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Study of Plasma Nitriding and Nitrocarburizing With Respect To Bearing Applications on M50 NiL

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Abstract: Applications requiring high strength stainless steels are growing rapidly. The aim of this project is to study the effect of plasma nitriding and nitrocarburizing on the surface hardening properties like micro hardness and wear for M50 NiL steel. M50 NiL steel is a low carbon steel with Cr, Mo, V so as to enable it to surface hardening so that it should have soft core to absorb the shocks and hard surface for low wear. It has got high level of fracture toughness i.e., 43Mpa at core. It is used for high temperature applications with good fatigue strength and wear resistance. It is costly alloy because of its alloying contents. The bearing made out of this steel is operated under heavy loads and high speeds in Air craft engine up to temperature 316°C. In this work an MDN 50 NiL bearing steel was plasma nitride and nitrocarburized under different temperatures. The plasma nitrided and nitro-carburized samples were characterized using optical and micro hardness test, and wear tests. The results showed that there are significant micro structural and morphological differences on the formed layers depending on the quantity of nitrogen and methane added to the plasma nitriding atmosphere.

Keywords: plasma nitrocarburizing, plasma nitriding, micro hardness, optical microstructures and wear test.

I. INTRODUCTION

The challenge with most engineering materials is finding something that is soft and enough to be formed into a useful shape and then strong enough to be particular use. Steel, in modern age, has found a vast application in almost every field. India's 33 percent growth in steel production in the last five years was second only to china among the top five producing nations. Steels represent the most important group of engineering materials as they have the widest diversity of applications of any of the engineering materials [1]. Bearing used under high temperature and high speed conditions are required to satisfy a high degree of reliability; therefore, these bearings use materials with cleanliness that is much better than ordinary bearing steel. These highly clean steel materials are resistant against internally propagate cracks that often begins at non-metallic inclusions within steel, resulting in longer life.

Unfortunately, bearings are not always lubricated with clean oil, and operating conditions for bearings with contaminated lubricant may be very harmful. In such severe operating conditions, contamination will dent bearing races before internally propagated cracking occurs, and the resulting indentations can provide propagation points for cracks. In this demanding situation, bearing life is shorter than under internally propagated cracking mode and can also vary depending on a variety of factors including quantity of contamination, and the size and the hardness of foreign contaminants.

When a bearing fails due to contamination, the failure usually occurs suddenly and unexpectedly. The failure mode is particularly critical for safety in aerospace engine bearings since engine trouble during flight can require an emergency landing. Also, for economic reasons, longer bearing life under contaminated lubrication and extended bearing replacement intervals are needed.

To make bearings more resistant against failure caused by foreign contamination in lubrication oil, various techniques are available including improved heat treatment techniques. Surface hardening process for forming a hardened layer on bearing raceway surfaces. Plasma nitriding also referred to as ion nitriding, is a variation of a low temperature nitriding process. M50 NiL used for bearings under high temperature and high speed conditions, and the plasma nitriding process is suitable for these high alloy steels.[2]

Depending on process conditions, low temperature nitriding process can cause a chemical compound layer (also known as white layer) to occur on surface of steel or cause precipitates to occur in grain boundary. These phenomena can adversely affect the fatigue rolling life of the bearing. A certain degree of nitride case depth needs to be achieved such that the surface of bearing components withstands the adverse effects caused by contaminants in lubrication oil.

In service periods, bearing steel parts undergo heavy working stresses. As a result wearing and tearing take place in the body parts. The quality of the steel products depends on the condition of their surfaces and on surface deterioration due to use. Surface deterioration is also important in engineering practice; it is often the major factor limiting the life and performance of machine components. Wear may be defined as the unintentional deterioration resulting from use or environment. It may be considered essentially a surface phenomenon. Wear is one of the most destructive influences to which metal are exposed, and the importance of

wear resistance needs no amplification. The displacement or detachment of metallic particles from a metallic surface may be caused by contact with (1) another metal (adhesive or metallic wear), (2) a metallic or non-metallic abrasive (abrasive wear) or (3) moving liquids or gases (erosive wear). The above wear types are subdivided into wear under rolling friction and sliding friction and, further according to whether lubrication can or can't be used.

II. LITERATURE SURVEY

A bearing is a machine part, which support a moving element and confines its motion. Since there is a relative motion between the bearing and the moving element, a certain amount of power must be absorbed in overcoming friction, and if the surface actually touches, there will be a rapid wear. A bearing consists of an inner and outer member separated either by a thin film of lubricant, or a rolling element. A bearing may have to sustain severe static as well as cyclic loads while serving reliably in difficult conditions. Bearing consists of rolling elements (balls, cylinders, or barrel shape) and rings which forms the raceways. The manufacturing process for the rolling elements involves the high reduction rate, plastic deformation of raw, cast materials into billets. The deformation helps to break up the cast structure and to close porosity. The billets are then reduced in section by further rolling or drawing, heat treated to a softened state and cut into lengths suitable for the manufacture of the balls or rolls; the finished rolling elements are then quenched and tempered, or isothermally transformed, to the required hardness. Bearing rings can be made from seamless tubes produces by hot rolling and similarly hardened by careful machining and grinding to the final dimensions and surface finish.

Bearing steels are made from raw materials that vary in composition and overall consistency. These steels often contain 1% Carbon, 1.5% Chromium, 0.25% Nickel and 0.25% Molybdenum. Bearing steels are one of the most demanded for high quality steel applications. The bearing industry uses different materials for the production of various bearing components. The materials are processed to achieve desirable properties to maximize bearing performance and life. The materials most commonly used are Steel - Chrome steels - Stainless steel - Martensitic Stainless Steel - AISI 440C - ACD34 / KS440 / X65Cr13 - SV30 – Plastics - Ceramic bearings - Hybrid bearings.

A. Effect of Alloying Elements on Bearing Steels

Steel is basically iron alloyed to carbon with certain additional elements to give the required properties to the finished melt. Carbon is the basic element, iron, is alloyed with carbon to make steel and has the effect of increasing the hardness and strength by heat treatment but the addition of carbon enables a wide range of hardness and strength[5].

Manganese is added to improve hot working properties and to increase strength, toughness and hardenability. Silicon is used as a deoxidizing agent in the melting of the steel. Chromium is added to increase resistance to oxidation. Nickel is responsible for a great toughness and high strength at both high and low temperatures and also improves resistance to corrosion. Molybdenum is added to chromium-Nickel austenite steel, it improves the resistance to pitting corrosion, when added to low alloy steel, molybdenum increases the high temperature strength and hardness. Vanadium is a strong deoxidizer and promotes fine grain structure. It helps steel resist softening at elevated temperatures. Vanadium forms hard carbide VC which imparts excellent wear resistance and resistance to tempering.

Bearings used under normal service conditions also experience the effects of vibration, shock, misalignment, debris, and handling. Therefore, the fabrication material must provide toughness, a degree of temper resistance, and microstructural stability under temperature extremes. The material must also exhibit the obvious requirement of surface hardness for wear and fatigue resistance.

Bearing steels are classified as

- 1) *Through hardening steels:* Through hardening steels possess a carbon content of more than 1% and the hardness is uniform throughout the volume. The retained austenite content is high in through hardening steels. They can carry somewhat higher contact stresses, such as those encountered in point contact loading in ball bearing. They have a simpler heat treatment than carburizing i.e., by tempering and quenching. They are mainly used for ball bearing applications and can be used at some higher temperature. *Through hardening processes*
Annealing – Normalizing – Quench and temper – Stress relief
- 2) *Case hardening steel:* Case hardening steels contains a carbon content of less than 0.2% but the hardness varies from case to core. It is highest at the case and minimum at the core. The retained austenite is low as compared to through hardened steel. They have a higher level of core toughness to resist through section fracture under severe fracture conditions. They are better used for roller bearings and used upto a temperature of 163°C. A compressive residual stress to resist bending loads imposed on the ribs of the roller bearings and to reduce the rate of fatigue crack propagation through the cross section.

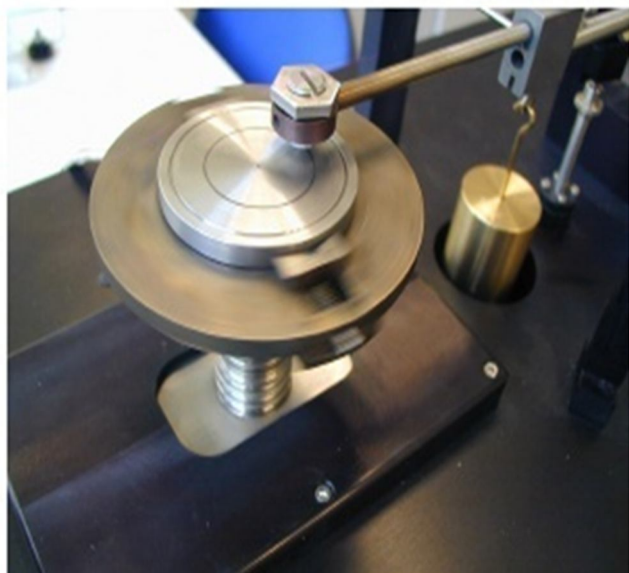
a) *Case hardening processes*

- i). *Diffusion methods:* Carburizing – Carbonitriding – Nitriding – Nitrocarburizing – Boriding
- ii). *Nitrocarburizing:* Nitrocarburizing is a surface-hardening process that uses both carbon and nitrogen, but with more nitrogen than carbon, when compared to carbonitriding. Carbonitriding produces a martensitic case with nitrogen levels less than carbon levels. In contrast, nitrocarburizing involves higher levels of nitrogen with a compound layer. There are two types of nitrocarburizing: ferritic and austenitic. Ferritic nitrocarburizing occurs at lower temperatures in the ferritic temperature range and involves diffusion of nitrogen into the case. Austenitic nitrocarburizing is a more recently developed process with process temperatures in the range of 675 to 775°C. Austenitic nitrocarburizing differs from ferritic nitrocarburizing in the ability for deeper case depths with a better load-carrying capability but may result in greater part distortion because of the higher processing temperatures and the required quenching process [6].
- iii). *Nitriding process* : Different methods exist for introducing atomic nitrogen, or both atomic nitrogen and atomic carbon, into the surface of steel. Various nitriding/nitrocarburizing atmospheres can be included:
 - Gas nitriding and gas nitrocarburizing in which gas (NH_3 - H_2) mixture is used.
 - Liquid nitriding in which salt (cyanate-cyanide) baths are used.
 - Plasma nitriding and plasma nitrocarburizing uses ionized gases.
- iv). *Plasma nitriding:* Plasma nitriding (Direct Current plasma nitriding - DCPN) is based on the creation of gaseous plasma under vacuum conditions. The process gases can be selected in whatever ratio suits the required surface metallurgy. In other words, the formation of the compound layer can be single phase, dual phase, or diffusion zone only. The surface metallurgy can be manipulated to suit both the application and the steel. Ion nitriding has opened the door to many applications that were not possible with conventional nitriding techniques. The first industrial applications of plasma nitriding took place at the beginning of the seventies, but the process is stagnated due to handling and process problems. Wear: Wear can be defined as the progressive loss from one or both surface when two surfaces are in relative motion with each other. Wear occurs in Single-phase wear or Multi-phase wear. Wear mechanisms - Several types of wear can be found in machinery as Abrasive wear - Adhesive and sliding wear - Solid particle erosion - Fretting wear - Corrosive wear - Impact wear. Symptoms of wear - Wear is a characteristic of the system and its surrounding and are influenced by many parameters. So it is necessary to understand the wear mechanism to protect the metal. Laboratory scale investigations, individuals of tribo-systems are carefully controlled and studied the effects of different variables on the wear behavior of the coating. Tribology - The meaning of tribology is the “science of rubbing” of interacting surfaces in relative motion and of related subjects and practices. It also includes parts of physics, chemistry, solid mechanics, fluid mechanics, heat transfer, materials science, lubricant rheology, reliability and performance. The economic aspects of tribology are significant. Surface engineering - Surface engineering means ‘engineering the surface of a material or component to confer surface properties which are different from the bulk properties of the base material. The purpose may be to reduce wear, minimize corrosion, increase fatigue resistance, reduce frictional energy losses, act as a diffusion barrier, provide thermal insulation, exclude certain wave lengths of radiation, promote radiation, electronic interactions, electrically insulate or simply improve the aesthetic appearance of the surface.

III. EXPERIMENTAL PROCEDURE

The material for present study has been finalized by standard chemical composition. The chemical composition analysis is conducted on the sample accordance to standards. The elements like C, Si, Mn, Cr, Ni, V and Mo are analyzed using optical emission spectrometer in accordance to with the standards ASTM E 354. After receiving the test material, 10mmx10mmx12mm samples and 12mmx6mm were cut down by using CNC Wire cut E.D.M for plasma treatment, hardness measurement and microstructural studies. The Carburized M50-NiL VIM-VAR was preheated to 820 – 870° C and soaked for 1 hr/inch thickness. The temperature was then raised at a high rate of heating to 1090 – 1110° C and soaked for five minutes followed by oil quenching. Tempering was carried out at 525 – 550° C for 2 hr/inch thickness followed by air cooling. The process of tempering and air cooling was repeated twice. The plasma nitriding treatment process is carried out by furnace chamber and vacuum chamber. Then the Samples for evaluating the microstructure was prepared by mounting and polishing process. The samples to be examined for optical microscopy were first polished using different grades of emery paper grades like 180, 400, 600, 800 and 1000 (SiC papers). After preliminary polishing, the samples were final polished on the cloth with various grades like 9μ, 3μ, 1μ and 0.5 μ using diamond paste. The samples were then thoroughly cleaned with acetone. The cleaned samples were subsequently etched using an etchant

consisting of nitric acid 10 ml and methanol 100 ml. processes. After etching samples were observed under metallurgical microscope at 100X and 200X magnifications and the resulted microstructures are discussed below.



CNC Wire cut E.D.M



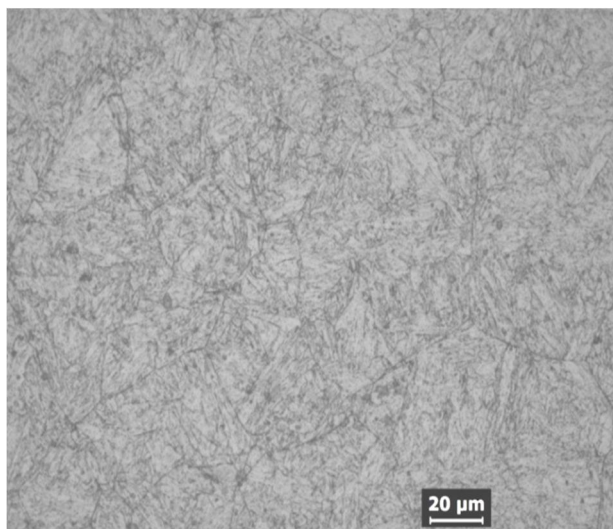
Wear test - Ducom Pin-on-disc apparatus

Then the Micro hardness was measured on all the samples by using the BUEHLER Vickers's micro hardness tester. As a result of the indenter's shape, the impression on the surface of the specimen was a square. The hardness measured across the diameter of the sample and the hardness values was reported. Wear testing was done using a testing pin-on-disc test rig as shown in Figure. Wear results are reported as volume loss in cubic millimeters for the pin and disk separately. The coefficient of friction also determined. Thus wear is studied.

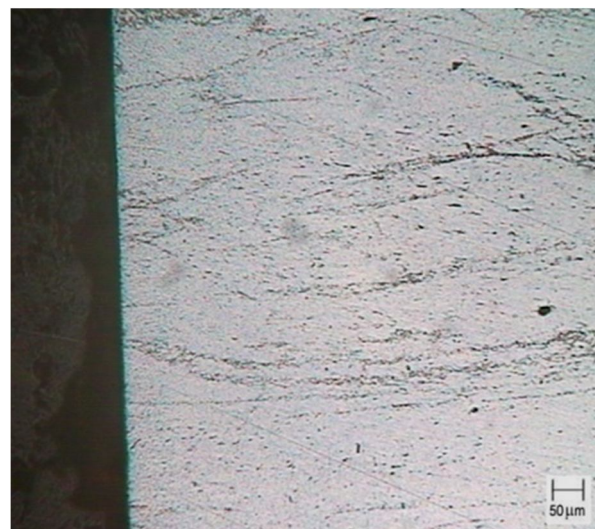
IV. RESULTS AND DISCUSSIONS

A. From optical microscope: Micro structural evaluation

The etched samples were examined under optical microscope with 100X, 200X, 500X magnification, and microstructures are captured. The low magnification (200X) optical images of the samples plasma nitrocarburized at 450, 500 and 550 °C are provided in following figures. All samples are nitrocarborised for 6 hr. As expected the thickness of the nitrocarburized layer increases with increase in the nitriding temperature. There is formation of compound layer only above 500°C. Higher case depth in the PN 550 sample is due to the fact that at higher nitriding temperature the rate of nitrogen diffusion is high.



Microstructures sample in etched condition.



Microstructure of Plasma Nitro Carburizing sample at 450°C with no compound layer



(a)

(b)

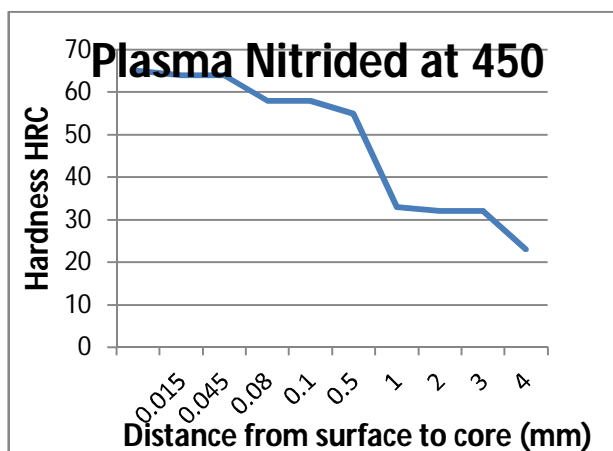
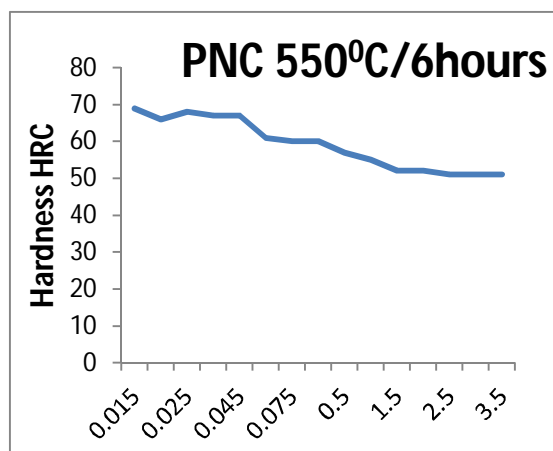
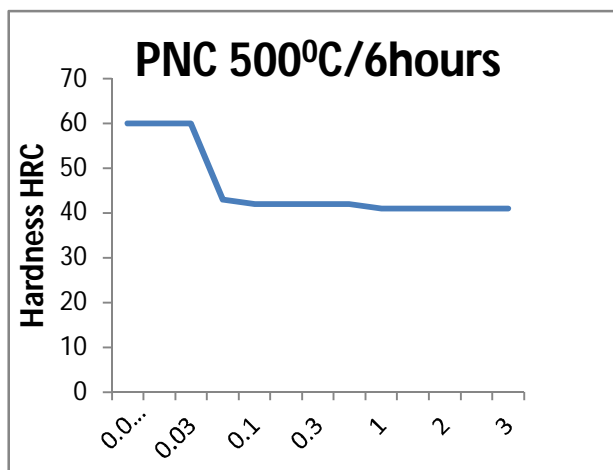
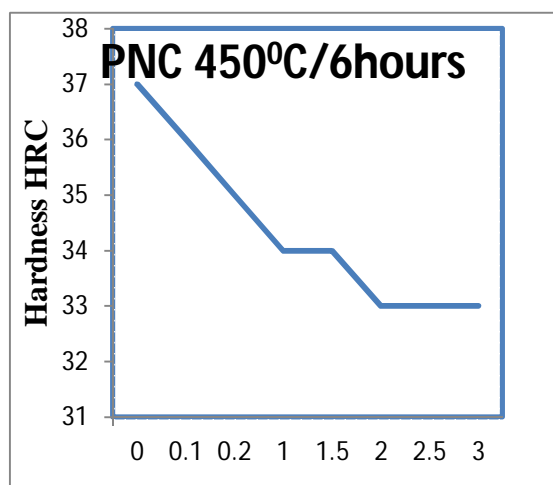
Microstructures of Plasma Nitrided samples in etched condition at 200X mag.

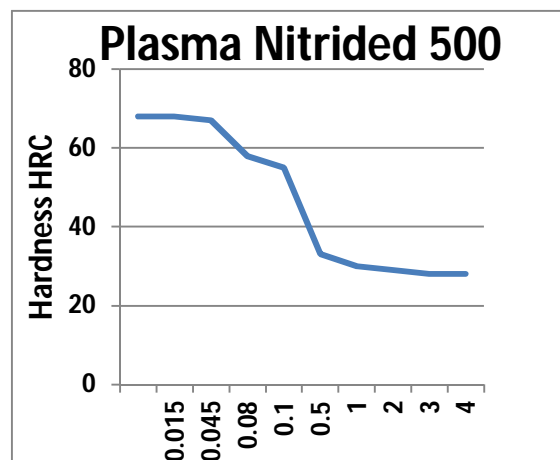
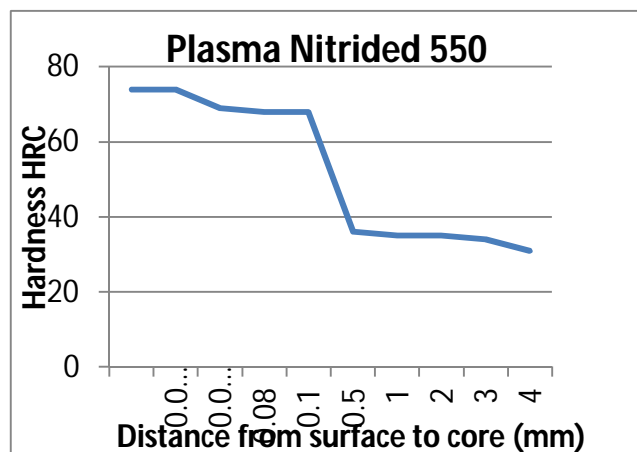
at 500°C with nitrided compound layer thickness is 142 μm

at 550°C with nitrided compound layer thickness is 176 μm

B. Micro hardness

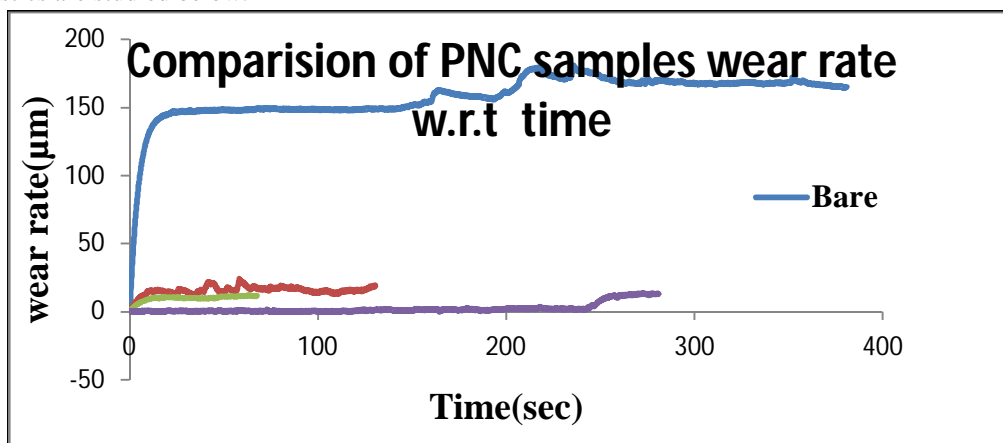
After nitriding treatment micro hardness test is conducted on micro Vickers hardness equipment. The variation of microhardness as function of distance from the surface for the samples plasma nitrocarburized at 450, 500 and 550 °C is shown in following Graphs.





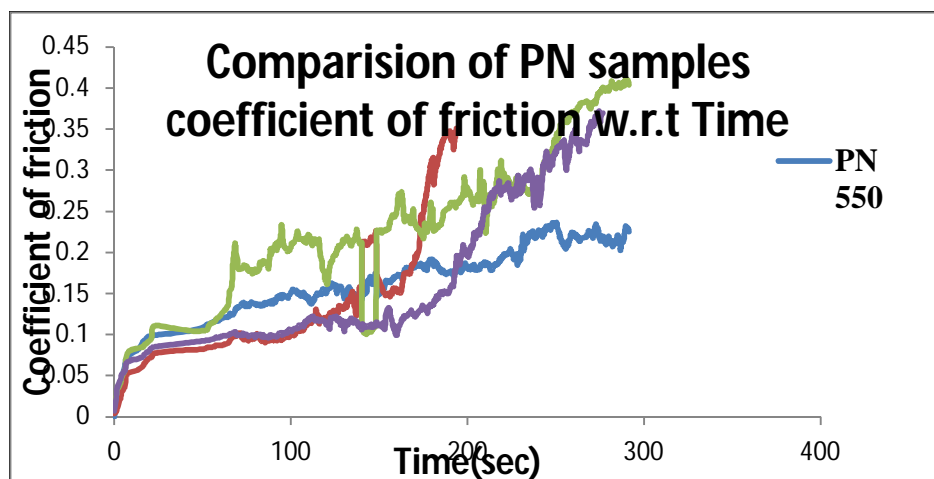
C. Wear rate and friction behavior:

Wear rate, coefficient of friction with respect to time are graphically represented by Winducom software for different samples and its wear characteristics are studied below:



Plasma nitrocarburized samples of different temperatures, wear rate w.r.t time results comparison

The above figure shows that the samples with higher plasma nitrocarburised layer i.e., at 550°C has lower wear rate. A high wear rate is observed in bare material i.e., 140 μm within few seconds. The fluctuations indicate that the material is attaining wear resistance.



Plasma nitrided samples of different temperatures, coefficient of friction w.r.t time results comparison

V. CONCLUSIONS

- A. Microstructural observation, with increase in temperature compound layer thickness is increased. For plasma nitriding treatment, maximum nitriding layer thickness is $176\mu\text{m}$ at 550°C and $142\mu\text{m}$ at 500°C and no layer is observed at 450°C . For plasma nitrocarburizing treatment, maximum nitrocarburized layer is $230\mu\text{m}$ at 550°C and $57\mu\text{m}$ at 500°C and no layer is observed at 450°C .
- B. With increase in temperature of nitriding and nitrocarburizing hardness increased which shows that at high temperature carbon and nitrogen has got more tendencies to diffuse in to the bare material, which gives rise to formation of precipitates of nitrides.
- C. Plasma nitrided material has high hardness against Plasma nitrocarburized material. The maximum nitriding surface hardness is up to 1083HV and nitrocarburized surface hardness is up to 964HV. The hardness profiles shows decrease from maximum surface hardness to core hardness.
- D. Wear rate of plasma nitrocarburizing shows the minimum wear rate of $2\mu\text{m}$ at 550°C upto 250sec which can be considered as optimised condition for plasma nitrocarburizing whereas wear data of plasma nitriding shows the minimum wear rate of $10\mu\text{m}$ at 550°C upto 400sec which can be considered as optimized condition for plasma nitriding.
- E. It is to be concluded that plasma nitrocarburizing has superior properties than plasma nitriding in terms of compound layer formation, wear resistance and low coefficient of friction.

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