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Full-annealing and its effect on the Mechanical Properties of Alloy 304H Stainless Steel

Anthony Edozie Onwudili^{1*} and Sunday Chukwuka lweka²

¹Department of Mechanical Engineering, University of Benin, Benin, Nigeria. ²Department of Mechanical Engineering Technology, Delta State Polytechnic, Ozoro, Nigeria.

Authors' contributions

This work was carried out in collaboration among all authors. Author AEO designed the study, performed the statistical analysis, and wrote the first draft of the manuscript. Author SCI wrote the protocol, managed the analyses of the study, and the literature searches. All authors read and approved the final manuscript.

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ABSTRACT

Stainless steel is an alloy of steel which contains at least 10.5% chromium, less than 1.2% carbon, and other alloying elements. It is widely used in many industries globally and their properties are highly influenced by their microstructure, heat treatment or by plastic deformation. But due to hardness, poor wear, and corrosion resistance, leading to short service life, there is need to investigate the effect of annealing on the mechanical properties of alloy 304H stainless steel and how the mechanical properties can be improved with a view of improving its service life and optimizing engineering usage. Sixteen (16) samples of the alloy were used. Twelve (12) samples were annealed at three different temperatures of 950°C, 1000°C and 1050°C inside a muffle furnace. At each temperature four samples were heat-treated inside the muffle furnace for 30 minutes. The result showed that the yield strength decreased from un-annealed sample to annealed samples at 950°C with a value of 504.8MPa and increased at 1000°C with a value of 610MPa and a decrease of 323.8 MPa was obtained at 1050°C. Also, the ultimate tensile strength showed an increase from 950°C with a value of 826.3MPa to 1000°C with a value of 930MPa but there was a slight decrease at 1050°C for all samples. The ultimate tensile strength at 1000°C with a value of 930MPa was the highest in all the samples. The annealed samples at 1000°C had the

*Corresponding author: Email: christislove222@gmail.com, chukaiweka@yahoo.com;

highest percentage elongation of 13.57% which shows an increase in the ductility of the material. The hardness of the material decreased from 157.25 BHN at 950°C to 134.00BHN at 1000°C. An increase to 169.50BHN was however obtained at 1050°C. Thus, full-annealing of alloy 304H stainless steel at 1000°C increases in ductility as hardness decreases.

Keywords: Full-annealing; steel; microstructure; mechanical properties; heat treatment.

1. INTRODUCTION

Stainless steel is the "green material" par excellence and it can be recycled. It plays an important role in various industries, like energy, transportation, building, research, medicine, food and logistics. It has an excellent corrosion resistance and good ductility [1-2]. Austenitic stainless steels, including AISI 316L and 304, are widely used as structural materials in industries such as petrochemistry, transportation, ultra-supercritical power and nuclear power plants, mostly due to their high specific strength, ductility, fracture toughness and excellent corrosion resistance [3-7]. However, hydrogen embrittlement sensitization and formation of different carbides and sigma phase can also affect the mechanical properties. The properties of stainless steel are highly influenced by their microstructure, heat treatment or by plastic deformation. The various heat treatments are: Tempering, Quenching, Normalizing, Annealing etc.

In addition, stainless steels with further improved mechanical properties and corrosion resistance could be the next generation of metallic materials for the aerospace and some niche industries [3] [8-9]. Hence, the need to investigate the effect of full-annealing. Annealing is the heating to and holding at a suitable temperature above recrystallization followed by cooling at a suitable rate in a switch off furnace for such purposes as to induce softness and enhance cold work [10-11] And it's done as a final treatment or as an operation preceding other further treatments. It may be classified into two basic types, depending on phase transformation features: first-type annealing and second-type annealing.

Full-annealing, isothermal annealing and partial annealing are examples of second-type or second-order annealing because the end result of the process is essentially due to phase transformation that takes place; and it involves heating the metal to temperatures above the transformation temperatures, holding at that temperature and subsequently slow cool. The annealed metal has low hardness and strength, but very high ductility.

In isothermal annealing, process is usually applied to forgings and other blanks of small size. It is not suitable for heavy components as the resulting hardness and structure will be nonuniform across the section due to the difficulty of cooling them rapidly and uniformly to the isothermal temperature at which phase transformation occurs.

In partial annealing, the steel is only partly recrystallized. It reduces hardness and increases ductility [12]. Hence, the need for Full-annealing of 304H.

Full-annealing consists of heating to about 30 -50°C above the upper critical temperature for hypoeutectoid steels. The metal is held at this temperature for a specific time, followed by very slow cooling such as cooling in the furnace or in an insulated container. Their very slow cooling causes the steel to have a homogenous austenitic structure. (A hypoetectoid steel having a carbon content less than 0.8 percent. Austenite is a solid solution of carbon in face-centeredcubic iron, in which the carbon atoms occupy interstitial positions in the iron lattice). The purpose of full-annealing is to improve ductility, remove residual stresses completely, obtain better magnetic and electrical properties, and refine the grain structure. Full annealing is usually applied to rolled stock, forgings and castings. Hence, the need to study the effect of full-annealing on the mechanical properties of this alloy 304H stainless steel and how the mechanical properties can be improved with a view of improving service life and optimizing engineering usage. According to Tukur et al. [13], the highest hardness value was obtained at a temperature of 1090°C for solution annealed samples. Also, Ofuyepone et al. [14] reported that the strength and hardness of alloy that is solution annealed at 1045^oC was due to substitute solid solution strengthening through the dissolution of more intermetallic compounds and complex carbides. Thus, investigating the effect of the various annealing temperatures on the tensile strength of alloy 304H stainless steel, examine the effect of annealing temperatures on the hardness of alloy 304H stainless steel and determine how annealing affect the microstructure of alloy 304H stainless steel. And this will be achieved by conducting preparation sample test, full-annealing test, which involved the heating of samples to different temperatures of 950°C,1000°C and 1050°C, tensile test, metallography and optical micrographs, and also hardness test.

2. LITERATURE REWIEV

Stainless steel can be divided into martensitic, ferritic, and austenitic steels [10-11]. Steel is an alloy of iron and carbon. Stainless steels are steels which contains at least 10.5% chromium, less than 1.2% carbon, and other alloying elements. Corrosion resistance of Stainless steel and its mechanical properties can be enhanced more by adding other elements, like nickel, molybdenum, titanium, niobium, manganese, etc. When stainless steel come in contact with oxygen, a chromium oxide layer is formed on the surface of the material. This passive layer protects it and has the unique ability to repair itself as shown in Fig.1 [15].

2.1 General Properties of Stainless Steel

Stainless steel has the best fire resistance of all metallic materials, when used in structural applications, having a critical temperature above 800°C. Considering at least 10.5% of chromium

content, stainless steel is continuously protected by a passive layer of chromium oxide that forms naturally on the surface through the reaction of the chromium with oxygen from air or water. If the surface is scratched, it regenerates itself. This particularity gives stainless steel their corrosion resistance as shown in Fig.1. Its items are easy to clean, and they fully meet the requirements of decoration and cooking utensils that require frequent and effective washing. Its great variety of surface finishes makes stainless a unique and aesthetic material. When compared with other materials, stainless steel has strong mechanical properties at ambient temperatures. It is used in difficult metal forming modes (deep stamping, flat bending, extrusion, etc.) when it is combined with ductility, elasticity, and hardness, while offering resistance to heavy wear (friction, abrasion, impact, elasticity, etc.). In addition, it has good mechanical behaviour at both low and high temperatures. Also, it is the "green material" par excellence and can be recycled. Within the construction sector, its actual recovery rate is close to 100%. [16].

2.2 Grade 304H Stainless Steel (UNS S30409 ie BS 304S51)

Type 304H has a higher carbon content making the steel more suitable for use in applications where elevated temperatures are present unlike 304L. 304H is an austenitic chromium-nickel steel alloy and its increase in tensile and yield strength is due to greater carbon content in it. Masteel UK Ltd [16].



Fig. 1. Composition of stainless steel

2.2.1 Benefits of using grade 304H stainless steel include

- Higher carbon content gives the material greater heat resistant qualities
- Higher tensile yield strength
- Greater short-term and long-term creep strength

2.2.2 Applications of 304H stainless steel

304H stainless steel is commonly found in the oil refining, gas, and chemical industry and is used in industrial boilers, pressure vessels, heat exchangers, pipelines, and condensers. The material is also used throughout the power generation industry.

2.2.3 Chemical composition and mechanical properties of grade 304H stainless steel UNS S30409 ie B.S. 304S51

Table 1 and Table 2 show the chemical composition and mechanical properties of grade 304H stainless steel according to [16] [15] [17] and [18] respectively. 304S51 material is B.S. BS 1501-3 Steel for pressure purposes. Specification for corrosion- and heat-resisting steels: plates, sheet, and strip.

3. MATERIALS AND METHODS

3.1 Materials

The material used for this research work is stainless steel 304H steel as supplied in a cylindrical stainless steel rod of diameter 16mm and 2m long.

3.1.1 Equipment

The equipment used for this research work are shown in the Appendix and they include: muffle furnace, universal tensile machine, optical metallurgical microscope

3.2 Methods

3.2.1 Sample preparation

The stainless steel 304H was annealed to various temperatures of 950°C, 1000°C and 1050°C and was later machined to size 11mm diameter and then cut with a lathe machine to various sizes. There were 16 samples, four of which are control samples and the remaining

twelve are shared into four each for the above temperatures and annealed.

3.2.2 Heat treatment

12 samples in total were annealed to required temperatures of 950°C, 1000°C and 1050°C inside a muffle furnace and held at that temperature respectively for 30minutes each in order to reduce the effect of sigma phase precipitating to form intergranular corrosion which reduces the mechanical properties

3.2.3 Tensile test

Tensile test was carried out at room temperature at a cross head speed of 10⁻⁵mls until a predetermined load was reached on the loaddisplacement curve. During the test, the tensile load and the extension of previously marked gauge length in the specimen was measured with the help of the load dial of the machine and extensometer respectively. The ends of the specimen were gripped in the machine and load was applied until failure occurred. Percentage elongation was also calculated.

3.2.4 Hardness test

The hardness test was carried out at the metallurgical training institute in Onitsha, Anambra state. It involved sawing; grinding, polishing and the final test were conducted on a brinell hardness testing machine.

3.3 Metallography (Microstructural Examination)

All samples were prepared for metallography and grounded using a set of abrasive emery papers. The 220, 320, 400, 600 grades were used in that order rotating it at 90° after each round of grounding and polishing was done using compound of alumina (Al₃O₃) in the first instance. The samples were then polished in the second instance on a nylon cloth-covered surface of a rotating polishing wheel using a diamond dust to produce a smooth and mirror-like surface, washed in water and dried with acetone. The sample were then etched with the Kalling's reagent (Cupine chloride) 12grams Hydrochloric acid (20ml) and Alcohol 225ml).

Furthermore, the reagent attacked the surface layer produced when polishing and other second precipitates produced from the optical microscope.

Table 1a. Chemical composition of grade UNS S30409 ie B.S. 304S51

UNS NO	Grade	С	Si	Mn	Р	S	Cr	Мо	Ni	Ν
S30409	304H	0.04/0.10	0.75	2.00	0.045	0.030	18/20	-	8/10.50	0.10

Table 1b. Mechanical properties of grade UNS S30409 ie B.S. 304S51

UNS NO	Grade	Proof Stress 0.2% (MPa)	Tensile Strength (MPa)	Elongation A5(%)		Hardness HB	Max HRB
							TIKB
S30409	304H	205	515	40	201		92
			0 11 1 1 1 1 1 1 1 1 	A1			

Source: Masteel UK Ltd (2009); [16]

Table 2a. Chemical composition of grade UNS S30409 ie B.S. 304S51

	Carbon	Silicon	Manganese	Phosphorus	Sulphur	Nickel	Chromium
304	0.08	0.75	2.00	0.045	0.03	8 - 10.50	18 - 12
304L	0.03	0.75	2.00	0.045	0.03	8 – 12	18 - 20
304H	0.04- 0.10	0.75	2.00	0.045	0.03	8 – 10.50	18 - 20

Source: (www.australwright.com.au/304-stainless-steel/)[15], (steelss.com/materials/Stainless-Steels/304S51_31_5593.html)[18]

Table 2b. Mechanical properties of grade B.S. 304S51 ie UNS S30409

Yield R _{p0.2} (MPa)	Tensile <i>R</i> _m (MPa)	Impact KV (J)	Elongation A (%)	Reduction in cross section on fracture Z (%)	As-Heat-Treated Condition	HBW
132 (≥)	572 (≥)	23	21	12	Solution and Aging, Annealing, Ausaging, Q+T,etc	112
		Courses: ounorallous not/a	radaa/atainlaaa ataala/21	EE02/2010E1 atool html [17]		

Source: superalloys.net/grades/stainless-steels/31/5593/304S51-steel.html [17]

4. RESULTS AND DISCUSSION

4.1 Results

The material was also tested at universal steel to determine the composition of stainless steel 304H grade which is of high carbon content according to Standard UNS S30409 and BS 304S51, because the carbon content classification ranges from a minimum of 0.04% to maximum of 0.1%. as shown in Table 3. Thus, it's of high corrosion resistance because of its high nickel content and very low manganese content as compared to Table 1a and 2a.

4.2 DISCUSSION

4.2.1 The effect of annealing on the tensile strength of alloy 304H stainless steel

From Fig. 2, it was observed that ultimate tensile strength increased from 826.3MPa to 930 MPa when temperature was increased from 950°C to 1000°C. It however decreased to 435MPa at 1050°C. The decrease at 1050°C is in line with the earlier finding by Tukur et al. [13], who studied the effect of heat treatment temperature on the mechanical properties of AISI 304 stainless steel, and stated that the decrease was due to the dissolution of carbide in chromium enriched region of the matrix. From Fig. 3, it was observed that the yield strength reduced in all the annealed sample at different temperatures.

From Fig. 4, it can be observed that percentage elongation of annealed samples increased from 950° C to 1000° C, and the increase has its peak at 1000° C with a value of 13.57% elongation, which was the highest elongation and decreased slightly at 1050° C with a value of 12.86%. Fig. 5, shows a comparison of yield strength and ultimate tensile strength samples. The sample at 1000° C had the highest tensile strength with a value of 930MPa among the annealed samples.

4.2.2 The effect of microstructure on the tensile properties of alloy 304H stainless steel

Plate 1 to 13 shows the microstructures of the as-received and the heat-treated sample. The as-received sample in Plate 1 shows a combination of ferrite and cementite. The annealed sample show finer pearlite microstructure. The finer pearlite structure is due to slow cooling from austenitic temperature. As the temperature drops, the structure of the pearlite becomes finer. The Plate 2 shows a fine pearlite precipitate dispersed in ferrite matrix, Plate 3 a combination a carbide in ferrite, Plate 4 carbide in pearlite, Plate 5 fine pealites in carbide, Plate 6 combination of carbides in ferrite, Plate 7 carbides entrapped in ferrite matrix, Plate 8 and 9 has carbide in ferrite matrix, Plate 10 carbides in ferrite matrix, Plate 11 fine pearlite dispersed in carbide matrix, Plate 12 and 13 has carbides dispersed in ferrite matrix.

	R _{p0.2} (MPa) 587 (≥)
R _m (MPa)	869 (≥)
AKV (J)	32
A (%)	21
Z (%)	44
Hardness	HBW 221
	Ocument standard some (medanisla (Otobialanda Otobialanda (Otobialanda (Otobialanda (Otobialanda (Otobialanda (

Table 2c. Mechanical properties of grade B.S. 304S51 ie UNS S30409

Source: steelss.com/materials/Stainless-Steels/304S51_31_5593.html [18] Note: B.S. standard material 304S51 belongs to the UK while UNS S30409 belongs to the US.

Clement	AISI 304 stainless steel material (% by weight)	
Carbon	0.10	_
Chromium	18.70	
Nickel	11.10	
Manganese	1.50	
Silicon	1.45	
Molybdenium	0.25	
Phosphorus	0.03	
Sulphur	0.002	
Iron	66.688	

Fable 3. Chemical composition	on (w%) of	AISI 304H	stainless	steel
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Fig. 2. Ultimate tensile strength versus temperature of annealed samples



Fig. 3. Yield strength versus temperature of annealed samples *The variation in strength as against as-received samples and anneal samples temperature.*

4.2.3 The effect annealing on the hardness properties of alloy 304H stainless

The hardness of the material showed a decrease at the temperature of 950° C to 1000° C with values 157.25BHN, 134BHN respectively but showed a slight increase at 1050° C with a value

of 169.50BHN Moreover the control has a hardness of 197.25BHN. This can be supported by Tukur et al. [13] who stated that the decrease in hardness was due to dissolution of the carbides in the chromium enriched region of the matrix.

Temperature(°C)	Percentage elongation(%) 1 st	Percentage elongation (%) 2 nd	Percentage elongation(%)3 rd	Percentage elongation(%) 4 th	Average(%)
Control	0	0	0	0	0
950	10.2	10.5	9.7	9.9	10.08
1000	13.65	12.802	13.90	13.94	13.57
1050	12.8	12.67	12.97	12.99	12.86

Table 4. Percentage elongation

Table 5. Ultimate tensile strength (UTS)

Temperature ([°] C)	Ultimate Tensile Strength 1 st (MPa)	Ultimate Tensile Strength2 nd (MPa)	Ultimate Tensile Strength3 rd (MPa)	Ultimate Tensile Strength 4 th (MPa)	Average(MPa)
Control	800	810	820	850	820
950	805	810	830	860	826.3
1000	900	930	940	950	930
1050	410	420	440	470	435

Table 6. Hardness test

Temperature(°C)	Hardness 1 st (BHN)	Hardness 2 nd (BHN)	Hardness 3 rd (BHN)	Hardness 4 th (BHN)	Average Hardness(BHN)
Control	197	198	197	197	197.25
950	184	185	129	131	157.25
1000	145	144	122	125	134.00
1050	218	219	119	122	169.50

Table 7. Yield strength

Temperature(°C)	Yield Strength 1 st (MPa)	Yield Strength 2 nd (MPa)	Yield Strength 3 rd (MPa)	Yield Strength 4 th (MPa)	Average(MPa)
Control	750	760	770	774	763.5
950	500	500	502	517	504.8
1000	600	610	605	625	610.0
1050	310	325	320	340	323.8

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Fig. 4. Percentage elongation versus temperature of annealed samples The variation in elongation of as received and annealed samples



Fig. 5. Ultimate tensile strength and Yield strength versus temperature of annealed samples

4.2.4 The effect of annealing on the ductility of alloy 304H stainless

Fig.3 show the percentage elongation which a measure of ductility. It is observed that the it

increased from 950° C to 1000° C with a value of 10.08% to 13.57% and the 13.57% at 1000° C was the highest This can be supported by tukur et al. [13] who stated that annealing increases the number of planes on annealed samples for

dislocation movement to occur.it also decreased to 12.86% at 1050^{0} C. This could be due to dislocations movement impeded by the

chromium carbides precipitated at the grain boundaries.



Fig. 6. Average hardness versus temperature of annealed samples This graph shows a decrease in hardness from 950° C to 1000° C, there is slight increase at 1050° C with as-

received or control having the highest hardness.



Plate 1. Control shows pearlite structure embedded in ferrite matrix at magnification of 400X



Plate 2. Sample annealed at 1050^oC at magnification of 400X and shows fine pearlite precipitate dispersed in ferrite matrix



Plate 3. Sample annealed at 1050°C (Shows Chromium Carbides in ferrite matrix due to slow cooling) at magnification of 400X



Plate 4. Sample annealed at 950°C (Carbide embedded in pearlite matrix) at magnification of 400X, pearlite (Light color) and Carbide (Darkened) carbides embedded in pearlite matrix



Plate 5. Sample annealed at 1000°C (Fine pealites in carbide matrix) at magnification 400X, Mix of coarse and fine precipitate particles within the ferrite matrix it shows a reduction in the inter particles distance



Plate 6. Sample of annealed at 1000°C

Shows a coarse precipitate particle dispersed within the ferrite matrix400X



Plate 7. Sample annealed at 950°C (Carbides entrapped in ferrite matrix at magnification of 400X)



Plate 8. Sample annealed at 950°C (Carbide embedded in ferrite matrix at magnification of 400X)



Plate 9. Sample annealed at 950°C (Carbide embedded in ferrite matrix at magnification of 400X)



Plate 10. Sample annealed at 1000°C (Carbide dispersed in ferrite matrix at magnification of 400X)



Plate 11. Sample annealed at 1000°C (Fine pearlite dispersed in carbide matrix at magnification of 400X)



Plate 12. Sample annealed at 1050°C (Carbide dispersed in ferrite matrix at magnification of 400X)



Plate 13. Sample annealed at 1050°C (Carbide dispersed in ferrite matrix at magnification of 400X)

5. CONCLUSIONS

The results obtained on the effect of fullannealing heat treatment on the mechanical properties of alloy 304H stainless steel. Indicate that samples annealed at 1000^oC had the highest tensile strength over other samples, annealing greatly increased the percentage elongation of alloy 304H, an evidence of highest ductility, good machinability and decrease in

hardness. And also, the un-annealed 304H samples was harder than the annealed samples. Generally, we can conclude that full-annealing of alloy 304H stainless steel at range 1000°C - 1050°C can appreciably increase in the yield strength and ultimate tensile strength while hardness value decreases at 1000°C but increase at 1050°C when compared with literature reports in Table 1b, 2b and 2c [15-18].

Hence, it can be used in power plant, breweries, food, beverages, and other heavy industries because of their tensile strength, ductility, durability and corrosion resistance to the betterment of mankind and its environment.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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APPENDIX

Results Table A1

	Area	Diamete r	Lengt h	Maximum Slope (Automati c Young's)	Modulus (Automati c)	Energy at Yield (Zero Slope)	Extensio n at Yield (Zero Slope)	Load at Yield (Zero Slope)	Tensile strain at Yield (Zero Slope)	Tensile extensio n at Yield (Zero Slope)	Tensile stress at Yield (Zero Slope)	Tensile stress at Maximum Tensile extensio n	Tensile strain at Maximum Tensile extensio n	Load at Maximum Tensile extensio n
1	(cm^2)	(mm)	(mm)	(N/mm)	(MPa)	(J)	(mm)	(N)	(mm/mm)	(mm)	(MPa)	(MPa)	(mm/mm)	(N)
	0.00196	0.5	42	3262.425	1704.066	3.14555	11	555.5375	0.2619	11	282.9329	444.3173	2.01708	872.415

Tensile Sample

Temperature (°C)	Yield Strength (MPa)	UTS(MPa)
Control	760.7	820
950	503.4	826.3
1000	610.0	930
1050	323.7	435

Temperature(°C)	Yield Strength (MPa)	Ultimate Tensile Strength(MPa)	Yield Strength (MPa)	Ultimate Tensile Strength(MPa)	Ultimate Tensile Strength(MPa)
Control	750	800	760	810	820
950	500	805	800	810	830
1000	600	900	620	930	940
1050	31	410	325	420	440

Tensile Strain

Temperature(°C)	Tensile Strain	Tensile Strain	Tensile Strain	Tensile Strain	Average
Control	0.94269	0.95002	0.96501	0.96501	1
950	1.63655	1.56	1.7	1.7	2
1000	2.01709	2.5	2.36	2.36	2
1050	1.71308	1.81002	1.85	1.85	2

Tensile Stress

Temperature(°C)	Tensile Stress 1 st (N/mm ²)	Tensile Stress 2 nd (N/mm ²)	Tensile Stress 3 rd (N/mm ²)	Tensile Stress 4 th (N/mm ²)	Average(N/mm ²)
Control	95.95	96.528	98.95	98.954	97.59
950	100.651	100.80	101.20	101.204	100.964
1000	445.31	465.316	475.36	475.364	465.3375
1050	84.299	85.338	86.858	86.38	85.72

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