International Journal of ISSI, Vol.5 (2008), No. 2, pp. 21-28

Effects of Homogenization Conditions and Hot Rolling Parameters on Grain Refinement of an As-Cast 301 Stainless Steel

M. Eskandari[1], A. Najafizadeh[2], A. Kermanpur[3*], M. Karimi[4]
Department of Materials Engineering, Isfahan University of Technology, Isfahan 84156-83111, Iran

Abstract

In this work, effects of homogenization time of 3 to 13 h at 1200 °C on the grain refinement of as-cast AISI 301 stainless steel after different hot rolling conditions were investigated. The results showed that the minimum grain size of 16±7 μm was achieved when homogenization took place at 1200 °C for 9 h followed by hot rolling at temperature range of 1000–1200 °C with strain of 0.8 and strain rate of 1.2 s^{-1} and annealing at 1050 °C for 3 min. Effect of Nb element on the retardation of recrystallization was pronounced. It seems that there is an optimum solute segregation below which retardation and above which grain growth of recrystallized grains is dominant. It was also found that this optimum homogenization condition was dependent upon the hot rolling parameters; higher the strain and strain rate resulted in lower homogenizing time at 1200 °C.

Keywords: Grain refinement; 301 stainless steel; Homogenization; Hot rolling.

1- Introduction

Grain refinement of coarse as-cast ingot structure in steels is a primary objective during the initial stages of ingot breakdown [1)]. Incipient grain refinement, where solute and second phases collect during solidification and cooling of ingots at grain boundaries and grains, depend on correct design of homogenization treatment. The solute and second phases can retard the incipient grain refinement by recrystallization at grain boundaries, during hot rolling due to the suppression of grain boundary migration. The suppression of grain boundary migration due to micro-alloying is caused by either: (1) the solute dragging effect because of segregation of alloying elements in the boundaries, or (2) the pinning effect due to precipitates of carbonitride of alloying elements at grain boundaries [2)].

The austenitic recrystallization was studied by Yamamoto *et al.*[3)] in an iron-manganes-niobium system containing extremely low carbon and nitrogen which facilitated investigation of the solute niobium effect. The result showed that the solute niobium evidently retarded the austenitic recrystallization. It was confirmed that this retardation effect by solute niobium was extremely large among various solute elements. Aust and Rutter [4)] showed that the rate of grain boundary migration could be reduced dramatically even by small average concentrations of solute drag. Qingbo *et al.*[5)] showed that in the steel containing micro content of Nb, these atoms are easier to segregate at grain boundary. Akben and Jonas [6)] presented the solute retarding parameter for niobium, molybdenum and vanadium in the steels with 0.05 wt% C bearing one or more of 0.30 %Mo, 0.03 %Nb or 0.11%V. Enomoto *et al.*[7-8)] investigated the grain boundary segregation of Nb atoms in 0.13 wt %C–1% Mn steels bearing different amounts of niobium. They showed that carbides such as niobium carbides can be dissolved at a quite high temperature upon a short-term heating (about 60-150 s). Zaky Farahat *et al.* [9)] showed that the niobium carbide films surrounding the austenite grains were nearly disappeared by solution treatment at temperatures above 1050 °C followed by rapid quenching. The above results show that by selecting the correct homogenization temperature and time, the second phases could be dissolved and consequently segregation of alloying elements suppressed.

Although homogenization is a classic process in industry and many works have so far been carried out on this process for different alloy steels, superalloys and other engineering materials, less attention has been paid on the relationship between the homogenization and subsequent hot rolling conditions and grain refinement. The purpose of this investigation was to study the influence of homogenization time and temperature on subsequent hot rolling parameters in order to refine grain size of an as-cast 301 stainless steel to become suitable for fabrication of the nanocrystalline structure.

2- Materials and Experimental Procedures

Cast ingots of AISI 301stainless steel were prepared using an induction furnace under air atmosphere. The chemical composition of this alloy

---

[*] *Corresponding author:*
Tel: +98-311-3915738 Fax: +98-31- 3912752
E-mail: ahmad_k@cc.iut.ac.ir
Address: Dept. of Materials Engineering,
Isfahan University of Technology,Isfahan84156-83111,Iran
1. M.Sc. Student
2. Professor
3. Associate Professor
4. M.Sc. Student

is shown in Table1. The ingots were homogenized at 1200 °C for 3 to 13 h under air atmosphere. The homogenized ingots were then hot rolled from 18 to 8 mm thickness according to the schedule illustrated in Table 2. The samples were then heated to 1200 °C and held for 20 min and rolled with total strain of 0.8 and strain rate of 1.2 s^{-1}. To study the influence of the strain and strain rate on solute drag effect during recrystallization, the 5 and 7 h homogenized samples were hot rolled at strain rate of 1.2 s^{-1} with strain of 0.4-1.5 and strain rate of 0.4-2 s^{-1} with strain of 0.8, respectively. All samples were quenched in water after hot deformation immediately. In order to fulfill recrystallization process, some specimens were annealed at 1050 °C from3 to 20 min.

Optical and SEM (LEO 1455 VP) examinations as well as EDS analysis were carried out, respectively, on the samples after electro-etching in 65% nitric acid solution. The line scan analysis was performed across the grain boundary. The software Clemex was used for grain size measurements based on the intercept method. Almost 100 grains were selected by the software for grain size measurements. Hardness of the samples was measured by Vickers method. X-ray diffraction analysis with Cu Kα radiation was used for phase identification.

3- Results and discussion

3-1- Influence of homogenizing time on grain refinement

The microstructure of the as-cast sample was consisted of two phases of austenite with grain size of 1.02± 0.49 mm, intergranular delta ferrite (9 vol%) and some carbide precipitates inside the grains as shown in Fig. 1a. The XRD pattern shown in Fig. 1b confirms presence of the two phases in the as-cast sample.

The microstructure of the as-homogenized samples at 1200°C for 3, 5 and 9 h are illustrated in Figs. 2a-2c. As it can be seen, the austenite grain size is increased gradually with increasing the homogenizing time. Also, the as-cast carbide precipitates are nearly disappeared after homogenization treatment. The lack of second phases is not unexpected because the homogenization temperature was higher than the carbide-forming temperature and the quenching conditions prevented carbides formation during the cooling stage [9]. Fig. 2d shows XRD pattern of the homogenized sample for 9 h. This figure shows that ferrite peaks are removed compared to the as-cast condition (Fig. 1b). The grain size variation of austenite for different homogenizing times is shown in Table 3.

Table 1. Chemical composition AISI 301 stainless steel (wt %).

Fe	C	Si	Mn	Ni	Cr	Mo	Cu	Nb	Ti	V	W	Sn	P	Co
74.3	0.11	0.67	0.65	6.91	16.20	0.27	0.53	0.003	0.02	0.06	0.02	0.04	0.02	0.09

Table 2. Thermo-mechanical processing used in this work.

Pass number	strain	Strain rate (s^{-1})	Temperature (°C)
1	0.13	1.2	1200
2	0.13	1.2	1100
3	0.13	1.2	1010
Reheating in furnace for 2 min at 1200°C			
4	0.2	1.2	1200
5	0.2	1.2	1020

Table 3. The austenite grain size after homogenization at 1200 °C from 3 to 13 h.

Sample condition	As-cast	As-homogenized					
		3 h	5 h	7 h	9 h	11 h	13 h
Grain size (mm)	1.02	1.13	1.46	1.74	2.28	2.46	2.60

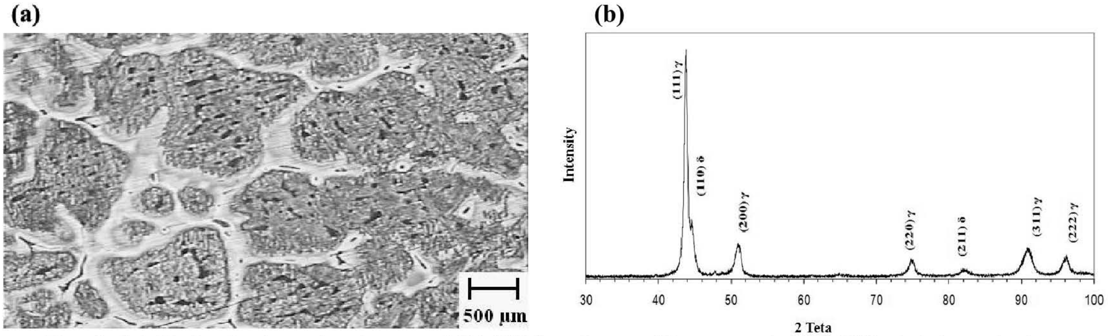

Fig. 1. (a) Optical microstructure and (b) XRD pattern of the as-cast AISI 301 stainless steel.

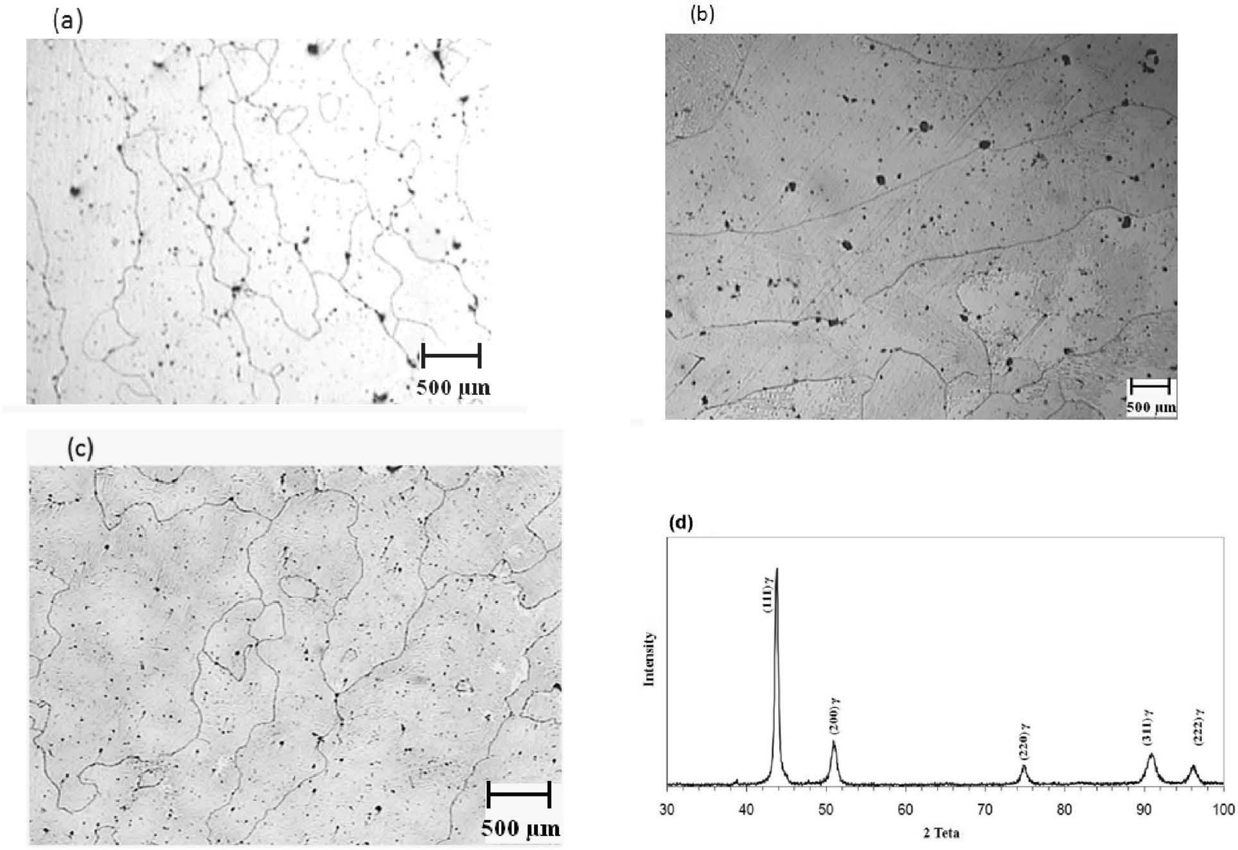

Fig. 2. Microstructure of the as-homogenized samples at 1200 °C for (a) 3 h, (b) 5 h and (c) 9 h. (d) XRD pattern of the sample homogenized at 1200 °C for 9 h.

The microstructure of specimens after hot rolling under strain of 0.8 and strain rate of 1.2 s^{-1} for homogenizing times from 3 to 13 h are shown in Fig. 3. As it can be seen, new small grains have been formed at the homogenized time of 3 h. The fraction of dynamic recrystallization (DRX) during hot rolling gradually increases with increasing the homogenization time till 9 h. At this time, the highest fraction of recrystallized grains with the average grain size of about 35±30 µm is achieved. It should be noted that in some grains the original austenite grain boundary show no evidence of DRX. This behavior is characteristic of heterogeneous DRX process [10-12].

The influence of homogenizing time on the hot rolled austenite grain size and hardness are presented in Fig. 4. As it can be seen, the homogenization time of 9 h represented the minimum average grain size and consequently maximum hardness. The minimum grain size is corresponded to a critical time in which a balance between two mechanisms of segregated particle dissolution and grain growth taking part during homogenization is achieved. EDS analysis was conducted to make sure for dissolution of the segregated particles as explained below.

The result of EDS micro-analysis on the grain boundary of the homogenized samples for 1, 3 and 9 h is shown in Fig. 5. As shown, Nb intensity peak is decreased by increasing homogenization time. The atomic arrangement at grain boundaries is relatively disordered and the interval among the atoms at grain boundaries is bigger. It provides a large driving force for segregation of elements such Nb and is inevitable for the Nb atoms to segregate to the grain boundaries. When the grain boundaries begin to move due to hot activation, the grain boundaries have to drag the Nb atoms to move together because of the segregation of Nb atoms. The diffusion velocity of the metal atoms of matrix influences the diffusion velocity of Nb atoms. Because the diffusion coefficient of Nb atoms in the ferrous material is less than that of grain boundary of Fe, the migration rate of grain boundary (f) is decreased. Based on Eq.1:

$$f = D_G/RT \qquad (1)$$

f is the migration rate of grain boundary, R the gas constant, T the absolute temperature, and D_G is the diffusion coefficient of grain boundary. Meanwhile

$$D_G = D_0 \exp(-Q_G/KT) \qquad (2)$$

Q_G is the diffusion activity energy of grain boundaries and D_0 is the diffusion constant. The diffusion coefficient (D_G) would be replaced by the coefficient of segregated atoms in the matrix so that the value of D_G greatly reduces. So, the grain-boundary movement is controlled by the diffusion rate of segregated atoms. Thus in insufficient homogenization time, f is small due to the high segregation of Nb atoms at grain boundaries and leads to retardation in recrystallization.

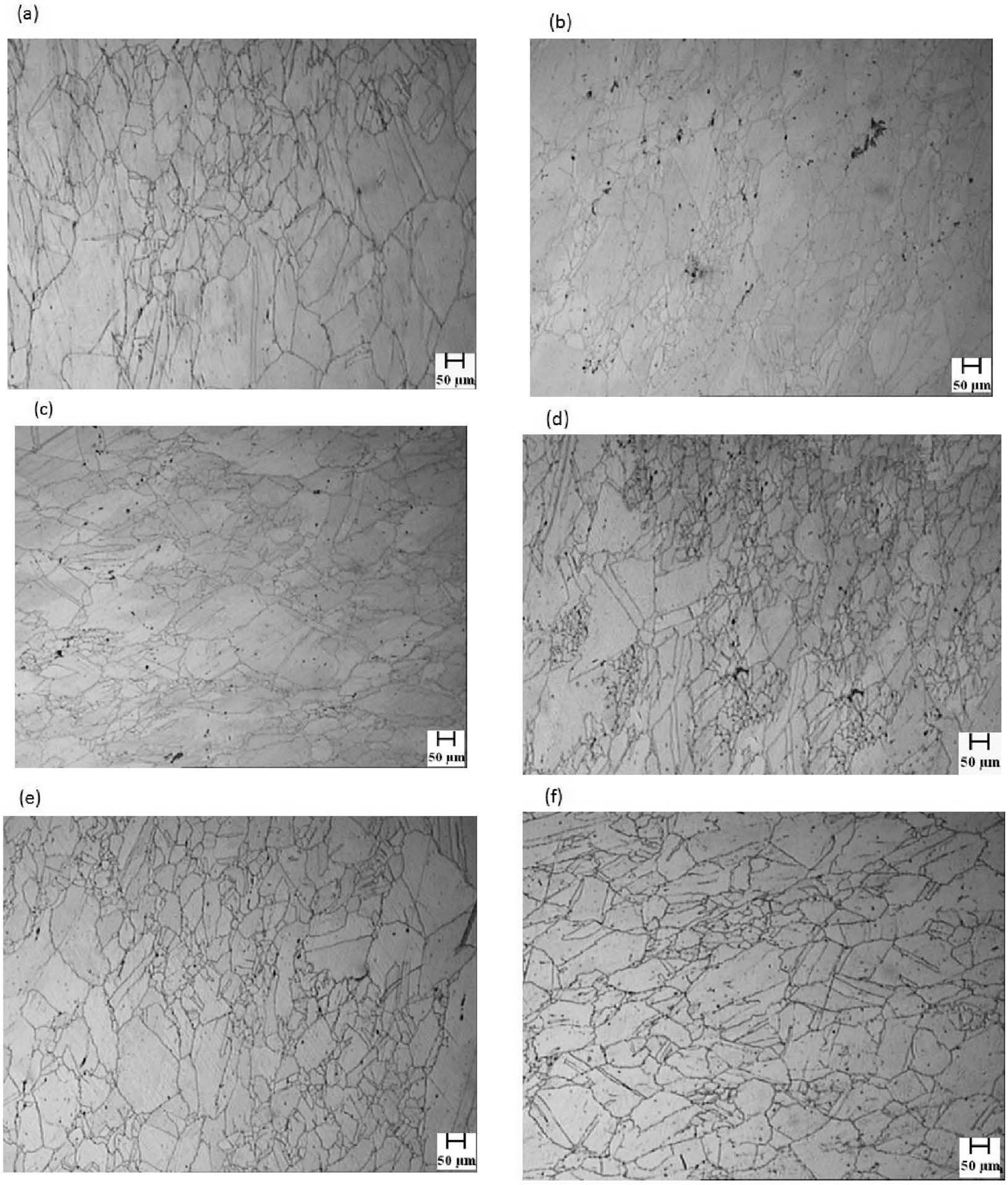

Fig. 3. Microstructure of the as-homogenized samples at 1200 °C for (a) 3 h, (b) 5 h, (c) 7 h, (d) 9 h, (e) 11 h and (f) 13 h, after hot rolling under strain of 0.8 and strain rate of 1.2 s^{-1}.

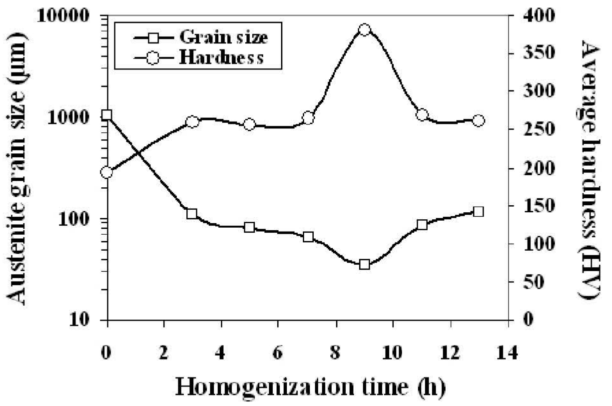

Fig. 4. Effect of the homogenization time on (a) austenite grain size and (b) hardness of the hot rolled samples.

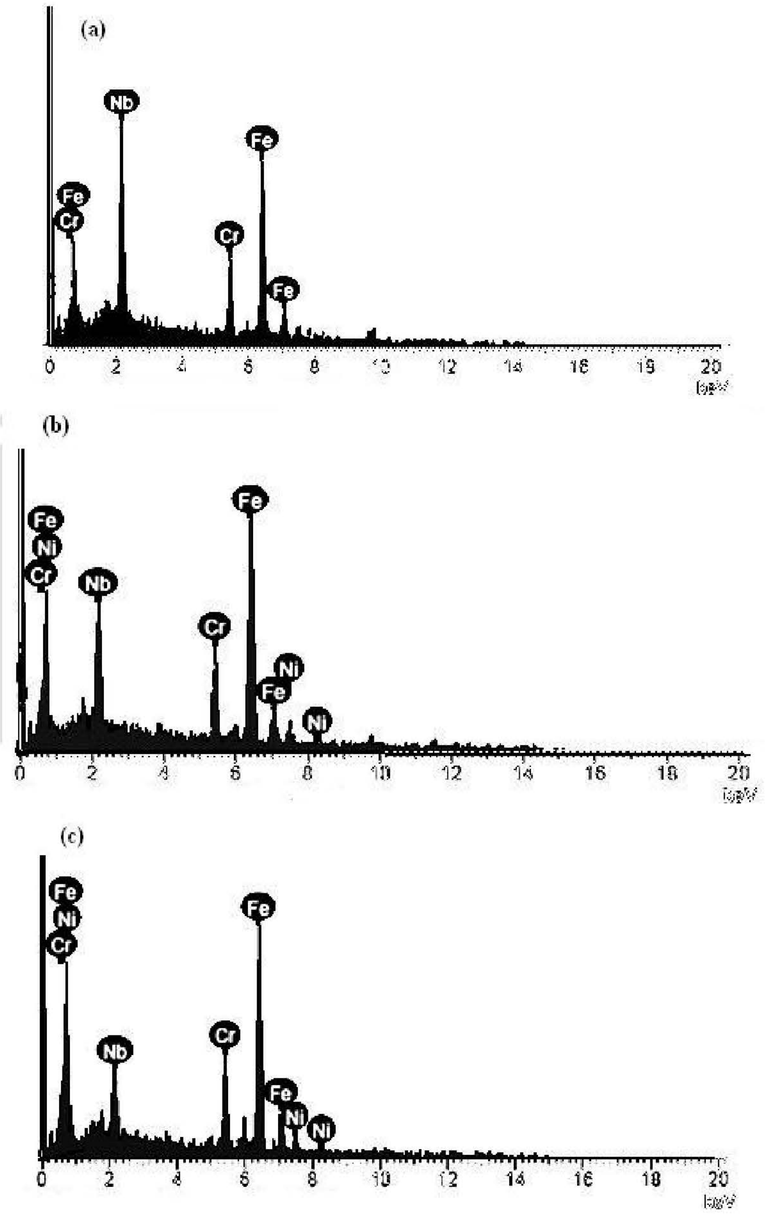

Fig. 5. The EDS analysis for various homogenization times: (a) 1 h, (b) 3 h and (c) 9 h.

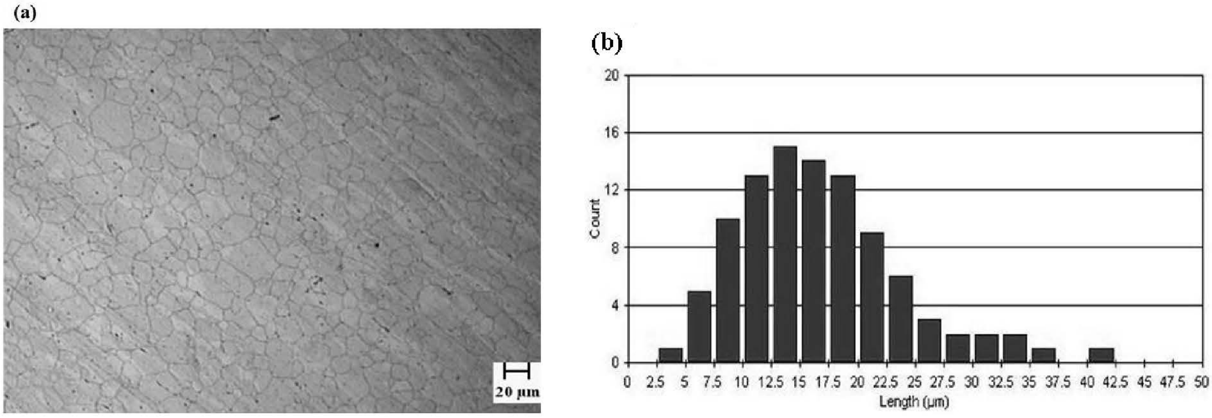

Fig. 6. (a) Microstructure of the 9 h homogenized sample after hot rolling with strain of 0.8 and strain rate of 1.2 s^{-1} after annealing at 1050 °C for 3 min, (b) grain size distribution.

Based on Eq.3, υ is the grain growth rate, f the migration rate of grain boundary, and F is the net driving force.

$$\upsilon = fF \qquad (3)$$

The grain growth rate in recrystallization process is determined by both f and F. Because of the segregation elements such as Nb atoms at austenite grain boundaries, the grain boundary movement is inhibited. With increasing the homogenization time, solubility of Nb atoms in lattice is increased and the grain boundaries begin to cast off the cloud. In excessive homogenization time, the migration rate of grain boundary suddenly increases and driving force dragged by Nb atoms begins to release so that the austenite grain rapidly grows up [2,5)]. Since volume fraction of the segregating element such as niobium depends upon the homogenization temperature and time, there should be a critical value of the homogenization time (t_c) at each temperature for optimum solution of such elements. This affects the retardation of DRX during subsequent hot rolling. Below this critical time, grain boundary mobility is slow due to the presence of high segregation fraction, but above this time because of excessive segregation dissolution, the grain boundaries mobility is increased and grain growth occurred.

The microstructure of the 9 h homogenized time after hot rolled at strain of 0.8 and annealed at 1050 °C for 3 min is shown in Fig. 6. As it can be seen, the average grain size is decreased from 35 to 16±7 µm after this annealing [2)]. In fact, the microstructure is further refined by static recrystallization.

3-2- Influence of strain rate on grain refinement

Influence of the hot rolled strain rate on the average grain size after homogenizing at 1200 °C for 5 and 7 h are illustrated in Figs. 7 and 8, respectively. As can be seen in these figures, the average grain size is decreased with increasing the strain rate for both homogenization times. It seems that increasing strain rate causes a lower retardation effect of elemental segregation. Under small strain rate, the solute atoms arrest dislocations and result in a decrease of their energy. Therefore, the force needed for the movement of dislocations will be increased. By increasing strain rate, speed of dislocation movement will be increased more than the diffusion rate of solute atoms, and the force needed for the movement of dislocation can reach to the minimum value [13)]. It means recrystallization can occur easier, and higher amount of grains are recrystallized.

3-3- Influence of strain on grain refinement

Influence of the hot rolled strain on grain refinement of austenite after 5 h homogenizing time is illustrated in Fig. 9. The first evidence of DRX is obvious by the formation of a limited number of new small grains at strain of 0.4 (arrows in Fig. 9a). More new small grains are formed with deformation to higher strain (ε = 0.8) (Fig. 9b). With increasing strain, most of the pre-existing boundaries are decorated by new DRX grains. With further deformation, the formation of new grains can continue without any major change in DRX grain size.

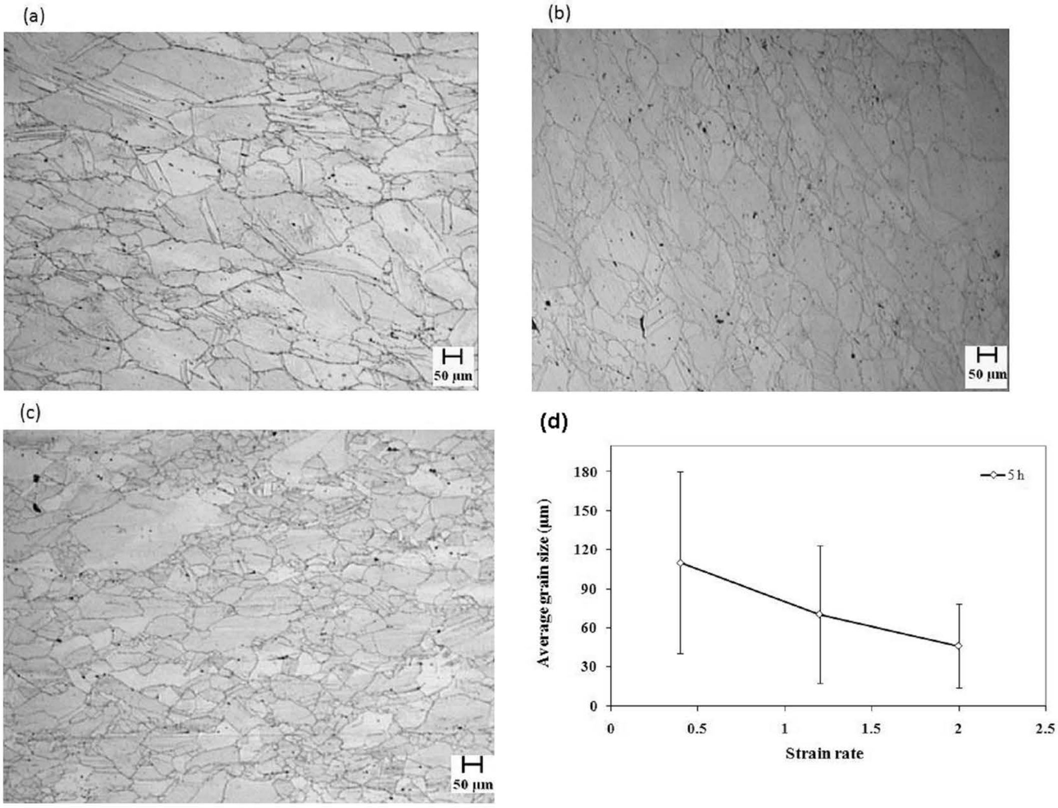

Fig. 7. Effect of hot rolled strain rate (at constant strain of 0.8) on grain refinement of the 5 h homogenized sample: (a) 0.4, (b) 1.2, (c) 2 s^{-1} and (d) average austenite grain size.

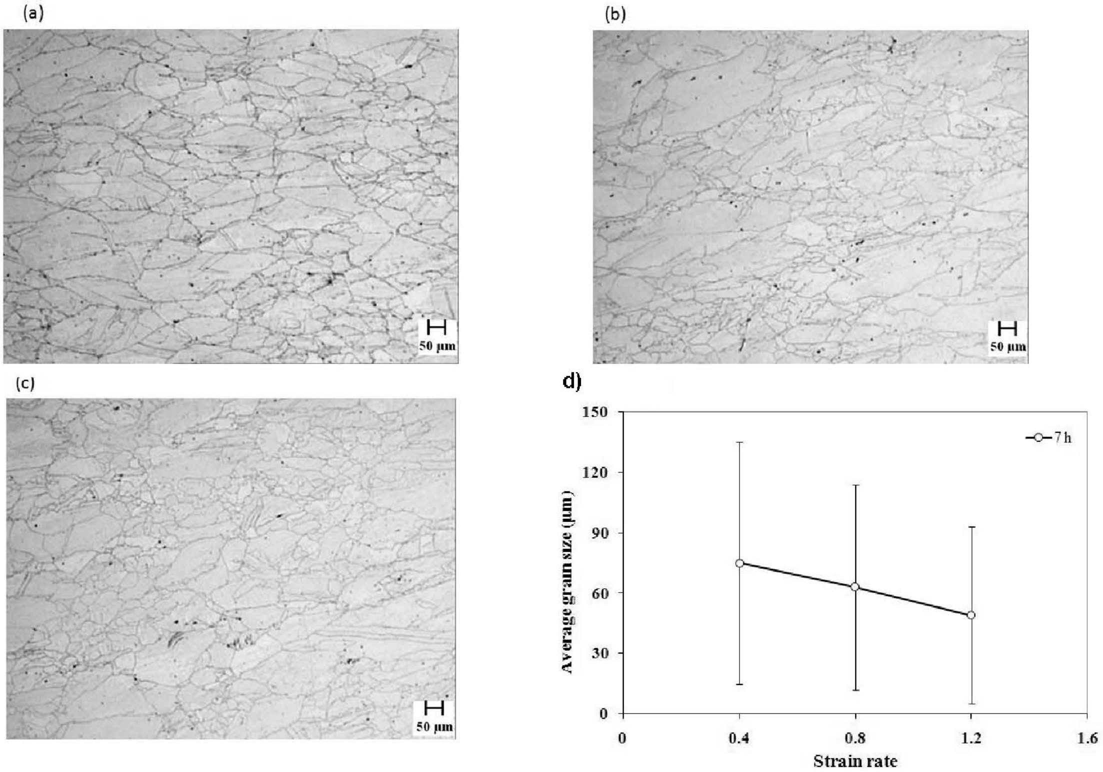

Fig. 8. Effect of hot rolled strain rate (at constant strain of 0.8) on grain refinement of the 7 h homogenized sample: (a) 0.4, (b) 0.8, (c) 1.2 s^{-1} and (d) average austenite grain size.

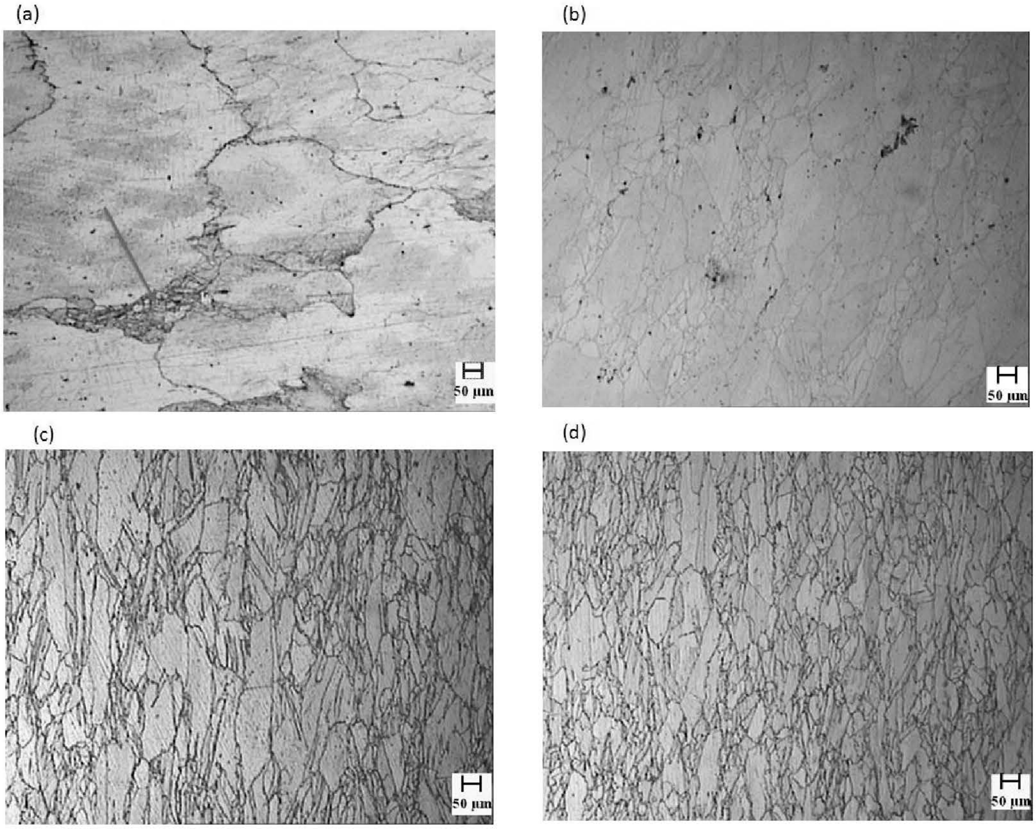

Fig. 9. Effect of hot rolled strain (at constant strain rate of 1.2 s^{-1}) on grain refinement of the 5 h homogenized sample: (a) 0.4, (b) 0.8, (c) 1.2 and (d) 1.5.

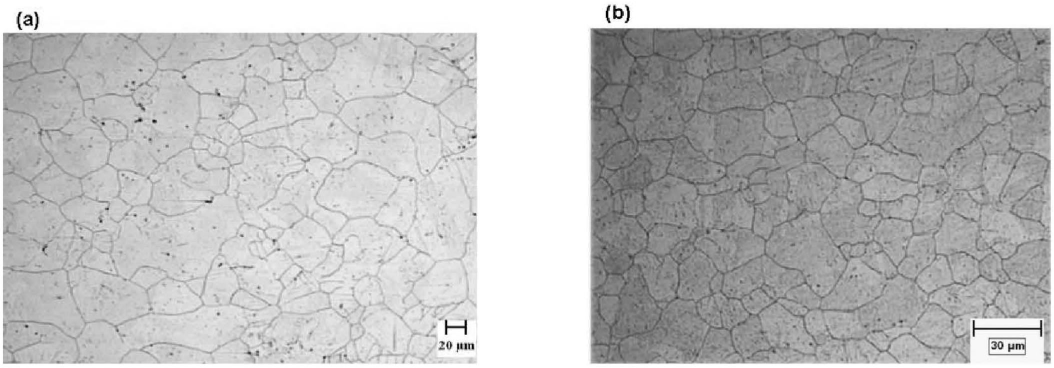

Fig. 10. Microstructure of the 5 h homogenized time after hot rolling with strain rate of 1.2 s^{-1} at strains of (a) 0.4 and (b) 1.5, followed by annealing at 1050 °C for 5 min.

The microstructures of the 5 h homogenized sample deformed under strain rate of 1.2 s^{-1} with two strains of 0.4 and 1.5 after annealing at 1050 °C for 5 min are illustrated in Figs. 10a and 10b, respectively. As it can be seen, with increasing strain from 0.4 to 1.5, the average grain size is reduced from 65 to 19 μm.
It can be seen in Figs. 7-9 that with decreasing strain rate and strain, the effects of segregating elements are more pronounced on the retardation of DRX 2). This clearly shows that for the homogenization time smaller than t_c, increasing the strain rate and strain could lead to more grain refinement.

4- Conclusion

The main conclusions obtained from the present work are the followings:

- The optimum homogenization time for the as-cast 301 stainless steel for subsequently hot rolled at temperatures of 1000 – 1200 °C with strain and strain rate of 0.8 and 1.2 s^{-1} respectively, is about 9 h.
- The optimum homogenization time depends upon the subsequent hot rolling conditions, i.e. increasing strain or strain rate would decrease the homogenizing time.
- Effect of Nb element on the retardation of recrystallization was pronounced. Also it seems that, there is an optimum of solute segregation fraction below which retardation is dominant and above which grain growth of the recrystallized grains.
- Effect of solute drag on primary grain refinement in as-cast condition is more pronounced. Therefore, selecting the suitable homogenization time and temperature, strain and strain rate during subsequent hot rolling have an important role to neutralize the solute drag effect on retardation of recrystalliziation and grain refinement.

5- Acknowledgements

The authors would like to acknowledge Dr. M. R. Toroghinejad for his useful comments.

References

[1] H. J. McQueen, J. J. Jonas: Appl Metalworking, 3(3) (1984), 233.
[2] I. Tamura, H. Sekine, T. Tanaka, C. Ouchi: Thermomechanical Processing of High-Strength Low-alloy Steel, 1st ed., Butterworth, (1988), 95.
[3] S. Yamamoto, C. Ouchi, T. Osuka: Thermomechanical processing of microalloyed austenite, American Institute of Metallurgical Engineers, (1981), 150.
[4] K. T. Aust, J. W. Rutter, Trans. MIME, 215 (1959), 119.
[5] Y. Qingbo, Y. Sun, Mate. Sci. Eng. A, (2006), 420.
[6] L. M. Storozheva, Metal Sci. Heat Treat., 31 (9-10) (1990), 692.
[7] M. Enomoto, H. I. Aaronson, Scripta Metall, 19 (1985), 1.
[8] M. Enomoto, Y. Sato, Mater. Trans A, 35 (1994), 862.
[9] A. I. Zaky Farahat, T. El-Bitar, Eman El-Shenawy: Mater. Sci. Eng. A, 492 (2008), 161.
[10] A. Belyakov, H. Miura, T. Sakai, Mater. Sci. Eng. A, 255 (1998), 139.
[11] W. Roberts, B. Ahlblom, Acta Metall., 26 (1978), 801.
[12] E. Brunger, X. Wang, G. Gottstein, Scripta Mater., 38 (1998), 1843.
[13] W. F. Hosfor, R. M. Caddell, Metal Forming-Mechanics and Metallurgy, Second ed., Prentic Hall, 1993.