Archives of Materials Science and Engineering

Volume 59
Issue 1
January 2013
Pages 14-21

International Scientific Journal published monthly by the World Academy of Materials and Manufacturing Engineering

Microstructure evolution after thermo-mechanical treatment of X11MnSiAl25-1-3 TWIP-type steel

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Received 21.11.2012; published in revised form 01.01.2013

ABSTRACT

Purpose: The purpose of the article is to present the results of research of the effect of thermal and thermoplastic working on the structure of high-manganese austenitic TWIP steels.

Design/methodology/approach: Plastometric tests were performed with DSI (Dynamic System Inc.) Gleeble 3800 instrumentation being the equipment of the Scientific and Didactic Laboratory of Nanotechnology and Materials Technologies of the Institute of Engineering Materials and Biomaterials. Changes in the microstructure after individual stages of hot plastic deformation were determined on the basis of detailed microstructure tests with the light microscope and scanning electron microscope. An X-ray phase qualitative analysis of the examined materials in the condition after casting and after thermoplastic working was carried out with the XPert diffractometer by Philips.

Findings: It was concluded based on the tests performed that the structure of the examined austenitic high-manganese steel in the initial condition is represented by austenite with numerous annealing twins. The results obtained for investigations in a continuous compression test will enable to establish power and energy parameters and design a hot compression process, consisting of several phases, of axisymmetric specimens, simulating the final rolling passes.

Practical implications: By elaborating the detailed data concerning structural changes and power and energy parameters of the thermoplastic working process of the investigated high-manganese austenitic TWIP steel type, it will be possible to design appropriately the final passes of the hot rolling process to obtain an optimum size of grains, which will in turn influence the improved strength properties of the investigated high-manganese austenitic X11MnSiAl25-1-3 steel.

Originality/value: The application of thermoplastic working of high-manganese austenitic TWIP steel.

Keywords: Superplastic materials; TWIP-type steel; Hot-working; Dynamic recrystallization

Reference to this paper should be given in the following way:

A.D. Dobrzańska-Danikiewicz, W. Borek, J. Mazurkiewicz, A. Jagiełło, Microstructure evolution after thermo-mechanical treatment of X11MnSiAl25-1-3 TWIP-type steel, Archives of Materials Science and Engineering 59/1 (2013) 14-21.

MATERIALS

1. Introduction

Despite substantial technological progress in the motor industry, steel continues to enjoy the largest share among the materials for car construction (99% of undercarriages in passenger cars; parts made completely or partially of steel make up 60-70% of a car's weight) [1, 4-10]. The requirements set for cars, forced by the market and such organisations as EURO NCAP [2] or NHTSA [3] and legislation, e.g. European Emission Standard EURO 5, continue to become stricter as the automobile market is advancing. Automobile makers are striving for a reduction in fuel consumption, improved active and passive safety, reduced mass of vehicles, enhanced corrosion resistance, lowered emissions of environmentally harmful substances or a reduction in manufacturing costs. Passive safety of passengers mainly depends on the properties of materials used for car construction. Efforts are thus made to improve impact energy absorption with materials applied for side and front impacts and car overturning. Stacking Fault Energy (SFE) expressed as mJ/m^2 is used in metallic materials for determining a material's ability to absorb impact energy. The lower the factor the more energy is absorbed for the impact [11-15].

High manganese TWIP type (Twinning Induced Plasticity) austenitic steels is one of the groups of steels with a low stacking-fault energy value of 15–30 mJ/m^2. The steels are characterised by a high content of manganese ranging between 15 and 30% ensuring a homogenous austenitic structure at room temperature [1]. If manganese content is below 15%, steel exhibits an austenite and martensite structure reducing a material's plasticity. The fraction of manganese above 30-32% causes the precipitation of brittle phase β-Mn [4]. Approx. 3% of Al and approx. 3% of Si is also added most often, aiding to stabilise austenite at room temperature and during plastic cold deformation [16-20]. In addition, aluminium slightly lowers steel density, which has a favourable effect on a reduction in the mass of structural components made of such type of steels, therefore decreasing a vehicle's total mass. Such steels possess very good plastic properties as a result of an intense curve of twinning during cold plastic deformation (TWIP effect, Twinning Induced Plasticity). Mechanical properties of steel with manganese contents of approx. 25%, including especially tensile strength, is 600 MPa, while uniform elongation is about 80% [7]. TWIP steel with uniform elongation of approx. 35%, and tensile strength of 1400 MPa can be obtained with appropriate modification of chemical composition of TWP type steel and appropriately selected hot working and/or thermomechanical processing according to Arcelor Auto and Thyssen Krupp consortium [20-28].

2. Material and experimental procedure

The investigations were carried out for high manganese X11MnSiAl25-1-3 TWIP steel with chemical composition given in Tab. 1. The steel investigated has a very low concentration of S and P impurities and gases.

Melts of the tested steels with the weight of 25 kg were performed with a laboratory vacuum induction furnace VSG-50 by Balzers. The mass of feedstock for the furnace was 23.0 kg. The feedstock consisted of materials with a known chemical composition and with adequate physical form. The basic feedstock was ARMCO 04JA grade iron with the following chemical composition: 0.02% C, 0.009% P, 0.036% Al, 0.23% Mn, 0.006% S, 0.02% Ni, 0.02% Si, 0.02% Cu, 0.02% Cr. The required chemical composition of steel was supplemented with an addition of: carbon (graphite), pure silicon, electrolytic manganese and aluminium. Mischmetal (~50% Ce, ~20% La, ~20% Nd) was used for modifying non-metallic inclusions. The investigated steel was cast in the argon atmosphere into a cast-iron ingot mould with the dimensions as given in Fig. 1. After casting the melt, it took 60 minutes to wait for the ingot head to solidify, and then the furnace chamber was opened and the ingot was cooling down in air. The ingot was removed in the cold state after about 20 minutes following casting.

Preliminary hot plastic working of the ingot was performed with open die forging with a hydraulic press by Kawazoe with the pressure of 300 tonnes. The ingot was heated for forging in a gas forging furnace. Forging temperature ranged between 1200 and 900°C with reheating between operations so that the material was not cooling down to the temperature below 900°C. The ingot body without the head and bottom of the ingot was subjected to forging, cut about 3 cm high. 20x210 mm flat sections were obtained as a result of this process.

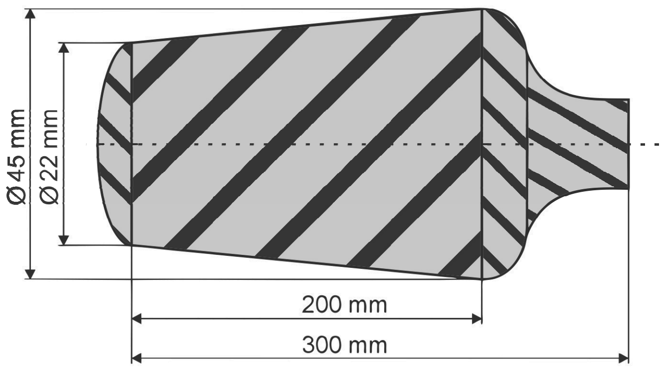

Fig. 1. Diagram of ingot obtained with sizes

By means of the cogged ingot, a set of axisymmetric specimens was made for plastometric tests and for tests performed with the thermomechanical processing simulator DSI Gleeble 3800 by DSI (Dynamic System Inc.) being the equipment of the Scientific and Didactic Laboratory of Nanotechnology and Materials Technologies, Institute of Engineering Materials and Biomaterials of the Silesian University of Technology. The shapes and sizes of the specimens are shown in Fig. 2.

The Gleeble 3800 system is equipped with a direct resistance heating system maintaining the set temperature with the accuracy of up to 1ºC. Precision controlling of specimens' temperature is enabled by a signal conveyed by thermoelements. The specimens are held by jaws made of a material with high heat conductivity, and a specimen can be cooled with high speed. The system equipped with a hydraulic mechanical system enables to apply pressure of 200 kN during pressing and to make tests with a deformation rate of 0.0001 to 200 s^{-1}. Linear variable differential transformers and tensiometric sensors for pressure measurement produce feedback securing the exact execution and repeatability of the set mechanical values of the defined technological process.

Very thin graphite and tantalum foils were inserted between the specimen surface and the die surface in order to reduce friction, and a nickel-based lubricant was applied onto the both surfaces.

The specimens were resistance heated in vacuum at a rate of 3°C/s to, respectively, 850, 950 or 1050°C at the first stage of the tests and then heated at such temperature for 30s to equalise the temperature and homogenise the tested steels' structure. Then, the specimens were hot-worked plastically with at a plastic working rate of $\dot{\varepsilon}$ =0.1, 1 and 10 s^{-1}. A hot pressing process, consisting of several phases, of axisymmetric specimens, simulating the final rolling passes, was designed in the next stage of plastometric tests.

The experiment was also carried out using the hermomechanical processing simulator Gleeble 3800. The degrees of deformation, plastic working speeds and intervals between the subsequent plastic working operations were selected to consider the conditions of the planned hot rolling of flat sections with the initial thickness of about 4-5 mm to obtain 2 mm thick specimens. Apart from the determination of power and energy parameters of hot plastic working, the specimens were supersaturated in water and air.

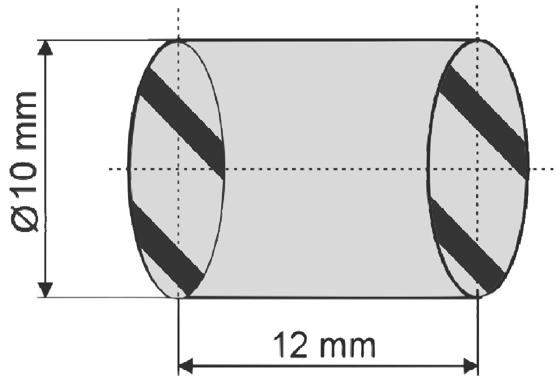

Fig. 2. Axisymmetric specimen diagram together with dimensions given, used for plastometric tests

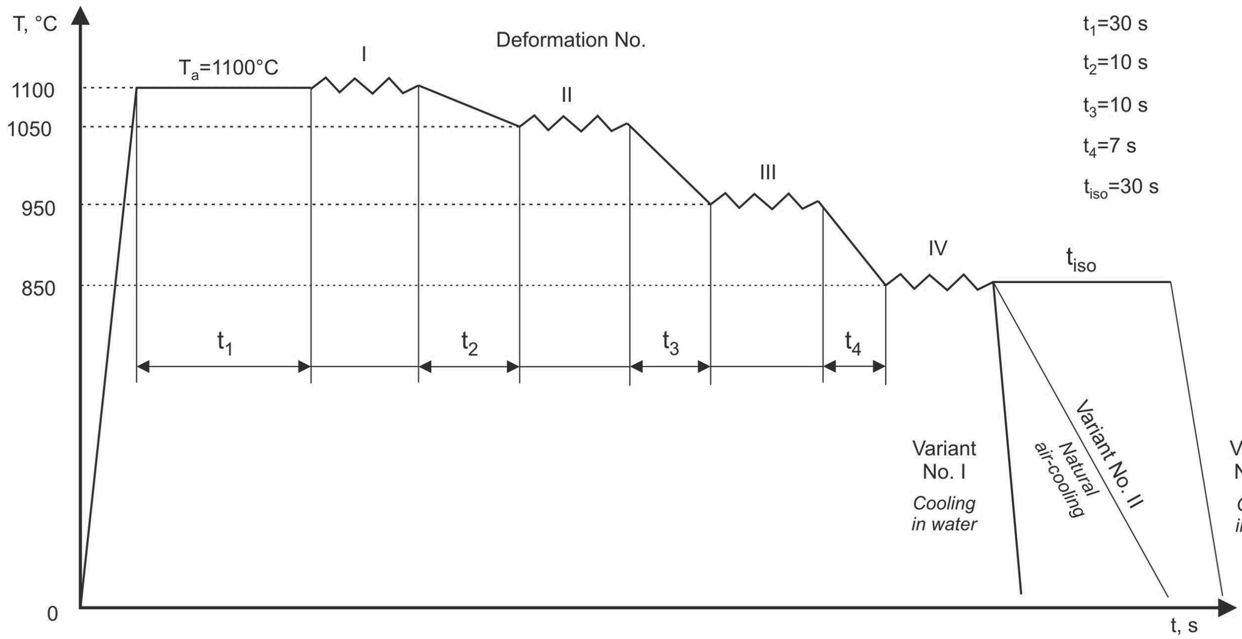

Fig. 3. Thermomechanical processing scheme for axisymmetric specimens made of X11MnSiAl25-1-3 TWIP type steel worked plastically using a Gleeble 3800 simulator

Table 1.
Chemical composition of the analysed steel

Steel grade	Mass concentration of elements, %												
	C	Mn	Si	Al	Nb	Ti	P_{max}	S_{max}	Ce	La	Nd	O ppm	N ppm
X11MnSiAl25-1-3	0.11	24.93	1.2	3.24	-	-	0.002	0.006	0.001	0.0005	<0.0005	5	58

Thermomechanical processing was started from steel austenising at 1150°C. The specimens were then deformed by pressing with a degree of plastic deformation of 4x0.23 (Fig. 3). Following the last plastic working at 850°C, some specimens were directly supersaturated in water, while others in air, and the remaining specimens were subjected to isothermal heating at the temperature of the last working of 850°C for 30 s and then supersaturated in water to freeze the examined steel's structure.

Metallographic tests were made with specimens made of X11MnSiAl25-1-3 steel embedded in thermosetting resins. The embedded specimens were ground with an automatic STRUERS grinder-polisher with sandpapers with the grain size of 220-1200 $\mu m/mm^2$. Another stage of specimens preparation was mechanical polishing using a diamond suspension with the grain size of 6 and 1 µm.

An etching reagent as a 10% nitric acid solution in ethanol was used to expose grain boundaries and deformation bands in the structure of high manganese austenitic steel specimens. Etching lasted 5÷10 seconds. The structural observations of the examined materials were carried with the Axio Observer Z1m light microscope by Zeiss with magnification of 200 to 1000×. An X-ray phase qualitative analysis of the examined material in the subsequent steps of the experiment was made with the XPert diffractometer by Philips, with the stepwise recording of 0.05° and counting time of 5 seconds, using filtered $K\alpha 1$ X-ray radiation, with the wavelength of λ=1.54056 nm coming from a lamp with a 40 kV copper tube with 30 mA filament current intensity. Measurements were made within the angle range of 2θ between 20 and 140°. The phases were identified using data from the International Centre for Diffraction Data (ICDD).

3. Results and discussion

Newly developed high manganese X11MnSiAl25-1-3 TWIP type steel exhibits a homogenous austenite structure with numerous annealing twins (Fig. 4). The austenite grain size in the tested steel is about 100-150 µm. A single-phase structure of the steel in the initial state is confirmed with the results of the X-ray phase analysis examinations presented in Fig. 5.

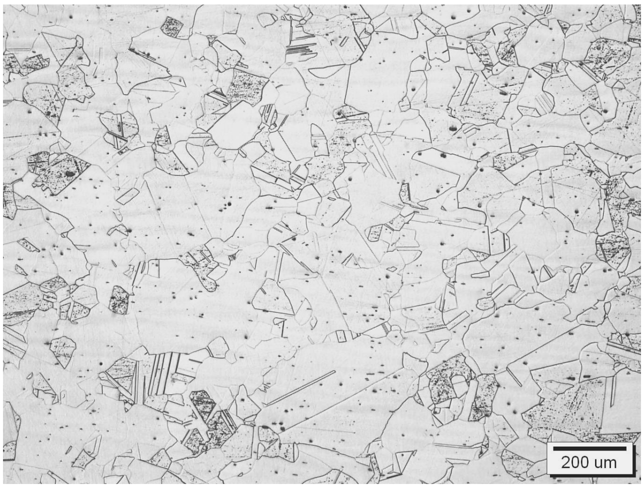

Fig. 4. Structure of X11MnSiAl25-1-3 steel in the initial state

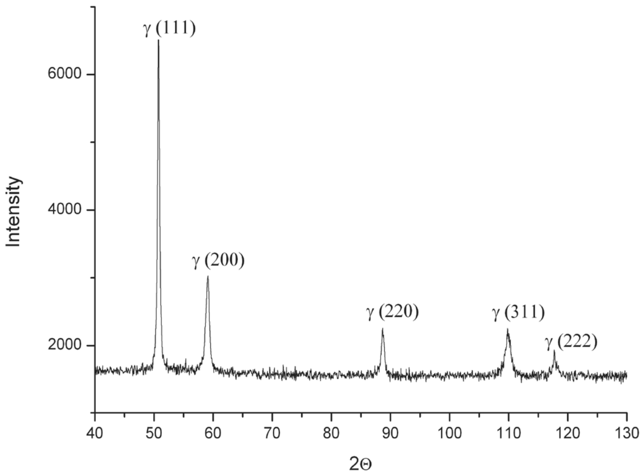

Fig. 5. X-ray diffraction pattern of the investigated X11MnSiAl25-1-3 steel in the initial state

Figures 6-8 present stress-strain curves obtained during a continuous pressing test for X11MnSiAl25-1-3 TWIP type steel and it was concluded on such basis that the range of yield stresses is within the range of 125 and 360 MPa. The values of yield stresses read from the diagrams and the corresponding deformation values are shown in Table 2. It was concluded based on the research results that a higher yield stress with a reduced pressing temperature or plastic deformation speed is accompanied by the shift of the maximum stress towards higher plastic deformation values. High yield stress values of 300-360 MPa arise from a strengthening effect of the concentration of main elements such as manganese (25%) and aluminium (3%). Increased plastic working speed at the constant deformation temperature increases a yield stress. A flow stress for large plastic

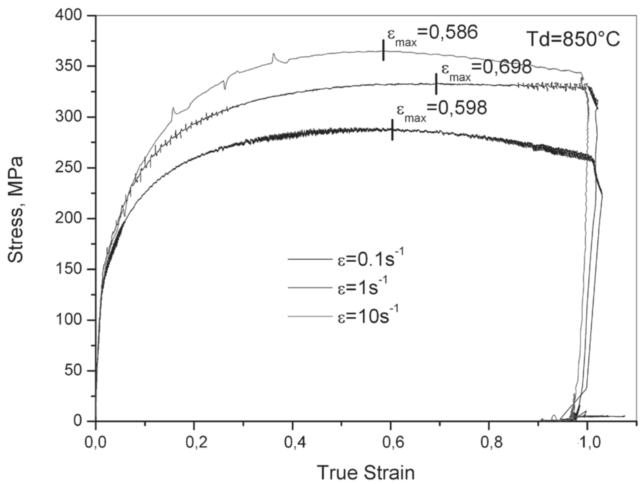

Fig. 6. Effect of deformation speed on the shape of stress-strain curves of X11MnSiAl25-1-3 steel after true deformation: ε=1, deformation temperature: 850°C

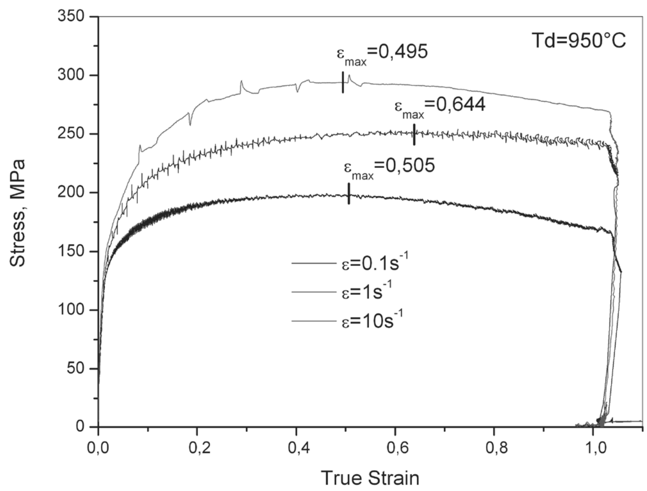

Fig. 7. Effect of deformation speed on the shape of stress-strain curves of X11MnSiAl25-1-3 steel after true deformation: ε=1, deformation temperature: 950°C

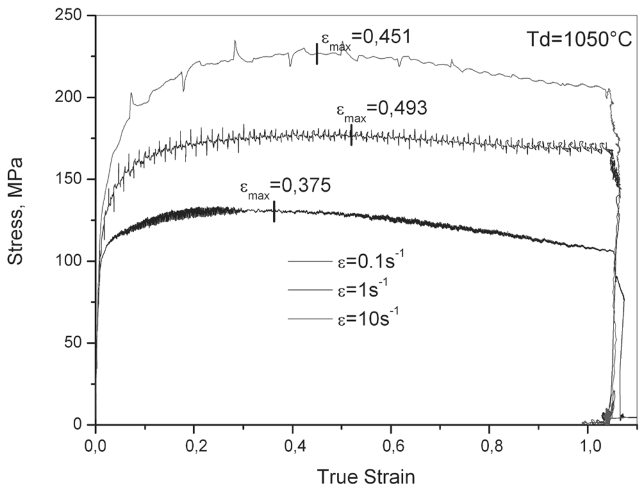

Fig. 8. Effect of deformation speed on the shape of stress-strain curves of X11MnSiAl25-1-3 steel after true deformation: ε=1, deformation temperature: 1050°C

deformations does not change almost, as a result of the state of equilibrium between strain hardening and dynamic processes eliminating the effects of deformation, i.e. dynamic recovery and/or dynamic recrystallisation.

The stress-strain curves of the examined high manganese austenitic TWIP type steel deformed plastically in accordance with the three variants of thermomechanical processing (rys. 3) assumed in the article are illustrated in Fig. 9. The individual thermomechanical processing variants differ only with a cooling method after the last stage of deformation at 850°C. The specimen tested in the variant I was cooled in water to maintain the structure of the examined steel. The specimen in variant II was cooled naturally in air, and in variant III, the specimen after the last deformation at 850°C was heated isothermally at the thermomechanical processing end temperature for 30 s, and then cooled in water to maintain the structure and analyse the effect of static or metadynamic recrystallisation progress on the refining of austenite grains in the investigated steel.

Table 2.
Effect of deformation temperature and rate on yield stress values of the examined high manganese X11MnSiAl25-1-3 steel

Deformation temperature	$\dot{\varepsilon}$, [s^{-1}]	ε_{max}	σ, [MPa]
850°C	0.1	0.586	280
	1	0.698	330
	10	0.598	365
950°C	0.1	0.495	195
	1	0.644	250
	10	0.505	290
1050°C	0.1	0.451	130
	1	0.493	175
	10	0.375	250

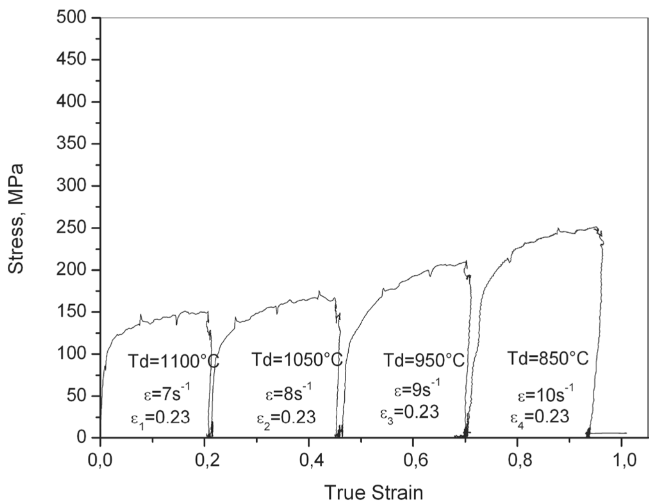

Fig. 9. Stress-strain curves for hot pressing in four stages with true deformation of 4x0.23 of axisymmetric specimens made of X11MnSiAl25-1-3 steel according to the thermomechanical processing scheme presented in Fig. 3

It can be concluded by analysing the shape of the stress-strain corves for X11MnSiAl25-1-3 TWIP steel that dynamic recovery and metadynamic or static recrystallisation occurring between the subsequent deformations are the main process removing the effects of strain hardening. Yield stress values after four-stage hot pressing of the axisymmetric specimens within the plastic deformation temperature ranging between 1050 and 950°C are higher by about 25-50 MPa as compared to the values obtained in continuous pressing tests at the rate of 10 s^{-1} (Fig. 9). The progress of dynamic recovery during hot plastic working is possible by using a true deformation of 0.23 in all the stages of pressing the axisymmetric specimens of the examined steels and of metadynamic recrystallisation in between relevant deformations as indicated by the lack of maximum stress values. A cyclic plastic deformation and the progress of partial metadynamic and static recrystallisation between the subsequent phases of plastic deformation result in reaching the maximum values of stress ε_{max} for lower values of deformation on the curve σ-ε for the last phase of plastic deformation as compared to the continuous pressing curve at 850°C.

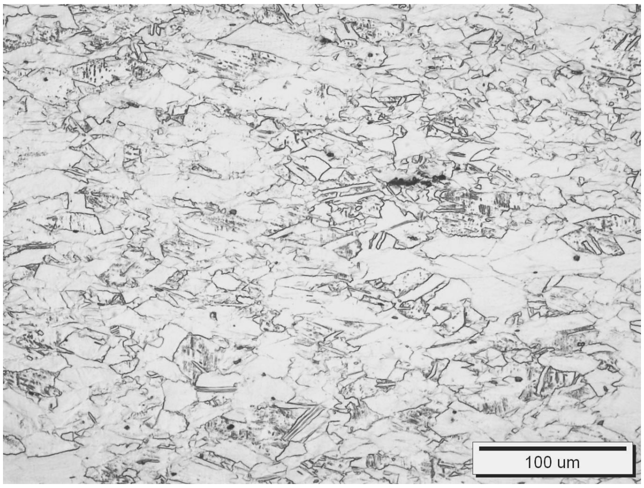

Fig. 10. Structure of X11MnSiAl25-1-3 TWIP type steel after four stages of hot pressing with the Gleeble 3800 simulator and after cooling in water after the last deformation at 850°C

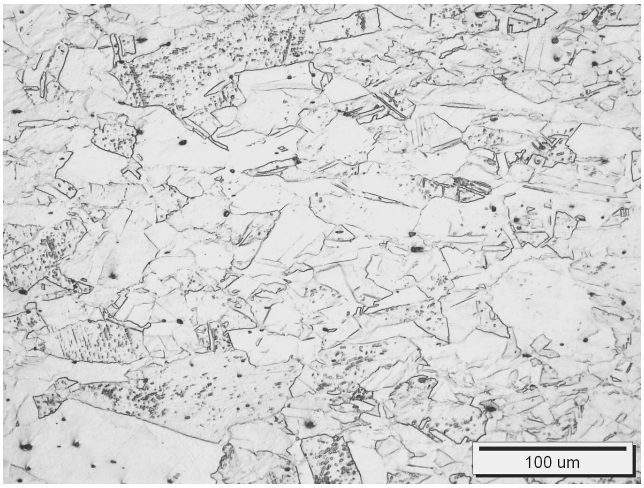

Fig. 11. Structure of X11MnSiAl25-1-3 TWIP type steel after four stages of hot pressing with the Gleeble 3800 simulator and after cooling in air after the last deformation at 850°C

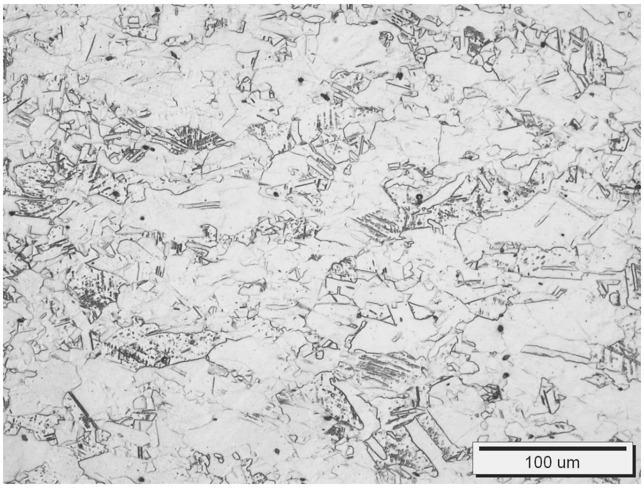

Fig. 12. Structure of X11MnSiAl25-1-3 TWIP type steel after four stages of hot pressing with the Gleeble 3800 simulator and isothermal heating at the temperature of final deformation of 850°C for 30 seconds, and then supersaturated in water

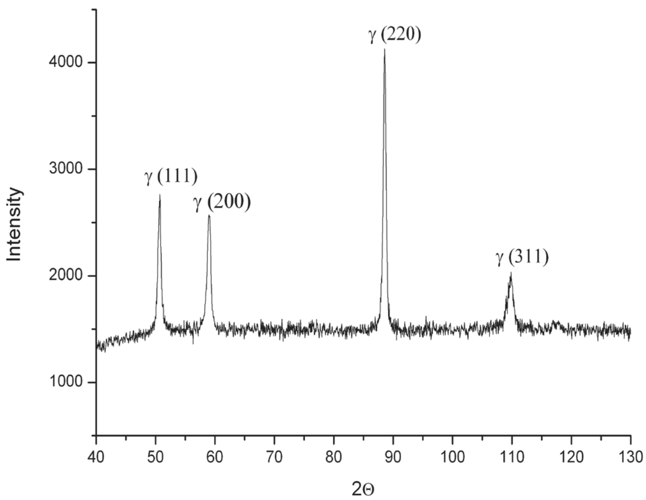

Fig. 13. X-ray diffraction pattern of X11MnSiAl25-1-3 steels after four stages of hot pressing with the Gleeble 3800 simulator and after cooling in water after the last deformation at 850°C

Figures 10-12 present the results of metallographic tests of the examined high manganese austenitic TWIP type X11MnSiAl25-1-3 steels performed with a light microscope. Dynamic recovery and static and metadynamic recrystallisation occurring between the subsequent deformations is the main process removing the effects of strain hardening. TWIP type steel deformed according to the variant I of thermomechanical processing presented in Fig. 3 features the average grain size of about 20-25 μm (rys. 10). By using natural cooling in air after the last deformation at the temperature of 850°C (according to the variant II of thermomechanical processing), once thermomechanical processing is completed, metadynamic recrystallisation is initiated causing growth in the grain size to the value of about 50-70 μm (Fig. 11). Due to isothermal heating at the temperature of the last deformation, i.e. 850°C, for 30 s, static and metadynamic recrystallisation is the main process removing the effects of strain hardening. The average size of austenite grains is 25-30 μm (Fig. 12).

Figures 13-15 present X-ray diffraction patterns of the examined high manganese austenitic X11MnSiAl25-1-3 TWIP type steels after thermomechanical processing with the Gleeble 3800 simulator. In the cast state, the investigated steel has a homogenous austenite structure (Fig. 5), and if different cooling variants are employed, hence different rates of cooling after thermomechanical processing, the phase composition remains unaltered.

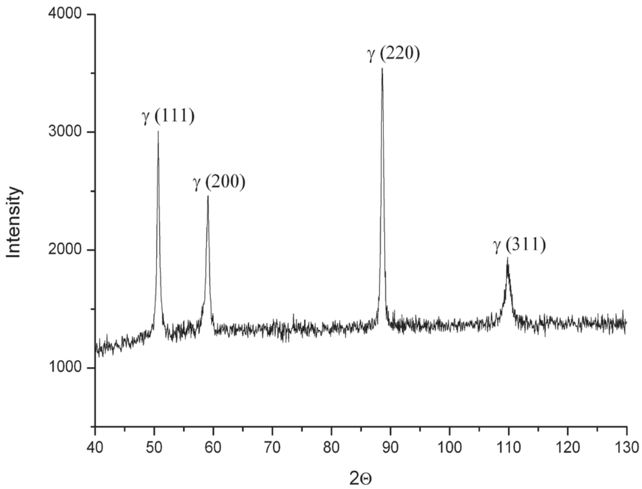

Fig. 14. X-ray diffraction pattern of X11MnSiAl25-1-3 steels after four stages of hot pressing with the Gleeble 3800 simulator and after cooling in air after the last deformation at 850°C

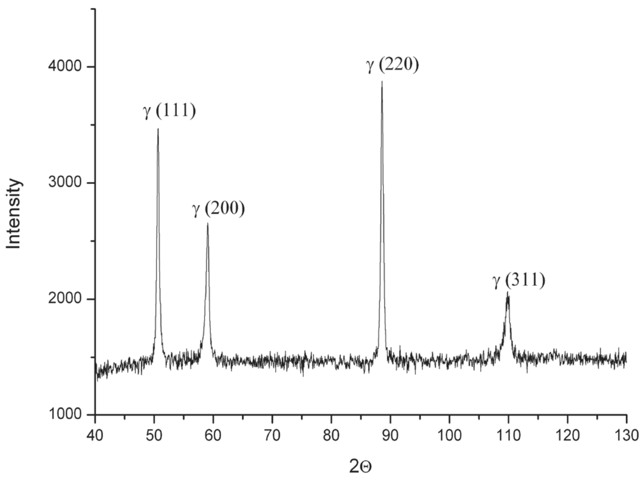

Fig. 15. X-ray diffraction pattern of X11MnSiAl25-1-3 steel after four stages of hot pressing with the Gleeble 3800 simulator and isothermal heating at the temperature of final deformation of 850°C for 30 seconds, and then supersaturated in water

4. Conclusions

It can be concluded according to the outcomes of the investigations in the initial condition and the outcomes obtained using the thermomechanical processing simulator Gleeble 3800, consisting of continuous pressing at the temperature of 1050 to 850°C at different hot working speed rates and after the hot pressing, in multiple stages, of axisymmetric specimens that the newly developed high manganese austenitic X11MnSiAl25-1-3 TWIP type steel has, in the initial condition, an austenitic structure with multiple annealing twins. Yield stress values within the range of 125-360 MPa were identified by determining stress-strain curves. A higher yield stress with a reduced pressing temperature or plastic deformation speed is accompanied by the shift of the maximum stress towards higher plastic deformation values. By applying the hot pressing of axisymmetric specimens in the Gleeble 3800 simulator with the deformation of 4x0.23 in four stages, dynamic recovery within the entire range of deformation and temperature is the process controlling the consequences of strain hardening. Steel supersaturated in water directly after deformation at temperature is characterised by a smaller size of austenite grains of about 20-25 μm. Further refinement of the microstructure can be obtained by isothermal holding of the specimens in a finishing hot-working temperature for 30 s. Repeated recrystallization and corresponding grain refinement causes that the thermo-mechanically processed steel is characterized by uniform structure.

Acknowledgements

Project was founded by the National Science Centre based on the decision number DEC-2012/05/B/ST8/00149.

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