Understanding the Effect of Aluminum Addition on the Forming of Second Phase Particles on Grain Growth of Micro-Alloyed Steel

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Abstract—**The formation of second phase particles in the steel matrix during melting and casting plays an important role in controlling the grain size of steel. An attempt is made in the present work to find the role of nitrogen on forming nitride particles either with aluminum or titanium. Two steel samples with the same titanium and aluminum weight percent in their chemical composition were collected after the hot rolling process. Solution heat treatment at 1350°C for 60min holding time was used to dissolve the particles and then the steel samples were reheated at 800°C for 60min, water quenched and their microstructure was revealed by usual grinding and polishing process using 2% Nital. A transmission electron microscope connected with EDS was used to reveal the morphology of the second phase particles. The samples for TEM analysis were prepared by the replica extraction method in 5% Nital solution. The samples were then caught in 3mm copper grid for TEM analysis. TEM micrographs revealed the second phase particles in the matrix of steel. EDS peaks were studied and titanium peaks were found in both samples and surprisingly there was not any peak found for aluminum.**

Keywords-aluminum; steel; grain; morphology; micrographs

I. INTRODUCTION

Researchers working on grain refinement add deliberately aluminum in combination with nitrogen to form second phase particles, commonly known as aluminum nitride (AlN) particles, which can be formed during melting and casting [1-5]. These are strong impurity elements which get together around the grain boundaries and provide dragging force that stops the mobility of the grain boundary when the steel is reheated or during heat treatment. Consequently, they retard the grain growth and the grains remain finer [5-11]. This is a common and widely used technique adopted for increasing the strength of steel. It was found that it is the most cheap and easy method of grain refinement, but during the last twenty years there was not paid attention to the fact that the nucleation sites for forming these nitride particles are more valuable regarding their effect on limiting the grain boundary movement [11-14].

II. EXPERIMENTAL PROCEDURE

Grain size control is always a priority in obtaining small grains which simultaneously increase hardness and toughness. Second phase particles are therefore a useful source for controlling the grain growth and grain growth is a decisive factor in increasing steel's strength. When aluminum and titanium are combined with nitrogen, they can form aluminum nitrides and titanium nitrides respectively because nitrogen has good affinity with both elements. Whenever titanium and aluminum are the alloying elements at the same time, it is quite difficult to find if nitrogen will be combined with only aluminum, only titanium or with both elements to form nitride particles, which, due to their higher melting point and stability, remain in the solid state when the steel is cooled from its melting point. This investigation took place in order to study and understand this effect. In this regard, two steels were obtained by hot rolling as shown in Table I and Figure 1.

TABLE I. STEEL SPECIMENS COMPOSITION

Steel	A	B
C	0.202	0.2
Mn	1.04	1.04
Al	0.042	0.042
Ti	0.025	0.025
Nb	0.047	-
B	0.0018	0.0019
N	0.0058	0.0061
Si	0.25	0.251
Cr	0.144	1.21

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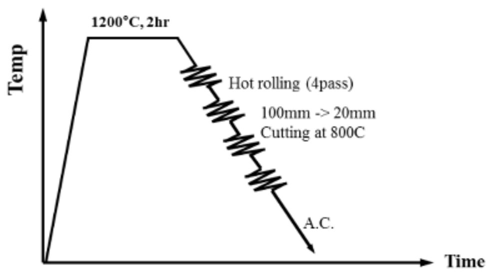

Fig. 1. Coooling curve

After hot rolling, both samples, A and B, were solution heat treated at 1350℃ for 60min holding time to dissolve the particles in the solution at high temperature. After holding, the samples were rapidly cooled in tap water as shown in Figure 2.

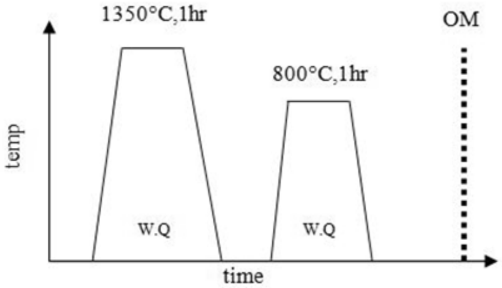

Fig. 2. Heat treatment cycle

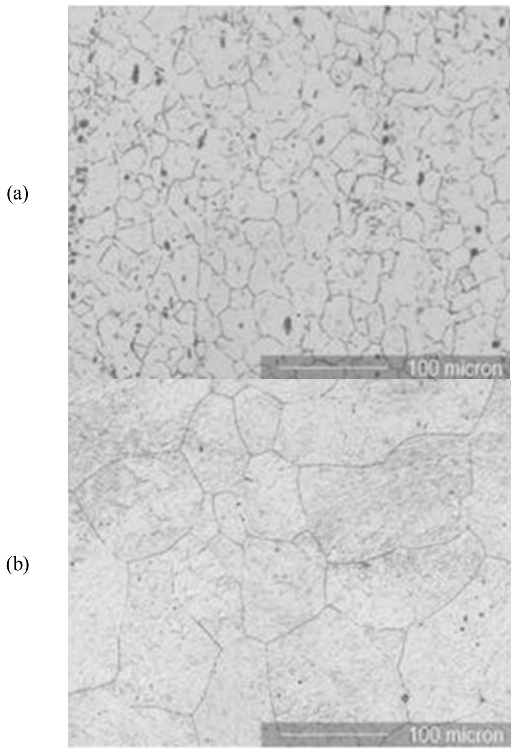

Fig. 3. Optical micrographs (a) 800℃, (b) 1350°C

To investigate the effective influence of particles, both samples were re-heated at the moderate temperature of 800°C for one hour and were then quenched. After the heat treatment process, the conventional grinding and polishing steps followed by etching in 2% Nital and thus the samples revealed their microstructure (Figure 3). Immediately afterwards the samples were prepared for transmission electron microscopy (TEM) by using the carbon extraction replica method as shown in Figure 4. Etching is an important method in this technique. Usually it is performed in 3% to 5% Nital solution because the precipitates exist in the steel matrix and therefore it is necessary to etch the steel matrix with etchant (5% Nital) to drag the particles outside. The particles were then kept in the copper grid and were used for TEM analysis. TEM, connected with the EDX, is a powerful instrument in revealing the second phase particles.

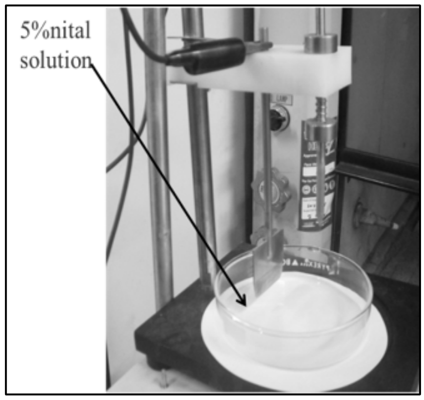

Fig. 4. Extraction of carbon replica by using etching cell

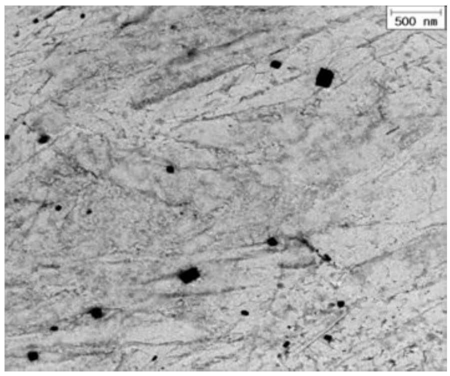

Fig. 5. TEM micrographs for sample A

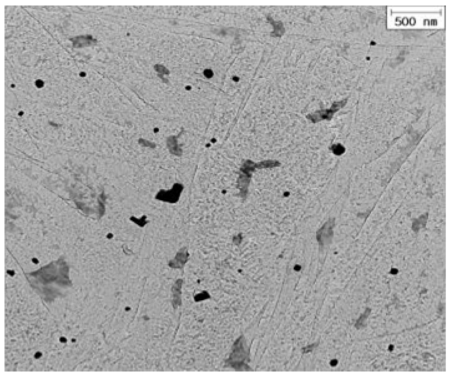

Fig. 6. TEM micrographs for sample B

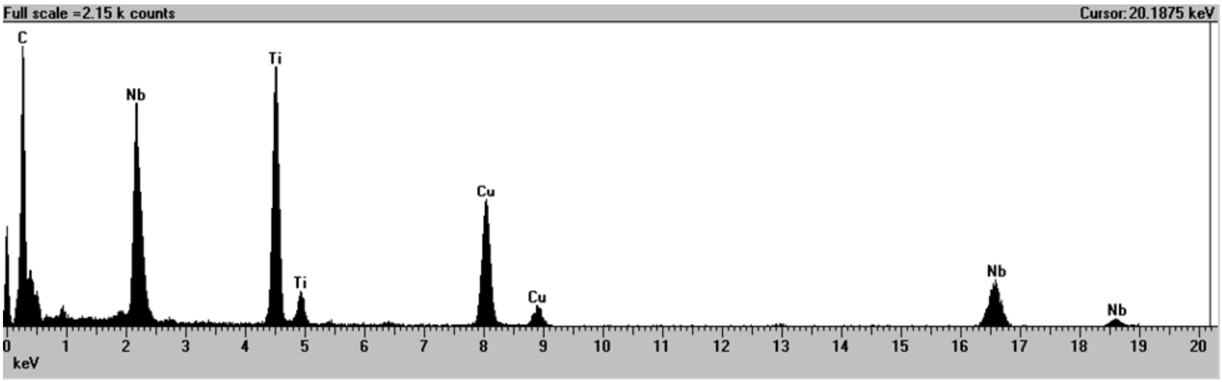

Fig. 7. EDS peaks of sample A

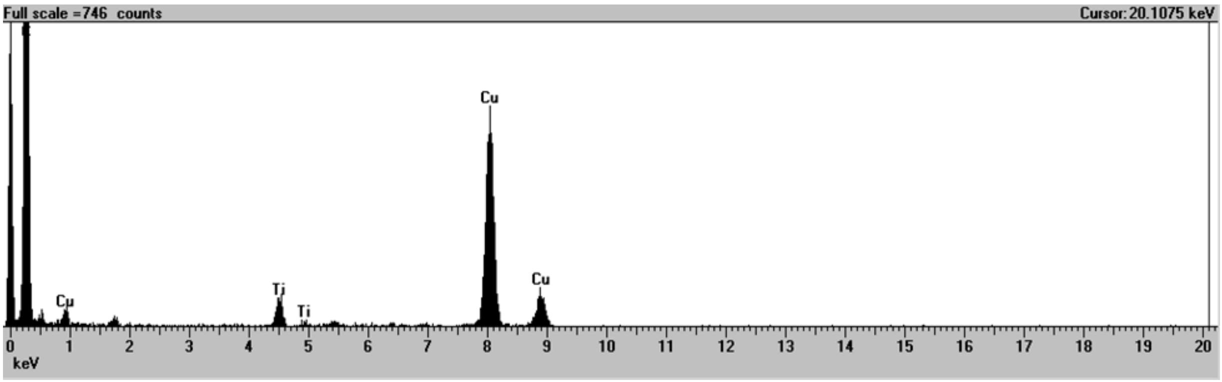

Fig. 8. EDS peaks of sample B

III. DISCUSSION

To understand and discover the effect of aluminum addition on the forming of second phase particles on grain growth of micro-alloyed steel, a thorough investigation has been taken in this work. Optical microscopy and TEM analysis have been utilized by keeping in view the EDS peaks. The relative atomic mass of Titanium is 48 and of Nitrogen is 14. The weight percent of titanium in steel sample A and steel sample B is 0.025. The weight percent of nitrogen required for forming TiN is $(0.025\times14)/48=0.0073$. It is therefore observed that although aluminum always shows affinity with nitrogen to form nitrides, in this case aluminum was combined with Titanium to form titanium nitrides. The reason behind this is that the amount of nitrogen required to form the aluminum nitrides is not sufficient and a large portion of nitrogen has been consumed by the titanium.

IV. CONCLUSION

On the basis of stoichiometric calculation, it is concluded that all the nitrogen atoms present in steel A and steel B were consumed by titanium to form TiN. Therefore, the EDS spectrum from TEM extraction replica experiments show only the peaks for titanium in sample A and B. AlN probably did not form at all and the EDS spectrum of particles in steel A and steel B confirms that result.

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