Journal of Metals, Materials and Minerals. Vol. 14 No.1 pp. 1-9 , 2004.

STUDY ON TRANSFORMATION BEHAVIOUR OF BULK Si-Mn TRIP STEEL

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ABSTRACT

In the last decade, a lot of effort has been made to optimise the thermomechanical processing of TRIP steels.. The precise characterization of the resulting multiphase microstructure of low alloyed TRIP steels is of great importance for the interpretation and optimisation of their mechanical properties. The results obtained from *in* situ neutron diffraction laboratory experiment concerning the austenite to ferrite transformation in Si-Mn bulk TRIP steel specimens, displaying the TRIP, effect are presented. The advancement of ferrite formation during transformation in conditioned austenite is investigated at different transformation temperatures and has been monitored by the neutron diffraction method. The relevant information on transformation proceeding is extracted from neutron diffraction spectra. The integrated intensities of austenite and ferrite neutron diffraction profiles developed during the transformation are then assumed as a measure of the phase volume fractions of both phases in dependence on transformation temperature and austenite conditioning. According to the yielding information on ferrite volume fractions from isothermal transformation kinetics data the thermomechanical processing of bulk specimens was designed in order to support austenite stabilization through bainitic transformation. The volume fractions of retained austenite resulting at alternating transformation conditions were measured by neutron and X-ray diffraction respectively. The stability of retained austenite in bulk specimens during room temperature mechanical testing was characterized by *in situ* neutron diffraction experiments as well.

Keyword : Si-Mn TRIP Steel, Transformation Behaviour, Neutron & X-ray diffraction Analysis

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INTRODUCTION

Research conducted to develop materials with enhanced strength and ductile characteristics has resulted in steels that contain different structural phases. Steels, in dependence on alloying, can be processed to retain a certain amount of untransformed austenite at room temperature. The final structure consists of equilibrium ferrite, bainite and retained austenite (RA) islands. The RA is able to transform into martensite when plastic deformation is applied increasing both the strength and the ductility of the steel. As results of actual mechanisms involved in promoting the strength and plasticity simultaneously, these steels are known as TRIP steels, that stand for transformation-induced plasticity (Zackay, *et al.* 1967; Itami, *et al.* 1994; and Sugimoto, *et al. 1994*). Concerning the formable high strength steels, associated with the transformation induced plasticity of retained austenite, polygonal ferrite of the dual phase PF steels is replaced by bainitic ferrite matrix and became attractive for some automotive applications because of excellent stretch formability Sugimoto, *et al.* (1994) and high impact absorbed energy (Song, *et al. 2000*). TRIP aided steels by this way present complex multiphase microstructures with metastable austenite, which is stabilised by concentrating the carbon through specifically designed thermal or thermomechanical (TM) treatment (Zackay, *et al.* 1967).

Various process routes for TRIP steels are either already in use or are subject to discussion depending on products. The processing of low alloy multiphase and TRIP steels is still a matter of current research. Special attention has to be paid to the cooling strategy when producing hot rolled multiphase steels. The temperature – time schedule used for the desired structure development, where lower cooling rates are concerned, appears more difficult when great thermals gradients are developing across the specimen during cooling. The processing should be carried out in the way that the final microstructure comprises of 50 to 60 % ferrite, 25 to 40 % bainite, and 5 to 15 % metastable retained austenite uniformly distributed in the bulk specimen. A variation of the cooling rate and cooling temperature support affects the change of the transformation behavior and may vary the strength level and ductility in a wide range (Block, 2002).

The precise, fast and cheap measurement of the amount of retained austenite in the multiphase structure is an essential item for further research work, improvement and development control of TRIP steels. The difficulty in identifying the different constituents of multiphase high strength steels in the final structure is persistent. For determination of retained austenite content in TRIP steels, the X-ray diffraction measurements are preferentially used, sometimes also classical metallography, Mössbauer spectroscopy and magnetic methods. If available, the neutron diffraction (ND) analysis can be helpful as well (Zhao, *et al.* 2001; and Xie, *et al.*1994).

The purpose of this study is to contribute a better understanding of the factors governing the development of multiphase microstructures in bulk specimens of alloyed 0.2C-1.9Si-1.4Mn TRIP-aided steel. The *in situ* neutron diffraction experiment have been used to evaluate the austenite decomposition in the course of the isothermal austenite to ferrite transformation process. In dependence on the transformation temperature and prior compressive deformation the volume fraction of non-transformed austenite and transformed ferrite have been quantified. On the basis of this information the detailed procedure of bulk specimens and the TM processing was designed to provide multiphase structure. In order to quantify the volume fraction of RA in the resultant multiphase structure the ND and X-ray diffraction were employed. The *in-situ* deformation tests have been performed on specimens with different RA volume fraction in multiphase structure while monitoring the phase transformation by means of ND.

EXPERIMENTAL

The chemical composition of the as-received micro-alloyed TRIP-aided steel grade is stated in Table 1. The specimens for TM processing and *in-situ* neutron diffraction monitoring were machined to a diameter of 6 mm with a gauge length of 15 mm. The thermocouple, needed for temperature setting, was welded directly on the specimen surface.

Table 1 Chemical composition of TRIP-aided steel in wt.%.

C	Mn	Si	P	S	Cr	Ni	Cu	Al	Nb
0.19	1.45	1.9	0.02	0.02	0.07	0.02	0.04	0.02	0.003

The TM processing of specimens consisted of heating to solution temperature of 1000°C for 5 min, and then cooling to 900°C followed by 25% compressive deformation in the deformation rig assembled in the neutron diffractometer. The temperature was consequently decreased to the different transformation temperatures T_t of 800, 775, 750, 700°C, which were derived from the ferrite transformation temperature region according to continuous cooling transformation diagrams of the experimental steel, Figure 1. The holding time at different isothermal transformation temperatures was 20 minutes in all cases. Finishing the holding time, the specimens were cooled down to the room temperature. The diagram for TM processing is presented in Figure 2.

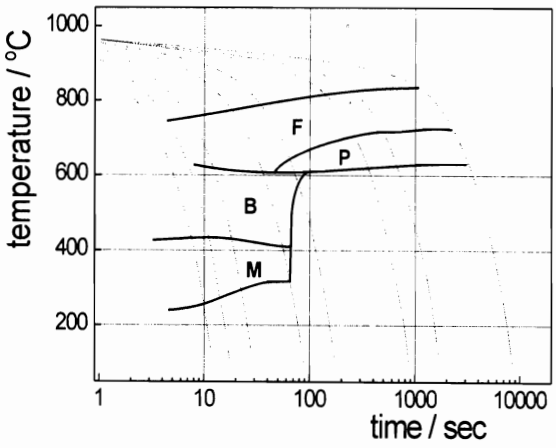

Figure 1 CCT diagram of the studied TRIP steel.

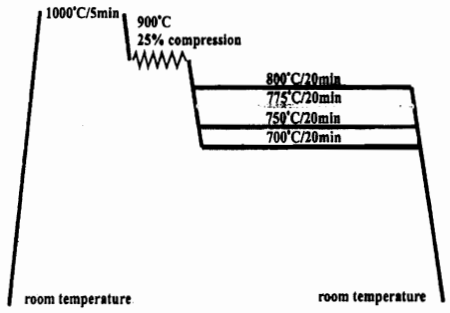

Figure 2 The diagram of applied TM process.

Neutron diffraction experiments were realized at the dedicated high-resolution stress/strain diffractometer TKSN-400 in NPI Řež (instrumental resolution of $\Delta d/d \cong 2x10^{-3}$). This facility is mainly used for in situ investigations of the deformation processes in different materials (Lukas, *et al.* 1999; Sittner, *et al.* 2002; and Tomota, *et al. 2003*). The instrument is equipped with a special deformation rig for tension/ compression loading up to the force of 20 kN. The resistant heating system is mounted directly to the water-cooled and modified grips of the deformation machine. By using the EUROTHERM thermo controller, a relatively good temperature stability of ±0.5°C in an overall working temperature range is reached. This fact represents the main benefit of the experimental method. The PC controlling system enables the independent control of the deformation machine, cooling system and also the diffractometer. Such a flexible controlling

system allows even a rather complicated scheme of thermomechanical loading. The diffractometer is dedicated stress/strain instrument with a linear PSD providing diffraction spectrum in a relatively narrow 2θ band of about $2\theta = 10°$. The detector window was set to cover both ferrite (110) and austenitic (111) reflection.

Simultaneously out of *in situ* ND experiment, however considering the obtained transformation kinetics data from isothermal austenite transformation, the TM processing of bulk cylinder specimens of 25 mm in diameter were carried out. The structure evolution in time of austenite conditioning prior to transformation and in time of isothermal transformation of conditioned austenite was carried out. The light microscopy was used in order to detect the structure evolution either in time of processing or after finishing the TM processing. The several TM schedules were carried out to monitor the austenite evolution in time of conditioning varying the specimen thermal and deformation conditions. On the basis of data evaluation, received from high temperature *in situ* ND experiments and from the structure analysis of austenite resulting from the conditioning of bulk specimens, the final temperature-time schedules for the hot pressing of Si-Mn steel bars were designed. The bainitic ferrite transformation condition, in order to prepare the multiphase structure consisting of ferrite, bainite and RA are also included in the study. X-ray and ND diffraction methods have been employed to detect the RA content in the multiphase structure of TRIP steel. *In situ* ND characterization was proposed to evaluate the RA transformation as a function of tensile load.

RESULTS AND DISCUSSION

The initial structure of austenite, resulting from thermomechanical treatment carried out in the ND experiment corresponding to solutioning condition (T_{sol} =1000°C/1h/H_2O) is documented in Figure 3. The structure of conditioned austenite, developed in deformed specimen by 25% reduction in specimen diameter which was introduced following the solutioning at temperature corresponding to spontaneous recrystallization, is presented in Figure 4. The applied deformation resulted

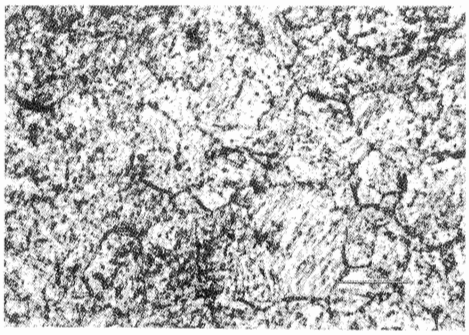

Figure 3 Structure of solutionzed austenite.

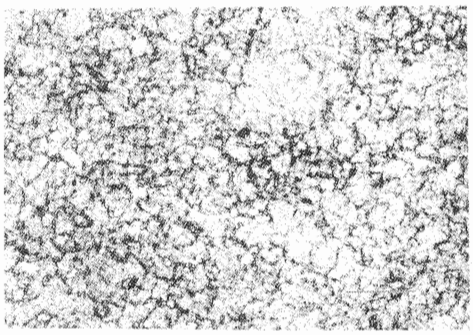

Figure 4 Structure of conditioned austenite.

in austenite grain refining. The conditioned austenite was then subjected to isothermal transformation at different temperatures. During all this thermal exposures, the neutron diffraction spectra were collected in 30 sec sequences, recording the evolution of (110) ferrite and (111) austenite reflection. The example of diffraction profiles is shown in Figure 5. The relevant information on transformation characteristics can be extracted from integrated intensities and angular positions of the individual profiles. The austenite and ferrite integrated intensities can be assumed as a measure of the phase volume fractions whereas the profile positions can be used for the estimation of the elastic

lattice strains evolving in both phases during transformation. The integrated intensity of the diffraction profile is proportional to the phase volume fraction, however, it also strongly depends on the temperature. To eliminate the temperature effect, the calibration dependence of the (110) intensity as a function of the temperature was measured in a single ferrite phase state of the steel. The time evolution of

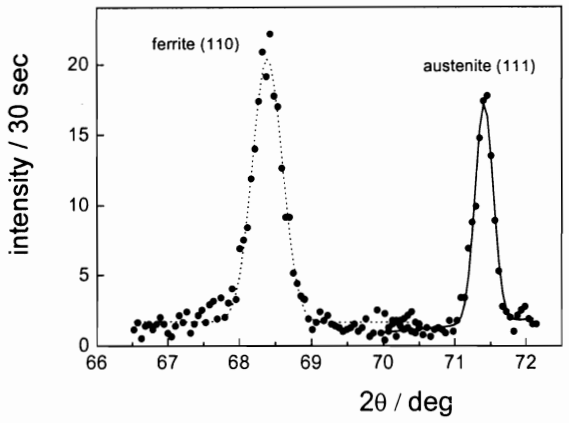

Figure 5 The neutron diffraction profiles of (110) ferrite and (111) austenite.

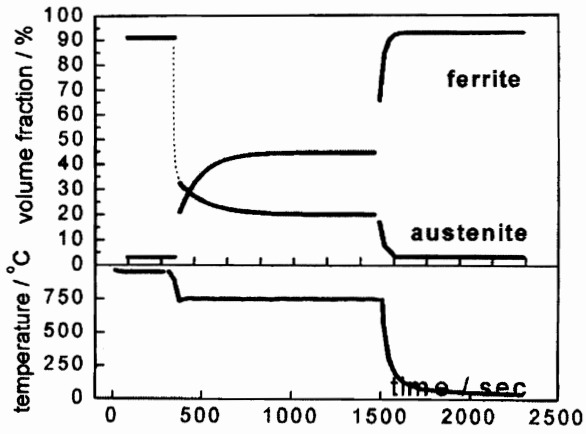

Figure 6 The time dependence plot of the volume fraction evolution of the ferrite phase during the isothermal holding at transformation temperature.
(a) Transformation temperature T_t=750°C;
(b) Temperature record.

the volume fraction of both phases was determined in the course of the thermal loading. An example of such a record for the transformation temperature $T\text{-}_t$= 750°C was shown in Figure 6. The integrated intensities of the austenite and ferrite diffraction profiles can be assumed as a measure of the phase volume fractions yielding thus information on kinetics of isothermal transformation. The obtained results are summarized in Figure 7.

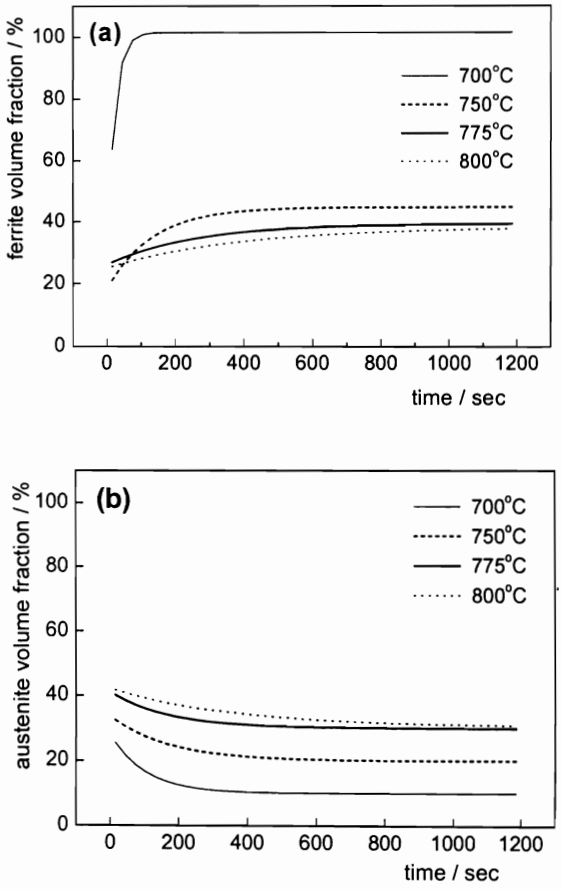

Figure 7 The evolution of the ferrite (a) and austenite (b) volume fractions, for different transformation temperatures, as a function of time.

The volume fraction of ferrite and austenite were determined from independent measurement, so that the sum of (v_F + v_A) was received was less than 100%. This discrepancy can be explained either by the

presence of the third phase (carbides) or by a statistical error roughly estimated ± 5%. Generally, the results pointing out to the amount of phase transformed are in good agreement with the CCT diagram (Figure 1) of experimental steel. The volume fraction of RA has been found ranging between 2.6 and 3.2 % in all cases and was predetermined by transformation conditions (T and time).

The data received from this high temperature *in situ* ND experiment have been employed at further experimental procedure to design the strain, thermal and holding time parameters in austenite conditioning of bulk specimens for TM processing producing multiphase steel. According to the results of ND experiment the austenite to ferrite transformation temperature of T = 750°C for TM processing of experimental Si-Mn TRIP steel have been defined.

The integration of mechanical shaping and decomposition of austenite into TM processing resulted in a multiphase microstructure that should display the TRIP effect. Optimization of such processes requires understanding of how the high temperature deformation alters the transformation mechanisms and kinetics. To investigate the effect of hot deformation austenite conditioning on austenite decomposition, laboratory deformation schedules using a hydraulic press have been performed. The major difficulty is the thermal matching, since the heat losses from a small laboratory specimen are proportionately greater than from an industrial slab.

The experimental procedure for the investigation of the effect of hot deformation on austenite transformation was as follows. The cylinder specimen of 25 mm in diameter and 25 mm in height was heated in an air furnace at 1000°C for 1 hour. Three different TM schedules regarding temperature-time-deformation employing have been conducted as follows:

1. Heating 1000°C/1h → ε_1= ø25mm → t = 13mm → T= 750°C/180s → H_2O/ 3s cooling → 420°C/300s→ air
2. Heating 1000°C/1h → ε_1= ø25 mm → t = 17mm →25s air cooling ∟90° → ε_2= h 30mm →t 13mm → T= 750°C/300s → H_2O/ 3s cooling → 420°C/300s→ air
3. Heating 1000°C/1h → ε_1= ø25mm → t = 17mm →25s air cooling ∟90° → ε_2= h 30mm →t 16mm → T= 750°C/300s → H_2O/ 3s cooling → 420°C/300s→ air

The samples structure morphology corresponding to austenite decomposition at 750°C and corresponding to transformation at bainite temperature region were examined by optical microscopy. ND and X-ray diffraction measurements are used as a reference to deduce austenite content in steel after completing the TM processing. Tensile specimens were machined to study retained austenite transformation during mechanical testing. To understand, and finally control, the TRIP effect, the stability of RA during mechanical testing was studied. The specimen for these tests were cut off from bulk specimens received after hot pressing. *In situ* ND analysis during strain induced transformation to characterize RA transformation was performed.

The compressive deformation of the austenite above 800°C has an effect on the ferrite transformation. The reaction is accelerated by deformation, especially when the second successive specimen deformation is performed in the non-recrystallization region of austenite (Sittner, *et al.* 2002). The deformation effect depends on the temperature of forming and the strain introduced. It was also noted that the higher deformation inserted in the specimen has raised the transformation temperature. The proeutectoid ferrite precipitation, in dependence on the amount of specimen straining was observed for along austenite grain boundaries for schedule (2) where the highest specimen height reduction appeared, Figure 8.

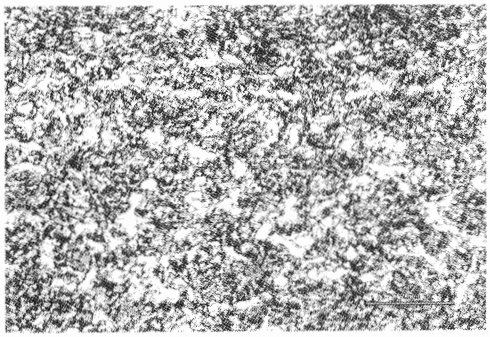

Figure 8 Micrograph of proeutectoid ferrit islands.

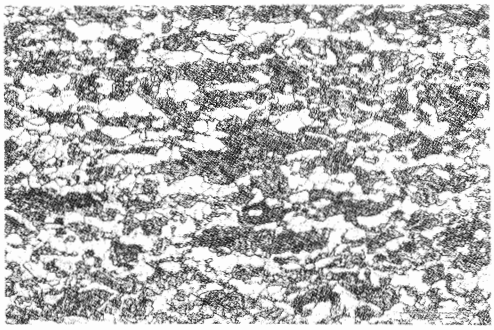

Figure 9 Micrograph of multiphase F-B-RA final structure.

One of the prerequisites, of which the TRIP effect would function in steels is that the final microstructure comprises 5 – 15% metastable RA which is stabilized and does not transform to martensite at room temperature. From this point of view, to enable the transformation of austenite to ferrite, associated with carbon enrichment of the austenite, the austenite volume fraction prior to transformation to bainite can appear as crucial. Performing controlled forging experiments, the structure with the highest fraction of ferrite was observed in specimens processed according to schedule (2), Figure 9.

The RA is the results of a purposeful TM processing that have been performed. The measurements based on ND and X-ray diffraction provided the following fractions of RA with realized schedules in Table 2.

Table 2 Volume fractions (in %) of retained austenite measured in multiphase structure.

	TM Schedule (1)	TM Schedule (2)	TM Schedule (3)
ND	0	13	23
X-ray	7	14	20

In the second part of the ND experiment, the neutron diffraction experiments were carried out to evaluate the stability of RA as a function of a tensile load during the strain induced transformation. Three specimens containing different volume fractions of RA (0%, 13% and 23%) were tested. During the tensile test, the specimens were loaded by a constant crosshead displacement in incremental continuous steps until failure; the deformation step of 1.3% was uniform in all successive deformation steps. The diffraction data were collected after each deformation step during 1 hour intermissions at constant deformation. The position sensitive detector was set to record the evolution of (110) ferrite and (111) austenite reflections. The absolute initial value of phase composition was determined by the X-ray phase analysis, relative changes of the RA volume fraction during the mechanical test were then determined from relative changes of the integrated intensities of (111) austenite the profile only.

To compare the strength and ductility of all specimens as a function of RA content, the stress values were plotted against the deformation of total gauge length, Figure 10. As can be seen the specimen containing a larger amount of austenite shows higher formability-lower yield point and higher elongation whereas the strength values are nearly identical.

The observed austenite volume fraction as a function of strain are plotted in Figure 11. Diffraction information on volume fractions is related to the irradiated central part of the specimen, the macroscopic strain

of this gauge volume was measured by an extensometer.

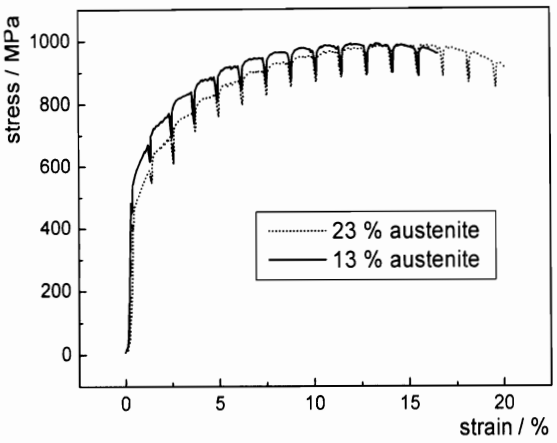

Figure 10 Strain-stress curves of studied specimens.

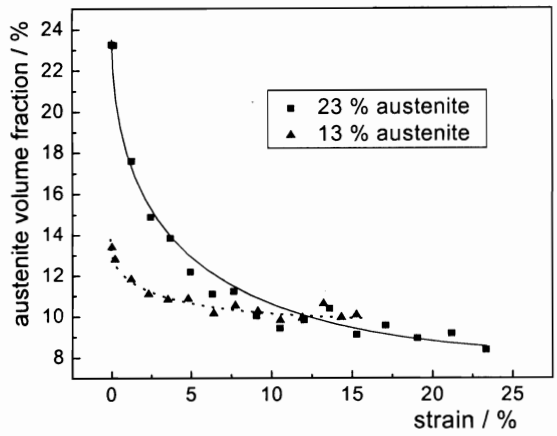

Figure 11 Austenite volume fraction as a function of macroscopic deformation.

The specimens containing a larger amount of retained austenite exhibits very good transformation behaviour; only about 8% of untransformed austenite has been detected in the final microstructure after failure whereas the specimen containing 13% of retained austenite shows a moderate TRIP effect and a higher content of 10% of untransformed austenite after failure. The reliable comparison of diffraction and mechanical response, respectively, can be done in the strain region below 12%, above this limit both specimens were necked. As can be seen in Figure 11, the essential part of transformation takes place below the strain of 5%, so that the disturbing necking effect is not of crucial importance.

CONCLUSIONS

The neutron difraction method applied *in situ* upon combined thermomechanical processing has been used for characterization of the austenite to ferrite transformation in TRIP steels. The advancement of ferrite formation during transformation in conditioned austenite at different transformation temperatures has been monitored. The transformation temperature of 750°C has been proposed as the most favorable for austenite transformation. The transformation data obtained from this *in situ* high temperature neutron diffraction experiment have been obtained in the laboratory experimental route, designing straining, thermal and holding time criteria in austenite conditioning to produce multiphase structure in bulk material. The X-ray and neutron diffraction were used to deduce retained austenite content in Si-Mn TRIP-aided steel. The room temperature tensile tests have provided information on the stability of retained austenite during mechanical testing. The higher formability and transformation ability were detected in steel specimens of higher austenite content. The obtained results provide good experimental judgment on developing the TRIP effect in bulk materials and offer a challenging new manufacturing route in high strength levels.

ACKNOWLEDGEMENT

The co-authors (P. Lukaš, P. Jenčuš, O. Muransky) gratefully acknowledge the financial support of present research provided by the Grant Agency of the Academy of Sciences of the Czech Republic (contracts 202/03/0891 and 106/02/1521/).

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(Revised version accepted September 11, 2004)