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Microstructural engineering of dual phase steel to aid in bake hardening

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Abstract. Low carbon steel of composition 0.05C – 0.18 Mn – 0.012 Si is intercritically annealed at temperatures 750^{o}C, 775^{o}C and 800^{o}C. The equilibrated alloys of different amounts of austenite with varying carbon contents are quenched in iced water. The same alloys are subcritically annealed at 675^{o}C and 700^{o}C for varying periods of times; the subcritically annealed alloy samples are quenched in iced water. Optical, scanning electron and transmission electron microscopy are carried out for all the samples. The dislocation structure, its distribution and density present in the above prepared duplex ferrite martensite steels are studied. The martensites are found to be highly dislocated due to lattice invariant deformation. At the same time ferrite adjoining the martensite areas also exhibits quite a high dislocation density. The high dislocation density is favorable for strain ageing and hence bakes hardenability. EDS analyses were carried out for both martensite and ferrite phases; it is found that the degree of supersaturation in ferrite together with carbon content in martensite varies with the process parameters. The microhardness test results show that the hardness values of different phases differ appreciably with process parameters. The microstructures and the corresponding microanalyses reveal that differently processed steels contain phases of varying compositions and different distribution.

Keywords: bake hardening; intercritical annealing; dual phase steel; martensite; Cottrell atmosphere

1. Introduction

Continued thrive for reduction in vehicle weights has led to the emergence of a variety of advanced high strength steels for automotive applications. In recent times extra low carbon (ELC) steel or more precisely, the interstitial free steels are widely used in automobile sectors due primarily to their excellent formability and high weldability (Bleck (2012), Mongkut's *et al*. (2013), Traint *et al*. (2007)). Owing to favorable texture achievable in the interstitial free steel (IF), it possesses excellent deep drawability (Parker *et al*. (2002), Mamuzić *et al*. (2006)). However the steel suffers from the deficient dent resistance owing to its low strength.

Major microstructural parameters to determine the mechanical properties of this class of steel are the size and shape of ferrite grains which in general constitute its microstructure. However extra strengthening of finished components may be possible through paint baking operation (Cooman *et al*. (1999), Haldar *et al*. (2012), Baier (2006)). Hardening during paint baking at about

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160-170 C takes place due to classical strain ageing effect. The processing of steel components is such designed as to retain approximately 20 ppm carbon in ferrite at room temperature; upon heating during paint baking the ferritic carbon migrates to dislocations and create Cottrell - Lomer atmosphere. The dislocations need extra stress to tear away the barrier due to this atmosphere for their further movement. This leads to an increase in yield strength of the steel.

Concurrently, demand for crash worthiness has led to the emergence of a variety of advanced high strength steels which are capable to provide higher strength at high level of ductility.

Duplex ferrite-martensite steel is one such steel, which is in use in automobile industries for quite some time due the advantage of its high formability, low yield/tensile ratio and high specific strength (Prawoto *et al*. (2012), Molinari.*et al* .(2011)). Microstructurally, it is composed of soft ferrite with dispersed islands of harder martensite phase. Effectiveness of bake hardening depends upon the availability of flux of carbon atoms (i.e. the degree of super saturation of ferrite), availability of nearby dislocations for early migration of carbon atoms and the mobility of carbon atoms.

Martensites themselves are internally faulted with dislocations due to lattice invariant deformation (Banerjee *et al*. (2001), Datta *et al*. (2004)); also volume change associated with martensite transformation is liable to generate dislocations in the surrounding matrix made of soft ferrite. Dislocation content of martensite and density of dislocations created around martensite islands depends upon the carbon content of martensite.

Since the process parameters of DP steel can tailor the degree of supersaturation of ferrite, carbon content of martensite and the amount along with distribution of these phases, it is anticipated that superior bake hardening response may be achieved in ELC steels of ferrite-martensite microstructure than the one with ferrite being its lone microstructural constituent.

The present investigation aims to understand the potential of dual phase microstructures of extra low carbon steel (~0.05wt%C) to determine its potential for enhanced bake hardening ability with reference to bake hardenability of similar steel with classical ferritic microstructure.

2. Experimental

The steel used for the present investigation is supplied by Tata Motors Ltd. in the form of sheet. The chemical composition of steel is given in Table 1.

Sample coupons of 25 mm × 25 mm area were cut by abrasive cut off machine. The samples, each of thickness 5 mm were intercritically annealed at 750˚C, 775 ˚C and 800 ˚C for 12 minutes. These temperatures are chosen as they are above the A1 (~720˚C) temperature of the steel and the equilibrium microstructure at these temperatures have been a mixture of austenite and ferrite as per dictates of metastable iron carbon equilibrium diagram After annealing within two phase field, the samples were rapidly quenched in iced brine. A few samples were annealed at the subcritical

Table 1 Chemical Composition of experimental Steel

Element	C	Mn	Si	S	P	Al	Cu	N
Amount (wt%)	.05	0.20	.012	.006	.014	.049	--	--

temperatures, 675 °C and 700 °C for 1 hr, 2hrs and 4hrs; the samples were then quenched in iced brine. The purpose of this experiment is to produce solute rich austenite through competitive process of austenite formation.

Optical microstructure study is conducted by the common metallographic technique of polishing the samples mirror finished followed by etching in 2% Nital solution. Representative photomicrographs are recorded for further analyses. Micro hardness testing of all the samples is carried out at 25 gm load with 15 sec on load dwell time. Microhardness test at such low load gave rise to small indentation depth and the depth of indentation was far less than one tenth of the sample thickness and therefore hardness measurement was quite in order. Scanning electron microscopy has been performed in a Nova Nano SEM 450 of FEI make. Transformation electron microscopy is carried out in FEI make Tecnai G^2 20 S-Twin electron microscope fitted with Bruker make EDS Quantax 200. Following mechanical thinning in Buehler polishing machines, the samples were subjected to dimpling and finally thinned electron transparent by ion milling in Gatan make PIPS II ion milling equipment. Conventional jet polishing was not done to exclude the possibility of any artifacts in TEM micrograph and also for ease in sample preparation. Texture studies of as received and intercritically annealed samples were carried out in a PANalytical X'Pert diffractometer.

3. Results and discussion

The optical microstructure of as received sample is shown in Fig. 1 as a reference. The optical microstructures of the steel samples equilibrated at various intercritical temperatures and then ice brine quenched have revealed dual phase microstructures in general, which are comprised of fined grained ferrite matrix embedded with martensite islands. The number density of martensite islands is seen to decrease with increase in equilibration temperatures (Figs.2-4). It is found that the martensite volume fraction is around 12 vol% when it is annealed at 750°C for 12 minutes. Upon increasing the annealing temperature to 775°C the martensite volume fraction is reduced to about 5%. However for still higher temperature, 800°C one would notice a very small amount of martensite (less than 2%) in the microstructure (Fig. 4) From the nature of steel panel of iron carbon diagram one may notice that the fraction of equilibrium austenite increases by a small extent with increasing annealing temperature within the two phase region (austenite +ferrite).;at

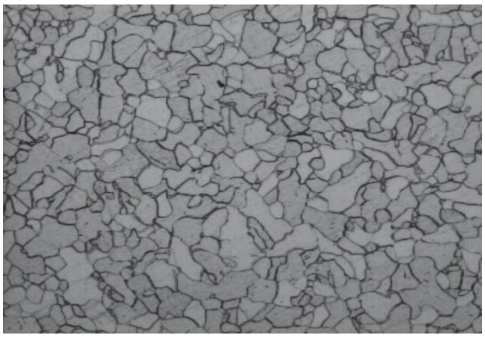


Fig. 1 Microstructure of experimental steel annealed in received condition (Normalized)

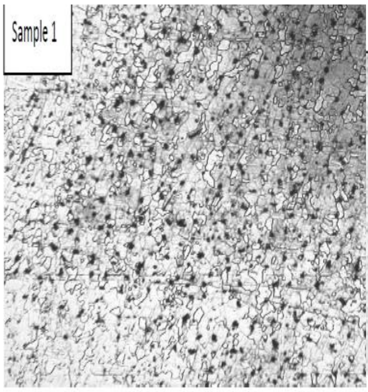


Fig. 2 Optical microstructure of steel annealed at 750˚C

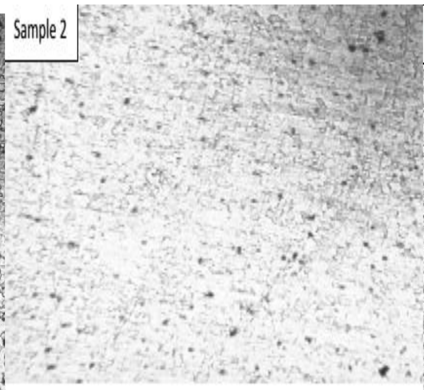


Fig. 3 Optical microstructure of steel annealed at 775˚C

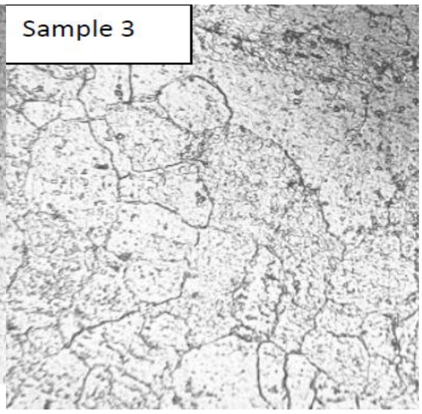


Fig. 4 Optical microstructure of steel annealed at 800˚C

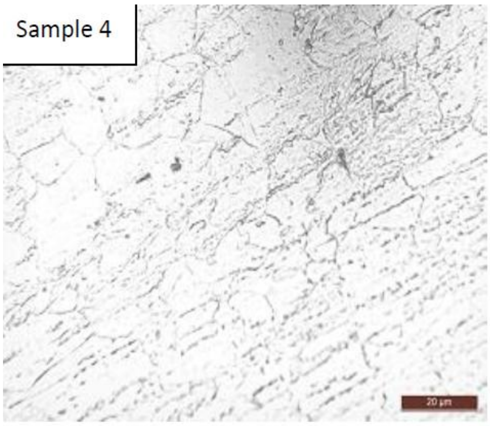


Fig. 5 Optical microstructures of steel annealed at 675˚C for 1hr

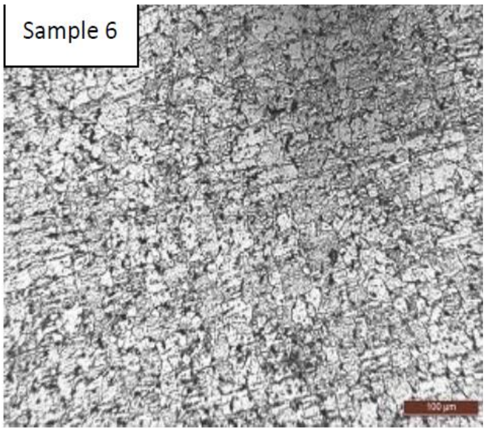


Fig. 6 Optical microstructures of steel annealed at 675˚C for 4 hrs

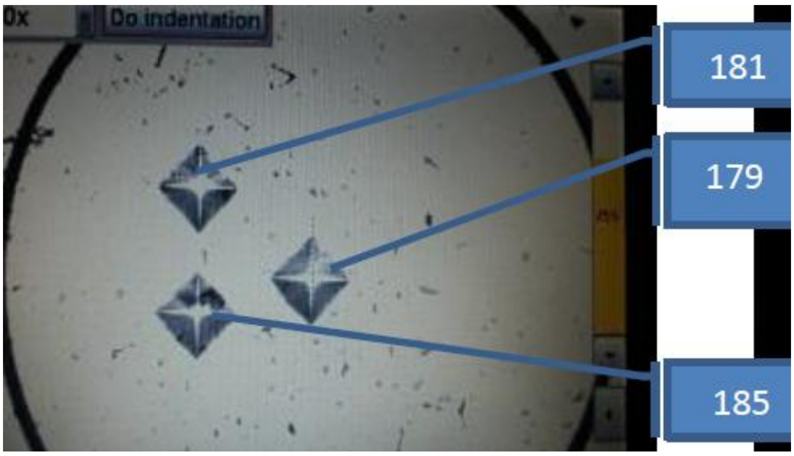


Fig. 7(a) Microhardness results of steel annealed at 750˚C

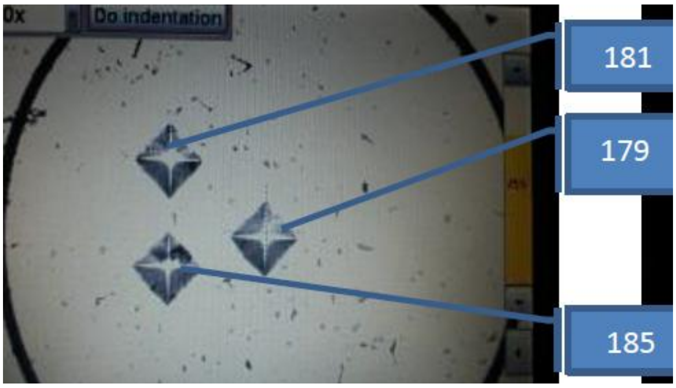


Fig. 7(a) Microhardness results of steel annealed at 750˚C

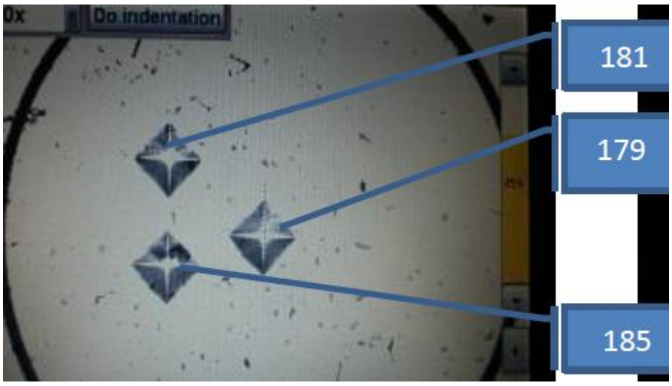


Fig. 7(b) Microhardness results of steel annealed at 675˚C for 2h

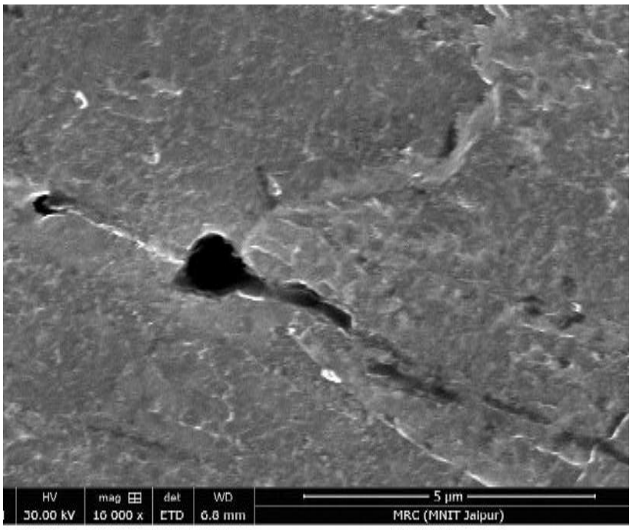
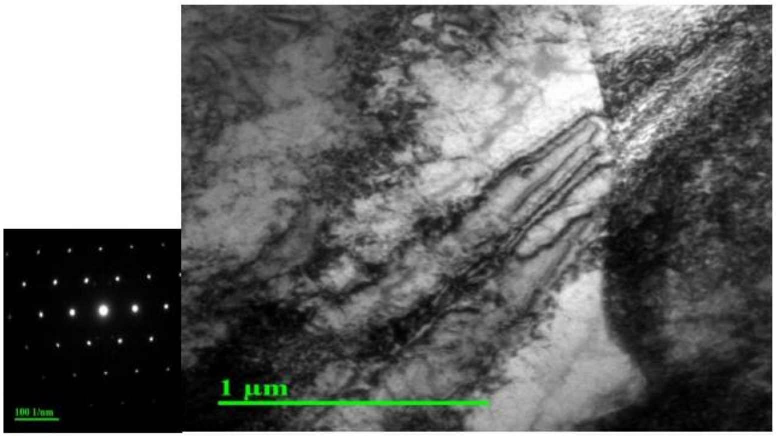
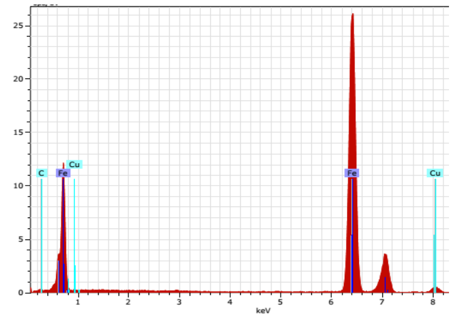


Fig. 8 Scanning electron micrograph of steel annealed at 700˚C





Spectrum: Acquisition 97

El	AN	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (1 Sigma) [wt.%]
Fe	26	K-series	97.17	97.17	96.72	2.96
Cu	29	K-series	2.62	2.62	2.29	0.13
C	6	K-series	0.21	0.21	0.99	0.08
		Total:	100.00	100.00	100.00	

Fig. 9 Transmission electron micrograph shows ferrite crystal with embedded martensite; SADP of the area typifies bcc ferrite and ferrite adjoining martensite is seen to contain dislocations. EDX shows 0.2 wt% C in martensite

Microstructures of representative samples studied by FEGSEM tend to confirm the findings in optical microscopy (Fig. 8). Results of FEGSEM study is supportive of the fact that with increase in time of holding at a subcritical annealing temperature, higher carbon austenite is formed and this, upon cooling, leads to the formation of martensite. The black islands at the triple points of ferrite grains of the sample held for four hours at 700°C is morphologically identical to martensite in conventional dual phase steels. The corresponding micro-hardness values are also found to be quite high to imply that these particles are martensites of higher carbon content. EDS analysis of these black particles proves that solute partitioned austenite of high carbon content (~0.2wt %) does form after holding for four hours at the subcritical annealing temperatures.

the same time there is a reduction in equilibrium carbon content of austenite. Since there is no major hardenability raising alloying elements in the experimental steel, the potential for martensite formation will depend only on the carbon content of austenite. It is conjectured that the austenite with low equilibrium carbon content has not produced sufficient martensite; rather it could produce non equilibrium ferrite of higher carbon content due to fast brine quenching. It may be mentioned that this ferrite of higher carbon concentration is very prone to be relieved of supersaturation through some diffusional precipitation of carbon.

When subcritical annealing at 650˚C and 700˚C are done for various time, formation of competitive austenite, rich in austenite stabilizing solute is a possibility. It is easy to reconcile that this austenite will begin to form at the ferrite grain boundary only with a high solute content. Upon cooling in iced brine, the solute partitioned austenite is converted to martensite provided its hardenability is sufficiently high (Fig. 6). It appears that shorter holding time at subcritical temperatures has not provided adequate incubation time to enable formation of solute rich austenite through diffusional means and as a result martensite formation is limited (Fig. 5). Representative micro hardness test results are shown in the (Figs. 7 (a),(b)). In general the hardness of pure ferrite is found to be around 150-180 VPN if there is no interference from the adjoining harder phases. The maximum hardness of martensite is recorded as 225VPN. The intermediate hardness values are due to the influence of the adjoining phases of differing hardness values. The hardness of martensite measured less than 225 VPN in some areas is ascribed to some ferrite phase being included within the indented area. Lower hardness values of black constituent in microstructures of some samples annealed at higher temperature is attributed to lower carbon content of martensite as well as the its small size to include the ferrite phase within the area of indentation.

The transmission electron micrograph of sample equilibrated at 750˚C and then quenched in iced brine shows a ferrite region with a small island of martensite; the ferrite is found to contain dislocations in the form of sub boundaries (Fig. 9). The SADP of the matrix phase is also shown. Martensite with dislocations as internal faults is delineated in Fig. 10. The adjoining ferrite area is seen to contain huge dislocations. The presence of these dislocations stems from the volume change accompanying martensite transformation. The volume expansion due to formation of martensite strains the surrounding matrix, and accommodation of this transformation strain takes place through the plastic deformation of ferrite. This approach of creating dislocations around martensite islands, distributed throughout the matrix, envisages a faster formation of effective Cottrell atmosphere, and hence superior bake hardening. The micro analysis of martensite and ferrite reveals the partitioning of carbon between ferrite and austenite (Fig. 9). The dark field photograph in Fig. 11 reveals martensite morphology along with its high dislocation density. High resolution photograph (Fig. 12) exhibits lattice fringes which seem to be discontinued in some regions of 2-3 mm dimension. A close look at these regions of the image reveals differently oriented fringes characterizing the presence of some other particles. It is known that carbon atoms in ferrite migrate to the nearby dislocations and create Cottrell atmosphere; in case the concentration of carbon atoms coming to dislocations is higher than usual, carbon atoms are arranged in lines along the dislocation cores; such condensed atmosphere leads to the nucleation of carbide. At somewhat lower temperature as room temperature, €--carbide of HCP structure may form. Although this hypothesis could not be verified by TEM, (De. A.K, Vandeputte.S, and De Cooman.B.C(1999)) the present observation of particles of 2 nm dimension with different lattice fringes lends support to the surmise that there are € carbide particles present in fine form within ferrite matrix. The lattice image in Fig. 12 clearly shows vacancies and dislocations where €-carbide is known to form because of lattice match between BCC and HCP crystals; the strain field around the coherent particles is discernable in Fig. 13.

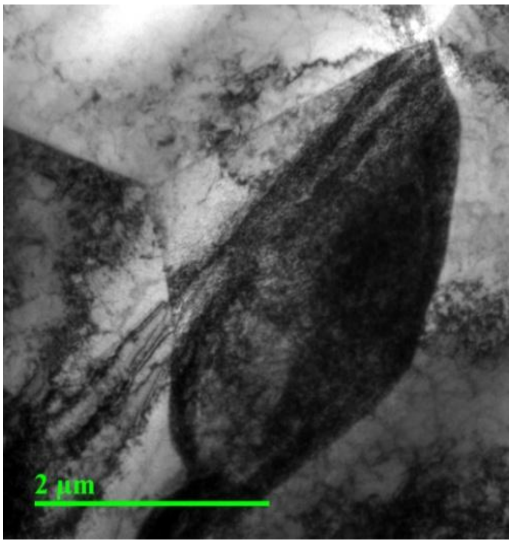


Fig. 10 Martensite in ferrite matrix, BF

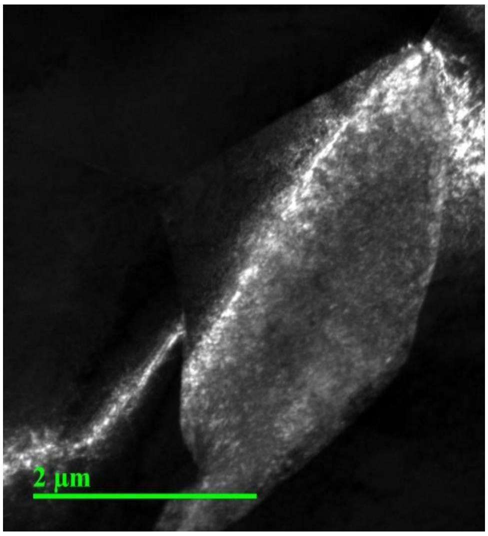


Fig. 11 Dark field photograph of martensite

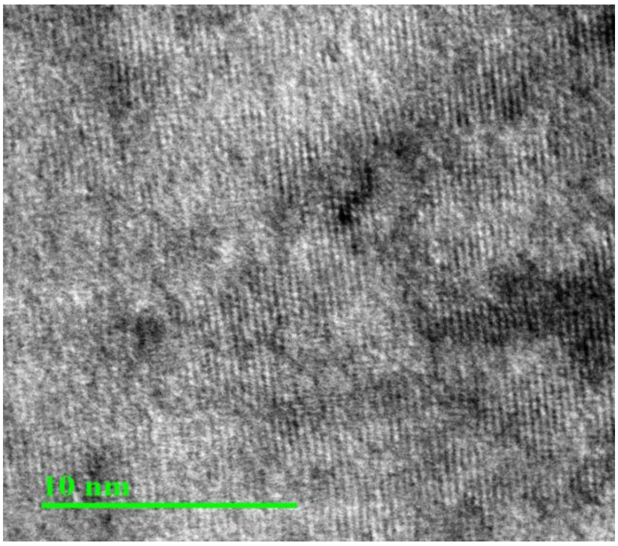


Fig. 12 Lattice image of steel annealed at 750˚C

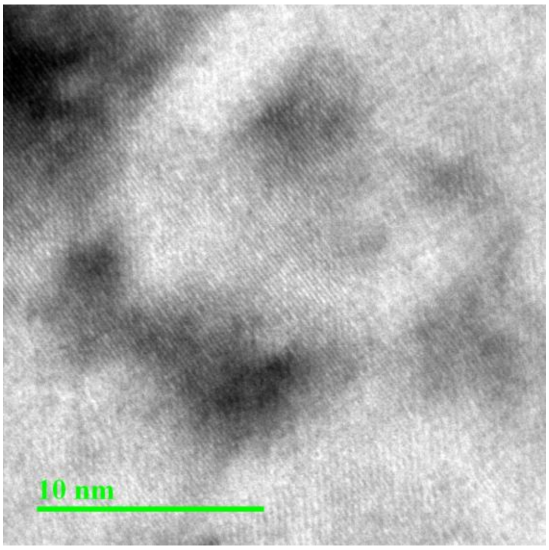


Fig. 13 Lattice image of steel as in Fig. 12 shows evidence of carbide nuclei

The ODF data of the as received sample exhibits rolling texture with well defined α-fiber (φ=10, φ_2=0) and is present with φ_1=0 to φ_1=90. The maximum intensity of rolling texture is obtained at φ_2=0, φ=90 at φ_1=90 ^{0}C; whereas the minimum intensity of the texture is formed at φ=70, φ_2 =45 and φ_1=25 ^{0}C.The three dimensional ODF data exhibits grain orientation along α fiber (Fig. 14).

When the steel is inter-critically annealed at 750 ^{0}C and then quenched in iced brine, there is a remarkable change in texture. The ODF with constant φ_1 values in the range 0-90^o has delineated a well-developed tube shape γ fiber. The stronger texture is observed at φ=45^o and φ_2=40^o at φ_1=45 ^{0}C such type of γ fiber is earlier reported in titanium micro-alloyed IF steel. In the present case the microstructure contains very fine martensite islands interacting with grain boundary. However α fiber is also seen to have been present with φ=0^o to φ_2 =90^o.The three dimensional ODF has given convincing evidence of strong γ fiber (Fig. 15)

If the annealing temperature is increased to 8000°C, there is once again change in texture. Relatively weak γ fiber occurs at φ =55^o and φ_2=45^0; Predominance of α fiber may be noticed φ_2=0-80, φ=10^o at φ_1=70^o. It is noticed that a mixture (α+γ) fiber is present at φ_1 =70 ^{0}C where both the textures are prevalent. The three dimensional ODF has clearly shown the presence of both γ and α fiber, though γ fiber is relatively strong. It therefore appears that for a dual phase structure more favorable recrystallization texture is obtained; this hints upon the fact that the formability of IF steel can be further improved if suitable microstructural engineering is done to secure a duplex ferrite plus martensite structure (Fig. 16) .

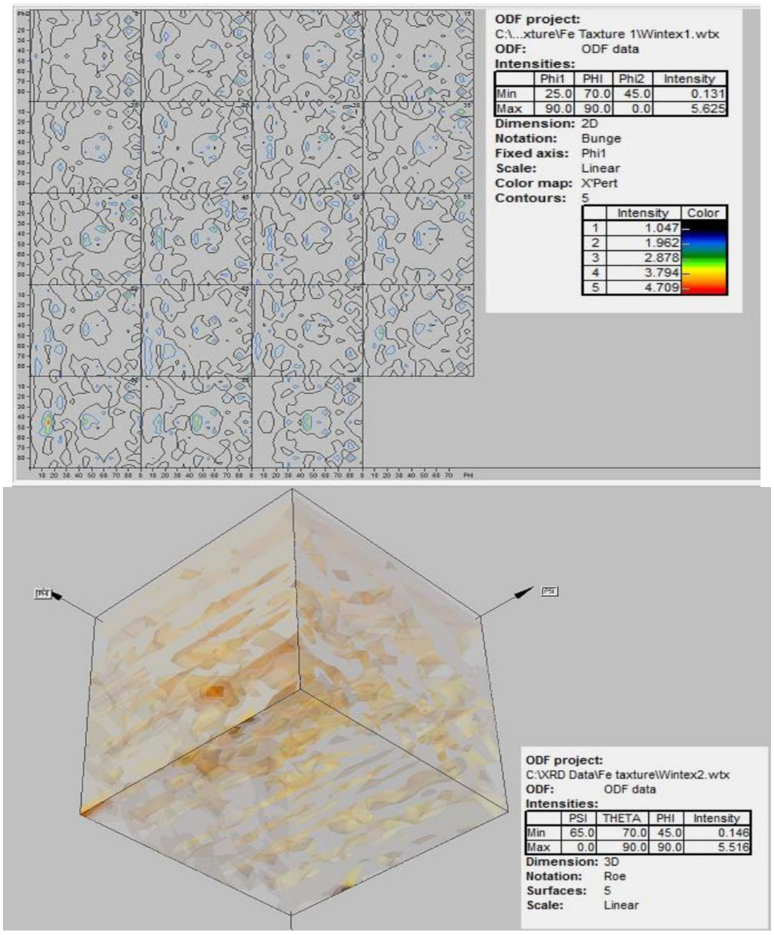


Fig. 14 ODF of as received IF steel; three dimensional ODF is shown

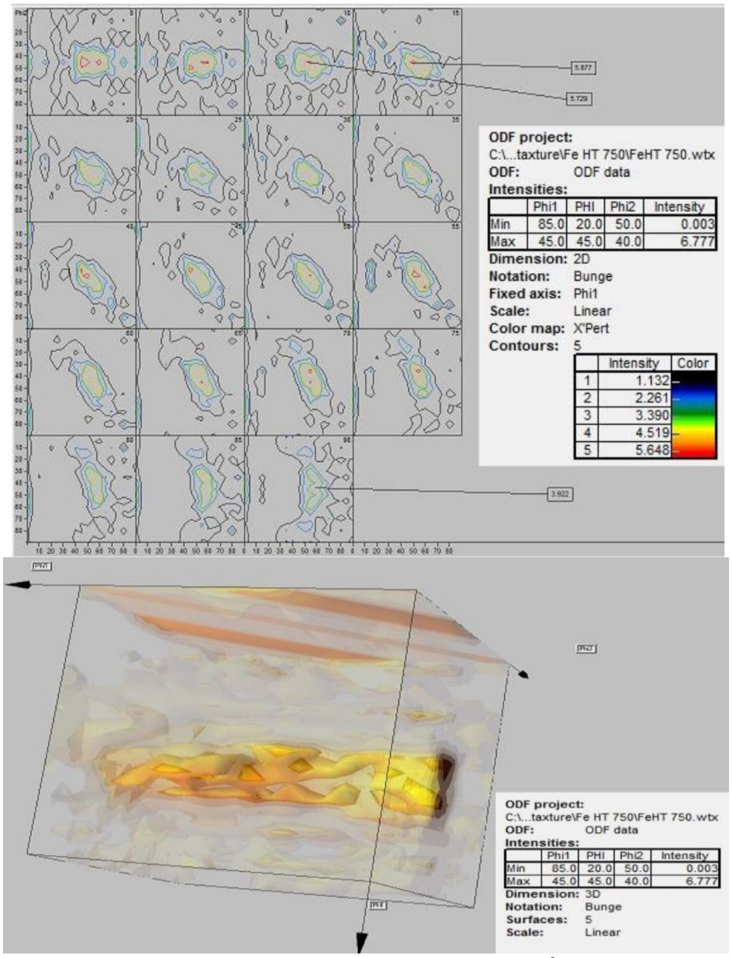


Fig. 15 ODF of steel annealed at 750˚C

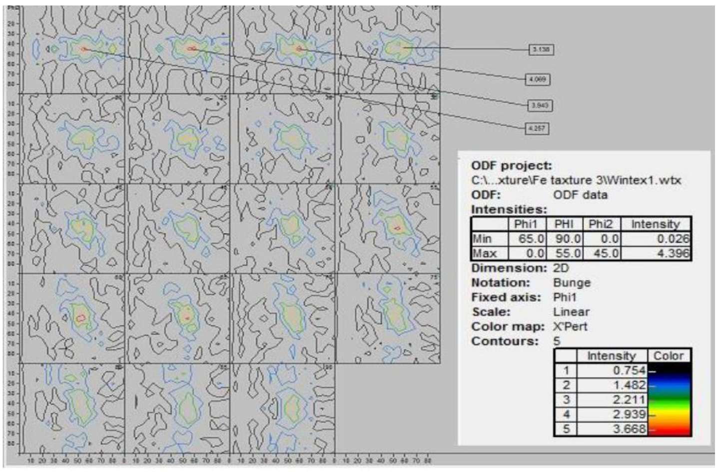


Fig. 16 ODF of steel annealed at 800˚C

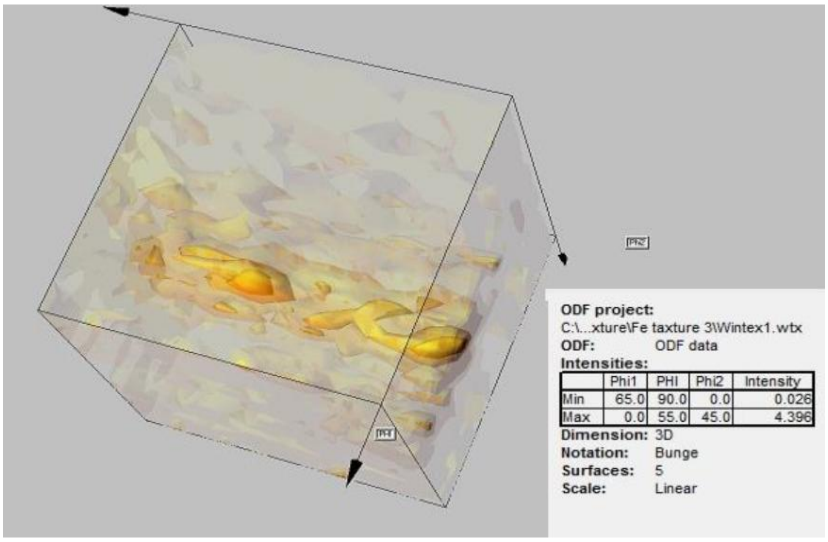


Fig. 16 Continued

4. Conclusions

The author wishes to conclude that both inter-critical and subcritical annealing if done judiciously, may lead to the formation of carbon rich austenite which transforms to martensite upon rapid quenching.

In duplex steel with ferrite+martensite structure the ferrite matrix around martensite Island contains high dislocation density. Also it is concluded that the super-saturation of ferrite brought about by fast quenching after equilibration treatment insures the formation of supersaturated ferrite which aids in transport of carbon atoms to nearby dislocations.

Due to high carbon concentration, nucleation of € carbide of size 2-3 nm takes place. Also, ELC steel with ferrite plus martensite structure yields a favorable recrystallization texture.

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