

## Identification of Retained Austenite, Ferrite, Bainite and Martensite in the Microstructure of TRIP Steel

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### Abstract

Transformation induced plasticity (TRIP) steels have a vast application in automotive industry because of their high strength, high ductility and hence excellent energy absorption capacity. These characteristics of TRIP steels are due to the existence of retained austenite in their microstructures in the ambient temperature, which transforms to the martensite phase during deformation. The microstructure of TRIP steel contains various phases and in the past-published studies mainly the volume fraction of retained austenite was investigated and there is not a quantitative comprehensive investigation about all phases in the microstructure of this steel. The main goal in this study is a comprehensive qualitative and quantitative investigation in various phases of TRIP steel microstructure. Therefore, a TRIP steel with chemical composition of 0.2C+ 1.43Si+ 1.58Mn was produced and its complicated microstructure which contained ferrite, bainite, martensite and retained austenite was investigated using X-Ray Diffractometry (XRD), optical microscopy (OM), field emission scanning microscopy (FE-SEM) and Electron backscatter diffraction (EBSD). The OM and FE-SEM results were used only to qualitative studies and identification of the morphologies of the phases but the EBSD results and functions were used to qualitative and quantitative studies. The volume fractions of retained austenite, ferrite+bainite and martensite phases were calculated and the amounts of 11%, 82% and 7% were obtained, respectively. The volume fraction of retained austenite was also measured with XRD and the amount of 14.3% was obtained.

*Keywords:* TRIP steel; XRD, FE-SEM; EBSD; Retained austenite.

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### 1. Introduction

The quest for improving vehicle fuel economy through weight reduction has motivated the development of advanced high-strength steels (AHSSs). In transformation- induced plasticity (TRIP)-assisted steels, ductility is improved through the strain-induced martensitic transformation of retained austenite to martensite <sup>1</sup>, making them good AHSS candidates. Although TRIP-assisted steels consist of complex multiphase microstructures, one of their key characteristics is the fraction of retained austenite available for the ductility-enhancing martensitic transformation during plastic deformation. Due to the low alloy con-

tent of these steels, the stability of the retained austenite at room temperature is mostly controlled through its level of carbon enrichment, which in turn can be manipulated through judicious design of the steel's phase constitution via heat treatments <sup>2</sup>.

The microstructure of TRIP steel can be obtained through a two- stage heat treatment processing route. First, the material is annealed between  $A_{c1}$  and  $A_{c3}$  to dissolve the original microstructure. This step is known as intercritical annealing (IA). After the IA, the steel is rapidly cooled to a temperature between martensite start temperature ( $M_s$ ) and bainite start temperature ( $B_s$ ), where bainite would form. This step is also known as bainitic isothermal transformation (BIT) <sup>3-5</sup>. In the open literature, some studies have examined the effects of IA and BIT on the microstructure and mechanical properties of this class of materials <sup>2, 6-10</sup>.

The microstructure of TRIP steel has been investigated in previous studies <sup>6, 10, 11</sup>. Shena et al. <sup>10</sup> investigated the volume fraction and morphology of retained austenite using SEM and EBSD. Also, they studied the effect of retained austenite volume fraction on the mechanical properties of TRIP steel. Blonde et al. <sup>11</sup>

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applied a high energy X-ray diffraction to measure the retained austenite and then studied the mechanical stability of this phase. In another work Seong et al.<sup>12)</sup> provided a review about quantitative study of phases in TRIP steels by means of neutron diffraction. They focused on retained austenite in this work and did not consider other phases.

Also, EBSD was used to measure bainite in TRIP steel by Zaefferer et al.<sup>13)</sup> They applied the kernel average misorientation (KAM) function to distinguish the bainitic ferrite in microstructure of TRIP steel. Some researchers have used the EBSD for martensite phase detection qualitatively and not quantitatively<sup>14)</sup>. By reviewing the past literature it is obtained that most of the researchers focused on the measurement of the volume fraction of retained austenite in TRIP steels and did not consider the volume fraction of the other phases. Moreover, there is not a comprehensive quantitative investigation on the volume fraction of various phases in TRIP steel. Therefore, in this study a comprehensive quantitative investigation on the microstructure of a 0.2C+1.43Si+1.68Mn TRIP steel was done using XRD, OM, FE-SEM and EBSD.

## 2. Materials and Methods

The chemical composition of TRIP steel contained 0.2 C, 1.68 Mn, 1.43 Si, 0.03 Cr, 0.01 Ni, 0.016 Al and 0.009 S (wt.%). The steel was prepared as a 5 kg ingot in an air induction furnace. The cast ingot was forged and small blocks were cut from forged stock. These blocks were soaked at 1200 °C for 1h, and hot rolled to a thickness of 4mm. Hot rolled plates were annealed at 900 °C for 1h to reduce the hardness for easier cold rolling process. The hot-rolled air annealed steel plates were cold rolled to achieve a final thickness of 1.2 mm.

The temperature of austenite to ferrite transformation ( $A_{c3}$ ) and the temperature at which the eutectoid reaction occurs ( $A_{c1}$ ) were calculated using Thermo-Calc database<sup>15)</sup> and the temperatures of 836 °C and 705 °C

obtained, respectively. To obtain the microstructure of TRIP steel a two-step heat treatment was conducted. The intercritical annealing was done at the temperature of 790 °C for 10 minutes. The obtained intercritical microstructure had special  $M_s$  and  $B_s$ , which were calculated by MUCG83 software and the amounts of 310 °C and 479 °C were obtained. After intercritical annealing, the steel sample was quenched to temperature of 350 °C (this temperature should be between  $M_s$  and  $B_s$ ) and was hold for 12 minutes at this temperature. Finally the steel sample was quenched in the water to room temperature.

The microstructure of TRIP steel was studied using OM, FE-SEM, EBSD and XRD. The Sample preparation for metallographic and microstructure investigation was performed according to the procedures provided in Table 1.

The samples for investigation with FE-SEM were etched in Nital 2% for 2-3 seconds. For OM various etchant are possible. It was obtained that the metabisulfite solution ( $Na_2S_2O_3 \cdot H_2O$  10 g +  $H_2O$  100 ml) is the best etchant solution and the all of the four phases can be determined using it. Using a metabisulfite solution as the etchant reveals the retained austenite, ferrite, and bainite/martensite phases in white, gray and black contrast, respectively<sup>3)</sup>.

The volume fraction of retained austenite was measured by XRD using Cu-K $\alpha$  radiation. A step scan within the  $2\theta$  interval between 40° and 120° at 0.2° was done.

Electron backscatter diffraction (EBSD) was performed with an EDAX/TSL EBSD system. The acceleration voltage during EBSD data collection was 12-15 kV, beam current 2.5-5.5 nA, the working distance 8 mm and the step size 20-60 nm. The TSL OIM software was used to analyze the EBSD output file. This file contains several automated functions for characterization of EBSD maps. The image quality (IQ) which describes the quality of an EBSD pattern. Grain Average IQ (GAIQ) which calculates the average IQ of the entire grain. kernel Average Misorien-

Table 1. Steps performed in sample preparation for metallography.

Preparation step	OM	FE-SEM	EBSD	XRD
Mounting	*	*		*
Grinding #220	*	*	*	*
Grinding #500	*	*	*	*
Grinding #1000	*	*	*	*
Grinding #4000	*	*		*
Polishing 1 $\mu$ m	*	*		*
Polishing OP-S			*	
Electropolishing			*	
Etching	*	*		*

tation (KAM) which is the misorientation between a point in the center of a predetermined kernel and the points in the perimeter of the kernel and Grain Average Misorientation (GAM) calculates the misorientation between neighboring points in a grain.

### 3. Results and Discussion

Fig. 1 shows the XRD pattern of the TRIP steel. After calculation of the phases according to ASTM E975-0<sup>16</sup>. It is obtained that volume fraction of retained austenite in the microstructure is 14.3% which means 85.7% of the microstructure contains the bcc structure. The XRD could determine only the bcc and fcc structures and could not determine the ferrite, martensite and bainite. Therefore OM, SEM and EBSD should be used to identify martensite and bainite.

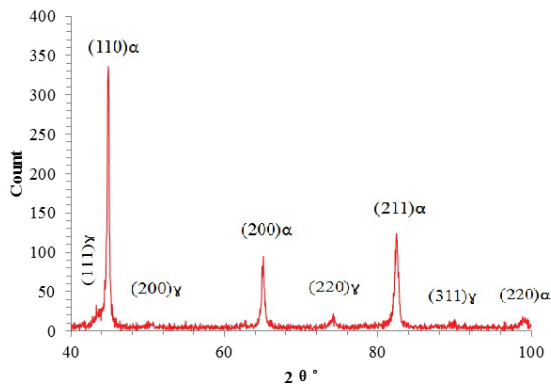


Fig. 1. XRD pattern of Studied TRIP steel.

Fig. 2 displays the OM of TRIP steel. Ferrite, bainite, austenite, and martensite can be seen in Fig. 2. The major phase which is observed in this figure is ferrite. The retained austenite is seen in white color and by block and thin film morphology. In TRIP steels bainite is a combined structure of lamellae of retained austenite and bainitic ferrite and this lamellar morphology of bainite is identified in the microstructure in Fig. 2 (red ellipses). It is possible that these regions are bainite, so that the elongated bright grains are austenite, and in-between the austenite grains, bainitic ferrite is located. EBSD was used to confirm this matter. The brown grains are the most probable to be martensite. In Fig. 2 some large bands of martensite are marked with rectangular. This martensite does not correspond to the findings with EBSD, where retained austenite was found with this morphology. This is related to the preparation method applied before OM characterization, which contains mechanical grinding and polishing, induces austenite to martensite transformation. In fact, the EBSD is the only useful method for quantification of the martensite in the BM microstructure.

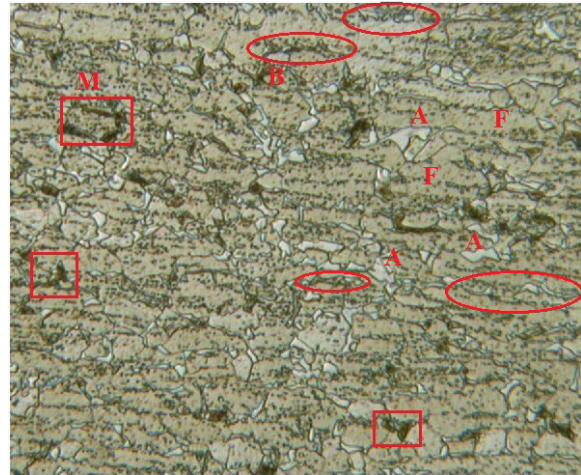


Fig. 2 OM microstructure of studied TRIP steel. A: Austenite, F: Ferrite, B: Bainite and M: Martensite.

Fig. 3 shows the FE-SEM micrograph of TRIP steel which is obtained after etching 2-4 second with naital 1%. FE-SEM micrograph has a very good resolution comparing to OM; hence, small details are much better resolved. Ferrite is present as the large etched grains with a similar morphology as the beige grains in the OM micrograph in Fig. 2. Bainite is obviously observed as the areas with lamellar structure containing bainitic ferrite and retained austenite (marked with ellipses in Fig. 3). Retained austenite is detected as un-etched grains with a mellow appearance. Martensite is recognized because it has a rough appearance. Only the rough appearance of martensite allows the separation between martensite and retained austenite in a SEM image. A helpful method was recommended to separate martensite and retained austenite, is to perform an annealing treatment (200-260 °C) prior to SEM investigation, which would reveal temper the martensite reveals its rough appearance<sup>17, 18</sup>. But, in present study, the martensite had a rough appearance normally. Still, it is not completely confirmed that the small grains comprise retained austenite, as indicated in the OM and SEM analysis. EBSD is the only method in which can actually prove this.

The EBSD IQ map and phase map are represented in Fig. 4a. The bcc phase has not identified in this map. But in fact, all of the grains not including the green grains are bcc.

The EBSD map indicates that the small grains are retained austenite. The volume fraction of retained austenite in EBSD map is 11% which is different to XRD results (14.3%). This deference is related to preparation method of these two methods. The bainitic ferrites have been formed between retained austenite which is shown in EBSD image. This morphology has been verified in OM and FE-SEM; EBSD verifies the existence of austenite in the lamellar structure.

Grains with lower IQ in Fig. 4a represent the martensite, but also some lamellae of retained austenite are seen nearby the martensite grain. In the martensite grains the lattice is distorted contains large density of dislocations which cause poor diffraction conditions and so reducing the IQ.

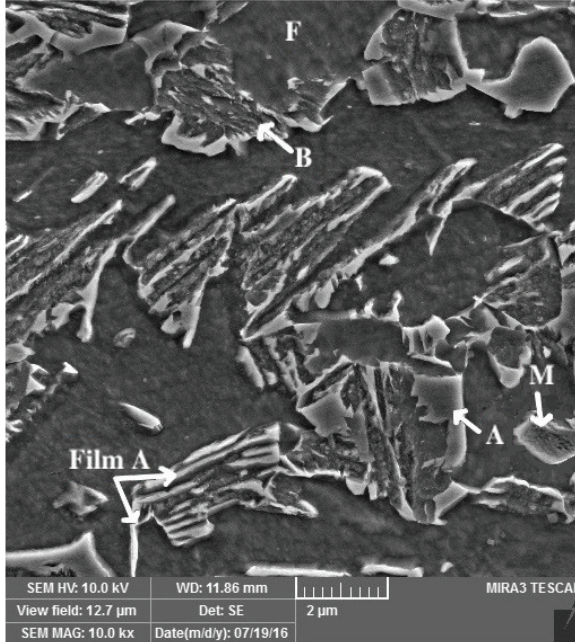


Fig. 3. FE-SEM micrograph of TRIP steel. A: Austenite, Film A: Film austenite, F: Ferrite, B: Bainite and M: Martensite.

Bainitic ferrite can be identified using its specific property. Heat treatment process of TRIP steel includes two stages: Firstly, the steel is heated up to intercritical region (790 °C in this study) to form ferrite and austenite and then follow an isothermal bainitic transformation in a lower temperature (350 °C in this study). During cooling, the growth of ferrite and bainitic ferrite occurs from the same austenite grains, but there is a small change in the growing mechanism, which will result in a minor orientation gradient from ferrite to bainitic ferrite<sup>19)</sup>. This orientation gradient can be measured by EBSD and used for the difference between ferrite and bainitic ferrite.

To study the possible orientation gradient among ferrite and bainitic ferrite five point-to-origin lines were drew (lines given in Fig. 5a). Fig. 5b displays the continuation of an orientation gradient between ferrite and bainitic ferrite. This orientation gradient confirms the existence of bainitic ferrite in-between the retained austenite lamellae.

Kernel Average Misorientation (KAM) is an EBSD function, which has been previously used as a method to recognize bainite in steel<sup>13)</sup> KAM determines local orientation gradients between a point and its inclosing points, in a kernel of a predetermined size. Therefore, KAM can be used to identify the orientation gradient,

which is present between bainitic ferrite and ferrite in TRIP steel. In Fig. 4b an EBSD map is shown that includes phase map and the red regions show bcc points with a KAM value in the range of 0.7-5°. This figure is in good agreement with result of Fig. 5.

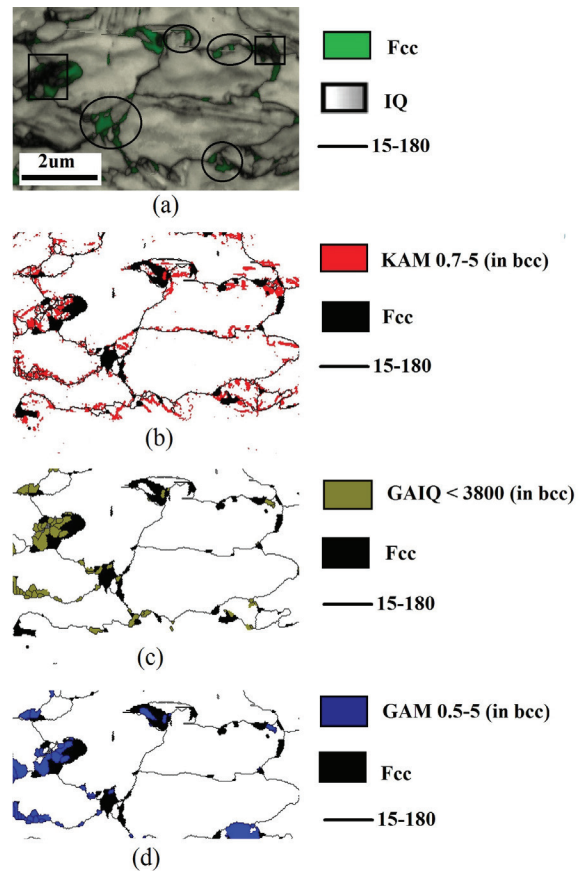


Fig. 4. (a) IQ and Phase map, (b) KAM and phase map, (c) GAIQ and phase map and (d) GAM and phase map.

The boxes in the Fig. 4a represent the martensite phase. It should be noted that, martensite can be wrongly identified as bainitic ferrite if KAM is the only method to identify bainitic ferrite. Therefore, it is very important that the grain boundaries, IQ and orientation gradients be controlled before final concluding. In conclusion, the KAM function is a good method to identify bainitic ferrite, but since martensite is showing the same behavior in KAM analysis, other methods must be used to determine the martensite. Martensite has some typical properties that differ from bainitic ferrite and they can be used for an unmistakable separation of them. Martensite grains have low IQ, which is shown in Fig. 4a. The IQ of bainitic ferrite is similar to neighboring ferrite grains. Another property of martensite is the existence of numerous low angle misorientations. The structure of lath martensite in steel includes packets, blocks, sub-blocks and laths (Fig. 6)<sup>20)</sup>.

The boundaries of Sub-block are known to be low

angle boundaries (LAGBs), and so are the lath boundaries, albeit with smaller misorientation angles<sup>20</sup>). The low IQ and existence of LAGBs in martensite can be used to recognize the martensite grains in EBSD. Two functions can be used to detect the martensite grain. One of them is the Grain Average Misorientation (GAM) function which calculates the misorientation between neighboring points in a grain. In the whole grain, the average misorientation amount is calculated and assigned to all points within the grain. Also, the Grain Average Image Quality (GAIQ) can be used to determine the martensite, which calculates the average IQ of the grain.

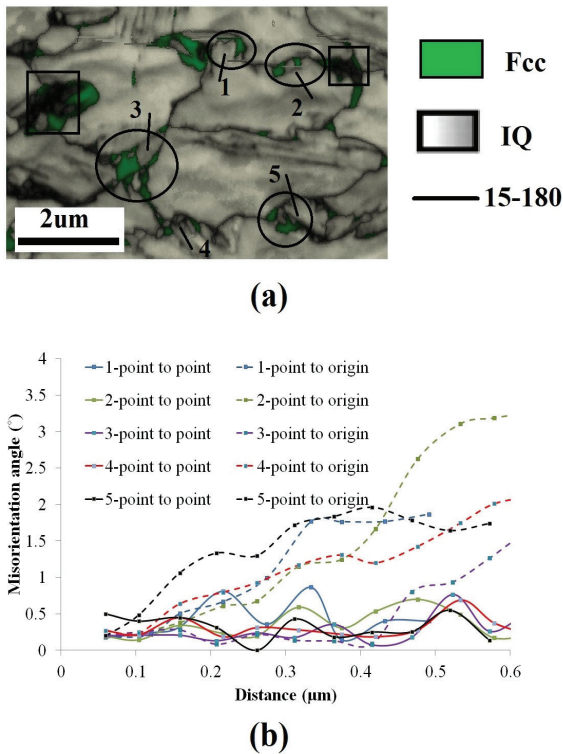


Fig. 5. (a) IQ and phase map with bainite (ellipses) and martensite (rectangular) positions and (b) point to point and point to origin orientation gradients.

In Fig. 4a bainite and martensite are shown in ellipses and boxes, respectively. Bainitic ferrite is

identified in Fig. 4b using the KAM function but it is clear that also the martensite is identified by KAM. To identify the martensite the GAM and GAIQ functions are used and the results are shown in Fig. 4c and d. By comparing the KAM and each of GAM and GAIQ the positions of bainite can be obtained. The volume fraction of martensite using GAM and GAIQ was obtained 6.5% and 7%, respectively.

Although the position of the bainite were determined by EBSD, the volume fraction of this structure could not be identified. Firstly, because bainite is a combined structure of bainitic ferrite and retained austenite, and it is not possible to distinguish between austenite grains and bainite. Secondly, bainitic ferrite cannot be quantified, because the KAM method only identifies the transition region between ferrite and bainitic ferrite. Therefore the sum of bainite and ferrite volume fractions was calculated 82%. Table 2 represents the ability and limitation of each method which was used in this paper. In this table the possibility of qualitative or quantitative investigation in each phase by each method is shown.

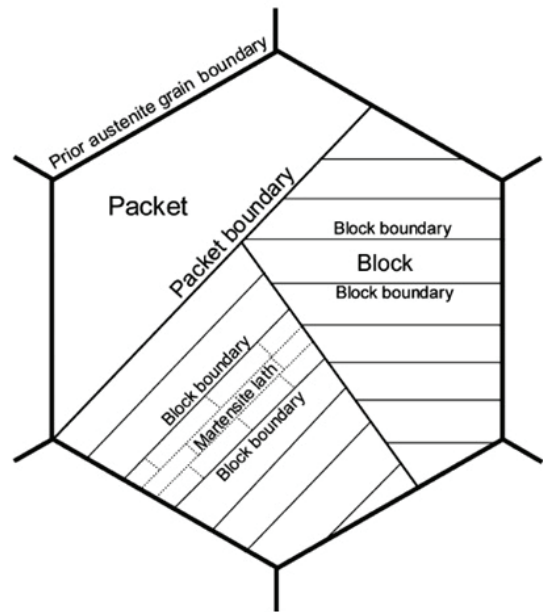


Fig. 6. lath martensite structure which includes packets, blocks and lathes.

Table 2. The possibility of qualitative or quantitative investigation in each phase by each method.

phase \ Method	OM	FE-SEM	EBSD				XRD
			IQ	KAM	GAM	Phase map	
austenite	qualitative	qualitative	-	-	-	qualitative	qualitative
ferrite	qualitative	qualitative	-	-	-	qualitative	-
bainite	qualitative	qualitative	-	qualitative	-		-
Ferrite+bainite	-	-	qualitative		-		-
martensite	qualitative	qualitative	qualitative	-	qualitative		-

#### 4. Conclusions

The microstructure of a TRIP steel was investigated using XRD, OM, FE-SEM and EBSD and the following results obtained:

- The microstructure contains four phases: ferrite, bainite, martensite and retained austenite
- The volume fraction of retained austenite was calculated using XRD and the amount of 14.3% obtained.
- The OM and FE-SEM results were used only to qualitative studies and identification of the morphologies of the phases.
- The EBSD results and functions were used to qualitative and quantitative studies. The volume fractions of retained austenite, ferrite+bainite and martensite phases were calculated and the amounts of 11%, 82% and 7% were obtained, respectively.

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