Optimization of heat treatment of TRIP steels

S. Nemecek, Z. Novy, H. Stanková

In transformation induced plasticity (TRIP) steels, proper microstructure is essential. The TRIP effect provides the material with high plasticity owing to high volume fraction of ductile phases (ferrite and austenite) in microstructure. In consequence, it provides high hardening through deformation and transformation. The hardening is caused by transformation of retained austenite to martensite. Suitable microstructure for TRIP effect can be obtained by either thermomechanical or heat treatment. Present paper describes development of an intercritical annealing technology, which guarantees fine-grained microstructure formation consisting of appropriate fractions of ferrite, lower bainite and retained austenite. Such structure is suitable for further cold forming with TRIP effect hardening.

Key words: heat treatment, TRIP steel, microstructure, mechanical properties

INTRODUCTION

TRIP (transformation induced plasticity) steels are promising materials for automotive industry [1]. They exhibit favourable strength and considerable amount of plasticity after cold working. During plastic deformation the austenite transforms to martensite, which markedly enhances the material properties and causes hardening. Strain propagates uniformly through the whole workpiece which disables local accumulation of deformation. An analogous growth mechanism produces deformation induced martensite plates, which can serve as barriers against cracks, thereby increasing fatigue life of material.
Optimization of all parameters is needed to obtain proper technological and mechanical properties. The parameters include mainly chemical composition, primary microstructure and heat treatment procedure, which ensure appropriate volume fractions of individual phases and stabilize austenite for the TRIP effect. The heat treatment is performed at intercritical temperatures between A_1 and A_3. Resulting microstructure contains typically 50% of ferrite, 30-35% of bainite and 15-20% of retained austenite. During processing the austenite provides (through formation of proeutectoid ferrite) high plasticity of material. Upon final cold deformation, the austenite transforms to martensite and (together with bainite), thus forming a strengthening microstructural component [2].
One possible route to obtaining desired mechanical properties in TRIP steel is heat treatment. Recrystallization processes in deformed structure and pearlite dissolution occur during heating and intercritical temperature soaking. At the same time, free carbon diffuses into retained austenite. Consequent cooling triggers proeutectoid ferrite formation and further enrichment of austenite with carbon. Holding time at the bainite temperature results in transformation of part of austenite into bainite and consequent strengthening. Considerable influence can be seen not only on the side of temperature but also in the holding time. General features of the heat treatment procedure are described in Fig. 1.

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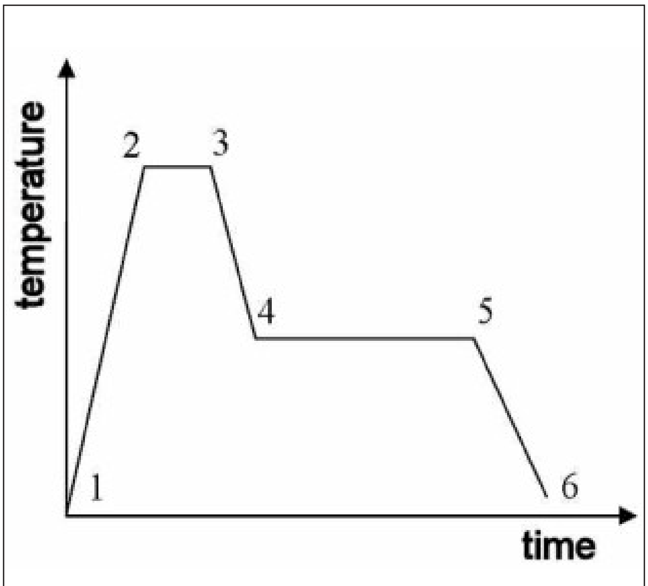

Fig. 1 – Diagram of heat treatment of TRIP steel:
1-2 HEATING: recrystallization, carbide precipitation, cementite dissolution
2-3 DELAY (810°C, 830°C/30 min): pearlite and ferrite transformation to austenite, carbon segregation, grain growth, precipitation of carbides
3-4 COOLING FROM AUSTENITE TEMPERATURE: transformation to ferrite
4-5 BAINITIC HOLDING TIME: (quenching salt 410°C, 430°C/3 to 8 minutes): transformation to bainite, cementite precipitation
5-6 AMBIENT TEMPERATURE COOLING (water): part of austenite transforms to martensite.

Fig. 1 – Diagramma di trattamento termico per un acciaio TRIP:
1-2 SALITA: ricristallizzazione, precipitazione di carburi, dissoluzione della cementite
2-3 MANTENIMENTO: (810°C, 830°C per 30'): trasformazione di perlite e ferrite in austenite, segregazione del carbonio, crescita dei grani, precipitazione di carburi
3-4 PRIMO RAFFREDDAMENTO (al di sotto del campo austenitico): transformazione in ferrite
4-5 PERMANENZA IN CAMPO BAINITICO: (bagno di sali a 410°C, 430°C per 3' - 8'): transformazione in bainite, precipitazione della cementite
5-6 RAFFREDDAMENTO A TEMPERATURA AMBIENTE (acqua): parte dell' austenite si trasforma in martensite.

Chemical composition	C	Mn	Si	P	S	Cr	Ni	Cu	Al	Nb
[%]	0.19	1.45	1.9	0.015	0.015	0.07	0.03	0.04	0.02	0.003

Table 1 – Chemical composition of experimental material.

Tab. 1 – Composizione elementare dei materiali.

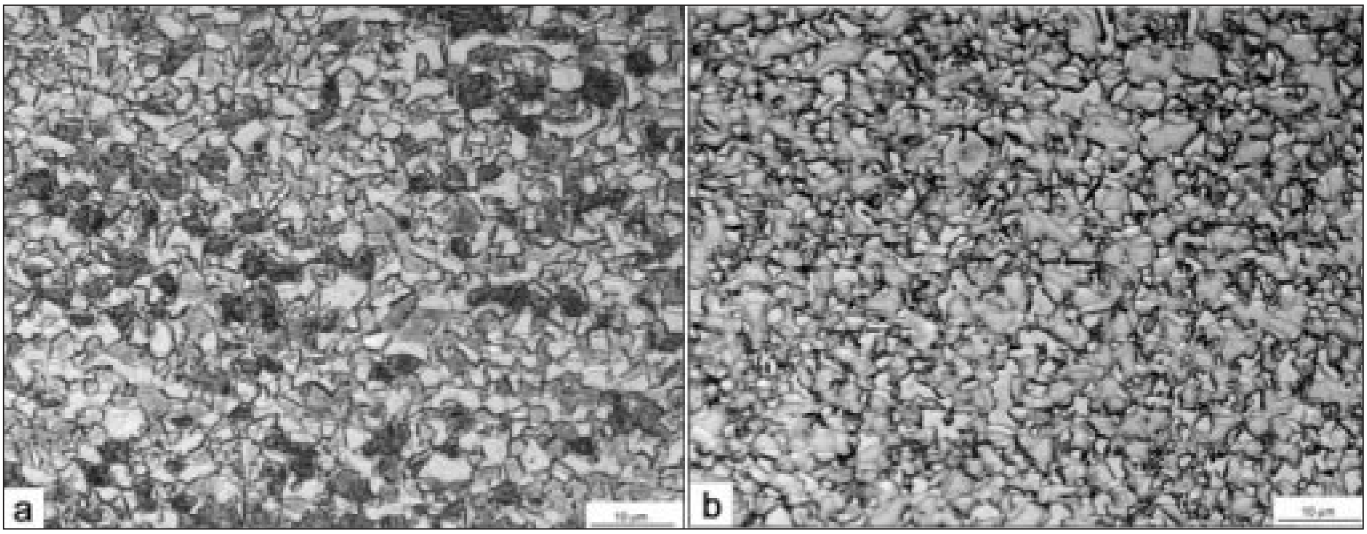

Fig. 2 – Microstructures upon intercritical annealing: (a) after quenching to water, (b) after delay at 410°C; etched with Nital and 10% aqueous solution of $Na_2S_2O_5$ (bright gray ferrite, dark martensite and bainite, white austenite).

Fig. 2 – Microstrutture dopo trattamento intercritico: (a) dopo raffreddamento in acqua, (b) dopo mantenimento a 410°C; attacco chimico con Nital e 10% di soluzione acquosa di $Na_2S_2O_5$ (ferrite grigio brillante, martensite e bainite scure, austenite bianca).

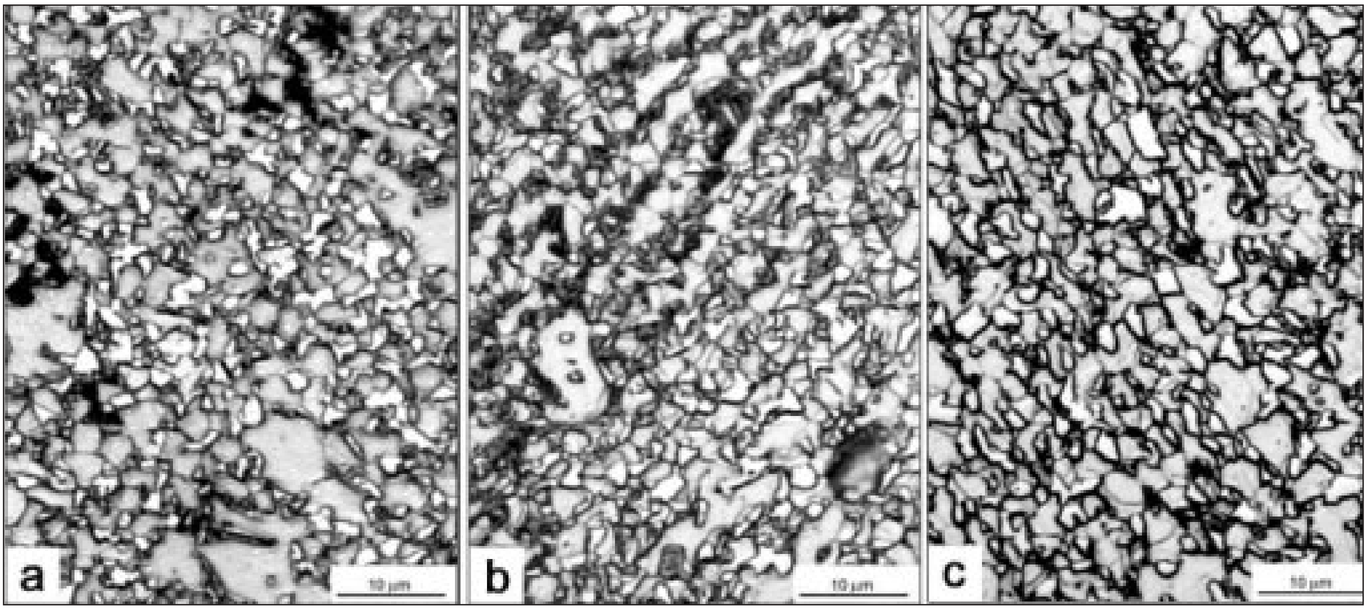

Fig. 3 – Microstructure of samples with different austenitizing temperature: (a) 810°C/30´+410°C/3´, (b) 830°C/30´+ 410°C/3´, (c) 850°C/30´+410°C/3´ (light gray ferrite, dark bainite, white austenite).

Fig. 3 – Microstruttura di campioni a diverse temperature di austenitizzazione: (a) 810°C/30´+410°C/3´, (b) 830°C/30´+ 410°C/3´, (c) 850°C/30´+410°C/3´ (ferrite grigio brillante, martensite e bainite scure, austenite bianca).

Mechanical properties are dependent on 4 main morphological factors: volume fractions of phases, size, distribution and shape of their particles or grains. Therefore, it is possible to determine dependencies between microstructure features and obtained mechanical properties by quantitative metallography.

MATERIALS AND METHODS

The experiments were conducted on Mn, Si steel with TRIP effect (Tab. 1). The carbon content was selected with the aim to stabilize necessary amount of retained austenite. The low carbon content is given by required good weldability. The elements such as Si and Mn serve similar purpose. Si suppresses carbide precipitation in bainite and facilitates carbon enrichment of austenite by proeutectoid ferrite precipitation. Mn decreases austenite transformation temperature and promotes carbon solution in austenite.

The samples were thermomechanicaly processed in order to obtain fine-grained microstructure. Such structure is not only the appropriate for good mechanical properties but also for shortening diffusion paths of carbon, which strongly affects retained austenite formation. Thermomechanical pro-

Table 2 – Dependency of mechanical properties on austenitizing temperature.

Tab. 2 – Dipendenza delle caratteristiche meccaniche dalla temperatura di austenitizzazione.

Heat Preatment Procedure	Proof Stress [MPa]	UTS [MPa]	Ductility [%]
810°C/30 min+ 410 °C/3 min	469 430	1032 1022	21 22
830°C/30 min+ 410 °C/3 min	457 473	993 984	20 20
850°C/30 min+ 410 °C/3 min	457 473	944 948	23 23

Table 3 – Mechanical properties in dependence on bainite transformation holding time.

Tab. 3 – Dipendenza delle caratteristiche meccaniche dalla durata della trasformazione bainitica.

Heat Preatment	Proof Stress [MPa]	UTS [MPa]	Ductility [%]
810°C/430°C-4 min	466	1082	17
810°C/430°C-5 min	458	1065	20
810°C/430°C-8 min	539	1001	26
810°C/410°C-3 min	522	1095	17
810°C/410°C-5 min	470	1021	23
810°C/410°C-8 min	519	976	26

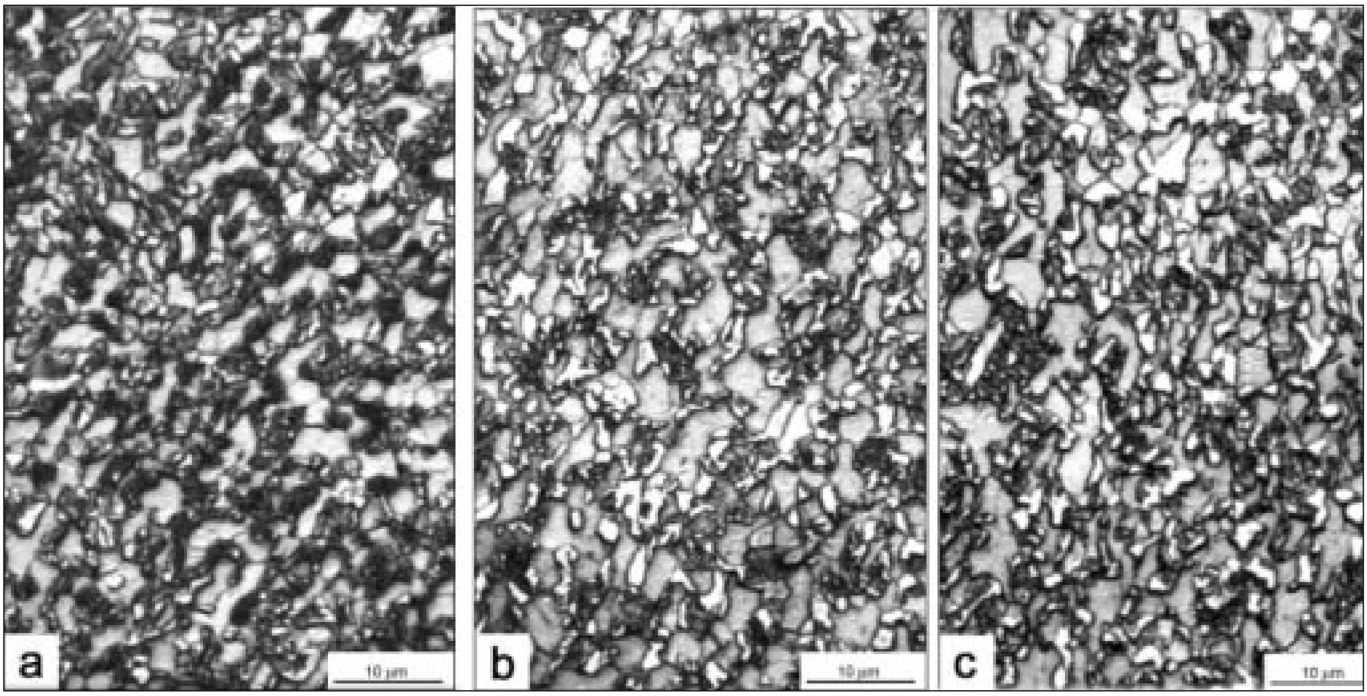

Fig. 4 – Microstructure of samples with different bainite transformation holding time: a) 830°C/30´+410°C/ 3´, b) 830°C/30´+410°C/5´, c)830°C/30´+410°C/8´; (light gray, ferrite, dark bainite, white austenite).

Fig. 4 – Microstruttura di campioni con diversa permanenza a T di trasformazione bainitica: a) 830°C/30´+410°C/ 3´, b) 830°C/30´+410°C/5´, c)830°C/30´+410°C/8´;(ferrite grigio brillante, martensite e bainite scure, austenite bianca).

cessing was performed by controlled series of deformations starting from 980°C. Experimental intercritical annealing range was used with temperatures from 750 °C to 890°C with 30-minute holding time. Metallographic observation confirmed that the microstructure complies with previously measured mechanical properties and an optimized temperature was used for subsequent scheduled experiments.

RESULTS AND DISCUSSION

Two samples were quenched after intercritical annealing – first one to water, second one was air-cooled (upon bainitic holding time) - Figs. 2 and 3, Tab. 2. With respect to obtained results, intercritical annealing 810°C was chosen for further experiments. To optimize the bainite volume fraction, 3 different holding times were tested with temperatures of 430 °C and 410 °C (Figs. 4 and 5, Tab. 3).

After optimization of austenitizing temperature, part of samples was water-quenched, Fig. 2a. Their matrix contains ferrite and martensite with very small portion of austenite. Volume fraction of individual phases has not changed significantly with temperature increasing from 810 to 850 °C. The fractions were as follows: about 26 to 30 % of ferrite and 70 to 74 % martensite. However, increasing temperature causes austenite grain coarsening.
Upon three-minute isothermal holding time at 410°C, majority of the austenite was stabilized, while minor portion transformed to bainite (Fig. 2b). The microstructure contains ferrite, austenite and bainite (Fig. 3). Tensile strength shows about a 10% decrease with increasing austenitizing temperature, while no significant changes occur in ductility and yield strength (Fig. 3 and Tab. 2).
In the following step, bainite transformation temperatures and holding times were estimated. Six modes (after heating at 810°C) at 410°C a 430°C (Tab. 3) with delay from 3 to 10

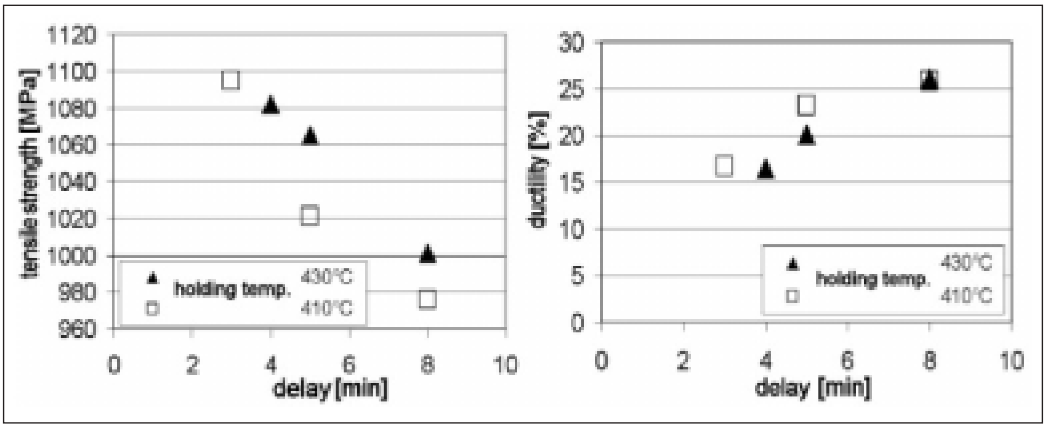

Fig. 5 – Dependence of tensile strength and ductility upon holding times at the 410 and 430°C temperatures.

Fig. 5 – Dipendenza del carico di rottura a trazione e della duttilità dal tempo di permanenza alle temperature di 410 e 430°C.

Product	Proof Stress [MPa]	UTS [MPa]	Ductility [%]
tubes after intercritical HT	435	917	13
tubes after cold deformation	860	926	13
tubes after thermomechanical processing			
spring sheets	460	592	17
sheets after HT	407	790	14

Table 4 – Mechanical properties of the real products (mean value from all samples).

Tab. 4 – Caratteristiche meccaniche di prodotti finiti (valori medi).

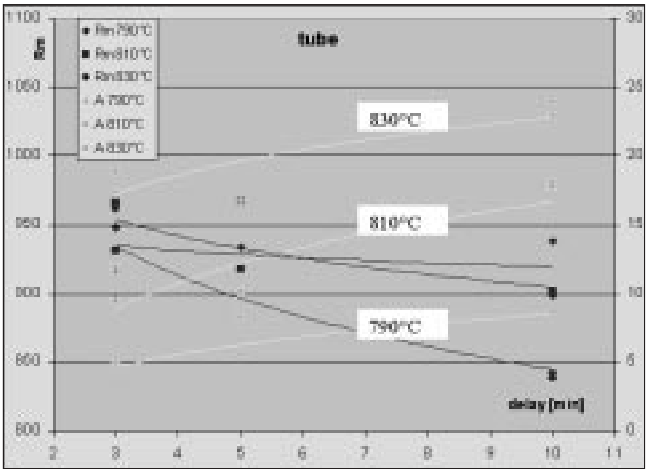

Fig. 6 – Ultimate tensile stress (Rm) (blue curves, left y-axis) and toughness (yellow curves, right y-axis) versus delay at bainite transformation temperature.

Fig. 6 – Carico di rottura a trazione (Rm) (curve blu, asse y di sinistra) e tenacità (curve gialle, asse y di destra) in relazione al tempo di permanenza alla temperatura di trasformazione bainitica.

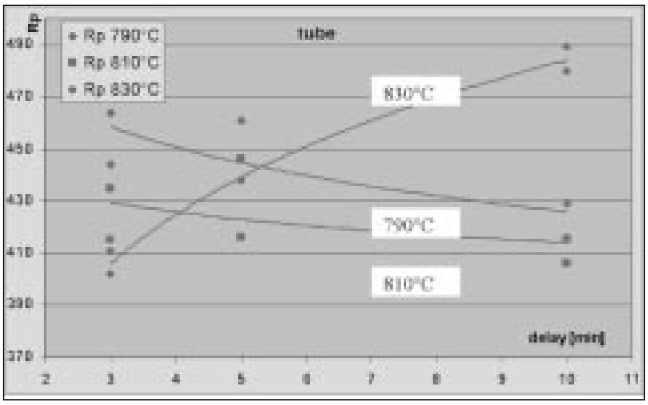

Fig. 7 – Proof stress (Rp) dependencies on time delaying on bainitic transform temperature.

Fig. 7 – Dipendenza delle carico di snervamento (Rp) dal tempo di permanenza alla temperatura di trasformazione bainitica.

minutes were performed. Besides ferrite and the strengthening phases, the structures contain also fine-grained retained austenite, Fig. 4. The longer the bainite formation holding time, the more the toughness increases and strength decreases, see Fig. 5. Yield strength remained relatively low and did not show observable changes.

For obtaining better strength combined with approximately the same plasticity, low austenitizing temperatures seem to be more appropriate. It is relatively surprising because, as was assumed, growing volume fraction of austenite (which occurs during intercritical annealing) causes strength increase due to large ratio of deformation induced martensite. This assumption was not confirmed. Measured data show the opposite, see Tab. 2. The phases did not differ in their volume fractions in dependence on intercritical annealing temperature. Nevertheless, higher temperatures cause grain growth, which is one of the reasons for strength decrease resulting from higher annealing temperature.

The temperature of isothermal holding time in the range of 410°C to 430°C has no significant influence on properties. On the contrary, the time of the isothermal holding time at the bainite transformation temperature is quite important. The longer the holding time, the lower the strength and the better the plasticity of material. This effect can be caused by shifts of the volume fraction between martensite and bainite. With the short holding time, the austenite probably transforms to (thermally induced) martensite. During longer delay, more austenite is transformed to bainite. Martensite – bainite ratio can be the main parameter for strength decrease and toughness increase.

Contrary to assumption, structures contain relatively low amount of fine-grained austenite, which probably occurs predominantly on ferrite grain boundaries, as well as the hard phases do. It is not quite clear if they are represented by bainite or martensite because the light microscope has lower resolution than the size of their patterns. For this reason it is necessary to perform an observation with an electron microscope. Based on those results, the real products were produced as tubes and sheets, Fig.6 and table 4.

The real products were mechanically tested in the same manner as experimental samples. The curves of strength (blue), yield strength (red) and toughness (yellow) in Figs. 6 and 7 show changes depending on time of delay and also on bainite transformation temperature. With extending holding time, the strength decreases and toughness increases. For yield strength, the situation is more complicated because of the second parameter – temperature. At lower temperatures the yield strength decreases but at 830°C it increases rapidly. The lower the temperature, the better the ultimate and yield strengths and lower toughness.

CONCLUSIONS

1. Higher intercritical temperature causes grain coarsening. Increase in volume fraction of retained austenite coupled with decrease of bainite and martensite fractions cause strength decrease but its impact on toughness values is ambiguous.
2. Increasing holding time at bainite transformation temperature causes decrease of strength and increase of toughness of tested material. Results show that as bainite holding temperature increases from 410° to 430°C, the toughness and strength do not change. Main differences occur with holding times from 3 to 10 minutes – toughness increases from 16 to 26% and strength level decreases about 10%.
3. Microstructure evaluation is difficult due to large amount of phases (it is not easy to differentiate them by etching) and small size of objects (about 1mm, which is resolution limit of LM).
4. Detailed experiment allowed us to make products such as sheets and tubes with high end-use properties from high strength TRIP steel while maintaining very good toughness and formability.

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ABSTRACT

OTTIMIZZAZIONE DEL TRATTAMENTO TERMICO DEGLI ACCIAI TRIP

Parole chiave: trattamenti termici, acciaio

Negli acciai TRIP (TRansformation Induced Plasticity), è essenziale un'appropriata microstruttura. L'effetto di TRIP conferisce al materiale un'elevata plasticità grazie alla presenza, nella microstruttura, di una alta frazione in volume di fasi duttili (ferrite ed austenite). Di conseguenza, ciò permette di raggiungere un'elevata durezza tramite deformazione e trasformazione. L'indurimento è causato dalla trasformazione dell'austenite residua in martensite. Una microstruttura adatta all'effetto di TRIP può essere ottenuta mediante trattamento termomeccanico o trattamento termico. Il presente articolo attuale descrive lo sviluppo di una tecnologia di ricottura intercritica, che garantisce la formazione di una microstruttura a grano fine costituita da appropriate frazioni di ferrite, di bainite inferiore e austenite residua. Tale struttura è adatta alla successiva formatura a freddo con indurimento per effetto TRIP.