against the inverse square root of grain size. Ultimate tensile strength increases with decreasing mean grain size of austenite according to Hall-Petch relationship as given on figure 6c. Additionally, the effect of mean grain size on uniform elongation of samples were shown in figure 6b. It can be noticed that in this condition, the Hall-Petch relationship is also useful for expressing the relation between the uniform elongation and mean grain size of austenite in investigated high-manganese TWIP-type steel.

## CONCLUSIONS

Heat treatment after prior thermo-mechanical treatment, consisting of an appropriate chosen parameters such as temperature (in the range of 850 to 1000 °C) and annealing time (300 and 900s) allows a controlled growth of the austenite grains and a change of the mechanical properties of austenitic high-manganese X13MnSiAlNbTi25-3-3 TWIP-type steel. The main mechanism of the structure shaping of the investigated steel during cold plastic deformation is mechanical twinning. Qualitative X-ray phase analysis revealed that the tested steel in both cases in initial state and after the heat and thermo-mechanical treatment, as well as after cold plastic deformation has a homogeneous austenite structure.

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