

Impact of Nano sized Aluminum Nitride Second phase Particles on Gamma and Alpha Phase Transformation in Less Carbon added Manganese Steel

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Abstract

A scientific investigation has been performed in this research work on the basis of experimental result Ramification to divulge the advancement of second phase particles and its influence on ferrite and austenite phase transformation in less carbon-manganese added steel. Two steels have been engaged in this competition with and without aluminum addition along with nitrogen. To fade out the already present particles, as received steel under investigation was solution heat treated at 1200°C and then both the steel samples were heat treated at 740°C and 800°C for 10, 15, 30, and 60 sec incubation time in the cyanide added liquid bath furnace and were rapidly cooled from elevated temperature at room temperature in ordinary water. Metallography was performed after etching technique in two etchant solutions to dispose the internal microstructural features with advanced Olympus GX51 optical microscope with polarized light. To further enlarge the important microstructure SEM with tungsten filament was used to capture the micrographs. To distinguish the lower critical AC₁ and upper critical AC₃ temperature for both the steels were measured with the help of Dilatometer results were plotted by using Origin data software. It was concluded that aluminum combine with nitrogen stimulate the fine aluminum nitride (AlN) particles and these particles were the major source to hinder the grain boundary mobility and consequently phase transformation of alpha and gamma was interrupted in aluminum added steel and it lowers the critical temperatures. Surprisingly there was no such attitude was observed.

Key Words: Phase transformation, AlN particles, Austenite, Dilatometry, aluminum

1. Introduction

Aluminum conventionally is used as de-oxidant element by steel smelters, the second phase particles commonly known as aluminum nitride (AlN) particles, and they can form during melting and casting. [1-7]. They are strong impurity elements which get together around the grain boundaries and provide dragging force to stop the mobility of grain boundary when the steel is reheated or during heat treatment, consequently they retard the grain growth and grains remain finer [7-12]. This technique is commonly and widely used since long time and is adopted by many researchers for increasing the strength of the steel and found that this is the most cheap and easy method of grain refinement but during the last decay there is very few work to be done to know that AlN particles does not only refine the grain size but also it delays the formation of austenite which can be turned into martensitic after quenching and if it delays the transformation characteristics of gamma then first transformation temperature AC₁ and second transformation temperature AC₃ and the amount of martensite can be result as lower in quantity. No one has given

attention that aluminum nitride particles can be useful for grain refinement technique but it also affects the austenite formation kinetics limiting the grain boundary movement. The distribution of the AlN particles present either outside the grain boundary or inside the grain boundary is more effective to make the fine grain steel [13-15]. This riddle and unrevealed features of AlN particles has been studied in the present research work. [16-19].

2. Materials and Methods

Two steel samples were used in this investigation steel A and steel B. steel A has no aluminum content and steel B has aluminum present in it. The content of nitrogen is also present in both the steels under investigation in this work. The chemical composition of both the steels in wt% is:

Steel A: 0.45C 0.20Si 0.8Mn 0.005N

Steel B: 0.42C 0.20Si 0.7Mn 0.152Cr
0.034Al 0.012N

As received material was obtained through the process as shown in diagram below in figure 1. Steps: Solution treatment (two hours at 1200°C) hot rolling (rolling reduction of 80% over a four - pass) Specimens cut at 800°C. Specimen cutting was performed to reduce the size of the sample. For making the easy experimental process 10x10x10 cm samples from the bulk steel were cut from the center of the steel plate in sufficient amounts and quantity. After applying cold mounting the samples were easily gripped by hand. The un-mounted samples were brought into heat treatment process as shown in cycle below in fig.3: Both the steel samples were heat treated with 10°C per minute in the lead bath furnace. Four heat treatment temperatures were selected, first sample was heat treated at 710°C much below the recrystallization temperature to make sure the nucleation of AlN particles was not supposed to appear at 710°C to set a bench mark, similarly second sample was performed at a higher temperature than first one and it was done at 740°C approximately near or above the AC1 line of the steel to observe the nucleation sites for AlN particles formation. Third sample was taken to heat at about the inter-critical annealing zone of the steel and this temperature was 770°C and the last sample of the experimental steel was heated in the austenitic domain at 800°C. This series of heat treatment temperatures was used to know the morphology of phase transformation start and phase transformation finish temperatures and this investigation was helpful to know the actual nucleation sites of AlN particles. For all the above mentioned temperatures the samples were dipped in salt bath furnace for a holding time of 10, 15, 30, 60 seconds respectively and each sample was quenched in tap water. Before going to the sequence of heat treatment temperatures, both the steel were solution treated at 1250°C for dissolving the second phase particles, if they were present. After the long heat treatment work the samples were cut from the center, perpendicular to rolling direction to observe the surface microstructures from inside. Heat treated samples were mounted in a resin solution in the cold mounting state and were then brought to grinding and polishing start from the 200 grade to 2000 grade emery grinding paper, then immediately these samples were polished using 0.05 and 0.02 % alumina solution in the soft polishing cloth simultaneously. This grinding and polishing process was performed both the steel and steel B in as rolled condition as well as after heat treatment for better understanding and finding the nucleation sites, as rolled or as received condition was compared with the steel A and Steel B heat

treated sample to make sure that AlN particles are not present in as rolled samples. After reaching the mirror image stage the samples were then ultrasonically cleaned in 100 ml ethanol for 5 minutes, after cleaning these samples were dried with hot air. The classical etchant for developing the microstructure was used for etching. Two reagents were brought to reveal and develop the microstructure. The reagent 1 was prepared separately and reagent 2 was also prepared separately in glass beaker then fifty ml from quenchant 1 and fifty ml from reagent 2 was collected and mixed in another beaker by using magnetic stirrer. Quenchant 1 includes 1gm of Sodium metabisulfite in hundred ml of distilled water and quenchant 2 includes 4gm solid C6H3N3O7 diluted in 100 ml ethanol. The revealed microstructure is shown in fig. 1.

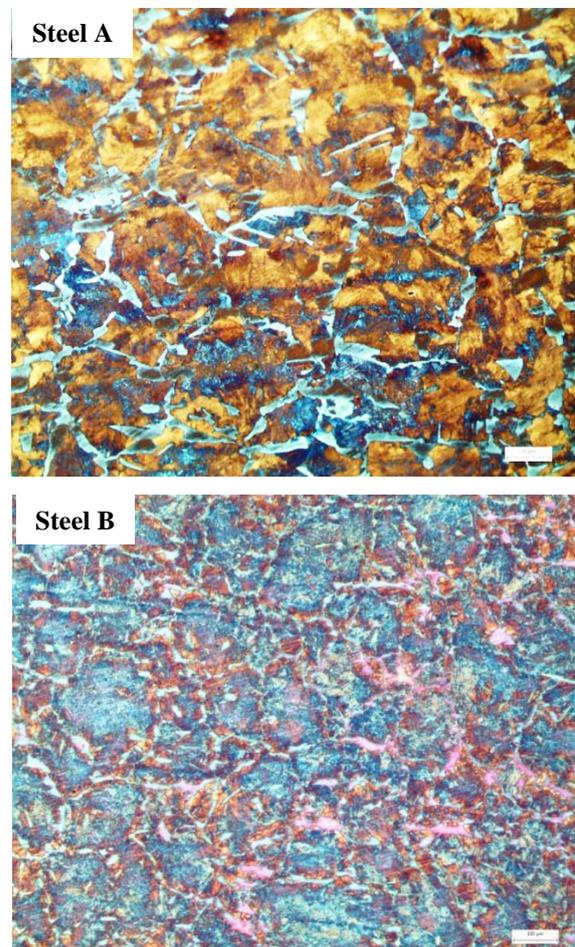


Fig. 1 (a): As rolled microstructure of steel A and steel B showing the ferrite and pearlite network. In steel A, coarse grains are existing but in steel B fine grain boundary network exists. This microstructure is before heat treatment in as hot rolled condition

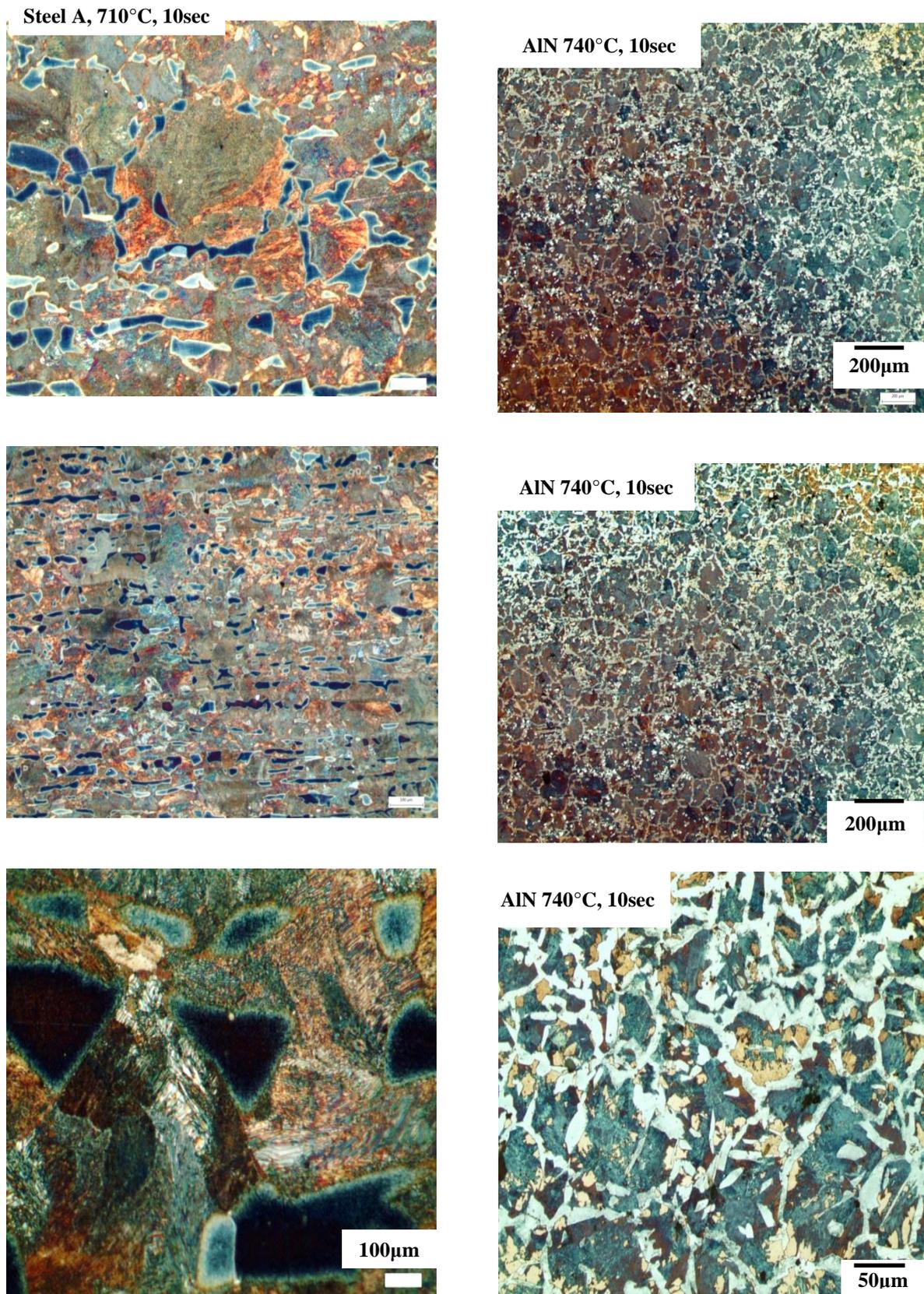


Fig. 1. (b): Microstructure of steel A and steel B at 740°C for 10 sec

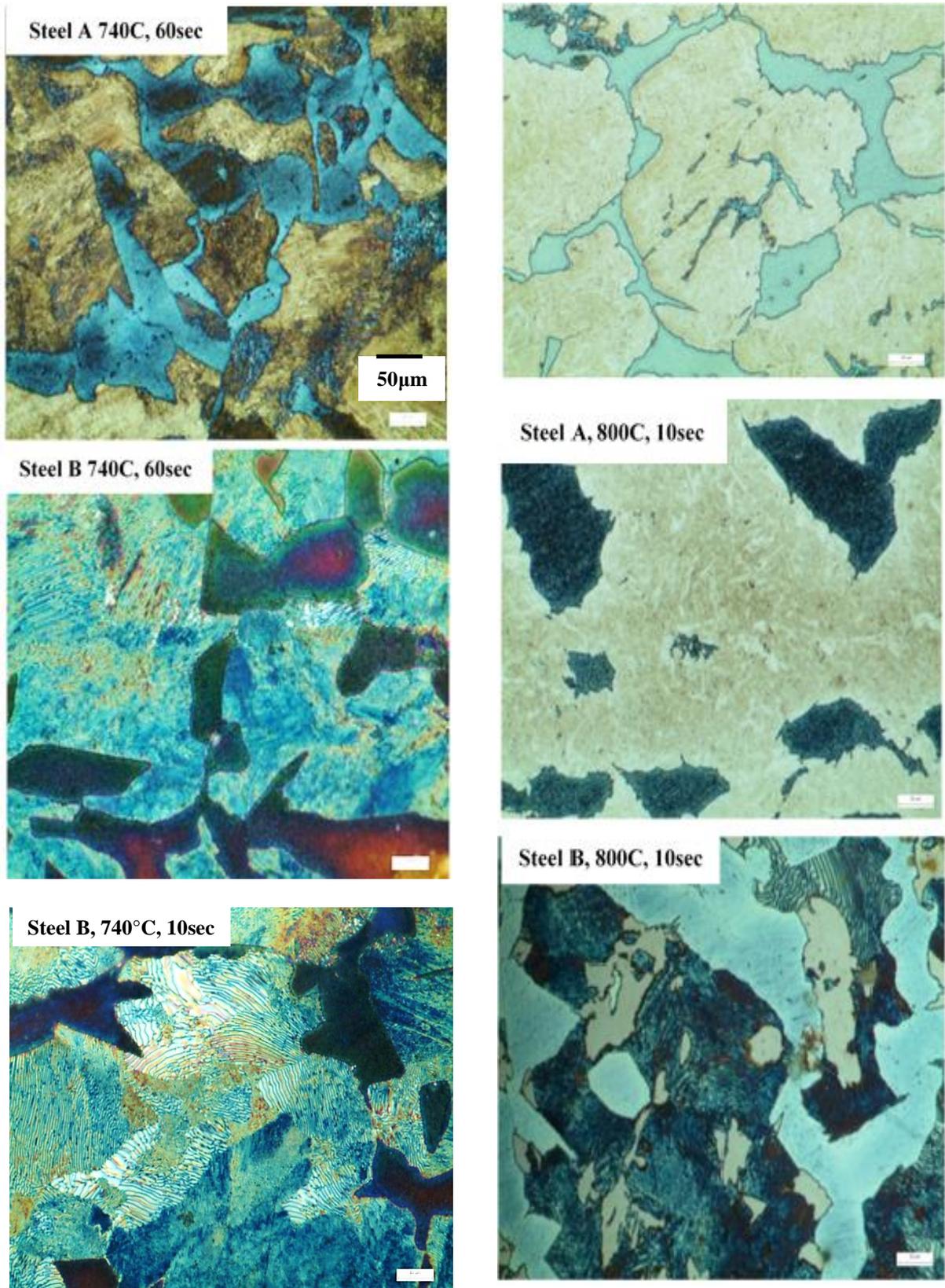


Fig 1. (c): Microstructure at 740°C for 10sec and 800C for 10sec showing the austenite has started to transform by consuming pearlite in steel A but in steel B austenite formation is lower than steel A due to the strong effect of AlN Nano particles that hinder the grain growth

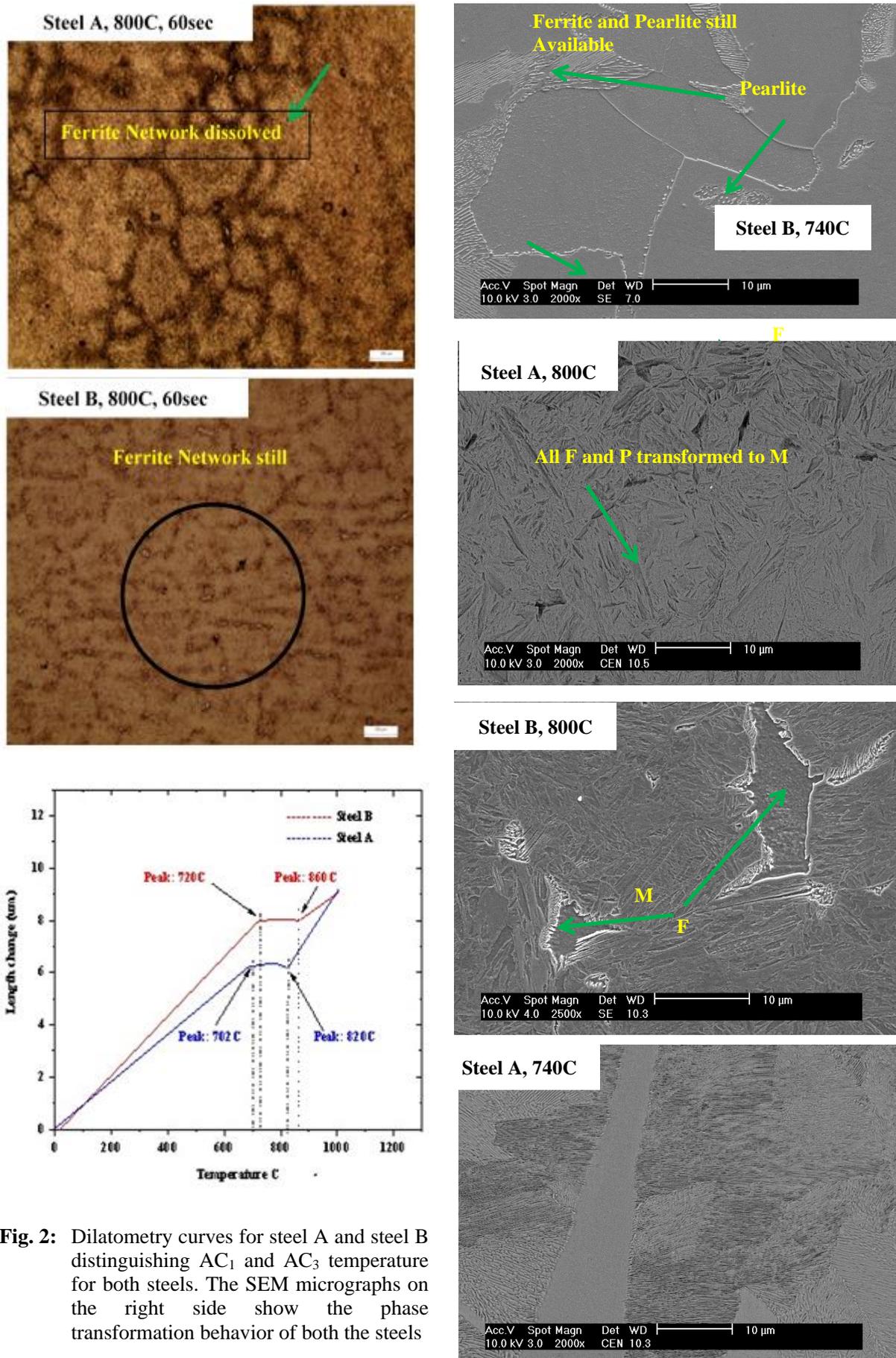


Fig. 2: Dilatometry curves for steel A and steel B distinguishing AC₁ and AC₃ temperature for both steels. The SEM micrographs on the right side show the phase transformation behavior of both the steels

The microstructure shown in fig 2 showing the austenite has started to transform by consuming pearlite in steel A but in steel B austenite formation is lower than steel A due to the strong effect of AlN particles that hinder the grain growth, as well as 800°C for both the steels A and B. In steel A at 740°C all the ferrite and pearlite has transformed to martensite after reaching at 800°C after 60sec holding. But in steel B even after reaching the temperature at 800°C still some ferrite network exists that shows the delaying the transformation and increasing the upper and lower critical temperatures due to the pinning of boundaries by AlN second phase particles.

3. Results and Discussion

Steel A (without AlN particles)

Microstructure at 740°C, 10,15,30,60 sec, soaking.

The austenite nucleation appears mostly at 740°C in our experimental steel. The holding time plays an important role to nucleate the new austenite nucleation. Nucleation of new gamma grains stimulated within pearlite colony. At 740°C, holding time of 10 and 15 sec respectively there is no change or negligible change in microstructure. The Aus. nucleation and growth were observed at the holding time of 30 and 60sec. The nucleation and growth sites are mostly pearlite areas with increasing time ferrite packet size increases a little, but pearlite phase slowly and gradually decreases to give rise to gamma phase nucleate. In the pearlite phase cementite plates provide carbon source to nucleate the Aus. grain at the ferrite-pearlite interface and also it is shorter distance to help for Aus. nucleation and growth.

Microstructure at 800°C, 10,15,30,60 sec

At higher temperature ranges nucleation and grain growth occurs at high rate. Microstructure becomes homogenous and packet size of the grains grown at large extent.

Steel B, (with AlN particles)

Microstructure at 740°C, 10,15,30,60 sec, soaking.

Combination of Nital and LePera solution has been applied to observe the microstructural features and it has been concluded that LePera etchant gives good contrast. Widmanstten Ferrite plates grow at austenitic temperature which is just above T₀, thus crystal structure change has been achieved by this deformation, and this change is in the form of martensite. In AlN steel, AlN particles

pin the prior grain boundaries above the lower critical temp; and hence hinder the grain growth as a resultant grain size is refined. In AlN steel phase transformation is observed to be slow as compared to steel A the possible reason is that kinetics of phase transformation is partially controlled by substitutional diffusion of Cr or Mn, Mn retards the reaction and these elements partition b/w the parent and product phase. Volume fraction of austenite in steel A is higher than AlN ST so it is strongly believed that AlN refines the grain size of prior austenite.

Microstructure at 800°C, 10,15,30,60 sec, soaking

In the aluminum contained steel three phases can be observed very clearly martensite, ferrite and some amount of bainite so it can be said that there is an inhomogeneity in the structure as compared to steel A. The amount pro-eutectoid is not completely disappeared and also the Black contour is finer than steel A, obviously the upper critical temperature is lower than Steel A. The substructure network in AlN steel is finer than steel A and it can be concluded that the ability of austenite formation in aluminum contained steel has been delayed. The velocity of pro-eutectoid ferrite to austenite transformation depends on how the supply of carbon source is available so the concentration of carbon near this black contour is lower in aluminum steel than steel A so this black contour which is assumed to be the original prior austenite grain boundary is finer than steel A.

a) Dilatometer observation

In order to better understanding make sure that AlN particles retard the grain growth of ferrite and pearlite when steel is subjected to heat treatment and it also delays the phase transformation of austenite and ferrite consequently the 1st transformation temperature AC₁ and the 2nd transformation temperature AC₃ will directly affect and it lowers in steel B as compare to the steel A as has been shown in the dilatometry curves. The first peak in steel A appears at a temperature of 702°C it means austenite transformation start at that temperature and forms the AC₁ line, similarly second peak in the steel a starts at 820°C that means austenite transformation finishes and it forms the AC₃ line. The case is different in the steel B, the first peak starts at 720°C and finishes at 860°C so it is clear and clear that in steel B AlN particles are the main source to alter the AC₁ and AC₃ temperature as well as amount of Austenite transformation is higher in steel A and lowers in steel B.

b) SEM micrographs

SEM micrographs were taken to support the optical micrographs. SEM result were observed at 740°C and 800°C for both the steel it was shown that at the 740°C after 60sec holding time in lead bath furnace in steel A some of the volume percent of eutectoid ferrite and pearlite has been dissolved to austenite and then martensite. This temperature is assumed to be near the lower critical line AC₁. Similarly at 800°C after 60sec almost all the proeutectoid ferrite as well as eutectoid ferrite and pearlite has been dissolved to austenite this is due to the absence of AlN particles. With respect to steel A, in steel B the process is different, at the end of 60sec at 800°C proeutectoid network has been broken and it does not dissolve completely and also same is the case with eutectoid ferrite and pearlite so it is concluded that lower critical and upper critical temperature of steel B is higher than steel A

4. Conclusion

Impact of Nano sized Aluminum Nitride Second phase Particles on Gamma and Alpha Phase Transformation in Less Carbon added Manganese Steel was studied and observed through experimental and metallographic examinations and it was found that Austenite growth is higher in pearlite colony due to shorter diffusion distances. Finger type growth morphology is observed Perpendicular to pearlite lath. Aluminum addition itself has also thermodynamic effects like, it delays the austenite phase transformation thus it can increase the lower and upper critical temperatures. AC₁ and AC₃ as shown in dilatometry curves. It can increase the grain coarsening temperature.

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