TRIP STEEL THERMOMECHANICAL PROCESSING SIMULATION AND SUBSEQUENT MICROSTRUCTURE EVALUATION CONCEPTS

Žáček O.[1], Kliber J.[2], Schindler I.[2]
[1] VÍTKOVICE – Research and Development Ltd., Pohraniční 693/31, 706 02 Ostrava, Czech Republic
[2] Department of materials forming, VSB – Technical University of Ostrava, 17. listopadu 15, 708 33 Ostrava, Czech Republic,
E-mail: ondrej.zacek@vitkovice-vyzkum.cz, jiri.kliber@vsb.cz, ivo.schindler@vsb.cz

ZPŮSOBY SIMULACE TERMOMECHANICKÉHO ZPRACOVÁNÍ A NÁSLEDNÉHO VYHODNOCOVÁNÍ MIKROSTRUKTURY TRIP OCELI

Žáček O.[1], Kliber J.[2], Schindler I.[2]
[1] VÍTKOVICE – Výzkum a vývoj, spol. s r.o., Pohraniční 693/31, 706 02 Ostrava, Česká republika
[2] Katedra tváření materiálu, VŠB – Technická univerzita Ostrava, 17. listopadu 15, 708 33 Ostrava, Česká republika,
E-mail: ondrej.zacek@vitkovice-vyzkum.cz, jiri.kliber@vsb.cz, ivo.schindler@vsb.cz

Abstrakt

Oceli s vysokou pevností a dobrou tvařitelností se používají zejména v automobilovém průmyslu pro redukci hmotnosti vozidla. TRIP oceli jsou jedněmi z těchto vysoko pevnostních ocelí. Zpracováním ocelí s využitím efektu transformačně indukované plasticity (TRIP) lze dosáhnout výborné kombinace mechanických vlastností - vysoké pevnosti (až 1200 MPa) a mimořádné plasticity (tažnost až 35%). Podstatou tohoto jevu je stabilizace podstatného množství zbytkového austenitu při termomechanickém zpracování až do nízkých teplot a jeho následná přeměna na deformačně indukovaný martenzit v důsledku plastického přetvoření provázená vysokou plasticitou materiálu. Stabilita zbytkového austenitu proti deformací indukované martenzitické deformaci (Strain Induced Martensitic Transformation – SIMT) je hlavním faktorem ovlivňujícím plasticitu materiálu. Mikrostruktura TRIP oceli je tedy tvořena matricí polygonálního feritu, perlitem, bainitem a významným množstvím zbytkového austenitu. V článku jsou představeny způsoby simulace termomechanického zpracování TRIP oceli a způsoby následného metalografického vyhodnocování mikrostruktury. Cílem je stanovení vlivu jednotlivých parametrů termomechanického zpracování na mikrostrukturu TRIP oceli a na základě těchto výsledků optimalizovat režim termomechanického válcování TRIP oceli. Termomechanické zpracování TRIP oceli bylo simulováno jako laboratorní termomechanické válcování vzorků stupňovitého tvaru a také hladkých vzorků. Válcování vzorků stupňovitého tvaru bylo využito pro stanovení trendů jednotlivých závislostí, hladké vzorky pak byly využity pro stanovení konkrétních hodnot mechanických vlastností. Při metalografickém vyhodnocování výsledné mikrostruktury pak byly zkoušeny různé způsoby leptání vzorků, optická světelná mikroskopie, elektronová mikroskopie včetně EBSD a také RTG difrakce.

Abstract

High strength steels with good formability are used for weight reduction in automobile industry especially. TRIP steels are one of these groups of high strength steels. It can be achieved excellent combination of mechanical properties – high strength (to 1200 MPa) and eminent formability (ductility to 35%) by treatment of steels with TRIP (Transformation Iinduced Plasticity) effect utilisation. Fundamental of this feature is stabilisation of substantial amount of retained austenite down to the ambient temperature at thermomechanical processing and its subsequent transformation for strain induced martensite in consequence of applied plastic deformation. Stability of retained austenite against SIMT (Strain Induced Martensitic Transformation) is main factor affecting plastic properties of material. Microstructure of TRIP steels made up by multiphase structure of polygonal ferrite, pearlite, bainite, acicular ferrite and certain amount of retained austenite. TRIP steel thermomechanical processing simulation concepts and subsequent microstructure evaluation concepts are presented in this paper. The aim of the work is to specify single thermomechanical processing parameters influence on TRIP steel microstructure. The thermomechanical hot rolling condition of TRIP steel will be optimized in terms of mentioned results. TRIP steel thermomechanical processing was simulated by way of step shape samples and flat samples laboratory thermomechanical rolling. Rolling were performed on step shape samples for exact trend determination of single relations whereas plain samples were thermomechanically rolled to define mechanical properties particular values. Utilization of different etching methods, light optical microscopy and electron microscopy including EBSD and even X-ray diffraction were verified within final microstructure metallography evaluation.

Keywords: TRIP steel, retained austenite, thermomechanical processing, laboratory rolling, metallography

1. Introduction

TRIP steels have been known since 1960's when Zackay [1] and others described properties of a steel containing 0.3% C, 9% Cr, 8% Ni, 4% Mo, 2% Mn, 2% Si. TRIP steels, however, have attracted more attention as late as recent two decades. This was due to conception of novel car designs involving steel sheets, aiming at considerable reduction in car weight. The latter will result in decreasing fuel consumption and emissions while maintaining the passenger safety. Thanks to advances in the fields of new materials and rolling techniques, this weight reduction can be achieved by using low-thickness strips from different types of high strength low-alloyed steels made by hot and cold rolling, such as TRIP steels.

Unlike the original high-alloyed steels using the transformation plasticity effect (particularly the metastable stainless steels), which contained ferrite and martensite, the present-day TRIP steels are low-alloyed steels with multiphase microstructure consisting of polygonal ferrite, bainite and retained austenite. These TRIP steels contain 0.1 to 0.4% C, 1.0 to 3.0% Mn and 1.0 to 3.0% Si (or Al). Typically, their composition is 0.2% C, 1.5% Mn and 1.5% Si (or Al). Manganese and silicon (or aluminium) are used in these steels to stabilize retained austenite. Silicon (or aluminium) delays carbide precipitation in the course of austenite decomposition, causing considerable carbon enrichment of the still untransformed austenite, which contributes to its stability [2].

Production of parts from modern steels, including the steels with the transformation induced plasticity effect, combines special alloying and appropriate thermomechanical treatment. Therefore, the thermomechanical schedule profoundly affects the resulting mechanical properties of TRIP steel [3]. Optimization of thermomechanical treatment schedules is conveniently performed with physical simulation using conditions closely approaching the real-world process. In this case, laboratory thermomechanical rolling of different types of samples is used. The method used for evaluation of results is essential for interpretation of physical simulation. With this in mind, different methods of microstructure evaluation in TRIP steel are presented here.

2. Laboratory Thermomechanical Rolling

Thermomechanical rolling of stepped samples was mainly aimed at finding the relationship between the magnitude of strain during hot forming (or forming in the intercritical region) and the volume fractions of individual phases in microstructure of the TRIP steel. Such relationship is also strongly affected by the thermomechanical treatment temperature, by the forming history of individual samples and so forth. When individual flat samples are used, only one strain value can be achieved in each sample. Owing to the laboratory rolling method, it is not possible to guarantee identical rolling conditions for all samples, and hence, the determination of the said relationship might be distorted.

Using the stepped samples (involving four stages) yields four different strain magnitudes in a single pass (within the time on the order of milliseconds) on the mill stand during the laboratory thermomechanical rolling. Strong effects of different thermomechanical treatment temperatures and forming history are thus suppressed, as the comparison is drawn between results obtained in a single specimen. A drawing of a stepped sample is shown in Fig. 1.

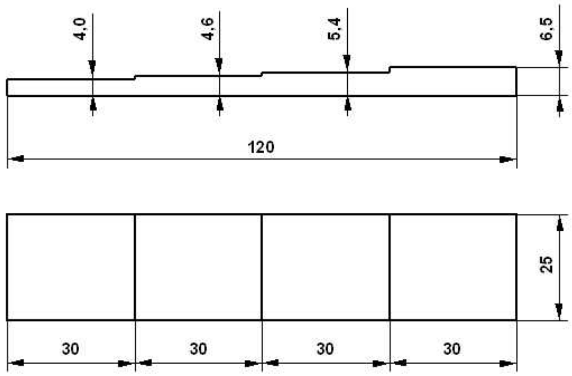


Fig.1 Drawing of a stepped sample

Unfortunately, the dimensions of individual steps in the sample are insufficient for machining a standard tensile test bar and for evaluation of the mechanical properties dependence. Enlarging the samples would produce dimensions exceeding the inner dimensions of furnaces. Therefore, microtensile testing bars have been made of the individual steps of samples. Values of mechanical properties found by testing these bars are affected by the bar dimensions. However, this method is sufficient for their comparison and for determining the trends.

For measuring particular mechanical values and finding the relationships between them, flat samples (with no steps) have been used with dimensions of 6.5x20 – 120 mm. To avoid discrepancies resulting from different thermomechanical treatment conditions, the results have been compared with the trends found by using the stepped samples [4].

Diagram of the thermomechanical rolling process is shown in Fig. 2. Thermomechanical rolling has been performed on a TANDEM laboratory rolling mill - see Fig. 3.[5]. The TANDEM rolling mill with two reversing two-high stands is primarily intended for modelling of hot rolling of flat products.

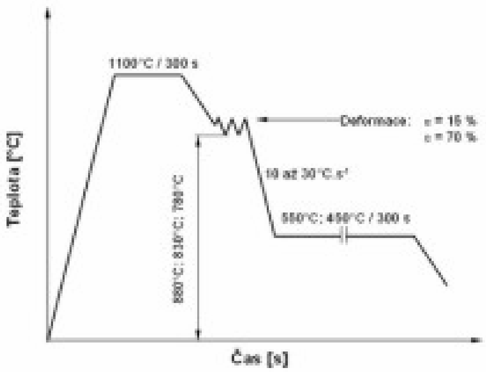


Fig.2 The diagram of the thermomechanical rolling process

Fig.3 The TANDEM rolling mill (view of both stands)

3. Methods of Microstructure Assessment

3.1 Etching Techniques

The metallographic preparation of TRIP steel specimens is performed in a standard fashion. In order to reveal the microstructure, an appropriate etchant is required which attacks various phases to different extent, producing a surface relief. This may be achieved using nital for overetching the sample (with long etching times between 30 and 60 s). Still better results can be obtained with two-stage etching using nital and 10% $Na_2S_2O_5$ solution. Upon grinding and polishing, the specimens are etched with 3% nital (HNO_3) for 5 seconds to reveal grain boundaries. For the next step - one minute etching - 10% solution of $Na_2S_2O_5$ is used. In addition to height differentiation of phases, this step colours the hardened phases (bainite or martensite) brown, whereas retained austenite remains white and ferrite grains turn slightly grey – see Fig. 4.

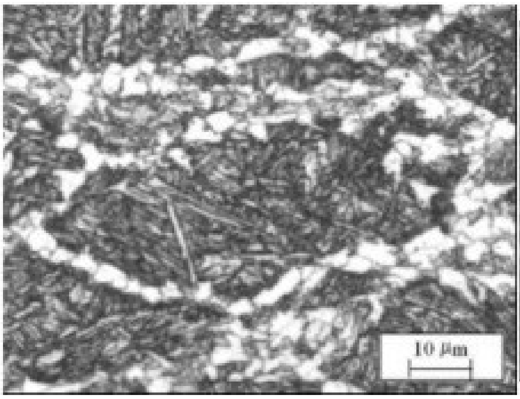


Fig.4 Light micrograph of TRIP steel microstructure upon two-stage etching with nital and 10% $Na_2S_2O_5$ solution (1,000 magnification)

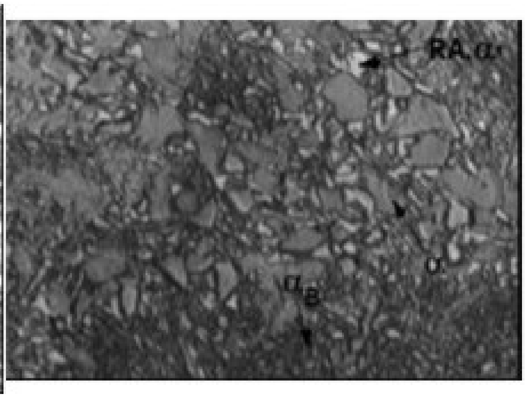


Fig.5 Light micrograph of TRIP steel microstructure upon etching with Le Pera reagent (1,000 magnification)

Le Pera was another tested etchant. It differentiates ferrite, bainite and martensite. Distinguishing martensite and retained austenite is difficult, as both phases appear white when observed with a light microscope - see Fig. 5.

Appreciable surface relief of the microstructure is also produced by the Marble reagent (HCl, $CuSO_4$ and alcohol). This etchant has been used primarily for samples for electron microscopy and X-ray diffraction. Scanning electron micrograph of TRIP steel microstructure magnified 1,500 times is shown in Fig. 6.

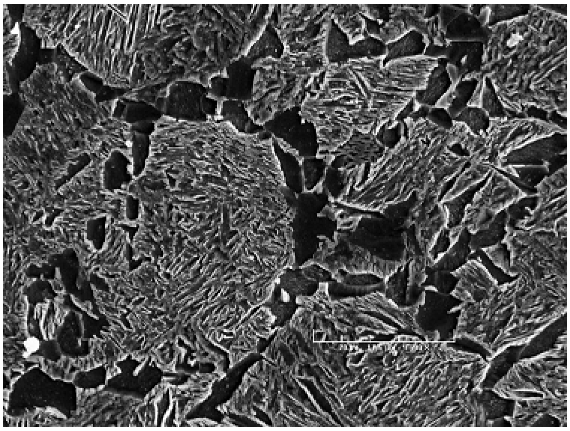


Fig.6 Scanning electron micrograph of TRIP steel microstructure upon etching with Marble reagent (1,500 magnification)

Upon the selective etching produces the surface relief, a light microscope with a polarizing prism can be used as well. With this type of imaging, different phases appear in different colours, which aids identification and quantitative evaluation of microstructure – see Fig. 7.

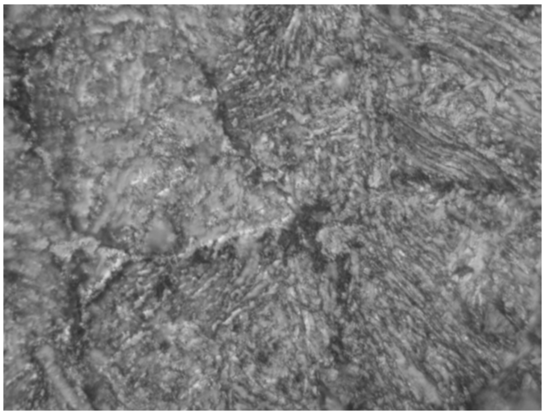


Fig.7 Light micrograph of TRIP steel microstructure (taken with a polarizing prism, shown in grey scale; 1,000 magnification)

3.2 Evaluation of Volume Fractions of Phases

X-ray diffraction was used as a basic method for determination of volume fraction of retained austenite. A Philips XPert diffractometer was employed for measuring the retained austenite fraction on the basis of diffraction maximum intensity ratios in the spectra. The spectra were further processed with Rietveld algorithm to reduce the effects of the forming texture. The accuracy of measurement of volume fraction of a phase is between 3 and 5%. The main problem with using X-ray diffraction concerns very small volume fractions of phases, as the minimum resolved amount is about 5%.

Another technique tested for measuring the retained austenite volume fraction was EBSD (Electron BackScatter Diffraction). This technique is based on electron backscattering. The sample is placed in a scanning electron microscope chamber. The angle of incidence of the electron beam on the sample surface is 20°, which increases the fraction of backscattered electrons and diffraction occurs [11]. The electron reflection from the sample surface produces a radiation pattern – see Fig. 8. The data of the pattern is used for evaluation.

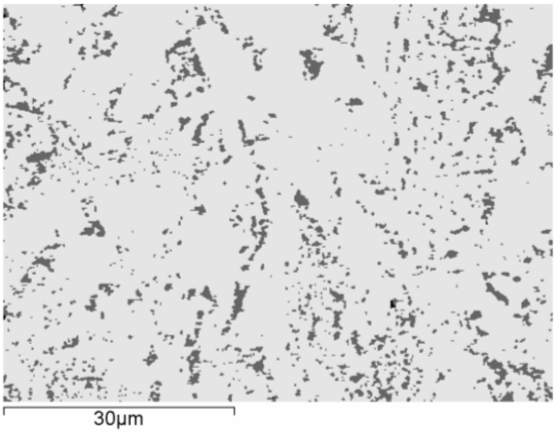
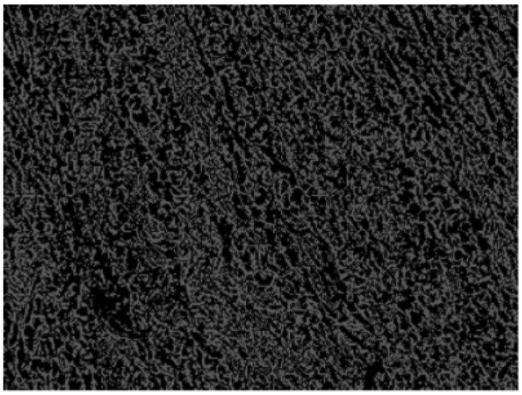


Fig.8 EBSD pattern of TRIP steel (shown in grey scale)

The EBSD disadvantage lies in the fact that owing to the crystallographic orientation it is not possible to distinguish bainite from martensite (the yellow area). EBSD can only be used for measuring the volume fraction of retained austenite (red area). It should be noted that, due to the minimum step of 1 μm, the EBSD data is subject to error resulting from 25 to 30% fraction of non-indexed (ignored) structure, which was calculated by the software in proportion to yield the above values. The fraction of non-indexed structure could be cut down markedly, resulting in higher accuracy of measured data, if a second electron gun was used – the so-called FAG (Field Affected Gun). Unfortunately, there is no such instrument available in the Czech Republic as yet.

The volume fractions of other phases (i.e. ferrite and bainite) were determined by image analysis using the scanning electron micrographs. Volume fractions of structure components were measured by means of image analysis using scanning electron micrographs with 500 magnification taken in the secondary electron imaging mode. Spirit image analysis software has been used for the evaluation.

The TRIP steel microstructure etched with the Marble reagent shows deep-etched ferrite areas, almost intact retained austenite regions and bainite carbides. The secondary electron images have been taken with such adjustment as to show the ferrite areas, where possible, in black and the remaining microstructure constituents in grey. Considering the above described appearance of the etched relief and its imaging in the secondary electron mode, it was impossible to distinguish bainite carbides from retained austenite, as their brightness difference was insufficient. A histogram segmentation has clearly identified only the ferrite areas (both within the actual ferrite grains and in bainite) and their area fraction could be calculated. The fraction of the two remaining constituents is then the calculated remainder to make up the 100%. The fraction of bainite alone can be calculated (within certain tolerance) using the known volume fraction of retained austenite, measured by X-ray diffraction. An example of the image analysis is shown in Fig. 9.



Width(μm): 210,39 Height(μm): 157,79 μm/pixel: 0,205

Scope magnification: 500X

Phase	Left Bound	Right Bound	Area Fraction
■ 1	0	0	62,29%
■ 2	1	1	37,71%

Fig.9 A result of image analysis of the TRIP steel micrograph - 62% fraction of ferrite (black phase), 38% fraction of bainite + retained austenite (red phase – shown grey scale))

4. Conclusion

Physical simulation should, to greatest extent possible, approach the conditions of the real-world process. For this reason, the use of laboratory thermomechanical rolling appears to be a very suitable means of optimization of the industrial schedule of thermomechanical treatment (rolling) of TRIP steel.

The above overview of techniques for evaluation of TRIP steel microstructure and for determination of volume fractions of phases does not represent by any means an exhaustive list of existing methods. These are merely the methods we have used and verified. The presentation of capabilities, advantages and disadvantages of individual methods clearly shows that none of them can provide microstructure evaluation independently. At least two different methods must be used at all times.

Acknowledgment

This work came into being during solution of projects MSM6198910015 (Research Plan financed by the Ministry of Education of the Czech Republic) and 106/04/0601 (Czech Science Foundation).

Literature

[1] Zackay V. F., Parker E. R., Fahr D., Busch R.: *The Enhancement of Ductility in High-Strength Steel.* Transactions of the ASM, V60, 1967, pp 252-259

[2] Jacques P. J., Ladrière J., Delannay F.: *On the influnce of interactions between phases on the mechanical stability of retained austenite in TRIP multiphase steels.* Metallurgical and materials transactions A, volume 32A, November 2001

[3] Kliber J., Masek B., Zacek O., Stankova H.: *Transformation Induced Plasticity (TRIP) Effect Used in Forming of CMnSi Steel.* Materials Science Forum, Vol. 500-501, November 2005, pp. 461-468

[4] Žáček O., Kliber J., Schindler I.: *Vliv parametrů termomechanického zpracování na mechanické vlastnosti TRIP oceli* (Influence of Parameters of thermomechanical treatment on mechanical properties of TRIP steel). In METAL 2006. Hradec nad Moravicí: TANGER, s. r. o., Ostrava, 2006, paper n. 175 (electronic medium).

[5] www.fmmi.vsb.cz/model