Determination of Austenite Grain Size in Titanium and Titanium Free Micro-alloyed Steels by the Combination of Heat Treatment and Thermal Etching TE

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Abstract—**To reveal the prior-austenite grain boundaries in micro-alloyed steels there is different techniques. This study has been carried out over a wide range of temperature (950-1150 ^{o}C) and it has been found that the tested procedure based on the combination of heat treatment and thermal etching (TE) method give excellent results for the Titanium and Titanium free medium carbon micro-alloyed steels at all the austenitization conditions tested.**

Index Terms: **Micro-alloyed steel: Prior austenite grain size, Thermal etching.**

I. INTRODUCTION

The mechanical properties of heat-treated alloy steels are strongly influenced by the grain size of the parent austenite phase before quenching. But revealing the prior-austenite grain boundaries (PAGB) can be quite difficult depending upon the alloy and its microstructure [1] and the development of effective etchants for revealing the prior-austenite grain boundaries in steels of different composition, Microstructure and properties are of significant importance in many areas, for example, in the development of new alloys, in structure property correlation, in steel quality evaluation, and in failure analysis [2,3]. Also the structural changes play an important role in determining the final microstructure and properties of high strength low alloy steels, also as we know, the prior austenite grain size (PAGS) exerts an important influence on the decomposition of austenite [4]. For that reason the accurate determination of the prior-austenite grain size (PAGS) of materials become very important in metallurgical studies. The effect of austenitizing temperature on as reheated γ grain size is given in series of literatures [5-7, 14] i.e grain refinement depends on as–reheated grain size of austenite. So the aim of this work is to study the influence of austenitization temperature Tγ ($^{\circ}$**C**) and austenitization time tγ (s) on austenite grain size of Ti/Ti-free micro-alloyed steels and effect the combination of heat treatment and thermal etching (TE) on the ability to reveal the austenite grain boundaries in the whole range of austenitization temperatures tested.

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II. EXPERIMENTAL

Two micro-alloyed medium-carbon forging steels with and without 0.01%Ti addition have been studied , the chemical compositions of these steels are given in table 1. The steel used for this investigation were made by laboratory and full-scale casting and fabricated into 22 and 19 mm diameter bars by full-scale hot-rolling without subsequent heat treatment. Representative hot-rolled steels were normalized from temperature range of 950-1300 ^{o}C .

Table 1. Chemical Composition Of The Experimental Steels By (wt%)

Steel Type		Chemical Composition by wt%
A	**B**	
0.309	0.256	C
0.485	0.416	Si
1.531	1.451	Mn
0.0077	0.0113	P
0.0101	0.0112	S
0.011	0.002	Ti
0.123	0.099	V
0.0221	0.0235	N

III. HEAT TREATMENT

The samples with 19 and 22 mm diameter and 12mm in length of the two steels were used to reveal grain boundaries by thermal etching (TE) method. For that purpose the surface of samples polished and finished with 1 µm diamond paste. Later on, those samples were heat-treated in radiation furnace at different heating temperatures to the austenitization condition listed in Table 2. In order to yield distinct prior-austenite grain size (PAGS). After that the samples were quenched in water and afterwards tempered at 450 °C, for 24 h, then slowly cooled in natural air to room temperature. A scheme of this heat treatment is shown in Figure 1. As mentioned in many studies the temperature against time needed to outline the prior-austenite grain boundaries by promote the formation of allotriomorphic ferrite or proeutectoid ferrite homogeneously precipitated at grain boundaries [8, 9]. Etching reagents based on saturated aqueous picric acid plus a wetting agent seem to give the best results in quenched and tempered steels [9]. The chemical etchant that gave positive results for this steel revealing the prior-austenite grain boundary at all austenitization temperatures was a solution formed by100 ml of distilled water, 10 g of picric acid ($C_6H_3N_3O_7$), 50 ml of sodium alkylsulfonate (Teepol), 1 ml of HCl. However, it is relevant to point out that this solution is used hot (90 °C), then the polished specimen was immersed into the solution. The etchant time is usually in the range of 25 sec.

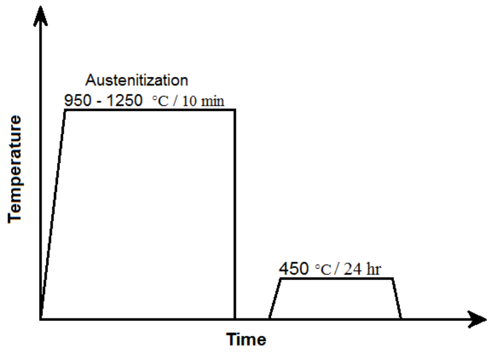


Figure 1. A Scheme Of Heat Treatment.

Table 2 : Austenitization Condition

Steel	Tγ (°C)	tγ (s)
A	950, 1050,1100,1150,1250	600
B	950, 1050,1150,1150,1250	600

Tγ is austenitization temperature and tγ is austenitization time.

IV. GRAIN SIZE MEASUREMENTS

The austenite grain size control is an important factor in development of the final mechanical properties of the product [10]. There are three types of errors that can influence grain size estimates. The first arises from experimental limitation for example limited resolution, poor grain boundary delineation, over-etching, inaccurate test line length measurement, miscounting. The second error type is due to improper sampling and the third error type arise from the representativeness of the chosen areas and specimens to the entire material, the last two error types are usually the greatest source of errors [1]. The standards for the determination of grain size are set in ASTM E112 and DIN EN ISO 643 [11, 12].

Table 3. Prior Austenite Grain Size (PAGS) Results

Steel	T_{VN} dissolution temperature, °C
A	1130
B	1114

V. RESULTS AND DISCUSSION

The combination of heat treatment and chemical etchant was able to reveal the austenite grain boundaries in the whole range of austenitization temperatures tested (Table 2). Figure 3 gives evidence of the success of this procedure to reveal the grain boundaries in Ti and Ti-free steel. The experimental results of the PAGS for this steel are listed in table 3 and also shown in Figure 2. As we can see from the Figures 3 and data listed in the Table 3, the austenite grain size was still smaller when the austenitization temperature Tγ is equal 950 °C (Figure 3 a,b), slightly start increase when Tγ = 1050 °C (Figure 3c,d) and above this temperature the grains coarsen rapidly (Figure 3 e - j). The reason is because we are in temperature ramge higher than the precipitation dissolution temperature.

Table 4. Calculated Temperature For Complete Dissolution Of VN

Austenitization temperature Tγ, °C	Prior austenite grain size (PAGS), µm	
	Steel A	Steel B
950	9 ± 1	9 ± 1
1050	35 ± 5	28 ± 3
1100	60 ± 5	50 ± 5
1150	64 ± 3	75 ± 5
1250	80 ± 10	100 ± 10

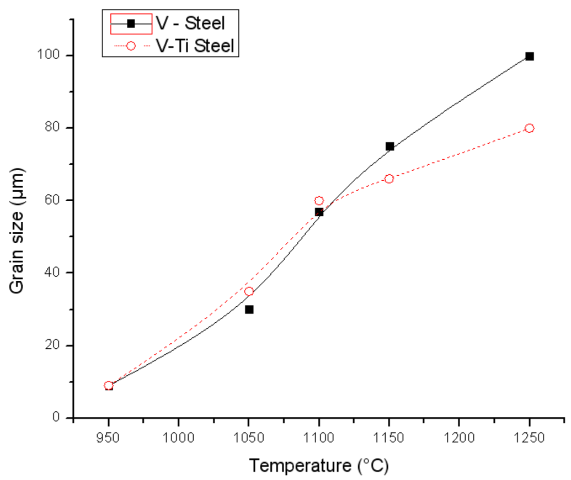
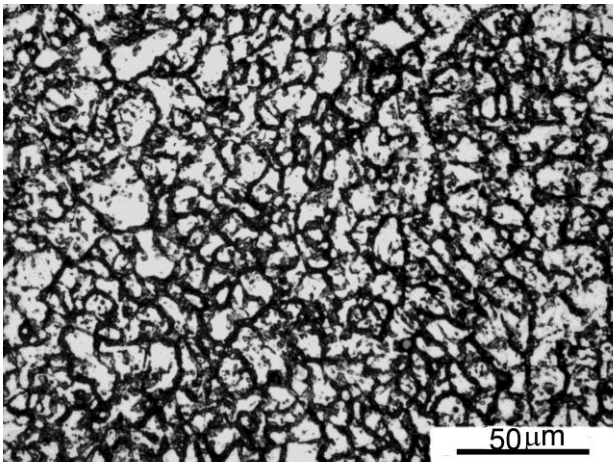


Figure. 2 The Grain Size As a Function Of Austenitization Temperature (Soaking Time 600 s.).

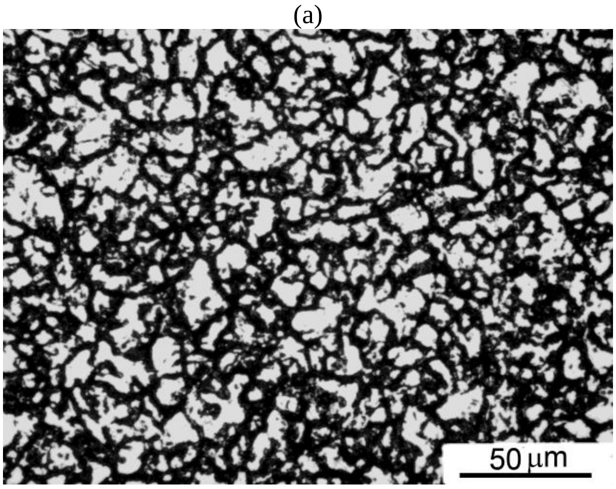
The equilibrium temperatures for complete dissolution of VN was calculated according to the equation (1) [13] and the corresponding results are given in Table 4.

$$\log[V][N] = -7840/T_{VN} + 3.02 \quad (1)$$

Also the result indicate that, when the precipitate is in the solid solution, it acts as solute drag (weak pinning),but if temperature lower than precipitation dissolution temperature the MEA will precipitate and retard grain growth i.e grain size inhibitor and acts as strong pinning.



(a)



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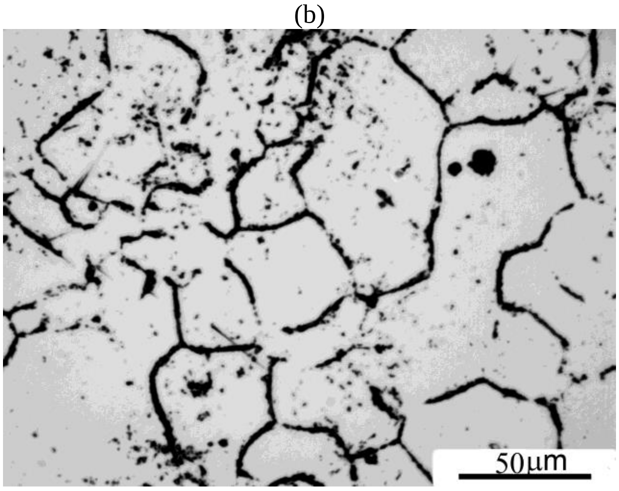
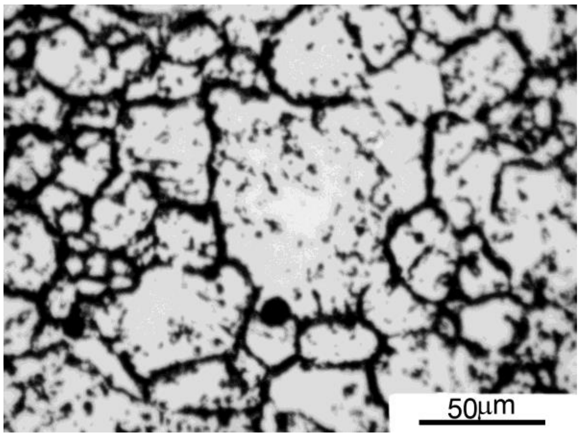


(c)



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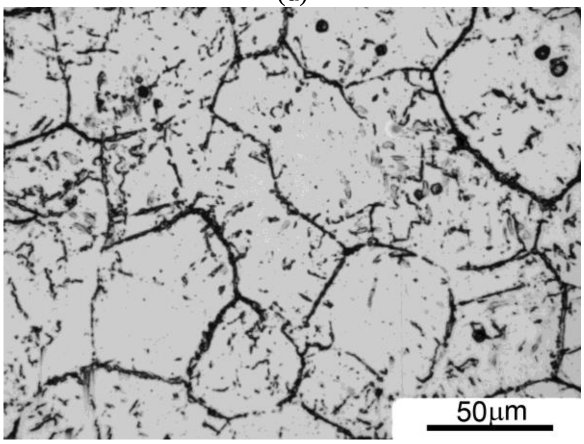
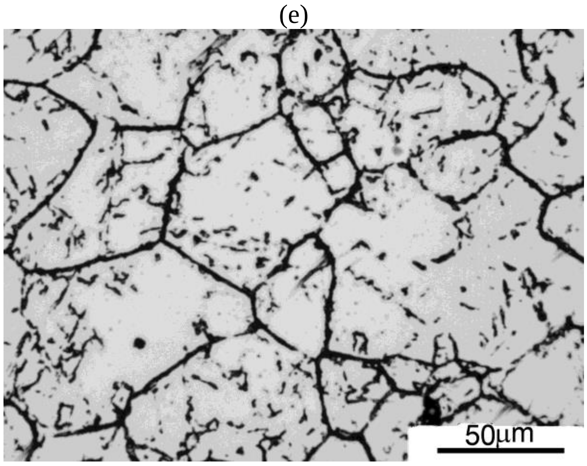
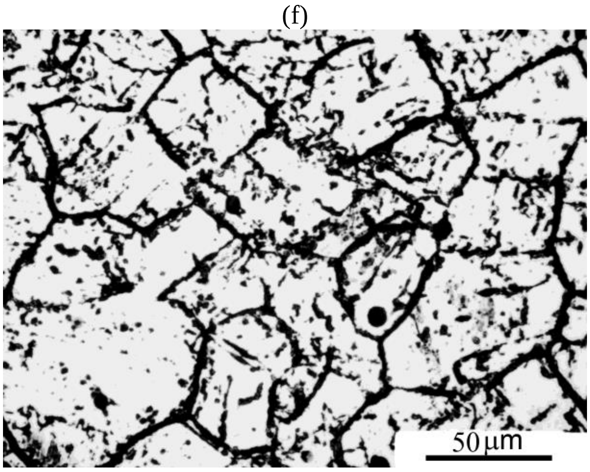


(e)



(f)



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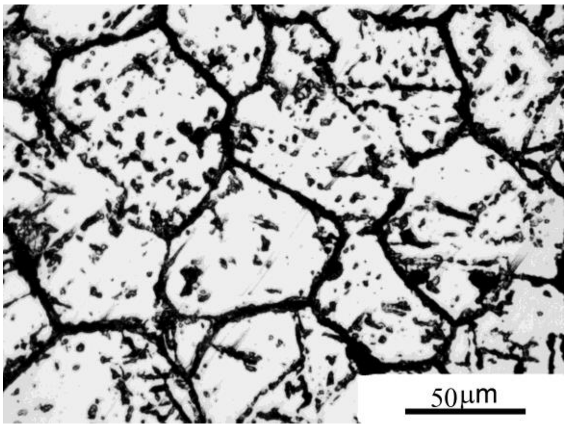

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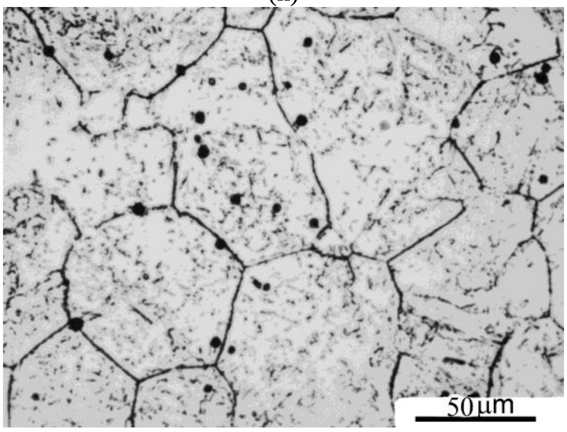
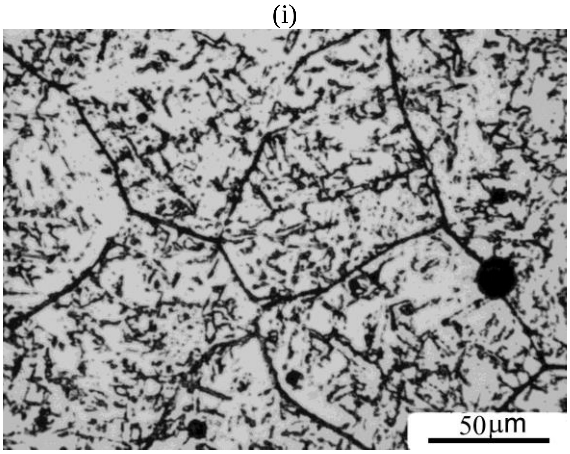


(i)



(j)

Figure. 3. Light optical micrographs showing revealed prior austenite grains at different austenitization conditions (a, b) 950 °C, (c, d) 1050 °C, (e, f) 1100 °C, (g, h) 1150 °C and (i,j) 1250 °C, for steel A and B revealed by the combination of heat treatment and TE.

VI. CONCLUSIONS

The tested procedure based on the combination of heat treatment and thermal etching (TE) method for revealing the prior-austenite grain boundaries gives excellent results for the Ti/Ti-free medium carbon microalloyed steels at all the austenitization conditions tested.

The austenite grain size control is an important factor in the development of the final mechanical properties of the product. The experimental results of the prior-austenite grain size (PAGS) for the steels investigated is smaller when the austenitization temperature $T\gamma$ is equal 950 ^{o}C , start slightly to increase when $T\gamma$ = 1050 ^{o}C and above this temperature the grains coarsen rapidly due to the effect of precipitation dissolution temperature.

REFERENCES

[1] Practical Application of Quantitative Metallography, V. Voort, American Society for Testing and Materials, 1984.

[2] K Cho, H Sim, J Kim, J Choi, K Lee, H Yang. A novel etchant for revealing the prior austenite grain boundaries and matrix information in high alloy steels. Mater Charact 2008, 59:786-793.

[3] L. Zhang and D. C. Guo. Ageneral Etchant for Revealing prior-Austenite Grain Boundaries in Steel. Mater Charact 1993,30:299-305.

[4] C. Garcia de andre, S. C. Capdevila, D. San martin, F. G. Caballero, J. Of Mat. Sci. 20, 2001, 1135 – 1137.

[5] Roman M .Kuziak and Yi –Wen Cheng , Microstructural Evolution In Microalloyed Medium –Carbon Forging steels During Thermomechanical Processing , international conference on processing , microstructure and properties of microalloyed and other modern high strength low alloy steel , Ed by A .J .DeArdo , The iron and steel socity , pittsburgh (1991) , p51

[6] Isao Kozasu , Recent developments of microalloyed steel plate , , in HSLA steels technology & application , Ed . Michael Korchynsky , American Society For Metals , Pennsylvania (1984), p593

[7] C. Garcia – Mateo, C. Capdevila, D, F. G. Caballero and C. Garcia de andre, ISIJ, 48, 2008, 1270-1275.

[8] C. Garcia de Andres, F.G. Caballero, C. Capdevila, D. San Martin. Revealling austenite grain boundaries by thermal etching: advantages and disadvantages. Mater Charact 2003,49:121-127.

[9] C. Garcia de Andres, M.J. Bartolome, C. Capdevila, D. San Martin, F.G. Caballero, V. Lopez. Metallographic techniques for the determination of the austenite grain size in medium-carbon microalloyed steels. Mater Charact 2001,46:389-398.

[10] M. Prikry, A. Kroupa, G.C.Weatherlt, and S.V.Subramanian, (precipitation behavior in a medium carbon Ti-V-N microalloyed steel.

[11] J. Reiter, C. Bernhard, H. Presslinger. Austenite grain size in the continuous casting process: Metallographic methods and evaluation. Mater Charact 2008,59: 737- 746.

[12] G. V. Voort. Tech-Notes. Buehler Ltd. Vol.1, Issue 5.

[13] W. Roberts and A. Sandberg, Report IM-1489, Swedish Institute for Metals Research (1980)

[14] F.A. Khalid, D.A. Gilroy, and D.V.Edmonds, p.67 in ref. [15]

[15] Processing, Microstructure and Properties of Microalloyed and other Modern High Strength Low Alloy Steels, A.J.De Ardo, ed., Iron and Steel Soc. Inc. , (1992).