

Investigation of the Grain Growth Evolution in the AISI 304H Austenitic Stainless Steel

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Abstract: The austenitic stainless steels usually present an excellent combination of corrosion resistance and mechanical properties such as ductility in the annealed condition and high yield strength after cold deformation. Solution annealing in the AISI 304H is recommended before deformation process in order to improve ductility. However, long annealing during solution annealing can cause GG (grain growth) or AGG (abnormal grain growth) in the AISI 304H. In these cases, ductility is strongly decreased. Therefore, GG or AGG must be avoided during solution annealing. In this article, grain growth during solution annealing of the AISI 304H samples was determined. Samples of the AISI 304H were annealed at 1,100 °C for solution-annealing times varying from 15 min to 180 min. The results show that AGG took place for samples annealed at 1,100 °C for 30 min. In this condition, grain size reached $70 \pm 10 \mu\text{m}$. After annealing solution at 1,100 °C for 180 min, grain size reached $120 \pm 20 \mu\text{m}$. In summary, the results shown that solution annealing at 1,100 °C even for relatively short annealing promotes the prompt increase of the grain size.

Key words: AISI 304H, austenitic stainless steel, solution annealing, grain growth, abnormal grain growth.

1. Introduction

Austenitic stainless steels play a key role in important industrial sectors such as chemical, petrochemical and pharmaceutical. These steels are non-magnetic and they are not hardenable by heat treatment. Austenitic stainless steels have excellent properties such as corrosion resistance, weldability and ductility [1]. In some applications, due to the low yield strength in these steels, work hardening is an important alternative for the increasing in mechanical resistance [1]. In order to improve the ductility, the solution annealing in the austenitic stainless steels is commonly specified before usage of these steels in the forming process. In this process, the carbides which present previously in the microstructure are dissolved. The main carbides formed in austenitic stainless steels are those chromium-rich carbides such as M_{23}C_6 type Cr (M = Cr, Fe or Mo). Due to the slow dissolution of these carbides during solution annealing, normal

and abnormal grain growth may occur in temperatures above 1,150 °C. For AISI 304H austenitic stainless steel, temperatures between 1,000 °C and 1,150 °C are reached during the solution annealing. For Ti-stabilized or Nb-stabilized austenitic stainless steels, solution annealing temperature should not exceed about 1,050 °C [2]. For these steels, the occurrence of abnormal grain growth, also known as secondary recrystallization, is rather frequent as the precipitates present in the microstructure are partially dissolved [1, 3, 4].

The kinetics of grain growth have been modeled by various authors over the past few decades [5, 6]. The grain boundaries distribution (mesotexture) has also been the goal of recent researches [2, 7, 8]. From the point of view of technological application, the grain growth is prevented in most cases, since this phenomenon promotes losses in mechanical properties. However, grain growth annealing are widely used in the silicon steel (Fe-3% Si, weight percent) because in this case, the texture obtained is suitable for applications in induction motors [3]. In this article, the grain growth

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Table 1 Chemical composition of the AISI 304H used in this work (weight percent).

	AISI 304H
C	0.044
Si	0.634
P	0.029
S	0.001
Cr	18.079
Ni	8.039
Cu	0.102
Co	0.182

in austenitic stainless steel AISI 304H samples annealed at 1,100 °C for times ranging from 15 to 180 min was investigated. The microstructural evolution using SEM (scanning electron microscopy) was also investigated.

2. Experimental

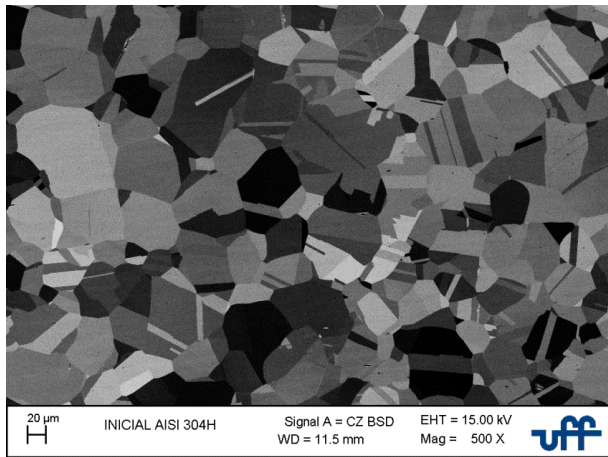
The AISI 304H austenitic stainless steel sheets were provided by the Aperam South America. The chemical composition of the samples is shown in Table 1. The sheets were previously hot rolled with final rolling temperature close to 1,000 °C (as-received condition). Samples of these hot-rolled sheets were cut into an Isomet 1000 Precision Saw (Buehler) with dimensions of about $10 \times 10 \times 4 \text{ mm}^3$. Then, the samples were solution annealed at 1,100 °C for annealing times varying from 15 min to 180 min in air using a tubular furnace (Carbolite). After solution annealing, the samples were metallographically prepared using the following steps: (1) Mounting using phenolic resin; (2) Grinding using 600, 800, 1,200, and 2,400 grit SiC abrasive paper (P-grade standard); (3) Initial polishing using 3-micron diamond paste (Buehler); (4) Final polishing using colloidal silica (OP-S/Struers). The micrographs were obtained using the SEM. The equipment used was the Zeiss EVO MA10 with LaB₆ filament. The grain size measurements were made using linear intercept method according to the ASTM E-112.

3. Results and Discussion

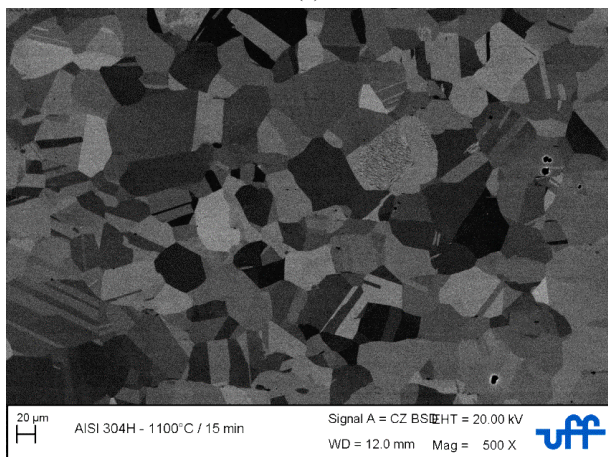
The evolution of the grain growth in AISI 304H samples is shown from Figs. 1-3. In some micrographs

is possible to observe the presence of residual strain-induced martensite. It was indicated by arrows in Fig. 1. This hard phase was generated during sectioning or grinding of the AISI 304H samples. So, this phase was considered a metallographic preparation defect and its analysis will be neglected. In Fig. 1a, SEM micrograph shows the AISI 304H microstructure after hot rolling (as-received condition). In this micrograph, it can be observed a reasonably homogeneous grain size distribution. It is not possible to observe the presence of the other phases, for instance, chromium-rich carbides such as $M_{23}C_6$ in the microstructure. This result suggests that final rolling temperature (1,000 °C) was sufficient to dissolve all of the previously formed carbides. Furthermore, the presence of the annealing twins is evident in most of the grains. Results in the literature [3] show that the carbide dissolution during solution annealing can promote abnormal grain growth [2]. In this work, the carbides have been dissolved previously during hot rolling. In this case, twinning boundaries ($\Sigma 3$) can act as barriers to the movement of grain boundaries during solution annealing, therefore, reducing the grain boundary mobility [3]. The twinning mechanism during annealing has been studied by some authors for the pure nickel. The formation of this kind of defect is still an open question in the literature [9, 10]. In Fig. 1b, the micrograph shows the samples annealed for 15 min. In this micrograph, a relative similar grain-size distribution can be observed. The micrograph also shows the presence of intense annealing twins and a reasonably homogeneous grain size distribution. In contrast, the micrograph of Fig. 1c (annealing time of 30 min) shows a substantial increase in grain size, in addition to annealing twins.

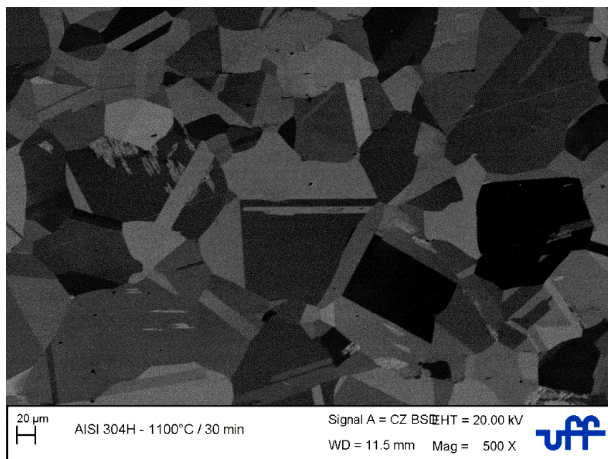
In Fig. 2, the samples were annealed between 60 min and 150 min. The results show that some grains have reached a size greater than 100 μm . These microstructure containing large grains should be avoided due to the losses in the mechanical properties [3]. In Fig. 3, the micrograph shows the sample



(a)

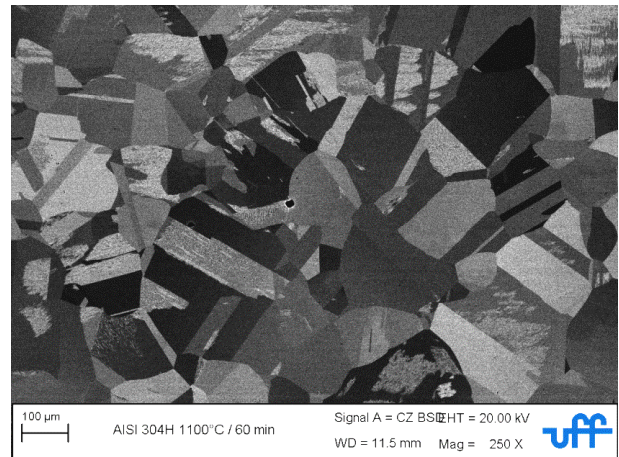


(b)

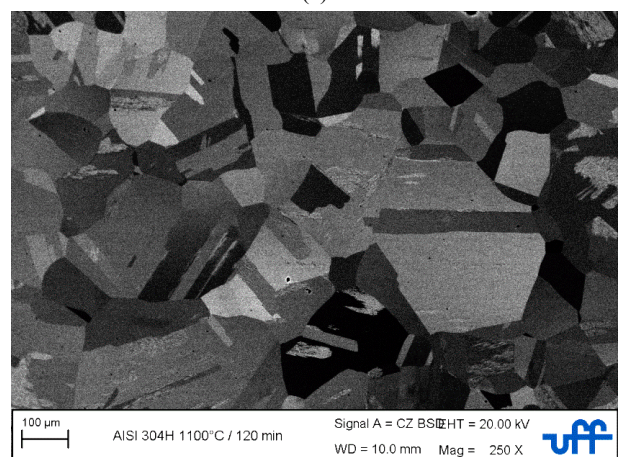


(c)

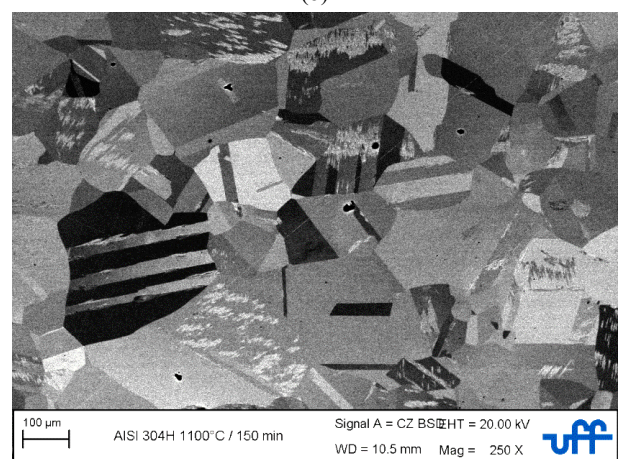
Fig. 1 SEM micrographs of the rolling plane of the AISI 304H samples after solution annealing: (a) as-received condition, (b) 15 min and (c) 30 min.



(a)



(b)



(c)

Fig. 2 SEM micrographs of the rolling plane of the AISI 304H samples after solution annealing: (a) 60 min, (b) 120 min and (c) 150 min.

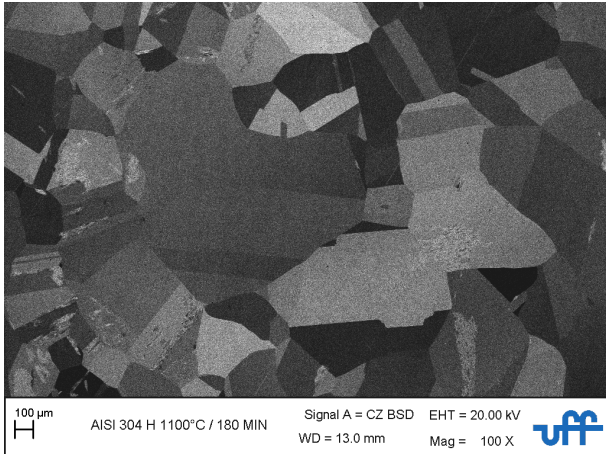


Fig. 3 SEM micrographs of the rolling plane of the AISI 304H samples after solution annealing. Holding time was 180 min.

annealed at 180 min. For this annealing time the micrograph reveals a wide grain-size distribution. It is possible to observe grains of more than 300 μm and others with only 50 μm are also observed. These grain size changes can occur due to both the nature of grain boundaries (mesotexture) and also due to interaction between grain and twin boundaries. Another example of grain size variation was observed in the micrograph of Fig. 4. The micrograph shows a sample annealed at 30 min. In the central part of the micrograph it is possible to observe a grain that grew abnormally in the microstructure. In general, the results show that the grain growth occurs in a homogeneous (normal) manner. However, the abnormal grain growth was observed discretely in some samples and cannot be ignored in this microstructural characterization.

In this article, the grain growth was measured using the linear intercept method and the results are summarized in Fig. 5. The grain growth has occurred promptly for samples annealed up to 60 min reaching close to $100 \mu\text{m} \pm 19 \mu\text{m}$. In the as-received condition, the average grain size of the sample was 30 μm . For samples annealed for a longer time than 60 min, grain growth progresses slowly and grain size does substantially not change. For instance, the sample annealed for 180 min, the average grain size is about $120 \mu\text{m} \pm 24 \mu\text{m}$. This result is expected for samples annealed for long time (60 min or more). For

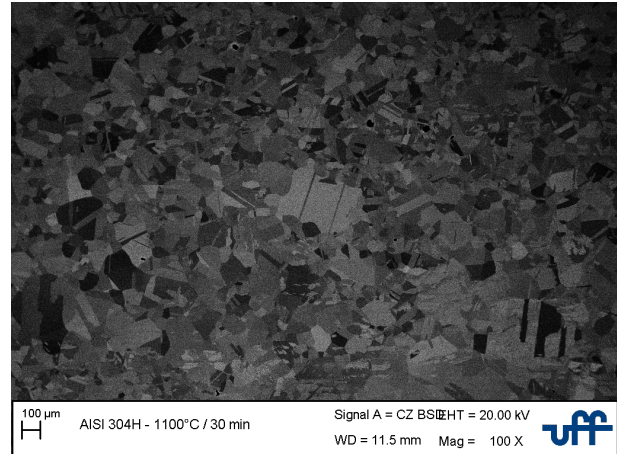


Fig. 4 SEM micrographs of the rolling plane of the AISI 304H samples after solution annealing at 1,100 °C for 30 min. Abnormal grain nucleation is visible in the center of the micrograph.

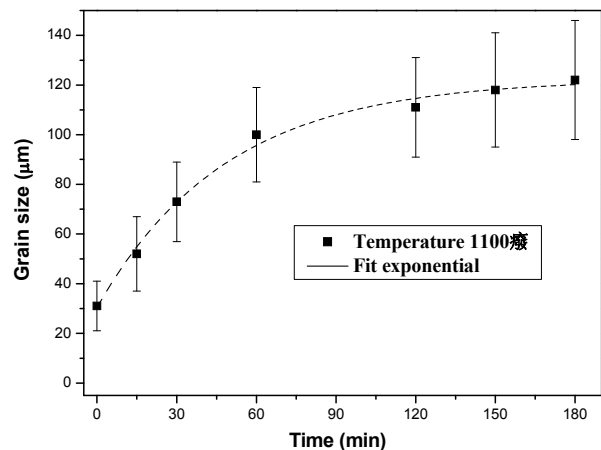


Fig. 5 Evolution of the grain size with annealing time in AISI 304H samples.

these samples, the grain boundaries fraction, the driving force of the grain growth, is reduced in this condition. Therefore, the grain growth kinetic is also remarkably reduced [3].

4. Conclusions

In this paper, the grain size evolution of the AISI 304H austenitic stainless steel was investigated. The solution annealing temperature was of 1,100 °C for annealing times ranging between 15 and 180 min. Generally, grain growth took place quickly for samples annealed up to 60 min. However, from this time annealing, the grain size increases slowly in the microstructure due to reducing of the grain boundary

fraction (driving force) present in the microstructure. It is worth mentioning that some evidence of abnormal grain growth was observed locally in sample annealed at 1,100 °C for 30 min.

References

- [1] Totten, G. E., ed. 2006. *Steel Heat Treatment: Metallurgy and Technologies*. Boca Raton, p. 695.
- [2] Mandal, S., Bhaduri, A. K., and Sarma, V. S. 2009. "Studies on Twinning and Grain Boundary Character Distribution during Anomalous Grain Growth in a Ti-modified Austenitic Stainless Steel." *Materials Science and Engineering A* 515: 134-40.
- [3] Humphreys, F. J., and Hatherly, M. 2004. *Recrystallization and Related Annealing phenomenon*, Pergamon. Oxford: Elsevier Science Ltd.
- [4] Gladman, T. 1997. *The Physical Metallurgy of Microalloyed Steels*. Cambridge: The Institute of Materials.
- [5] Hillert, M. 1965. "On the Theory of Normal and Abnormal Grain Growth." *Acta Metallurgica* 13: 227-38.
- [6] Gladman, T. 1966. "On the Theory of the Effect of Precipitate Particles on Grain Growth in Metals." In *Proceedings of Royal Society London A294*, 298-309.
- [7] Kumar, R. B., Das, S. K., Mahato, B., Das, B., and Chowdhury, S. G. 2007. "Effect of Large Strains on Grain Boundary Character Distribution in AISI 304L Austenitic Stainless Steel." *Materials Science and Engineering A* 454-455: 239-44.
- [8] Jones, R., and Randle, V. 2010. "Sensitisation Behaviour of Grain Boundary Engineered Austenitic Stainless Steel." *Materials Science and Engineering A* 527: 4275-80.
- [9] Jin, Y., Lin, B., Bernacki, M., Rohrer, G. S., Rollett, A. D., and Bozzolo, N. 2014. "Annealing Twin Development during Recrystallization and Grain Growth in Pure Nickel." *Materials Science and Engineering A* 597: 295-303.
- [10] Chen, X. P., Li, L. F., Sun, H. F., Wang, L. X., and Liu, Q. 2015. "Studies on the Evolution of Annealing Twins during Recrystallization and Grain Growth in Highly Rolled Pure Nickel." *Materials Science and Engineering A* 622: 108-13.