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AN EXPERIMENTAL INVESTIGATION INTO THE EFFECT OF TREATMENT TEMPERATURE ON THE GRAIN SIZE OF LOW CARBON AND MICROALLOYED STEELS

بحث معملى فى تأثير درجة حرارة المعالجة الحرارية على حجم الحبيبات للصلب منخفض الكربون و الصلب منخفض السبائك.

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الخلاصـــة :

يودى التركيب البللورى للمادة دورا مهما فى خصائصها الميكانيكية . فتأثير حجم الحبيبات على الخصائص الميكانيكية للمادة يكون معقدا حيث ان حدود الحبيبات وشكلها لهما تأثير على مدى مقاومة او ليونة المادة. لذا فقد أجريت سلسلة من اختبارات المعالجة الحرارية والتبريد على كل من الصلب منخفض الكربون والصلب ومنخفض السبائك باستخدام وصلة حرارية مثبتة فى العينات المختبرة لقياس درجة الحرارة . وقد تم بحث سلوك نمو الحبيبات عند درجة حرارة منخفضة وقد تم التنبؤ بمتوسط حجم الحبيبات عند اى درجة حرارة للصلب منخفض الكربون والصلب منخفض السبائك. وذلك باستخدام المعادلة الاتية :-

$$d\gamma = k_1 . t^{k_2} \exp(-Q_0/RT)$$

وقد اظهرت النتائج المعملية ان هناك زيادة ملحوظة فى حجم الحبيبات مع زيادة درجة الحرارة عند مدى (١١٠٠ °م) الى (١٣٠٠ °م) . وقد تمت مقارنة النتائج المعملية والنظرية لكل من حجم الحبيبات عند درجات الحرارة المختلفة ووجد أن المعادلة المستخدمة للتنبؤ بحجم الحبيبات تعطى توافقا مع النتائج المعملية عند درجه حرارة من (١١٠٠ °م) الى (١٣٠٠ °م) لكل من الصلب منخفض السبائك والصلب منخفض جدا للكربون ، ومن (٩٠٠ °م) الى (١٠٥٠ °م) للصلب منخفض الكربون .

ABSTRACT

The microstructure of material plays a significant role in its mechanical properties. The influence of grain size on mechanical properties is complex since grain boundaries may either act as obstacle to dislocation motion (strengthening effect) or provide a positive contribution to deformation of the material (softening effect). A series of heat treatment and quenching tests of extra-low carbon, low carbon steels and Nb-V microalloyed steels are conducted. The grain growth behaviour at different high temperatures is investigated. An empirical relation of grain growth behavior has been used to predict the austenitic grain size of the used materials which are subjected to different austenitization treatment temperatures. Experimental results indicate that there is an increase in grain size with temperatures ranging from 900 to 1300 °C. Grain size also increases with the reheating time when treated at the same temperature. Comparison of the predicted and experimental grain size at different temperatures is made. It is found that the empirical relation of grain growth behavior gives good agreement at temperature ranges from 1100 to 1300 °C for Nb-v microalloyed steels and extra-low carbon steels, and 900 to 1050 °C for low carbon steels.

KEYWORDS

Heat treatment, phase transformation, grain coarsening, accelerated cooling, mechanical properties, microstructure evolution, grain growth modeling.

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1. INTRODUCTION

During the past two decades, microstructure engineering in hot-strip mills has gained significant attention with goal being to develop a predictive tool that quantitatively links the processing parameters in the mill to the properties of the hot-rolled steel product. During hot rolling of low carbon steel and microallyoed steels, austenite grain growth is the dominant process in the reheating furnace. Hence, the final ferrite grain size and associated mechanical properties depend on the conditions of the reheating treatment process of the steel. Microstructure changes during solution heat treatment of steel result from the complex interaction between thermal and metallurgical phenomena. Grain size is one of the most important parameters determined in quantitative metallography, owing to the importance of this microstructural feature in influencing mechanical properties of steels [1,2]. The influence of grain size on mechanical properties is complex since grain boundaries may either act as the obstacle to dislocation motion (strengthening effect) or a positive contribution to the deformation of the material (softening effect). The importance of these two opposite effects depends on temperature as pointed out by Kutumba et al [3]. He found that the flow stress depends on the microstructure and on the deformation rate for a given alloy. In most cases the flow stress at various temperature depends on the initial grain size according to the following relationship :

$$\sigma = \sigma_o + kd_o^{-1/2}$$

Sakai et al. [4] have been carried out a series of austenitized of 0.06% C-1.43% Mn steel at of temperatures ranging from 900 to 1260°C selected to produce initial grain sizes of 60 to 375 μm. Their experimental results during axisymmetric compression tests indicated that cyclic stress-strain behaviour was observed at temperature ranges from 900 to 1000 °C, while single peak behaviour was obtained at high temperatures. Efforts by Anelli et al. [5,6] were initially concentrated on the development of microstructure and hot strength models for plate rolling of C-Mn and microalloyed steels. Cuddy and Raley [7-8] presented an experimental investigation of grain coarsening behavior in three types of steels heated half hour at temperatures in the range 900 - 1250°C. Their experimental results indicate that Nb content decreases grain-coarsening behavior at austentitic temperatures. Akben et al. [9] Investigated the effect of chemical composition of steel on austenitization temperature, austenite grain size and transformation temperature. Morrison [10] found that the austenite grain size increases linearly with increase of austenitising temperature for plain carbon steel and Nb steel. Almond and Irani [11] investigated the variation in grain size with soaking temperature for different carbon steels heated one hour at temperatures between 1000 and 1250 °C, indicating that the increasing of carbon contents leads to increasing the grain coarsening. Inagaki [12] discussed experimentally the change of the microstructure of 0.1 C-1.35 Mn and 0.03 Nb steels due to annealing twins in the specimens heated for 1 hr at temperatures of 950, 1050, 1150 and 1250 °C and quenching in water. He reveals that the increasing of reheating time leads to increasing of grain size. Ichii et al. [13] found that the grain size of 0.07C-0.031N and 0.057 Al steel is large at solution treatment of 1300 °C compared with obtained at 1085 °C. Militzer et al [14] indicated that the pinning force and austenite grain growth are a function of the pre-heat treatment schedule. Sun et al [15] investigated the effect of mean initial grain size on the recrystallized grain size obtained after deformation at different temperatures. showing that the recrystalized grain size decreases with decreasing the initial grain size. Their results reveal that the volume ratio between the recrystallized and the initial austenite grains, decreases with increasing initial grain size. Siwecki [16] found that the grain size existing after reheating has no effect on the final austenite grain size resulting from the recrystllization rolling, as long as the rolling schedule involves more than 4-5 passes. Elkassas et al [17] have discussed briefly the role of the microstructure features on the various properties of different steel grades. The reheat temperature affects the state of the precipitates which in turn influences the recrystallization kinetics and subsequent grain growth [18].

The present article investigates the effect of austenitising temperature on the austenite grain growth of extra-low carbon, low carbon steels and Nb-V microalloyed steels in the as-reheated condition and develops an improved description of this process. The austenite grain coarsening has to be considered as an important process for grain size development. The austenite grain size represents the initial grain size for the subsequent hot rolling process. An attempt is made to control carefully the experimental conditions for microstructure evolution during hot rolling. Empirical relation during austenitic treatment is coupled with temperature and reheating time to simulate austenite grain growth for the used materials. The verification of the grain growth empirical relation has been performed by comparing calculated values with the experimental data. The empirical relation provides an important insight to predict the initial structure of hot rolling process.

3. EXPERIMENTAL EQUIPMENT, MATERIALS AND PROCEDURE

3.1. Materials Description

Extra-low carbon, low carbon steels (**A** & **B**), respectively and Nb-v microalloyed steel are received in a cylinderical shape with 10 mm diameter and 15 mm length. The chemical composition of the three materials (wt-%) is given in Table 1.

3.2. Equipment

A Lindberg furnace is used to heat the specimens to the solution treatment temperatures. The DASH-8 data acquisition board is connected to a universal analog input expansion submultiplexer/ amplifier system by which the signal from thermocouples are amplified and recorded.

3.3. Experimental Procedure

The experimental is setup as shown in Fig. (1). The Lindberg furnace is heated to specified temperature (900 to 1300 °C). The specimens are put in Quartz test tube and connected to vacuum system. The test tube is evacuated until the vacuum gage reads a minimum vacuum of 0 Torr. Then the test tube is put in the furnace through a small hole drilled in the door of the furnace. the specimens are heated for specified time (15, 20 and 30 minutes) and quenched in ice water. It must be noted that time for taking the specimen out of furnace and quenching in ice water is only 3-4 seconds.

Table 1 Chemical Compostion (wt-%) of the used materials

Steel	C	Mn	S	P	Si	Al	M	V	Mo	Mb	Cr	Cu	Fe
A	0.07	1.58	0.009	0.008	0.25	0.03	-	-	-	-	-	-	rest
B	0.19	0.75	0.007	0.009	0.05	-	-	-	-	-	-	-	rest
Nb-V	0.043	1.43	0.006	0.011	0.312	-	0.007	0.005	0.252	0.075	0.014	0.007	rest

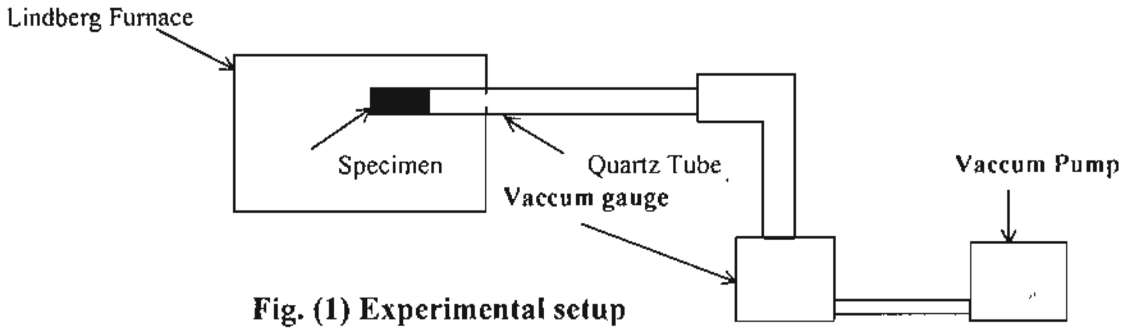


Fig. (1) Experimental setup

3.4. Metallographic Examination

The grain structure of the specimens are examined by the conventional optical technique. The procedure consists of mounting the solution treated specimens in resin, polishing, etching and photographing. The most successful etching solutions are picric acid fully saturated in distilled water with addition of two weight percent teepol for Nb-V microalloyed steel and 3% nital for carbon steel [19]. The time of etching was 4-5 seconds and 15 to 20 seconds for Nb-V microalloyed steel and carbon steel, respectively. The grain boundaries are much easily revealed if the samples are tempered at 500 °C. This thermal treatment leads to remove texture variations and anisotropy [19]. The samples are tempered at 500 °C for one hour. The metallographic software Java is used in measuring grain size.

4. RESULTS AND DISCUSSION

4.1. Grain Coarsening Behavior at Different Solution Treatment Temperatures

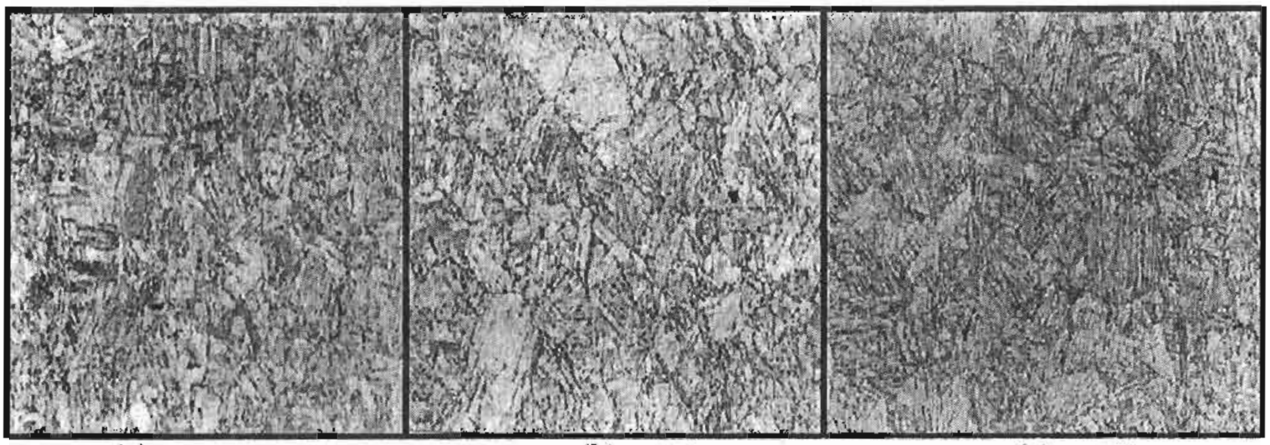
Grain size is measured using linear intercept method [20]. It can be shown that true face -to-face diameter of Kelvin ployhedra, is related to the surface to volume ratio S/V of such solid , as S/V =6.70/d, or, for ployhedra in contact to fill space, S/V =3.35/d. Also for convex shape, S/V and l could be related as S/V =2/l . Therefore true volume grain diameter is given by

$$d = 1.68\,L = 1.68\,(l / m\,n) \qquad (1)$$

The grain diameter is calculated using the above formula. The results are tabulated in Table 2. The prior-austenite grain structure are shown in Figs. (2. a, ,b. c) and (3. a, b, c) at different temperatures and reheating time for extra low carbon steels. The micrographs of the austenite grain size for low carbon steels, indicating the undeformed grains. are presented in Fig.(4. a, b, c, d & e) as the average austenite grain size after reheating for 15 minutes at temperatures from 900° to 1100 °C. It is observed that the increase of C and Mn percentage increase the grain size diameter at the same temperature and heating time. Hence, it can be said that the small changes in carbon content have effected the grain coarsening by comparing the results of the used materials. Table 2. shows that the grain sizes of Nb-V microalloyed steels are smaller as compared to extra-low carbon and low carbon steels. This is because the alloying elements hinder the grain growth. Impurities in material also effect the grain growth. It can be concluded that Nb and V contents have a significant effect on the grain-coarsening temperature.

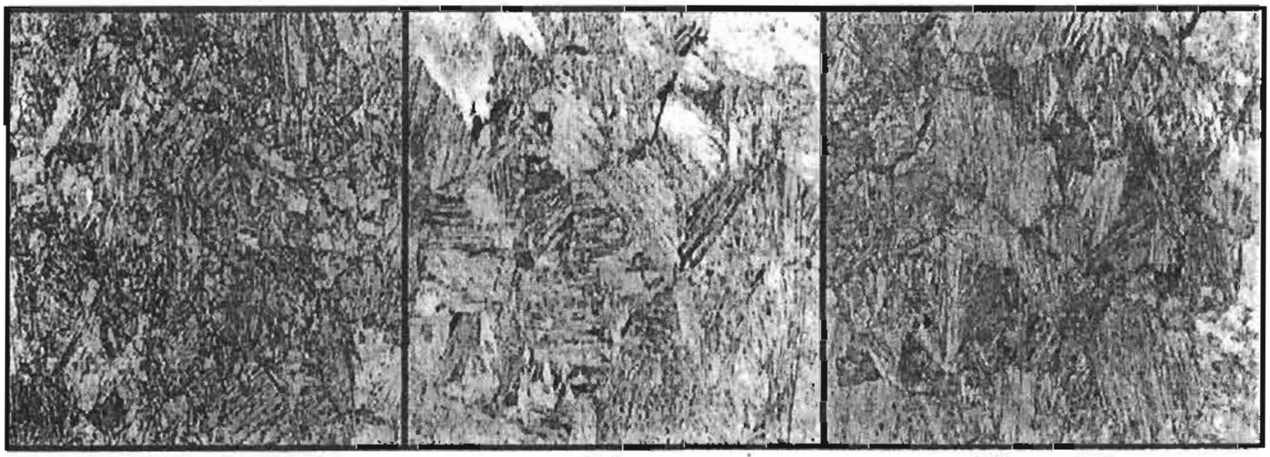
Table 2. Grain size for Extra low, low carbon steels and Nb-V Microalloyed steels

Temperature °C	reheating Time (in min.)	Av. Grain size (in µm) Extra Low carbon steels	Av. Grain size (in µm) (Nb-V) steels
Untreated	-	34	9.1
1100	20	109.03	31.2
	30	128.4	37.14
1200	20	209.45	126.9
	30	240.4	147.6
1300	20	403	325.5
	30	462.3	371.6
		Av. Grain size (in µm) Low carbon steels	
1080	15	132.18	
1048	15	127.96	
994	15	106.60	
940	15	88.73	
890	15	85.71	



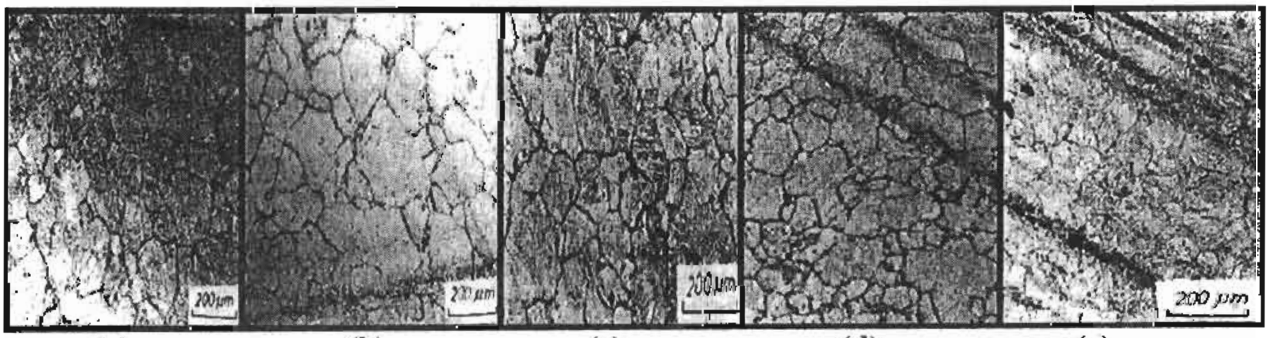
(a) 200 µm (b) 200 µm (c) 200 µm

Fig. (2) Micrograph of extra-low carbon steel (A) at 1100, 1200 and 1300 ^{0}C for 20 min.



(a) 200 µm (b) 200 µm (c) 200 µm

Fig. (3) Micrograph of extra-low carbon steel (A) at 1100, 1200 and 1300 ^{0}C for 30 min.



(a) (b) (c) (d) (e)

Fig. (4) Micrograph of low carbon steel (B) at 1080, 1048, 994, 940 and 890 ^{0}C in the centre of samples for 15 min.

4.2. Influnce of austenitising Temperature and Reheating Time on Microstructure Evolution of Austenite

The effect of the reheating temperature on the grain size diameter of the low carbon, extra-low carbon steels and Nb-V microalloyed steels are shown in Figs (5) and (6), respectively. It is clear that as the reheating temperature increases, the average grain size increases due to thermal activated which cause higher grain growth rate of the reheated material. Moreover when compared with published results given in Table 3, it is observed that the nature of the trend of the experimental results are similar. The results are not comparable due to difference in the experimental conditions and the composition of materials. Some comparison between the published results and our experimental results can be shown in Tables 2 and 3, respectively. It can be seen that reasonable comparisons are found. The control of grain coarsening behaviour of steels is an important step in design of thermomechanical process striving to achieve fine-grained products.

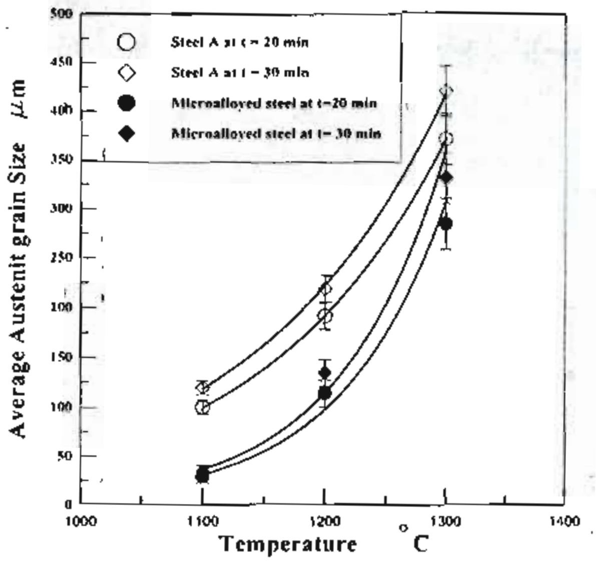


Fig. (6) Average austenite grain size as function of solution treatment temperature for extra-low carbon steels and Nb-V Microalloyes steels at the centre of the samples.

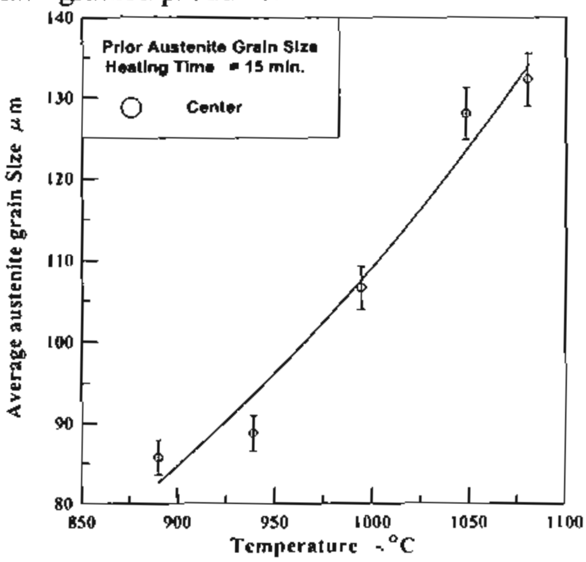


Fig. (5) Average austenite grain size as function of solution treatment temperature for low carbon steels at the centre of the samples.

Table 3. Some of the published results

References	Steel type	Grain size in (μm), t =30 min.		
		1100 °C	1200 °C	1300 °C
L. Cuddy et al [7-8]	0.065C-0.007N	125	220	-
	0.058C-0.014N	40-75	150	-
	steel 0.048 Nb	31	140	-
	steel 0.11Nb	31	105	-
Akben et al.	0.06C-1.43Mn-0.24Si-0.025Al	110 at 1030 °C	200 at 1140 °C	-
[9]	0.04C-0.034Nb-0.115V-0.31Mo	115	200	-
W. Morrison [10]	0.1C-1.42Mn	89.8	254	-
	0.11C-1.5Mn-0.045Nb	53.4	359	-
E. Almond et al [11]	0.057C-1.9Mn-0.14V-0.11Si	25	112.5	-
	0.053C2.03Mn-0.14V-0.12Si	125	-	-
H.Inagaki [12]	0.1C-1.35Mn-0.03Nb	62 at 1150 °C	320 at 1250 °C	-
K. Ichii et al [3]	0.07C-0.031N-0.057Al	5.95 at 1085°C	-	365

4.3 Empirical Relation of Grain Coarsening Behavior

In order to ascertain the austenitising grain size as function of temperature, isothermal reheating studies are performed on extra-low carbon, low carbon steels and Nb-V microalloyed steels specimens. Different temperatures spanning the austenite phase field are employed for the heat treatment studies. The austenite grain coarsening behaviour of the three steels during reheating and hot working are an important factor in achieving fine-grained products.

Obviously, reheating process results in grain coarsening. As-reheated austenite grain size which is thermally grown, should be estimated in order to achieve the required fine-grained products. This step is important to predict the microstructure evolution in the subsequent hot rolling processes. Therefore, the average size of austenite grains in (µm) is given for isothermal heat treatments by the following relationship [21]:

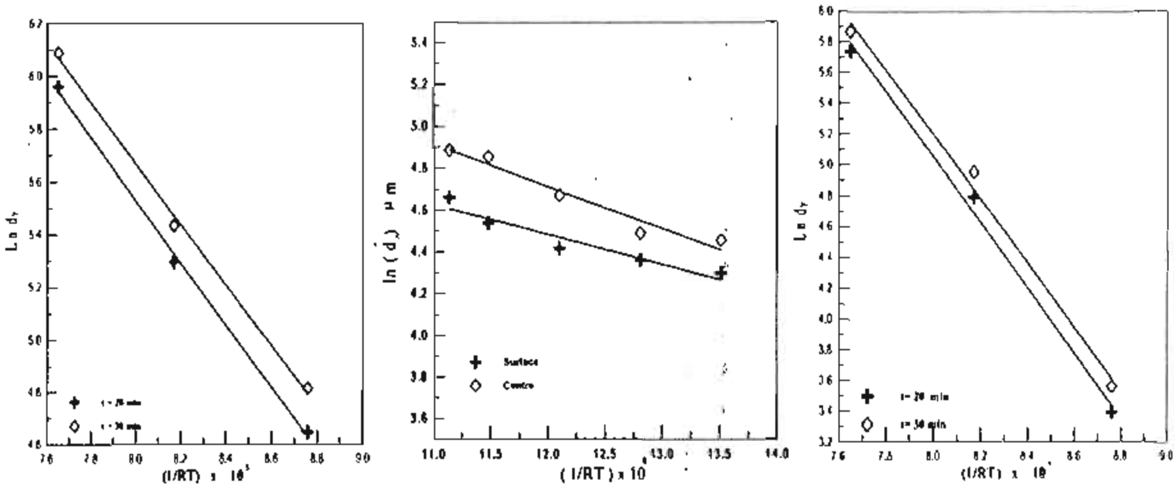
$$\bar{d}_{\gamma} = k_1 \cdot t^{k_2} \exp(-Q_o / RT) \qquad (2)$$

where t_r is the time in hour (t< 1), T the absolute temperature and R the gas constant. The constants k_1 , k_2 and Q_o have been identified for each steel on the basis of measurements of $\bar{d}_{\gamma}$ performed on samples subjected to different austenitization treatments.

On the basis of the present laboratory test results, the thermal activation energy can be calculated as follows:

$$Q_o = -R \cdot \left[\frac{\partial \ln(\bar{d}_{\gamma})}{\partial (1/T)} \right] \qquad (3)$$

The value of the thermal activation energy is obtained from the universal gas constant and the measured values of $\partial(\ln(\bar{d}_{\gamma}))/\partial(1/T)$ (see Fig. (7)). and the measured value of the relation slope, leading to Q_o for extra-low, low carbon steels and Nb-V microalloyed steels as shown in Table 4. Constants k_1 and k_2 were calculated using the experimental data and regression analysis, leading to the empirical values as shown in Table 4.



Extra-low carbon steel | Low carbon steel | Micro-alloyed steel

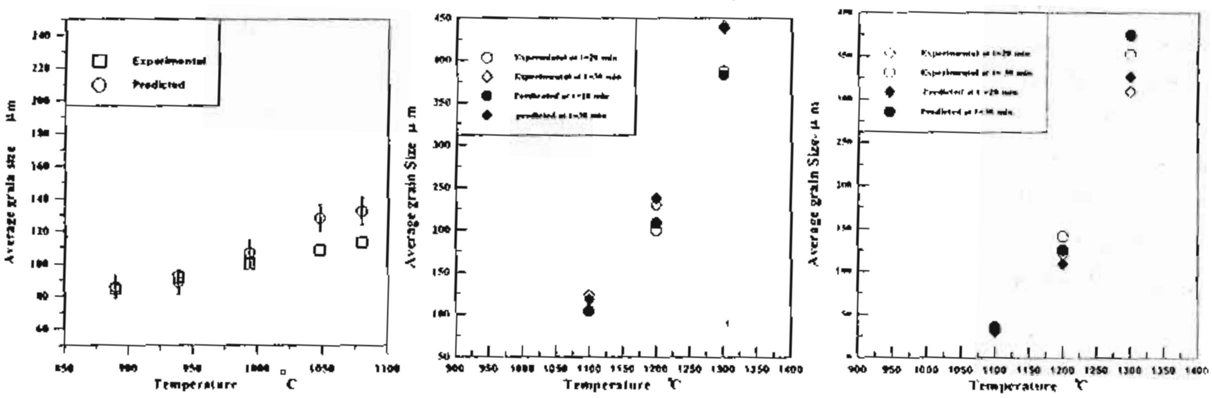
Fig.(7) Determination of the activation energy for Extra low, low carbon steels and Nb-V Microalloyed steels : The ln d_{γ} against (1/RT).

Table 4. Values of empirical coefficients

	Empirical coefficients	Extra low carbon steels	Low carbon steels	Micro-alloyed steels
γ grain growth	k_1	0.33	0.04	0.34
	k_2	4.5x10^6	717	4994.126x10^6
	Q_o (J/mol)	117770	20183.5	2115000

4.4. Predictabiliy of empirical relation for Grain Growth behavior during Reheating Process.

Figs.(8) (a), (b) and (c) indicate that the predictability of austenite grain size is reasonable at different treatment temperatures and reheating time for extra-low carbon, low carbon steels and Nb-V micro-alloyed steels. It is noted that the increasing of reheating time and temperature for extra-low carbon steels and Nb-V microalloyed steels leads to increase the deviation between the predicted and measured values of average grain size. The used constitutive relations give a good agreement at temperatures up to 1200 °C and reheating time up to 20 min for extra-low carbon and Nb-V microalloyed steels. These relations gives good agreement for low carbon steel at temperatures range to 950 °C. Fig.(9), also shows clearly the temperatures and reheating time rang which give agreement for all the materials. These data can be used successfully as input data for the microstructure evolution model during hot rolling process. The control of grain coarsening behaviour of steels due to the reheating process is an important step in design of thermomechanical process aiming at achieving fine-grained products. Also, reheating temperature affects a formation of so-called deformation bands which play an important role during subsequent grain restoration process [22].



(a) Low Carbon steels (b) Extra-low carbon steels (c) Micro-alloyed steels

Fig.(8) Comparison of the predicted and experimental austenite grain size at different temperatures for Extra-low carbon, low carbon steels and Nb-V microalloyed steels.

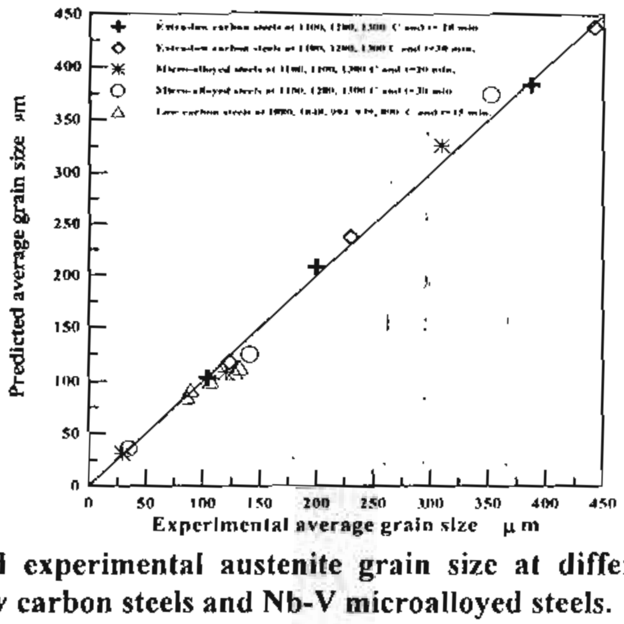


Fig.(9) Predicted and experimental austenite grain size at different temperatures for Extra-low carbon, low carbon steels and Nb-V microalloyed steels.

5. CONCLUSIONS

The austenite grain size at different solution temperatures is determined metallographically from quenched test samples for extra-low, low carbon steels and Nb-V microalloyed steel. The austenite grain size at near surface and center is observed at temperatures range from 900 ° to 1300 °C for all materials. The results show that at higher temperatures, the thermally activated grain growth is higher. The grains of Nb-V microalloyed steels are smaller compared to extra and low carbon steels, where the alloying elements hinder the grain growth. Also, grain growth is effected by impurities in the material.

Empirical relation of Grain growth behavior which is function of temperature and isothermal reheating time, have been used. A comparison between the measured and predicted values using the existing relation of austenite grain diameter is made for extra-low carbon, low carbon steels and Nb-V microalloyed steels at different temperatures and reheating times. Analysis of the results show that empirical relation of grain growth behavior examined here provide good estimates of grain diameters at temperature ranges from 1100 to 1300 °C for Nb-V microalloyed steels and extra-low carbon steels, and from 900 to 1050 °C for low carbon steel.This takes place due to increasing of carbon content. In general, conclusion regarding the ability of empirical relation for predicating the evolution of the metallurgical structure during reheating process can be applied at with caution because the relation has been driven for particular steel grades.

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NOMENCLATURE

d_0 Average grain size, μm

d_γ Average grain diameter austenit phase, μm

k, k_1, k_2 Material constants

Q_0 Activation energy for grain growth, kJ/mol

L The mean linear intercept, μm

l Total line length employed, μm

m Magnification of photograph

n Numbers of boundaries intersected by test line.

R Gas constant, kJ/mol ^{o}C

t Time, hour

T Absolute temperature, ^{o}k

σ Flow stress, MPa

σ_0 Yield flow stress, MPa