

# Effect of Solution Annealing Heat Treatment on the Corrosion Resistance and Mechanical Properties of an Austenitic Stainless Steel

Ali Alavi Shoushtari<sup>1,a</sup>, Meysam Sharafi<sup>2</sup>, Reza Moravej Aleali<sup>3</sup>

<sup>1</sup> IKA Engineering and Technical Inspection Co., Tehran, Iran

<sup>2</sup> Material Science Department, Islamic Azad University of Najafabad, Iran

<sup>3</sup> Nalco Chemical Company, UAE

<sup>a</sup> [Alialavi.sh@gmail.com](mailto:Alialavi.sh@gmail.com)

## Abstract

One of the most applicable grades of austenitic stainless steel is 304 which is used widely in storage tank to chemical transportation. Although this alloy has suitable properties for engineering uses, its poor resistance to intergranular corrosion restricted its use in industries. Meanwhile, this problem lead to reduction in the average working hours in equipment in which 304 alloy is used. To remedy this problem, there are many different methods has been introduced in the recent decades. However, finding a method which increase corrosion resistance of alloy without any collapse in other alloy's properties still a challenge. In this study, different heat treatment cycles on the 304 alloy were applied and then the corrosion rate was measured. Furthermore, mechanical tests were carried out to find out which cycle resulted in optimum properties. The results illustrated that reducing carbide participate led to better mechanical properties as well as corrosion resistance.

**Keywords:** annealing, heat treatment, stainless steel, corrosion, mechanical properties

## Introduction

The austenitic stainless steels generally contain from 6 to 22% Ni to stabilize the austenite at room temperature. They also contain other alloying elements, such as chromium for corrosion resistance, and smaller amounts of manganese and molybdenum. This grade of steel is so applicable, particularly during recent decades because of their special properties. However, these alloys are sensitised to some type of corrosion.

The most important type of corrosion in the austenitic stainless steel groups is intergranular corrosion. This type of corrosion is occurred due to deplete Chromium in grain binderies. Chromium is the most effective element (again) in protect

stainless steel against various kinds of corrosion [1,2]. The minimum amount of Chromium to protect steels from corrosion is 11 percentages [3]. If austenitic stainless steels expose in the 500oC to 800oC temperature range, a new phase, Cr<sub>23</sub>C<sub>6</sub>, is appeared and if steels hold in this range, the phase Precipitates form solid solution [4,5]. In this condition, the grain boundary zones empty out of Chromium so that the stainless steel seems like plain steel without any anti-corrosion character. The point is that in this converted steel the areas adjacent to empty from Chromium zones are so Sensitive to corrosion [5,6]. Solution annealing heat treatment is a practical and useful method to resolve this problem and improves the corrosion resistance characters. As the temperature range of this heat treatment is high (1080 to 1160°C), it is essential to find a optimum holding time to do operation [7,8].

Holding the steel in the high temperatures more than enough could enhance resistance to intergranular corrosion. In contrast, it causes collapse in surface quality and mechanical properties due to increasing grain size [9,10]. There are many tests and experiments to determine types of corrosion in austenitic stainless steels, but the measurement of intergranular corrosion by using standard chemical solutions according to ASTM A262 is the most famous and acceptable one [11].

In this paper, the effect of solution annealing heat treatment on the corrosion rate and mechanical properties of 304 stainless steels was studied to attain the optimum temperature and holding time.

## Experimental

The Regards microstructure of sample under investigation, we used two methods: using Iron Sulphate – boiling sulphuric acid solution, and using boiling nitric acid to measure intergranular corrosion and sketch the data on a diagram. For this reason an austenitic stainless steel sheet grade 304 with 2 millimetre thickness was used. Table 1 shows the chemical composition of sheet. A sample was taken from sheet to determine its microstructure according to ASTM A262 and compare it with standard metallographic samples. For this purpose, after doing mechanical abrasive grinding, electropolishing was done by using A2 electropolish solution made by Struers Company. Afterward, samples were electroetched by oxalic acid (table 2). To improve quality of etching, temperature of was maintained below 50°C.

Samples' microstructure was observed by optical microscopy in 500 X fold. Several specimens in the dimension 55\*44 millimetres were prepared from the sample and categorized in four groups and exposed in different solution annealing heat treatments. The treatment temperature was 1100°C [9]. The specimens were annealed in the mentioned temperature in the period of 5 to 50 minute in the 5 minute time interval at the vacuum furnace with  $10^{-5}$  mille Bar. Then, all specimens were cooled form solution annulling temperature in the water. To obtain better consequence, the surface of all specimens polished, and then they weighted with (0.0001) accuracy to use in evaluation of intergranular corrosion in the corrosive mediums.

Table 1– Chemical Composition of Sample

<b>Cu</b>	<b>Ni</b>	<b>Mo</b>	<b>Cr</b>	<b>S</b>
0.46	9.78	0.33	18.73	0.017
<b>P</b>	<b>Mn</b>	<b>Si</b>	<b>C</b>	<b>Fe</b>
0.012	1.43	0.66	0.08	Rest

Table 2– The etch condition

<b>Electrolyte</b>	H2C2O4.2H2O
<b>Cathode</b>	Austenitic Stainless Steel
<b>Current density</b>	1.5 A/m2
<b>Etching Time</b>	2.5 min
<b>Preparation after etching</b>	Washing by 10% nitric acid to remove yellow layer on specimens

For Iron Sulphate – sulphuric acid media, the surface of specimens were contacted with the solution. After that a condenser system with a heater was installed on the solution container to boil sulphuric acid. The specimens were held continuously for 120 hours in the constant temperature. Afterward, the specimens were putted out, washed with water and acetone, and dried. Subsequently, they weighted to compare with previous weight.

We did same procedure to do experiment in the boiling nitric acid. However, the experiment was stopped after 48 hours to change the solution. This procedure was repeated five times. Then the grain size of all specimens (in both mediums), which were exposed in solution annealing heat treatment, were calculated according to ASTM E112 [12], and after the corrosion tests, the tensile test was carried out in accordance with ASTM E8 [13].

## Discussion

Figure 1 is shown the metallographic image of the sample. In comparison with ASTM A262 samples, it was clear that this specimen's microstructure belonged to 3th type of microstructures on which surrounded with thick layer of carbides which most of them are chromium carbide. So, the type B and C test from the mentioned standard could be done:

A) Results from method B (Iron Sulphate – boiling sulphuric acid solution): the corrosion rate of 15 specimens in millimetre per month was calculated. Figure 2 shows the variation of corrosion rate on time of solution annealing heat treatment. Regards the chart, the negative slope was high in the beginning of heat treatment, and sharply decreased by increasing of heat treatment time. Meanwhile, the corrosion rate reduced during the process. The noticeable point in the calculation according to method B of ASTM A262 was that after a specific annealing time, holding in 1100°C did not have any particular effect on the corrosion rate and just caused energy loss and a collapse in mechanical properties due to increase in grain size. The optimum time which caused lowest effects on mechanical properties was determined 25 minute.

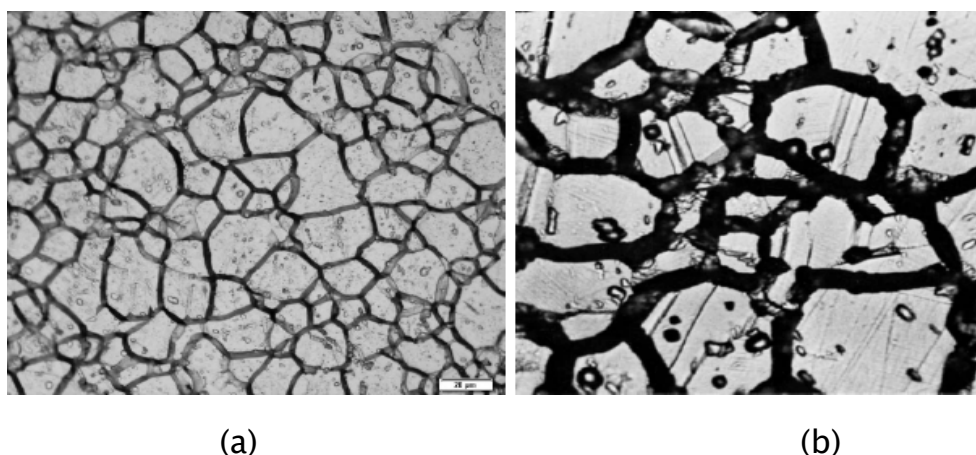


Fig 1.a) Type three of ASTM A262 sample. b) 304 grade stainless steel solution annealed in 1100°C (scale: 20  $\mu$ m).

B) Results from method C (boiling nitric acid): the corrosion rate of 15 specimens calculated in millimetre per month demonstrated in figure 2. Regarding to test results, the corrosion rate of the specimens in this medium far less than another medium. Yet, the decrease in corrosion rate was happened after 20 minute holding

in 1100°C. As a result, the calculation illustrated that the most portion of chromium carbides was solved after the mention time.

Figure 3 showed the chart of variation of tensile strength and the grain size of annelid specimens vs. annealing time. As can be seen from chart, at the beginning increasing in annealing time did not have significant effects on grain size and tensile strength. Whereas, grain size raised, and consequently the mechanical properties collapsed after 25 minute. Thus, although after 25 minute the corrosion rate dropped, the mechanical properties slumped, too.

## Conclusion

According to test results, stainless steel grade 304 corrosion properties improved by solution annealing heat treatment, but this improvement stopped after specific time. Furthermore, for this grade of steel with 2 millimetre thickness, the best heat treatment time is about 20 to 25 minute in 1100°C. Therefore, in the mentioned condition we could improve the corrosion resistance character accompany with the desired mechanical properties.

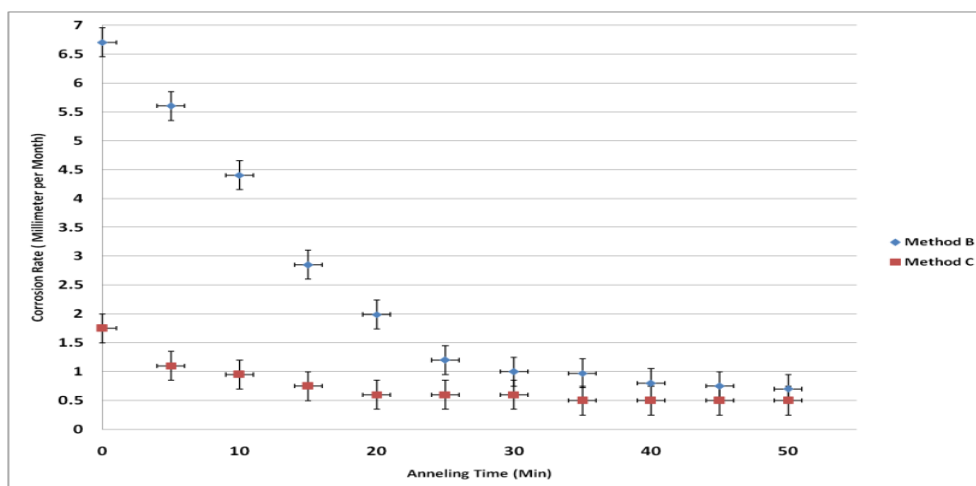


Fig 2. Chart of variation of corrosion rate in two methods vs. annealing time

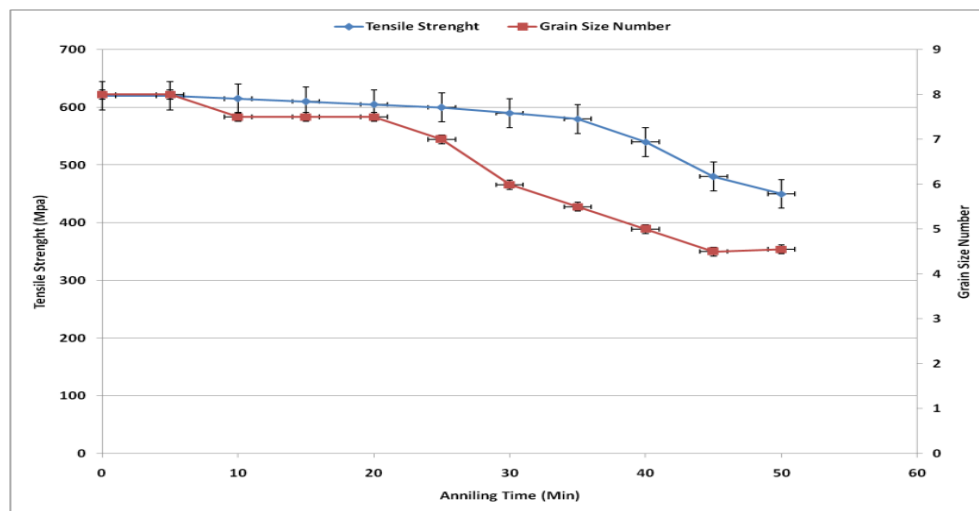


Fig 3. Chart of variation of grain size number and tensile strength vs. annealing time

## References

- [1] E.C.Bain, R.H.Abon, and J.J.B.Ruterford, *Trans. Am. Steel Treating Society*, 1933, Vol.21, pp.481–509.
- [2] M.A.Gaudett, and J.R.Scully, *Metallurgical and Materials Transactions A*, 1994, Vol.25A, pp.775–787.
- [3] ASM Handbook, Volume 4, Heat Treating, ASM, 1991.
- [4] G.H.Aydogdu, M.K.Aydinol, *Corrosion Science*, 2006, Vol.48, pp.3565–3583.
- [5] E.Almanza, L.E.Murr, *Journal of Materials Science*, 2000, Vol.35, pp.3181–3188.
- [6] C.J.Alvarez, E.Almanza, *Journal of Materials Science*, 2005, Vol.40, pp.2965–2969.
- [7] Y.Zhou, K.T.Aust, U.Erb, and G.Palumbo, *Scripta Materialia*, 2001, Vol.45, pp.49–54.
- [8] G.E.J.Talbot, and J.D.R.Talbot, *Corrosion Science and Technology*, 1998, CRC Press LLC.
- [9] S.S.M.Tavares, M.P.Cindra, *Journal of Materials Science*, 2003, Vol.38, pp.3527–3533.
- [10] P.Muraleedharan, H.S.Khatak, J.B.Gnonmoorthy, and A. Podriguez, *Metallurgical and Materials Transactions A*, 1985, Vol.16A, pp.285–289.
- [11] Standard test method for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels, ASTM A262, American Society for Testing Materials, Philadelphia, PA, 1988.
- [12] Standard Test Methods for Determining Average Grain Size, ASTM E112, American Society for Testing Materials, Philadelphia, PA, 2010.
- [13] Standard Test Methods for Tension Testing of Metallic Materials, ASTM E8, American Society for Testing Materials, Philadelphia, PA, 2011.