SPRÁVY

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Z činnosti výboru Vedeckej spoločnosti pre náuku o kovoch pri SAV

Dňa 28.4.2011 sa uskutočnilo Zasadanie výboru Spoločnosti v rámci programu konferencie Konštrukčné materiály 2011 v Žiline. Celkom bolo prerokovaných 5 bodov programu. Podrobnejšie informácie v prípade záujmu podajú jednotliví členovia výboru na pobočkách. Najdôležitejším bodom bolo zabezpečenie činnosti Spoločnosti v súvislosti so zmenou financovania zo strany SAV. V súčasnosti je spoločnosť schopná svojej činnosti bez obmedzenia pričom boli prijaté niektoré úsporné opatrenia bez dopadu na členov Spoločnosti. Informácie o spôsobe financovania Vám v prípade záujmu podá Ing. Pavol Štefánik, CSc. - hospodár Spoločnosti.

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Blahoželáme

RELATIONSHIP BETWEEN MICROSTRUCTURE AND MECHANICAL PROPERTIES OF COLD ROLLED INTERCRITICALLY ANNEALED TRIP STEEL MICRO-ALLOYED WITH TITANIUM

VZŤAH MEDZI MIKROŠTRUKTÚROU A MECHANICKÝMI VLASTNOSŤAMI ZA STUDENA VALCOVANEJ INTERKRITICKY ŽÍHANEJ TRIP OCELE MIKROLEGOVANEJ TITÁNOM

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ABSTRACT / ABSTRAKT

The present contribution describes the microstructure, precipitation state and mechanical properties of cold rolled Ti micro-alloyed multiphase TRIP (TRansformation Induced Plasticity) steel subjected to the heat treatment at continuous annealing line. The results were compared with conventional TRIP steel with a tensile strength of 780 MPa. By an addition of Ti to lean TRIP composition an excellent combination of tensile strength and total elongation, namely $R_m x A_{80}$ *exceeding 15000 MPa x %, was achieved. A pronounced increase in yield and tensile strength of Ti micro-alloyed TRIP steel compared to the reference material is attributed to the partial recrystallization of ferrite during intercritical annealing along with grain refinement and precipitation strengthening mainly caused by small TiC precipitates, whereas its exceptional formability is related to the presence of sufficient amount of retained austenite needed for ensuring desirable TRIP effect.*

Tento príspevok popisuje mikroštruktúru, stav precipitácie a mechanické vlastnosti za studena valcovanej Ti mikro-legovanej viacfázovej TRIP (TRansformačne Indukovaná Plasticita) ocele podrobenej tepelnému spracovaniu na kontinuálnej žíhacej linke. Výsledky boli porovnané s konvenčnou TRIP oceľou s pevnosťou v ťahu 780 MPa. Pridaním Ti k základnému TRIP zloženiu bola dosiahnutá vynikajúca kombinácia pevnosti v ťahu a celkového predĺženia, a to $R_m x A_{80}$ *presahujúca 15000 MPa x %. Výrazné zvýšenie medze sklzu a pevnosti v ťahu Ti mikro-legovanej TRIP ocele v porovnaní s referenčným materiálom je pripísané čiastočnej rekryštalizácii feritu počas interkritického žíhania spolu so zjemnením zrna a precipitačným spevnením spôsobeným hlavne malými TiC precipitátmi, zatiaľ čo jej výnimočná tvárniteľnosť súvisí s prítomnosťou dostatočného množstva zvyškového austenitu potrebného pre zaistenie požadovaného TRIP efektu.*

1. INTRODUCTION / ÚVOD

In order to decrease the car weight, its fuel consumption and in turn CO_2-emissions by a simultaneous improvement of passenger safety, the automotive industry constantly ask for novel steel grades, which achieve high strength levels along with appropriate formability properties. TRIP steels are a well-suited candidate to fullfil these requirements due to their excellent combination of high strength and superior formability, resulting from the strain-induced transformation of retained austenite to martensite in a triple phase microstructure [1-3].

TRIP steels with a strength level from 600 MPa to 780 MPa are currently available on the market [4-6]. To reduce thickness of safety related parts of vehicles even further, the automotive industry requires TRIP steels with a strength level higher than 780 MPa.

The easiest way to increase the strength of TRIP steels is to increase the amount of alloying elements, such as C, Mn, P, Si etc. This is however directly accompanied with a deterioration of their weldability properties. The strength of steel can also be increased by an addition of micro-alloying elements, such as Nb, Ti and V. Precipitation strengthening and grain refinement, resulting from the formation of small carbides or carbonitrides of micro-alloying elements during processing of such a steel, increases its strength without deteriorating the weldability [7].

In the present paper an addition of Ti was used in order to increase the yield and tensile strength of TRIP steels by maintaining acceptable formability properties. The microstructure evolution and mechanical properties of the Ti micro-alloyed TRIP steel were evaluated after the heat treatment at the continouos annealing line (CAL) and were compared with the reference TRIP steel with a tensile strength level of 780 MPa. Furthermore, in detail observation of Ti containing precipitates served as the basis for discussion of their strengthening potential in a TRIP microstructure.

2. PROCESSING OF TI MICRO-ALLOYED TRIP STEEL / SPRACOVANIE TI MIKROLEGOVANEJ TRIP OCELE

Precipitation state during hot rolling stage: The various modes of formation of Ti containing precipitates is expected already during casting, reheating and hot rolling of Ti micro-alloyed TRIP steels. If the steel contains both Ti and N, TiN precipitation already starts in the liquid phase during the casting process (T>1400 °C). $Ti_4C_2S_2$ and TiS precipitation can take place during and after the reheating stage (T>1200 °C). Due to their lowest solubility, TiC particles usually form by strain-induced precipitation or transformation-induced precipitation during hot rolling and subsequent coiling in a temperature range from 1200 °C to 600 °C [8].

Annealing stage: After hot rolling and coiling, the steel is pickled, cold rolled and annealed in order to obtain its TRIP characteristics. A schematic representation of the heat treatment and possible precipitation of the Ti micro-alloyed TRIP steel containing ~0.2 % C is shown in Figure 1. The initial microstructure before intercritical annealing consists of cold rolled ferrite and pearlite, the latter containing some 0.8 % C. In the initial stage of intercritical annealing, pearlite transforms into austenite. The C content of the intercritical austenite then gradually decreases to approximately 0.3 - 0.4 %. The intercritical annealing temperature is high enough to ensure possible dissolution or further growth of TiC precipitates already formed in the previous stages of the processing. The formation of freshly made TiC cannot also be exluded.

Fig. 1: Schematic representation of the heat treatment for cold rolled Ti micro-alloyed TRIP steel: α – ferrite, α_B –bainite, γ – austenite, γ_r – retained austenite, α'- martensite.
Obr. 1: Schematické znázornenie tepelného spracovania za studena valcovanej Ti mikrolegovanej TRIP ocele: α – ferit, α_B –bainit, γ – austenit, γ_r – zvyškový austenit, α'- martensit.

After intercritical annealing the steel is cooled to the overageing temperature. Isothermal bainitic transformation in this stage results in a further C enrichment of retained austenite to about 1.5 %. The TiC precipitation is not expected during this stage due to the insufficient diffusivity of Ti at that temperature [8]. The final microstructure consists of ferrite, bainite, retained austenite and some athermal martensite and contains a number of Ti reach precipitates. The metastable retained austenite transforms to martensite during straining of the steel, resulting in an increased work hardening rate at higher stains. By employing this so called TRIP effect [9] an excellent strength-ductility balance of such a steel can be achieved.

3. EXPERIMANTAL PROCEDURE/METODIKA EXPERIMENTU

The chemical compositions of the investigated TRIP steels are listed in Table 1. The reference material represents the CMnAlSi TRIP steel with a tensile strength level of 780 MPa, abbreviated as ref. TRIP. The Ti micro-alloyed TRIP steel of the same basic composition as the reference material is marked as Ti TRIP.

Tab. 1: Chemical composition of the investigated TRIP steels in wt. %

Tab. 1: Chemické zloženie skúmaných TRIP ocelí v hmot. %

Material	***C***	***Si***	***Al***	***Mn***	***Cr+Mo***	***Ti***	***S***	***N***
ref. TRIP	*~0.2*	*<1.0*	*<1.0*	*1.5-2.5*	*<0.5*	-	0.002	0.003
Ti TRIP	*~0.2*	*<1.0*	*<1.0*	*1.5-2.5*	*<0.5*	*<0.15*	0.002	0.003

The steels were industrially cast in a continuous casting unit and subsequently cut into slabs with a length of 12 m. After reheating at 1200 °C for about 2 h, the slabs were subsequently hot rolled down to a thickness of 3 mm. The finishing temperature was 900 °C. The hot rolled band was coiled at an intermediate coiling temperature. After hot rolling, the band was pickled and cold rolled to a final thickness of 1.5 mm. The cold rolled sheets were annealed at CAL at the intercritical annealing temperature of ~800 °C followed by the overageing at 400 °C for several minutes.

The samples for metallographic observation of the final microstructure were mechanically polished in the sheet direction and etched in LePera reagent [10]. In this etching technique, ferrite is colored blue, bainite is brown and the martensite/retained austenite phase is white.

In order to reveal the precipitation state in the Ti micro-alloyed TRIP steel, mechanical and electrolytical thinning of samples was carried out. The thin foils were examined in a PHILIPS CM20 STEM (Scanning Transmission Electron Microscope) operating with an acceleration voltage of 200 kV. Electron diffraction was used to identify individual Ti containing precipitates. The size of about 200 TiC particles was measured in ferrite in the as annealed condition.

The volume fraction of retained austenite was determined by means of saturation magnetization method [11].

The mechanical properties of the investigated TRIP steels were measured according to European Standard DIN ISO 6982 involving tensile samples with a gauge length of 80mm (A_{80}) .

4. RESULTS / VÝSLEDKY

4.1 MICROSTRUCTURE / MIKROŠTRUKTÚRA

The microstructure of the investigated TRIP steels after the heat treatment at CAL is shown in Figure 2. LePera etching revealed in both steels a typical TRIP microstructure consisting of the ferritic matrix (blue) with bainitic compounds (brown) surrounded by the islands of metastable retained austenite and/or athermal martensite (white/light brown). It is worth noting that Ti micro-alloying evidently refined the overall TRIP microstructure and resulted in a partial recrystallization, so called pancaking, of the ferritic phase. Both mechanisms can effectively strengthen the Ti micro-alloyed TRIP steel.

Fig. 2: Microstructure of the investigated TRIP steels (LePera etching): ref. TRIP (left), Ti TRIP (right)
Obr. 2: Mikroštruktúra skúmaných TRIP ocelí (LePera leptanie): ref. TRIP (vľavo), Ti TRIP (vpravo)

4.2 PRECIPITATION STATE / STAV PRECIPITÁCIE

Figure 4 shows the STEM micrograhs of Ti containing precipitates formed at the early stages of processing of the Ti micro-alloyed TRIP steel. Large rectangular TiN particles with a size of about 1 μm were rarely found in the microstructure. These precipitates are supposed to form even in the liquid phase, as already mentioned aforehand. A number of TiS precipitates, formed during the reheating and hot rolling stage, was also found in the steel microstructure. These precipitates with a globular morphology had a size of approximately 40 nm. Due to their large size, both TiN and TiS are not expected to contribute to the overall strengthening of the Ti micro-alloyed TRIP steel.

Fig. 4: Precipitates formed in the earlier stages of the processing of the Ti micro-alloyed TRIP steel revealed by STEM: TiN (left) and TiS (right)
Obr. 4 Precipitáty vytvorené v skorších štádiách spracovania Ti mikrolegovanej TRIP ocele odhalené pomocou STEM: TiN (vľavo) a TiS (vpravo)

Figure 5: Bright field (left) and dark field (middle) micrographs of numerous TiC particles identified by means of electron diffraction (right)
Obr. 5: Svetlé (vľavo) a tmavé pole (stred) početných TiC častíc identifikovaných pomocou elektrónovej difrakcie (vpravo)

Figure 5 represents the bright and dark field images of small TiC precipitates identified by electron diffraction. From the dark field micrograph is evident that TiC precipitates mostly formed in the ferrite, while only small amount of TiC particles was found within the retained austenite. The size of the precipitates present in the ferrite was

(14±1) nm, which is small enough to contribute to a satisfactory grain refinement and precipitation strengthening of the steel.

4.3 MECHANICAL PROPERTIES AND AMOUNT OF RETAINED AUSTENITE / MECHANICKÉ VLASTNOSTI A MNOŽSTVO ZVYŠKOVÉHO AUSTENITU

The mechanical properties and volume fraction of retained austenite for the reference and Ti micro-alloyed TRIP steel are listed in Table 2.

Tab. 2: Mechanical properties and volume fraction of retained austenite for the investigated TRIP steels
Tab. 2: Mechanické vlastnosti a objemové množstvo zvyškového austenite skúmaných TRIP ocelí

Material	$R_{p0.2}$ [MPa]	R_m [MPa]	A_{80} [%]	γ_r [vol. %]
ref. TRIP	*493*	*800*	*27.2*	*12.8*
Ti TRIP	*750*	*952*	*16.2*	*13*

Compared to the reference material, the addition of Ti increased the yield and tensilte strength of the Ti micro-alloyed TRIP steel of 257 MPa and 152 MPa, respectively. The increase in strength is related to the partial recrystallization of ferrite during intercritical annealing in combination with the precipitation strengthening and grain refinement mainly associated with the presence of small TiC particles in the steel microstructure.

The R_mxA_{80} ratio of the Ti micro-alloyed TRIP steel exceeded 15000 MPa x % at the total elongation of 16.2 %. Such elongation is quite exceptional among the advanced high strength steels (AHSS) of the same strength level. This can be attributed to the presence of a sufficient amount of retained austenite needed for ensuring desirable TRIP effect. The effect of micro-alloying on retained austenite stability with respect to the overall TRIP effect was investigated more in detail elsewhere [1, 8, 12].

Despite the acceptable total elongation achieved in case of the Ti micro-alloyed TRIP steel, the addition of Ti caused its evident decrease of 9 % compared to the reference material. The mechanisms, which may govern this decrease, are as follows [13-15]:

- Partial recrystallization: results in a higher density of mobile dislocations and decreases total elongation by an increased interaction between dislocations,

- ferrite grain size: smaller grain size causes a decrease of total elongation,
- presence of precipitates: in addition to acting as obstacles against the motion of mobile dislocations, precipitates can also serve as the nucleation sites for void linking. Smaller void spacing results in an easier void linking, which in turn causes a decrease of total elongation.

5. CONCLUSIONS / ZÁVER

The present contribution aimed at characterising the microstructure and precipitation state and their influence on the mechanical properties in the cold rolled intercritically annealed TRIP steel micro-alloyed with Ti. The obtained results were critically compared with the reference TRIP steel with a tensile strength level of 780 MPa. The main conclusions can be summarized as follows:

- The addition of Ti clearly refined the overall TRIP microstructure and resulted in a partial recrystallization of the ferritic phase.
- Numerous Ti containing precipitates were found in the microstructure of the Ti micro-alloyed TRIP steel, namely large TiN and TiS particles mainly precipitated during casting and reheating and small TiC precipitates formed during the hot rolling, coiling and annealing stage.
- A pronounced increase in the yield and tensile strength of the Ti micro-alloyed TRIP steel compared to the reference material is mainly associated with the partial recrystalization of ferrite in conjunction with its precipitation strengthening and grain refinement thanks to small TiC precipitates.
- An excellent ratio of $R_m x A_{80}$ exceeding 15000 MPa x % of the Ti micro-alloyed TRIP steel was achieved due to the presence of a sufficient amount of retained austenite necessary for ensuring satisfactory TRIP effect.
- A decrease of the total elongation for the Ti micro-alloyed TRIP steel compared to the reference material is mainly attributed to a higher amount of obstacles hindering the motion of mobile dislocations. This includes an expected higher density of mobile dislocations as a result of partial recrystallization of the ferritic phase, grain size refinement and presence of Ti reach precipitates in the steel microstructure. Nevertheless, the total elongation of the Ti micro-alloyed TRIP

steel is still quite exeptional among AHSS of the same strength level.

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Prednáška „Kvantitatívna analýza štruktúry materiálu“

Dňa 17.3.2011 sa na Ústave výrobných technológii MTF STU v Trnave uskutočnila prednáška organizovaná trnavskou pobočkou Spoločnosti v spolupráci s Ústavom výrobných technológii na tému Kvantitatívna analýza štruktúry materiálu.

Doc. Ing. Maroš Martinkovič, PhD.
Prednášajúci a spoluorganizátor

Konferencia „Konštrukčné materiály 2011“

Dňa 28.4.2011 sa uskutočnila vedecká konferencia s medzinárodnou účasťou Konštrukčné materiály 2011 organizovaná žilinskou pobočkou Vedeckej spoločnosti pre náuku o kovoch pri SAV. Konferencie sa zúčastnilo 25 účastníkov, zo Slovenska a z Českej republiky. Bolo prednesených 14 príspevkov s bohatou diskusiou. Všetky zaslané príspevky boli recenzované a recenzentmi odporučené príspevky budú publikované v časopise Acta Metallurgica Slovaca (ISSN 1335-1532) a Materials Engineering (ISSN 1335-0803).

Prof. Ing. Radomila Konečná, CSc.
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