

SALT BATH NITRIDING OF CP TITANIUM GRADE-2 AND TI-6AL-4V GRADE-5

J. R. Deepak¹, V.K. Bupesh Raja² K.Arun Kumar³, Varun Radhakrishnan.H⁴, Sonu Skariah Thomas⁵,

School of Mechanical Engineering, Sathyabama University Chennai- 600119

Email id : deepakcape@gmail.com

Abstract. Titanium is known for its strong affinity towards nitrogen. The metal forms a nitride form case of high hardness when the salt bath nitriding is carried out. The selection of Grade 2 Titanium and Grade 5 Ti6Al4V Titanium alloy, stands with the fact that CP Grade 2 Titanium is the most formable and corrosion resistant amongst the pure grades of Titanium and grade 5 alloy is biocompatible and also has excellent tribological properties. This research work attempts to solve the problem of galling by comparing the morphology of the nitride case produced in Commercially Pure Grade 2 Titanium and the nitride formations produced in the Titanium Ti6Al4V alloy through Salt bath nitriding for a time span of 24 hours. Salt Bath Nitriding imparts unique improvements in Roughness, Hardness and Wear resistance of the samples thereby widening the applications of the material.

Index Terms—Surface Engineering Salt Bath Nitriding, Ti-6Al-4V, EDM

1. Introduction

Although many researchers have studied about the nitriding of common ferrous metals, the proper mechanism regarding the nitriding of Titanium and its alloys has not been completely understood. This paper presents the effect of different Salt Bath Nitriding parameters on the structure of nitrided zone of Commercially Pure Titanium (Grade 2) and



Grade 5 (Ti6Al4V) alloy and their influence on mechanical properties on the samples. The salt bath nitriding process introduces nitrogen into the surface of metal parts to improve tribological properties and performance. Nitriding is performed at sub critical treatment temperatures, typically at 1075 F (580°C) in a molten salt bath. Process Time can be reduced if the thickness is compromised. Liquid nitriding is considered as the benchmark for uniformity, consistency and flexibility. It provides the best corrosion resistance and short process time. The nitride surface layer is well integrated with bulk; hence it is not susceptible to flaking or peeling. Liquid nitriding achieves results by means of chemical reaction (at temperatures lower than conventional heat treatment) that leaves a hard nitride compound layer on the surface of the component. The liquid nitriding process is carried out for duration of 24 hours and is post processed by imparting the process of Air quenching and Water Quenching on distinct samples and the final values of Hardness, Roughness and Wear are noted. These test results are then compared with the results of the raw samples of both the grades of Titanium and the usefulness of introducing a nitride layer on the surface of grade 2 and grade 5 Titanium is understood by comparing the results before and after nitriding.

2. Experimental Setup

Liquid nitriding is a process in which anhydrous ammonia is introduced into cyanide - cyanate bath. The salt bath process uses the principle of the decomposition of cyanide to cyanate and the liberation of nitrogen within the salt for diffusion into the steel surface. The work piece has been cut into the required size (40mm*25mm*5mm), all the edges of the work piece must be cleaned and polished to ensure that no impurities are present on the metal surface. The cleaned work pieces are loaded into the nitriding chamber. Maintenance of dimensional and geometric stability during liquid nitriding is enhanced by hardening of parts prior to nitride treatment. Four plates each of Grade 2 and Grade 5 are obtained for the experiment. The apparatus used for the experiment is an enclosed chamber or a bath known as the Salt Bath. Inside the chamber salts of ammonia or cyanide are supplied prior to the Nitriding process. The specimens are then held on a fixture which is placed inside the molten salt bath containing the nitriding salts. The individual components should not be in contact with each other during the process. The overall process temperature is maintained in the range of (560 – 580)°C for a time period of 24 hours. The hardness and depth of the diffusion zone will determine fatigue strength. In addition to case depth and hardness also the obtained compressive residual stress state in the case is of major importance.

Factors affecting Nitriding are as follows:

- Quantity of parts per batch.
- Process Temperature, typically (500-580) degree celcius.
- Complexity of the components in terms of blind holes which adds the time involved in cleaning the component after the process.
- Ancillary steps like cleaning, polishing, impregnation etc.

3. Results and Discussion

The Nitriding of the 2 different grades of Titanium in Salt bath apparatus for 24 hours led to introduction of a surface coating of lustrous silver/black colour on the metal. This is the nitrided layer which acts as a surface coating for the metal and the values of Hardness, Roughness and Wear of the nitrided samples of CP Titanium grade 2 and grade 5 alloyed Titanium were noted down and comparison was made with the raw samples.

3.1. Hardness Test

The Vickers method is based on an optical measurement system. The Micro hardness test procedure, ASTM E-384, specifies a range of light loads using a diamond indenter to make an indentation which is measured and converted to a hardness value. The following observations were made post the hardness test

Table1. Vickers Hardness Values of Liquid Nitrided (24 Hours) Ti Grade-2

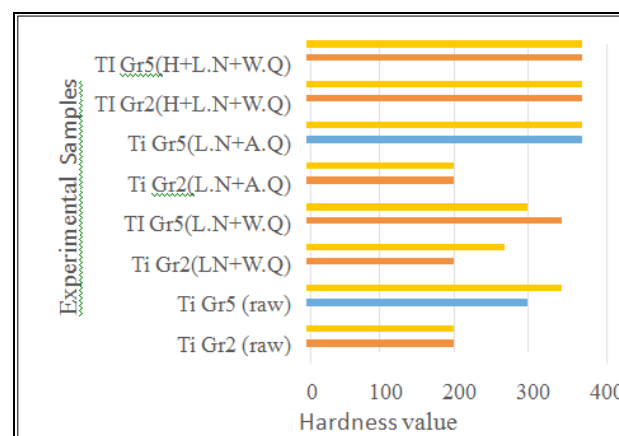
L.N+W.Q	L.N+A.Q	H+L.N+W.Q
187.2	186.3	370.2
189.2	220.4	369.5

250.2	215.8	371.4
266.9	221.2	372.2

Table2. Vickers Hardness Values of Liquid Nitrided (24 Hours) Ti6Al4V Grade-5

L.N+W.Q	L.N+A.Q	H+L.N+W.Q
349.3	368.6	367.3
351.1	368.9	369.7
352.3	368.9	370.2
353.1	370.6	370.7

It is found that the Average value of hardness has considerably increased in case of Liquid Nitrided samples of CP Grade 2 Titanium and Grade 5 Titanium alloy. The Pre Hardening of both grades of titanium increased the Hardness values to a notable degree and Water Quenching post Nitriding in Salt Bath furnace increased the Hardness of the component. This is of major importance in applications like manufacturing of fasteners, automotive components like nuts, bolts and also in environments where the metal is subjected to continuous stress. Maximum hardness value was observed to be 372.2 in case of CP Titanium grade 2, Hardened, Liquid Nitrided and Water Quenched sample compared to 202 in case of raw sample of CP Titanium. This clearly shows the advancement in hardness after nitriding. Also when the same process was carried out in grade 5, high hardness is achieved

**Figure 1.** Comparison of Micro Hardness values of Ti & Ti6Al4V Raw and Liquid Nitrided (Water & Air Quenched)

3.2. Roughness Test

Roughness is a component of surface texture. It is quantified by the vertical deviation of real surface from its ideal form. If these deviations are large, the surface is rough. Roughness test results displayed almost no major deviation in the surface of the samples but due to the presence of nitrided layer, a slight increase in roughness was observed while interpreting the Arithmetic mean value. This might be due to the fact that the components which were prone to liquid media for a time period of 24 hours increased their roughness in certain samples. This can be avoided by reducing the nitriding process time and carrying out the process in a more controlled environment.

Table3. Roughness Value of Liquid Nitrided Ti Grade-2 & Ti6Al4V Grade-5

Sample	Layer1		Layer2	
Roughnessvalue	R _a μm	R _z μm	R _a μm	R _z μm
Grade2Raw	7.28	55.2	4.12	52.7
Grade5Raw	3.65	48.8	4.53	46.8
Grade2(L.N+A.Q)	4.92	49.6	4.33	61.3
Grade2(H+L.N+W.Q)	2.91	31.0	3.35	50.9
Grade2(L.N+W.Q)	4.27	48.4	7.77	62.0
Grade5(L.N+A.Q)	6.92	46.6	3.63	52.5
Grade5(H+L.N+W.Q)	8.94	46.2	8.24	51.2
Grade5(L.N+W.Q)	4.72	49.7	4.62	49.7

Note: R_a refersto Arithmeticaveragevalueof Roughness
R_z refersto Mean Roughness depthvalue

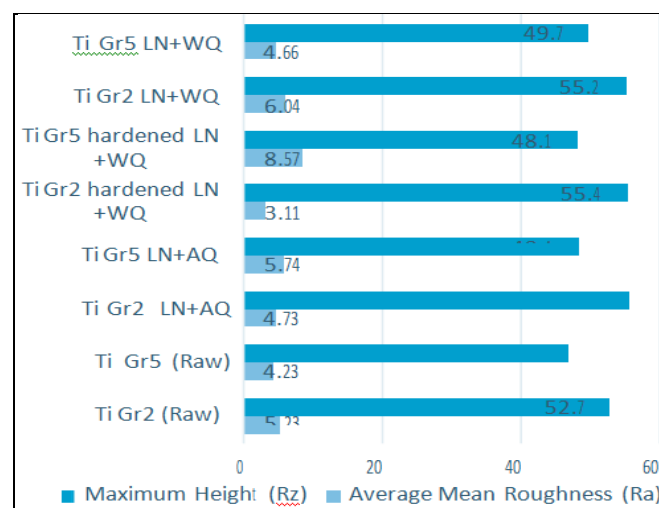


Figure 2. Comparison of Roughness Comparison Graph of Liquid NitridedTi Grade-2&Ti6Al4V Grade-5

3.3 Wear Test

The wear test was carried out in Ducom TR-20LE-PHM-250. Pin on disc rig to test the wear resistance of Titanium grade 5 alloy liquid nitrided for 24 hours, water quenched as the sample showed deviations from other samples. A pin on disc tribometer consists of a stationary "pin", which is Titanium metal in this case, under an applied load in contact with a rotating disc. The disc in which the pin is placed is made of hardened high Carbon, high Chromium Steel, 60HRC hardness which has excellent wear resistance. The pin surface is slid against the surface of the rotated disc. The time and sliding distance determine the amount of wear in the surface of the pin. A test load of 5 N was introduced in case of raw sample and a test load of 10 N was introduced on the nitrided sample to infer the rate of wear resistance in nitrided sample. The load is also directly proportional to the wear caused in the surface of the pin. This is the Wear and friction test rig. The Wear, Time, Sliding diameter, coefficient of friction are the various test parameters. The presence of nitride layer in the sample improved the wear resistance of the metal. The final value of amount of wear in the nitrided sample was observed to be 23.56 micrometers whereas the raw sample showed a value of 53 micrometers during the wear test for half the load. Liquid nitriding is therefore suggested for Titanium parts and components in industries where the metal is exposed to high wear and friction applications along with high corrosion resistance.

TABLE-4: WEAR TEST VALUES

Time(seconds)	Wear (micrometers)	Frictional Force(N)
55.14	5.0	3.58
85.52	9.70	3.29
130.72	15.63	3.31
190.42	21.28	3.20
236.64	23.56	3.76

Post test examination led to some interesting observations and results. The Grade 5 raw sample showed very high wear resistance values. This stands with the fact that Titanium is a wear resistant metal and Grade 5 which is widely used for manufacture of Aerospace components and medical implants, is amongst the most wear resistant grades available. The amount of wear is determined by measuring appropriate linear dimensions of both specimens before and after the test, or by weighing both specimens before and after the test.

4. Conclusions

The test results and the observations give a conclusion that the liquid nitriding process improves the tribological properties of the titanium grade 2 and Ti-6Al-4V alloy. Grade 2 Titanium which is usually not used in harsh saline and chemical environment due to its pure state has been benefited by nitriding in Salt Bath media. This is a very important fact as nitriding Commercially pure Titanium makes it equally competent with alloyed Ti grade 5 and it can be used in more useful and new areas which was once restricted and did not make its place into. The compound layer is responsible for the improved wear properties observed in Ti-6Al-4V alloy. In comparison with the roughness values, it is studied that Ti Grade 5 6Al-4V alloy liquid nitrided for 24 hours plus Water Quenched displayed excellent wear resistance and roughness characteristics. The wear can be further reduced by subsequent treatment by a dry film lubricant coating on one member of the couple. We have hereby presented the effect of introducing a nitride coating on the surface of Grade 2 CP Titanium and Grade 5 Ti-6Al-4V alloy in a salt bath media and the usefulness of using this age old technique of surface treatment. Vigorous research work is being carried out around the globe for improving the surface properties of Titanium, especially Grade 5 Titanium which is the work house industry metal and it is concluded that liquid nitriding is the most effective nitride process for improving tribological properties of the metal with minimum distortion among other advantages like short process time and the most cost-effective method of nitriding. The Trademark Arker process Salt Bath nitriding is amongst the most industrially accepted processes with almost no environmental impact and hazards, is effective in nitriding Titanium grade 2 and grade 5 and usage of the metal in more useful, high-end applications such as in Supersonic jet engines and parts of high performance super cars to an advanced level. Thus we conclude the research work by standing with the fact that further research work and experiments regarding the tribological properties of Titanium and by carrying out surface engineering techniques on the metal will open ways to further beneficial and cost-effective exploitation of Titanium.

References

- [1] Anandan. C, Rajam. K.S , MicroRaman study of the effect of oxide layer on nitriding of Ti-6Al-4V Vol. 254, Applied Surface Science, 2008.
- [2] Budzynsk. P, Surface modification of Ti-6Al-4V alloy by nitrogen ion implantation Vol. 261 ,Wear, 2006.
- [3] Cuthill.J.R, W.D. Hayas, R.E. Seebold, Nitriding Phenomena in Titanium and the 6Al-4V Titanium alloy Vol. 64.A, No.1, Journal of Research of the National Bureau of Standards, A. Physics and Chemistry, 1959
- [4] Frank Czerwinski, Thermochemical treatment of metals Vol. 80, Intechopen, 2012.
- [5] George Vander Voort, Metallographic Preparation of Titanium and its Alloys Vol. 3, Issue 3 , Beulher, 2015
- [6] Gotman.I, E.Y Gutmanas, Department of Materials Science and Engineering, Technion, Israel Institute of Technology, Titanium nitride-based coatings on implantable medical devices, Advanced biomaterials and devices in medicine, Haifa, Israel 2014.
- [7] Guilherme AS, Henrique GE, Zavanelli RA, Mesquita MF, Surface roughness and fatigue performance of commercially pure titanium and Ti-6Al-4V alloy after different polishing protocols, 2005

- [8] Kenneth G. Budinski, Tribological properties of titanium alloys Vol .151, Wear, 1991.
- [9] Koyuncu.E, F.kahraman,O. karedeniz, Investigation of surface properties of high temperature nitrided titanium alloys Vol 37, Journal of achievements in material and manufacturing engineering, 2009.
- [10] Li Yuan-hui, Luo De-fu, Wu Shao-xu, School of Material Science and Engineering, Jiangsu University, Zhenjiang Jiangsu, QPQ Salt Bath Nitriding and Corrosion Resistance Vol. 118, Solid state phenomena, China,2006.
- [11] Mittemeijer.E.J, Max Planck Institute for Intelligent Systems, ASM Handbook, Fundamentals of Nitriding and Nitrocarburizing Vol. 4A, Steel Heat Treating Fundamentals and Processes, 2013
- [12] Veiga.C, J.P. Davim,A.J.R. Loureiro, Department of Mechanical Engineering, Coimbra Institute of Engineering, Rua Pedro Nunes, Quinta da Nora, Properties and applications of Titanium Alloys: A brief review, Reviews on Advanced Material Science, Coimbra , Portugal 2012.
- [13] Weisenburger JN, Hovendick SM, Garvin KL,Haider H, How durable are titanium nitride coatings on total hip replacements, J Bone joint surg,2011.
- [14] Zhecheva A, Sha W,Malinow S,Long A, Enhancing the microstructure and properties of titanium alloys through nitriding and other surface engineering methods. Surf coat technol, 2005.

