Influence of aisi 304 austenitic stainless steel by aqueous soluted nitriding and gas nitriding

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Abstract. AISI 304 austenitic stainless steel has great corrosion resistance,but it lacks in hardness,strength and wear resistance.So in order to improve these various hardening techniques are followed.In the present research salt bath nitriding and gas nitriding were carried out to improve hardness and wear resistance.Three samples are treated with salt bath nitriding and the other three samples are treated by gas nitriding and the one sample is kept aside to compare the results.The samples were wear tested on pin on disc apparatus after the heat treatment.Then the samples are subjected to Scanning electron microscope test and then the results were compared

1 Introduction:

Stainless steel is a contemporary material that, due to its resistance to corrosion, is referred to by this general term for different grades of steel.Because it is less likely to discolor, requires little maintenance, and is recyclable.It is described as an alloy of ferrous metals with at least 10% chromium. The bulk of steel alloys are made of iron, which has a carbon percentage that ranges from 0.02% to 1.7% by weight and varies depending on the grade.[1-2]

Austenitic Stainless steel have become the extensively accepted. The austenitic stainless steel microstructure is the result of the expansion of nickel, nitrogen, manganese. At substantially higher temperatures, it has a structure that is identical to that of traditional steels[3-4]. These steels' distinctive blend of formability, weldability is attributed to their structure. The addition of nitrogen, chromium, molybdenum can enhance erosion protection. These properties cannot be cured with heat treatment method, but it has advantage that they can work cured to a high level of quality while maintaining a valuable level of flexibility and strength[5-6]. Austenitic stainless steels with chromium and nickel are included in the 300 series. Alloys made of chromium, nickel, and manganese are included in the 200 series. There are many different compositions and qualities of austenitic

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stainless steels, yet they all share a lot of characteristics. Although they can be cold worked, they cannot be heated. All of them lack magnetic properties when annealed, while some may acquire some magnetic properties after being cold worked. They have excellent cold working strength, remarkable moldability, and outstanding corrosion resistance[7-8].

Improvements in austenitic stainless steels have been made during the 1980s. Low temperature applications, such those found in nuclear power plants, petrochemical operations, and food processing facilities, commonly make use of these materials. Unfortunately, there are significant limitations to this material's utilization on surfaces because of its poor resistance to wear. Using standard nitriding methods, nitrogen was added for wear resistance.

The wear and tear of machine parts or equipment, which results in financial losses for society, is one of the most prevalent issues in today's industry. Downtime and replacement cost expenses are also included in financial depreciation losses. Wear is identified as the source of abrasive wear[9-10]. Abrasives are the primary source of most industrial wear issues. Thus, when researching the literature, it was noted that the wear of agricultural equipment and the mill, as well as many of the factors responsible for abrasive wear, are the main problem. So there are a number of variables with this problem that are very economical to deal with these problems. Wear has been called the miracle of design to evacuate material from one surface, and sometimes from both surfaces in strong contact, due to the mixing of sliding and movement on the other.

2.Experimental Procedure

2.1 Specimen preparation:

AISI 304 stainless steel was cut into small cylindrical specimens of 40mm long and 10mm diameter using lathe operation. The filleting is done to the specimen. Then the composition test has been performed.After the composition test materials are heat treated.Before being heat treated samples were cleaned using SiC paper. Samples were quenched and tempered at 980°C and 650°C, respectively, to increase their hardness and toughness. While the other three samples underwent gas nitriding, the first three samples underwent salt bath nitriding. The samples were nitrided at 570°C for a variety of durations (60, 120, and 180 minutes), allowing nitrogen to infiltrate the steel surface before cooling in air. This method allows nitrides to enter the inside of the steel surface. The pieces are submerged in a liquid shower during the nitriding salts process, and the nitride salts diffuse through the steel surface to create Fe3N links.The entire procedure was carried out in a furnace using sodium nitride salts and potassium nitrate (KNO3) , which were heated to 560-570 °C (Na2NO3).
To enhance the hardness and toughness samples were subjected to Quenching and tempering at 980°C and 650°C respectively. Three samples went through the salt bath nitriding procedure, while the other three went through the gas nitriding process.The samples are nitrided in aqueous soluted nitriding oven at 570 degree centigrade for time variables 60 minutes, 120 minutes, 180 minutes respectively in furnace.Gas nitriding was done at temperature from 450-500° Centigrade. During nitriding procedure, all samples are placed in an NH3 gas and alumina crucible is passed through the tube with a flow rate of 90 ° C –100 ml/min. After cooling to room temperature, the specimens were taken out from furnace for 6 hours, 12 hours, and 18 hours, respectively.
Gas nitriding was performed at the temperature from 450-500° Centigrade. During nitriding procedure, all samples are immersed in an NH3 gas and an alumina crucible is passed through a tube with a flow rate of 90 ° C -100 ml/ min. Gases, nitrogen and hydrogen, are separated throughout the process, and the nitrogen diffuses over the steel surface. The

complete procedure was carried out in a vacuum at 570° C. Despite fact that gas nitriding takes a long time, the end result is a large body depth and high hardness. Gas nitriding can provide strong corrosion resistance of steel due to its simple operation, high stability, and more efficient method of generating the CrN phase.The nitriding procedure was then carried out in the plant at 570°C. The specimens are removed from furnace after cooling to room temperature for six hours, twelve hours, and eighteen hours, respectively. Then after the heat treatment all samples were subjected to wear tests with normal loads of 20N and 30N on pin on disc machine.

3. Results and Discussions:

Table 1: Readings of wear test at 20N load at 600rpm

Samples	**Weigh before test(grams)**	**Weigh after test (grams)**	**Weigh-Loss (grams)**	**volume wear loss in mm^3**
Untreated sample	38.456	38.394	0.062	7.812
SBN 1 (60 min)	38.574	38.528	0.046	5.75
SBN2(120 min)	38.586	38.552	0.034	4.32
SBN3 (180 min)	38.609	38.583	0.026	3.25
GN1 (6 hrs)	38.586	38.562	0.023	2.91
GN2 (12 hrs)	38.608	38.587	0.0201	2.52
GN3 (18 hrs)	38.620	38.605	0.0145	1.62

Table 2 : Readings of wear test at 30N load at 600rpm

Samples	**Weigh before test(grams)**	**Weigh after test (grams)**	**Weigh-Loss (grams)**	**volume wear loss in mm^3**
Untreated sample	38.392	38.341	0.0504	6.312
SBN 1 (60 min)	38.451	38.408	0.0425	5.314
SBN2(120 min)	38.482	38.447	0.0340	4.26
SBN3 (180 min)	38.526	38.497	0.0289	3.62
GN1 (6 hrs)	38.436	38.409	0.0265	3.32
GN2 (12 hrs)	38.418	38.396	0.0220	2.76
GN3 (18 hrs)	38.582	38.568	0.0138	1.73

Before and after surface treatment, the surface characcteristics , microstructure of the AISI 304 SS were studied using a scanning electron microscope

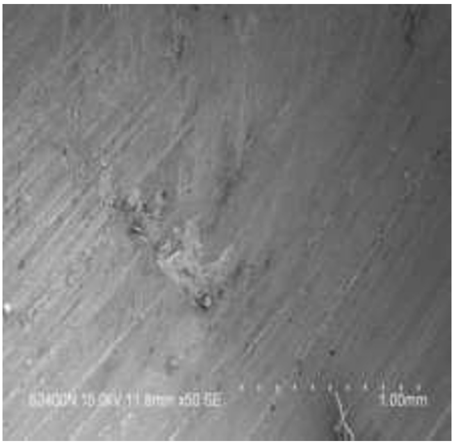


Fig 1: Untreated sample

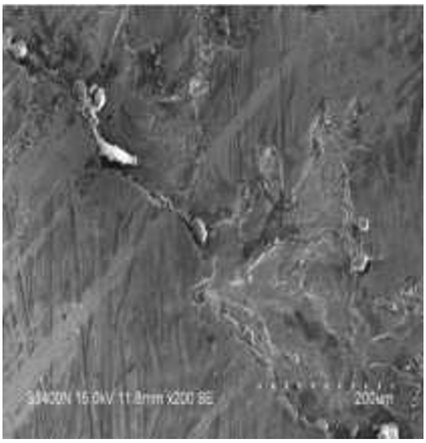
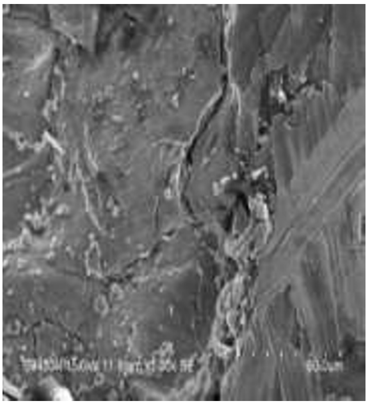


Fig 2: SEM image of SBN1 after pin on disc at 30N load

The above SEM image i.e. Fig 1 shows that the loss of material is more for the untreated sample due to strong abrasion, adhesion,plastic deformation.

The findings presented in Figure 2 indicate that the wear resistance of the substrate is impacted by the sparsely formed varieties of nitrides when a small amount of nitrogen diffuses below the compound zone. Conversely, when a larger amount of nitrogen diffuses below the compound zone, the wear resistance is restored and enhanced by the formation of more nitrides. There was less material peeloff in the treated sample compared to the untreated sample.



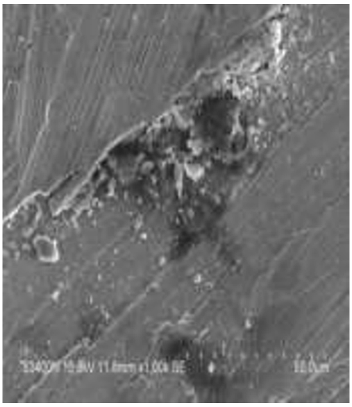
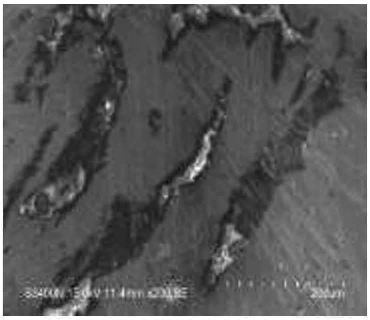


Fig 3 : SEM image of SBN2 after pin on disc at 30N load

Fig 4: SEM image of SBN3 after pin on disc at 30 N load

Figure 3 illustrates how the amount of nitrogen on the surface rises with nitriding time. The result of these processes is an expanded austenite (c-layer) known as the S phase layer. Austenite was reduced to a CrN layer in the nitrided latter phases. There is equal nitrogen dispersion and less material peel as the nitriding duration rises.As the nitriding time rises, the wear loss diminishes.

It can be seen from fig. 4 that there was very little material flaking off the specimen. The surface was entirely covered with chromium nitride (CrN) layers, which gave rise to a variety of intermediate layers such Roaldite, siderazot, and others. The specimen's hardness has increased the material's resistance to wear and extended life.



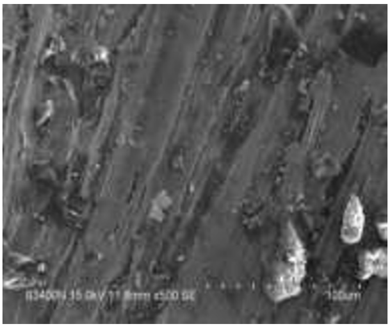


Fig 5 : SEM image of GN1 after pin on disc on 30N load

Fig 6: SEM image of GN2 after pin on disc at 30N load

From fig5 the formation of expanded layers was observed at low temperatures, indicating that gas nitriding is a useful surface engineering technique for improving austenitic stainless steel wear resistance by forming expanded austenite. When compared to an untreated sample, the material's peel was shown to be extremely low. Chromium nitride (CrN), which is assumed to be crucial in enhancing wear resistance, was accumulated on specimens as a result of the material sputtering during the diffusion process.

Based on figure 6 the researchers provide new understandings of the nitriding procedures based on a planned deposition layer that coincides with the grain orientation of polycrystalline specimens. Wear resistance was greatly increased by adding nitrogen to surface layer at temperatures as high as 550°C. Comparing the specimen that was nitrided for six hours with the specimen that was left untreated, it was discovered that the wear loss was minimal.

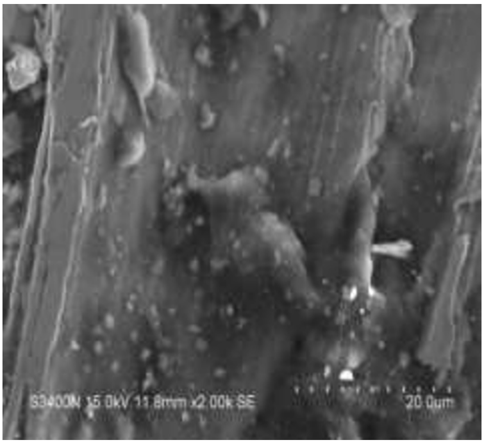
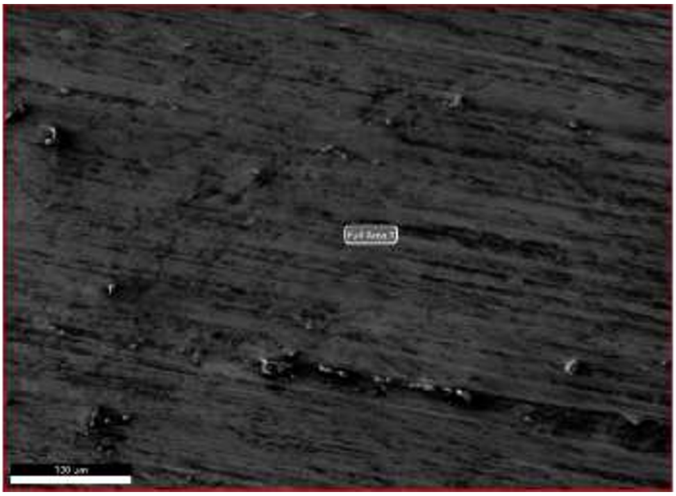


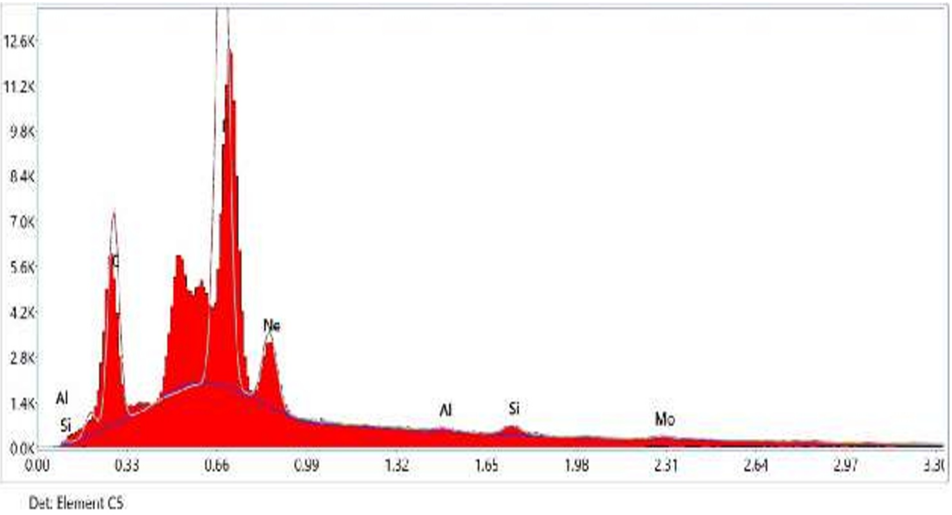
Fig 7: SEM image of GN3 after pin on disc at 30N load

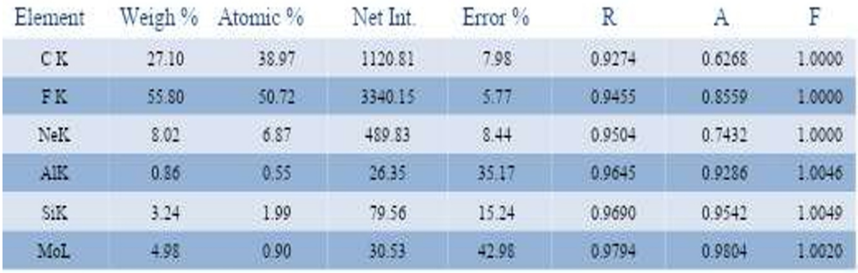
The material's wear resistance is greatly enhanced by the enlarged austenite. It was discovered when coated with chromium nitride to surface of AISI 304 SS increased material's strength, hardness under applied loads.

3.1 Energy Dispersive X-ray spectroscopy :

SEM is used for Edax analysis. After pouring liquid nitrogen into a cylindrical tube and allowing it to cool to room temperature for 2-3 hours, the SEM machine is turned on. After inserting the samples into the machine, the microscopic images were displayed on a computer screen, and the spectrum was created in any area of the SEM image to scan, analyse, and provide the result edax.



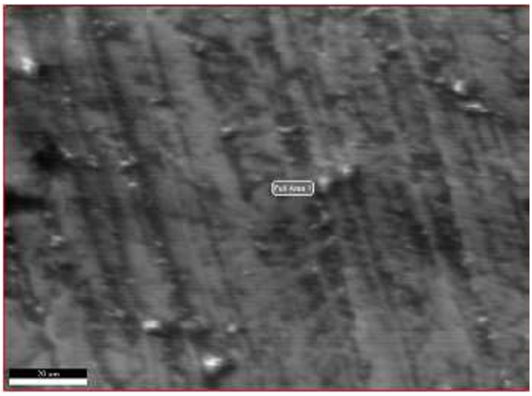


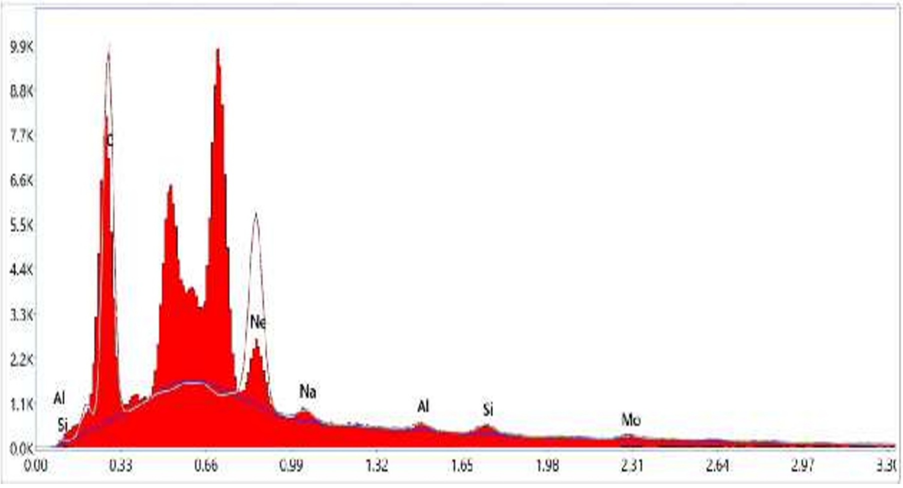


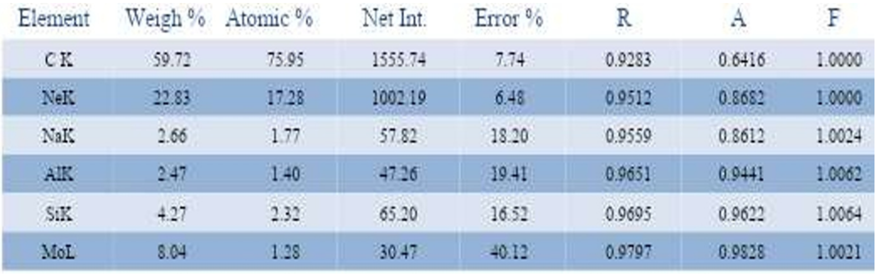
Element	Weigh %	Atomic %	Net Int.	Error %	R	A	F
C K	27.10	38.97	1120.81	7.98	0.9274	0.6268	1.0000
F K	55.80	50.72	3340.15	5.77	0.9455	0.8559	1.0000
NeK	8.02	6.87	489.83	8.44	0.9504	0.7432	1.0000
AlK	0.86	0.55	26.35	35.17	0.9645	0.9286	1.0046
SiK	3.24	1.99	79.56	15.24	0.9690	0.9542	1.0049
MoL	4.98	0.90	30.53	42.98	0.9794	0.9804	1.0020

Fig 8 : Untreated specimen

The layer was mostly ferrite in the untreated AISI 304 specimen. It was discovered that nitrided layers consist of an expanded austenite (γ-phase) after nitriding. The peaks of the pattern shows an expanded austenite called as S-phase.



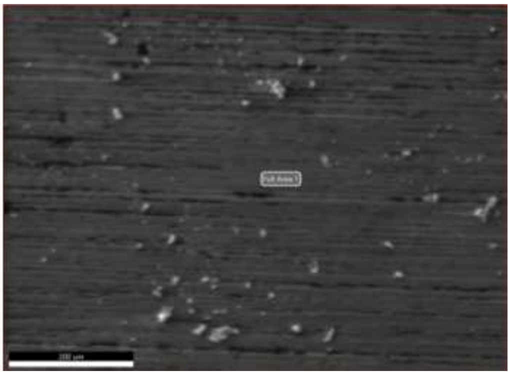


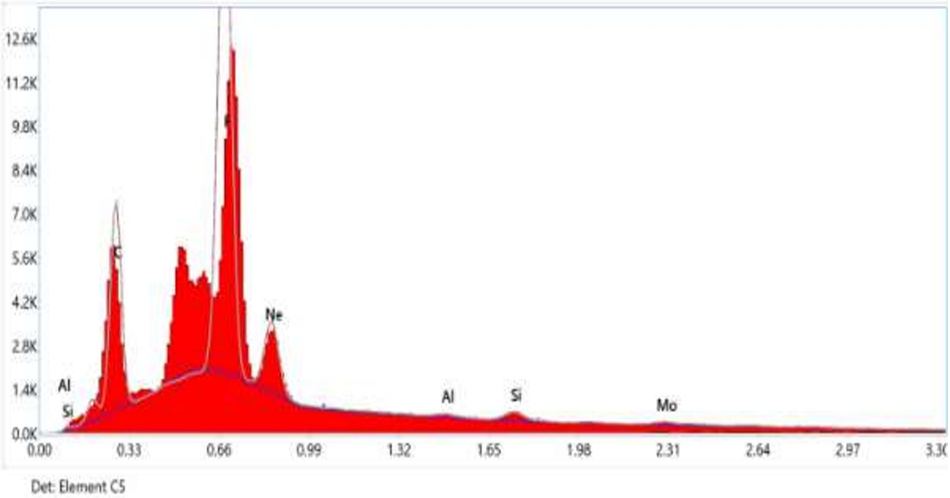


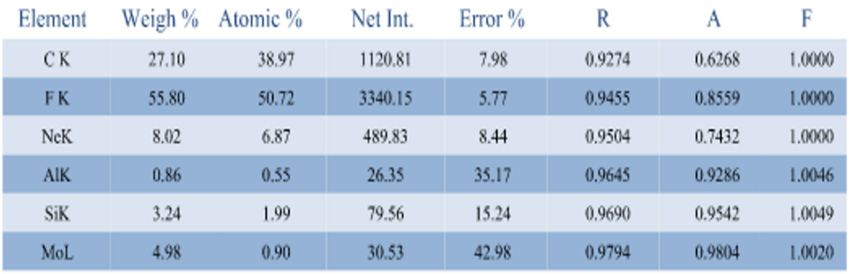
Element	Weigh %	Atomic %	Net Int.	Error %	R	A	F
C K	59.72	75.95	1555.74	7.74	0.9283	0.6416	1.0000
NeK	22.83	17.28	1002.19	6.48	0.9512	0.8682	1.0000
NaK	2.66	1.77	57.82	18.20	0.9559	0.8612	1.0024
AlK	2.47	1.40	47.26	19.41	0.9651	0.9441	1.0062
SiK	4.27	2.32	65.20	16.52	0.9695	0.9622	1.0064
MoL	8.04	1.28	30.47	40.12	0.9797	0.9828	1.0021

Fig 9 : Salt bath nitride specimen

In salt bath nitriding of 180 minutes specimen Carbon percentage increased as compared to that of untreated sample.







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MoL	4.98	0.90	30.53	42.98	0.9794	0.9804	1.0020

Fig 10: Gas Nitrided specimen

The above figure shows edax analysis for gas nitrided specimen.

3.2 Hardness behavior :

Rockwell hardness tests was performed on nitrided samples to evaluate the impact of nitrogen.All of the nitrided samples had their surface microhardness evaluated.

Readings to measure hardness:

Sample	Load(Kgf)	Hardness (RHN)
Untreated	100	38
SBN(60 min)	100	47
SBN(120min)	100	50
SBN(180min)	100	59
GN (6 hours)	100	75
GN (12 hours)	100	77
GN (18 hours)	100	96

For the test, a load of 100 kgf was used. As the hardness increased on surface layer composite and diffusion varieties of nitrides are formed.

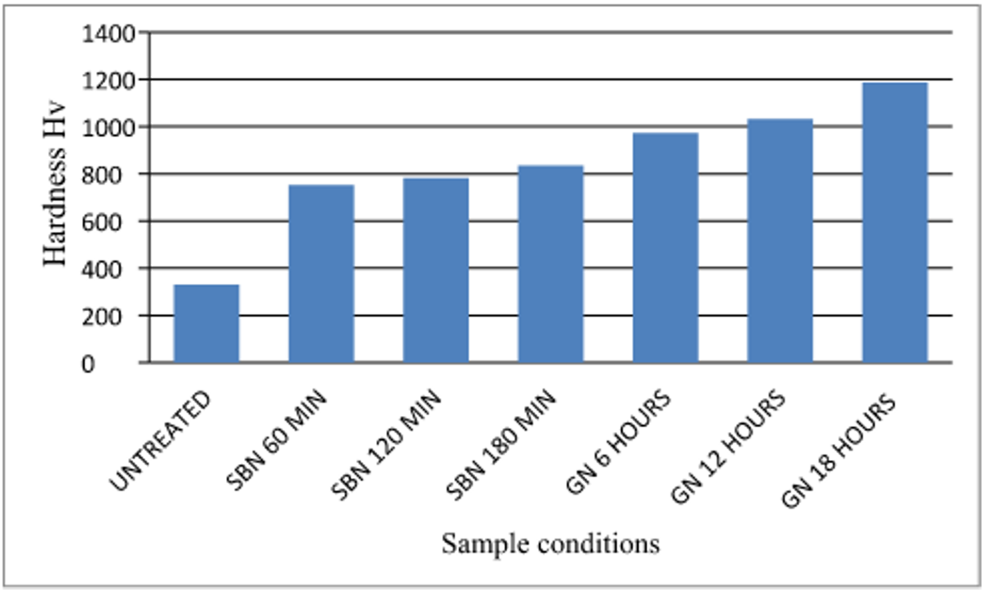

Fig 11: Hardness vs sample conditions

The above graph shows that the hardness of gas nitride specimen is more compared to that of salt bath nitride specimen.

4 Conclusions:

- SEM research revealed that in both salt bath nitrided and gas nitrided samples, a white layer known as the S phase, or enlarged austenite layer, developed.
- Then it decomposed to a CrN layer during gas nitriding for 18 hours. It was terminating that the distribution of nitrides increases with increasing nitriding time.
- Strong adhesion, plastic deformation, and abrasion cause the untreated sample to continuously lose material.
- From wear study it was found that gas nitrided sample has minimum wear loss compared to salt bath nitrided and untrated sample.
- Due to the increased presence of nitrides on the surface, the sample nitrided with gas (18hours) has the maximum hardness of all the samples.Compared to untreated and salt bath nitride specimens, gas nitride specimens has more hardness.

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