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 grain growth (GG) or abnormal grain growth (AGG) 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 1100°C for solution-annealing times varying from 15 min to 180 min. The results show that AGG took place for samples annealed at 1100°C for 30 min. In this condition, grain size is about 70 ± 10 μm. After annealing solution at 1100°C for 180 min, grain size reached 120 ± 20 μm. In summary, the results shown that solution annealing at 1100°C even for relatively short annealing promotes the prompt increase of the grain size.*

Keywords: *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 (Totten, 2006). In some applications, due to the low yield strength in these steels, work hardening is an important alternative for the increasing in mechanical resistance. In order to improve the ductility, the solution annealing is commonly used in the austenitic stainless steels before their application in order to dissolve the carbides presents previously in the microstructure. 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 1150°C. For AISI 304H austenitic stainless steel, temperatures between 1000°C and 1150°C are reached during the solution annealing. For Ti-stabilized or Nb-stabilized austenitic stainless steels, solution annealing temperature should not exceed about 1050°C (Mandal *et al.*, 2009). 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 (Humphreys, 2004; Gladman, 1997).

The kinetics of grain growth have been modeled by various authors over the past few decades (Hillert, 1965; Gladman, 1966) The grain boundaries distribution (mesotexture) has also been the goal of recent researches (Kumar *et al.*, 2007; Jones and Randle, 2010; Mandal *et al.*, 2009). 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 (Humphreys, 2004). In this article, the grain growth in austenitic stainless steel AISI 304H samples annealed at 1100°C for times ranging from 15 to 180 min was investigated. The microstructural evolution using scanning electron microscopy (SEM) was also investigated.

2. EXPERIMENTAL

AISI 304H austenitic stainless steel sheets were provided by the Aperam South America. The chemical composition of the samples is shown in the Table 1. The sheets were previously hot rolled with final rolling temperature close to 1000°C (as-received condition). Samples of these hot-rolled sheets were cut into an Isomet 1000 Precision Saw (Buehler) with dimensions of about 10 x 10 x 4 mm^3. Then, the samples were solution annealed at 1100°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: i. Mounting using phenolic resin. ii. Grinding using 600, 800. 1200 and 2400 grit SiC abrasive paper (P-grade standard). iii. Inicial polishing using 3-micron diamond paste (Buehler) iv. Final polishing using colloidal silica (OP-S/Struers). The micrographs were obtained using the scanning electron microscopy (SEM). The equipment used was the Zeiss EVO MA10 with LaB_6 filament. The grain size measurements were made using linear intercept method according to the ASTM E-112.

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

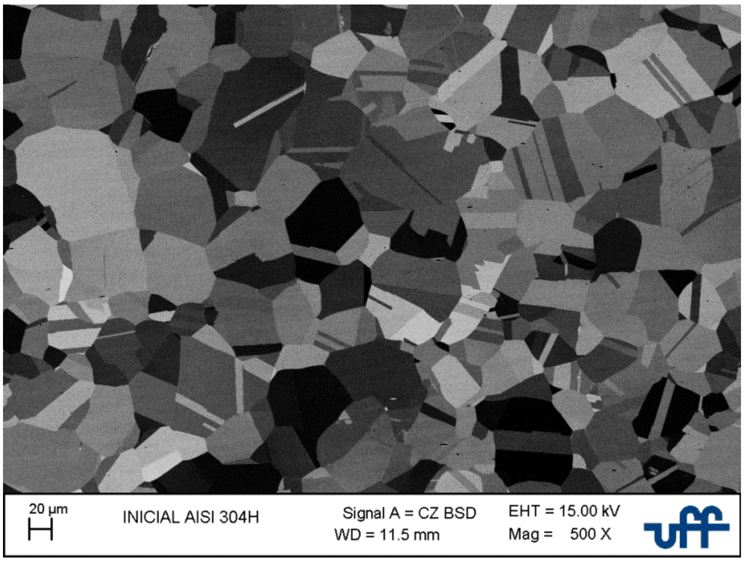
	c	Si	P	S	Cr	Ni	Cu	Co
AISI304H	0,044	0,634	0,029	0,001	18,079	8,039	0,102	0,182

3. RESULTS AND DISCUSSION

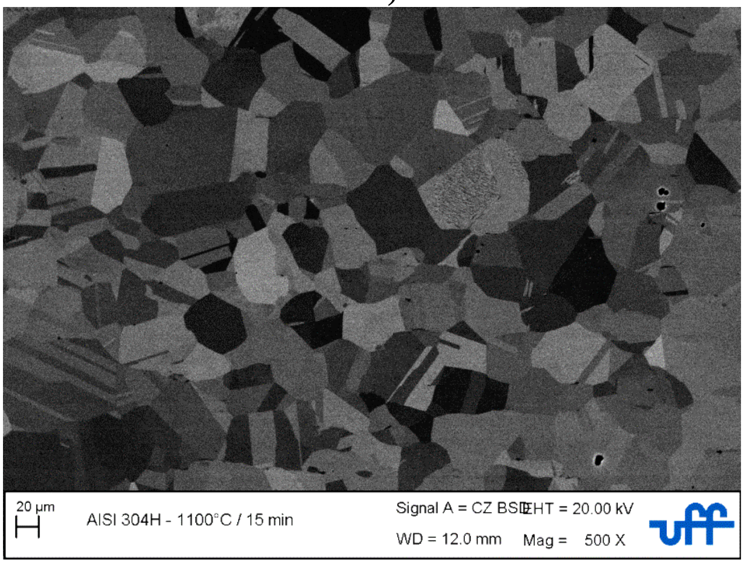
The evolution of the grain growth in AISI 304H samples is shown from Figure 1 to Figure 3. In some micrographs is possible to observe the presence of residual strain-induced martensite. This hard phase was generated during sectioning or grinding of the AISI 304H samples. So, this phase was considered a metallographic preparation defect and its analysis will be neglected. In the Figure 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 (1000 ° 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 (Humphreys, 2004) show that the carbide dissolution during solution annealing can promote abnormal grain growth (Mandal *et al.*, 2009). 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 (Humphreys, 2004). 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 (Jin *et al.*, 2014; Chen *et al.*, 2015). In Figure 1b, the micrograph shows the samples annealed for 60 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 the Figure 1c (annealing time of 30 min) shows a substantial increase in grain size, in addition to annealing twins.

In the Figure 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 (Humphreys, 2004). In the Figure 3, the micrograph shows the sample annealed at 180 min. For this annealing time the micrograph reveals a wide grain-size distribution. It´s 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 Figure 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.

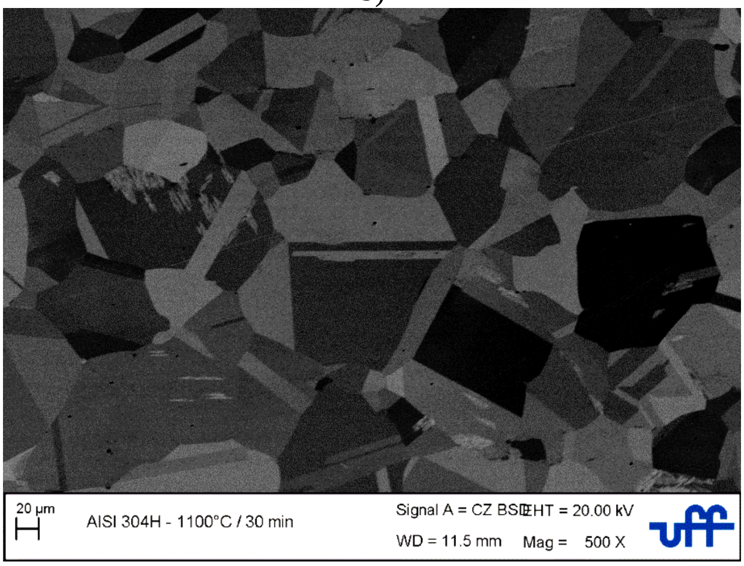
In this article, the grain growth was measured using the linear intercept method and the results are summarized in the Figure 5. The grain growth has occurred promptly for samples annealed up to 60 min reaching close to 100 $\pm$ 19 µm. In the as-received condition, the average grain size of the sample 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 $\pm$ 24 µm. This result is expected for samples annealed for long time (60 min or more). For 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 (Humphreys, 2004).



a)

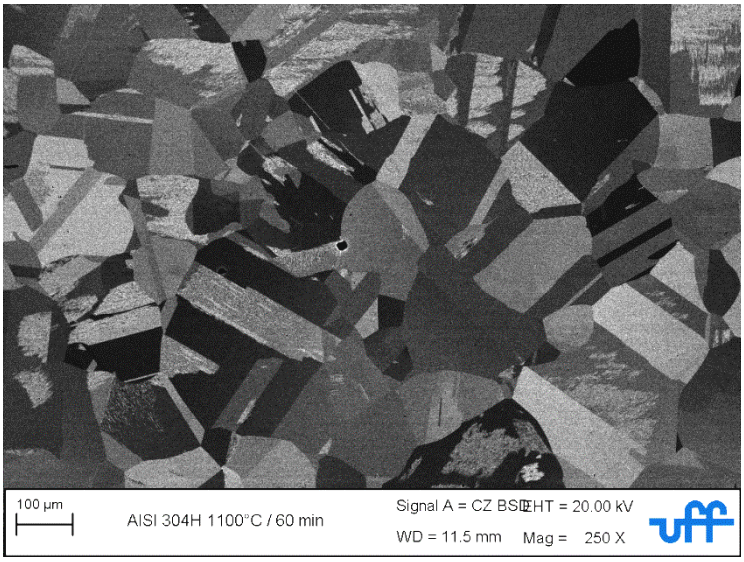


b)

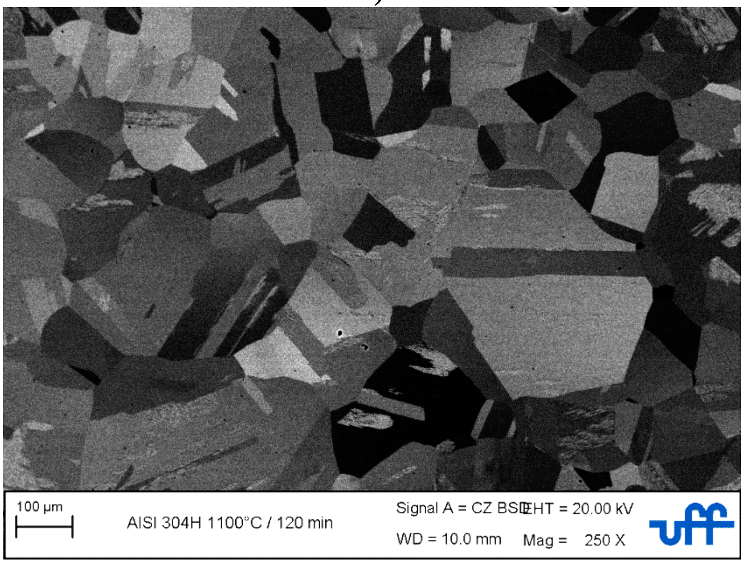


c)

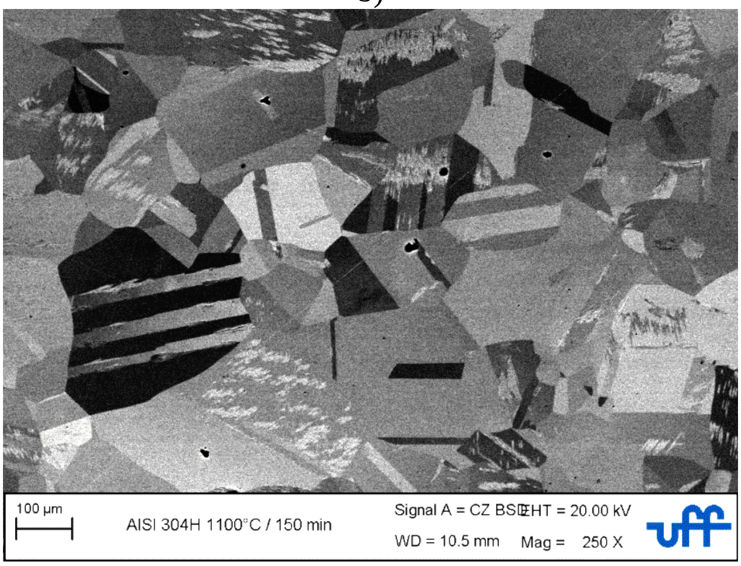
Figure 1 – SEM micrographs of the rolling plane of the AISI 304H samples after solution annealing: (a) as-received condition, (b) 15 min and (c) 30 min.



a)



b)



c)

Figure 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.

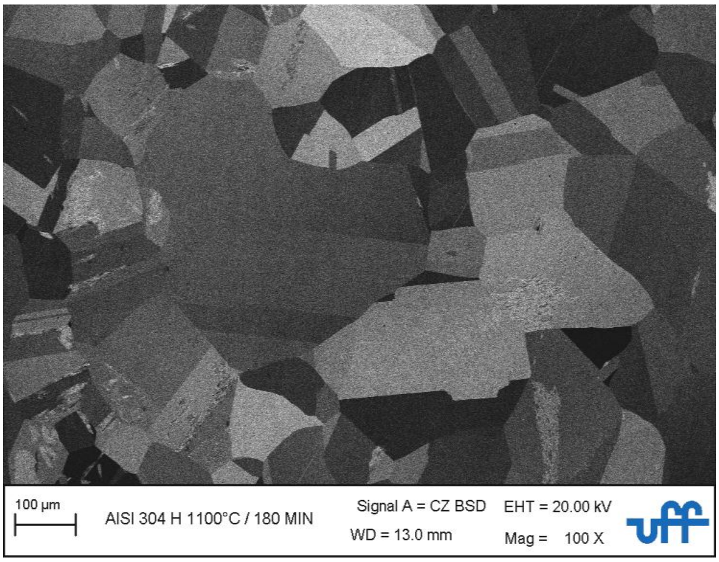


Figure 3 – SEM micrographs of the rolling plane of the AISI 304H samples after solution annealing. Holding time was 180 min.

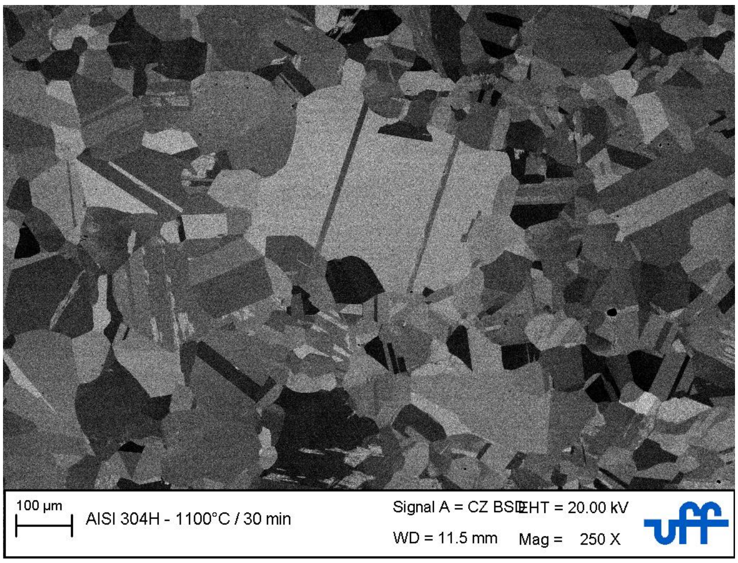


Figure 4 – SEM micrographs of the rolling plane of the AISI 304H samples after solution annealing at 1100°C for 30 min. Abnormal grain nucleation is visible in the center of the micrograph.

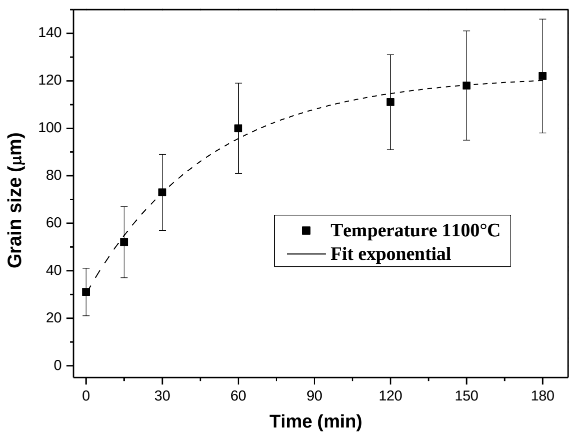


Figure 5 – Evolution of the grain size with annealing time in AISI 304H samples.

4. CONCLUSION

In this paper, the grain size evolution of the AISI 304H austenitic stainless steel was investigated. The solution annealing temperature was of 1100°C for anneling 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 1100 °C for 30 min.

5. REFERENCES

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