



Q-P-Q Salt Bath Nitriding and its effect on Steels:Review

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Abstract—

Nitriding is a case-hardening process of enriching the solid steel surface with nitrogen at a low temperature, normally in the range of 500-575°C (when the steel is ferritic). The nitrogen combines with the iron to form an iron-nitride compound layer that provides improved surface properties; e.g., resistance to wear, friction, corrosion, and fatigue. "QPQ" stands for "Quench-Polish-Quench", which describes a sequence of secondary steps following the liquid nitriding step. These steps entail the sequence: (1) OXIDATION: 2-3 microns of the surface layer is transformed to an iron oxide. This is done by immersing the parts in specially formulated 'salts' between 400°C - 425°C (750°F -800°F); (2) POLISHING: to improve surface finish and (3) RE-OXIDATION: to recover the oxide layer thickness that may have been lost during the polishing step. QPQ is prescribed when a smooth surface finish and maximum corrosion protection are required. In industrial application mostly the problem faced is corrosion, surface roughness and tribological behavior of material after application of QPQ salt bath nitriding. The QPQ

process gives better result for Cr bearing material but as per as industry problem is concerned cost is one more factor to be considered. EN8 steel is used in various industrial application like piston rod of gas spring, the gas spring is used in various automobile as well as household applications. The paper reviews the Q-P-Q Salt bath nitriding and its effects on steels.

Keywords— Corrosion resistance, QPQ, Nitriding, Surface roughness, Wear Resistance

I. INTRODUCTION .

The QPQ process is particularly suitable for all kinds of low-carbon steel, constructional steels, tool and mold steels, and cast iron. Many researchers have done work on the microstructural analysis, corrosion behavior, tribological behavior of a medium carbon steel and a high-speed steel. It is found that the nitrided surface consists of three layers: an oxide film in the outer layer; a nitride layer (compound layer) in the middle; and a diffusion layer near to the base metal. The peak value of the oxygen content

is secured in the outer layer and the peak values of the nitrogen and carbon content obtained in the nitride layer [1].

A. Salt bath nitriding.

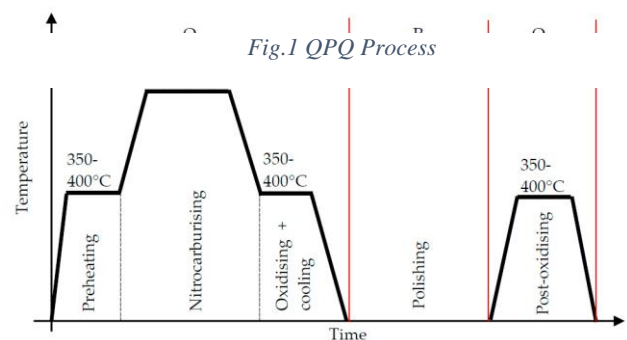
In salt bath nitriding, the medium used is a salt containing nitrogen such as a cyanide salt, or potassium nitrate. Cyanide salt baths have the same, or an even higher, nitrogen potential than ammonia. The nitriding behavior can be reasonably described by nitrogen diffusion into the work pieces. During nitriding, most nitrogen is interstitially dissolved. After nitriding, a slight oxidation has been found. Actually, it is a nitrocarburizing process, because the salts used usually also donate carbon to the work piece surface, and the two elements generally diffuse into the workpiece surface simultaneously [2]. As with the other nitriding methods, the modified case is comprised of a compound layer and a diffusion zone from the surface towards the core. The compound layer consists of nitrides formed with the main elements and alloying elements. The diffusion zone is composed of a solid solution with N and nitride precipitates. Salt bath nitriding has the advantages of easy operation, less processing time, low cost. However, the salts used are highly toxic when heated to a high temperature. The temperature used is typical for all nitrocarburizing processes, i.e. 580-650°C. It would seem that salt bath nitriding should have been eliminated from industrial use because of the high toxicity of the salts. However, the process is still widely used because the old toxic salts have been replaced by non-toxic salts.

B. Quench Polish Quench

The QPQ surface heat treatment, namely the Quench-Polish-Quench salt bath technology, mainly consists of six steps: 1) degreasing, 2) preheating, 3) nitriding, 4) oxidizing, 5) washing and 6) oil immersion. It is a nitrocarburizing process in a cyanide-cyanate salt bath with post-oxidation in a nitrate-nitrite salt bath.[3] It is widely used for tools, molds and gears to increase their strength, wear and corrosion resistance and fatigue strength, and to obtain less distortion [1]

QPQ is distortion-free and adaptable for all grades of steels. It has been established that a compact and integrated magnetite film (Fe_3O_4) can be developed through this low temperature thermo-chemical process, which can greatly improve wear resistance [4]

An example of QPQ treatment process is shown in Fig.1 applied heat treating process, also known as quench-polish-quench (QPQ), mainly consists of the following steps (i) pre heating, (ii) nitrocarburizing, (iii) oxidizing, (iv) cooling, (v)



polishing and, finally, (vi) post-oxidising. The specimens were preheated at about 3500C in air and then nitrocarburized in salt bath at about 5800C. After that the samples have been oxidized and cooled in water; then they were polished and, finally, exposed to further post-oxidation in salt bath at about 3500C and then cooled in water to room temperature [5]

II. EFFECTS OF QPQ PROCESS ON STEELS

AISI 4140 steel was nitrided in salt bath to study and analyze the behavior of the surface roughness. The structural surface characterization behavior of the nitrided steel was compared to the behavior of the same steel which was untreated. The nitriding process was implemented in the salt bath component at ten different times (from 1 h to 10 h) when the temperature was constant at (580°C). The influence of nitriding treatment on structural properties of the material was studied by scanning electron microscopy (SEM), microhardness tester and surface profilometer. It was found that salt bath nitriding was effective in improving the surface properties behavior of this steel. Experimental results showed that the nitrides ϵ -Fe₂-3(N,C) and γ' -Fe₄(N,C) present in

the compound layer increase the microhardness (406–502 HV0.3), the initial surface roughness values of nitrided samples were higher than those of un nitrided specimens, it also observed that the increasing the nitriding time increases the surface roughness parameters (Ra, Rq and Rz) [6]

Salt bath preoxidation was primarily conducted prior to salt bath nitriding, and the effect on salt bath nitriding was compared with that of conventional air preoxidation. Characterization of the modified surface layer was made by means of optical microscopy, scanning electron microscope (SEM), micro-hardness tester and x-ray diffraction (XRD). By observing the cross-sectional microstructure of specimens nitrided at 560°C for 120min with different preoxidation method compound layer (also called white layer) was formed at the outermost surface after salt bath nitriding under different conditions and the thickness of compound layer was significantly increased by salt bath preoxidation the result shows 60% increase in thickness. The effective hardening layer thickness was evidently increased with the salt bath preoxidation. The enhancement mechanism is that the sample treated by salt bath preoxidation has higher relative content of Fe₃O₄, which can react with active nitrogen atoms in salt bath nitriding to accelerate the growth of Fe₃N [7]

When C110 steel was subjected to liquid nitriding at 430 °C, the nitrided layer was almost composed of a thin ε-Fe₂-3N layer. When C110 steel was subjected to liquid nitriding at 640 °C, the phase composition of the nitrided layer was greatly changed. The nitrided layer depth increased significantly with increasing the treating temperature. The liquid nitriding effectively improved the surface hardness. After liquid nitriding, the absorption energy of the treated sample decreased and the tensile strength increased by Charpy V-notch (CVN) test. But the elongation of treated sample decreased. The reason is that the nitrided layer of sample is hardened and there is brittlement by diffusion of nitrogen atom. Despite of treatment temperature, the liquid nitriding can improve the corrosion. After being nitrided at 430 °C, the nitrided layer

of the C110 steel was mainly composed by ε-Fe₂-3N, which had an excellent corrosion resistance and high microhardness, the nitrided sample had the best corrosion resistance. After nitriding temperature over 580 °C, especially at 680 °C, the sample's surface was covered by the thick oxide layer, which had very low hardness and corrosion resistance. So, the corrosion resistance of samples was severely compromised [8]

The influence of nitriding on the corrosion resistance of martensitic stainless steel was investigated. Plasma nitriding at 440 °C and 525 °C and salt bath nitrocarburizing were carried out on X17CrNi16-2 stainless steel. Microhardness profiles of the obtained nitrided layers were examined. Phase composition analysis and quantitative depth profile analysis of the nitrided layers were performed by X-ray diffraction (XRD) and glow-discharge optical emission spectrometry (GD-OES), respectively. Corrosion behavior was evaluated by immersion test in 1% HCl, salt spray test in 5% NaCl and electrochemical corrosion tests in 3.5% NaCl aqueous solution. Results showed that salt bath nitrocarburizing, as well as plasma nitriding at low temperature, increased microhardness without significantly reducing corrosion resistance. Plasma nitriding at a higher temperature increased the corrosion tendency of the X17CrNi16-2 steel. [9]

The nitriding behavior of austenitic stainless steels (AISI 304 and 316) was studied by different cold work degree (0% (after heat treated), 10%, 20%, 30%, and 40%) before nitride processing. The microstructure, layer thickness, hardness, and chemical micro composition were evaluated employing optical microscopy, Vickers hardness, and scanning electron microscopy techniques (WDS microanalysis). The initial cold work (previous plastic deformations) in both AISI 304 and 306 austenitic stainless steels did not show special influence in all applied nitriding kinetics (in layer thicknesses). The nitriding processes have formed two layers, one external layer formed by expanded austenite with high nitrogen content,

followed by another thinner layer just below formed by expanded austenite with a high presence of carbon (back diffusion). An enhanced diffusion could be observed on AISI 304 steel comparing with AISI 316 steel (a nitrided layer thicker could be noticed in the AISI 304 steel). The mechanical strength of both steels after nitriding processes revealed significant hardness values, almost 1100HV, on the nitrided layers.[10]

The study was conducted to improve the fatigue resistance of the stainless steel 316L. The rotational bending method was used with constant and variable stresses at different times of (1, 3, 5) hours and at (5300 C, 6300 C). These tests were performed before and after nitration study showed that the fatigue resistance was directly proportional to the increase in nitrate time due to the increased depth of the hardened layer, but this resistance decreased when the temperature was 630 °C due to the formation of brittle phase with low resistance.[11]

The low-temperature liquid nitriding of stainless steels can result in the formation of a surface zone of so-called expanded austenite (S-phase) by the dissolution of large amounts of nitrogen in the solid solution and formation of a precipitate-free layer supersaturated with high hardness. Erosion–corrosion measurements were performed on low-temperature nitrided and non-nitrided 316L stainless steels. It was shown that low-temperature nitriding dramatically reduces the degree of erosion–corrosion in stainless steels, caused by the impingement of particles in a corrosive medium. The nitrided 316L stainless steels exhibited an improvement of almost 84% in the erosion corrosion resistance compared to their non-nitrided counterparts. The erosion-only rated and synergistic levels showed a general decline after low-temperature nitriding.

Low-temperature liquid nitriding could not only reduce the weight loss due to erosion but also significantly reduce the weight loss rate of interactions, so that the total loss of material decreased evidently. Therefore, 316L stainless

steels displayed excellent erosion–corrosion behaviors as a consequence of their highly favorable corrosion resistances and superior wear properties.[12]

In nuclear power plants (NPP), rod cluster control assembly (RCCA) tubes undergo impact under low-pressure contact against guides, leading to specific wear on the contact surfaces. To focus on this industrial issue, an experimental layout was designed to perform reciprocating fretting in different environments. A representative tube interface (nitrided AISI 316L)/plane (AISI 304L) was investigated in air and in a solution composed of 1000 ppm of bore and 130 ppm of lithium to be closed to a NPP primary water chemistry at atmospheric pressure and room temperature. Wear regimes were identified in terms of variation in parameters such as sliding amplitude (from $\pm 40 \mu\text{m}$ to $\pm 160 \mu\text{m}$), normal loads (from 2N/mm to 5N/mm) and test duration

(From 100,000 to 1.5 million cycles). Surface damage evolution was followed by 3D profilometry and several analyses (SEM, EDX, optical observation) were conducted on surfaces and cross-sections. Based on these results, fretting-wear mechanisms in the 304L SS plate and nitrided 316L SS tube were investigated. The first results in dry environment showed that, for $\delta g^* b \pm 120 \mu\text{m}$, plane profiles had a “W-shape” whereas for $\delta g^* N \pm 120 \mu\text{m}$ there was a U-shape. Thus, a W-shape morphology parameter M_w was defined, taking into account the third-body wear volume. The worn surfaces of the plane were smoother in solution, and wear was no longer detected on the tube specimens. A modified energy wear approach was then implemented to quantify reciprocating fretting wear rate according to ambient conditions [13]

The Study of premature failure of 316L valve cores by salt bath nitriding at 580 °C for 5.5 h, searching for some settlements to improve the service life of the valves was conducted. the premature failure of the valves was because of the excessive emergence of microcracks and compounds, which were induced by an improper

selection of nitriding techniques. The analysis of the phase composition of rust showed that the failure accords with the characteristic of the electrochemical corrosion in dry-wet cycling environment. The microstructure of the nitrided layer was studied. Wedge microcracks in the oxide layer and reticulate microcracks beneath the oxide layer were observed as rapid corrosion channels of the nitrided layer. Electrochemical measurements showed the samples with smaller grain size had higher corrosion potential and lower corrosion current density, and the polarization curves of the samples after the nitriding treatment had no obvious passivation area. Phase composition analysis of the nitrided layer showed that a mass of compounds emerges in the second and third sublayers, and the S phase with low nitrogen concentration emerged in the fourth sublayer. [14]

QPQ salt bath treatment of 45 steel was conducted by nitriding at 565 °C for various time (60 min, 90 min, 120 min, 150 min and 180 min), followed by the same post-oxidation process with heating temperature of 430 °C and holding duration of 40 min. Characterization of modified surface layers was made by means of optical microscopy, microhardness test, X-ray diffraction analysis, corrosion and wear resistance test.

The results showed the formation of a very thin Fe₃O₄ oxide layer during post-oxidation on the top of the bright nitrides compound layer formed during nitriding. The maximum microhardness value of 630 HV_{0.01} was obtained after nitriding at 565 °C for 120 min, which was two times higher than that of the untreated sample. The corrosion and wear resistance of 45 steel could be significantly improved by QPQ complex salt bath treatment, and the optimum nitriding duration for improving the wear and corrosion resistance was 120 min and 90 min, respectively [15]

It was shown that the two applied surface treatments markedly improved the wear resistance of the steel as a result of surface hardening and the compressive residual stress field thus induced, along with the formation of

surface layers of nitrides and oxides with excellent tribological features. These surface treatments also led to good performance when tested at high temperature simulated working conditions: no relevant stress relaxation and softening until 500°C, but gradual stress relaxation and partial softening after prolonged exposure at 600°C was observed. Furthermore, the application of a deep cryogenic treatment showed a significant reduction in the wear rate compared to that of conventional quenched and tempered H13 steel.[16]

The reviews of the two thermos chemical surface treatments (salt bath nitriding and gas nitriding followed by the post oxidation) applied to AISI 347 material. Morphological characterizations were carried out with the help of scanning electron microscope, X-Ray Diffractometer and Pin-on-disc wear testing machine. The detailed study made by authors the results shown that with gas nitriding the wear resistance of the material is improved more compared with the salt bath nitriding. [17]

QPQ salt bath treatment of H13 steel was conducted by nitriding at the same temperature of 565°C for various times, followed by the same post-oxidation process. Optical microscope, micro-hardness tester, X-ray diffraction and wear resistance tester were employed to characterize the microstructure, phase constituents, micro-hardness and wear resistance of the treated specimens. The results showed a compound layer mainly composed of ε-Fe₂₋₃N and diffusion layer were formed during salt bath nitriding and a thin oxide layer composed of Fe₃O₄ was formed by post-oxidation, and the compound layer thickness increases with the nitriding time. The maximum surface hardness value of 1441 HV_{0.3} was obtained after nitriding at 565°C for 150 min, which is as three times high as that of untreated sample. Meanwhile the wear resistance of H13 steel is significantly improved by QPQ treatment, 150 min is the optimum nitriding time to improve the surface hardness and wear resistance of H13 steel [18]

Titanium is known for its strong affinity towards nitrogen. The metal formed 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 was the most formable and corrosion resistant amongst the pure grades of Titanium and grade 5 alloy is biocompatible and also had 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 imparted unique improvements in Roughness, Hardness and Wear resistance of the samples thereby widening the applications of the material [19]

The QPQ complex salt bath treatment is a type of surface technology which contains mainly salt bath nitriding and post-oxidizing processes. The effect of QPQ oxidizing temperature and duration on the corrosion resistance of QPQ treated specimens had been explored by immersion tests and salt spray tests. All the specimens were post-oxidized after being nitrided at 5700C for 2 hours. The material used in this study were 1020 steel. In the immersion tests, the specimens were immersed in 3% H₂O₂ and 10% NaCl water solution. In the salt spray tests, specimens were salt spray tested using 5% NaCl neutral solution. From the experimental data, for high corrosion resistance, conclusions showed that :(1) appropriate temperature should be selected carefully in post-oxidizing stage. Too low or too high temperature would decrease the corrosion Resistance. (2) The best post-oxidizing duration should generate magnetite film in porous area and should not collapse. (3) In second oxidizing stage, the porous area has been partly eliminated, so the duration should be less than the duration of post-oxidizing [20]

Combined influence of liquid nitriding time and liquid nitriding temperature on the properties of nitrided region developed on 316 stainless steel

was investigated. Studies were restricted to low temperatures of 4250C and 4750C while the nitriding time was varied from 4 to 16 h. Developed nitrided layers were characterized by optical microscopy, scanning electron microscopy and X-ray diffraction. Mechanical and electrochemical properties of the nitrided layers were characterized, respectively, by Vickers micro-hardness measurements and cyclic polarization studies in 3.5 wt% NaCl solution. After 4-h nitriding at 4250C or at 4750C, the developed expanded austenite started to contract with simultaneous decrease in nitrided layer thickness, hardness and corrosion resistance. These prolonged nitriding time effects have been attributed to the decreased chemical potential of nitrogen in salt bath leading to the outward diffusion of N (from sample to salt bath). However, the 4-h nitriding at 4750C resulted in larger expansion of austenite as compared to 4-h nitriding at 4250C. Due to this, better mechanical and electrochemical property was realized after 4-h nitriding at 4750C suggesting that the increased amount of N solubility in sample led to simultaneous increase in hardness and corrosion resistance of stainless steels.[21]

AISI 304 austenitic stainless steel was low-temperature liquid nitrided in a molten salt bath at 703 K for 8 hours, which produced a 3-layered structure consisting of a top oxide layer, an intermediate nitrogen-rich layer and a bottom carbon-rich layer. The effect of nitriding on its corrosion resistance was investigated in a H₂S environment. The corrosion rate of the untreated sample is about 3.3 times that of the nitrided sample after H₂S corrosion. Corrosion pits can be clearly observed on the surface of the untreated sample, while the nitrided sample surface remained relatively intact. Both the oxide layer and the nitrogen-rich layer can help reduce the hydrogen permeation, which is beneficial for combating hydrogen embrittlement. The corrosion products mainly consisted of oxides, hydroxides, and sulfates. The nitrided layers can serve as a barrier to corrosion, thus preventing the corrosion of the substrate material. Active nitrogen in the nitrided layer reacts with H ions to form NH₄⁺, which effectively prevents

further acidification of the local area and inhibits the occurrence of pitting corrosion and the dissolution rate of the metal in the etching hole, thus improving the local corrosion resistance of the stainless steel [22]

III. CONCLUSION

It appears from the literature survey that QPQ process has been able to improve the surface properties of a number of ferrous materials. This process is found to have improved, wear, corrosion and fatigue properties of various steels with high surface finish. However, the application of QPQ treatment in the widely used EN8 steel is not well documented in literature.

On the contrary, EN8 steels are known for its wide application in automobile and other engineering sectors, where the service conditions require that the steel should possess a high fatigue strength with excellent surface finish, coupled with a very good wear and corrosion resistance. In view of this, it seems prudent to explore the scope of QPQ process for achieving the desirable benefit of improved properties. So, in the present investigation, attempts will be made to study the effect of QPQ salt bath nitriding on the structure and properties of EN8 steel.

Following factors can be studied for QPQ process

A. Corrosion Behaviour

The effect of QPQ duration on the corrosion resistance of QPQ treated specimens will be explored.

B. Surface Hardness

From the survey it is concluded that the surface hardness increases gradually with the nitriding duration, this is because the compact surface layer is getting thicker and thicker with nitriding time. It can also be seen that the hardness value decreases as the load increase at the same nitriding time. That is because when the load is small, hardness indenter completely hits the nitrided layer. With the gradual increase of the load, the contribution of the matrix to the hardness value will increase. Therefore, the tested surface hardness value decreases as the testing load increases.

C. Wear

The weight loss of QPQ treated samples is dependent on nitriding time and is decreases gradually with increase of time. This is because the surface layer treated by QPQ is composed of compact nitride layer with high hardness with low friction coefficient. Hence, wear becomes important parameter to be checked after QPQ treatment

D. Compound Layer

The nitrided layers can serve as a barrier to corrosion, thus preventing the corrosion of the substrate material.

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