# DESIGN OF THERMO MECHANICAL
# PROCESSING AND TRANSFORMATION BEHAVIOUR OF BULK Si-Mn TRIP STEEL

In the last decade, a lot of effort has been paid to optimising the thermomechanical processing of TRIP steels that stands for transformation induced plasticity. 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 *in situ* neutron diffraction laboratory experiment concerning the austenite to ferrite transformation in Si-Mn bulk TRIP steel specimens, displaying the transformation induced plasticity (TRIP), are presented. The advancement of ferrite formation during transformation in conditioned austenite is investigated at different transformation temperatures and has been monitored using 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 thermo mechanical processing of bulk specimen 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.

**Key words:** *transformation induced plasticity (TRIP), Si-Mn steel, thermomechanical processing, neutron and X-ray diffraction, mechanical testing*

**Termomehanička obrada i ponašanje pri transformaciji Si-Mn TRIP čelika.** Tijekom zadnjeg desetljeća uložen je veliki napor u optimiziranje termomehaničkog procesa TRIP čelika, tj. plastičnosti čelika inducirane transformacijom. Precizno objašnjavanje svojstava dobivene višefazne strukture niskolegiranih TRIP čelika je od velike važnosti za intervenciju i optimizaciju njihovih mehaničkih svojstava. Prikazani su rezultati dobiveni *in situ* laboratorijskog eksperimenta difrakcijom neutrona a u vezi transformacije austenita u ferit u uzorcima Si-Mn TRIP čelika koji pri tome pokazuju plastičnost induciranu transformacijom (TRIP). Razvijanje feritne formacije tijekom transformiranja u kondicionirani austenit istraživalo se pri različitim temperaturama transformacije i praćeno je uporabom metode difrakcije neutrona. Relevantna informacija o razvoju transformacije dobivena je iz spektra difrakcije neutrona. Integrirani intenziteti profila difrakcije neutrona austenita i ferita razvili su se tijekom transformacije i onda su uzeti kao mjerilo za volumski udjel obadvije faze u ovisnosti o temperaturi transformacije i kondicioniranje austenita. Prema informacijama o popuštanju volumskih udjela ferita dobiveni iz izotermnih podataka o kinetičkoj transformaciji projektirana je termomehanička obrada krupno uzorka radi podrške stabilizaciji austenita kroz transformaciju bainita. Difrakcijom neutrona, odnosno x-zraka, izmjereni su volumski udjeli zaostalog austenita pri promjenljivim uvjetima transformacije. Svojstva stabilnosti zaostalog austenita u krupnim uzorcima tijekom mehaničkog testiranja na sonoj temperaturi obilježena su i *in situ* eksperimentima s difrakcijom neutrona.

**Ključne riječi:** *transformacija potaknuta plastičnošću (TRIP), Si-Mn čelik, termomehanička obrada, difrakcija neutrona i x-zraka, mehaničko ispitivanje*

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## INTRODUCTION

Research conducted to developed 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 between 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 [1 - 3]. 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 [4] and high impact absorbed energy [5]. By this way TRIP aided steels present complex multiphase microstructures with metastable austenite, which is stabilised by concentrating the carbon through specifically designed thermal or thermo mechanical (TM) treatment [1].

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. A special attention has to be paid to the cooling strategy when producing hot rolled multiphase steels. The temperature - time schedule used for desired structure development, where lower cooling rates are concerned, appears more difficult when great thermals gradients are developing across the specimen in time of cooling. The processing should be carried out in 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 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 pretty wide range [6].

Precise, fast and cheap measurement of the amount of retained austenite in multiphase structure is an essential item for further research work, improvement and development control of TRIP steels. The difficulty in identifying different constituents of multiphase high strength steels in 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 [7, 8].

The purpose of this study is to contribute to a better understanding of the factors governing the development of multiphase microstructures in bulk specimens of high strength low alloyed 0,2C-1,9Si-1,4Mn TRIP-aided steel. The *in situ* neutron diffraction experiment has been used to evaluate the austenite decomposition in course of the isothermal austenite to ferrite transformation process. In dependence on the transformation temperature and prior inserted 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 the TM processing was designed to provide multiphase structure. In order to quantify the volume fraction of RA in resulted 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 PART

The chemical composition of the as-received low alloyed TRIP-aided steel grade is stated in Table 1. The specimens

Table 1.    **Chemical composition of TRIP-aided steel in wt. %**
Tablica 1.    **Kemijski sastav TRIP čelika izražen u tež. %**

| 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 |

for TM processing and *in-situ* neutron diffraction monitoring were machined to diameter of 6 mm with the gauge length of 15 mm. The thermocouple, required to temperature setting, was welded directly on the specimen surface.

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 deformation rig assembled in 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 diagram of the experimental steel, Figure 1. The hold time at different isothermal transformation temperature was of 20 minutes in all cases. Finishing the cooling the specimens were cooled down to the room temperature. The diagram for TM processing presented in Figure 2.

Neutron diffraction experiments were realized at the dedicated high-resolution stress/strain diffractometer TKSN-400 in NPI Řež (instrumental resolution of $\Delta d/d \cong 2 \times 10^{-3}$). This facility is mainly used for in situ investigations of the deformation processes in different materials [9 - 11]. 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.
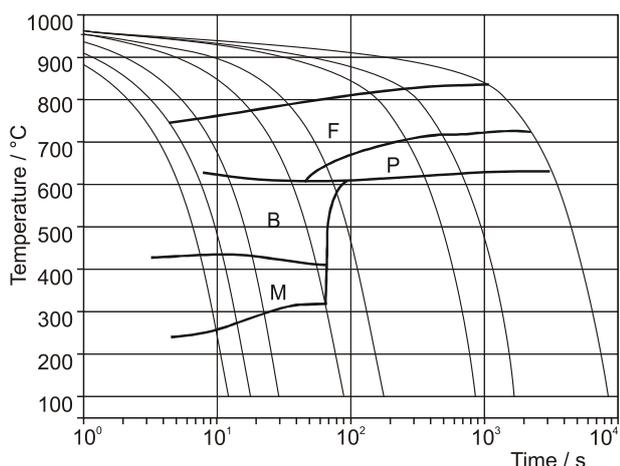
Figure 1.    **CCT diagram of the studied TRIP steel**
Slika 1.       **CCT dijagram ispitivanog TRIP čelika**

By using the EUROTHERM thermo controller, a relatively good temperature stability of ± 0,5 °C in overall working temperature range is reached. This fact represents the main benefit of the used 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 rather complicated scheme of thermomechanical loading. The diffractometer is delicate stress/strain instrument with a linear PSD providing diffraction spectrum in a relatively narrow $2\theta$ band of about $2\theta = 10°$. The detector window was set to cover both ferrite (110) and austenitic (111) reflection.
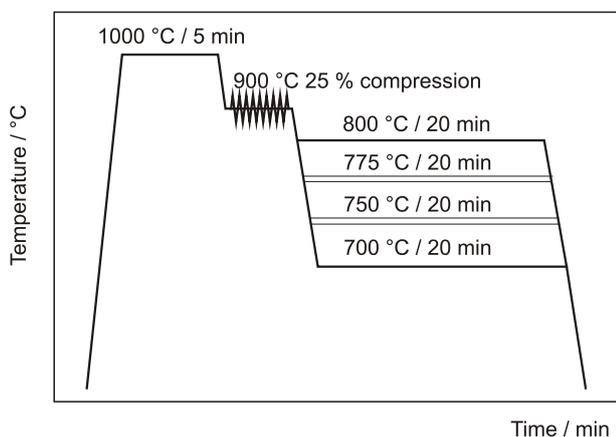


Figure 2.    **The diagram of applied TM process**
Slika 2.       **Dijagram primijenjenog termomehaničkog postupka**

Simultaneously out off *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 transformation and in time of isothermal

trnsformation 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 condition. On the basis of data evaluation, received from high temperature *in situ* ND experiment and from structure analysis of austenite resulting from conditioning of bulk specimens, the final temperature-time schedules for 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 multiphase structure of TRIP steel. *In situ* ND character-ization was proposed to evaluate the RA transformation as a function of tensile load applied.

**RESULTS AND DISCUSSION**

The initial structure of austenite, resulting from thermo mechanical treatment carried out off the ND experiment; corresponding to solutioning condition ($T_{sol}$ = 1000 °C /1h/$H_2$O) is documented in Figure 3. The structure of
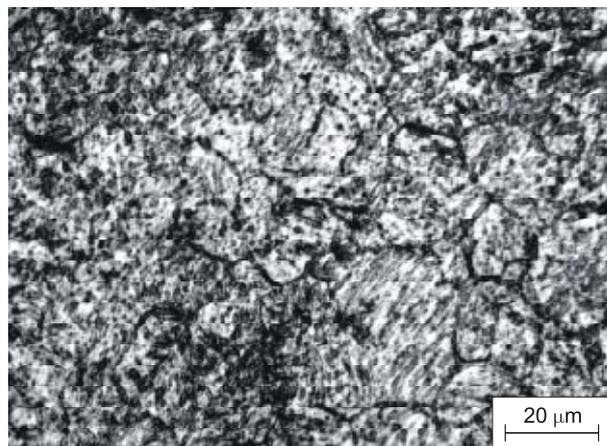


Figure 3.    **Structure of solutionzed austenite**
Slika 3.       **Struktura otopine austenita**

conditioned austenite, developed in deformed specimen as result of 25 % specimen diameter reduction, which was introduced after the dissolving, at temperature correspond-ing to spontaneous recrystallization is presented in Figure 4. The applied deformation resulted  in austenite grain refining. The conditioned austenite was then subjected to isothermal transformation at different temperatures. Dur-ing this thermal exposures the neutron diffraction spectra were collected in 30 sec sequences, recording evolution of (110) ferrite and (111) austenite reflection, respectively. The example of diffraction profiles is shown in Figure 5.
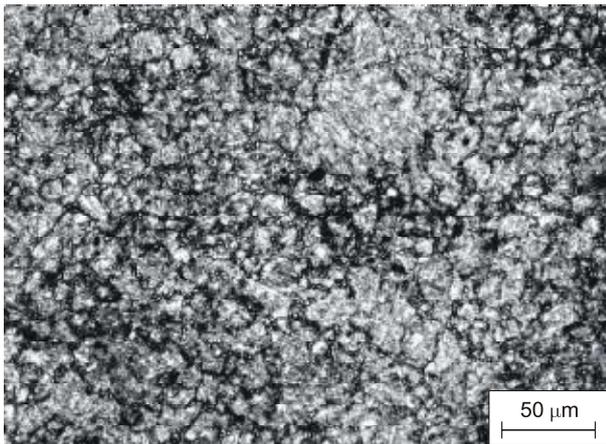
Figure 4.    **Structure of conditioned austenite**
Slika 4.      **Struktura kondicioniranog austenita**

The relevant information on transformation charac-teristics 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 estimation of the elastic lattice strains evolving in both phases during transformation. The integrated intensity of the diffraction profile is proportional
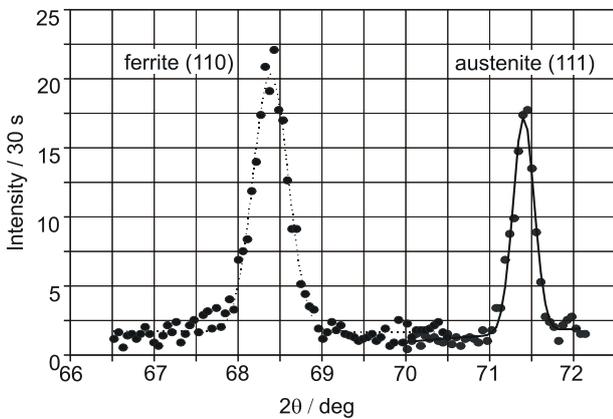


Figure 5.    **The neutron diffraction profiles of (110) ferrite and (111) austenite**
Slika 5.      **Profili difrakcije neutrona ferita (110) i austenita (111)**

to the phase volume fraction, but also strongly depends on the temperature. To eliminate the temperature effect, the calibration dependence of the (110) intensity as a func-tion of the temperature was measured in a single ferrite phase state of the steel. The time evolution of 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 $T_t = 750$ °C is given in Figure 6. The integrated intensities of the austenite and ferrite dif-fraction profiles can be assumed as a measure of the phase
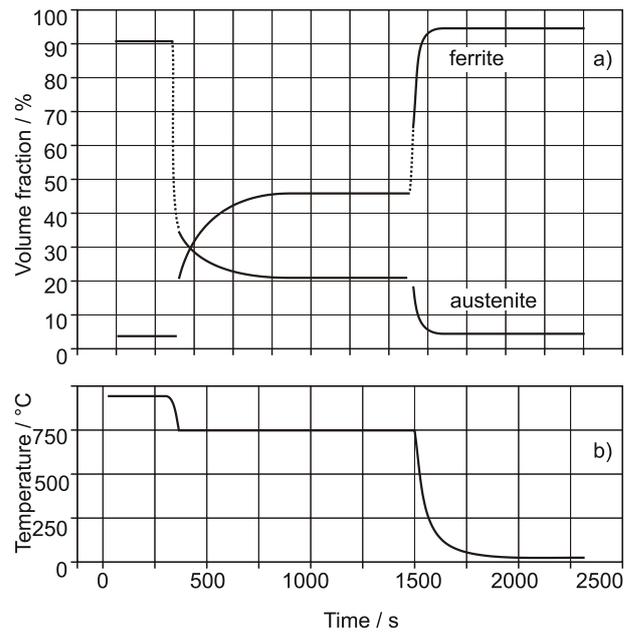


Figure 6.    **The time dependence plot of the volume fraction evolu-tion of ferrite phase during the isothermal hold at trans-formation temperature: a) Transformation temperature $T_t = 750$ °C; b) Temperature record**
Slika 6.      **Dijagram vremena i nastajanja volumenskog udjela feri-tne faze tijekom izotermnog zadržavnja na temperaturi transformacije: a) temperatura transformacije $T_t = 750$ °C; b) temperaturni zapis**

volume fractions yielding thus information on kinetics of isothermal transformation. The obtained results are sum-marized in Figure 7.

The volume fraction of ferrite and austenite was deter-mined from independent measurement, so that sum of ($v_F$ + $v_A$) was received less than 100 %. This discrepancy can be explained either by the presence of the third phase (carbides) or by a statistic error roughly estimated ± 5 %. Generally, the results pointing out to amount on phase transformed are in good agreement with 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 condition ($T$ 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 hold time pa-rameters in austenite conditioning of bulk specimens for TM processing producing multiphase steel. According to the results of ND experiment the austenite to ferrite trans-formation temperature of $T = 750$ °C for TM processing of experimental Si-Mn TRIP steel has been defined.

The integration of mechanical shaping and decomposi-tion of austenite into TM processing resulted in multiphase microstructure that should display the TRIP effect. Opti-mization of such processes requires understanding of how the high temperature deformation alters the transforma-

tion mechanisms and kinetics. To investigate the effect of hot deformation austenite conditioning and on austenite decomposition, a laboratory deformation schedules using hydraulic press has 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.
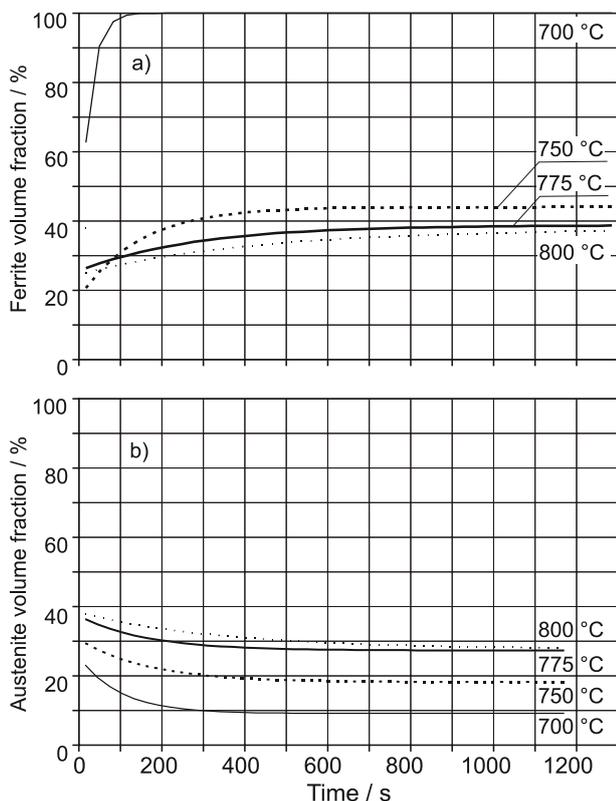


Figure 7.  **The evolution of the ferrite a) and austenite b) volume fractions, for different transformation temperatures, as a function of time**
Slika 7.    **Nastajanje volumske frakcije ferita a) i austenita b) za razne temperature transformacije kao funkcija vremena**

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/1 h $\rightarrow \varepsilon_1 = \varnothing$ 25 mm $\rightarrow t = 13$ mm $\rightarrow T = 750$ °C/180 s $\rightarrow H_2O$/3 s cooling $\rightarrow 420$ °C/300 s $\rightarrow$ air;

2. Heating 1000 °C/1 h $\rightarrow \varepsilon_1 = \varnothing$ 25 mm $\rightarrow t = 17$ mm $\rightarrow$ 25 s air cooling $\llcorner 90° \rightarrow \varepsilon_2 =$ h 30 mm $\rightarrow t$ 13 mm $\rightarrow T = 750$ °C/300 s $\rightarrow H_2O$/ 3s cooling $\rightarrow 420$ °C/300 s $\rightarrow$ air;

3. Heating 1000 °C/1 h $\rightarrow \varepsilon_1 = \varnothing$ 25mm $\rightarrow t = 17$ mm $\rightarrow$ 25 s air cooling $\llcorner 90° \rightarrow \varepsilon_2 =$ h 30 mm $\rightarrow t$ 16 mm $\rightarrow T = 750$ °C/300 s $\rightarrow H_2O$/3 s cooling $\rightarrow 420$ °C/300 s $\rightarrow$ air.

The samples structure morphology corresponding to austenite decomposition at 750 °C and corresponding to transformation at bainite temperature region was 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 for study of retained austenite transformation during mechanical testing. To understand, and finally control the TRIP effect, the stability of RA during mechanical testing was studied. The specimens 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.

The compressive deformation of the austenite in the range above 800 °C has the effect on the ferrite transformation. The reaction is accelerated by deformation, especially in case when the second successive specimen deformation is performed in non-recrystallization temperature region of austenite [10]. The deformation effect depends on the temperature of forming and the strain introduced. It was also noted that the higher deformation inserted in specimen has caused rising up of the transformation temperature. The proeutectoid ferrite precipitation, in dependence on the amount of specimen straining was observed along austenite grain boundaries for schedule (2) where the highest specimen height reduction appeared, Figure 8.
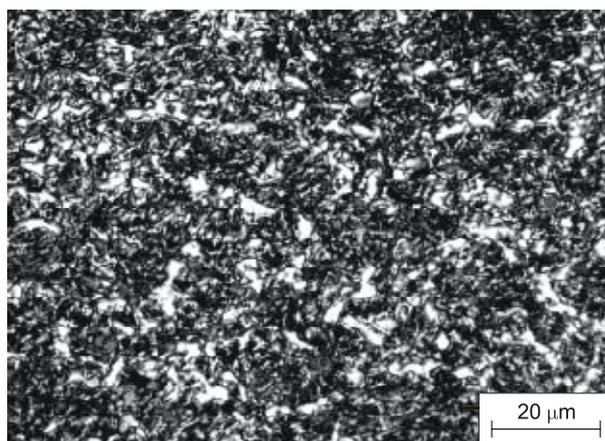


Figure 8.  **Micrograph of proeutectoid ferrite islands**
Slika 8.    **Mikrostruktura proeutektoidnih feritnih nakupina**

One of the prerequisites, the TRIP effect would function in steels, 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 austenite to ferrite, associated

with carbon enrichment of the austenite, the ferrite volume fraction prior transformation to bainite can appear as crucial. Performing controlled forging experiment the structure with the highest fraction of ferrite was observed in specimen processed according to schedule (2), Figure 9.
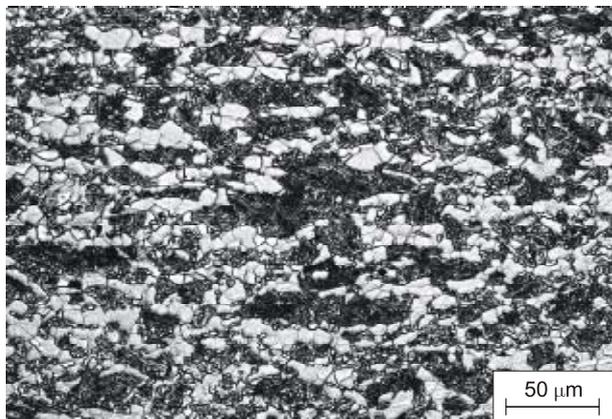


Figure 9.    **Micrograph of multiphase F-B-RA final structure**
Slika 9.     **Mikrostruktura višefazne završne strukture F-B-RA**

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

Table 2.     **Volume fractions (in %) of retained austenite measured in multiphase structure**
Tablica 2.   **Volumski udjeli (u %) zaostalog austenita izmjereni u višefaznoj strukturi**

|         | 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 fractions of RA (0 %, 13 % and 23 % resulting from performing TM processing were tested. During the tensile test, the specimens were loaded by a constant cross-head 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 stopovers at constant deformation. The position sensitive detector was set to record 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 integrated intensities of (111) austenite 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 larger amount of austenite shows higher formability - lower yield point and higher elongation whereas the strength values are nearly identical.
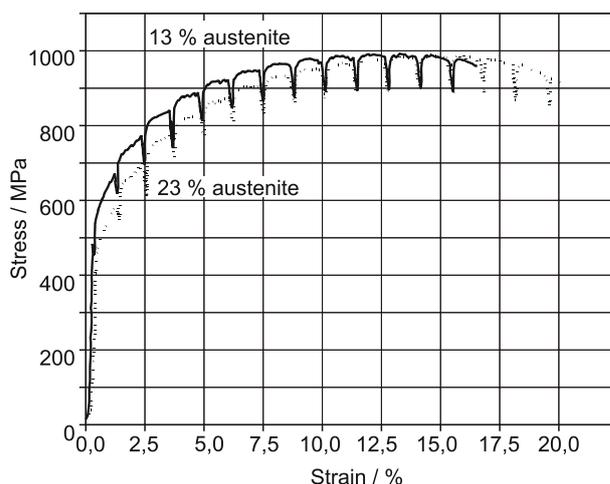


Figure 10.  **Strain-stress curves of studied specimens**
Slika 10.   **Krivulje deformacija-napreznje ispitivanih uzoraka**

The observed austenite volume fractions 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.
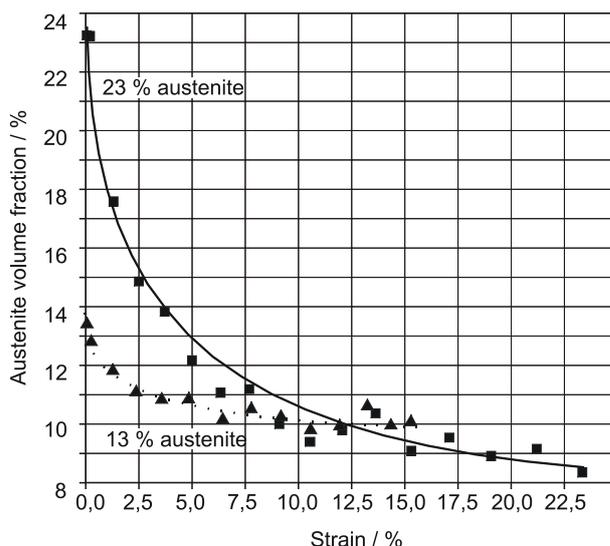


Figure 11.  **Austenite volume fraction as a function of macroscopic deformation**
Slika 11.   **Volumski udjeli austenita kao funkcije makroskopske deformacije**

The specimen containing larger amount of retained austenite exhibits very good transformation behaviour; only of 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 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 diffraction 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 employed at laboratory experimental route, designing straining, thermal and hold 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 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 TRIP effect in bulk materials and offer a challenging new manufacturing route in high strength levels.

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