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### Abstract

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Article

## Microstructure Evolution During the Production of Dual Phase and Transformation Induced Plasticity Steels Using Modified Strip Casting Simulated in the Laboratory

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**Abstract:** Instead of conventional steel making and continuous casting followed by hot and cold rolling, strip casting technology modified with the addition of a continuous annealing stage (namely, modified strip casting) is a promising short-route for producing ferrite-martensite dual-phase (DP) and multi-phase transformation-induced plasticity (TRIP) steels. However, at present, the multi-phase steels are not manufactured by the modified strip casting, due to insufficient knowledge about phase transformations occurring during in-line heat treatment. This study analysed the phase transformations, particularly the formation of ferrite, bainite and martensite and the retention of austenite, in one 0.17C-1.52Si-1.61Mn-0.195Cr (wt. %) steel subjected to the modified strip casting simulated in the laboratory. Through the adjustment of temperature and holding time, the characteristic microstructures for DP and TRIP steels have been obtained. The DP steel showed comparable tensile properties with industrial DP 590 and the TRIP steel had a lower strength but a higher ductility than those industrially produced TRIP steels. The strength could be further enhanced by the application of deformation and/or the addition of alloying elements. This study indicates that the modified strip casting technology is a promising new route to produce steels with multi-phase

Keywords: strip casting; heat treatment; DP steel; TRIP steel; phase transformation

#### 1. Introduction

The strip casting technology allows to produce solid metal strips from liquid metals with minimal amount of deformation in one hot rolling stand continuously operating with a casting unite [1]. This technique has been applied in industry for the production of aluminium, lead, carbon steel, silicon steel and stainless steel [2–4]. In laboratory the ferrite-martensite dual-phase (DP) steel [5–7], multi-phase transformation-induced plasticity (TRIP) steel [8,9] and twinning-induced plasticity steel [10] have also been successfully produced by strip casting. In comparison with a traditional way to produce steels by the sequence of steel-making, continuous casting, hot rolling and cold rolling, the strip casting technology requires a shorter production line, due to the strips being directly manufactured from liquid metals. Therefore, it is an efficient way to save energy, reduce CO<sub>2</sub> emission and increase profitability of a metallurgical enterprise with a minimal impact on environment [1,11].





Typical DP steels contain soft ferrite and hard martensite and they are manufactured in industry by hot or cold rolling followed by annealing [12]. The presence of martensite leads to a continuous yielding behaviour and a moderate work hardening capability [12,13]. Strength and ductility can be adjusted via controlling the volume fraction of martensite. An increasing martensite fraction decreases the ductility but increases strength [14]. For example, when the martensite fraction is 40%, the ultimate tensile strength (UTS) is 660 MPa with a total elongation (TE) of 46.7%; when the martensite fraction is 50%, the UTS is 670 MPa while the TE is 45.0% [14]. After cold rolling, the inter critical annealing in the ferrite-austenite two-phase region can control the ferrite fraction and the austenite fraction. During cooling to room temperature, the austenite will transform to martensite. Thus, the martensite fraction can be controlled through the adjustment of annealing temperature and time. A higher inter critical annealing temperature and longer holding time lead to a larger austenite fraction followed by a larger fraction of martensite after cooling to room temperature [13,14].

Multi-phase TRIP steels consist of ferrite, bainitic ferrite, granular bainite and retained austenite (RA); sometimes a small amount of martensite may be present [15–17]. These steels attract attention of many investigators and researchers all over the word because of their combination of high strength and good ductility. The reason for TRIP steels have a good balance between strength and ductility originates from the presence of RA grains. During deformation, the RA grains transform to martensite (namely, the TRIP effect) leading to enhanced ductility prior to fracture. This transformation in homogeneously occurs in a range of strain levels. Strains required to initiate the austenite-to-martensite transformation vary with the austenite stability ascribed to the austenite grain morphology, carbon content and type of neighbouring phases [17–19]. The TRIP effect delays the development of the local stress concentration and enhances the strain hardening ability, which results in an increased ductility [17,20,21]. Thus, the control of amount and stability of RA grains is a key factor for the enhancement of mechanical properties in TRIP steels [22,23]. The first stage of heat treatment schedule applied to obtain a TRIP steel microstructure is the inter critical annealing in the ferrite-austenite two-phase region. The temperature and holding time in the two-phase region can control the fraction and grain size of ferrite [15,24]. The second stage of heat treatment induces the bainite transformation, during which austenite is enriched in carbon, partitioning from bainite. The fraction, morphology and stability of RA grains can be controlled by adjusting the bainite transformation temperature, which affects the amount and morphology of bainite [16,20,25]. Bainitic ferrite comprises film RA clamped between bainitic ferrite laths, while granular bainite includes blocky RA surrounded by the irregular-shaped ferrite [17]. During the final cooling to room temperature, the austenite which is stable enough can be retained, while the unstable austenite transforms to martensite.

DP and TRIP steels are industrially produced by hot rolling or cold rolling followed by annealing [26]. In the automotive industry they have been successfully applied to manufacture various car parts such as B-pillar reinforcements, longitudinal beams and cross members [27]. Recent studies carried out by the present authors produced low-alloyed ferrite-martensite DP [8] and multi-phase TRIP steels [8] using strip casting simulated in the laboratory. Although tensile properties comparable with industrially hot/cold rolled DP and TRIP steels have been achieved, the microstructure is not uniform through strip thickness, due to the cooling rate gradient [8,9]. To avoid this problem, a furnace was introduced into the processing line of strip casting (Figure 1) for the first time in this work, namely the modified strip casting. An interrupted cooling in the first zone of the furnace should result in formation of a ferrite-pearlite microstructure, instead of bainite and/or martensite obtained in the conventional strip casting due to fast cooling. This stage requires further researches on the acceleration of pearlite formation in the future in order to shorten the furnace in industry. Following this, the second step of heat treatment allowed to produce the homogeneous microstructures of DP and TRIP steels. The phase transformations occurring during the simulated heat treatments for the modified strip casting technology have been investigated and presented below (Figure 1).

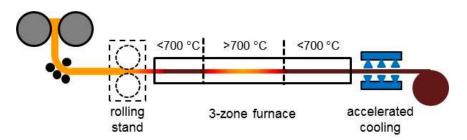


Figure 1. A schematic presentation of the modified strip casting.

#### 2. Materials and Experimental Procedures

#### 2.1. Materials

The received plates of  $36 \times 36 \times 1.2 \text{ mm}^3$  were cast using a dip tester (in-house manufactured at the University of Deakin, Victoria, Australia), which can simulate rapid solidification during strip casting [28]. Thus, the features of the microstructure in the strip casting can be simulated in the laboratory [5,8]. The technical parameters of dip tester can be found in Ref. [28]. The steel studied in this work (contained 0.172) C, 1.520Si, 1.610Mn, 0.195Cr (wt. %) and balanced Fe. The cast microstructure consisted of martensite and bainite as shown in Figure 2.

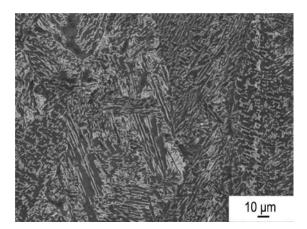
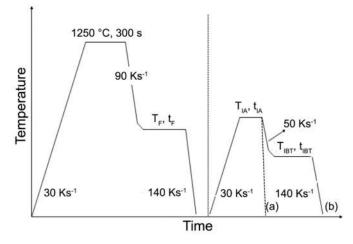


Figure 2. As-cast microstructure consisting of martensite and bainite observed by scanning electron microscopy (SEM).

#### 2.2. Processing Schedules

The processing routes to obtain DP and TRIP steels are illustrated in Figure 3a,b, respectively. They include two steps of heat treatments in order to simulate the modified strip casting in the laboratory: (1) the first step of heat treatment is to produce a ferrite-pearlite microstructure and (2) the second step of heat treatment is to produce microstructures of DP and TRIP steels. The heat treatment was carried out using a Theta Dilatronic III dilatometer (Theta Inc., New York, NY, USA) under a vacuum of ~ $6.7 \times 10^{-2}$  Pa. Flat samples of  $14 \times 6 \times 1$  mm<sup>3</sup> size were cut using wire cutting. To simulate the modified strip casting, the samples were heated at 30 Ks<sup>-1</sup> to 1250 °C and held for 300 s and this led to an average grain size of prior austenite ( $80 \pm 27 \mu m$ ) being similar to that which is usually observed in the strip casting [8]. After holding, the samples were rapidly cooled at ~90 Ks<sup>-1</sup> to different holding temperatures ( $T_F$ ), held for different times ( $t_F$ ) to study the ferrite and pearlite transformation during the modified strip casting and helium quenched at ~140 Ks<sup>-1</sup> to room temperature. To simulate the production of DP steels, the samples with a ferrite-pearlite microstructure were heated at 30 Ks<sup>-1</sup> to a selected temperature of  $T_{IA}$  (750 and 780 °C) in the two-phase region, held for a selected time period of  $t_{IA}$  (120, 180, 240 and 300 s) to allow formation of a certain volume fraction of austenite and helium quenched to room temperature to obtain martensite as the second phase. To simulate the production

of TRIP steels, the samples with ferrite-pearlite microstructure were heated at 750 °C for 240 s, which resulted in formation of ~50% ferrite and ~50% austenite and then quickly cooled at ~50 Ks<sup>-1</sup> to the isothermal bainite transformation temperature of  $T_{IBT}$  (400, 450 and 500 °C), held at this temperature for 900 s (15 min) to simulate coiling and finally helium quenched at ~140 Ks<sup>-1</sup> to room temperature.



**Figure 3.** Schemes of processing schedules applied to simulate the production of (**a**) Dual Phase (DP) and (**b**) Transformation-Induced Plasticity (TRIP) steels using the modified strip casting.

#### 2.3. Microstructure Characterization

The heat-treated samples were cut through thickness, mounted, mechanically polished and electropolished using an electrolyte containing 330 mL methanol, 330 mL butoxyethanol and 40 mL perchloric acid. Then, the samples were etched using 2 vol % nital. The microstructures were characterized using a Leica DMR research optical microscope (OM) (Leica, Wetzlar, Germany) and a JEOL JSM-7001F field emission gun scanning electron microscope (FEG SEM) (Jeol, Tokyo, Japan) operating at an accelerating voltage of 15 kV. On the basis of the pixel quantities of different grey scales in the micrographs, the ferrite fraction was calculated using ImageJ (Image-Pro Plus, Media Cybernetics Inc., Rockville, MD, USA). In order to distinguish between austenite, martensite and bainite, colour etching was employed according to Ref. [28]. A PANalyticalX'pert-Pro MRD goniometer (Malvern Panalytical, Eindhoven, Netherlands), equipped with Ni-filtered Cu K<sub> $\alpha$ </sub> radiation source scanning over a range of 2 $\theta$  = 60–110°, was used to measure the RA fraction. The RA fraction was calculated using the direct comparison method; the details are presented in Ref. [8].

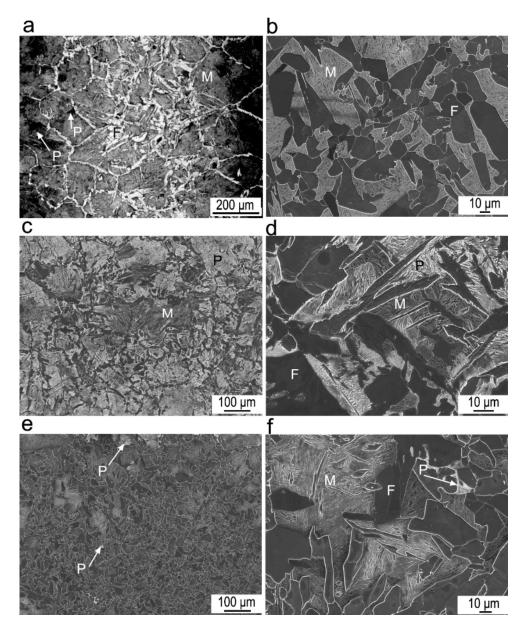
#### 2.4. Mechanical Properties

The dog-bone tensile samples (with a gauge length of 4.9 mm, width of 2.1 mm and thickness of 1 mm) were cut from the heat-treated samples. The uniaxial tensile testing was carried out using an in-house modified Kammrath and Weiss GmbH tensile stage (Kammrath & Weiss GmbH, Dortmund, Germany) at a constant cross-head speed of 2  $\mu$ m/s, which resulted in an initial strain rate of 4 × 10<sup>-4</sup> s<sup>-1</sup>.

#### 3. Results

# 3.1. The Formation of Ferrite and Pearlite Microstructure during the First Step of Simulated Heat Treatments in the Modified Strip Casting

The microstructure evolution with holding temperature ( $T_F$ ) and time ( $t_F$ ) (Figure 3) is shown in Figure 4 and the corresponding fractions of ferrite are listed in Table 1. Figure 3a was observed by OM where black is pearlite, white is ferrite and grey is martensite. Figure 3b–f were obtained by SEM where dark grey is ferrite, light grey is martensite and white is pearlite. With an increase in holding temperature from 620 to 650 °C, for a constant holding time of 180 s, the fraction of ferrite increased from  $0.14 \pm 0.02$  to  $0.46 \pm 0.02$  (Figure 4a,d,f) due to a shortened incubation time for ferrite nucleation and a correspondingly increased time for ferrite growth. With increasing the holding temperature up to 670 °C the fraction of ferrite decreased (as listed in Table 1 for the holding time of 60 or 300 s) due to an increased incubation time for ferrite nucleation. It means that the nose temperature (namely, having shortest incubation time for phase transformation) of ferrite transformation field of the continuous phase transformation diagram is 650 °C. When the holding temperature is 650 °C, the ferrite fraction increases with an increased holding time from 60 to 180 s (Table 1) due to an increased time for ferrite nucleation and growth. When the holding time was 180 s, a minor fraction of pearlite was observed at 620 °C (Figure 4a). A larger fraction of pearlite was obtained after holding at 630 °C (Figure 4c,d)due to a shortened time for pearlite nucleation. Whereas, only little pearlite was observed at a higher temperature of 650 °C (Figure 4e,f) due to an increased time for pearlite nucleation. Therefore, the nose temperature of pearlite transformation field was 630 °C.

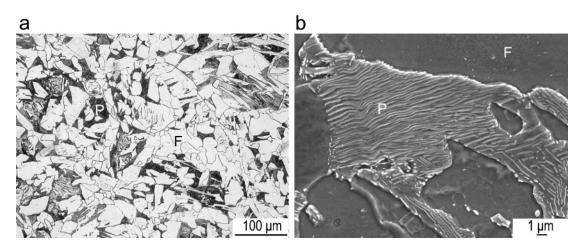


**Figure 4.** (a) Optical microscopy (OM) and (b–f) SEM microstructures after holding at (a) 620 °C for 180 s, (b) 670 °C for 300 s, (c,d) 630 °C for 180 s and (e,f) 650 °C for 180 s. *F* is ferrite, *P* is pearlite and *M* is martensite.

Time	620 °C	630 °C	650 °C	670 °C
60 s	-	-	$0.08 \pm 0.02$	$0.02\pm0.001$
180 s	$0.14\pm0.02$	$0.33 \pm 0.02$	$0.46\pm0.02$	-
300 s	-	$0.46\pm0.04$	$0.56\pm0.02$	$0.50\pm0.03$

**Table 1.** The fraction of ferrite as a function of holding temperature and time.

Since the nose temperatures of ferrite and pearlite transformation field were 650 and 630 °C, respectively, the holding for ferrite and pearlite formation was conducted between these two temperatures, namely 635 °C. This was supposed to enhance the austenite decomposition. Figure 5 shows that the corresponding microstructure consists of ferrite ( $0.75 \pm 0.02$ ) and pearlite. This microstructure was suitable for further processing to produce DP and TRIP steels because ferrite is a microstructural constituent for both of them and austenite prefers to nucleate at the pearlite with a large driving force.

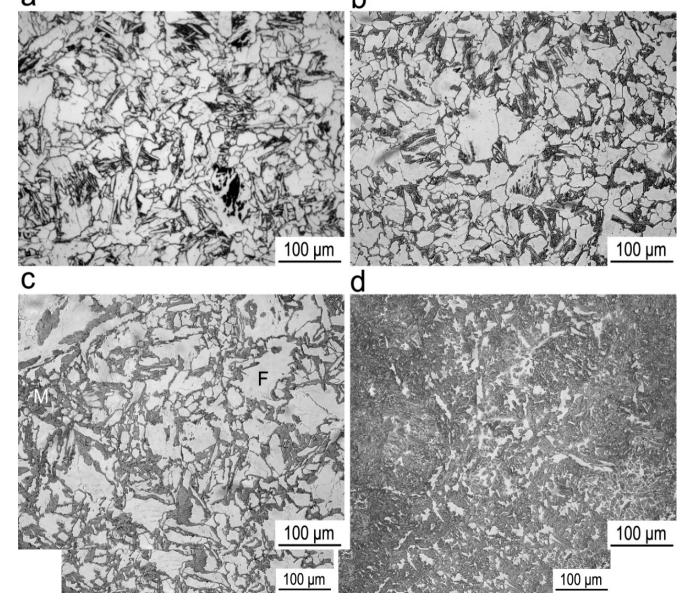


**Figure 5.** Microstructures after austenitizing for 300 s at 1250 °C and holding at 635 °C for 900 s (15 min): (a) OM imaging and (b) SEM imaging. *F* is ferrite and *P* is pearlite.

## 3.2. Production of DP Steel Microstructure during the Second Step of Simulated Heat Treatments in the Modified Strip Casting

Following the first step of simulated heat treatments in the modified strip casting in Section 3.1, the second step of simulated heat treatments was investigated for the production of DP steels (Figure 3a). Figure 6 shows the microstructures after inter critical annealing at different temperatures for different times within the two-phase region followed by direct quenching to room temperature. Quenching after the inter critical annealing led to the formation of microstructures consisting of ferrite and martensite (Figure 6). When the inter critical annealing temperature was 750 °C, the ferrite fraction decreased from  $0.79 \pm 0.01$  to  $0.42 \pm 0.02$  with the increase of the inter critical annealing time from 120 to 300 s (Figure 7). When the inter critical annealing time was 300 s, a higher inter critical annealing temperature of 780 °C led to a lower ferrite fraction of  $0.09 \pm 0.02$  (Figure 6d).

With an increased inter critical annealing time in the austenite-ferrite temperature region the austenite grains continued to nucleate and grow and this resulted in a lower ferrite fraction (Figure 7). Similarly, a higher temperature led to a larger driving force for austenite formation resulting in a less amount of ferrite. Thus, through the control of inter critical annealing temperature and time during the inter critical annealing, DP steels having different fractions of ferrite and martensite can be produced.



**Figure 6.** OM microstructures after inter critical annealing at (**a**) 750 °C for 120 s, (**b**) 750 °C for 180 s, (**c**) 750 °C for 300 s and (**d**) 780 °C for 300 s. White is ferrite (*F*) while grey is martensite.

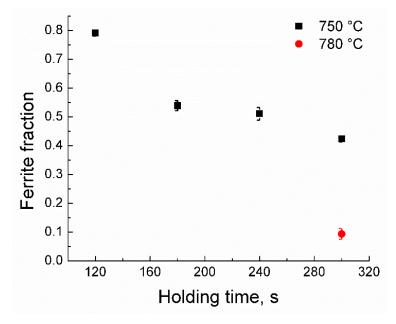
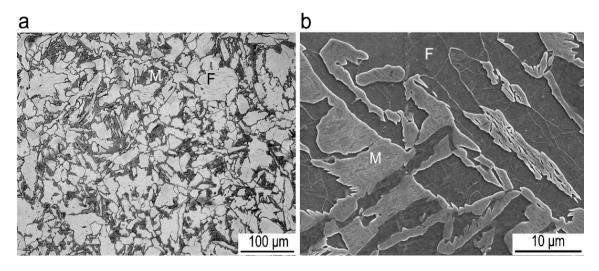


Figure 7. Effect of temperature and time on ferrite fraction during the inter critical annealing.

# 3.3. Production of TRIP Steel Microstructure during the Second Step of Simulated Heat Treatments in the Modified Strip Casting

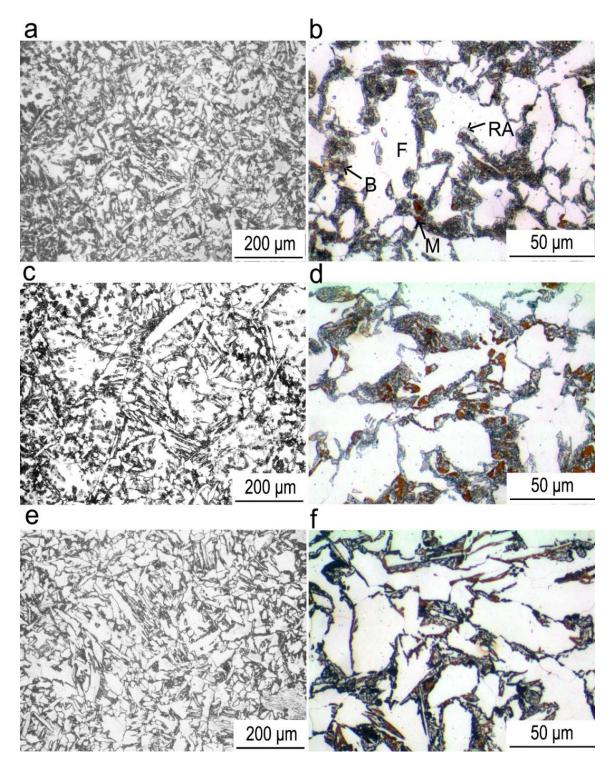
Following the first step of simulated heat treatments in the modified strip casting in Section 3.1, the second step of simulated heat treatments has been investigated for the production of TRIP steels (Figure 3b). Based on the published data, ~50% ferrite fraction provides the optimal combination of UTS and TE in the multi-phase TRIP steels [15,25]. According to Section 3.2,  $0.51 \pm 0.06$  ferrite was achieved after inter critical annealing at 750 °C for 240 s (Figure 8), this condition was selected for further processing to obtain bainite and RA required for TRIP steels.



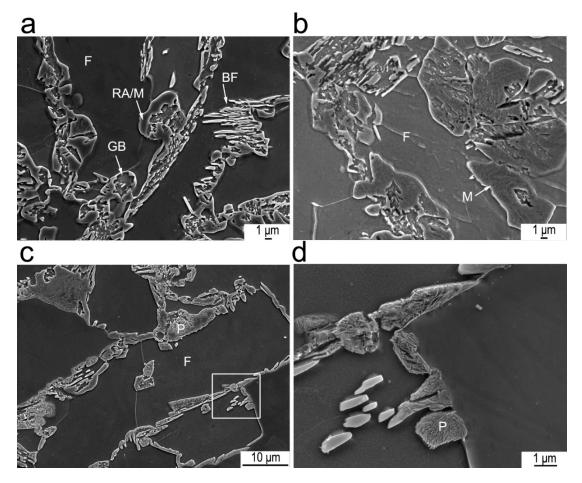
**Figure 8.** Microstructures after inter critical annealing at 750 °C for 240 s followed by quenching to room temperature: (**a**) OM imaging and (**b**) SEM imaging. *F* is ferrite and *M* is martensite.

The isothermal bainite transformation (IBT) was studied by holding at 400, 450 and 500 °C for 900 s (15 min). The representative and homogenous microstructures are shown in Figure 9 (OM) and Figure 10 (SEM), indicating the successful production of typical TRIP steel microstructures [26]. Figure 9b,d,f show the ferrite in grey/white, bainite in dark grey/bluish, martensite in brown and RA in grey/white. After holding at 400 °C, the microstructure consisted of ferrite, bainite, RA and martensite (Figure 9a,b). As seen in Figure 10a, bainite included bainitic ferrite, where film-shaped retained austenite/martensite constituent (RA/M) was clamped by ferritic laths and granular bainite, where globular RA/M was surrounded by irregular-shaped ferrite. After holding at a higher temperature of 450 °C the martensite fraction increased (Figures 9d and 10b). The increasing of holding temperature up to 500 °C slightly decreased the martensite fraction (Figure 9f) and promoted some pearlite formation (Figure 10c,d). It is well known that pearlite is harmful to the TRIP steels; pearlite consumes carbon and reduces the carbon content in austenite. A decreased carbon content in austenite decreases the austenite stability and the fraction of RA in the room temperature microstructure [29,30]. Due to the presence of pearlite after holding at 500 °C this heat treatment condition was regarded as inappropriate for the TRIP steel production and thus the RA fraction was measured only for the samples held at 400 and 450 °C (Figure 11). Based on the direct comparison method, the RA fraction was measured to be 5.2% and 4.2% after holding at 400 and 450 °C, respectively.

A



**Figure 9.** Optical microstructures after holding at (**a**,**b**) 400 °C, (**c**,**d**) 450 °C and (**e**,**f**) 500 °C for 900 s (15 min) following holding at 750 °C for 240 s: (**a**,**c**,**e**) nital and (**b**,**d**,**f**) colour etched microstructures. Ferrite is shown with grey/white, bainite with dark grey/bluish, retained austenite with grey/white and martensite with brown colours in (**b**,**d**,**f**). *F* is ferrite, *B* is bainite, *M* is martensite and *RA* is retained austenite.



**Figure 10.** Microstructures after holding at (**a**) 400 °C, (**b**) 450 °C and (**c**,**d**) 500 °C for 900 s (15 min) following holding at 750 °C for 240 s. *F* is ferrite, *BF* is bainitic ferrite, *GB* is granular bainite, *RA*/*M* is retained austenite/martensite constituent, *M* is martensite and *P* is pearlite.

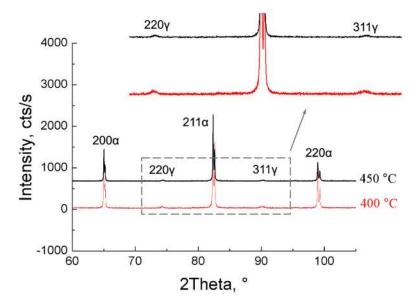


Figure 11. X-ray diffractograms after holding at 400 and 450 °C for 900 s (15 min).

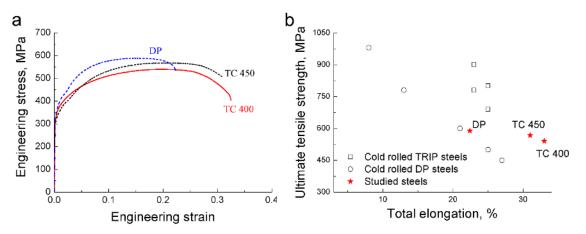
The critical driving force ( $G_{critical}$ ) for bainite transformation can be expressed by the following equation [31]:

$$G_{critical} = AT - B \tag{1}$$

where *A* and *B* are positive constants related to the steel chemical compositions [32,33] and *T* is the temperature. When the temperature decreased from ferrite-austenite two-phase region to bainite transformation region, the bainite formed during the holding and the carbon diffused from bainitic laths to austenite. Increasing the IBT temperature from 400 to 450 °C led to an increase in  $G_{critical}$ . This indicates that the transformation from austenite to bainite becomes more difficult with an increase in temperature, leading to a lower fraction of bainite. For a lower fraction of bainite formation, the content of carbon partitioning from bainitic ferrite laths into austenite was lower and thus the austenite became less enriched in carbon. Therefore, a less stable austenite transformed to martensite during quenching from 450 °C (c.f. Figure 9b,d) and a lower RA fraction was obtained. Similar results were reported in Ref. [34].

#### 3.4. Tensile Properties

Ferrite-martensite DP steels and multi-phase TRIP steels have been successfully produced in the laboratory using the modified strip casting technology proposed in the above sections. Figure 12 shows the engineering stress-engineering strain curves for a DP steel that has a ferrite fraction of 0.69  $\pm$  0.02 (designated as DP) and TRIP steels held at 400 and 450 °C (designated as TC 400 and TC 450, respectively). The curves indicate a typical continuous yielding behaviour observed in the multi-phase steels [35]. The DP steel showed a higher yield strength (YS) of 350 MPa, a higher UTS of 589 MPa and a lower TE of 22.4% than the TRIP steels. The TRIP steel held at 400 °C exhibited a lower UTS of 541 MPa and a larger TE of 33%, while the TRIP steel held at 450 °C had a higher UTS of 568 MPa and a lower TE of 31%. These two TRIP steels have similar YS of 332 (TC 400) and 328 (TC 450) MPa, respectively. The slightly larger TE in the sample held at 400 °C was probably ascribed to a higher fraction of RA for this processing condition (5.2% vs. 4.2%), leading to a stronger TRIP effect [8,36].



**Figure 12.** (a) Engineering stress-engineering strain curves and (b) comparison of mechanical properties for the studied steels and DP and TRIP steels conventionally cold/hot rolled in the steel industry [37].

The tensile properties obtained in the present study are comparable to the properties obtained in industrially cold rolled DP and TRIP steels (Figure 12b). The studied DP steel showed a little lower UTS than DP 590, however, together with a little larger TE. Obviously, for the studied TRIP steels, the TE is large enough, whereas the UTS is much lower in comparison with TRIP 690. Although a lower strength is obtained in the present study, the results indicate a feasibility to produce DP and TRIP steels using the modified strip casting. Recent studies have shown that the improvement of tensile properties can also be achieved through the deformation in the austenite temperature region followed by accelerated cooling and warm deformation in the ferrite temperature region, leading to

finer microstructures [38]. In addition, the microalloying with niobium, vanadium and molybdenum can also improve the tensile strength via precipitation and solid solution strengthening [39,40]. Thus, further research on the enhancement of tensile properties in DP and TRIP steels produced by the modified strip casting can be dedicated to the investigation of effects of thermo-mechanical control process and alloying.

#### 4. Conclusions

The modified strip casting with the addition of a continuous annealing stage was shown to be a promising technology for producing ferrite-martensite DP and multi-phase TRIP steels. The initial ferrite-pearlite microstructure was obtained via the adjustment of holding temperature during the first step of heat treatments of the modified strip casting. Following this, to produce DP steels, various temperatures and times of holding during inter critical annealing can result in different fractions of ferrite and martensite; to produce TRIP steels, at first ~50% ferrite should be obtained during inter critical annealing and bainite and retained austenite, together with a small amount of martensite, can be formed as a result of isothermal bainite transformation followed by cooling to room temperature.

The tensile strength was lower but the ductility was higher, in the studied steels than those in the conventionally produced DP and TRIP steels. Further enhancement of mechanical properties in strip cast steels can be achieved via the introduction of thermo-mechanical control process and microalloying, leading to the strengthening from grain refinement and precipitation. These require further investigation. The results presented in this work indicate a strong feasibility to produce homogenous microstructures of DP and TRIP steels in industry using the novel technology of the modified strip casting.

**Author Contributions:** E.V.P. and A.G.K. conceived the idea. Z.X. carried out the experiments. Y.Z. wrote the draft. All the authors reviewed this paper.

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