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Research Article

Aluminum and Aluminum Nitrides Effect on Nucleation Sites in Micro-alloyed Steel

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Abstract: In the present research work an investigation has been taken to understand the effect of aluminum addition on nucleation sites of AlN particles. Two steel samples have been taken with and without aluminum addition. Steel A has no aluminum and steel B has some aluminum added. These samples have been solution heat treated first at 1200°C to dissolve the particles if already present in the steel matrix, and then these samples were heat treated at 710°C, 740°C, 770°C and 800°C respectively with a holding time of 10 to 60 seconds and were then quenched. These samples were then grinded and polished using metallography steps and revealed the microstructure after etching in two solutions Nital and Picric acid simultaneously. After initial interrogation by using the optical micrographs the SEM was utilized to further disclosed the nucleation morphologies. It was found that the 740°C with holding time of 15sec is the most critical stage to observe the nucleation sites. After having the SEM, it was concluded that for steel A, has the most preferable nucleation sites are ferrite and pearlite regions and surprisingly in steel B having aluminum addition the nucleation sites were interface of pro-eutectoid ferrite and pearlite. Optical, SEM and TEM characterization techniques were utilized to study the behavior and morphology.

Keywords: Aluminum, AlN (Aluminum nitride) particles, Lead bath, Nucleation, Austenite formation.

1. INTRODUCTION

Most research work has been done so far on refining the grain size of steel by utilizing aluminum nitride particles (AlN) and these particles are directly involved to control the prior austenite grain size which in turn will form the martensitic upon quenching and martensitic increases the strength and hardness of the steel none of anyone has studied so far the effect of actual nucleation sites for austenite formation as a result of pining effect of AlN particles. Aluminum conventionally is used as deoxidant element by steel smelters, de-oxidation of steel with aluminum is very common practice today and plays a crucial role in ladle metallurgy [1, 4]. Aluminum forms aluminum oxide and decreases the amount of oxygen in the steel and facilitates the production of killed steels [5]. The addition of aluminum not only results in formation of alumina inclusions but also affects the inclusion chemistry [6]. As with other metallic elements such as vanadium, titanium and niobium, aluminum, in the presence of nitrogen, can form aluminum nitride [7]. Researchers working on grain refinement technique, they add deliberately aluminum in combination with nitrogen to form the second phase particles commonly known as aluminum nitride (AlN) particles, and they can form during melting and casting [8, 10]. 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 [11]. 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

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grain refinement but during the last twenty years none of anyone has given attention that aluminum nitride particles can be useful for grain refinement technique but nucleation sites for forming these nitride particles are more valuable to be studied and investigated to know their critical effect on limiting the grain boundary movement [12, 15]. 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 [16, 17]. This riddle and unrevealed features of AlN particles has been studied in the present research work. Aluminum conventionally is used as deoxidant element by steel smelters, de-oxidation of steel with aluminum is very common practice today and plays a crucial role in ladle metallurgy [18, 25]. The addition of aluminum not only results in formation of alumina inclusions but also affects the inclusion chemistry. As with other metallic elements such as vanadium, titanium and niobium, aluminum, in the presence of nitrogen, can form aluminum nitride [26, 32].

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 steel A will be termed as base steel and steel B will be termed as AlN steel throughout this manuscript. The content of nitrogen is also present in both the steels under investigation in this work. The chemical composition of both the steels is given in Table 1.

As received material was obtained through the process, as shown in Fig. 1. Steps: Solution treatment (two hours at 1200°C) hot rolling (rolling reduction of 80% over a four -pass) Specimens cut (800°C, after cutting air-cooling). 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.

The un-mounted samples were brought into heat treatment process as shown in cycle in Fig. 2. 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 Ac1or lower critical line of the experimental 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

Table 1. Chemical Composition of Experimental Steel

Weight%	C	Si	Mn	Cr	Al	N
Steel A	0.463	0.241	0.872	-	-	0.005
Steel B	0.454	0.255	0.778	0.152	0.033	0.011

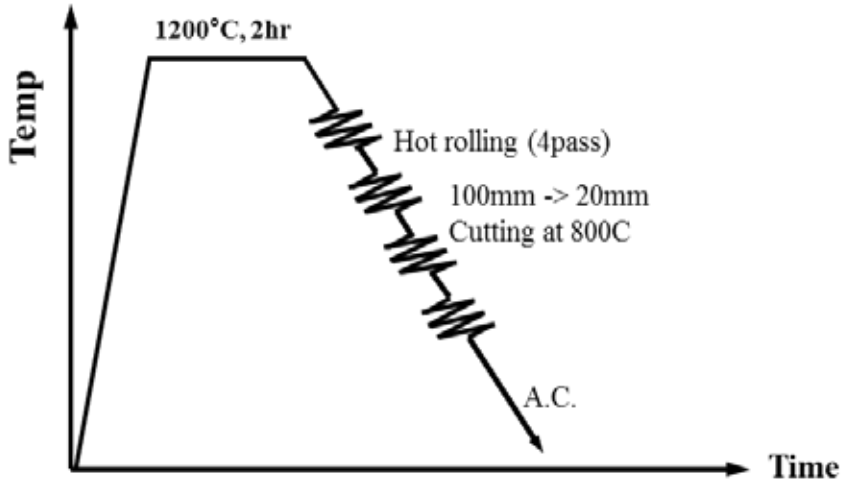

Fig. 1. Deformation Process of Experimental Steel A & Steel B

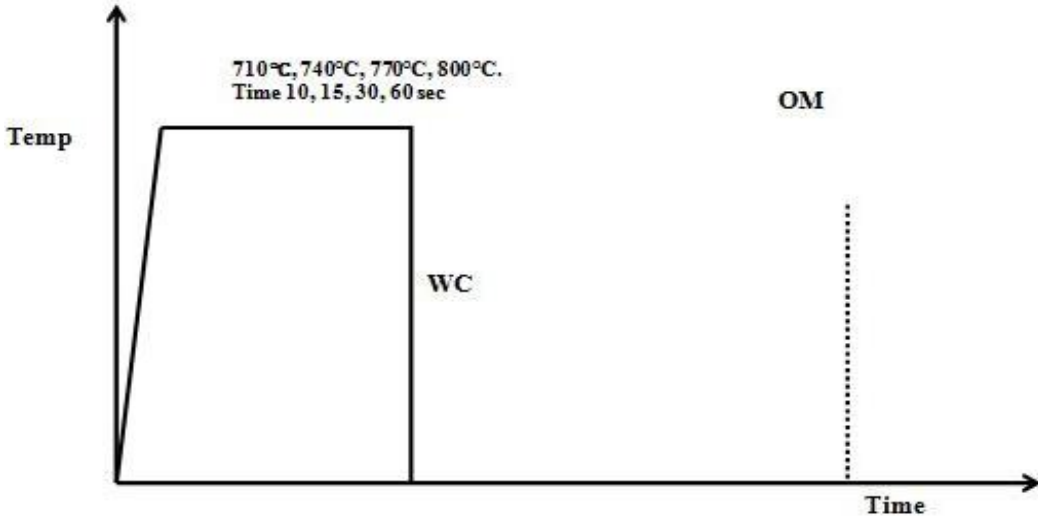

Fig. 2. Heat Treatment Cycle for Both Steel A & Steel B

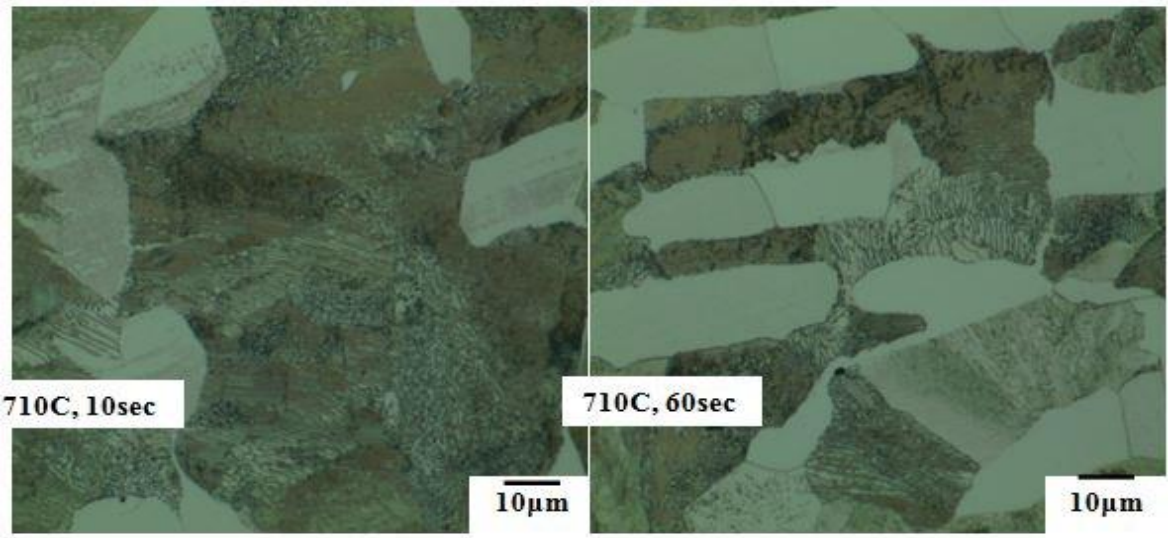

Fig. 3. Microstructure of as received samples

and polishing process was performed for 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 purpose. Two step etching was performed firstly in 2% Nital and then in 4% Picric acid solution total time for etching was 5 to 10sec in Nital and 20 to 40 sec in Picric acid. The revealed microstructure is shown in Fig. 4.

3. ANALYSIS AND DISCUSSION

The nucleation affects overall austenite formation kinetics in both the steel. In steel A, austenite phase transformation is rapid but in steel B austenite formation is retard by the AlN particles. This effect can be seen in optical micrographs, SEM images.

For keeping all the results of optical microscope and SEM it was found that the nucleation of austenite is not found at 710°C the reason for this is the 710°C is much lower than the first critical

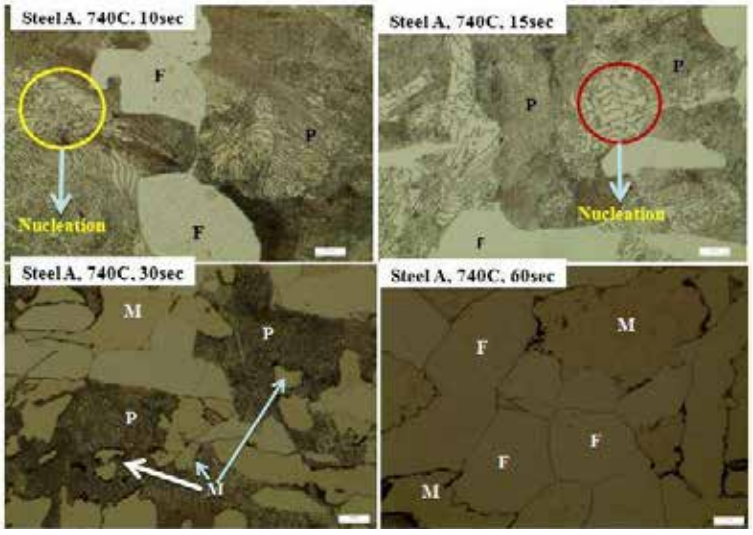

Fig. 4. Microstructure at 740°C, showing the possible nucleation sites.

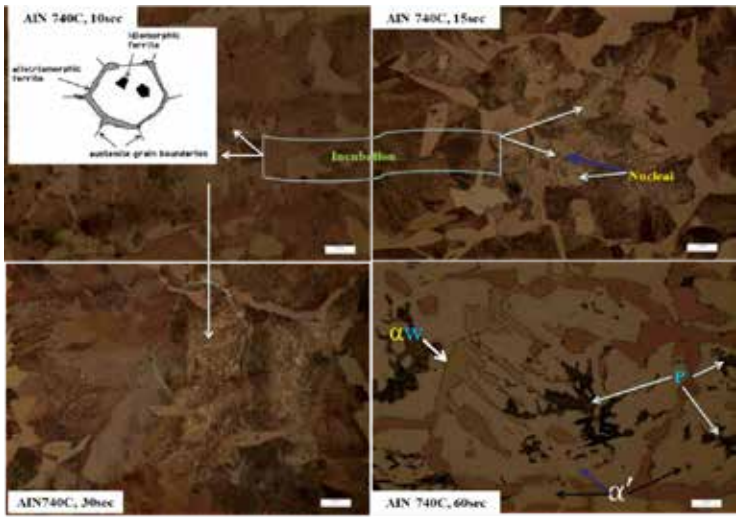

Fig. 5. Nucleation and austenite phase formation with respect to holding time.

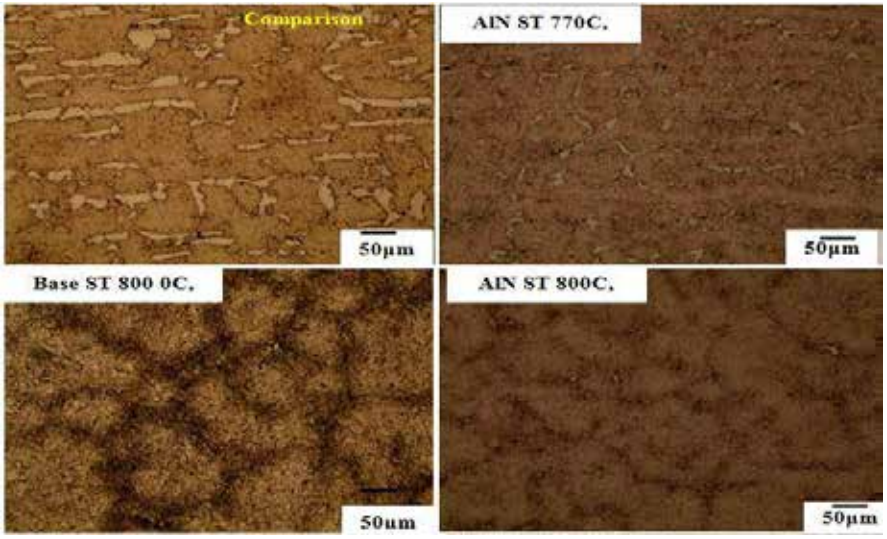

Fig. 6. Comparision of steel A and steel B. Aluminum contained steel shows delayed phase transformation due to presence of second phase particles.

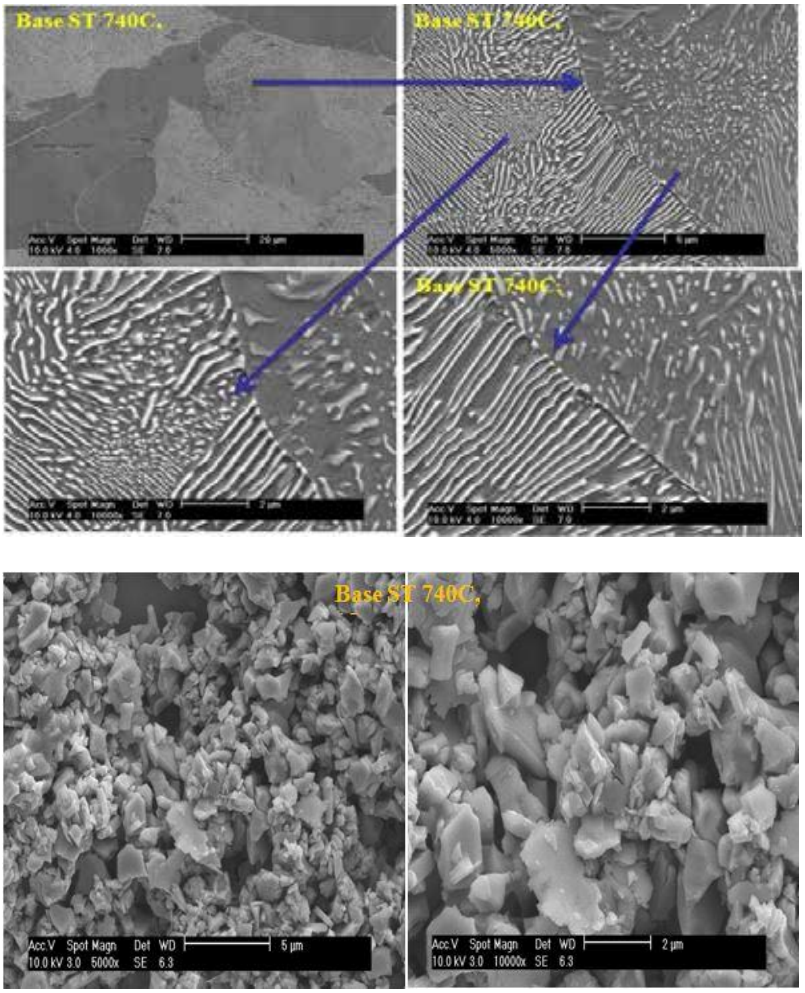

Fig. 7. SEM micrographs indicating the nucleation sites in steel A at 740°C

temperature i-e lower than AC1. The austenite nucleation stimulate from the 740°C for both the steels however holding time is the major difference between two steels at this temperature. Therefore 740°C temperature is the most important stage in all temperatures in this research work. The detail discussion is mentioned below:

3.1 For Steel A

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 Aus. grains takes place predominantly within pearlite colony. At 740°C, holding time of 10 and 15 sec respectively there is no change or negligible change in microstructure. The Austenite 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 Aus. 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. The driving force for austenite nucleation is the change in Gibbs free energy. In the pearlite phase cementite plates provide carbon source to nucleate the austenite grain at the ferrite-pearlite interface and also it is shorter distance to help for austenite nucleation.

3.2 For Steel B

In steel B 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 B so it is strongly believed that AlN particles refine the grain size of prior austenite. For steel B, its surprisingly to observe that the nucleation sites for austenite in this steel is not controlled by the pearlite region but it is stimulated from the pro-eutectoid ferrite and pearlite interface where the source of carbon is the cementite particles present.

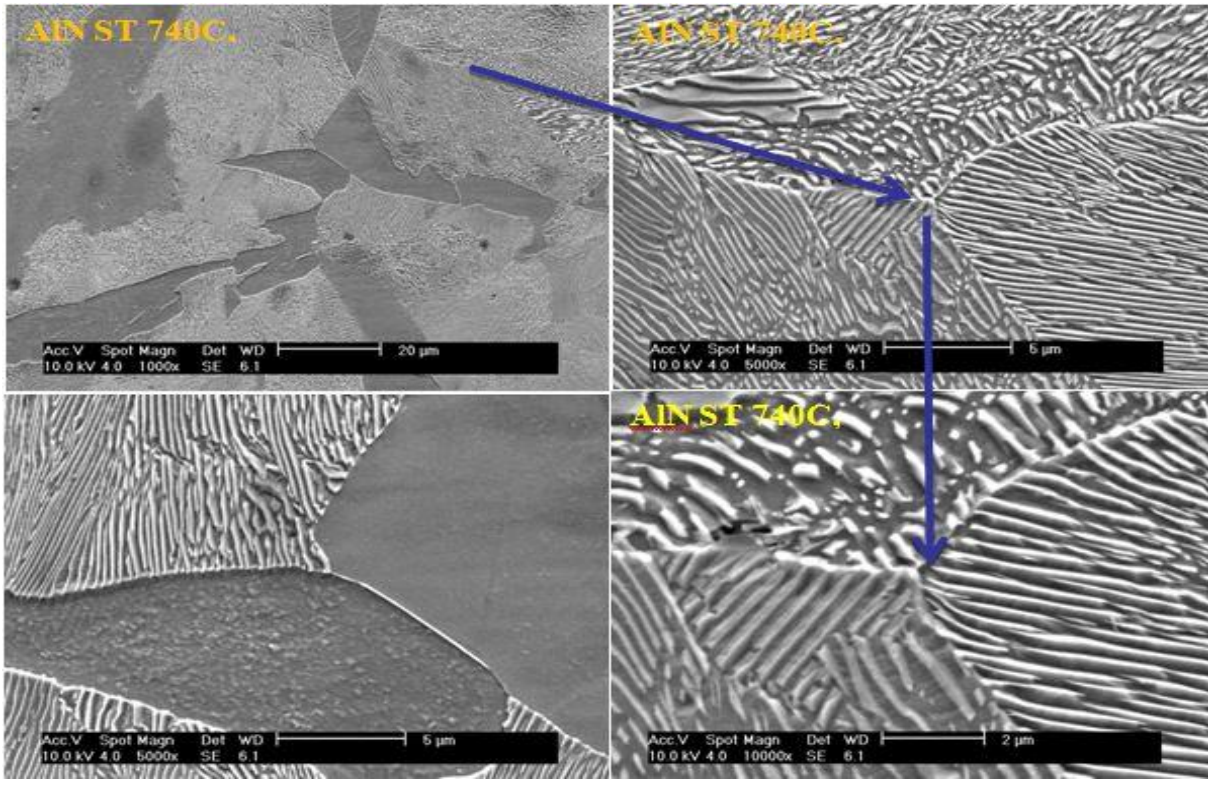

Fig. 8. SEM micrographs at 740°C indicating the nucleation sites in steel B

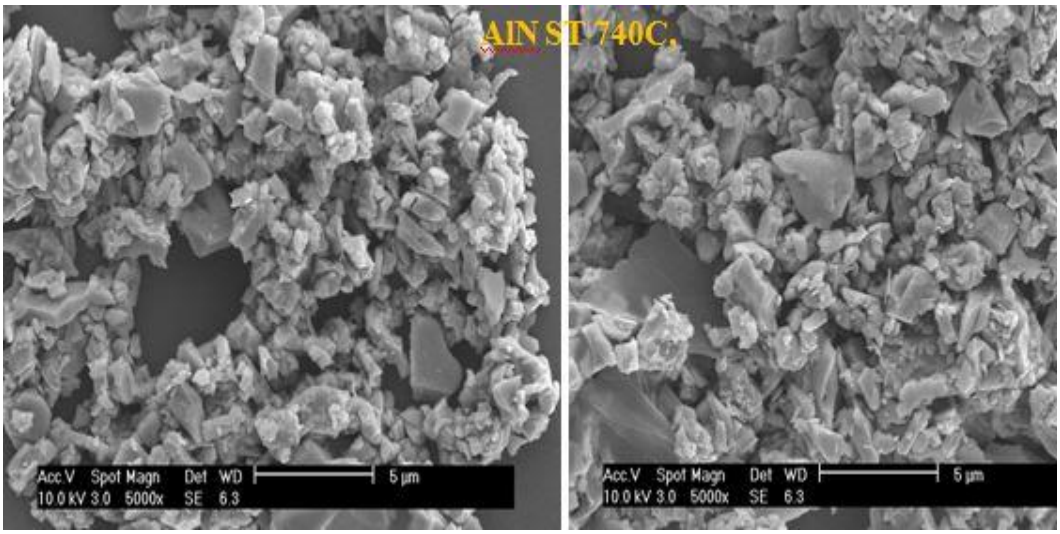

Fig. 9. SEM microstructure showing the comparision of both steels at 740°C

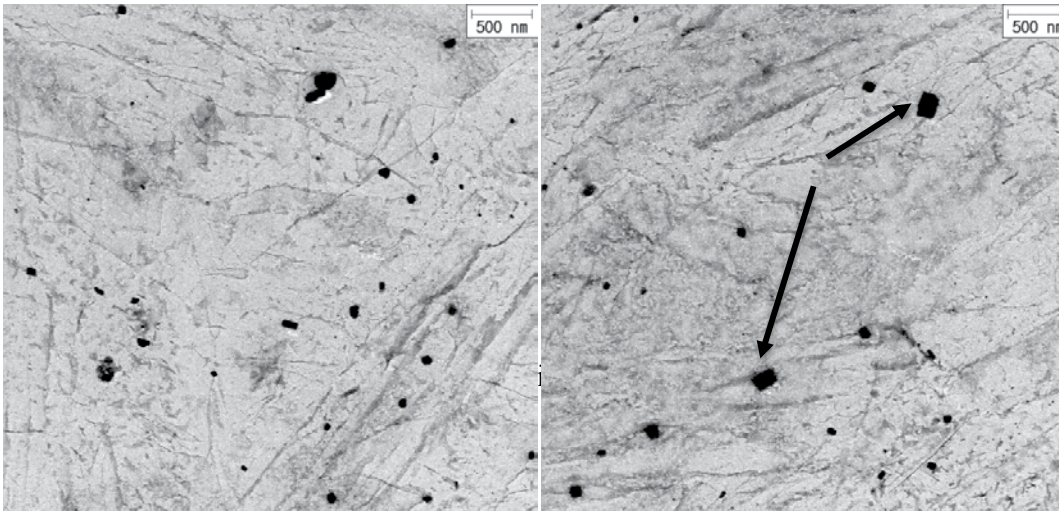

Fig. 10. TEM micrographs showing presence of AIN particles in steel B.

4. ACKNOWLEDGMENTS

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