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The effects of double intercritical annealing on microstructure and mechanical properties of 0.107C-2.39Mn-0.453Si dual phase steel

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Abstract

In this study, double intercritical annealing was applied to modify the mechanical properties of 0.107%C-2.39%Mn-0.453%Si dual phase steel. The effects of the double intercritical annealing (DIA) method were investigated via microstructure observation and tensile test, and then compared with the single intercritical annealing (SIA) method. By increasing the intercritical annealing temperature, yield and tensile strengths increase while ductility decreases primarily due to the increase of martensite fraction. DIA leads to a slight reduction of the ferrite size and the martensite fraction regardless of the intercritical annealing temperature. Tensile results showed that DIA increases ductility without losing significant amount of strength. The outcome implies that the DIA method can be used to modify the mechanical properties of DP steels without adding excessive complexity to the process.

1. Introduction

Dual phase (DP) steel is a type of advanced high strength steel (AHSS) which is widely used in automotive applications [1-4]. Although DP steel is a low-carbon steel which is generally used for low strength applications, the combination of ferrite and martensite microstructures achieved via a heat treatment process allows DP steel to display high mechanical properties e.g., high work hardening, tensile strength and ductility [5,6].

Nevertheless, due to the complexity of the microstructure of DP steel, many factors play a role on the mechanical properties. These factors include martensite fraction [7], grain size [5], phase distribution [8,9], and phase morphology [10]. For many years, there have been studies that try to improve the mechanical properties of DP steel by tailoring these factors, which result in a wide range of mechanical properties from low-moderate strength of 400 MPa to 500 MPa to high strength of 1000 MPa or more [5-15].

As the unique microstructure of DP steel is produced via heat treatment, the modification of mechanical properties of DP steel is mainly achieved via the heat treatment process [5-15]. The typical heat treatment process for DP steel consists of two stages, austenitizing and intercritical annealing. By adjusting the variables such as time, temperature, and cooling rate of these two processes, martensite fraction, grain size, phase distribution, and phase morphology can be optimized. Consequently, the improvement of mechanical properties can be accomplished.

Out of many heat treatment methods, multiple cycles annealing [16-18], is incredibly interesting. It was reported that grain size reduction was achieved by cyclic intercritical annealing which enhances the mechanical properties [16-18]. Moreover, a further reduction of grain size can be accomplished by introducing cold deformation between heat treatment steps [5,8,9,12]. However, the introduction of cold deformation introduces the difficulty into the process, therefore, it is not considered in this work.

The purpose of this experiment is to study the effects of the additional intercritical annealing step on the microstructure and mechanical properties of 0.107%C-2.39%Mn-0.453%Si dual phase steel. In addition, the effects of different intercritical annealing temperature were also investigated. The mechanical properties were studied via tensile test and discussed in conjunction with the observed microstructure.

2. Experimental Procedures

2.1 Materials and specimen preparation

The material used in this study is dual phase steel sheets with the composition as shown in Table 1. The samples were cold rolled to the thickness of 1.4 mm and were cut to the square shape of 30 cm × 30 cm. Then, they were machined into tensile test specimens according to the subsize specimen from ASTM E8 standard (Figure 1).

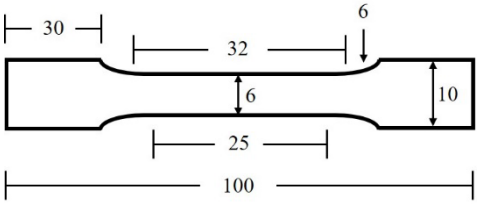


Figure 1. Schematic drawing of subsize tensile specimen (ASTM E8 standard).

Table 1. Chemical composition of steel used in this study.

Chemical composition (wt%)							
C	**Mn**	**Si**	**Cr**	**Mo**	**Ni**	**V**	**Cu**
0.107	2.39	0.453	0.023	0.0072	0.021	0.0015	0.014

2.2 Heat treatment procedures

In order to select the austenitizing and intercritical annealing temperatures used for the heat treatment process, A_1 and A_3 temperatures of the studied steel were calculated based on their chemical composition using equations (1) – (4) [19]:

Hougardy;

$$A_1 = 739 - 22C - 7Mn + 2Si + 14Cr + 13Mo - 13Ni \quad (1)$$

Trzaska;

$$A_1 = 739 - 22.8C - 6.8Mn + 18.2Si + 11.7Cr - 15Ni - 6.4\,Mo - 5V - 28Cu \quad (2)$$

Hougardy;

$$A_3 = 902 - 255C - 11Mn + 19Si - 5Cr + 13Mo - 20Ni + 55V \quad (3)$$

Trzaska;

$$A_3 = 937.3 - 224.5C - 17Mn + 34Si - 14Ni + 21.6Mo + 41.8V - 20Cu \quad (4)$$

Two different equations for each temperature were used in order to minimize the errors from the difference in chemical composition. The calculated values for A_1 from equations (1) and (2) are 721°C and 728°C, and A_3 from equations (3) and (4) are 857°C and 838°C, respectively.

To get a fully austenitic structure, the austenitizing temperature needs to be higher than A_3, thus, 950°C was selected. On the other hand, the intercritical annealing temperature must be done between A_1 and A_3 temperatures to obtain a mixture of ferrite and austenite phases before quenching. Furthermore, the variation of intercritical annealing temperature leads to the change in phase fraction which affects the mechanical properties. According to the calculation, the temperatures between 730°C and 830°C with 50°C intervals were picked for the intercritical annealing and the effects of intercritical annealing temperature were observed.

The heating profile for this experiment is shown in Figure 2. Firstly, all machined tensile specimens were austenitized in the box furnace at 950°C for 10 min, then, water quenched to room temperature (referred as "austenitized"). After that, they were intercritical annealed at 3 different temperatures for 3 min and water quenched (referred as "single intercritical annealing (SIA)"). Lastly, some of the intercritical annealed specimens were intercritical annealed again for 3 minutes at the similar temperature as the previous step, then water quenched (referred as "double intercritical annealing (DIA)"). Hereafter, the specimens are named regarding to its intercritical annealing temperature and heat treatment condition for example the specimen single intercritical annealed at 730°C is called "730SIA".

2.3 Tensile testing

In total, 7 conditions (Table 2) of heat-treated specimens were tensile tested. The test was operated at room temperature with the crosshead speed of 0.008 mm/sec during the elastic region and then changed to 0.16 $mm{\cdot}s^{-1}$ after plastic deformation started until failure for all specimens. Furthermore, each specimen condition was tested at least 3 times and the average values were used to discuss their mechanical properties and behavior.

2.4 Material characterization

The tensile tested specimens were cut from the grip part in order to observe the undeformed microstructure after each heat treatment condition. The cut specimens were grounded with sand paper up to 2000 grit. Lastly, they were polished with alumina powder and etched with Nital solution 2 vol%.

Optical microscope (OM) was used to observe the overall microstructure of all specimens in the rolling direction. Grain size measurement and phase fraction were determined from the optical micrographs using ImageJ software. Scanning electron microscope (SEM), JEOL JSM-6610LV, was used to further investigate the details of the microstructure and energy dispersive spectroscopy (EDS) was utilized in line scan mode to detect the element partitioning between each phase.

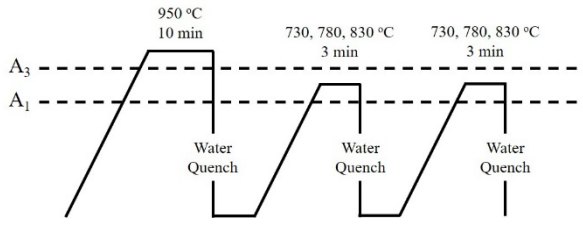


Figure 2. Schematic drawing of the heat treatment used in this study.

Table 2. Tensile test specimen's heat treatment conditions.

Condition	Name	Heat treatment condition
1	Austenitized	950°C, 10 min
2	730SIA	950°C, 10 min + 750°C, 3 min
3	780SIA	950°C, 10 min + 780°C, 3 min
4	830SIA	950°C, 10 min + 830°C, 3 min
5	730DIA	950°C, 10 min + 750°C, 3 min + 750°C, 3 min
6	780DIA	950°C, 10 min + 780°C, 3 min + 780°C, 3 min
7	830DIA	950°C, 10 min + 830°C, 3 min + 830°C, 3 min

3. Results and discussion

3.1 Microstructures

The micrographs of austenitized specimen are shown in Figure 3. The fully martensitic structure was observed in the optical micrograph (Figure 3(a)) and the martensite was clarify as lath type according to the SEM micrograph (Figure 3(b)). Furthermore, the rolling microstructure was completely destroyed, therefore, the microstructure before intercritical annealing was lath martensite packets, which formed inside equiaxed prior austenite grains.

Figure 4 represents the micrographs of SIA specimens near surface (Figure 4(a), 4(c) and 4(e)) and at the center of thickness (Figure 4(b), 4(d) and 4(f)). Every specimen shows the mixture of ferrite and martensite structure in which the martensite fraction is increased with increasing intercritical annealing temperature. Moreover, two shapes of ferrite, granular and lath, were observed in all specimens. Granular ferrite was observed mainly near surface of the 730SIA and at all areas of the 780SIA and 830SIA. In contrast, lath ferrite was observed primarily at the center for all specimens. Granular ferrite is ferrite which nucleates at the interface boundary due to recrystallization during intercritical annealing. On the other hand, lath ferrite originates from the reversion of lath martensite. The microstructures imply that during SIA, recrystallization of ferrite is more complete as the temperature increases.

Furthermore, while the microstructures near surface and at the center of 730SIA (Figure 4(a-b)) display considerably different morphologies, 780SIA (Figure 4(c-d)) and 830SIA (Figure 4(e-f)) do not. This suggests that the inhomogeneity occurs after SIA when the intercritical annealing temperature is low and disappears as the temperature increases. The result aligns with the observed ferrite morphology. Compared to the other works [5-15] the holding time during intercritical annealing in this study is short. Therefore, as the temperature is also low, the provided energy is insufficient to complete the recrystallization in all areas especially at the center thus, an inhomogeneous microstructure occurs for the 730SIA.

The microstructures of DIA specimens near surface ((a), (c) and (e)) and at the center ((b), (d) and (f)) are shown in Figure 5. Similar to the SIA specimens, the mixture of ferrite and martensite was observed and the martensite fraction is increased with increasing intercritical annealing temperature. Overall, the phase morphology of DIA and SIA specimens is similar for the intercritical annealing temperature of 780°C and 830°C. However, the microstructure of 730DIA (Figure 5(a-b)) are more comparable between near surface and at center compared to the 730SIA (Figure 4(a-b)) as more granular ferrite at the center and smaller ferrite size near surface were observed. This implies that the homogeneity of microstructure was improved after DIA. Nevertheless, the ferrite size and phase fraction data are needed to be discussed to clarify the differences in the obtained microstructure between DIA and SIA processes.

The ferrite grain area (representing grain size) of heat-treated specimens is shown in Table 3. For all conditions, the average ferrite size is smaller than 100 μm^2 although the size discrepancy is vast. Overall, the ferrite size of specimens intercritical annealed at 730°C is the largest, followed by 830°C and 780°C, respectively, for both SIA and DIA. Still, the ferrite size is slightly smaller for DIA ones. The larger ferrite size for specimens intercritical annealed at 830°C compared to 780°C is due to the grain growth phenomena at higher temperature. Furthermore, the smaller ferrite size in DIA specimens attributes to the repetitive recrystallization during the second intercritical annealing which reduces the ferrite size.

Furthermore, the ferrite size of 730SIA shows the largest difference value between near surface and center areas while the other conditions are comparable. This result is consistent with the observed microstructure (Figure 4-5). One of the reasons for this discrepancy in the ferrite size of 730SIA is regarded to the amount of two ferrite shapes in each area. As granular and lath ferrites are different in size, lath shape is smaller, having a larger amount lath ferrite in the center and mainly granular ferrite near surface results in this large discrepancy. For the other conditions, a more homogeneous microstructure was observed with a less discrepancy of ferrite size between near surface and center areas.

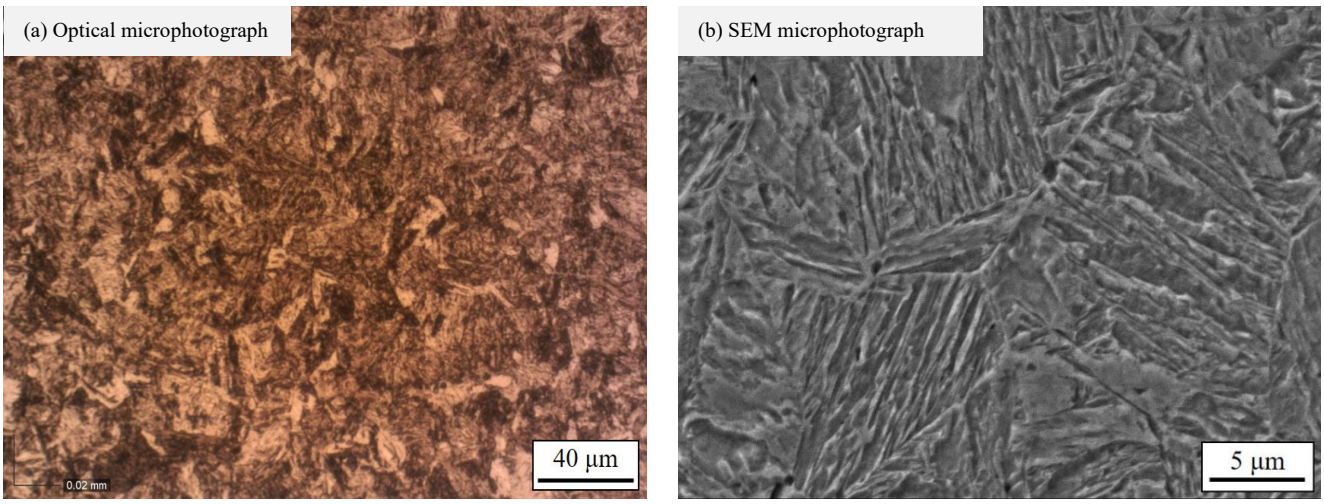


Figure 3. The microstructures of austenitized specimen.

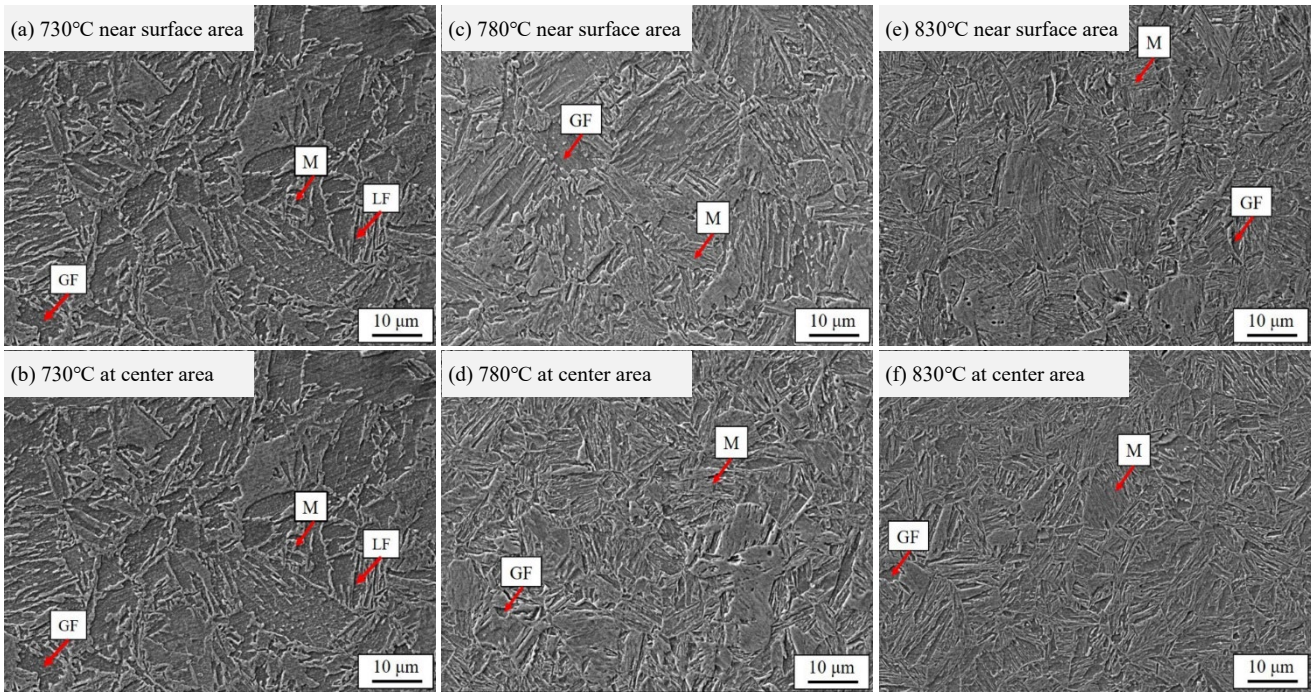


Figure 4. SEM micrographs of the single intercritical annealed specimens. LF: Lath ferrite, GF: granular ferrite, M: Martensite.

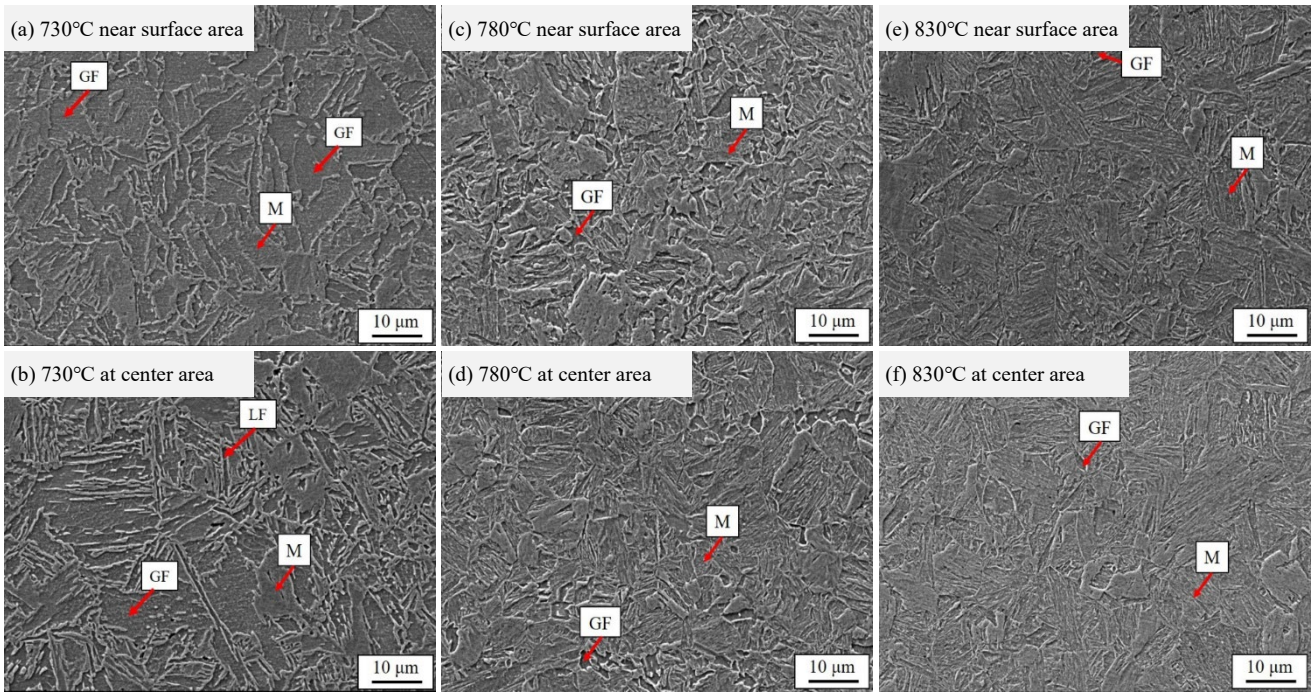


Figure 5. SEM micrographs of the double intercritical annealed specimens. LF: Lath ferrite, GF: granular ferrite, M: Martensite.

Table 3. Ferrite grain area of the intercritical heat-treated specimens.

Specimen name	Ferrite area – surface (µm²)		Ferrite area – center (µm²)		Overall (µm²)	
	Average	SD	Average	SD	Average	SD
730SIA	96.63	80.08	58.15	55.58	77.39	71.46
780SIA	48.04	61.87	36.86	35.04	42.45	50.51
830SIA	43.08	31.55	56.05	41.97	49.57	37.63
730DIA	66.22	51.55	54.36	48.07	60.29	50.11
780DIA	46.59	48.11	35.99	30.18	41.29	40.44
830DIA	47.01	38.92	48.46	39.58	47.74	39.19

Overall, the martensite fraction increases with the increase of intercritical annealing temperature (Figure 6), which is similar to other studies [20-22]. For all temperature, DIA results in a lower martensite fraction than SIA and a significant decrease of martensite fraction was observed in the 730DIA and 780DIA. This suggests that the change in phase fraction of ferrite and austenite during the first intercritical annealing at 730°C and 780°C is incomplete and DIA pushes forward the microstructure evolution to reach more equilibrium state.

Unlike the ferrite size, both the 730SIA and 730DIA specimens show a large difference of martensite fraction between near surface and center areas. This implies that at the intercritical annealing temperature of 730°C, the homogeneity is not achieved even after DIA. The reason is the insufficient amount of energy from short holding time, 3 min, at the low temperature of 730°C which cannot drive the microstructure evolution at the center to reach the similar morphology as near surface.

From the above results, the microstructure evolution during the SIA and DIA processes can be explained. For SIA, during the first intercritical annealing, fully martensitic structure changes into a mixture of ferrite and austenite. Then, after water quench, austenite transforms to martensite while ferrite is not affected thus, dual phase structure is achieved at room temperature after SIA. However, for the 730SIA and 780SIA, the phase fraction is not equilibrium and the higher martensite fraction than equilibrium state was observed. Also, for the 730SIA, the recrystallization process is unfinished especially at the center area which experienced shorter time at holding temperature. As a result, a higher fraction of non-recrystallized phase, lath ferrite, was observed at the center.

Afterward, for DIA, the second intercritical annealing is performed to cause repetitive recrystallization for grain size reduction. The dual phase structure after SIA changes back to ferrite and austenite at holding temperature then dual phase structure with smaller ferrite size is achieved via water quenching. After the second intercritical annealing, the phase fraction of the 730DIA and 780DIA becomes more equilibrium (lesser martensite fraction than SIA). However, for the 730DIA, the homogeneous morphology between near surface and the center areas is yet to achieved even though the homogeneity increases. Consequently, the smaller ferrite size and lower martensite fraction were observed after DIA, and the differences between SIA and DIA are more pronounced at lower temperature.

The SEM images with EDS line scan profiles of the 830SIA and 830DIA are presented in Figure 7. The results show that no significant partitioning of the substitutional alloying elements, Mn and Si, was observed while some fluctuation was noticed only for carbon profile. This suggests that even at the highest intercritical annealing temperature used in this study, 830°C, the diffusion of substitutional alloying elements is lacking and only the interstitial element, carbon, diffuses. The cause is the short holding time in this experiment which does not allow the diffusion of substitutional elements. As a result, phase transformation during SIA and DIA is mainly influenced by carbon diffusion.

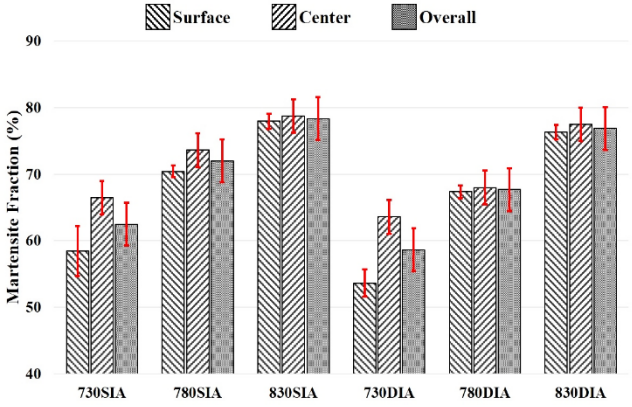
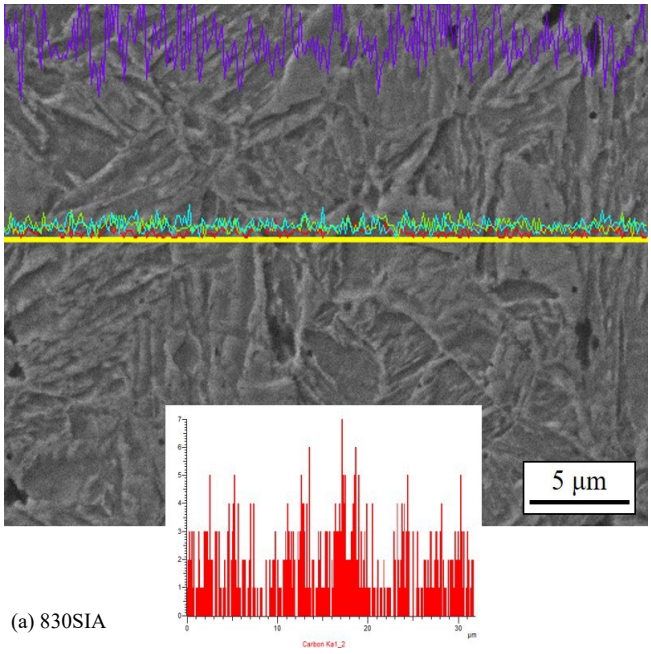
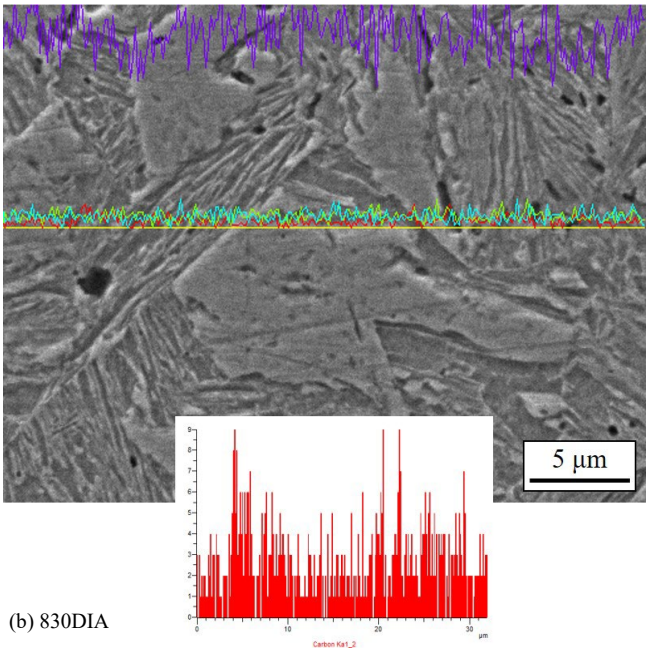


Figure 6. Martensite fraction of the intercritical heat-treated specimens.



(a) 830SIA



(b) 830DIA

Figure 7. Microstructures at the center of thickness with EDS line scan including carbon data of specimens.

3.2 Mechanical properties

The engineering stress strain curves for every heat treatment condition and their average values are displayed in Figure 8 and Table 4, respectively. The austenitized specimen exhibits high yield and tensile strengths with low elongation and virtually no post yield elongation. In case of SIA specimens, the yield and tensile strengths increases with increasing intercritical annealing temperature, while the elongation decreases. Tensile properties of SIA specimens vary from UTS around 700 MPa with 16% elongation (730SIA) to UTS of 1160 MPa with 7% elongation for the 830SIA. Compared to the austenitized specimen, the ductility of SIA specimens is higher. Moreover, the yield and tensile strengths of the 830SIA match the austenitized specimen but with larger post necking elongation. This suggests that the mechanical properties of DP steel can be modified through intercritical annealing process.

For the DIA specimens, the mechanical properties change from the SIA ones (Figure 8). As intercritical annealing temperature increases, the difference on mechanical properties between DIA and SIA specimens decreases. Overall, the reduction of strength values was observed in the DIA specimens compared to the SIA ones. The decrease in strength values is minor in the specimens intercritical annealed at 730°C and 830°C but significant for 780°C. On the other hand, the increase of elongation after DIA compared to SIA is significant for the intercritical annealing temperature of 730°C while lessens for 780°C and stayed the same for 830°C.

The total work hardening (sees from UTS – YS values) shows that the increase in strength after yielding is similar for both the SIA and DIA conditions at the same temperature (Table 4). However, the strain hardening curves, Figure 9, reveals that during plastic deformation region, strain hardening is slightly lower at low strain then became higher. Lastly, it maintains a positive value up to larger true strain for the 730DIA and 780DIA compared to their counterpart SIA condition. As for the 830DIA and 830SIA (Figure 9(c)), the difference in strain hardening is negligible. This is in agreement with the displayed uniform elongation of the 830DIA and 830SIA. Conversely, the uniform elongation increases for the 780DIA and largely enhances for the 730DIA when compared to their SIA equivalent. The results imply that DIA performed at a lower intercritical annealing temperature leads to a larger alteration on tensile behavior whereas the effect lessens at higher intercritical annealing temperature.

Table 4. Yield strength, tensile strength, uniform elongation and, elongation to failure of specimens heat-treated with various conditions.

Specimen	Yield strength (MPa)	Tensile strength (MPa)	Uniform elongation (%)	Elongation to failure (%)	UTS-YS (MPa)
Austenitized	897.54 ± 7.66	1164.20 ± 8.56	2.83 ± 0.44	3.98 ± 1.16	266.66
730SIA	503.50 ± 9.72	704.33 ± 4.33	10.37 ± 0.49	16.64 ± 0.72	200.83
730DIA	478.50 ± 2.78	695.57 ± 3.43	14.35 ± 0.48	21.98 ± 0.81	217.07
780SIA	551.20 ± 14.08	964.31 ± 9.75	7.70 ± 0.39	13.37 ± 0.66	413.11
780DIA	487.92 ± 8.96	899.10 ± 5.03	8.84 ± 0.29	15.06 ± 0.26	411.18
830SIA	894.29 ± 47.92	1163.74 ± 35.93	3.52 ± 0.23	7.26 ± 1.08	269.45
830DIA	908.01 ± 47.92	1171.83 ± 5.45	3.37 ± 0.03	7.55 ± 0.36	263.82

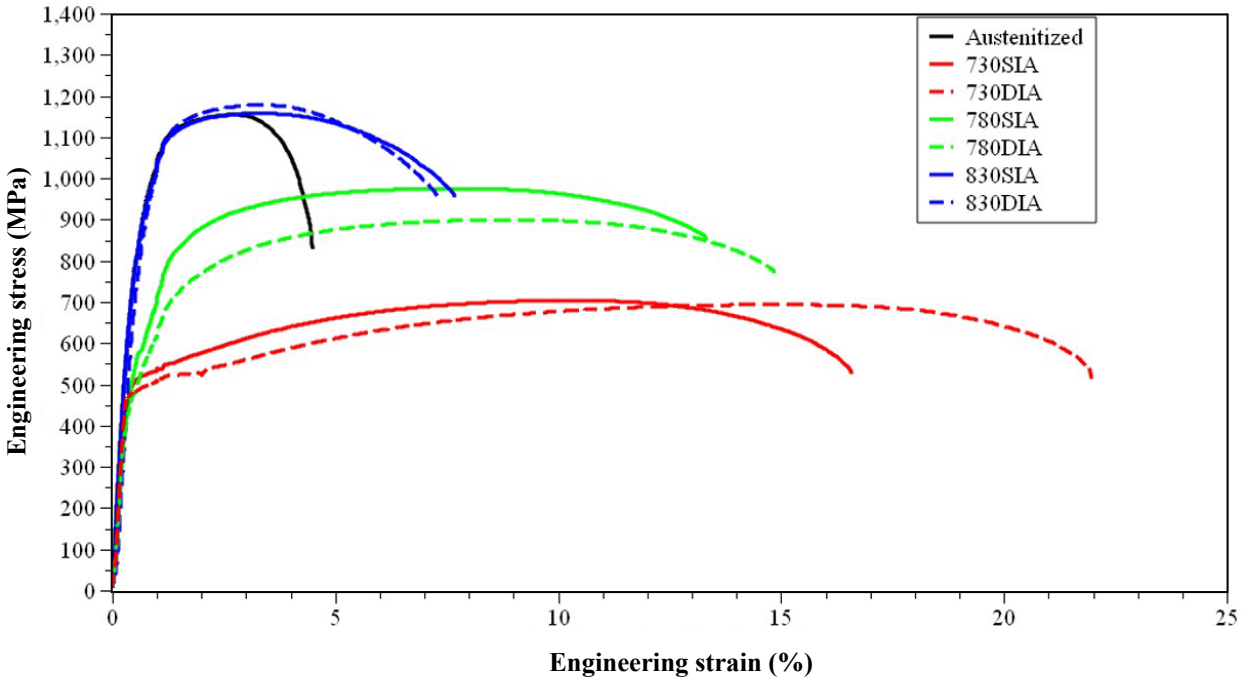


Figure 8. Engineering stress-strain curves of specimens heat-treated with various conditions.

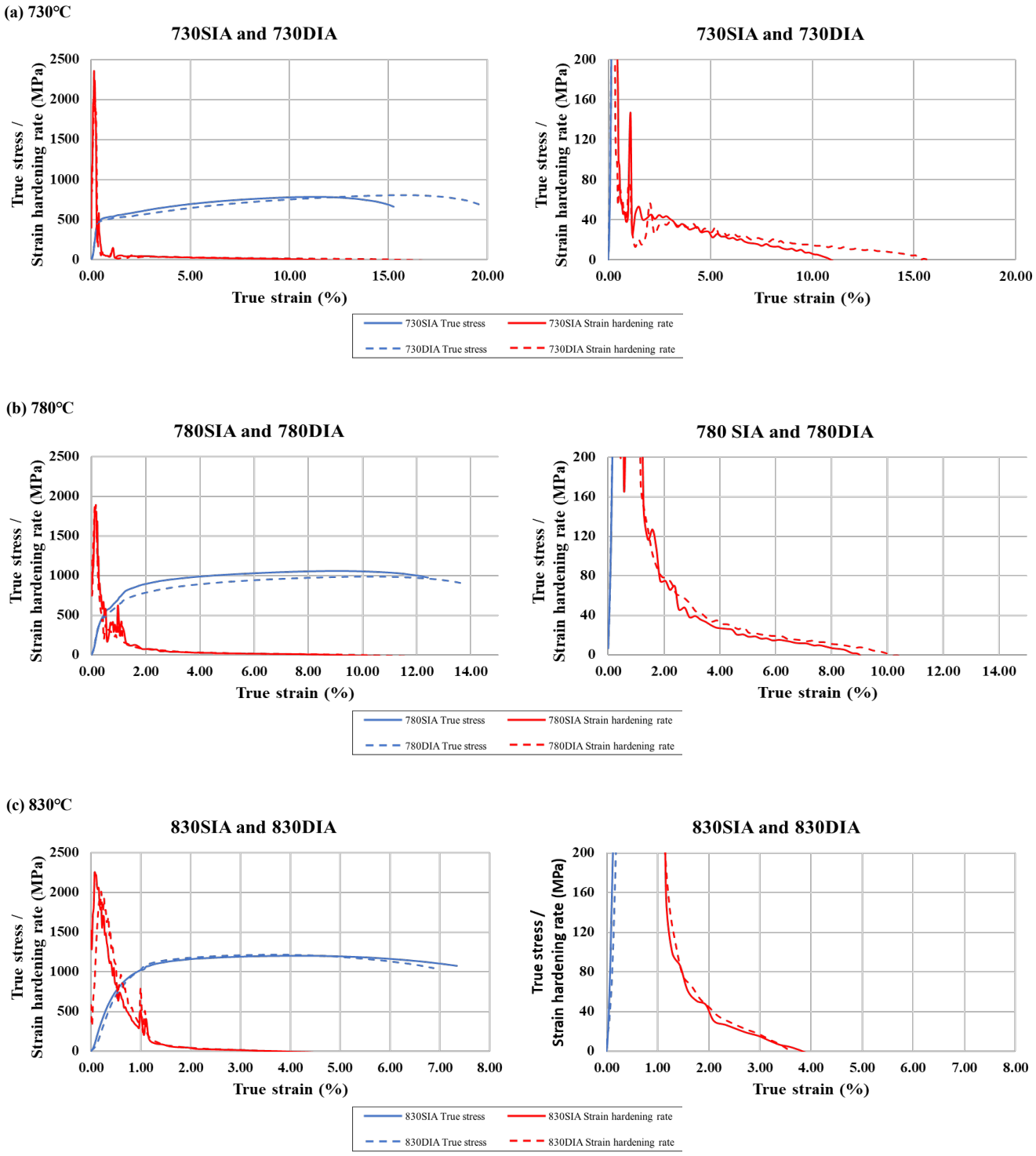


Figure 9. True stress-strain and work hardening curves of specimens intercritical annealed.

3.3 Relationship between microstructure and mechanical properties

According to the microstructure and mechanical properties results, the connection between them is discussed. DIA alters the microstructure of the specimens compared to SIA, which directs the variation of mechanical properties. The smallest change was observed in the 830°C intercritical annealed specimens. In this case, both the ferrite size and martensite fraction slightly decrease (Table 3 and Figure 6) whereas the mechanical properties (Table 4) and homogeneity (Figure 4(e-f), Figure 5(e-f)) stay almost unchanged. As the reduction of the ferrite size enhances yield strength and the decrease in the martensite fraction generally reduces yield strength [20-22], their effects cancel out each other.

A larger alteration was witnessed from the specimens intercritical annealed at 780°C. Though the ferrite size is almost similar (Table 3), about 4.5% decrease in martensite fraction (Figure 6) was found. The yield and tensile strength values decrease over 60 MPa after DIA even though the elongation is increased. As higher martensite fraction generally results in higher strength and lower elongation [20-22], thus, the observed mechanical properties of 780DIA are mainly related to its lower martensite fraction.

Lastly, the largest difference in microstructure and mechanical properties was found between the 730SIA and 730DIA (Figure 4 (a-b), Figure 5(a-b)). In both conditions, a larger ferrite size (Table 3) and less martensite fraction (Figure 6) were observed near surface compared to the center area. Nevertheless, the inhomogeneity decreases and the amount of granular ferrite increases at the center area after DIA. The yield and tensile strengths of the 730DIA are slightly lower than the 730SIA but the elongation is drastically higher (Table 4), and the strain hardening during plastic deformation sustains a positive value to a larger true strain (Figure 9(a)). It was reported that the lower martensite fraction leads to the higher C content in martensite and reduced the strain hardening exponent of DP steels.[23] However, the strain hardening in this study shows the opposite result. It was suggested that the increase number of ferrite enhances elongation [24], and the slight reduction of ferrite size is beneficial for strengths and ductility [5,8]. Moreover, a larger number of granular ferrite at the center area after DIA compared to mostly lath ferrite observed in the SIA specimen is beneficial for strain hardening behavior because less stress concentration is produced at the interface. As a result, the observed mechanical behavior of the 730DIA attributes to the increase in ferrite fraction, the reduction of ferrite size and, the higher amount of granular ferrite.

According to the results, the mechanical properties of DP steel vary greatly from the tensile strength of 700 MPa up to 1160 MPa and the elongation from 7% up to 22.5% with a different intercritical annealing process. These values fit within the usual mechanical properties of DP steel [4]. Compared to the previous studies of DP steel processed with various heat treatment conditions [5-18], the results are comparable although direct comparison is not precise due to the difference in the tensile testing condition. It was suggested that tailoring the microstructure with cold deformation before intercritical annealing promotes recrystallization and leads to better mechanical properties [5,8,9,12]. However, deformation between the heat treatment steps adds complexity to the process. Moreover, cyclic intercritical annealing was studied by S. Ghaemifar and H. Mirzadeh [16,18] as a potential method and was proved to be effective for the modification of the mechanical properties. Still, until recently the method was not well studied. According to this study, the results confirm that DIA method can modify and enhance the mechanical properties of DP steel. However, more studies on the variables such as intercritical annealing time, intercritical annealing temperature, heating and cooling rates should be performed in order to increase the efficiency of this method.

4. Conclusions

In this paper, dual phase steel with the composition of 0.107%C-2.39%Mn-0.453%Si was subjected to the DIA process annealing at 3 different temperatures. The effects on the microstructure and mechanical properties were studied and compared with the regular method, SIA. The significant results were observed as follows:

1. For both the SIA and DIA processes, the martensite fraction increases by increasing the intercritical annealing temperature which leads to the increase of yield and tensile strengths and the loss of elongation.

2. A slight reduction of the overall ferrite size and martensite fraction were observed after DIA. The effects of DIA on the microstructure are the largest at 730°C and decline with increasing intercritical annealing temperature. This is due to the insufficient amount of energy from 3 min holding at a lower temperature which cannot drive the microstructure evolution to reach homogeneity or equilibrium state.

3. The increase in elongation and slight reduction in strength values were observed after DIA. These effects decrease with increasing intercritical annealing temperature and are negligible at 830°C. The decrease in strengths attributes to the reduction of martensite fraction while, the decrease of ferrite size and the increase of ferrite fraction are responsible for the increase of elongation.

4. The change in ferrite morphology from lath to granular at the center area after DIA at 730°C compared to SIA is beneficial for the strain hardening behavior during plastic deformation which also contributes to the increase in elongation.

5. The results from this study establish that DIA leads to the modification of mechanical properties of DP steels and possibly enhances them. However, suitable values of the variables such as intercritical annealing time, intercritical annealing temperature, heating rate and cooling rate should be further studied to improve the effectiveness of this method.

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