



## Effect of temperature in intercritical treatment on microstructure, tensile properties and hardness in dual phase ST52 steel

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### Abstract

Dual phase ferrite-martensite steels are low alloy steels with high strength that have shown excellent ductility during tensile. It's Microstructure consisting of a soft ferrite matrix with hard martensite islands at the grain boundaries. In this study the effects of different intercritical annealing temperatures over mechanical properties of dual phase steel ST52 have been studied. For this purpose  $Ae_1$  and  $Ae_3$  temperatures are mathematically calculated on basis of interaction among chemical composition, anneal temperature and speed of critical cooling rates. Based on observed microstructure, increase in anneal temperature, decrease strain follow necking. This matter is related to increase of martensite volume fraction and suitable sites for crack nucleation. The best mechanical proprieties have been seen from specimen that have been annealed in 770 °C for 105 min and water quenched.

**Keywords:** Dual phase steel, ST52, Mechanical properties, Microstructure.

### 1. Introduction

Dual phase steel is new group of high strength low alloy (HSLA) steel. The main characteristic of this group is tensile strength of 550 Mpa and microstructure with 20% hard martensite which is propagated in soft and ductile ferrite background [1]. The dual phase refers to microstructures with two phases of ferrite and martensite. Ferrite and martensite are predominant phases and the other phases such as bainite, pearlite and residual austenite can be slightly found in the background. This type of structure can be made by heating steel up to the temperature above  $Ac_1$  which is in ferrite-austenite region and quenching it to room temperature. This process is normally different from the method which is used to make fully martensite structures because the steels does not being heated to austenite region to become fully austenite. Formed ferrite and austenite has enrich carbon microstructure. This heat treatment on dual phase steel is called intercritical annealing [2]. After quenching, dual phase steel gets heated in low temperature to become more ductile. Main characteristic of dual phase steel are low yield strength, high work hardening rate. When work hardening rate of steel is high, the strength of the steel increase rapidly while deformation [3, 4]. Researchers have suggested different diagrams for formation of austenite in this type of steel [5]. By using these diagrams, the time of austenite formation and the controlling kinetic processes in each steps of austenite formation can be easily recognized. For instance, for steels been heated at 780 °C the first step of forming austenite which is solving pearlite occurs very quickly and within 0.2 seconds %12 austenite is already made [6]. As time goes, austenite growth inside ferrite. Carbon diffusion into austenite is the speed controller factor and after 6 seconds around %20 austenite has been formed. When austenite growth into ferrite slows down, the manganese diffusion into ferrite becomes the main controlling factor and after 55 hours more than %50 austenite has been formed. Final step is to gentle

equivalence of manganese in austenite that manganese diffusion in austenite controls velocity of equilibrium. The final equilibrium occurs after 3000 hours [7]. The pearlite dissolving process happens very slowly (15 seconds to 8 hours) in low temperature between  $AC_1$  and  $740\text{ }^\circ\text{C}$ . In this condition the further austenite growth is only being controlled by manganese diffusion inside ferrite and there is no sign of carbon diffusion control phase [7, 8]. It has also been found that high strength micro alloy steels have 2 to 9 percent residual austenite on top of their ferrite [9, 10]. The residual austenite particles increase work hardening rate at initial percent of strain in tensile experiment. This phenomenon is the result of changing residual austenite to martensite under strain effect [11].

The present work examines the role of intercritical annealing temperature and microstructure on the tensile properties of ST52 steel. Dual phase steel has been developed at the different intercritical annealing temperature between  $Ae_1$  and  $Ae_3$ . The main aim of the research is to produce a reinforcement which will be strong and ductile.

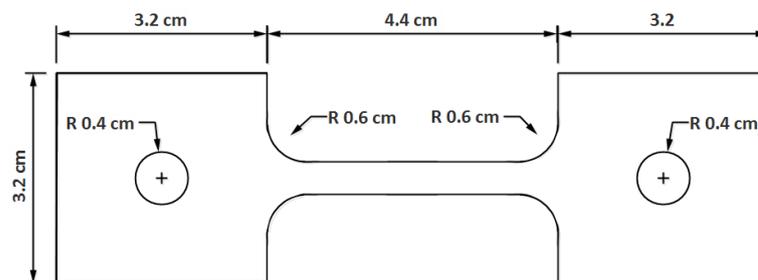
## 2. Experimental procedure

### 2.1. Materials and methods

The used material was in the form of 12 mm thick hot rolled plate with ferrite-pearlite structure. The chemical composition of the investigated sample was obtained through spark emission spectroscopy (Foundry Master Pro) and is shown in Table 1. Plane low carbon steel in the form of sheet is used to obtain tensile specimen with the help of punching die the sample for the tensile testing were cut from the sheet with the dimensions as shown in Fig. 1. All these samples were subcritically heated in muffle furnace at  $890\text{ }^\circ\text{C}$  for 1 h followed by air cooling. These samples are used for developing the dual-phase steels.

**Table 1:** The chemical composition of the investigated material.

Specimen Number	Chemical composition (wt.%)						
	C	Si	Mn	P	S	Cr	Mo
1	0.193	0.216	1.20	0.0110	<0.003	0.0081	<0.005
2	0.193	0.230	1.21	0.0092	<0.003	0.0112	<0.005
	Ni	Al	Co	Cu	Nb	Ti	Fe
1	0.0159	0.0396	0.0010	0.0110	0.0022	0.0030	Rest
2	0.0136	0.0307	0.0018	0.0120	0.0047	0.0023	Rest

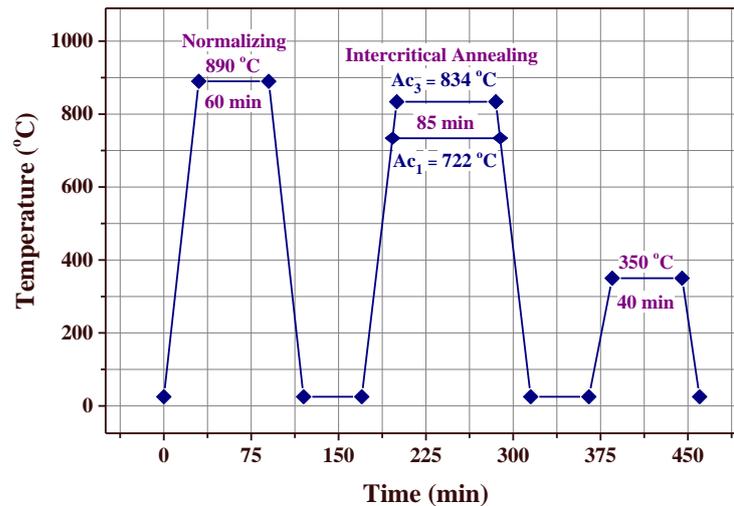


**Figure 1:** Schematic diagram of steel sample with dimensions.

With aiding mathematical calculations 1 and 2 [11], critical temperature of the steel ( $Ae_1$  and  $Ae_3$ ) were calculated  $722.3$  and  $834.2\text{ }^\circ\text{C}$ , respectively.  $740$ ,  $770$ ,  $800$  and  $820\text{ }^\circ\text{C}$  temperatures are used to achieve dual phase structures and  $860\text{ }^\circ\text{C}$  temperature is used to create Martensite structure. Intercritical anneal also occurred at this temperature. Figure 2 shows schematic of heat treatment cycle used in this study.

$$(1) \quad Ae_1 = 751 - 16.3C - 27.5Mn - 5.5Cu - 5.9Ni + 34.9Si + 12.7Cr + 3.4Mo = 722.3\text{ }^\circ\text{C}$$

$$(2) \quad Ae_3 = 881 - 206C - 15Mn - 26.5Cu - 20.1Ni - 0.7Cr + 53.1Si + 41.7V = 834.2\text{ }^\circ\text{C}$$

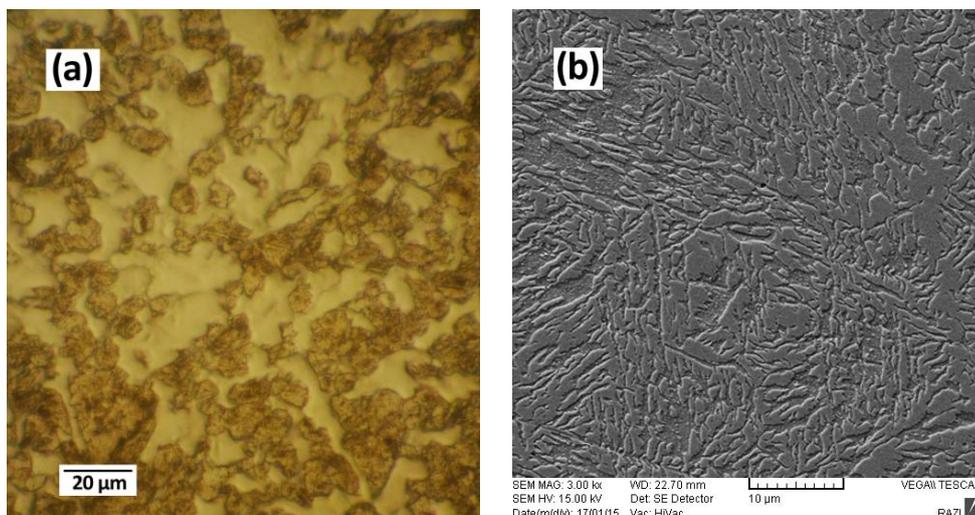


**Figure 2:** Schematic illustration of heat treatment cycle used in this study.

At first, 18 specimens have been prepared from wrought plate for intercritical annealing. One specimen is made as a raw sample, 3 specimens are used for each intercritical annealing and 3 specimens to create fully martensite structures. For base microstructure be seen, two specimens have been normalize in 890 °C temperature and cooled down in air. All specimens were put inside furnace for a particular time to reach the required temperature. Once the time was elapsed the specimens were removed and quenched in water and then isothermally held at 350 °C for 40 min followed by air cooling at room temperature. Sample for microstructure studies were prepared and etched with 2% nital solution. The etched specimens were mounted on a brass stud, sputtered with gold and examined using scanning electron microscope (SEM, VEGAII TESCAN) at 15 keV. Tensile tests were carried out at room temperature using an INSTRON tensile machine with a cross-head speed of 1 mm/min (strain rate of  $4.6 \times 10^{-4} \text{ s}^{-1}$ ).

### 3. Results and Discussions

The first step to create enrich carbon austenite in intercritical annealing is pearlite decomposition. This stage happened very quickly and no analysis can be done even by using thermo dilatometer analysis (TDA) [12]. However this analysis can be done in a better way in low temperature i.e. 740 °C. Annealing for longer period more than what is required for pearlite to fully dissolve, causes an increase volume fraction of austenite and austenite growth into ferrite [13]. Microstructure of heat treated specimen at 740 °C is shown in figure 3.

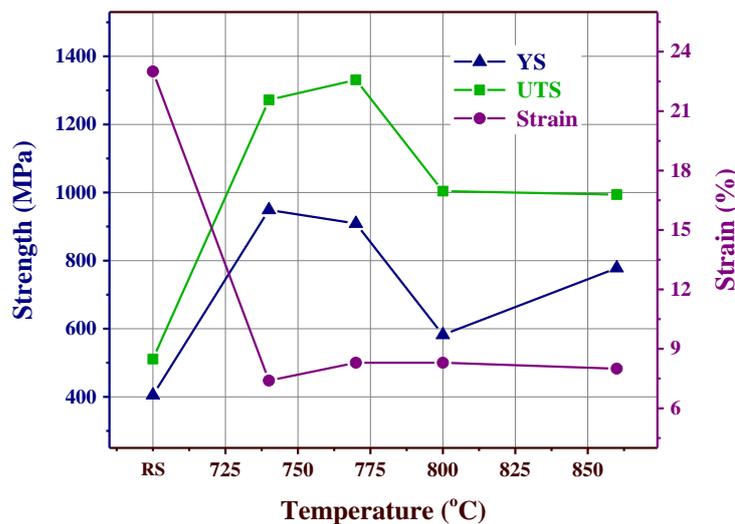


**Figure 3:** Optical and SEM micrographs for intercritical annealed specimen at 740 °C.

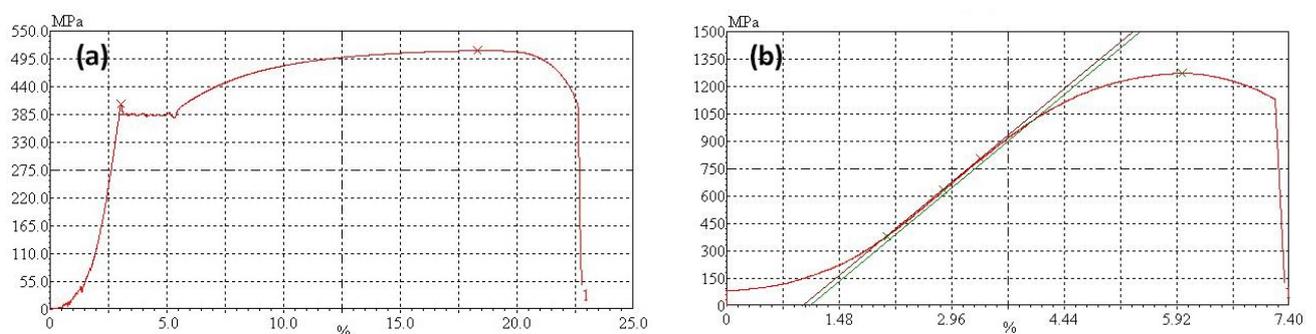
At this temperature, the first step of transformation occurs in dual phase zone that consist of pearlite dissolves and austenite growth into pearlite which is being controlled by carbon diffusion into austenite [6].The diffusion path is in interfaces between pearlite and austenite which is roughly equal to pearlite layers distance (0.2 micron). Due to this tight gap, austenite growth rate is very high. At this temperature, diffusion rate of alloy element such as Mn reduces and causing a significant decrease in growth rate. Since martensite is formed by enrich carbon austenite, it has high hardness (table 2) and has shown greater strength and lower elongation compare to normalized specimen (Fig. 4). Besides there will not be a sign of yielding phenomenon which is one of the dual phase steel's specifications (Fig. 5) [14, 15].

**Table 2:** Results of the steel samples after tensile test and hardness test.

Sample Number	Intercritical annealing temperature (°C)	Yield Strength (MPa)	UTS (MPa)	Strain (%)	Hardness HV30
1	Raw specimen	405	511	23	152 ± 3
2	740	949 Proof	1272	7.4	336 ± 5
3	770	909 Proof	1331	8.3	316 ± 6
4	800	582 Proof	1004	8.3	289 ± 4
5	860	778 Proof	994	8	371 ± 5

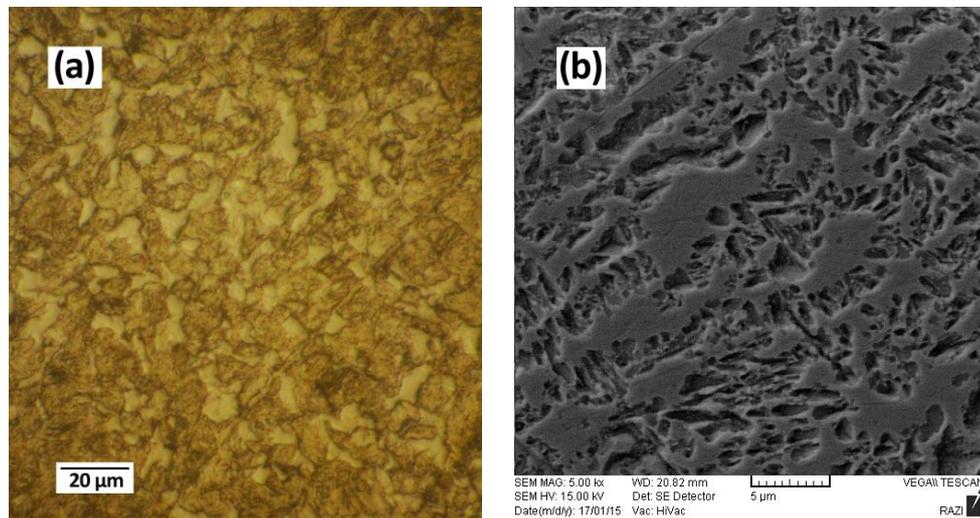


**Figure 4:** Variation of yield strength (YS), ultimate tensile strength (UTS) and strain versus intercritical annealing temperature.



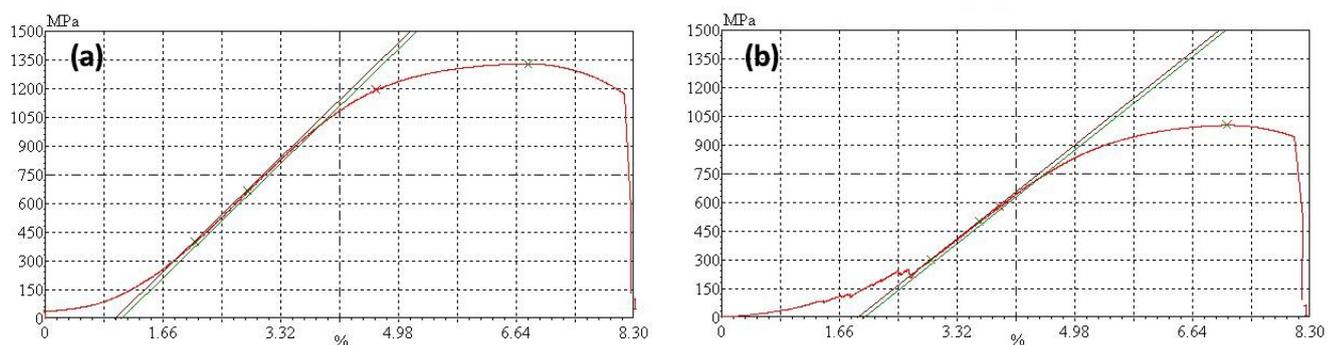
**Figure 5:** Engineering stress-engineering strain curves of (a) normalized specimen and (b) intercritical annealed sample at 740 °C.

As temperature increases, transition from substitution to interstitial diffusion control, increases the rate of pearlite dissolve dramatically [5]. It can be expected to have a large amount of martensite at 770 °C (fig. 6). At this temperature austenite nucleate in interface between ferrite and pearlite and growth into pearlite to complete the pearlite decomposition process. Austenite nucleation happens frequently and it is not considered as speed controller factor. Austenite growth rate at this stage is merely controlled by carbon velocity inside the austenite but at lower temperatures other elements can affect this process [16, 17].



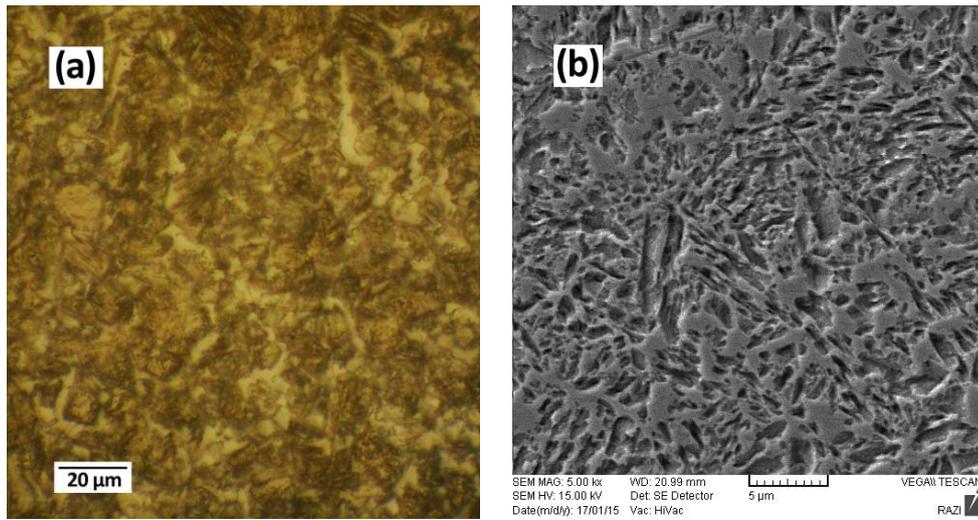
**Figure 6:** Optical and SEM micrographs for intercritical annealed specimen at 770 °C.

Increase in austenite growth into ferrite introduces the next step. Slow growth of austenite at this stage can be controlled by carbon diffusion in austenite or manganese diffusion in ferrite [18, 19]. At the end, there would be a slow balance between ferrite and manganese which is caused by manganese diffusion into austenite (this process is slower than manganese diffusion into ferrite) [20, 21]. In temperature higher than intercritical annealing, created austenite have taken a higher percentage of microstructures compare to main volume fraction of pearlite colonies. Therefore in low intercritical annealing temperature (770°C for ST52 steel) the amount of austenite volume fraction is minimal and cementite spheroidising in pearlite creates a small distribution of austenite which can successfully create martensite distribution [22]. This matter will result in great final strength (1331 Mpa) (Fig. 7a) and softness with hardness of 316 HV30 (Table 2).

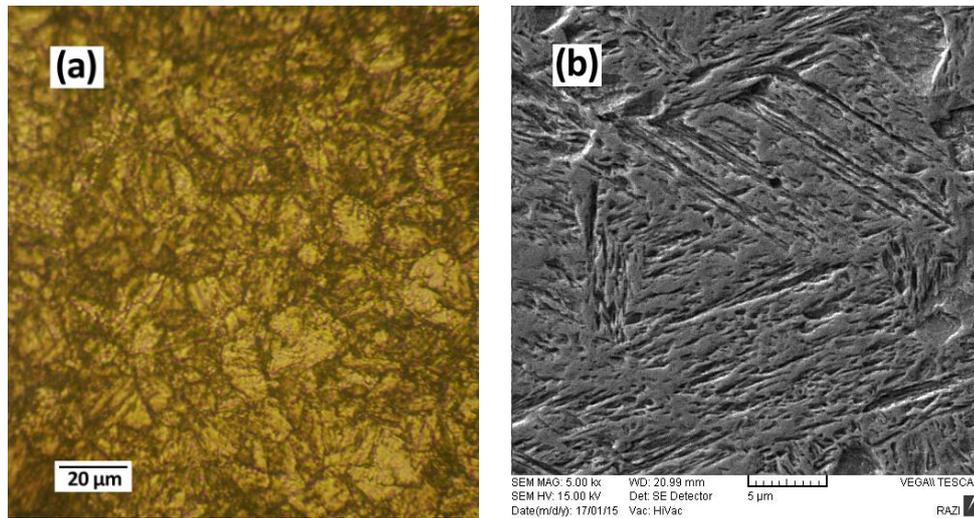


**Figure 7:** Engineering stress-engineering strain curves of intercritical annealed specimens at (a) 770 and (b) 800 °C.

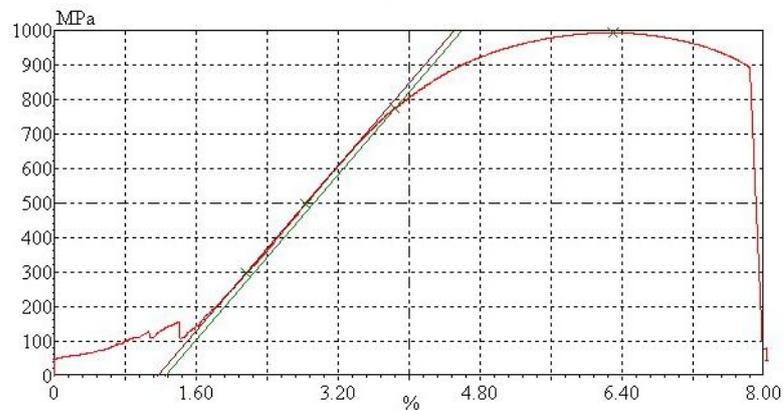
By increasing intercritical annealing temperature up to 800 °C the amount of martensite in microstructure increase (fig. 8). Due to decrease of carbon in austenite and creation of tough martensite, the ultimate strength diminishes (fig. 7b). The fully martensite structure will be created at 860 °C temperature (Fig.9). Since the martensite has a brittle structure it is prone to crack nucleation and as it is shown in figure 10 this issue causes a dramatic decrease in ultimate strength while the hardness is 371 HV30.



**Figure 8:** Optical and SEM micrographs for intercritical annealed specimen at 800 °C.



**Figure 9:** Optical and SEM micrographs for intercritical annealed specimen at 860 °C.



**Figure 10:** Engineering stress-engineering strain curves of intercritical annealed specimen 860 °C.

## Conclusion

Findings have shown that in all specimen lose their yielding point phenomenon when dual phase structure is formed and by increasing the intercritical annealing temperature the austenite volume fraction decreases (it has relationship with the amount of martensite after quenching) which results in increase of ultimate strength and ductility reduction is marginal. In low intercritical annealing temperature (steel ST52, 770 °C) small amount of austenite are formed and cementite spheroidising in pearlite, results in a small distribution of austenite which can successfully create fine grain martensite distribution. This matter will result in good combination of ultimate strength (1331 Mpa) and softness with hardness of 316 HV30. By increasing intercritical annealing temperature up to 800 °C the amount of martensite in microstructure increase. Due to decrease of carbon in austenite and creation of rough martensite, the ultimate strength diminishes. Temperature increase up to austenite phase zone creates fully martensite structures. During tensile tests because of brittle structure of martensite, the small cracks on specimens' surface will have low energy level and can easily growth and produce the ultimate strength of 993 Mpa.

## Reference

1. Zeytin H. K., C. Kubilay, H. A. Havva, Zeytin K., Kubilay C., Aydin H., *Mater. Letters* 62 (2008) 2651–2653.
2. Andrews K.W., ASM hand book, Vol. 4, U.S. (1990).
3. Akbarpour M. R., Ekrami A., *Mater. Sci. Eng. A* (2008) 306-310.
4. Chakraborti P.C., Mitra M.K., *Mater. Sci. Eng. A* (2007) 123-133.
5. Speich G.R., V.A. Demarest, R.L. Miller, *Metall. Trans. A*, 12 (1981) 1419-1428.
6. Speich G.R., ASM hand book, Vol. 1, U.S. (1990).
7. Juan-juan L., Wen S., Qun-fei H., *J Iron Steel Res Int*, 17 (2010) 54.
8. Hayat F., Demir B., Acarer M., *Mater. Sci. Heat Treat*, 49 (2007) 484.
9. Anazadeh Sayed A., Kheirandish Sh., *Mater. Sci. Eng. A* 532 (2012) 21–25.
10. Saai A., Hopperstad O.S., Granbom Y., Lademo O.G., *Procedia Materials Science* 3 (2014) 900–905.
11. Speich G.R., Metals Hand book, ASM, oh, U.S. (1990).
12. Qu S., Zhang Y., Pang X., Gao K., *Mater. Sci. Eng. A* 536 (2012) 136–142.
13. Zeytin H.K., Kubilay C., Havva H.A., Zeytin K., Kubilay C., Aydin H., *Mater. Letters* 62 (2008) 2651–2653.
14. Sirinakorn T., Uthaisangsuk V., Srimanosawapal S., *Procedia Eng.* 81 (2014) 1366–1371.
15. Zhao Z., Tong T., Liang J., Yin H., Zhao A., Tang D., *Mater. Sci. Eng. A* 618 (2014) 182–188.
16. Sodjit S., Uthaisangsuk V., *Mater. Des.* 41 (2012) 370–379.
17. Rosenberg G., Sinaiová I., Juhar L., *Mater. Sci. Eng. A* 582 (2013) 347–358.
18. Speich G.R., Demarest V.A., Miller R.L., *Metall. Trans. A*, 12A (1981) 1419-1428.
19. Lee S., Lee S.J., De Cooman B.C., *Acta Mater.* 59 (2011) 7546–7553.
20. Speich G.R., Miller R.L., United States Steel Research Laboratory, private communication (1975).
21. Arlazarov A., Goune M., Bouaziz O., Hazotte A., Petitgand G., Barges P., *Mater. Sci. Eng. A* 542 (2012) 31–39.
22. Kuang C.F., Li J., Zhang S.G., Wang J., Liu H.F., Volinsky A.A., *Mater. Sci. Eng. A* 613 (2014) 178–183.

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