

Synopsis of the thesis

**Studies on Rotating-Bending Fatigue Behavior of Surface Engineered
Low-alloy Steel Shaft Materials**

Submitted for the award of the degree

of

DOCTOR OF PHILOSOPHY

in

(Metallurgical and Materials Engineering)

by

U. N. Puntambekar
Department of Metallurgical and Materials Engineering,
Faculty of Technology & Engineering,
M. S. University of Baroda,
VADODARA

2013

Synopsis

Studies on Rotating-Bending Fatigue Behavior of Surface Engineered Low-alloy Steel Shaft Materials

Introduction:

AISI 4340 steel, a member of the family of low alloy steels, constitutes a very important engineering material employed in the manufacture of different structural components which encompass automotive crankshafts and rear axle shafts, aircraft crankshafts, connecting rods, propeller hubs, gears, drive shafts, landing gear parts and heavy duty parts of rock drills, among others [1]. It is a heat treatable, medium-carbon type of low alloy steel having superior mechanical properties with nickel, chromium and molybdenum as the principle alloying elements. After an appropriate surface modification treatment, this steel is recommended for applications involving severe conditions of wear, corrosion and cyclic loading.

Surface modification or surface engineering deals with 'engineering the surface' of a material or component to confer the surface properties, that are different from the bulk properties of the base material [2]. The purpose of surface engineering may be thus to reduce wear, minimize corrosion, increase fatigue resistance, act as a diffusion barrier, provide thermal insulation or simply improve the aesthetic appearance of the surface. using different surface modification or coating techniques.

Some of the surface modification or coating processes developed industrially to improve the wear, fatigue and corrosion resistance of a steel include processes like hard chrome plating, thermal spray coating, heat treatments like carburizing, nitriding, carbonitriding and induction hardening; methods involving non-uniform plastic deformation like shot peening, or methods such as ion implantation and chemical or physical vapor deposition, etc. However, it is realized that the improvement in surface properties of steel by means of coatings (so as to increase its performance against wear and corrosion) often significantly deteriorates its fatigue properties. It is found that quite a few of these surface modification techniques lead to reduction in fatigue strength, but for the exception of techniques like nitriding (based on gas, salt bath or plasma energy) that are known to enhance the fatigue strength. For instance, the thermal spray technique has emerged as a feasible alternative to replace electrolytic hard chromium (EHC) plating in a number of engineering applications. This is typically used to deposit wear and corrosion resistant coatings such as ceramic and metallic layers on materials. In thermal spray, molten or semi-molten materials are sprayed onto the surface of a substrate such as steel by means of the high temperature, high velocity gas stream, producing a dense coating. However, a major concern as regards the performance of components coated by thermal spray and subjected to cyclic loading both in air and in corrosive environments, is the adverse effect of such coatings on the fatigue properties of the substrate/base material.

Failure due to fatigue is a result of initiation of a crack, by and large at the surface of the component, and its subsequent propagation as a consequence of cyclic loading of the component. It is found that a component fails due to fatigue under cyclic loading condition at stress levels much lesser than even its yield strength. About 70% of total failures of cyclically loaded components are due to fatigue. Thorough understanding of fatigue behaviour is therefore essential for components subjected to constant variable amplitude loading.

Improvement in fatigue resistance of components can be derived primarily by decreasing the surface cyclic tensile stress or by increasing the surface yield stress, thereby increasing the resistance to fatigue crack nucleation. In practice this can be ensured by changing the characteristics of the surface region of a component by changing the composition and/or its surface microstructure using chemical conversion coatings and/or thermo-mechanical processes so as to induce residual compressive stresses at the surface leading to enhancement in the fatigue life.

Hard Chrome Plating

Hard chrome plating is a commonly used surface treatment for steel substrates because of its high corrosion & wear resistance. The success of electrolytic hard chrome (EHC) plating relies on the fact that it is cheap and simple; coatings can be obtained in a wide range of thickness, possess an excellent bond strength. Major advantages of hard-chrome plating are excellent surface finish, high hardness with a degree of toughness, excellent abrasion resistance, very low friction co-efficient, stable and non-corrodible surface etc.

Effect of chrome plating on fatigue life of steel substrates has been a subject of study for dynamical applications. Fatigue life of steel subjected to hard chrome plating has been found to depend on residual stresses in the coating, microcrack density and the nature of the coating/substrate interface [3]. In most of the cases, the fatigue life of steel substrate is found to have negative effect. Despite of this, hard chrome plating has been selected as a surface treatment in this investigation being one of the most frequently used and oldest surface treatments.

Thermal Spraying

Thermal spray processes are finding applications in the industries for its corrosion resistance and tribological properties. They are replacing the existing coating techniques such as chromium plating, which are otherwise also being phased out because of their environmental concerns. Effect of thermal spray coatings on fatigue life of various steel substrates has been studied, recently. Reports on effect on fatigue life of plain carbon (medium) steel, low-alloy steel grades 4140 & 4340 [3,4,5] are found in the literature. It is found that fatigue life decreases in most of the cases [4,5], while in few cases no effect on base material's fatigue life is observed [3], or it is found to increase. Thus the present understanding of fatigue behavior for AISI 4340 steel subjected to thermal spray coatings is inconclusive and so further study is required to be done in this area.`

One of the methods used to enhance the fatigue life of the rotating components that are subjected to cyclic loading, is nitriding. Nitriding is a group of surface-hardening processes based on the thermo-chemical principle involving the introduction of nitrogen into the surface of the steel. Commercially used various nitriding processes are gas nitriding, liquid or salt bath nitriding and plasma or ion nitriding. Nitriding produces a strong and shallow case with high compressive residual stresses on the surface of steel so as to improve its fatigue life. Rotating bending fatigue tests on nitrided steel samples show higher fatigue limit than their un-nitrided counter parts.

The effect of nitriding on fatigue life has been extensively studied by several researchers for a variety of steels including AISI 1045 steel [6], Cr-Ni-Mo steel [7,8], AISI 4340 steel [9], AISI 4140 steel [10-13], Cr-Mo steel [14] & Cr-Mo-V steel [15]. It has been found that the nitriding improves the fatigue strength of the material and increases its fatigue limit.

Nitriding is usually performed at temperature between 500 and 560 °C where the structure of the steel is still ferritic. Nitrogen diffusion modifies surface and near surface microstructure producing hard layers with altered mechanical properties. The layer on the nitrided steel consists of two parts, compound layer and diffusion layer. The outer thinner layer consists of mainly γ -Fe₄N and/or ϵ -Fe_{2,3}N inter-metallic phases. It is commonly called as ‘compound layer’ [16] or ‘white layer’ because of its white color after Nital etch on the microscopy and has a thickness of some micrometers. Thickness and phase content of compound layer depend on the treatment parameters such as specific gas composition, time and temperature of nitriding etc. [17-18]. The layer, beneath the compound layer, is known as ‘diffusion layer’ which consists of mainly interstitial atoms in solid solution and fine coherent nitride precipitates when the solubility limit is reached. The diffusion layer is relatively thick and strong. It is in the subsurface of a steel component with a gradual reduction in the hardness and nitrogen concentration towards the core, resulting in a diffuse case–core interface. The thickness of the diffusion layer is also known as ‘case depth’.

However, the conventional nitriding processes based on gas or salt bath have certain limitations being messy and less environment-friendly and hence plasma or ion nitriding process is preferred. Plasma nitriding is an advanced surface modification technology which has experienced substantial industrial development over the past 30 years [19]. It is used for increasing fatigue strength, surface hardness, and corrosion or wear resistance of industrial components for a wide variety of applications. Some of the advantages of the use of the plasma energy for nitriding in comparison with conventional gas or salt bath nitriding are: faster nitrogen penetration, cleanliness and economical aspects, minimum distortion of work piece as well as easier control of compound and diffusion layer thickness. The requirement of lower process temperatures, shorter process periods and suppressed compound layer formation are said to be the other advantages of ion nitriding [20].

There are three ways in which the surface treatment influences fatigue strength; firstly, by affecting the intrinsic fatigue strength of material at the surface, secondly by introducing or removing residual stresses in the surface layers and thirdly, by introducing or removing irregularities in the surface which act as stress raisers [21]. Plasma nitriding enhances fatigue strength by first two mechanisms stated above.

Plasma nitriding, is a process of surface hardening using glow discharge technology to introduce nascent (elemental) nitrogen into the surface of a metal part for subsequent

diffusion into the material. Under high vacuum, high-voltage electrical energy is used to form plasma, through which nitrogen ions are accelerated to impinge on the workpiece. Nitrogen ions are combined with alloying elements such as chromium to form a fine dispersion of alloy nitrides. This ion bombardment heats the workpiece, cleans the surface, and provides active nitrogen. Nitrogen diffusion modifies surface and near surface microstructure producing hard layers namely, compound layer and diffusion layer, as discussed earlier with altered mechanical properties.

The present work deals with the studies on fatigue behaviour of AISI 4340 steel subjected to various surface modification treatments like hard chrome plating, thermal spraying and plasma nitriding. The surface treated specimens were characterized for coating thickness, surface hardness and/or hardness profile, phase identification using XRD, optical and SEM fractography, microstructure and the effect of coating thickness on fatigue life and finite element analysis (FEA).

Scope of Work:

The review of literature indicates that researchers have established strong correlations between the number of non-material factors (such as the numerical value of the applied mean stress, magnitude of stress concentration, operating environmental temperature, size of the component, fabrication state, surface finish of component, etc) and the fatigue life of a material under investigation. However, the effect of material related factors such as the fatigue strength modification factor and cycle modification factor with respect to the surface engineering techniques used have not been fully studied.

In recent years, a number of surface modification treatments have been developed for steels used as shafts materials, including AISI 4340 steels. Since, the fatigue strength modification and cycle modification factors for these treatments are not available in open literature, machine designers are routinely forced to take recourse to large factors of safety in their design calculations resulting in highly non-optimal, costly, cumbersome designs. In view of this, it is contemplated in the present research work to examine the effect of three major surface modification techniques viz. hard chrome plating, thermal spraying with alumina, & plasma nitriding on the fatigue strength of AISI 4340 low-alloy steel in light of these factors.

An attempt has been made to modify the surface characteristics of the hardened & tempered AISI 4340 steel specimens using aforesaid surface modification treatments. The coated / surface modified specimens in each case were subjected to rotating bending fatigue testing followed by their characterization using techniques like X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), micro-hardness measurements and microstructural examination, etc. Basquin type power law relationship for alternating stress amplitude versus cycles to failure was derived in each case. Based on these plots, the surface treatment modification factors have been arrived at.

It also appears from the literature reviewed that for plasma nitriding process the effect of thickness of the compound layer (popularly known as white layer) on fatigue life of steel has not been fully understood. In view of this, during present investigation an attempt has also been made to examine the effect of variation in thickness of compound layer on fatigue strength of AISI 4340 steel. Further, the results obtained are correlated with the underlying physical processes associated with the various treatments studied.

Experimental work and results:

Specimen preparation

The test specimens were prepared from the 18.7 mm dia. AISI 4340 steel wire rods by machining to grip diameter of 17 mm and gauge diameter of 12 mm containing a semi-circular notch of depth 1.5 mm and root radius of 1 mm. This translates into a stress concentration factor of 2 (at notch root), as computed using analytical elastic stress analysis formulae. Figure 1 shows the schematic of the test specimen with dimensions duly marked over it.

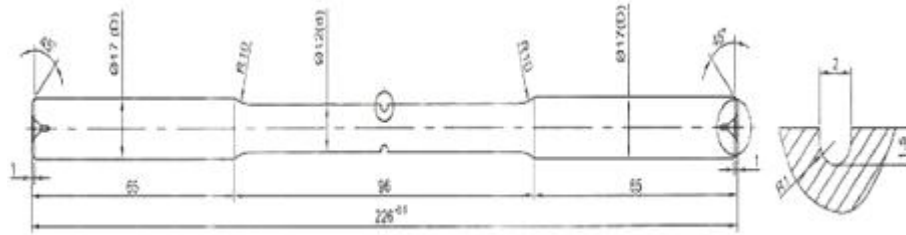


Figure: 1 Schematic of the fatigue test specimen

Before carrying out the surface modification treatments on these specimens, they were subjected to hardening & tempering. Specimens were preheated at a temperature of 500 °C, followed by hardening at 850-860 °C and quenching in oil. The as-hardened specimens were subsequently tempered at 520 °C \pm 10 °C to obtain a hardness of 32 \pm 2 HRC. The chemical composition of material and tensile properties of the heat treated specimens are given in Table-1.

Table: 1 Chemical composition and mechanical properties of AISI 4340 steel

Chemical composition		Mechanical properties	
Element	Composition, wt %	Parameter	Value
Carbon	0.396	Yield Strength σ_y , (MPa)	1073
Silicon	0.293	Ultimate Tensile strength σ_u , (MPa)	1142
Manganese	0.629	% Elongation, δ	13
Phosphorous	0.013	% reduction in area, ΔA	44
Sulphur	0.018	Hardness, HRC	32 \pm 2
Nickel	1.425		
Chromium	0.996		
Molybdenum	0.205		

Surface modification treatments

Heat-treated specimens were then subjected to various surface modification treatments such as hard chrome plating, thermal spray with alumina and plasma nitriding.

Hard-chrome plating:

Pre-treatment surface cleaning of specimen was done by acid pickling (in hydrochloric acid) followed by degreasing. Then the hard-chrome plating was done using an

electro-chemical process. The chrome-plated specimens were subjected to a baking treatment at 150 °C for removal of hydrogen diffused, if any, to avoid hydrogen embrittlement. Process parameters (current & time) were set by conducting plating of dummy samples initially and taking thickness measurements on the same.

Thermal Spraying:

Thermal spraying of alumina powder was done using a flame-spray gun. An oxy-acetylene flame was used as a heating source. A bond-coat of a nickel- base alloy was applied to ensure proper bonding of alumina coat. Powders to be coated were fed into the flame through a container mounted on the gun itself. The specimen was gripped at two ends in a fixture and rotated slowly. At the same time, the flame was directed over the specimen surface and moved over its length to ensure uniform application of coat all over the surface. This also ensured that temperature rise of specimen (during spraying) did not exceed 150 °C. Process parameters (fuel gas flow, time & feed rate of powders) were set by conducting coating of dummy samples initially and taking thickness measurements on the same.

Plasma Nitriding:

Plasma nitriding was carried out in a 500 mm diameter and 500 mm height bell shaped stainless steel vacuum chamber. The specimens were mounted on the sample holder and the chamber was evacuated to a base pressure of 0.05 mbar. The specimens were first sputter cleaned using N₂-H₂ gas mixture in 25% : 75% ratio and the pressure was raised to 1 mbar. Plasma was generated using a D.C. pulsed power supply. After completing the sputter cleaning process, the mixture of nitrogen and hydrogen gas was introduced in the chamber. Plasma nitriding of heat treated specimens was carried out to obtain three different categories of specimens with respect to compound layer thickness. The respective categories so developed are: specimens without any compound layer, specimens with less than 10 micron thickness of compound layer and the specimens with more than 10 micron thickness of compound layer, respectively.

In order to obtain variation in thickness of compound layer with similar diffusion zone, plasma nitriding was carried out in two batches with different operating parameters. While, the plasma nitriding temperature (560 °C), time (24 hrs) and gas pressure (4.6 mbar) were kept constant for both the batches, the composition of nitriding gas mixture was varied. The gas mixture compositions used were in the ratio of 80:20 and 65:35 of H₂ & N₂ gas, respectively. Accordingly, for higher nitrogen content (35 % vol) of 65H₂:35N₂ gas mixture, the compound layer thickness increased upto 20 micron, while it was 10 micron for lower nitrogen content (20 % vol) in a gas mixture of 80H₂:20N₂. Half of the specimens from the lot of 10 micron thickness were subjected to grinding to remove compound layer.

Rotating bending fatigue testing

In the present work, the methodology of smooth bar rotating bending fatigue testing, using a R. R. Moore type rotating bending fatigue testing machine is adopted. Essentially, the specimen is supported as a four point (pin hinged) beam. Two of the support points are the points of load application, while the remaining two points are reaction support points. When the specimen is rotated, the bending moment generated by the four points loading results in the creation of an alternating, fully reversed, compression-tension stressing of

fibers located away from the neutral axis of the specimen. The specimen was rotated at 2820 RPM for fatigue testing purposes. Figure 2 shows rotating bending fatigue machine used in these experiments.



Figure: 2 Pictorial view of rotating bending fatigue machine

Characterisation of surface- treated specimens

Coating thickness:

Coating thickness of specimens of three different surface treatments was measured microscopically on the transverse sections. Measurements were made in un-etched condition for hard-chrome and thermal sprayed specimens and for those of plasma nitriding were made in etched condition. Table 2 gives the data on average coating thickness of the specimens measured using an optical microscope.

Surface hardness:

Surface hardness of the specimens was measured by using an ultrasonic hardness tester.

Table: 2 Average thickness / depth of surface for surface treated specimens

Category	Average thickness /depth of hardened layer, μm	Hardness
Untreated specimen	N/A	32 \pm 2 HRC (Equivalent to 320 HV)
Hard chrome- plated	55 to 60	700 -750 HV
Thermal sprayed- Alumina	475 - 525	1100 -1200 HV
Plasma nitrided	400	700-750 HV

Microhardness profile:

Plasma nitrided specimens were also subjected to determination of micro-hardness profile using a microhardness tester at a load of 100g. Figure 3 gives the corresponding plots. The total case depth of nitriding was estimated using this data. Considering 400 HV

as threshold core-hardness, a total case-depth of 400 μm was observed in almost all the three categories.

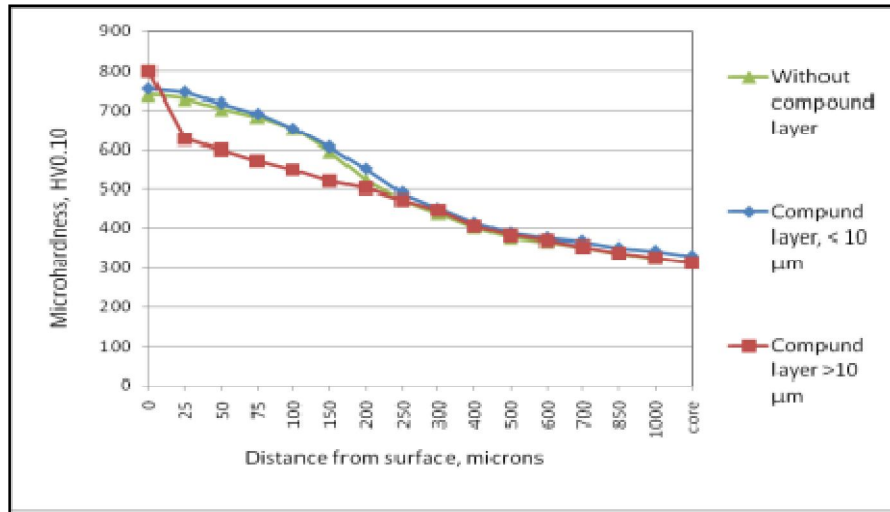


Figure 3: Microhardness profile for nitrided specimens for three different categories

The surface hardness of specimens with higher compound layer thickness ($>10 \mu\text{m}$) is higher than the surface hardness of the other two categories. While, there is a steep decrease in the hardness below the surface for more than $10 \mu\text{m}$ category, the hardness for the other two categories gradually decreases (smooth profile of curve) from surface to core. The hardness profile of the region below about $250 \mu\text{m}$ is similar for all the three categories.

This can be attributed to the nitrogen concentration gradient from surface (i.e. the compound layer) to diffusion zone. When the compound layer thickness is higher, the layer is rich in nitrogen as well as nitrides formed, and so the hardness value is higher. This permits diffusion of less amount of nitrogen in the region below compound layer, and so the hardness values drop drastically below the compound layer.

XRD analysis:

The X-ray diffraction method was used for the determination of phases present in the various surface treated specimens using $\text{Cu-K}\alpha$ radiation within the 2θ range of 35 to 90° . For plasma nitrided specimens emphasis was laid on determination of presence or absence of the $\gamma\text{-Fe}_4\text{N}$ and $\epsilon\text{-Fe}_{2,3}\text{N}$ phases in compound layer. Figure 4 (a) to (d) show the XRD profiles for the untreated and plasma nitrided specimens of three different conditions.

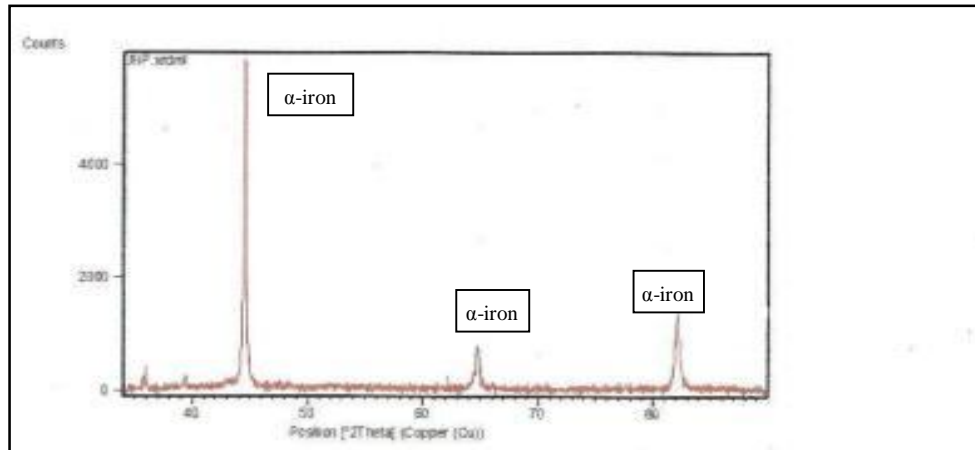


Figure: 4 (a) XRD profile for untreated specimen

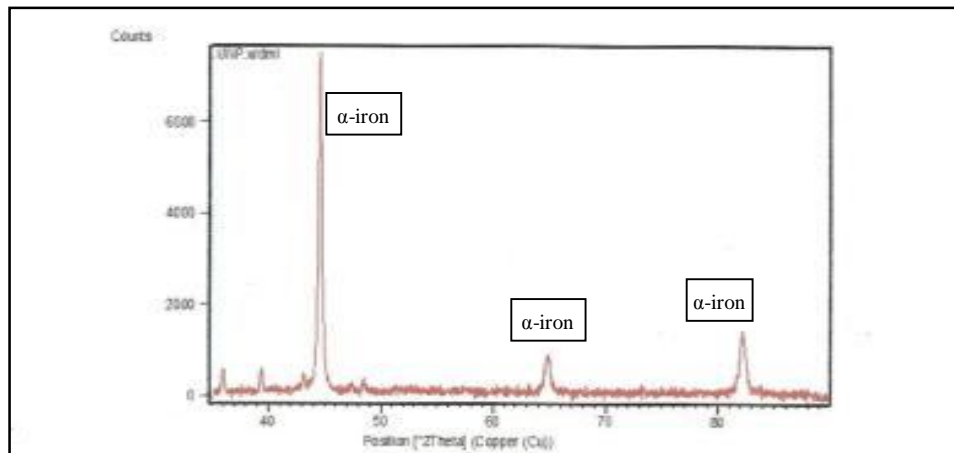
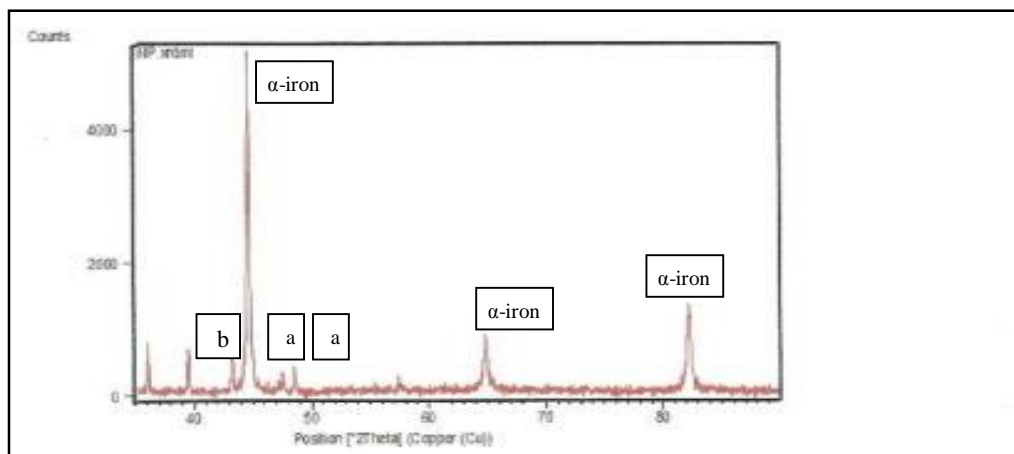
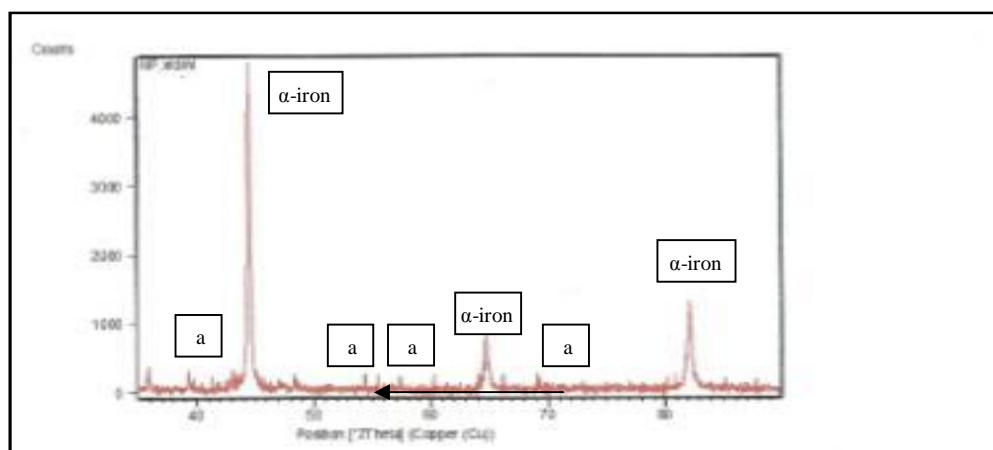


Figure: 4(b) XRD profile for plasma nitrided specimen - without compound layer



a – Indicates Fe₄N compound, b- Indicates Fe₃N compound

Figure: 4 (c) XRD profile for plasma nitrided specimen - with compound layer less than 10 μm .



a – Indicates Fe₄N compound

Figure: 4(d) XRD profile for plasma nitrided specimen - with compound layer more than 10 μm .

Fractography:

The fracture surface of fatigue tested specimens was examined under stereo microscope from macrofractography view point. The study involved the identification of nature of fracture as regards the high-stress low-cycle or low-stress high cycle fatigue, number of crack origins & their location and overall assessment of the fracture surface. From the fractographs it is observed that the central - final fracture core is more or less symmetrical with respect to the axis of the test sample.

From the fractographic point of view, all the specimens can be divided into two groups namely,

Group - I: Chrome-plated & thermal sprayed (alumina) categories

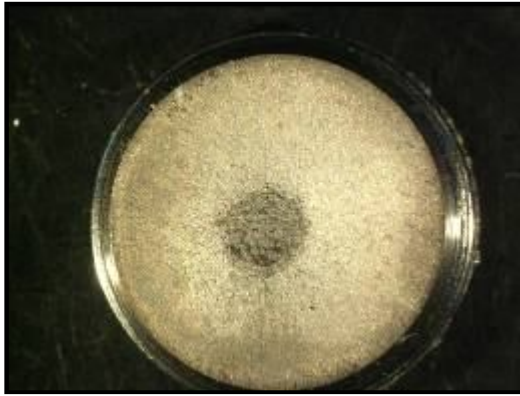
The important fractographic features of this group are presence of ratchet marks or radial marks starting from surface indicating the initiation of cracks from multiple crack origins typically under the influence of high stress concentration prevailing at surface.

Group - II: Untreated and Plasma nitrided categories

There is total absence of multiple crack origins indicative of no high stress concentration at surface. Fatigue cracks usually initiate from the materials surface. However, for materials hardened by plasma nitriding, the crack tends to initiate in subsurface rather than surface in high cycle fatigue. This is due to compressive residual stress formed in the diffusion layer, resulting in better resistance to cyclic slip. [22].

Figures 5 (a) to (f) show the macrofractographs of the untreated, hard chrome plated, thermal sprayed and plasma- nitrided fatigue test specimens of all categories.

The specimens were also subjected to SEM fractography in order to study the microscopic features of fracture surface of the specimens particularly for a closer understanding of the interface between coating and substrate. The SEM fractographs shown in Figures 6 (a) to (e), further confirm that hard chrome-plated & thermal- sprayed specimens show multiple cracking initiating from the surface. Whereas, untreated and plasma nitrided specimens (all types) do not show any multiple cracking from the surface.



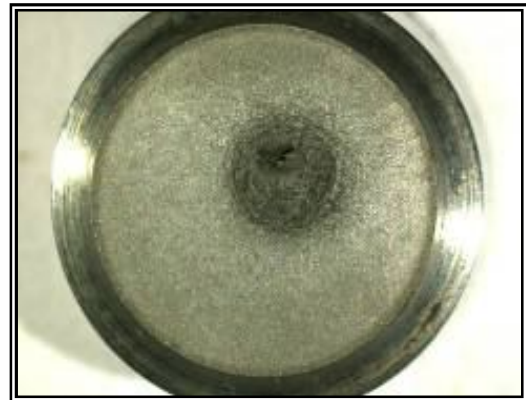
(a)



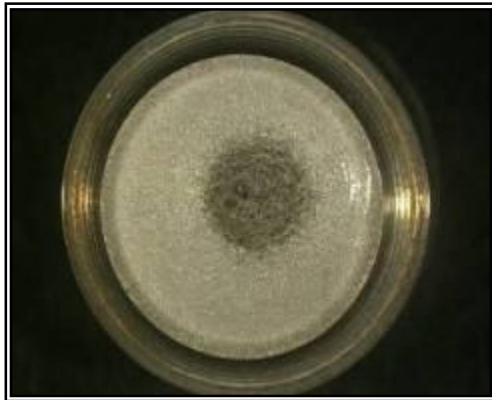
(b)



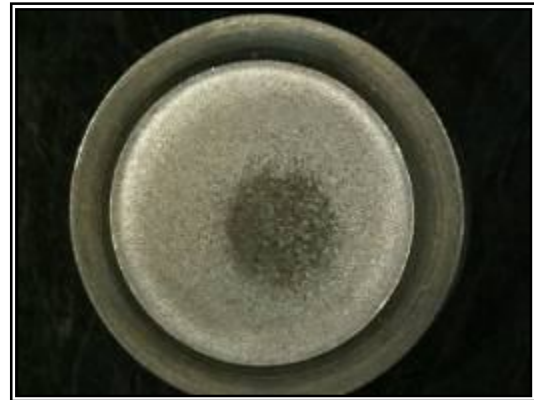
(c)



(d)



(e)



(f)

Figure: 5 Fractographs showing fatigue fracture surfaces for
 (a) Typical untreated specimen
 (b) Hard chrome- plated specimen
 (c) Thermal sprayed - alumina coating and
 (d) to (f) Plasma- nitrided specimen without compound layer, with
 compound layer of $< 10 \mu\text{m}$ and, with compound layer $> 10 \mu\text{m}$

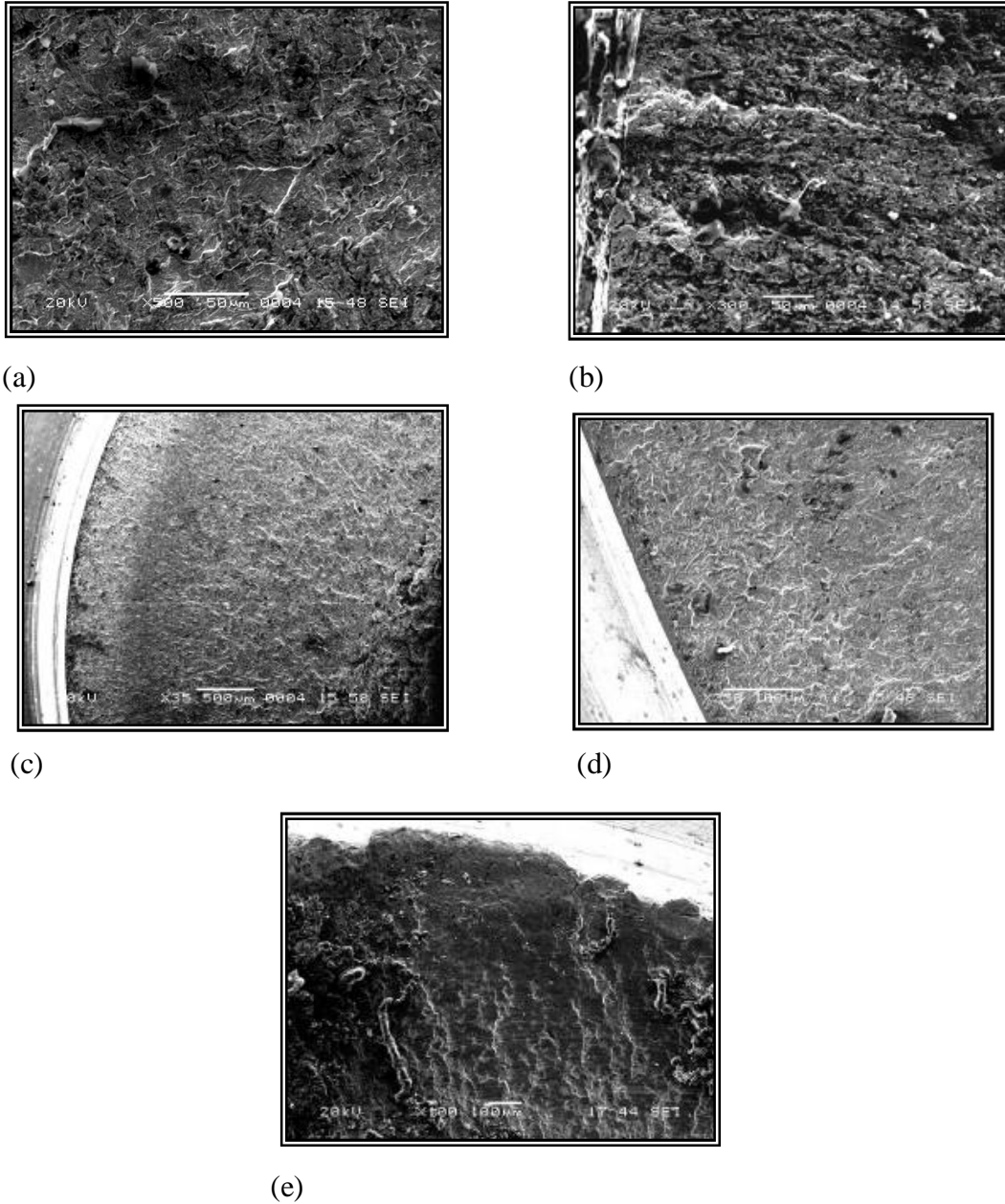
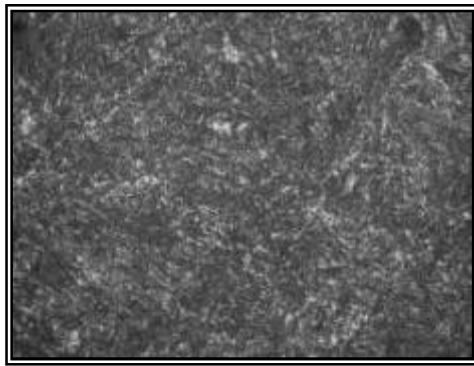


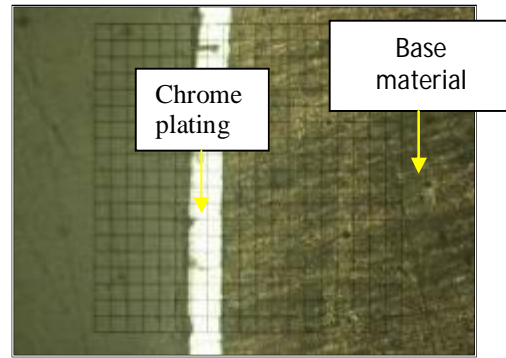
Figure: 6 SEM fractographs showing fatigue fracture surfaces for
(a) Hard chrome- plated specimen
(b) Thermal sprayed - alumina coating and
(c) to (e) Plasma- nitrided specimen without compound layer, with
compound layer of < 10 μm and, with compound layer > 10 μm

Microscopic examination:

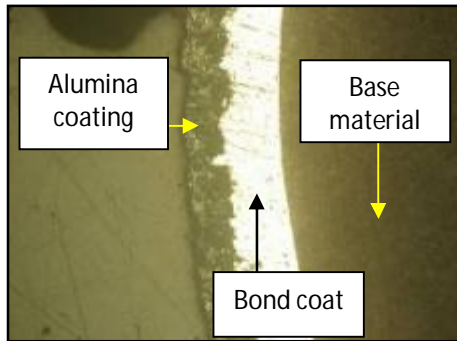
Microscopic examination of surface treated specimens was done on transverse section. Uniformity of coating and bonding between the coated layer & substrate were examined. For plasma nitrided specimens the compound layer and diffusion zone were examined in etched condition (using 2% Nital as etchant). Thickness of the compound layer was also estimated. Figure 7 (a) to (f) show photomicrographs of these specimens.



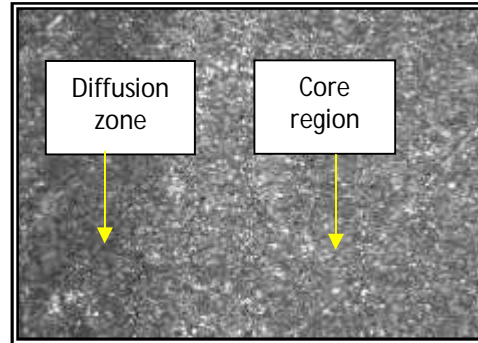
(a) Mag. 200 X



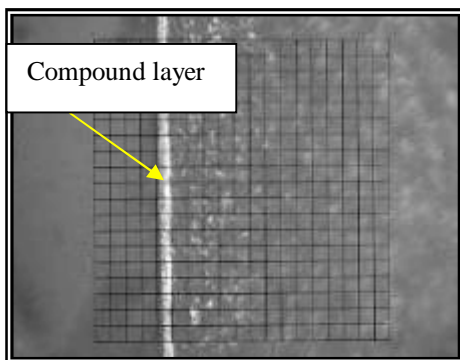
(b) Mag. 200X



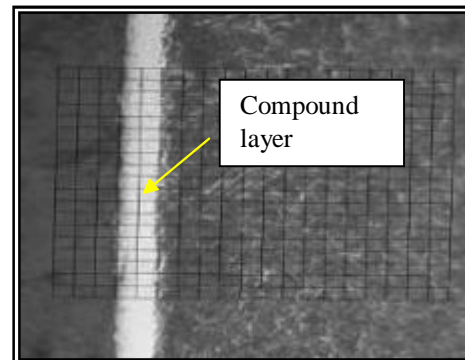
(c) Mag. 100 X



(d) Mag. 200 X



(e) Mag. 500 X



(f) Mag. 500 X

Figure: 7 Microstructures at surface region after various treatments

(a) Untreated material (b) Hard chrome plated (c) Thermal sprayed - alumina coating (d) Plasma nitrided - Without compound layer (e) Plasma nitrided - With less than 10 μm thickness, and (c) Plasma nitrided - With greater than 10 μm thickness. (Scale for grid - One division is 10 μm)

Determination of Basquin Relationship

Statistical analysis of fatigue test results

The data generated in the fatigue testing program is subjected to statistical analysis, and the extracted mean field parameters are modeled using the Basquin relationship:

$$\sigma_a = \alpha (N_f)^\beta \quad (\text{Eq. 1})$$

Where:

σ_a = alternating stress (stress amplitude)

N_f = Number of cycles to failure

α , β = Basquin pre-exponent and fatigue life exponent, respectively

Based on the above equation the derived data for fatigue life for various specimens were calculated and the results obtained have been reported in Table 3.

Table: 3 Derived values of fatigue life from Basquin exponents

Applied stress, Mpa	Untreated	Hard chrome plated	Thermal sprayed (Alumina)	Plasma nitrided (w/o Compound layer)	Plasma nitrided (Compound layer <10 μm)	Plasma nitrided (Compound layer >10 μm)
700	8.80E+05	7.84E+04	3.940E+04		3.95E+07	3.54E+04
770	3.81E+05	6.46E+04	2.910E+04		7.84E+06	2.30E+04
840	1.77E+05	5.41E+04	2.200E+04	6.84E+08	1.80E+06	1.55E+04
910	8.78E+04	4.59E+04	1.707E+04	3.52E+07	4.62E+05	1.07E+04
975	4.58E+04	3.95E+04	1.348E+04	2.26E+06	1.32E+05	7.66E+03
1045				1.75E+05		
1115				1.61E+04		

Based on the results reported in above Table, Basquin σ_a versus N_f plots (i.e. derived S-N curves) are presented in Figure 8.

Computation of fatigue stress and cycle modification factors:

Once the Basquin type relationship given by (Eq.1) have been developed for the untreated specimen and various treatment categories, the following additional parameters namely fatigue stress and cycle modification factors were computed using following equations.

a) Fatigue Stress modification factor, $(\theta_{\text{coat}})_i$

$$(\theta_{\text{coat}})_i = \frac{(\sigma_a)_i}{(\sigma_a)_b} = \frac{\alpha_i (N_f)^{\beta_i}}{\alpha_b (N_f)^{\beta_b}} = \left[\frac{\alpha_i}{\alpha_b} \right] \left[N_f^{(\beta_i - \beta_b)} \right] = \theta_{PEf} N_f^{\theta_{Ei}}$$

(Eq. 2)

Where:

i = ith treatment category

b = Base material (control category)

α_i, α_b = Basquin pre-exponent for ith treatment and control categories, respectively

β_i, β_b = Basquin exponent for ith treatment and control categories, respectively

$\theta_{PEi}, \theta_{Ei}$ = Life reduction pre-exponent and exponent, respectively for treatment i.

It is to be noted that the inverse Basquin exponents are derived by switching the role of dependant (N_f) and independent variables.

The higher values of Fatigue Stress Modification Factors (FSMF), $(\theta_{\text{coat}})_i$ are desirable for greater endurance limit and in turn enhanced fatigue life of any material.

b) Cycle modification factor, Ψ_i

$$\left(\frac{N_f}{N_f} \right)_i = \Psi_i = \frac{\xi_i (\sigma_a)^{\eta_i}}{\xi_b (\sigma_a)^{\eta_b}} = \left[\frac{\xi_i}{\xi_b} \right] \left[\sigma_a^{(\eta_i - \eta_b)} \right] = \Psi_{PEf} \sigma_a^{\Psi_{Ei}}$$

(Eq. 3)

Where:

i = ith treatment category

b = Control (base material) category

ξ_b, ξ_i = Inverse Basquin pre-exponent for control and ith categories, respectively

η_b, η_i = Inverse Basquin Exponents for control and ith categories respectively

Ψ_{PEi}, Ψ_{Ei} = Cycle modification pre-exponent and exponent, respectively for ith treatment.

Likewise, the higher values of Fatigue Cycle Modification Factors (FCMF), Ψ are desirable for enhanced fatigue life of any material.

As per above mentioned equations the FSMF & FCMF parameters were calculated and the results are presented in Figure 9 (a) & (b).

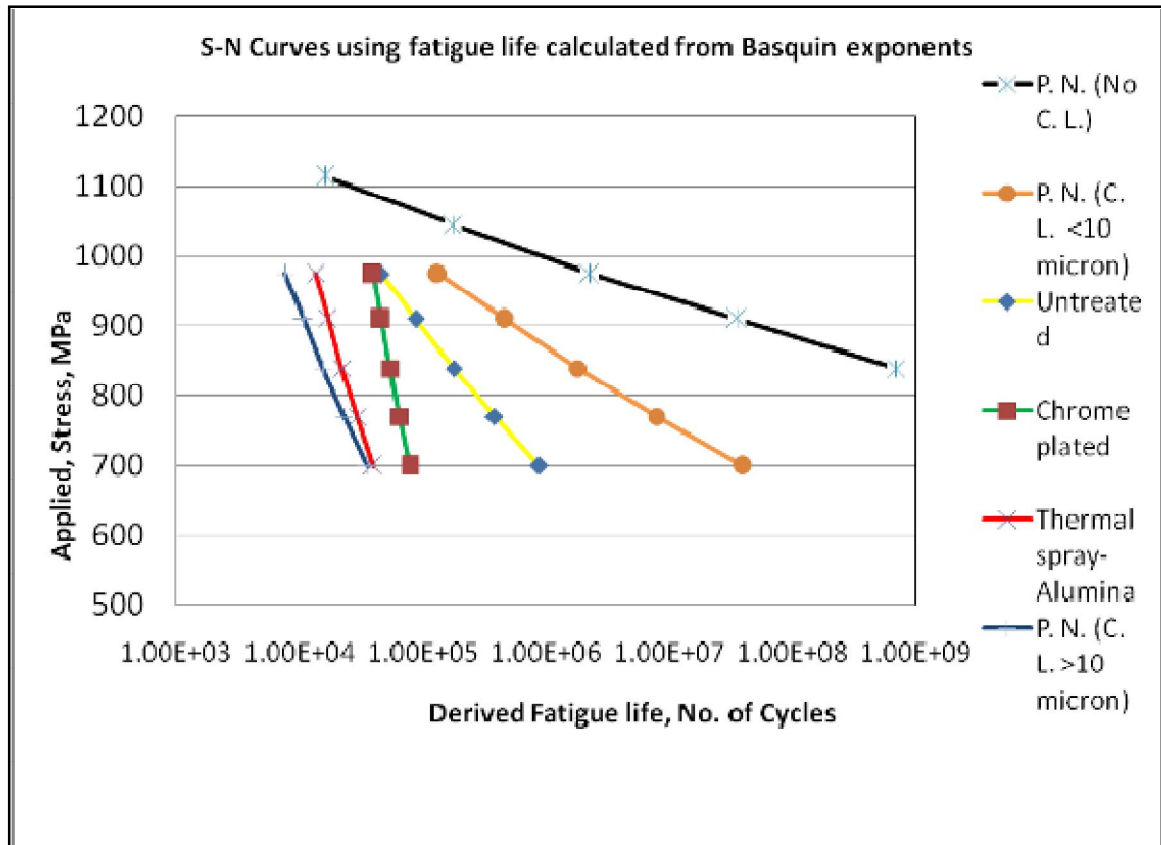
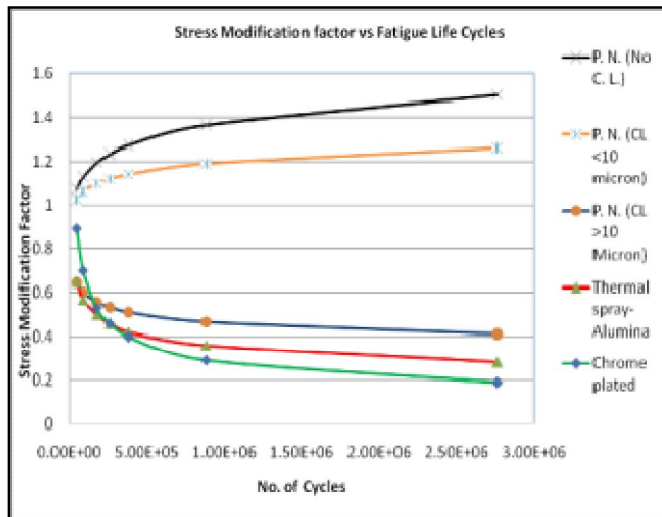
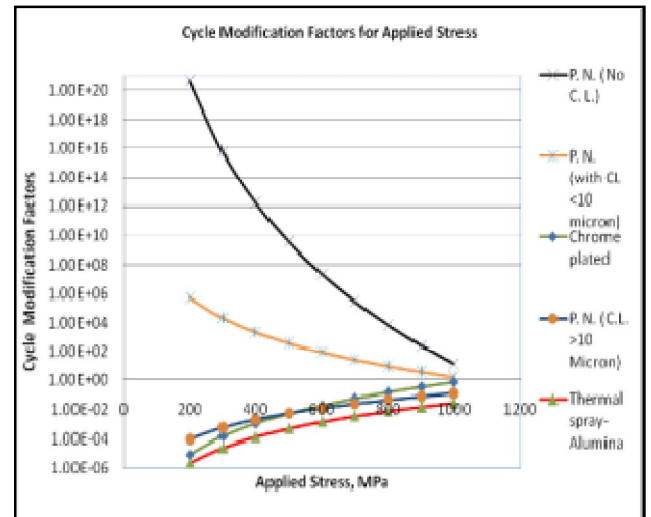


Figure: 8 Derived fatigue life curves / S-N curves (Basquin plots) for various treatments



a)



b)

Figure: 9 (a) Stress modification and (b) Cycle modification factors for various treatments

Interpretations of fatigue life curves:

It is seen from these plots that the maximum reduction in stress level occurs for the plasma nitrided category with compound layer greater than 10 μ m. For this category, the Fatigue Stress Modification Factors (FSMFs), $(\theta_{\text{coat}})_i$ are fractional (less than one), and undergo a monotonic decrease with an increase in the number of cycles to failure.

In other words, if one were to plot a superposition of the Basquin plots for this category (with thickness of compound layer greater than 10 μ m) with reference to the untreated category, the Basquin plots will be translated downward, parallel to the 'alternating stress axis' and to the left, parallel to the 'number of cycles to failure' axis.

On the other hand, for the plasma nitrided specimens, with compound layer less than 10 μ m as well as with 'absence of compound layer', the Fatigue Stress Modification Factors (FSMFs), $(\theta_{\text{coat}})_i$ are greater than one. The plasma nitrided category without compound layer shows the highest increase in the stress modification factor. The presence of compound layer less than 10 μ m is seen to degrade the fatigue resistance as compared to the fatigue resistance for the plasma nitrided category without the compound layer.

Thus, present investigation reveals that the Plasma Nitriding - in the presence of compound layer less than 10 μ m - does not degrade fatigue life/strength but, in fact, results in its increase. In other words, in contrast to the commonly held notion that the presence of compound layer (in any thickness) will degrade the fatigue life, the present study shows that this is not completely true. The exact effect of the compound layer on the fatigue life depends on two thickness regimes of the layer. Thus, when the compound layer thickness is greater than 10 μ m, the fatigue life is markedly degraded. On the other hand, for compound layer thicknesses less than 10 μ m, the effect on fatigue life is positive, albeit a little lower than the fatigue life of plasma nitriding coating without any compound layer.

Finite Element Analysis

Finite Element Analysis (FEA) was done to find-out stresses developed in the specimens (during testing) for various surface conditions/treatments. The methodology adopted for analysis is described below:

A 3D CAD model of the specimen was prepared. Finite Element Meshing was done of the specimen to generate elements. Properties were assigned to materials including base material and coatings. Boundary conditions were applied on the specimen for displacement (constraints).

Different values of loads were applied on specimens and the analysis program was run for each value of load. Output parameters (plots) in terms of stresses generated (Max. Von-mises and Max. principle stresses) were obtained. The stress values were correlated with behavior observed during fatigue testing. The FEM study is in progress and the results obtained by and large in agreement with the experimental values.

Conclusions

Based on the experiments carried out on the untreated specimens i.e. base material, hard chrome plated, thermal sprayed using alumina powder, and plasma nitrided specimens of three different categories and the analysis undertaken, the following conclusions have been drawn:

- The data generated is clearly distinguishable between the different categories of surface treatments which have positive and negative effect on rotating bending fatigue strength of AISI 4340 steel. The treatments can be divided in two groups as given below:
 - Categories, which increase fatigue strength are
 - Plasma nitrided with compound layer $< 10\text{ }\mu\text{m}$
 - Plasma nitrided without any compound layer
 - Categories, which decrease fatigue strength are
 - Hard chrome plated
 - Thermal sprayed using alumina powders, and
 - Plasma nitrided category of compound layer $> 10\text{ }\mu\text{m}$
- Based on detailed study on plasma nitriding, the following conclusions are drawn:
 - i. For the plasma nitrided categories with compound layer less than $10\text{ }\mu\text{m}$ and with absence of compound layer, the Fatigue Stress Modification Factors (FSMF), $(\theta_{\text{coat}})_i$ are greater than one whereas for plasma nitrided category with compound layer more than $10\text{ }\mu\text{m}$ plasma nitrided categories with compound layer less than $10\text{ }\mu\text{m}$ the FSMF value is less than one. The plasma nitrided category without any compound layer shows the highest increase in the Fatigue Stress Modification Factor. The higher values of FSMF are desirable for greater endurance limit and in turn enhanced fatigue life of any material.
 - ii. The difference in the fatigue strength of compound layer greater than $10\text{ }\mu\text{m}$ and less than $10\text{ }\mu\text{m}$, respectively, is essentially associated with interaction effects of compound layer thickness with the state of residual stress. It is known that compound layer tends to have principal residual and octahedral normal stresses in the tensile mode. Further, the probability of fatigue crack nucleation is directly proportional to the volume of compound layer. Thus, for larger thickness of compound layer, the probability of crack nucleation will be higher and will decrease as the thickness of the compound layer reduces. At a critical threshold value of the thickness, the probability of crack nucleation in the diffusion zone can easily become larger than in the compound layer. Similarly, below a threshold thickness of compound layer the residual stress can switch to the compressive mode leading to enhanced fatigue strength.
 - iii. An important finding of this work is that the presence of compound layer is not always detrimental to fatigue life. The results obtained clearly show that the presence of compound layer up to $10\text{ }\mu\text{m}$ thickness enhances the fatigue strength and fatigue life of the material and the drop in fatigue strength starts only when the compound layer thickness is greater than $10\text{ }\mu\text{m}$. Besides this, it is well known that plasma nitriding offers improvement in the wear and corrosion resistance of the material and thus an added benefit.

- iv Fatigue cracks usually initiate from the materials surface. For materials hardened by plasma nitriding, the crack tends to initiate in subsurface rather than surface in high cycle fatigue. This is due to compressive residual stress formed in the diffusion layer, resulting in better resistance to cyclic slip.

REFERENCES

- [1] E.S. Puchi-Cabrera , M.H. Staia , D.T. Quinto, C. Villalobos-Gutiérrez and E. Ochoa-Pérez, “Fatigue properties of a SAE 4340 steel coated with TiCN by PAPVD”, *International Journal of Fatigue* 29 (2007) 471–480
- [2] B. Kumar and N.C. Upadhyay, *IIM Met. News* 8 (2005) 5
- [3] M.P. Nascimento , R.C. Souza , W.L. Pigatin , H.J.C. Voorwald, “Effects of surface treatments on the fatigue strength of AISI 4340 aeronautical steel”, *International Journal of Fatigue* 23 607–618, (2001)
- [4] R.G. Bonora, H.J.C. Voorwald, M.O.H. Cioffi, G.S. Junior, L.F.V. Santos, “Fatigue in AISI 4340 steel thermal spray coating by HVOF for aeronautic application”, *Procedia Engineering* 2 1617–1623, (2010)
- [5] B. D. Sartwell, P. M. Natishan, I. L. Singer, K. O. Legg, J. D. Schell, J. P. Sauer, “Replacement of Chromium Electroplating Using HVOF Thermal Spray Coatings”,
- [6] J. Qian and A. Fatemi, “Cyclic deformation and fatigue behavior of ion-nitrided steel” *Int. J. Fatigue* Vol. 17, No. 1, pp. 15-24, 1995
- [7] J.D. Costa, J.M. Ferreira, A.L. Ramalho, “Fatigue and fretting fatigue of ion-nitrided 34CrNiMo6 steel”, *Theoretical and Applied Fracture Mechanics* 35 (2001) 69-79
- [8] Chang-Min Suh, Byung-Won Hwang, Ri-Ichi Murakami “Characteristics of Fatigue Crack Initiation and Fatigue Strength of Nitrided 1Cr-1Mo-0.25V Turbine Rotor Steels”, *KSME International Journal*, Vol, 16No. 8, pp. 1109--1116, 2002
- [9] A. Celik , S. Karadeniz, “Improvement of the fatigue strength of AISI 4140 steel by an ion nitriding process” *Surface and Coatings Technology* 72, 169-173, (1995)
- [10] M. M. Tomic, I. Terzic, R. Gligorićević, M. Ognjanović, “Fatigue improvements of glow-discharge-plasma-nitrided steel rotary specimens” *Surface and Coatings Technology*, 63 (1994) 73-83
- [11] Sule Yildiz Sirin, Kahraman Sirin, Erdinc Kaluc, “Effect of the ion nitriding surface hardening process on fatigue behavior of AISI 4340 steel” *Material Characterization* 59, 351-358,(2008)
- [12] Mohamed Ali Terres, Habib Sidhom, “Fatigue life evaluation of 42CrMo4 nitrided steel by local approach:Equivalent strain-life-time” *Materials and Design* 33, 444–450, (2012)
- [13] Akgu'n Alsaran, Irfan Kaymaz, Ayhan Celik, Fatih Yetim, Mehmet Karakan, A repair process for fatigue damage using plasma nitriding, *Surface & Coatings Technology* 186 (2004) 333– 338
- [14] Aleksander Nakonieczny Solemani, “Fatigue fracture of nitrided and Carbonitrided layers”, *Journal of Theoretical and Applied mechanics*, 44, 3, pp. 713-730, Warsaw (2006)
- [15] Kenan Genel , Mehmet Demirkol , Mehmet Capa, “Effect of ion nitriding on fatigue behaviour of AISI 4140 steel”, *Materials Science and Engineering*, A279, 207–216, (2000)

- [16] Celik A, Karadeniz S. Investigation of compound layer formed during ion nitriding of AISI 4140 steel. *Surf Coat Technol* 1996;80:283–6.
- [17] O'Brien JM, Goodman D. Heat treating, plasma (ion) nitriding. In: Davis JR et. al. Editors. *Metals Handbook*. 9th ed., vol. 4. Ohio, USA: ASM; 1991. p. 420–4.
- [18] Alsaran A, Karakan M, Celik A. The investigation of mechanical properties of ion-nitrided AISI 5140 low-alloy steel. *Mater Charact* 2002;48:323–7.
- [19] Zagone LF, Figueroa CA, Droppa Jr R, Alvarez F., “Influence of the process temperature on the steel microstructure and hardening in pulsed plasma nitriding”, *Surf Coat Technol* 2006; 201:452–7.
- [20] S.R.Hosseini and F. Ashrafizadeh, “Evaluation of Nitrogen Diffusion in Plasma Nitrided Iron by Various Characterization Techniques, International Journal of ISSI, Vol. 5, (2008), No.2, pp. 29-35
- [21] Abhishek Sharma, K. Sachdev, G. Zalab, J. Alphonsa, S. Mukherji and K. C. Swami “Effect of Plasma Nitriding on the Microstructure and Properties of Several Steels”, International Journal of Materials Physics, ISSN 0974-309X Volume 2, Number 2 (2011), pp. 141-147
- [22] K. Hussain, A. Tauqir, A. Haq, A.Q. Khan, *Int. J. Fatigue* 21 (1999) 163.
