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Effect of Nb addition on the grain growth of annealed high Mn austenitic steel structure

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الملخص

يهدف البحث الى دراسة تأثير إضافة النيوبيوم على خاصية نمو الحبيبات في البنية المجهرية لسبائك الصلب الاستتنيتي العالي المنجنيز المعالج حراريا. اختيرت السبية Fe30Mn محتوية على نسب مختلفة من عنصر النيوبيوم، حيث تم تقسيم العينات الخاضعة للدراسة الى مجموعتين. سبائك منخفض النيوبيوم من 0.05 الى 0.4% واخرى عالية النيوبيوم بنسب من 0.6 الى 1%. أجريت المعالجة الحرارية للعينات في درجة حرارة كافية لاذابة النيوببيوم واعلى من درجة اعادة التبلر 1200^oC ولفترات زمنية من 2 الى 60 دقيقة. خلص البحث الى ان النيوبيوم يعمل على عرقلة نمو الحدود الحبيبية ويتضح ذلك من خلال الحجم الحبيبي الصغير كلما زادت نسبة النيوبيوم في السبيكة. هذا الاستنتاج يوضح تاثير النيوبيوم نتيجة لتأثير عنصر المنجنيز على إذابة النيوم في طور الاستنيت. إتضح من نتائج البحث التطابق الواضح بين النتائج المعملية والنتائج النظرية لقياس حجم الحبيبات عند إعتماد ثابت مقارنة n=2 وان الفارق في حجم الحبيبات ليس نتيجة لتأثير النيوبيوم فحسب بل ايضا نتيجة لتأثير عنصر المنجنيز في إذابة النيوبيوم في الاستنيت.

الكلمات المفتاحية: صلب TWIP، نمو الحبيبات، مودل رياضي، ذوبانية عنصر النيوبيوم.

ABSTRACT

An investigation has been carried out to study the effect of Niobium (Nb) addition on the grain growth characteristics of Fe30Mn –TWIP steel alloy. The studied steel samples have been divided in to two groups which are low-Nb (0.05-0.4 Nb wt%) group and high Nb one (0.6 and 1Nb wt%). All samples were heated at dissolution temperature just above the recrystallization stop temperature of 1200^oC at different period of times. It has been found that simultaneous addition of Nb inhibits the austenitic grain growth markedly. However, Significant grain refinement was observed in high Nb group. This indicates a stronger grain boundary pinning effect due to the effect of Mn on the Nb solubility. The comparison between the experimental values of grain size at 1200°C and empirical modelling using ***n* = *2*** shows a reasonable agreement. variation of grain growth behavior could be due to the effect of Manganese on Nb solubility in austenite.

Keywords: TWIP steel, grain growth, empirical modelling, Nb solubility.

1. INTRODUCTION

High Manganese steels have full austenite stability at room temperature and low stacking fault energy. Thus variations in carbon and nitrogen levels in solution do not impact significantly on the stable phase [1]. The austenite grain size of high Mn steel is an important factor which controls the transformation characteristics and, thus, influences the resulting microstructure and mechanical properties [2]. Due to its retarding effect, Nb has been used to control the austenite grain growth during steel heat treatment, [1]. Manohar, P. [3], studied the effect Nb on the grain growth of austenite in high Mn steels during heat treatment and reported that increasing of Nb content retards the γ phase transformation and raises the Ar3 temperature during heat treatment.

Nb affects the grain boundary migration by the segregation of Nb solutes which can introduce a frictional drag on the moving boundary. Presence of Nb second phase particles can also decreases the grain boundary area and, hence, the overall grain boundary energy, thus producing a grain boundary pinning effect [4]. Austenite grain boundary pinning is particularly effective and important for the toughness of steels in which the final properties are obtained by quenching and tempering.

In this work, different Nb content Fe30Mn alloy, has been austenitized at 1200°C for different times in order to study the grain growth (G.G) behavior of Fe30Mn alloy microstructure. This work also uses a mathematical model from previous study [4], to explain and understand the effect of Nb on the (G.G) of annealed austenitic steel.

2. Experimental Part

The materials used in this work were (2.7x30x120) mm **of 73% hot rolled**

plate samples of Fe30Mn and other six Nb-Fe30Mn levels with chemical composition given in Table 1.

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

Alloy ID	Mn W%	Nb W%	C Wt%	N Wt%
Fe30Mn	29.2	0.0001	0.002	0.0026
0.05Nb	29.7	0.044	0.002	0.005
0.1Nb	29.9	0.078	0.004	0.007
0.2Nb	30.02	0.18	0.008	0.01
0.4Nb	30.1	0.46	0.008	0.011
0.6Nb	30.2	0.62	0.013	0.011
1Nb	30.2	1.0	0.021	0.012

All isothermal grain growth tests were performed on materials presented in Table 1 (as two groups; 0.05Nb, 0.1Nb, 0.2Nb and 0.4Nb wt% as group1, 0.6Nb and 1Nb wt% as group 2) using a muffle furnace at 1200^{o}C for 2, 10, 30 and 60 minutes. A 1200^{o}C annealing temperature has been selected as a temperature at which recrystallization was completed [5,6]. The samples were prepared using standard metallographic techniques and etched in saturated aqueous 7% solution of sodium Meta bi-sulphate for further optical observation and scanning electron microscopy. To obtain the average grain size, linear intercept method has been implemented on the SEM images and average grain size was calculated.

3. RESULTS AND DISCUSSION

3.1 Microstructure investigation

3.1.1 Fe30Mn alloy samples

Microstructure of Fe30Mn annealed samples exhibits fully recrystallized equaixed austenite grains with variety of annealing twins at 1200^{o}C for 2 minutes. As the dissolution time increases up to 30 min, the grain size increases. This can be explained by the reduction in the surface energy. Extensive grain growth was observed in Figure 1C and that could be attributed to few grains growth dramatically in expense of smaller recrystallized grains in order to reduce the grain boundary energy.

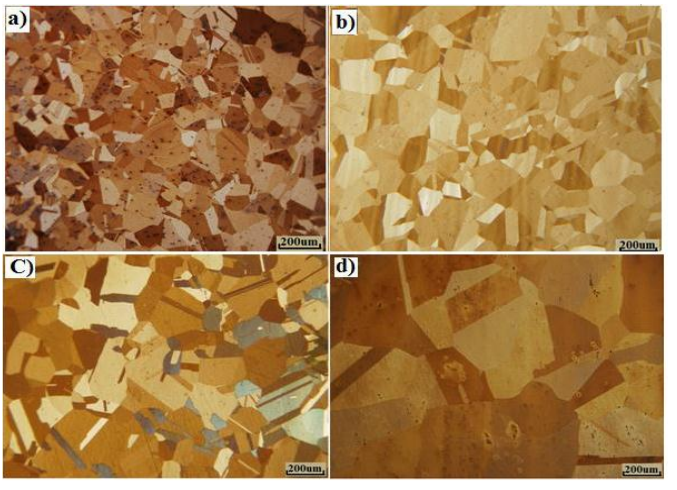

Figure 1: An optical micrograph of annealed Fe30Mn at 1200^{o}C for different times; a) 2 minutes, b) 5 minutes, c) 10 minutes and d) 30 minutes.

3.1.2 Nb-Fe30Mn alloy samples

Annealing at 1200^{o}C was performed for all Nb-Fe30Mn alloys and recrystallization occurred at short times. For the low-Nb group alloys (0.05-0.4Nb wt%), high recrystallization fraction was seen in the annealed structure at 1200^{o}C for two minutes and was fully recrystallized when all samples were held for 5 minutes. Grain growth was seen with increasing time (Figure 2, Figure 3 and Figure 4. From figures, This recrystallization and grain growth behavior is due to the difference in the presence of Nb precipitates.

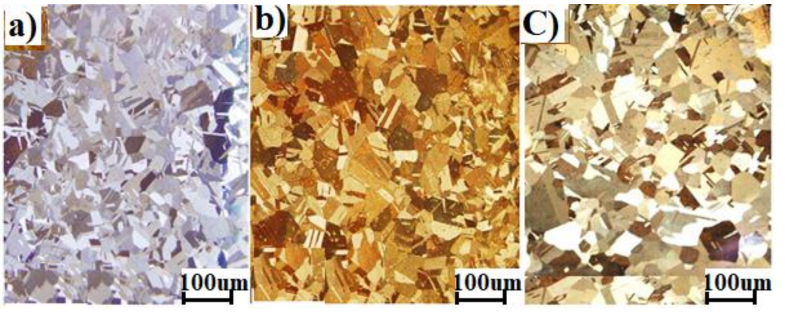

Figure 2: Optical micrographs show grain growth of grade 0.05Nb annealed at 1200°C for a) 5, b) 10, and C) 30 minutes.

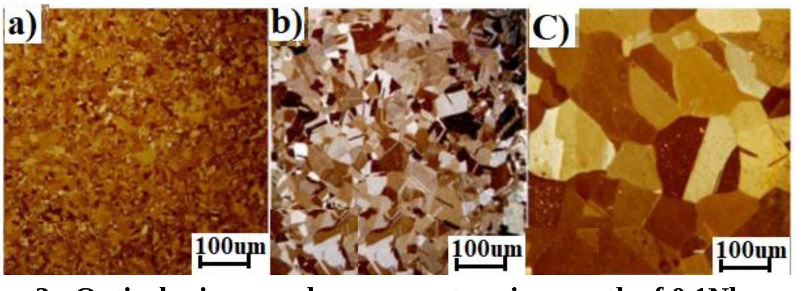

Figure 3: Optical micrographs represent grain growth of 0.1Nb samples annealed at 1200°C for a)5, b)10, and c) 30 minutes.

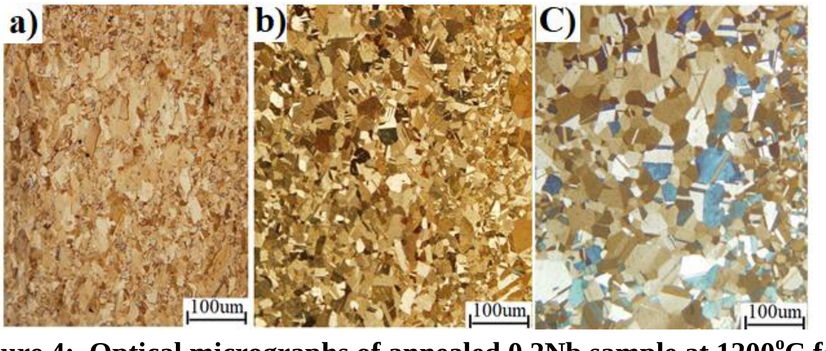

Figure 4: Optical micrographs of annealed 0.2Nb sample at 1200°C for a) 5, b) 10 and C) 30 minutes.

High Nb samples show partially recrystallized microstructures at soaking times of 2 minutes and 5 minutes Figure 5. This can be explained by the incomplete dissolution of Nb particles and that Nb carbides volume fraction were more effective in retarding recrystallization.

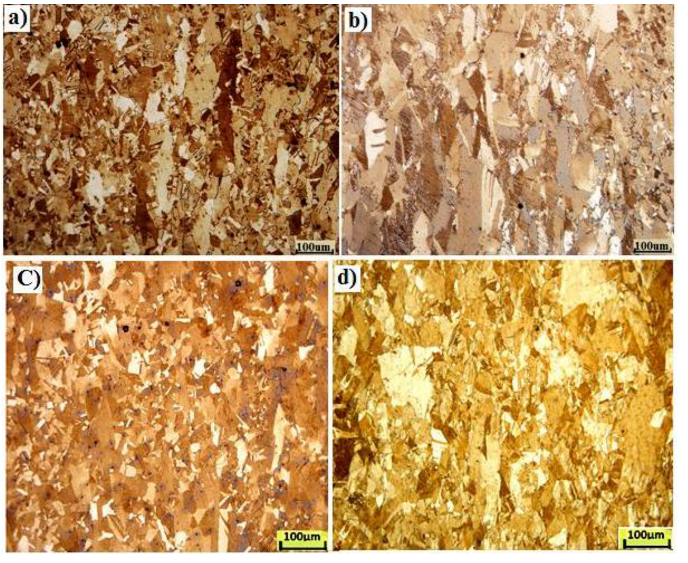

Figure 5: Optical microstructure of annealed at 1200°C a) 0.4Nb for 2 min, b)1Nb for 2 min, C) 0.4Nb for 5 min, and d) 1Nb for 5 min.

However, there was a difference in grain growth in the annealed high Nb studied samples. As Nb increased, the grain growth decreased and for the 1%Nb shows less growth even at longer time as seen in Figure 6.

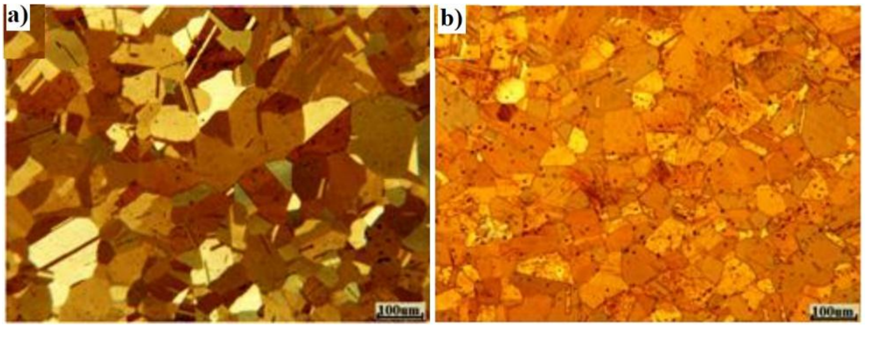

Figure 6: An optical micrograph of annealed a) 0.4Nb for 10 min, b) 1% Nb for 30 minutes.

3.2 Grain Size Measurements

Line interceot method was used to calculate the grain size of the annealed grains and the results have been cheked by ImagJ software. Five optical micrographs have been taken for each annealing time and the average grain size results were tabulated as shown in Table 2.

Table 2: Grain size measurement of the studied alloys at 1200^{o}C

Annealing time (min)	Grain Size (μm) at 1200^{o}C						
	Fe30Mn	0.05Nb	0.1Nb	0.2Nb	0.4Nb	0.6Nb	1Nb
0	152.4	136.8	128.7	120.2	117.6	63.3	33.8
5	218.1	155.9	141.3	134.2	130.3	77	40
10	265.8	184.6	180.2	174.6	166.8	88.6	51.6
30	425.3	246.5	239.6	224.2	216.5	106.8	83.8
60	538.9	359.1	328.2	294.3	271.6	133.2	127

Data presented in Table 2 indicates distinct stages in grain growth of Nb-Mn steel of different Nb contents. It also shows the effect of Nb on austenite grain growth which also affected by the presence of high Mn content.

4. DISCUSSION

In a comparison of the grain growth behavior of the two alloy groups, it can be observed that the grain sizes of the lower Nb alloys are always the largest, while those in alloy containing 6Nb and 1Nb are the finest. Figure 7, plots the grain size as a function of time. The base grade shows a grain size increase of about 3.5 times over the 60 minutes of annealing and the Nb grade displays a coarsening ratio of similar size (~4 times). Overall, the grain size is seen to increase by 2.5 - 4 times during annealing for 60 minutes at 1200^{o}C with no grain boundary pinning force due to influence of Nb on this parameter. However, the different starting grain sizes means that a further analysis is required; the starting grain size controls the driving pressure for grain growth.

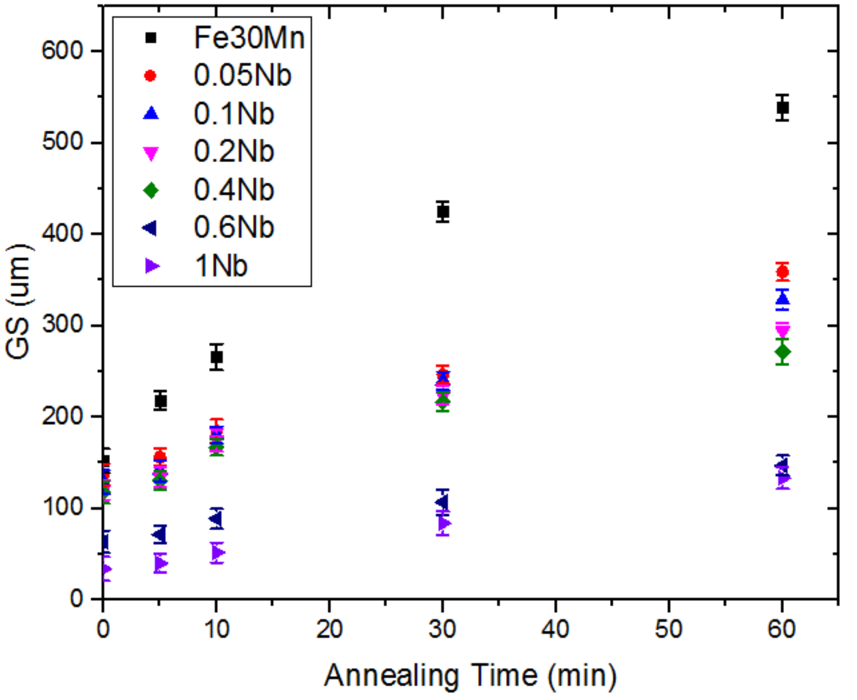

Figure 7: Grain size as a function of annealing time at 1200^{o}C.

Grain growth analysis

The aim of this section is to model the grain growth behavior of the studied alloys in order to understand the Nb effect on the annealing behavior at 1200^{o}C. Grain growth is typically described using the equations below:

$$D^n - D_0^n = K_1\, t$$

$$K_1 = k\, exp\left(-\frac{Q}{RT}\right) = \frac{D^n - D_0^n}{T}$$

where $\boldsymbol{D0}$ is the initial grain size (size of the grains at 1200^{o}C/2 min in the present case) and ***D*** is the final grain size, $\boldsymbol{K1}$ is a rate constant (µm2/sec) that depends on the temperature, activation energy for grain growth ***Q*** and a constant ***k.*** The comparison between the experimental values of grain size at 1200oC and empirical modelling using ***n = 2*** shows reasonable agreement as shown in Figure 8. And the reason behind choosing the value of time exponent (***n = 2)***is that the assumption of the effect of only solute drag on grain growth and very low of pinning effect due to the coarse particles and their low fraction as found in earlier work [4,7].

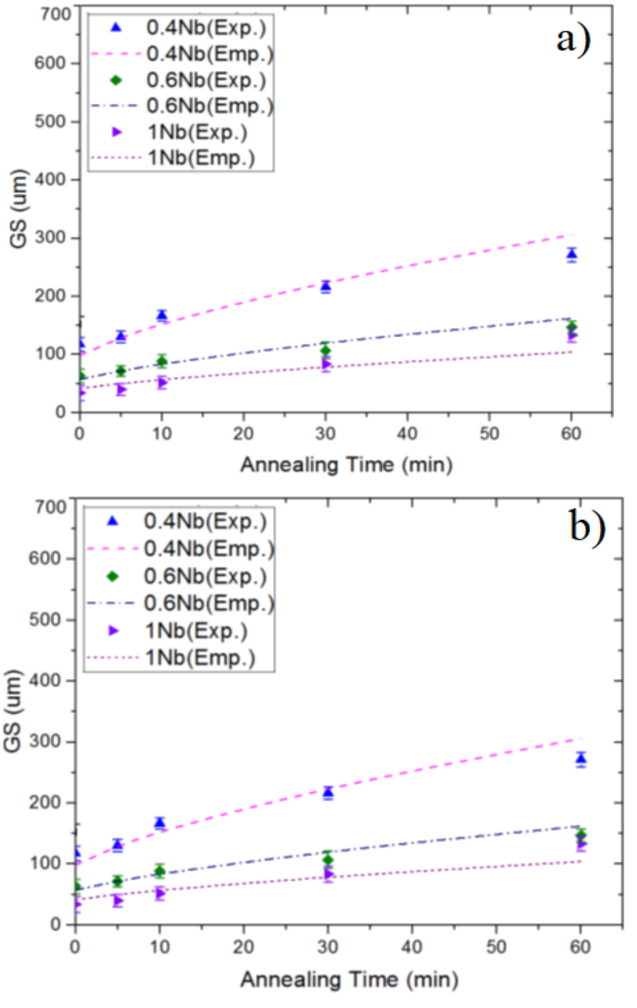

Figure 8: Comparison between experimental (Exp.) grain growth and empirical (Emp.) equation of grain growth of the studied alloys at 1200°C for the studied alloys (all divided in a) and b) graphs)

The experimental grain growth rate constant , K1, is plotted in Figure 9. It clearly shows the effect of Nb on grain growth. Over the range studied, Nb drops the rate of grain growth by over an order of magnitude. Interestingly, the addition of 0.05% Nb drops the rate by a factor of ~2. The rate constants for the 0.6 and 1.0Nb grades are particularly low, perhaps reflecting the presence of Nb(C,N)

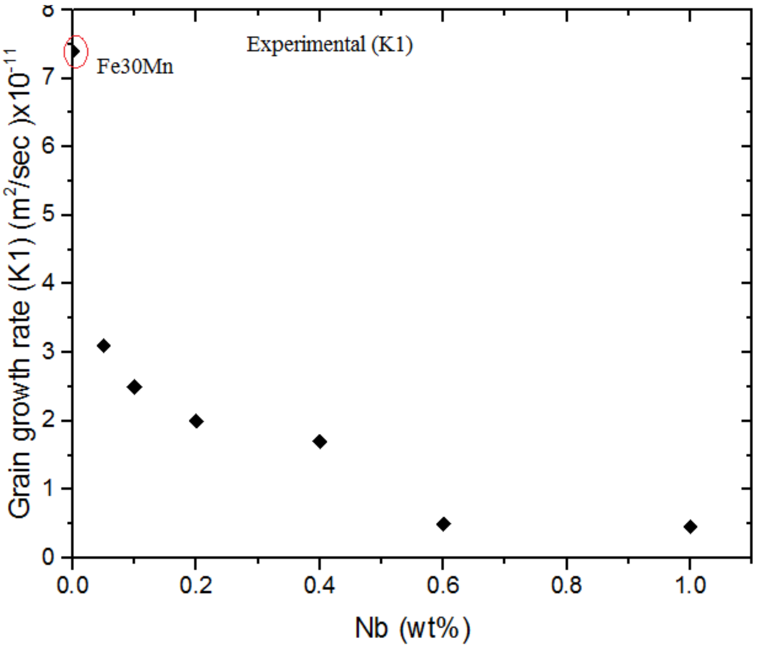

Figure 9: The effect of Nb on grain growth rate (K1) at 1200°C as experimental data results.

5. CONCLUSION

1- Microstructure investigation shows a grain growth of Fe30Mn alloy is a behavior of an austenitic microstructure where grain size increased as annealing time was increased.
2- The effect of Nb in low-Nb Fe30Mn alloys was clearly observed by the grain size measurements.
3- Grain growth of high-Nb Fe30Mn samples was less than that of low-Nb ones.

4- Overall grain growth of the studied alloys can be concluded as the lower Nb content alloy (Fe30Mn0.05Nb) was about 3.5 times faster than that of the growth in high-Nb content one

5.Austenite grain growth model provided satisfactory prediction of austenite grain size at high austenising temperature of 1200C.

6. Excessive grain growth behavior was observed in the Fe30Mn alloy. This indicates that the driving force for grain growth is the reduction in the grain boundary energy.

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