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Characterization of Gas Nitrided 304L Stainless Steel

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Abstract: At present scenario more demand by defence, automobile and nuclear sector for more requirements of 304L stainless steel to enhance the properties of components like wear resistance, fatigue resistance, and surface hardness without degradation of corrosion resistance. Gas nitride process able to fulfil the various sector requirements. Hence by laboratory trial proves the various parameters (i.e. time & temperature) achieve the more satisfactory results. On 304 L stainless steel perform gas nitriding process at different temperature (400°C, 450°C, 500°C, 550°C) & atmospherically flow rate of nitrogen 280 m bar lit / Sec. at certain times (3 hr, 4 hr, 5 hr). Finally it is observed that at 400°C for 4 hrs, gas nitriding treatment gives maximum case depth of 19µm.

Keywords: ASS (Austenitic stainless steel), Gas Nitriding, EDS, SEM

I. INTRODUCTION

For better life and serviceability, many engineering components should be provided with improved properties in the surface adjacent region of the component, as this is usually the location of most severe loading. Thus a hard case on a soft core can induce pronouncedly improved mechanical performance. The outer hard case enhances the wear and fatigue resistance while the soft core “bulk” retains the toughness to bear the impact loads. Nitriding is the most widely employed thermochemical surface treatment whereby Nitrogen is introduced into the surface of the solid metal by holding the metal at moderate or high temperature to enhance the mechanical (fatigue and wear) and chemical (corrosion) properties of ferritic steel components but here it is performed on Austenitic Stainless Steel components for study purpose which obtained the positive results. At the usual nitriding temperatures (450°C-580°C; below the Ac1 temperature of steel) no phase transformations, such as austenite-ferrite and austenite-martensite, can occur in the bulk of the steel components. Due to the absence of these bulk phase transformations, which involve large volume changes, good dimensional tolerance can be achieved, upon nitriding, which avoids the need of post-machining and/or post-heat treatments.

This is a distinct advantage of the nitriding process over other surface heat treatments, like, carburizing and carbonitriding. Several nitriding methods are available such as gas nitriding, salt bath nitriding, plasma nitriding and laser nitriding. Among these nitriding methods, gaseous nitriding is the only method which can be thermodynamically controlled due to the possibility of precise control of the chemical potential of nitrogen in the gas atmosphere. Owing to their good corrosion resistance, mechanical strength, heat resistance and high formability, austenitic steels have found application in many branches of industry. In the initial state, these steels exhibit, however, low hardness and abrasive wear resistance. However, the modification of the surface of austenitic steels is, as a rule, difficult, as the dense and tight Cr₂O₃ oxide film formed on the steel surface prevents other elements from penetrating into the material.

II. INTRODUCTION TO STAINLESS STEEL

Steel which contains more than 12% Chromium will form a thin passive oxide film which consists high corrosion resistance called Stainless Steel. Stainless steel are high alloy steels and possess excellent corrosion and oxidation resistance. Due to this characteristics, these steels find numerous application in nuclear plants, power generating units, paper and pulp manufacturing plants, food processing unit, and petrochemical units. [1]

Stainless steels are mainly five types based on their microstructures.

- 1) Ferritic stainless steel
- 2) Austenitic stainless steel
- 3) Martensitic stainless steel
- 4) Duplex stainless steel
- 5) Precipitation harden stainless steel

III. AUSTENITIC STAINLESS STEEL

Apart from other Stainless steel grade ASS having Ni as main element. Hence, addition of Ni to stainless steels can increase their ability to passivate in sulphuric acid, significantly improve corrosion resistance in hydrochloric acid of all concentrations at room temperature, and increase corrosion resistance in aqueous and molten sodium hydroxide. Nickel also improves toughness, cold formability and resistance of welds to certain agents. [2][3] Progressive addition of Ni displace delta ferrite zero to higher temperatures and increase the amount of austenite formed at higher temperatures. The stability of the austenite phase is also increased by Ni so that at approximately 8% Ni in an austenitic structure persists at room temperature. The parents of the austenitic stainless steel class are 302 (0.15% c) and 304 (0.08% c) which have approximately 18% chromium and 8% nickel. Therefore, Austenitic stainless steel is commonly known as 18-8 stainless steel. Based on 302 and 304 steels, a series of 3xx austenitic stainless steels have been developed by adjusting the chromium and nickel content or by addition of small amounts of other alloying elements.

The most common austenitic stainless steels are

- The basic 18-8 stainless steels (17-19% Cr, 8-10% Ni) 302 and 304;
- The higher nickel content 305 (10-13% Ni) to reduce the rate of work hardening and improve deep drawing;
- The highly corrosion resistant molybdenum-bearing 316 (2-3% Mo) and 317 (3-4% Mo);
- The stabilized grade 321 (about 0.4% Ti);
- The extra low carbon (0.03% c) grades, 304L and 316L;
- The oxidation resistant grades, 308 (19-21% Cr, 10-12% Ni), 309 (22-24% Cr, 12-15%Ni), 310 (24-26% Cr, 19-22% Ni).

IV. REASONS FOR SELECTING ASS (AUSTENITIC STAINLESS STEEL) FOR NITRIDING

Austenitic stainless steel of the 304L series are the most difficult to nitride; nevertheless, types 301, 302, 303, 304, 308, 309, 316, 321, and 347 have been successfully nitrided. These nonmagnetic alloys cannot be hardened by heat treating; consequently, core material remains relatively soft, and the nitrided surface is limited as to the loads it can support. This is equally true of the non-hardenable ferritic stainless steels. Alloys in this group that have been satisfactorily nitrided include types 430 and 446. With proper prior treatment, these alloys are somewhat easier to nitride than the 300 series alloys. So, it is a known fact that ASS. is difficult to Case harden as it contains Cr₂O₃ film which is generally hardtop remove. This experiment was generally based on research work for obtaining a thin case on the surface of 304L S.S. by varying the Time & Temperature. ASS. has better wear resistance it made to increase by Nitriding without degradation of other mechanical properties. The grain refinement of ASS. is also more difficult because of its single phase system. Also 304L ASS. is susceptible to sensitization which is carried out when > 0.03% C is there for these reason carburizing is not carried out on it so mainly nitriding process is carried out for surface hardening but, Due to certain reasons specified above, it was a challenging role to carryout Nitriding on 304L Stainless Steel.

V. WHY ONLY 304L GRADE FOR NITRIDING?

Type 304L stainless steel, also known as ASTM/AISI/SAE 304L, UNS S30403, A2 stainless steel or EN 1.4307, is the most versatile and widely used steel available in the market. It is “all purpose” grade characterized by good corrosion resistance, excellent formability and weldability. Additionally it is among the cheapest grades of stainless steels, making it the favourite choice of industry. It is particularly suitable for large structures such as application where localized heat resistance and wear resistance are require, in nuclear plant pipe lines where high temperature and erosion chances are more, pharmaceutical industry. as well as for exhaust systems within automotive industry. As other ASS, 304L is non-magnetic, even though some δ-ferrite (0-5%) is commonly found in the form of stringers. Controversial results have been reported regarding the effects of δ-ferrite, including resistance to stress-corrosion cracking and tendency to cracking during cold work. Applying Angel’s equation, it infers that deformation induced martensite can form at room temperature in 304L. This was experimentally demonstrated by several researchers in a number of experiments including uniaxial tensile tests and cold working. In our studies we also observed the formation of deformation induced martensite due to mechanical polishing through XRD and EBSD measurements.

VI. DESCRIPTION ABOUT 304L GRADE

Composition of 304L S.S.		Properties of 304L S.S.		Application of 304L S.S.
Components	Wt.%	Density	8 gm/cc	In chemical plant In medical device In building construction Interior decoration Kitchen furniture In windows frame
C	0.03max.	Melting point	1450 °c	
Cr	18-20	Hardness	158 BHN	
Mn	2 max	Tensile strength	564 MPA	
Ni	8-12	Yield strength	210 MPA	
P	0.045 max	Elongation %	58 %	
S	0.03 max	Modulus of elasticity	200 GPa	
Si	1.0 max	Charpy impact	216 j	

VII. EFFECT OF NITROGEN & IRON-N₂ PHASE DIAGRAM

Iron-nitrogen equilibrium diagram can be used to study the nitriding process. At the commonly used nitriding temperature (below 590°C), nitrogen dissolves in α-iron up to only 0.1% (called nitrogenous ferrite).

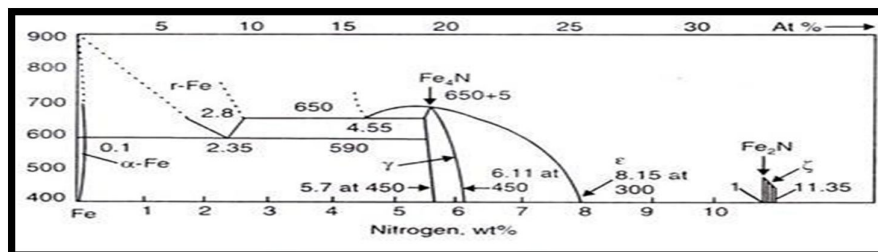


Fig. 1 Iron – Nitrogen phase diagram.

When the nitrogen dissolved in a-iron exceeds 0.1%, next phase stable at the temperature, i.e. γ' - nitride (a solid solution) is formed. When nitrogen content exceeds about 6%, ϵ -nitride (a solid solution) is formed. If the temperature of nitriding is below 500°C, further increase of nitrogen results in the formation of ζ -nitride which has nitrogen content of about 11%. Normally, temperatures are higher, and thus the nitrided layer has on the surface, ϵ -nitride, richest in nitrogen, and somewhat diffusing-in nitrogen during nitriding and internal precipitation of their nitrides takes place resulting in high surface hardness. Inside the steel, it has γ' - and then α -ferrite. Below Table given these intermediate phases with chemical formula. Thus, after cooling, the phases in nitrided case are in the order from surface towards the case:

Thus, plain carbon steels which don't contain any strong nitride forming elements, when nitrided in an atmosphere of high nitrogen activity, the iron nitrides are formed. The iron nitride case thus formed is generally brittle and does not have high hardness. But steels containing strong nitride forming elements like Al, Mo, Cr, Ti, V etc. interact with diffusing-in nitrogen during nitriding and internal precipitation of their nitrides takes place resulting in high surface hardness.

Table.1 Details about various phases present in Fe-N phase diagram.

Phase	Composition	Wt. % At % of N	Interstitial atop per 100 Fe atom	Bravais lattice
Ferrite (α)	Fe	0.10	-	B.C.C
Austenite (γ)	Fe	2.8	12.4	F.C.C
Martensite (α')	Fe	2.6	11.1	Body Central Tetragonal
Gamma (γ)	Fe ₂ N	5.9	25	Cubic
Eta (ϵ)	Fe ₂ N ₂	4.5-11	22-49.3	Hexagonal
Zeta (ζ)	Fe ₂ N	11.4	50	Orthorhombic

VIII. EXPERIMENT WORK

The 304L SS samples were Gas Nitrided at different temperature 400°C, 450°C, 500°C, 550°C for different time 3hr, 4hr, 5hr.

A. Gas Nitriding Process

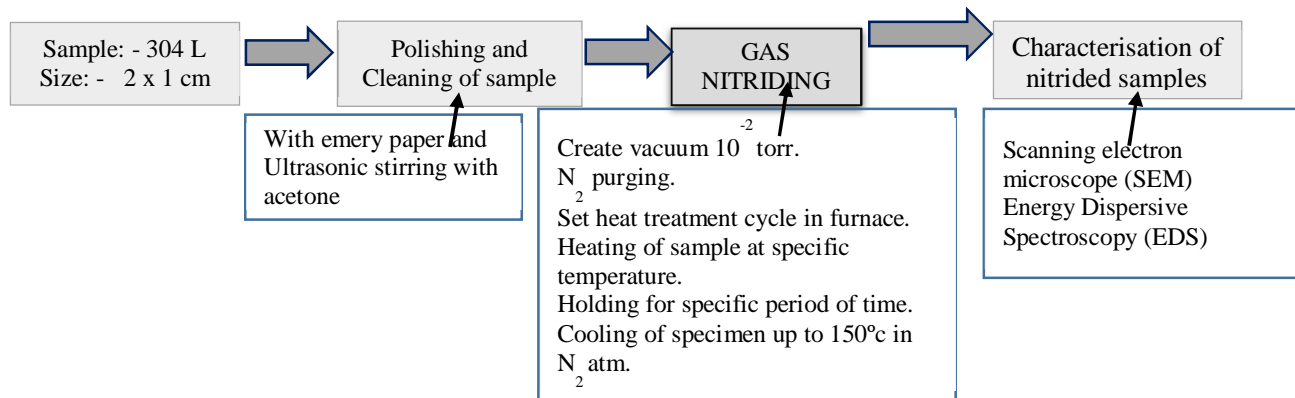


Fig. 2 shows the flowchart for gas nitriding process of 304L Austenitic stainless steel.

B. 304L Sample Preparation for Gas Nitriding

1) Sample Details :-

Sample grade : - 304L ASS
 Size of sample : - 2 x 1 cm
 Fabrication technique : - Rolling (Plate)
 Composition of specimen: - C-0.03%, Cr-18-20%,
 Ni-8-12%, P-0.045%,
 Mn-2%, Si-0.75%,
 S-0.03%, Fe-Balance



Fig. 3 Initial Condition of Sample.

C. Specimen Preparation for Nitriding

1) *Grinding and Polishing:* GRINDING removes saw marks and levels and cleans the specimen surface. Polishing removes the artifacts of grinding but very little stock. Grinding uses fixed abrasives—the abrasive particles are bonded to the paper or platen—for fast stock removal. Polishing uses free abrasives on a cloth; that is, the abrasive particles are suspended in a lubricant and can roll or slide across the cloth and specimen. The term lapping to mean grinding or coarse polishing with an abrasive slurry against a hard metal platen. Grinding is done by manually or mechanically but manual grinding allows better control of grinding depth than automatic grinding. For improve surface smoothness first rough grinding is carried out then

fine grinding i.e 1-4 emery no. After the finest grinding step and subsequent cleaning, manually polish the specimen on napless polishing cloths loaded with lubricant and Alumina paste. [15]

Fig. 4 polishing and grinding machine.



Fig. 5 Ultrasonic cleaning machine.



2) *Ultrasonic Cleaning*: Ultrasonic cleaning is the rapid and complete removal of contaminants from objects which are immersed in a tank of liquid that is flooded with high frequency sound waves. These non-audible sound waves create a gentle scrubbing action within the fluid, removing contaminants from all surface areas into contact with. The process is brought about by high frequency electrical energy that is converted by a transducer into high frequency sound waves – ultrasonic energy. Ultrasonic energy enters the liquid within the tank or bath, this causes the rapid formation and collapse of minute bubbles; a phenomenon known as cavitation. These bubbles travel at high speed within the tank, causing them to implode against the surface of any immersed object with an enormous release of energy. As the bubbles implode and cavitation occurs, the cleaning solution rushes into the gap left behind by the bubbles, gently lifting any contaminants, dirt and tarnishing from both the surface and innermost recesses of intricately shaped parts. [4] [5] But in our case the cleaning is carried out with water and acetone where the samples are kept in the beaker consisting samples completely dipped in acetone. The beaker is then kept in the cleaning chamber which is filled with water and the arrangement is such that water and acetone doesn't come in contact with each other. Required time for cleaning set in the programmer. (Generally 15 – 20 min in our case).

D. Gas Nitriding Furnace

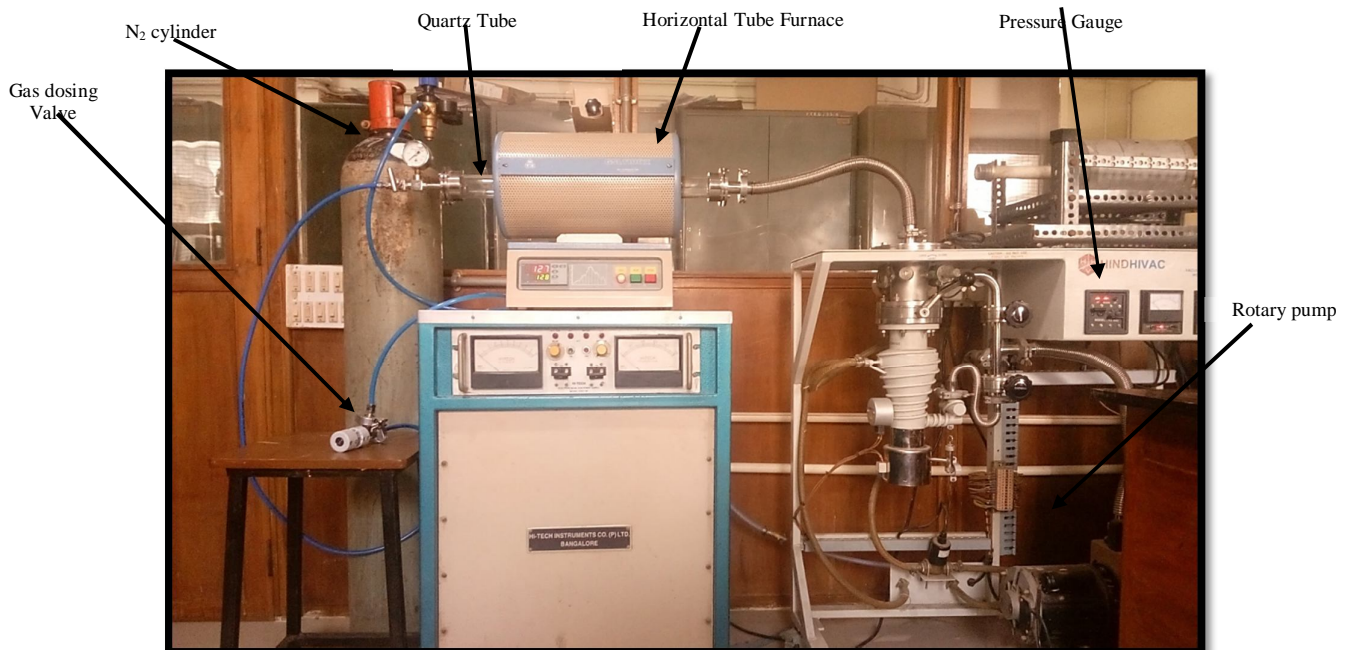


Fig. 6 Experimental setup of nitriding process

1) Procedure for Gas Nitriding:

- a) Clean the furnace using Acetone to remove dust particle from the furnace.
- b) A Quartz boat was used as a sample holder. Samples were placed in Quartz boat and treated in the Hot zone of the furnace.
- c) Both the ends of furnace are closed. At one end the furnace was connected to the vacuum system and at the other end it is connected to Gas Dosing Valve to Nitrogen cylinder
Now the set-up is ready for Gas Nitriding process.
- d) Initially using the Rotary pump, a vacuum of 10^{-3} torr is connected inside the furnace.

- e) Nitrogen Gas was then purged through gas dosing valve and flow rate is controlled such that pressure 1mbar is created inside the furnace.
- f) The furnace is switched on and the experiment was carried out as per the program set in PID display.
- g) Table 2. Gives the detailed parameter of Gas Nitrided experiments.
- h) Once the program is over, the samples are cooled in furnace in Nitrogen atmosphere upto 150°C.
- i) The samples are removed next day for characterization (SEM, EDS).

IX. RESULTS & DISCUSSION

Table. 2 Details of the Parameters in all Experiments.

Sample no.	Base pressure (mbar)	Time to reach at temp. (min)	Temperature (in °c)	Heating atm.	Holding time (in min)
S-1	0.02	40	400	N ₂	240
S-2	0.02	40	400	N ₂	300
S-3	0.02	45	450	N ₂	180
S-4	0.02	45	450	N ₂	240
S-5	0.02	45	450	N ₂	300
S-6	0.02	50	500	N ₂	180
S-7	0.02	55	550	N ₂	240

A. Heat Treatment Cycles of Nitrided Samples

1) Sample No. 1 (S-1):

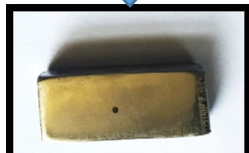
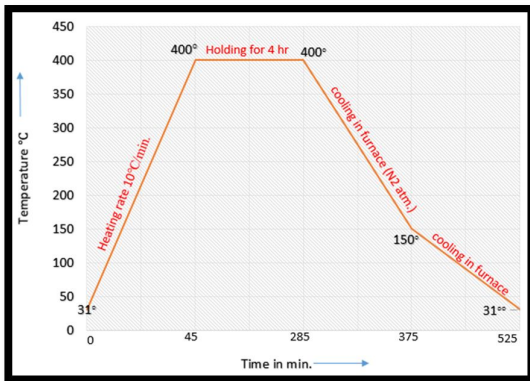


Fig.: 7 Heat treatment cycle of sample no. 1 and appearance of nitrided sample.

2) Sample No. 2 (S-2):

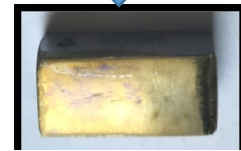
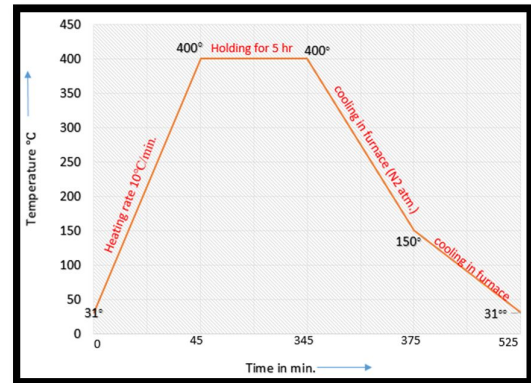


Fig.8 Heat treatment cycle of sample no. 2 and appearance of nitrided sample.

3) Sample No. 3 (S-3):

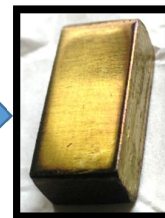
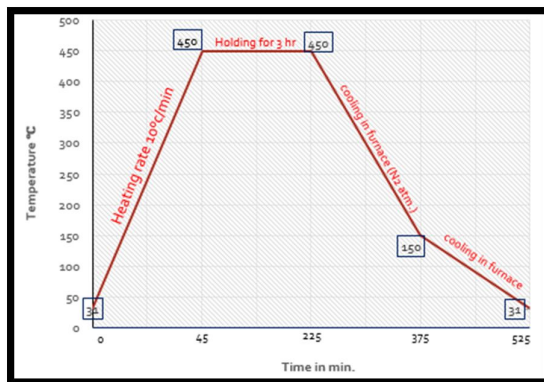


Fig. 9 Heat treatment cycle of sample no. 3 and appearance of nitrided sample.

4) Sample No. 4 (S-4) :-

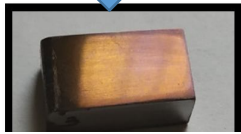
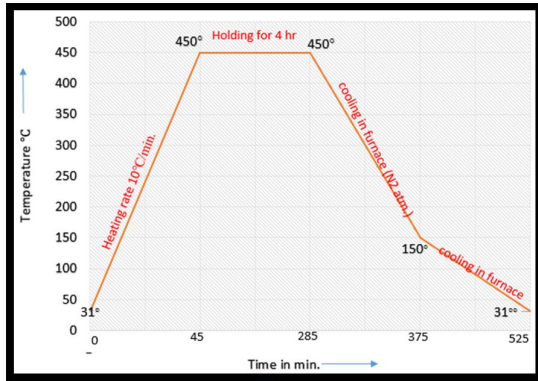


Fig. 10 Heat treatment cycle of sample no. 4 and appearance of nitrided sample.

5) Sample No. (S-5) :-

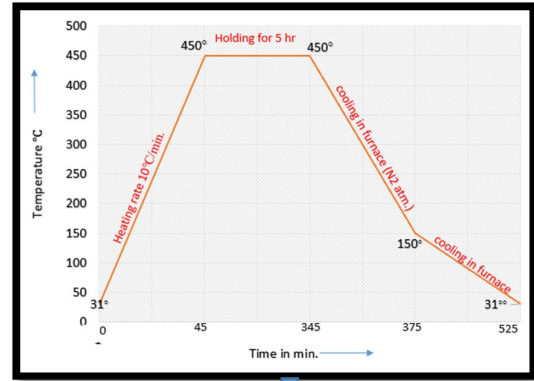


Fig. 11 Heat treatment cycle of sample no. 5 and appearance of nitrided sample.

6) Sample No. 6 (S-4) :

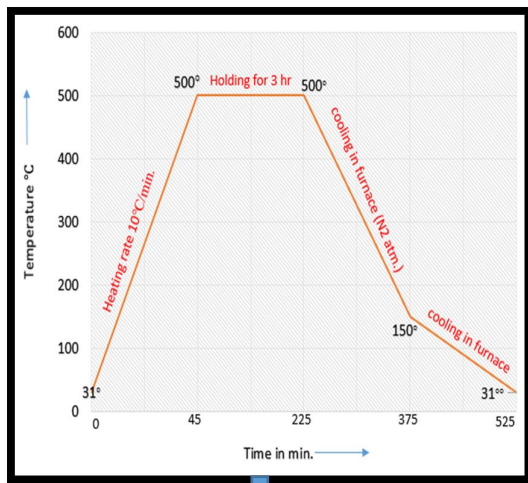


Fig. 12 Heat treatment cycle of sample no. 4 and appearance of nitrided sample.

7) Sample No. 7 (S-7) :

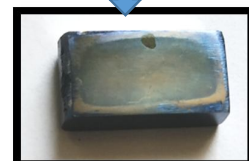
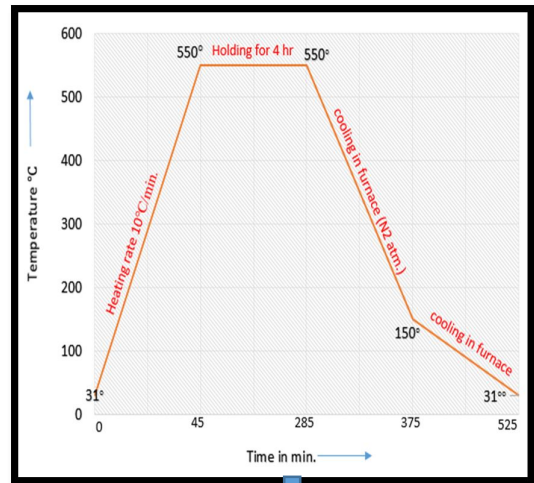


Fig. 13 Heat treatment cycle of sample no. 4 and appearance of nitrided sample.

B. Scanning Electron Microscope and EDS Results

1) *Sample No.1 (S-1)*: Fig. indicates the SEM micro photographs of Gas Nitrided sample S1 treated at 400°C for 240min. It is observed that two continuous thin layers are formed with an average thickness of 17µm and 11µm on the surface of the specimen.

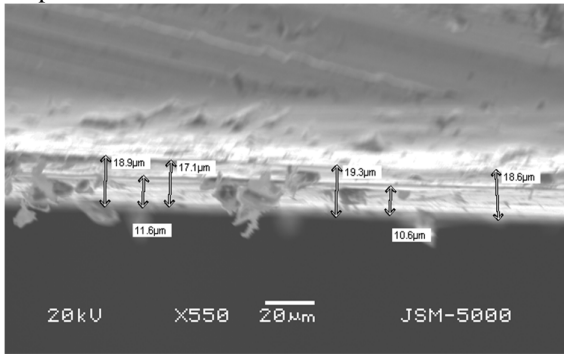


Fig.: 14 SEM analysis of Gas nitrided sample at 400°C for 240min at 550x.

Sample no :- S 1 Base pressure :- 0.02 mbar
 Nitriding temp. :- 400°C Magnification :- 550X
 Nitriding time :- 240 min

Table: 3 (S 1) composition of nitrided sample surface.

Element	Weight%	Atomic%
N K	2.06	6.97
O K	3.88	11.49
Si K	0.66	1.12
S K	0.73	1.08
Cr K	17.08	15.57
Mn K	1.81	1.56
Fe K	63.56	53.96
Ni K	10.21	8.25
Totals	100.00	

The Table shows the EDS analysis of the sample S1 Nitrided at 400°C for 240min. the micrograph shows the region where EDS is carried out. The Nitrogen content in the gas nitrided sample is 2.06% along with the presence of Cr 17.08 wt% and Fe 63.56 wt%.

2) *Sample No.2 (S-2)*: Fig. indicates the SEM micro photographs of Gas Nitrided sample S1 treated at 400°C for 300min. It is observed that a continuous thin layer is formed with an average thickness of 14-15µm on the surface of the specimen.

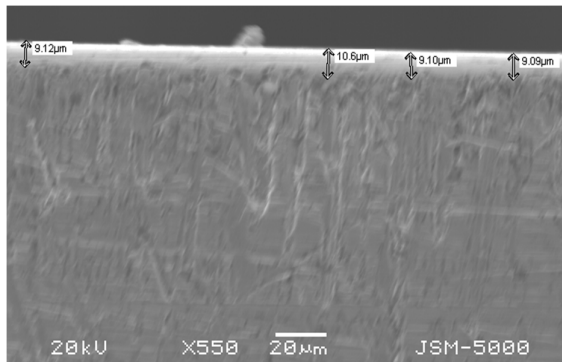


Fig.: 15 SEM analysis of Gas Nitrided sample at 400°C for 300min at 550x.

Sample no :- S 2 Base pressure :- 0.02 mbar
 Nitriding temp. :- 400°C Magnification :- 550X
 Nitriding time :- 300 min

Table: 4 (S 2) composition of nitrided sample surface.

Element	Weight%	Atomic%
Cr K	49.32	70.67
N K	3.40	4.18
O K	10.77	11.58
Na K	2.11	1.58
Mg K	0.37	0.26
Al K	0.38	0.24
Si K	0.85	0.52
S K	0.58	0.31
Cl K	2.43	1.18
K K	0.77	0.34
Ca K	0.68	0.29
Cr K	6.10	2.02
Mn K	0.57	0.18
Fe K	19.44	5.99
Ni K	2.24	0.66
Totals	100.00	

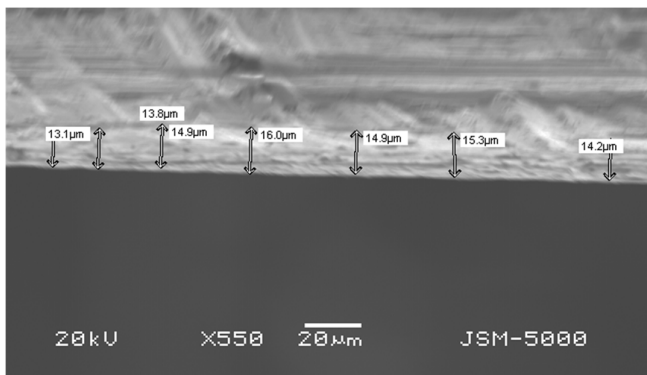
The Table shows the EDS analysis of the sample S1 Nitrided at 400°C for 300min. the micrograph shows the region where EDS is carried out. The Nitrogen content in the gas nitrided sample is 3.40% along with the presence of Cr 6.10 wt% and Fe 19.44 wt%

3) *Sample No.4 (S-4)*: Fig. indicates the SEM micro photographs of Gas Nitrided sample S1 treated at 450°C for 240min. It is observed that a very thin continuous layer formed with an average thickness of 9µm the surface of the specimen.

The Table shows the EDS analysis of the sample S1 Nitrided at 450°C for 240 min. the micrograph shows the region where EDS is carried out. Nitrogen was not found in this gas nitrided sample. The presence of Cr 16.25 wt% and Fe 59.60 wt% can be seen.

Sample no :- S 4 Base pressure :- 0.02 mbar
 Nitriding temp. :- 450°C Magnification :- 550X
 Nitriding time :- 240 min

Table: 5 (S 4) composition of nitrided sample surface.



Element	Weight%	Atomic%
C K	2.55	9.61
N K	0.16	0.51
O K	4.66	13.22
Si K	0.88	1.42
Cr K	17.35	15.13
Mn K	1.26	1.04
Fe K	65.03	52.81
Ni K	8.11	6.26
Totals	100.00	

Fig.: 16 SEM analysis of Gas nitrided sample at 450°C for 240min at 550x.

X. DISCUSSION AND CONCLUSION

304 L.S.S. samples are gas nitrided using nitrogen gas at 1 mbar pressure for 4 different temperature and 4 different time. The SEM and EDS analysis indicates the formation of nitrided layer on 304 L.S.S.

- 1) S1 400°C for 240 min N₂ % is 2.06 %.
- 2) S2 400°C for 300 min N₂ % is 3.40 %.
- 3) S4 450°C for 240 min N₂ % is 0.16 %.
- 4) S7 550°C for 240 min N₂ % is 0.48 %.

The 304 L sample gas nitrided at 400°C temperature and 240 min time shows max. thickness of 19 µm.

XI. ACKNOWLEDGMENT

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