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Surface Modification of A105 Steel using Tenifer Process of Ferritic Nitrocarburizing

Er. Ravideep Singh Sran¹, Er. Gora Ram Sharma²

^{1, 2}Bhai Maha Singh College of Engineering, Sri Muksar Sahib

Abstract: *The industrial valve seats are most susceptible to wear components in many valves due to various environmental conditions and operating practices the sliding mechanisms are intensively de-graded by wear. This study is concerned in reducing both damage and premature wear of industrial valve seats, which are intended for sliding wear resistance applications. Current study investigates the variation of various mechanical and surface properties of A105 medium carbon steel under different experimental and testing conditions. Micro-hardness, wear resistance, and microstructure tests were conducted on treated components. The results reveal significant changes in the microstructure after the heat treatment. A compound and diffusion zone is distinguished on the treated specimen. It is found that maximum hardness is achieved at 580 °C and hardness increases by increasing the soaking time. Furthermore the wear resistance tests were conducted on a pin on disc tribometer under dry conditions. The measurements were taken as weight loss in grams. As compared to untreated specimen the wear rate has significantly decreased in case of treated specimen.*

Keywords: *Tenifer, Post oxidation, Salt bath treatment, A105 steel.*

I. INTRODUCTION

With the advancement in industrial era, materials with high strength and good mechanical properties are desirable. Engineering materials are mostly tailored by different treatment processes to match their suitability with the industrial applications. Many surface modification techniques are used for altering the surface properties. Salt bath processes are one of the most commonly used due to their simplicity and economic viabilities. Nitrocarburizing process was developed to attenuate the effect of long nitriding times. Ferritic nitrocarburizing process is advantageously being used in today's industrial components, which are required to endure high fatigue, surface hardness, impact, wear resistance and corrosion protection properties. It is a thermochemical treatment and is one of the oldest and cheapest methods to enhance the material properties [1]. It is applied advantageously to textile machinery components, water pump parts, timing gears and a number of automotive parts which undergo sliding-rolling [2- 4]. Nitrocarburizing process was originally created as an alternative to gas nitriding, in order to produce a more uniform case [5]. Nitrocarburizing has many advantages over conventional surface treatment techniques; it has lower treatment temperature and shorter treatment time, high degree of shape and dimensional stability [6]. Usually nitrocarburizing process is done in three media; Liquid, gas, and plasma nitrocarburizing [7]. Out of these three process liquid nitrocarburizing is widely used in industries [8]. Nitrocarburizing and carbonitriding terms are often confused as similar, but both are different thermochemical treatments for surface hardening of steel components using diffusion of carbon and nitrogen on the work piece surface. Carbonitriding is a modification of carburizing process and it is not a form of nitriding process. Whereas, nitrocarburizing is modification of nitriding process and it is not a form of carburizing [9, 10]. Also the continuous improvements in the conventional Nitriding/ Nitrocarburizing processes are leading to the enhancements in various desirable mechanical properties. Some of the emerging techniques are surface mechanical attrition treatment (SMAT)[11], rare earth assisted salt bath nitriding [12]; Boro-nitriding coating [13]; large pulsed electron beam (LPEB)[14]; dual DC/ Radio Frequency Inductively Coupled Plasma (RFICP) source [15]. Usually in traditional salt bath carbonitriding, surface hardening is obtained by martensitic transformation; at a process temperature ranges from 800 to 850 °C. In nitrocarburizing, surface hardening is obtained by precipitation of nitrides and carbonitrides at temperatures 480 °C to 570 °C [16, 17]. The first Ferritic Nitrocarburizing treatment process was invented by UK chemical giant, Imperial Chemical Laboratories (ICL), and later on Degussa of Germany came up with an improved salt-bath process, which they called Tenifer [18]. For the current research we have studied the effects of Tenifer treatment on A105 medium carbon steel, which is extensively used in valve seats. As we know, the valve seats are the most susceptible to wear components. Due to various environmental conditions and operating practices, the sliding mechanisms are intensively de-graded by wear. This study is concerned in reducing both damage and premature wear of industrial valve seats, which are intended for sliding wear resistance applications.

II. EXPERIMENTATION

Two different surface treatments methods were studied: Conventional ferritic salt bath Nitrocarburizing process (Tenifer) and ferritic salt bath Nitrocarburizing technique with post oxidation. The process was done in a cyanide-cyanate salt bath with a trade name Tenifer® and using a post-oxidation process having a trade name OXYNIT® in a nitrate-nitrite salt bath. A 105 steel is selected for current study, as it is a medium carbon and an inexpensive steel. A105 steel specimens were prepared by grinding and degreasing to achieve a uniform surface roughness. The specimens were then pre-heated at a temperature of 300 °C for different time durations. After achieving the desired temperature, the set of specimens was dipped into the Tenifer salt bath at different temperatures and holding times. After the completion of Tenifer process, a post-oxidation process in salt bath of OXYNIT® solution for 10 min at 350 °C was performed. Tenifer salt bath Nitrocarburizing was performed at 530, 580 & 630 °C temperatures for a duration of 120, 180, and 240 min. respectively. To evaluate the effect of both the thermochemical processes on the mechanical properties and surface morphology of A 105 carbon steel, Taguchi’s L9 orthogonal array was used for experimental design. 18 experiments were conducted. Table 1 shows the experimental design for the current study.

Table 1: Design of experimentation

Experiment no.	Pre heating time (min.)	Soaking time (min.)	Treating temp. (°C)	Post-oxidation temp. . (°C)	Post- oxidation time. (min.)
Untreated	--	---	---	---	---
1	10	120	630	---	---
2	10	180	580	---	---
3	10	240	530	---	---
4	20	120	580	---	---
5	20	180	530	---	---
6	20	240	630	---	---
7	30	120	530	---	---
8	30	180	630	---	---
9	30	240	580	---	---
10	10	120	630	350	10
11	10	180	580	350	10
12	10	240	530	350	10
13	20	120	580	350	10
14	20	180	530	350	10
15	20	240	630	350	10
16	30	120	530	350	10
17	30	180	630	350	10
18	30	240	580	350	10

III. RESULTS

A. Characteristics of the Surface Treatments

1) *Optical Microscopy*: Optical microscopy was done using the METLAB inverted optical microscope. Micrographs were taken at magnifications 200X. After heat treatment process, the changes in microstructure were observed and corresponding layers were analyzed. Two distinguishable zones were present in micrographs; one is the compound zone and other is diffusion zone. Fig. 1 & 2 shows the optical micrographs of TF 9 and TFP 9 specimens at 200X magnifications respectively.

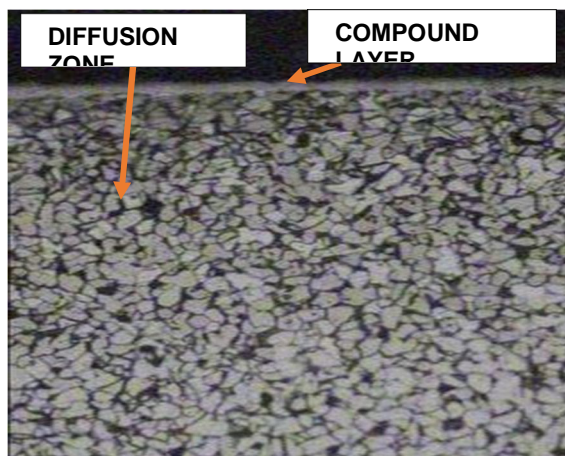
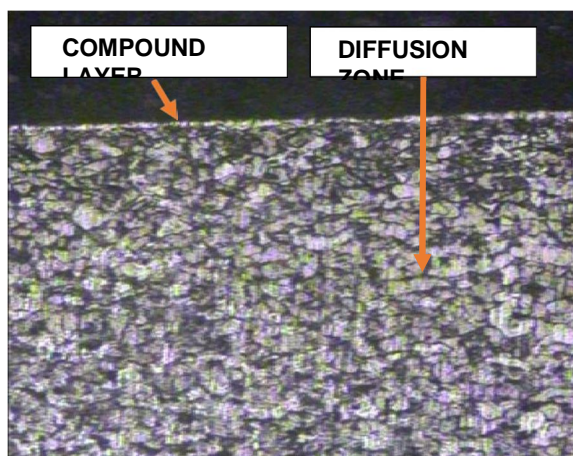


Fig.1: Optical Micrograph of TF9 Specimen at 200 X Fig. 2: Optical Micrograph of TFP9 Specimen at 200 X

The compound layer is an important parcel of Nitrocarburizing treatment, which helps in increasing the wear and corrosion resistance properties of the metal. In addition, the diffusion zone, which is immediate under the compound layer, helps to enhance fatigue strength. Diffusion area is a solution of nitrogen atoms into the α -Fe matrix. A matrix of A105 specimens comprising a polygonal ferrite structure having little amount of pearlite appears clearly in diffusion zone. The white compound layer's thickness varied from specimen to specimen, being thinner in the specimens at lower temperatures and holding times and thicker at higher temperature and holding times. Fig. 3 & 4 shows the optical micrographs of TF 2 and TFP 2 specimens at 200 X magnifications respectively.

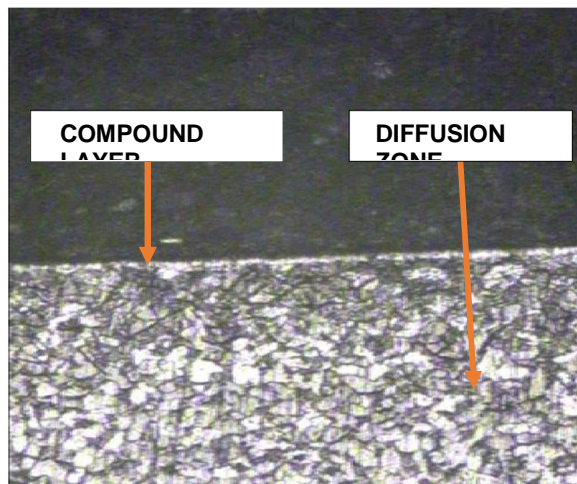
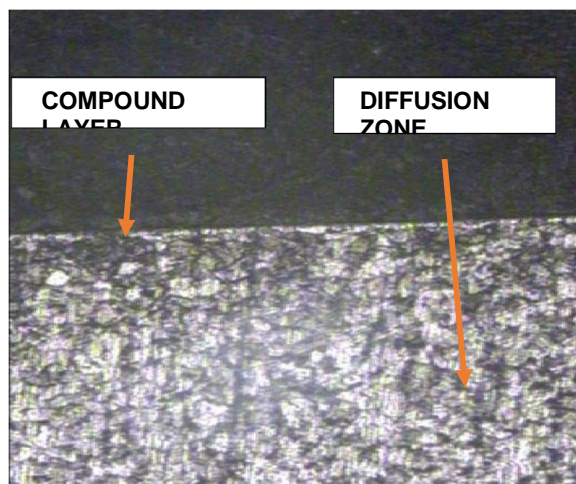


Fig. 3: Optical Micrograph of TF2 Specimen at 200 X Fig. 4: Optical Micrograph of TFP2 Specimen at 200 X

A similar compound and diffusion zones appeared in all the micrographs. It can be said that the compound layer thickness increased with increase in Nitrocarburizing temperature and Nitrocarburizing time. Diffusion zones are measured using the tracking markers and it appeared approximately 350 μ m. This compound layer produced has very high wear resistance properties. The compound layer is most often porous in nature.

This porosity, if in excess, sometimes leads to a compromise between some of the mechanical properties like hardness. But on the other hand it is also very useful in wear resistance properties. The micro holes produced during the process of sliding in a wet conditions retains some of the oil, and kept lubricating the surface in contact time to time, thus enhancing the life of the component by reducing the friction. These micro holes are somewhat filled up by oxide layer produced after the process of post oxidation.

B. Mechanical Properties of A105 Medium Carbon Steel

1) **Micro-Hardness:** The need to increase hardness has a side effect as it decreases the other properties like toughness of the material. Vickers micro-hardness measurements were made on test specimens and the results are shown in Fig. 5. The indentations were measured under the test force of 1.96133N 0.2kgf for a duration of 10 seconds and mean of the three readings were taken into consideration. The micro-hardness readings were taken from the top of the specimens and the diffusion zone hardness is observed from the specimen’s cross-section. It has been observed that by increasing the nitrocarburizing temperature and preheating temperature, an increase in hardness value has been observed. As it can be seen from the Fig. 5 that the micro-hardness has been increased from 146 (untreated) to 383 in TF9 and 390 in TFP9 specimens. It is observed that the hardness of the specimen after post oxidation does not vary much because additional 4-5 μm oxide layer doesn’t make much difference in hardness. The maximum hardness has been found in the 9th specimen in both cases i.e. TF9 and TFP9. As it can be said that hardness increases with nitrocarburizing time and temperature but after 580 °C it starts decreasing. Therefore, 580 °C can be said as the optimized temperature for the process. Fig. 6(a) & (b) shows the micro-indentations taken from cross-section. As it can be clearly seen the size of the indentations goes on increasing towards the core. It can be concluded that the core remains untreated; this is helpful in maintaining the toughness of the specimen, the combination of hard surface and soft core.

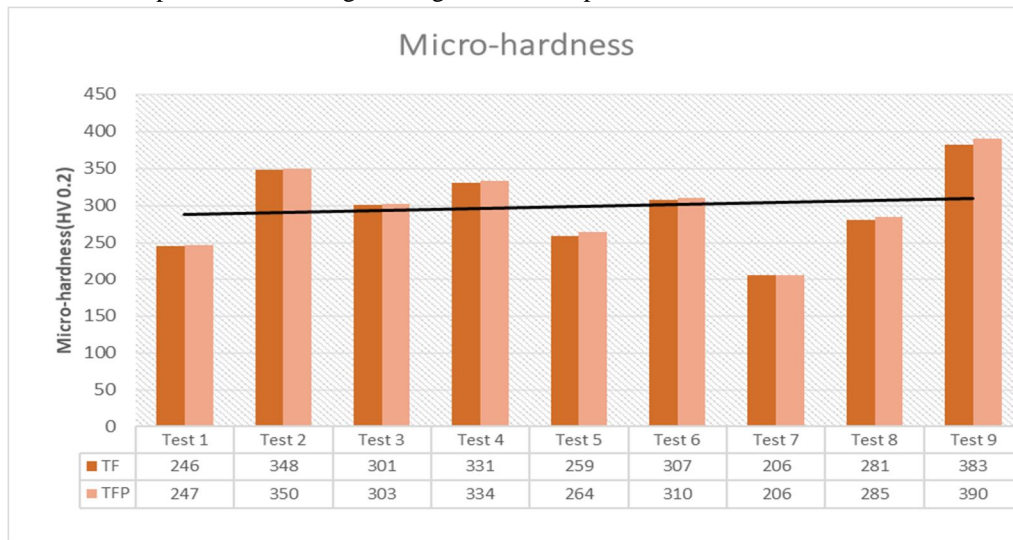


Fig. 5: Experimental conditions vs Microhardness of A105 steel at HV 0.2.

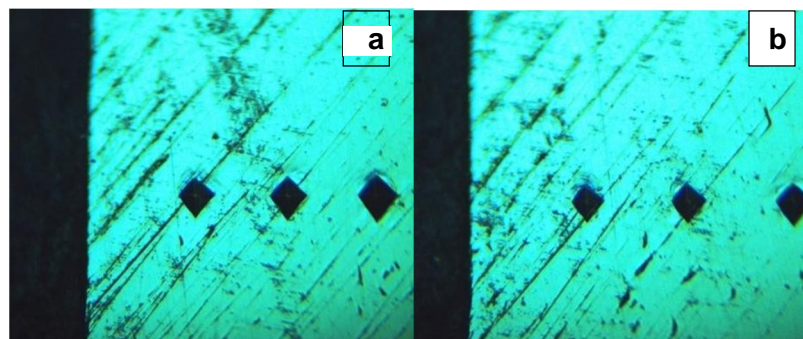


Fig. 6 (a) & (b): Micro-indentations of TF and TFP specimen.

The indentations were taken every 100 μm from each other. Fig. 7 shows a relationship between the distance of indentations and micro-hardness values. Both the TFP and TF processes are depicted on the graph. The values at 0 distance are surface values and other values are cross sectional values.

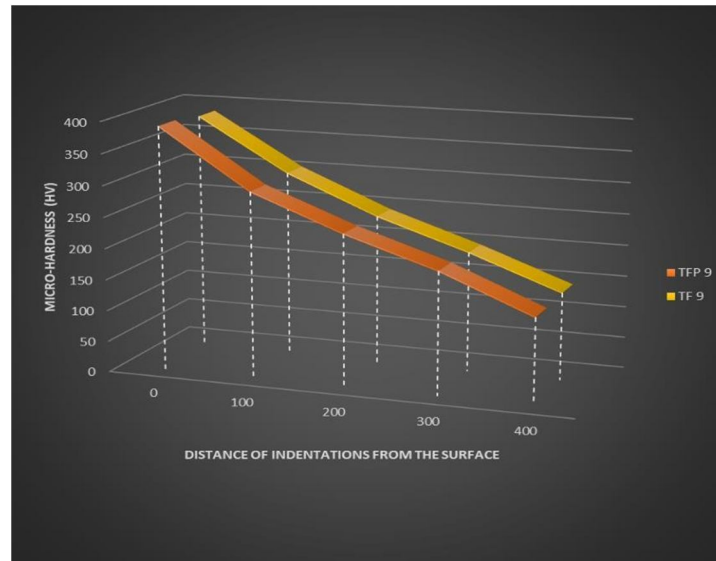


Fig. 7: Micro hardness of TF and TFP specimen Vs cross sectional distance.

C. Wear Properties of A105 Medium Carbon Steel.

1) *Wear Resistance:* Resistance to wear measurements is made on pin-on-disc equipment (Multiple Tribo Tester, TR-201). The testing was performed using track diameter 70 mm at speed 200 RPM, for 20 min under the load of 5 kg. Figure 8 shows the graphical representation of experimental conditions v/s Weight loss (gm). The plot reveals that loss of weight is almost similar in both the processes i.e. TF and TFP. However as compared to untreated specimen the weight loss has significantly decreased. Under the similar wear testing environments, weight loss is decreased with an increase in Nitrocarburizing temperatures and Nitrocarburizing time. This may be related to the hardness, toughness and thickness of the compound zone above the diffusion zone. Minimum wear loss has been recorded at experiment no. 9, where the nitrocarburizing temperature and nitrocarburizing time is maximum. Not much distinguishable weight loss has been observed between two processes as the oxide layer thickness isn't much as mentioned in micro-hardness results.



Fig. 8: Experimental conditions Vs Wear resistance at Track Diameter 70 mm, Speed 200 RPM.

IV. CONCLUSIONS

Current research was conducted to investigate the effects of Tenifer Salt bath Nitrocarburizing technique and post oxidation on the surface properties and mechanical properties of A 105 medium carbon steel.

Following conclusions can be made:

- A. The micro-hardness has been significantly increased after the treatment. The compound layer hardness values were found to be higher than that of the substrate. Furthermore, micro-hardness measurements prove that the higher carbon and nitrogen elemental intensities on compound layer helped increasing the hardness.
- B. Resistance to wear was measured as the weight loss of specimen after each run. Lesser weight loss was observed after the treatment of specimens. Lesser weight loss can also be associated with the high hardness values obtained after the process.
- C. Micrographic images revealed the formation of two distinguished zones i.e. compound zone and diffusion zone. A white layer at the surface is the compound layer and diffusion of the carbon into the surface which can be visible in micrographs is called diffusion zone.
- D. Nitrocarburizing time and Nitrocarburizing temperature played a significant role. However, increasing the temperature beyond 580 °C led to decrease in hardness as well as wear resistance. Similarly, with the increase in nitrocarburizing time, the properties of treated material were enhanced and most optimized values were obtained at 240 min of soaking time. Preheating time hasn't shown much significant changes in the behaviour of the specimens. Oxide layer formed was very less and that's why it does not affect any of the output responses much.

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