INFLUENCE OF THERMAL SPRAYING PROCEDURES ON THE PERFORMANCE OF OVER LAYS DEPOSITED ON THE STEEL SUBSTRATES

A DISSERTATION

Submitted in partial fulfillment of the requirements for the award of the degree

of

MASTER OF TECHNOLOGY

in

MECHANICAL ENGINEERING

(With Specialization in Welding Engineering)

By

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CANDIDATE'S DECLARATION

I hereby declare that the work which is being presented in this dissertation entitled **"Influence of Thermal Spraying Procedures on the Performance of Over Lays Deposited on the Steel Substrates"** in partial fulfillment of the requirement for the award of the degree of Master of Technology in the department of, Mechanical & Industrial Engineering with specialization in **"Welding Engineering"**, submitted in **Mechanical & Industrial Engineering Department, Indian Institute of Technology, Roorkee, (India)**, is as authentic record of my own work carried out during the period from July 2005 to June 2006 under the guidance of **Dr. D.K.Dwivedi**, Assistant Professor, Department of Mechanical & Industrial Engineering, Indian Institute of Technology, Roorkee, (India) and **Mr. Ajai Agarwal**, Assistant Professor, Department of Mechanical & Industrial Engineering, Indian Institute of Technology, Roorkee, (India).

The matter embodied in this dissertation has not been submitted by me for the award of any other degree of this institute or any other institute.

Dated: 30-06-2006

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CERTIFICATE

This is to certify that the above statement made by the candidate is correct to the best of my knowledge.

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ABSTACT

Thermal spraying is a process which is used to coat thin films of metallic or non metallic materials on to substrates. The study of these thermal sprayed coatings are useful, because these coatings are used to restore desired dimensions; to improve resistance to wear, corrosion, oxidation, or a combination of these; and to provide specific electrical or thermal properties.

Three types of powders named Ni based WC (1002ET), Ni-Cr eutectic alloy (1004EN), and Co and Ni based CrC (1006EE) are used to develop coatings on mild steel substrate by flame spraying process. We added some carbide particles like WC, CrC to reinforce the coatings. Heat treatment was done for all modified and unmodified coatings to enhance wear resistance.

It was studied the abrasive wear behavior of all coatings under different testing conditions like heat treatment, normal load, counter surface, percentage of hard carbide particles added. Tests like abrasive wear test, normal hardness test, microhardness test, SEM analysis of worn out surfaces, EPMA of coatings and microstructure study were carried out in order to understand wear behavior of coatings.

Microstructures were obtained for all coatings to understand the distribution of secondary phases in the metal matrix. EPMA test (quantitative test) was done to get weight percentage of different elements present in the coating and in secondary phase. Microhardness test was carried out to get the hardness value at individual phases, to explain wear behavior under different conditions. SEM analysis of worn out samples was done to understand wear mechanism of wear.

Heat treatment enhanced microhardness and reduced wear rates. Addition of carbide particles acts as reinforcement and so reduced wear rates. But heat treatment in modified powder coatings showed adverse effect on wear resistance.

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INTRODUCTION # CHAPTER-1

Today industry needs components of high wear resistive, corrosion resistive and hard facing surfaces. So a lot of attention is paid by so many research scholars in the field of industrial tribology. Surface engineering is a technology which deals with these tribological aspects. So surface engineering is widely used in different industrial sectors. It includes various technologies, such as thermal treatment for surface hardening; nitriding, shot peening, physical vapors deposition (PVD), chemical vapors deposition (CVD), ionic implantation; electro-chemical processes and many other techniques with involve different physical and chemical methods. Some of the surface treatments are shown in Table: 1.1. Using these techniques it is possible to obtain high quality structure and properties of the surface of the industrial components. [1]

Table1.1. Categories of surface treatments.[1]

ATOMIC DEPOSITION	PARTICULATE DEPOSITION	BULK COATING OR CLADDING	SURFACE MODIFICATION
Electrolytic deposition Electro plating Auto catalytic Fused salt Chemical displacement	**Thermal spray** Flame spray Arc spray Plasma spray HVOF Detonation spray Cold spray	**Wetting processes** Painting Dip coating	**Chemical conversion** **Electrolytic** Anodization Fused salt
Physical vapors Deposition Ion implantation Vacuum deposition Vacuum evaporation Ion beam	**Impact plating**	**Electrostatic spraying** Printing **Spin coating**	**Chemical** Thermal vapor Plasma vapor liquid
Plasma deposition Sputter deposition Ion painting Plasma polarization	**Fusion coatings** Thick film ink Enameling Thermal spray Spray&Fuse/Spray Fuse	**Cladding** Explosive roll bonding	**Mechanical Shot Peening**
Chemical vapor Deposition Spray pyrolysis		**Overlay** **Laser cladding** **Weld overlay**	**Thermal surface Enhancement** **Diffusion from bulk sputtering** **Ion Implantation**
		Self Propagating High Temperature Synthesis	

Among these methods, there exists thermal spraying. It consists of heating of material up to or above the melting temperature, material accelerated and sprayed on to a surface which is to be coated. Thermal spraying is widely used for surfacing applications to attain or restore desired dimensions; to improve resistance to abrasion, wear, corrosion, oxidation, or a combination of these; and to provide specific electrical or thermal properties.[1]

In all methods of thermal spraying, kinetic and thermal energy of a thermal source is transferred to material. This material may be, for example, in the form of powder or wire which is melted and sprayed onto a clean surface where it flattens and interact with the substrate. Coating is developed due to relative movement between the spraying gun and the substrate. The properties of the coating and the service performance depend essentially on the thermal spraying process selected. [1]

Heat transfer to the sprayed material facilitates its deformation during impact onto the substrate. Velocity and temperature of the material must correlate well in order to develop a deformed liquid splat which fits the morphology of the substrate surface or the previously deposited material. High velocity of the sprayed material contributes to an increase in the coating density and a decrease in the coating porosity that improves adhesive bonds between the substrate and the coatings.

1.1. Thermal Spraying

Thermal spraying is a generic term for a group of coating processes used to apply metallic or non metallic coatings. These processes are grouped into three major categories: flame spray, electric arc spray, and plasma arc spray. These energy sources are used to heat the coating material (in powder, wire, or rod form) to a molten or semi molten state. The resultant heated particles are accelerated and propelled toward a prepared surface by either process gases or atomization jets. Upon impact, a bond forms with the surface, with subsequent particles causing thickness buildup and forming a lamellar structure. The properties of the deposit may depend upon its density, the cohesion between the deposited particles, and its adhesion to the substrate.

A major advantage of thermal spray processes is the extremely wide variety of materials that can be used to produce coatings. Virtually any material that melts without decomposing can be used. A second major advantage is the ability of most thermal spray processes to apply coatings to substrates without significant heat input. Thus, materials with very high melting points, such as tungsten, can be applied to finely machined, fully heat-treated parts without changing the properties of the part and without excessive thermal distortion of the part. A third advantage is the ability, in most cases, to strip off and recoat worn or damaged coatings without changing part properties or dimensions. [2-6]

Thermal spraying coatings are often used to produce wear resistance coatings on several industrial components. So before that, it is custom to know about the wear and its behavior in different applications. So wear mechanism plays a major role in influencing these coatings. Wear may be defined as the removal of the material from solid surface as a result of mechanical action. It is a characteristic feature of the wear process that the amount of material removed is quit small.

1.2. Thermal spraying processes and techniques

Members of the thermal spray family of processes are typically grouped into three major categories: flame spray, electric arc spray, and plasma arc spray, with a number of subsets falling under each category. (Cold spray is a recent addition to the family of thermal spray processes. This process typically uses some modest preheating, but is largely a kinetic energy process). A brief review of some of the more commercially important thermal spray processes is given below. Selection of the appropriate thermal spray method is typically determined by

- Desired coating material
- Coating performance requirements
- Economics
- Part size and portability

1.2.1. Flame Spraying Processes

Flame spraying includes low-velocity powder flame, rod flame, and wire flame processes and high-velocity processes such as HVOF and the detonation gun process.

(a)Flame Powder Spraying: In the flame powder process, powdered feedstock is aspirated into the oxy fuel flame, melted, and carried by the flame and air jets to the work piece. Particle speed is relatively low (<100 m/s), and bond strength of the deposits is generally lower than the higher velocity processes. Porosity can be high and cohesive strength is also generally lower. Spray rates are usually in the 0.5 to 9 kg/h (1 to 20 lb/h) range for all but the lower melting point materials, which spray at significantly higher rates. Substrate surface temperatures can run quite high because of flame impingement.

(b)Wire Flame Spraying: In wire flame spraying, the primary function of the flame is to melt the feedstock material. A stream of air then atomizes the molten material and propels it toward the work piece. Spray rates for materials such as stainless steel are in the range of 0.5 to 9 kg/h (1 to 20 lb/h). Again, lower melting point materials such as zinc and tin alloys spray at much higher rates. Substrate temperatures often range from 95 to 205 °C (200 to 400 °F) because of the excess energy input required for flame melting. In most thermal spray processes, less than 10% of the input energy is actually used to melt the feedstock material.

(c) High-Velocity Oxy Fuel Spraying: In HVOF, a fuel gas (such as hydrogen, propane, or propylene) and oxygen are used to create a combustion jet at temperatures of 2500 to 3100 °C (4500 to 5600 °F).The combustion takes place internally at very high chamber pressures, exiting through a small-diameter (typically 8 to 9 mm, or 0.31 to 0.35 in.) barrel to generate a supersonic gas jet with very high particle speeds. The process results in extremely dense well bonded coatings, making it attractive for many applications. Either powder or wire feedstock can be sprayed, at typical rates of 2.3 to 14 kg/h.

(d)Detonation Gun Spraying: In the detonation gun process, pre-encapsulated "shots" of feedstock powder are fed into a 1 m (3 ft) long barrel along with oxygen and a fuel gas (typically acetylene). A spark ignites the mixture and produces a controlled explosion that propagates down the length of the barrel. The high temperatures and pressures (1 MPa, or 150 psi) that are generated blast the particles out of the end of the barrel toward the substrate. Very high bond strengths and densities as well as low oxide contents can be achieved using this process.

1.2.2. Electric Arc Spraying Processes: In the electric arc spray process (also known as the wire arc process), two consumable wire electrodes connected to a high-current power source are fed into the gun and meet, establishing an arc between them that melts the tips of the wires. The molten metal is then atomized and propelled toward the substrate by a stream of air. The process is energy efficient because all of the input energy is used to melt the metal. Spray rates are driven primarily by operating current and vary as a function of both melting point and conductivity. Generally materials such as copper-base and iron-base alloys spray at 4.5 kg (10 lb)/100 A/h. Zinc sprays at 11 kg (25 lb)/100 A/h. Substrate temperatures can be very low, because no hot jet of gas is directed toward the substrate. Electric arc spraying also can be carried out using inert gases or in a controlled atmosphere chamber.

1.2.3. Plasma Arc Spraying Processes

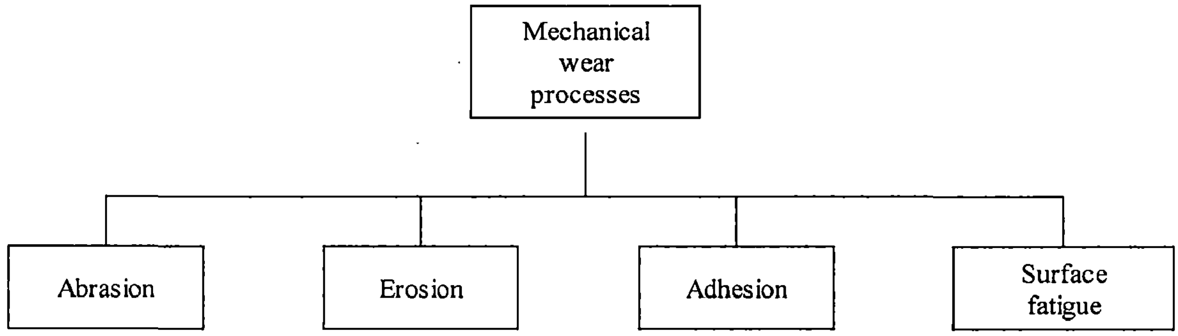
(a)Conventional Plasma spraying Process: The conventional plasma spray process is commonly referred to as air or atmospheric plasma spray (APS). Plasma temperatures in the powder heating region range from about 6000 to 15,000 °C (11,000 to 27,000 °F), significantly above the melting point of any known material. To generate the plasma, an inert gas—typically argon or an argon-hydrogen mixture is superheated by a dc arc. Powder feedstock is introduced via an inert carrier gas and is accelerated toward the work piece by the plasma jet. Provisions for cooling or regulating the spray rate may be required to maintain substrate temperatures in the 95 to 205 °C range.

(b)Vacuum Plasma Spraying Process: Vacuum plasma spraying (VPS), also commonly referred to as low-pressure plasma spraying (LPPS), uses modified plasma spray torches in a chamber at pressures in the range of 10 to 50 kPa (0.1 to 0.5 atm). At low pressures the plasma becomes larger in diameter and length, and, through the use of convergent/divergent nozzles, has a higher gas speed. The absence of oxygen and the ability to operate with higher substrate temperatures produce denser, more adherent coatings with much lower oxide contents.

1.2.4. Cold Spraying Process: Kinetics has been an important factor in thermal spray processing from the beginning. With the introduction of detonation gun, HVOF, and high-energy plasma spraying, the kinetic-energy component of thermal spraying became even more important. The latest advance in kinetic spraying is known as cold spray. Cold spraying is a material deposition process in which coatings are applied by accelerating powdered feed stocks of ductile metals to speeds of 300 to 1200 m/s (985 to 3940 ft/s) using gas-dynamic techniques with nitrogen or helium as the process gas. The process is commonly referred to as "cold gas-dynamic spraying" because of the relatively low temperatures (0 to 800 °C, or 32 to 1470 °F) of the expanded gas and particle stream that emanates from the nozzle. Powder feed rates of up to 14 kg/h (30 lb/h) are possible.

1.3. Wear mechanisms:

The phenomenon of wear was given a formal definition in 1968 by the OECD as 'the progressive loss of material from the operating surface of a body occupying as a result of relative motion at its surface'. Modern research has established that there are four main forms (Fig. 1.1) of wear and each process obeys its own laws.[7]



2 and 3 body:	cavitation	adhesion	pitting
Polishing-	liquid impact	fretting	flaky wear:
Low stress abrasion	solid impact	scuffing	delamination
High stress abrasion	slurry erosion	galling	ratcheting
Gouging			

Fig. 1.1. Classification of mechanical wear processes.[21]

(a)Adhesive wear: This is the form of wear which occurs when two smooth bodies are slid over each other, and fragments are pulled off one surface to other. Later, these fragments may come off the surface on which they are formed and be transferred back to the original surface, or else from loose wear particles. This wear arises from the strong adhesive forces set up whenever atoms come in to intimate contact. During sliding, a small patch on one of the surfaces comes in to contact with a similar patch on the other surface, and there is a probability, small but finite, that when this contact broken the break will occurs at not at the original interface but within one of the materials.

(b)Abrasive wear: This is the form of the wear which occurs when a rough hard surface, or a soft surface containing hard particles, slides on a softer surface, and ploughs a series of grooves in it. The material from the grooves is displaced in the form of wear particles, generally looses ones. Abrasive wear occur under two conditions.[25,26]

- **Two body abrasion:** In this condition, one surface is harder than the other rubbing surface e.g. in mechanical operations such as grinding, cutting, and meeting.
- **Three body abrasion**: In this case a third body, generally a small particular of grit of abrasive, lodges between the two rubbing surfaces; abrade one or both these surfaces.

(c) Erosion: The damage produced by sharp particles impinging an object is closely analogous to that produced by abrasion. The main difference is that in erosion the surface roughness produced may become relatively greater, because an impinging particle may readily remove material from a low point on the surface.

(d) Surface fatigue wear: This form of wear is observed during repeated sliding or rolling over a track. The repeated loading and unloading cycles to which the materials are exposed may induce the formation of the surface or subsurface cracks, which eventually will result in the break-up of the surface with the formation of large fragments, leaving large pits in the surface.

Other forms of wear: The other phenomena which some times classified as wear are fretting, cavitation, erosion.[7]

(a) Fretting wear: It is not a primary form of wear, but a phenomenon which occurs when other wear mechanisms act together under oscillating sliding conditions. This form of wear arises when contacting surfaces undergo oscillatory tangential displacement of small amplitude.

(b) Corrosive wear: This form of wear occurs when sliding takes place in a corrosive environment. In the absence sliding the products of corrosion would form a film on the surfaces, but the sliding action wears the film away, so that the corrosive attack can continue.

(c) Cavitation: When a portion of liquid is under tensile stresses, it may boil. Later, the bubble may collapse suddenly, producing a mechanical shock. A nearby solid surface may be damaged by this shock, leading to the removal of the particle. This process is known as cavitation.

1.4. Literature review

A lot of work was carried out by so many research fellows in thermal spraying. They have conducted a verity of spraying procedures such as flame spraying, plasma spraying, detonation gun spraying, HVOF spraying, and etc. They have also tried different types of post spraying and pre spraying techniques to enhance the results. These tests were conducted for different types of spraying powders such as single-phase materials, composite materials, layered or graded materials. [1-5]

Many people are interested in investigation of mechanical properties, metallurgical characteristics of different types of coatings. They have tried to modify the coatings by changing the compositions of different elements present in them. Most of the research fellows which are interested in tribological properties of coatings have studied mechanical and metallurgical characteristics of ceramic coatings like Ni, Cr, Co, WC, Chromium carbide, Mo, B, and Si etc. A lot of investigations were carried out about abrasive wear behavior of flame spraying coating using Ni-WC, Ni-Cr-Co-Si-B, Co-Ni-Cr-WC, powders and tried to relate wear results with its mechanical properties like microhardness.[5]

John A. Williams explained about wear and wear particles in detailed. He said that when material is lost from a loaded surface either entirely or principally through some form of mechanical interaction the concentration, size and shape of the debris particles carry important information about the state of surfaces from which they were generated and thus, by implication, the potential life of the contact and of the equipment of which this forms a part. The full exploitation of this information and the ability to be able to predict quantitatively the future performance or life requires an understanding of the sources and mechanisms of generation of the extracted and sampled particulate debris. In many cases, it is instructive to display the running conditions of a given contact on some form of operational or wear map. This both enables the implications for wear of changes in design, material or operating parameters to be assessed and allows sensible correlations to be made between laboratory-based experimental investigations and observations in the field.21]

The surfaces of materials, components and products are subjected to corrosion and wear. Protective coatings can counteract such effects. For the development and the selection of suitable wear protective measures, material behavior has to be investigated under well defined tribological conditions and characterized by data relevant to wear15]. J.M. Guilemany and et.all "The wear of WC-Co involves an initial removal of the binder phase mainly produced by abrasion of both the counter face two-body abrasive wear and the fine debris three-body abrasive wear , followed by pull out of the carbides. Adhesive wear is not as important as abrasion and is only observed when a steel metallic ball is used". [11]

According to Antonio Cesar Bozzi et al. there are some mechanisms of abrasive wear that can act in cobalt-based alloys made by powder metallurgy such as the detachment of the carbides and formation of large pits followed by the micro cracking of the edges of carbides. Another mechanism that could be acting in this case is binder extrusion; this acts mainly in the ''mild'' regime for cemented carbides proposed by Larsen-basse. The binder is forced to move outside the surface of the material by the stress of compression of the abrasive particles, due to the fact that the hard phases WC, W_2 C deform only slightly. Thus, almost all deformation occurs in the softer and more ductile matrix. The hard phase has only a small displacement from the normal and tangential stress applied by the abrasive particles. Multiple and repeated contacts of the abrasive particles are required before a significant amount of material can be removed; this cyclic loading results in an extrusion of the matrix between the hard phase particles. After this removal, microcracking and or detachment of hard phase occurs when the support and compressive loading of the matrix are no longer present. [22]

P.Wu et.al characterized wear mechanism in Ni based WC was by deep and fairly long continuous grooves, micro cutting [18]. Hyung. Jun. Kim et.al said that coating of 35% WC with NiCrBSiC shows the best quality in terms of hardness and porosity. However, 25% WC addition is showing the best wear resistance for Sugaru abrasive wear test, while 40% WC addition is showing the best wear resistance for DSRW abrasive wear test [16]. J.M.Miguel et.al said that Spray&fused coating has the best sliding wear resistance (similar to the HVOF sprayed NiCrBSi). The main wear mechanism is adhesion, but abrasion and delamination also take place. The fatigue wear process (intersplat delamination) in the HVOF sprayed coating is not as important as in the plasma sprayed coating due to the best mechanical bonding among splats. [10]

Gang-Chang Ji et.al characterized Cr3C2-NiCr coatings and explained the wear mechanism in that coating. They said that the successive removal of NiCr binder phase leads to carbide particles to be exposed to the surface. Then, the removal of carbide particles occurs by fracturing or loosening followed by subsequent pulling out by abrasive particle. Therefore, an improvement of the hardness of matrix alloy and bonding strength of carbides to the matrix may enhance the abrasive wear resistance of the coating. [17]

1.5. Formulation of problem

The world wide attention on reclamation of worn out metal parts and enhancement of wear life has been provoked the technologists for various type of hard surfacing of conventional material to a great extent. Depending upon service requirements various types of hard facing materials has been used so far, where the process of hard surfacing such as the weld metal deposition, plasma spraying, thermal spraying etc has been primarily decided on process economy and suitability of application to the components. Out of various hard surfacing processes the thermal powder spray technique has been found as a comparatively cheaper and versatile process.

Attempts were made to find wear rates using different kind of setups. But abrasive wear of a pin on reciprocating flat was not found. Understanding of wear behavior under reciprocating conditions is useful for special conditions, where relative motion between two surfaces is reciprocating.

The hard surfacing materials are generally eutectic alloys based on cobalt, nickel, tungsten, chromium, Molybdenum etc having dispersion of various types of carbides. And various metallic carbides have different microstructures and different physical properties like wear resistance which in terms governs life of a component. So it is useful to study various types of metallic coatings produced by flame sprayed on the steel substrates. Thus during deposition of powder at high temperature the occurrence of various types of transformation in it is inevitable which governs significantly the properties of coatings. More over, during deposition some kind of powders may form some meta-stable phases in the coatings which may become stable by further transformation through a diffusion process under a post spray heat treatment process and may provide influence towards the wear resistance property of the coating.

In this present report, the coatings were developed by powder flame spraying on the base material of mild steel by using Ni based WC, Co & Ni based CrC, and Ni-Cr Eutectic alloy surfacing powders. Characterization of coatings was done by microstructure examination, microhardness measurement, normal Vickers hardness, EPMA, and SEM analysis of wear test samples.

This chapter discuss about all procedures followed during the experimentation of the work. The procedures and equipment used for thesis work. Mainly this chapter discussed about the set-up fabricated to do wear analysis; and the equipment used for metallurgical characterization such as microscopy, SEM, EPMA, micro-hardness and normal hardness.

Coatings were developed on mild steel by flame spraying process. The transverse section cut samples were prepared and these were used for wear test, microstructure analysis, microhardness test, hardness test and E.P.M.A analysis. After the wear test the samples were examined by S.E.M. The detailed procedure of each process was discussed below.

2.1. Thermal spraying

Thermal spraying on mild steel (substrate material) was carried out by flame spraying using surfacing powders type EWAC (1002 ET, 1004 EN, and 1006 EE) and by modified powders of the 1004EN and 1006EE. Modification of the surfacing powders was done by adding Chromium carbide and Tungsten carbide in different compositions (10-20%). The contents and composition of each type of unmodified powders and modified powders were given in Table: 2.1.

Table: 2.1. Different types of powders and their compositions.

Type of coating	Contents
1002ET	Nickel based Tungsten Carbide
1004EN	Ni and Cr Eutectic Alloy
1006EE	Cobalt and Nickel based Chromium Carbide
Modified 1004EN-10%	90%-Ni&Cr Eutectic alloy + 10%- CrC
Modified 1004EN-20%	80%-Ni&Cr Eutectic alloy + 20%- CrC
Modified 1006EE-10%	90%-Co& Ni based CrC + 10%-WC
Modified 1006EE-20%	80%-Co& Ni based CrC + 20%-WC

The flame spraying was performed under practically neutral flame of oxy-acetylene gas, where the pressure of oxygen and acetylene was maintained at 3 and 1.2 kg/cm^2 respectively. Spraying was always carried out at a torch speed of 10cm/min. Before going to the flame spraying the surface of mild steel substrate was cleaned mechanically by using emery paper of grade 400 followed by scrubbing with acetone. Thermal spraying was carried on the substrate preheated at around 350°C. Preheating of the substrate was carried out by the oxy-acetylene torch and the temperature was measured by thermocouple attached to digital meter. The coated samples were given a post heat treatment inside an electrical muffle furnace at around 800°C for 2hr, followed by cooling in still air at room temperature.

Table: 2.2. Conditions of flame spraying used in present study

Oxygen cylinder pressure	3 kg/cm^2
Acetylene cylinder pressure	1.2 kg/cm^2
Torch type	L&T, Super-jet Eutalloy torch
Distance of torch nozzle tip from specimen	20mm
Torch angle	90°
Torch speed	10cm/min
Preheating temperature	350 °C
Post heat treatment conditions	800 °C for 2hr

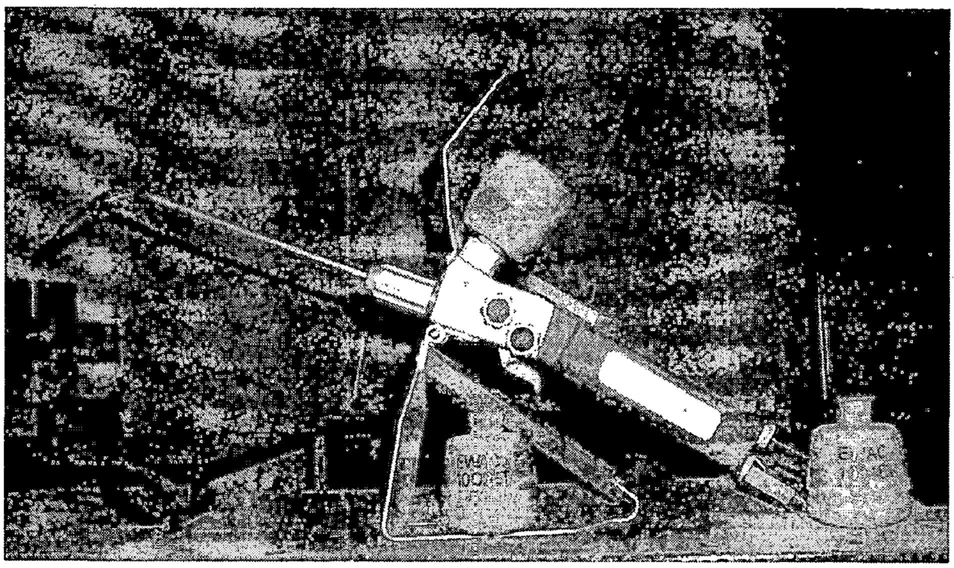


Fig. 2.1. Photograph of powder flame spraying torch.

2.2. Metallographic Study

The transverse section of the coated samples, obtained by sectioning the coating, was prepared by standard metallographic procedure. The polished specimens were etched with suitable etching agent (Table: 2.3) and studied under optical microscope (ZEISS microscope).

Table: 2.3. Etching agents used for microstructure study

Coating type	**Etching agent**
1002-ET	1ml HCl + 10ml HNO_3 + 10ml H_2O
1004-EN, modified powder of 1004	30ml Acetic acid + 30ml HNO_3 + 10ml Glycerin
1006-EE, modified powder of 1006	30ml Acetic acid + 30ml HNO_3 + 10ml Glycerin

2.3. Hardness measurement

Normal Vickers hardness was measured by using (AMSLER OTTO WOLPERT WERKE-GMBH Germany) universal hardness tester under 50N load. Hardness was measured at three points and the average was taken for comparison purpose. Diamond indenter was used for indentation and the average of the two diagonals was calculated. And the hardness value was taken from the standard tables, and represent in terms of VHN.

Microhardness of the coating was measured (Leitz Weltzar microhardness tester made in Germany) across the coating at equal intervals from the interface of coating and substrate material. Diamond indenter was used and 100gr load (0.981N) was applied for the test. The diagonals of the indenter were measured and the average length was calculated. The microhardness value can be obtained directly from the standard tables or from the following formulae, and represented as Hv_{100}.

$$Hv_{100} = 189.03 \times 10^3 \times f / d^2$$

Where f is in N;

d in μm.

2.4. Scanning Electron Microscope (S.E.M) Analysis

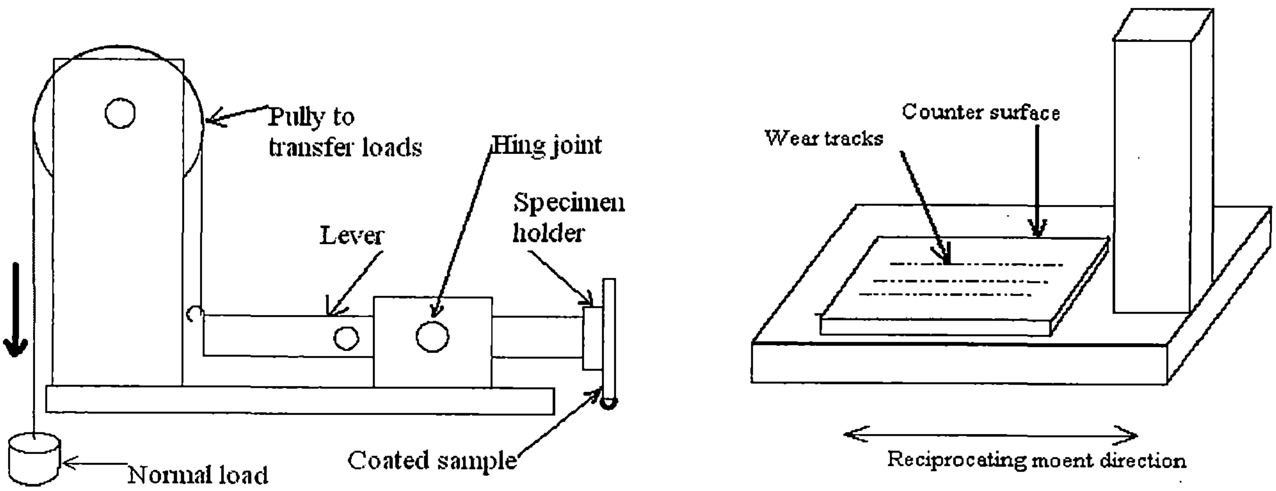
The wear test samples were examined under Scanning Electron Microscope (435VF model, LEO Electron Microscopy ltd, England), to observe the topography of the wear surface tested against 120grit and 600grit abrasive medium under different loads.

2.5. Electron Probe Micro Analysis (E.P.M.A)

EPMA was done by using electron probe micro analyzer (JXA-8600M Super probe, JEOL, Japan). EPMA was carried out for both unmodified and modified powder coatings. EPMA was carried out across the coating from substrate to towards coating. Quantitative analysis of coating was done to analyze chemical composition of coating. Also test was carried out across the second phase (carbide phase).

2.6. Wear test

The wear characteristics of the coatings were studied by pin on reciprocating flat method as schematically shown in Fig. 2.2.



(a) Specimen holder (b) counter surface holder

Fig. 2.2.Schematic diagram of wear testing setup.

Wear testing was carried out based on a reciprocating pin on flat system. The original set-up was shown in Fig. 2.3. This implies to some particular applications where reciprocating motion present in-between two surfaces. This setup was fabricated as a part of project. The transverse section of the coating was cut as a testing specimen, so that the specimen can be hold by the specimen holder.

2.6.1. Working principle

The transverse section of the coated surface was cut into number of pieces, so that each specimen can be hold easily in between the fixed and movable jaws. The desired amount of load can be applied on the coated surface by selecting the same weight and kept on the tray, which was stringed by steel wire through hinged pulley. The counter surface was fixed on a holder as shown in Fig. 2.2. This counter surface holder was kept in shaper tool post. And the specimen holder was fixed by the jaws of the work post of shaper. When reciprocating ram moves the counter surface holder reciprocates, and the coated specimen comes into contact with the counter surface with the applied load.

By running the reciprocating mechanism at a particular speed for a given time we can obtain the desired wear distance. The above set up was used to measure abrasive wear. The normal load and type of counter surface are the main variables for this abrasive wear test. Wear test was conducted on a shaper as it gives reciprocating mechanism. The average speed of the sample during wear test is 0.0953 m/s. Total distance traveled by the sample is approximately 40m in 10min times at a track length of 55mm.

Wear test was carried out under 5N, 10N, 15N, and 20N load. We used 120grit and 600grit emery papers of water proof quality as abrasive medium. We measured the weight of the coating sample before and after the wear test. We used an electronic weighing balance whose accuracy is up to one micro gram (1μgr). The difference in weight (before test - after test) of the tested sample represents the loss of material during wear test. Wear rate, represented in terms of mg/km, is defined as mass loss per unit distance traveled.

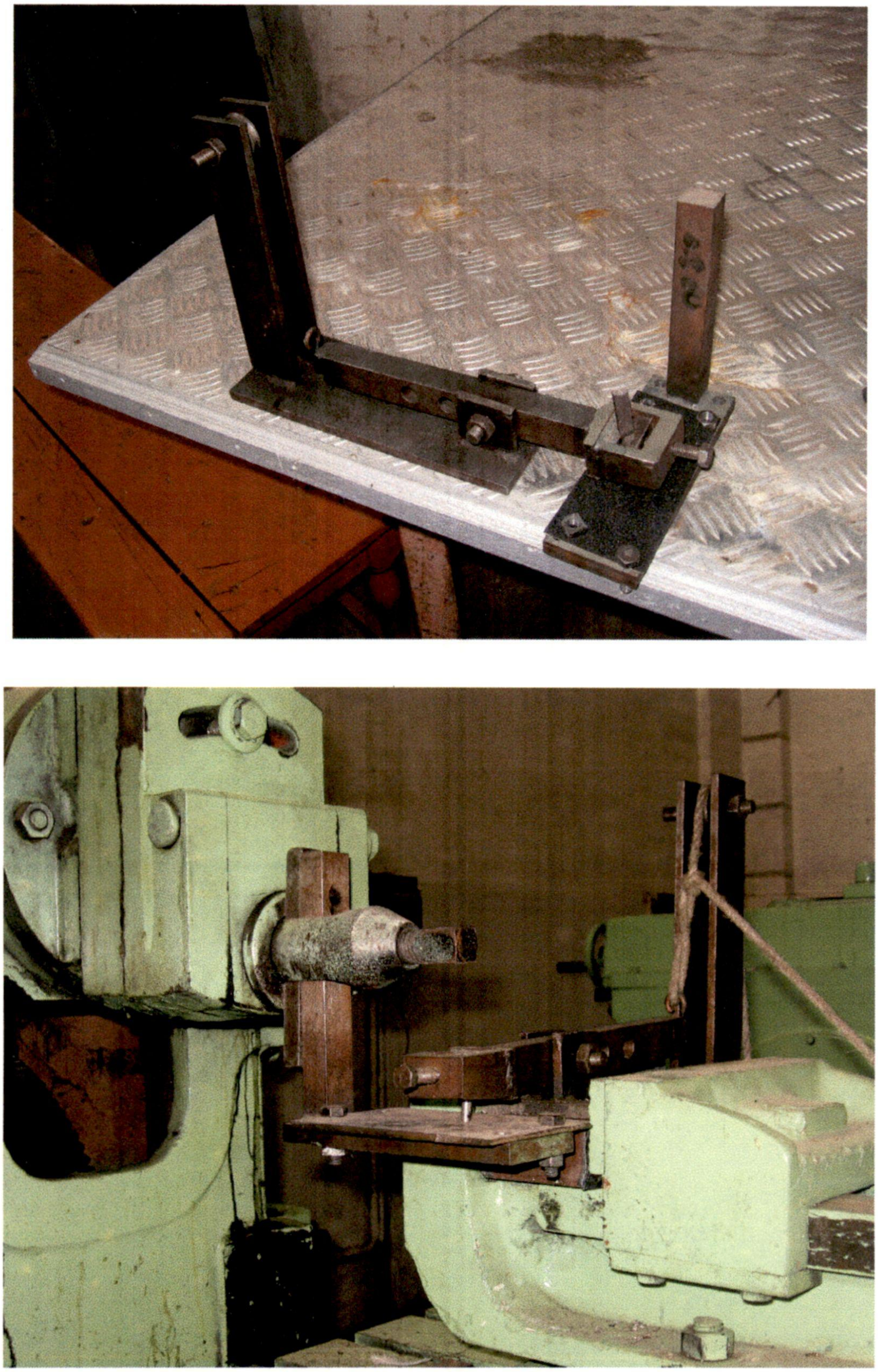


Fig. 2.3. Photograph of wear testing set-up.

RESULTS AND DISCUSSIONS

This chapter discusses about the mechanical properties and metallurgical properties like microhardness, wear rate, EPMA, SEM analysis, and normal hardness of Nickel based Tungsten carbide coatings (EWAC 1002ET) subjected to different testing conditions.

3.1. Microstructure

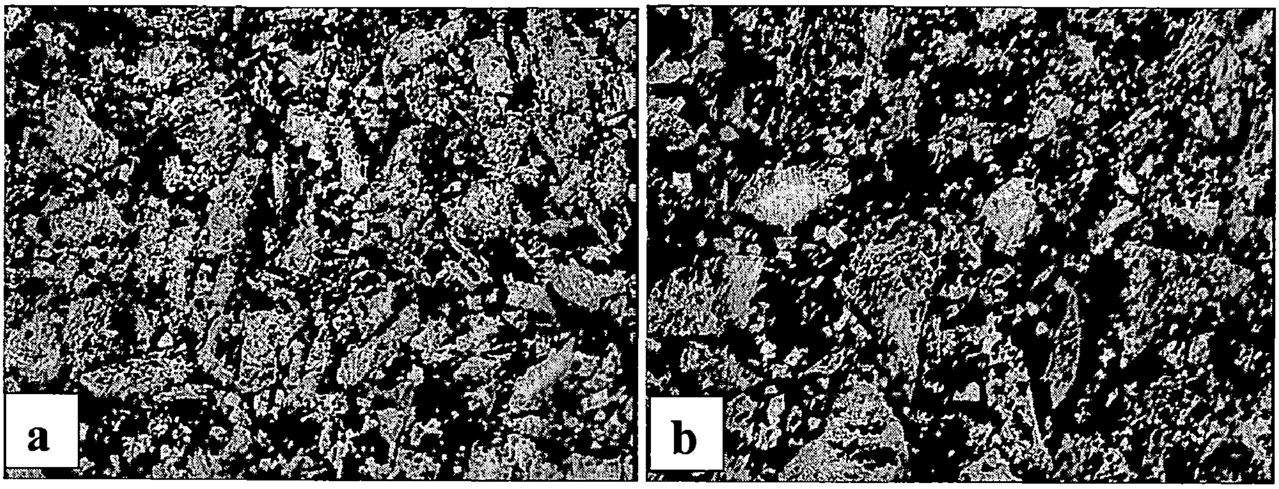


Fig. 3.1.Optical microphotographs of Ni-WC coating (320X)
(a) non heat treated coating (b) heat treated coating

The microstructures of the non heat treated coating and heat treated coating were shown in Fig. 3.1. The white particles in the photos were Tungsten Carbide particles. In non heat treated condition the carbide particles are uniformly and evenly dispersed in the Nickel eutectic matrix (Fig. 3.1a). In heat treated condition the carbide particles are accumulated together and formed larger particles and unevenly disturbed (Fig. 3.1b). The percentage of the carbide particles in heat treated condition is 37%, and in non heat treated condition 41%.

3.2. Electron probe micro analysis:

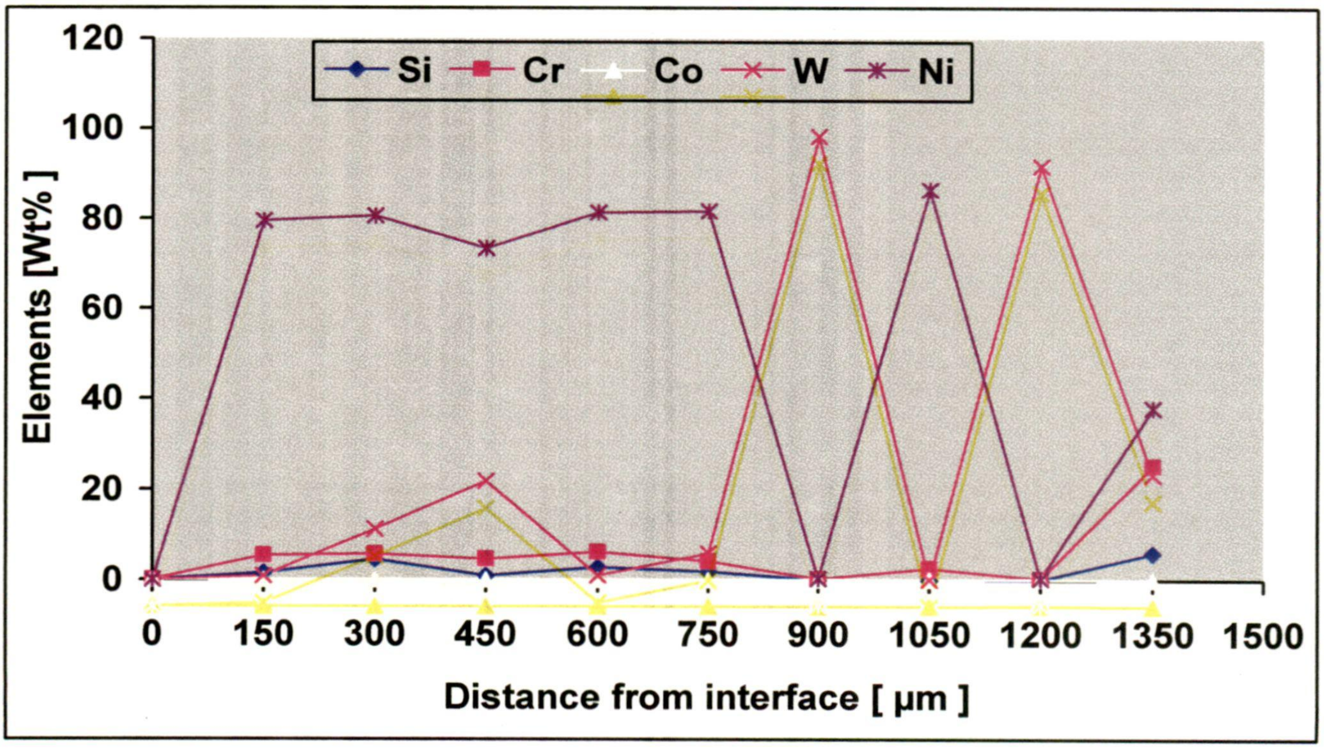


Fig. 3.2. EPMA analysis of Ni-WC coating.

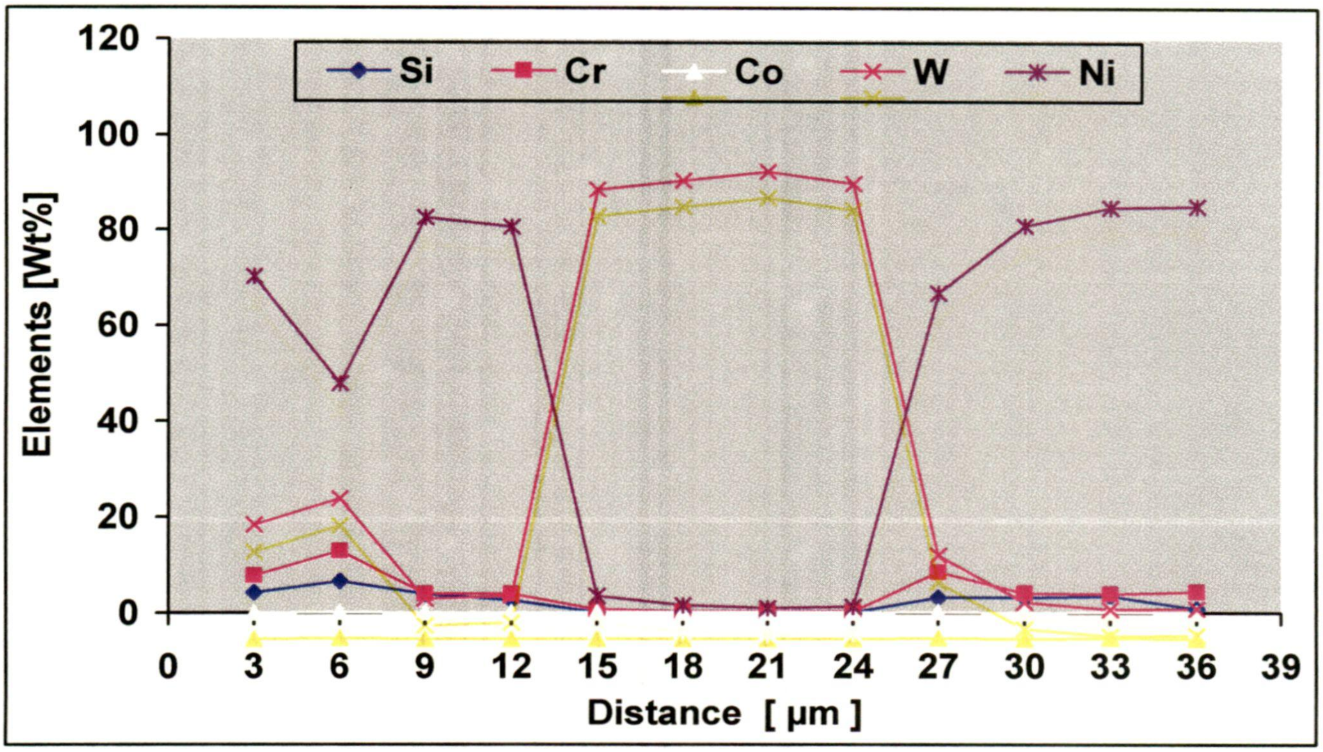


Fig. 3.3. EPMA analysis of carbide particle present in Ni-WC coating.

Electron probe micro analysis was done, for heat treated coating, across the coating from the interface of base and coating. The weight percentage of different elements (mainly Ni, W, Co, Cr, and Si) present across the coating from interface, towards the coating was shown in Fig. 3.2. Across the coating the approximate weight percentage of different elements is 80-85%Ni, 5-10%Cr, 5-8%W, 3-6%Si. At some points in the coating the percentage of Tungsten is high, signifying that the point of analysis lied on a carbide particle (Fig. 3.2). This will be clearer as we observe (Fig. 3.3) EPMA results done across carbide particle, visible in microstructure (Fig. 3.1b). This carbide particle is rich in Tungsten and a minimum of 90% to a maximum of 98% is present at this particle.

3.3. Hardness and wear behavior

Normal Vickers hardness was measured for both heat treated and non heat treated coatings, under 50N load. In non heat treated condition VHN is 1246 and in heat treated condition VHN is 1033. In non heat treated condition the carbide particles are uniformly distributed, so the hardness number is higher in non heat treated condition.

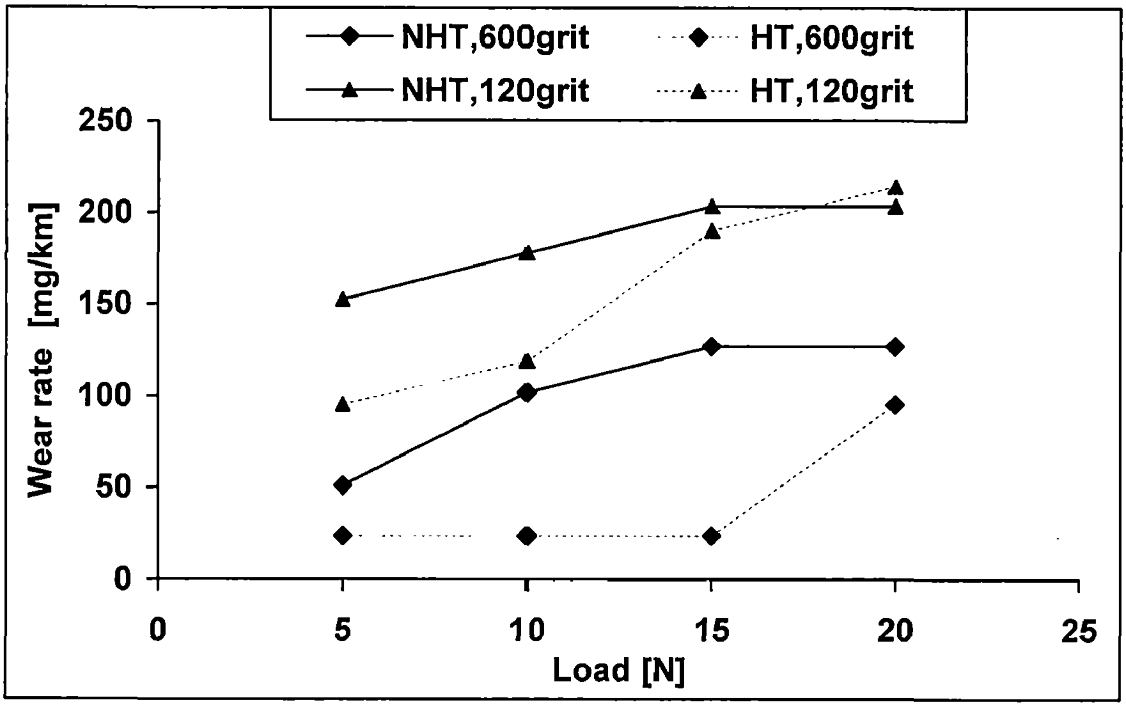


Fig. 3.4. Wear rate Vs applied load in Ni-WC coating.

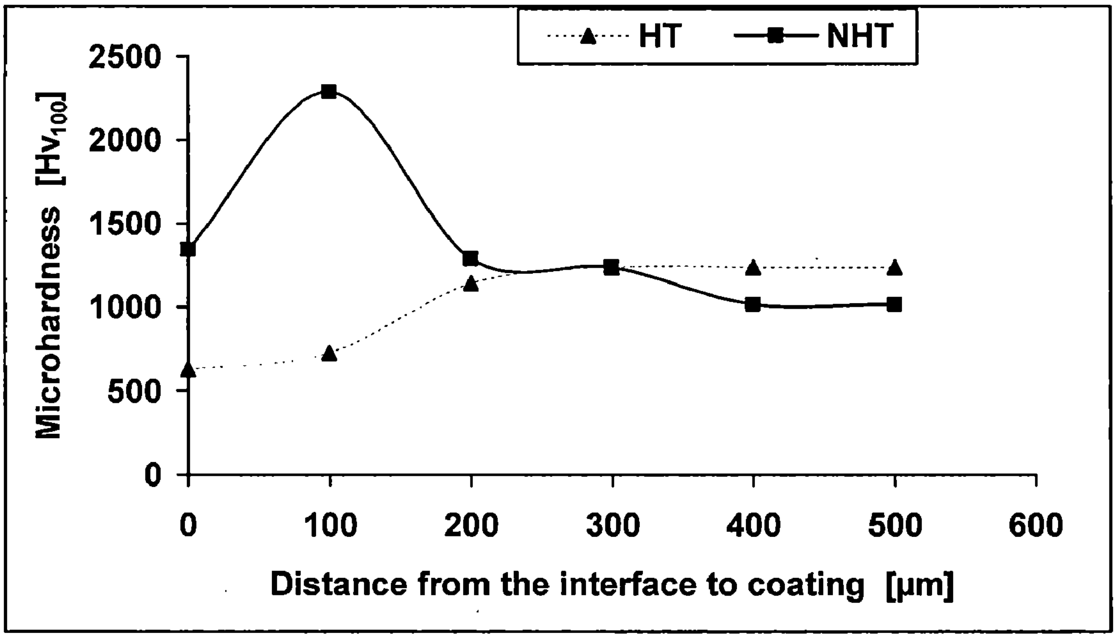


Fig. 3.5.Microhardness Vs distance from interface in Ni-WC coating.

The wear rates Vs normal applied load under different testing conditions is shown in Fig. 3.4. Against the 600grit abrasive counter surface at all load conditions, heat treated coating samples showed lower wear rates than non heat treated coating samples. Against 120grit abrasive medium the wear rate at lower loads (5, 10,15N) is less in heat treated coatings and against higher load (20N) wear rate is a little bit high in heat treated coating. The wear rate is mainly dependent on microhardness. Microhardness Vs distance from the interface of coating and base was shown in Fig. 3.5.The microhardness of the heat treated coating was higher than the non heat treated sample. So in all the cases, except against 120grit abrasive medium and 20N normal load, the wear rate is less in heat treated coatings and more in non heat treated coatings.

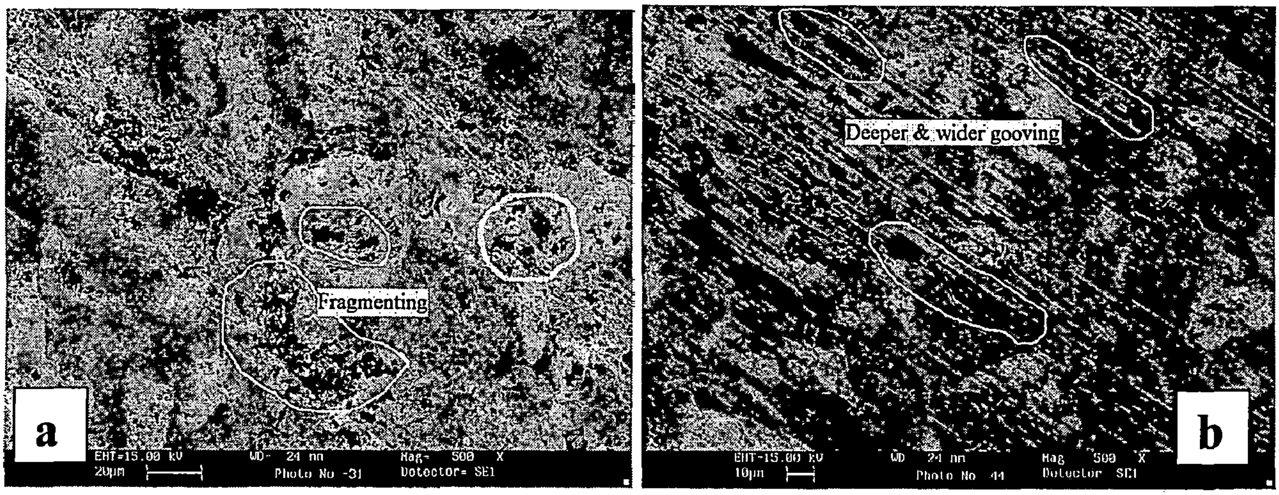
The secondary phase of carbide particles embedded in the eutectic matrix has higher micro-hardness than the remaining eutectic matrix. The average microhardness of the carbide particles present in non heat treated condition is around 5035Hv and in heat treated condition is around 4450Hv. Also the normal Vickers hardness number in heat treated condition (1033VHN) is less than in non heat treated condition (1246VHN). This may be probably due to the difference in microhardness of carbide particle in heat treated and non heat treated condition.

At higher loads (20N) and against 120grit abrasive medium, wear rate was controlled by the carbide particle. And the micro-hardness of these carbide particles in non heat treated coating is more than the heat treated coating. So wear rate against 120grit and 20N load is less in non heat treated condition. The peak in non heat treated condition is probably due to partial fall of the indenter on carbide phase (Fig.3.5).

3.4. SEM analysis

SEM images of worn out surfaces of coating samples tested against 600grit were shown in Fig. 3.6. In non heated coating (Fig. 3.6 b) we can observe deep wear tracks in matrix and also scratches across the carbide particles. Where as in heat treated coatings there are no tracks across the carbide particles, instead along the grain boundary we can observe wear tracks. Under an applied normal load of 20N the material was removed along the carbide particle grain boundary (Fig. 3.6 c).

SEM images of worn out surfaces of coating samples tested against 120grit are shown in Fig. 3.7. Coatings tested under 5N load are shown in Fig. 3.7 a&b, and (Fig. a) is heat treated coating and (Fig. b) is non heat treated coating. We can observe less wear tracks in heat treated coatings (Fig. 3.7a) and more tracks in non heat treated coatings (Fig. 3.7b), which is inline of wear results (Fig. 3.4). At higher loads (20N) coating in heat treated condition is subjected to more wear rate than in non heat treated condition. Worn out coatings under testing conditions of 120 grit abrasive medium and 20N load were taken SEM images and are shown in Fig. 3.7 c&d. In heat treated condition (Fig. 3.7c), we can observe wear tacks or scratches formed on the carbide particle and wedging effect taken place. But in non heat treated condition (Fig. 3.7d) we can see the carbide particles with no wear marks on them. After heat treatment, carbide particles will wear out easily under higher loads. Thus more wear rate under higher loads (20N) after heat treatment.



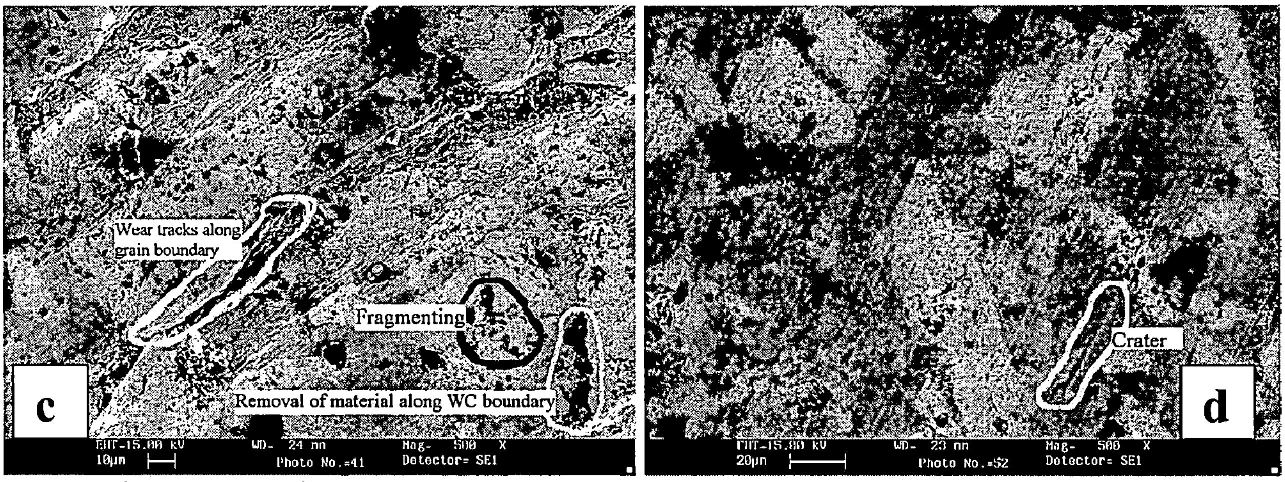


Fig. 3.6. SEM images of the worn out samples against 600grit abrasive medium.
a) Heat treated-5N; b) Non heat treated-5N; c) Heat treated-20N; d) Non heat treated-20N

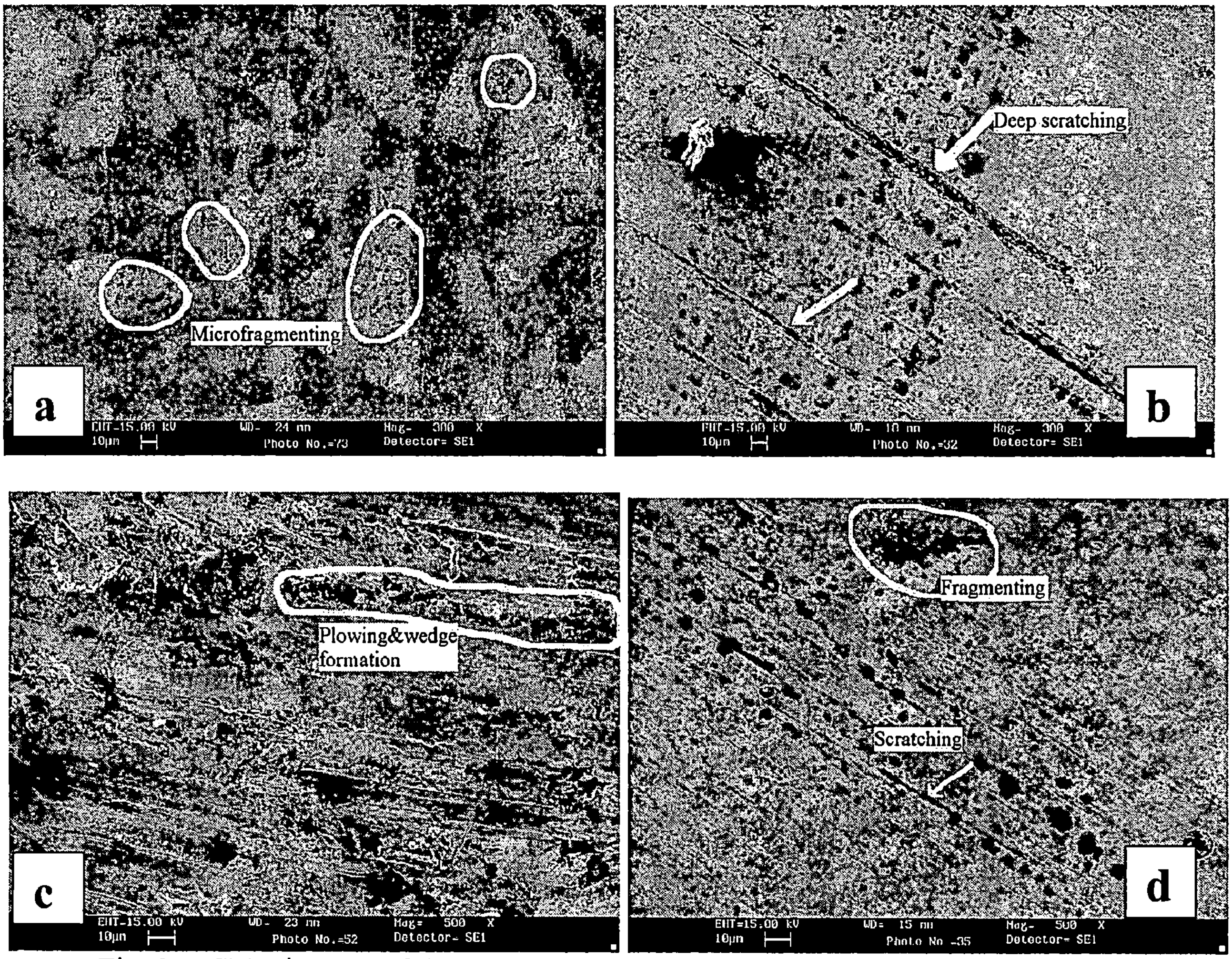


Fig. 3.7. SEM images of the worn out samples against 120grit abrasive medium
a) Heat treated-5N; b) Non heat treated-5N; c) Heat treated-20N; d) Non heat treated-20N

3.5. Summary

- Heat treatment increased the grain size of carbide particle and uneven distribution in nickel eutectic matrix.
- In non heat treated coatings carbide particles are evenly distributed in Ni eutectic matrix.
- Heat treatment improved microhardness in Ni based WC coatings.
- Similarly wear rates are reduced after heat treatment.
- The embedded carbide particles reduced the wear rates as carbide particles are harder than Nickel matrix.
- Heat treated coatings under higher loads showed higher wear rates than non heat treated coatings.

The mechanical and metallurgical characterization such as microstructure study, normal hardness, microhardness, EPMA, SEM analysis of unmodified powder (Ni-Cr-Si-B) coatings and modified (by adding Chromium carbide) powder coatings were done.

4.1. UNMODIFIED POWDER COATINGS

Unmodified powder coatings were developed on mild steel substrate with surfacing powder of Ni-Cr-Si-B (EWAC 1004EN). Mechanical and metallurgical characterizations of coatings were done with the help of different tools available.

4.1.1. Microstructure and EPMA

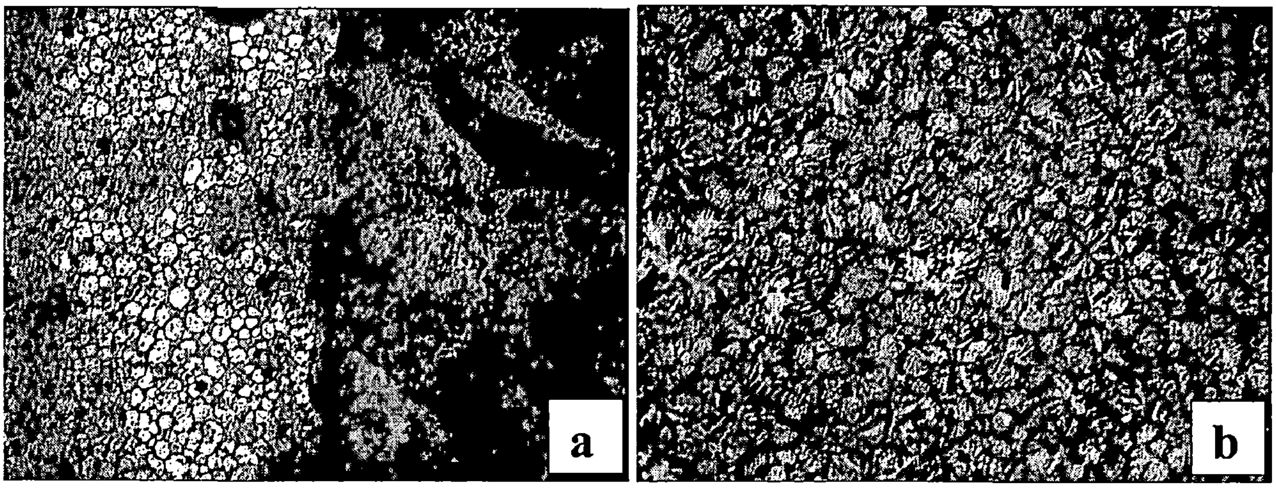


Fig. 4.1. Micro-structures of unmodified powder coated samples.
a) Non heat treated coating (320X) b) Heat treated coating (500X)

The microstructures of the non heat treated and heat treated coatings are shown in Fig. 4.1 (a, b). The microstructure shows cellular grains of Ni solid solution formed in eutectic matrix. EPMA was carried out for a heat treated sample of unmodified powder (EWAC 1004EN) coating. EPMA analysis was carried out across the coating from interface (Fig. 4.2) and across the cellular grain (Fig. 4.3). The major element present in the coating is Ni (Fig. 4.2). It is more or less uniform through out the coating (97-99%). Low melting eutectic phase present at grain boundary (Fig. 4.2). EPMA analysis across the cellular shape grain (Fig. 4.3) reveals that these grains are Ni solid solution, formed in the matrix during solidification of coating.

The weight percentage of different elements present in unmodified coating is 95-98%Ni, 2-3%Si, and Cr, Co are less than 1%. And across the Ni cellular grain there is only Si around 5% and remaining is Ni. The weight percentage of different elements present in the eutectic matrix is 38% Si, 3.4% Ni, 0.012% Co, and 0.047% Cr. In the EPMA report across the coating (Fig. 4.2) at a distance of 400μm from interface we can observe that the testing point fall on the eutectic matrix. Formation of nickel solid solution was started with pure nickel particle and then coring effect was taken place during solidification. This phenomenon can be observe in Fig. 4.3, where at a point of 12μm distance the weight percent of other elements except Ni is very less (Si--0.77%, Cr--0.185%, Co--0.11%).

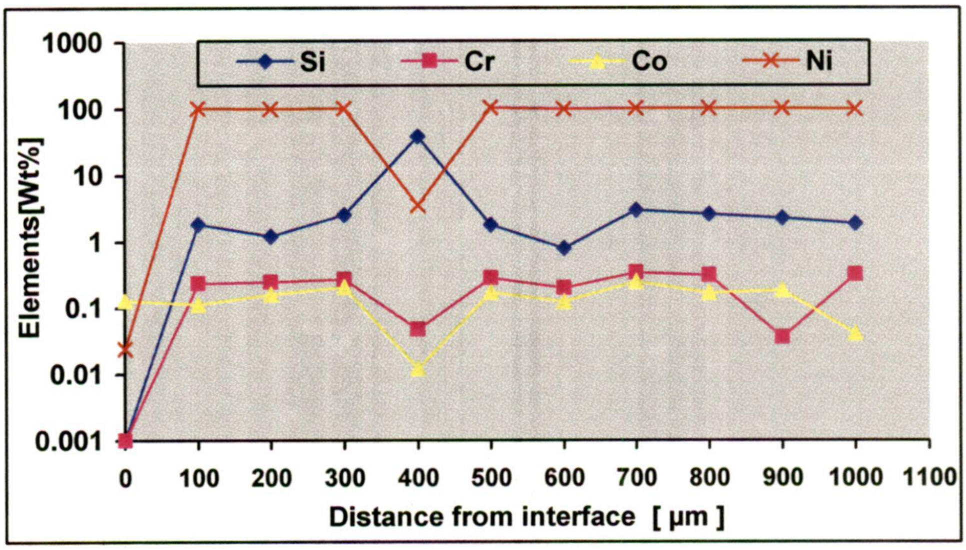


Fig. 4.2.EPMA analysis of unmodified powder coating from interface.

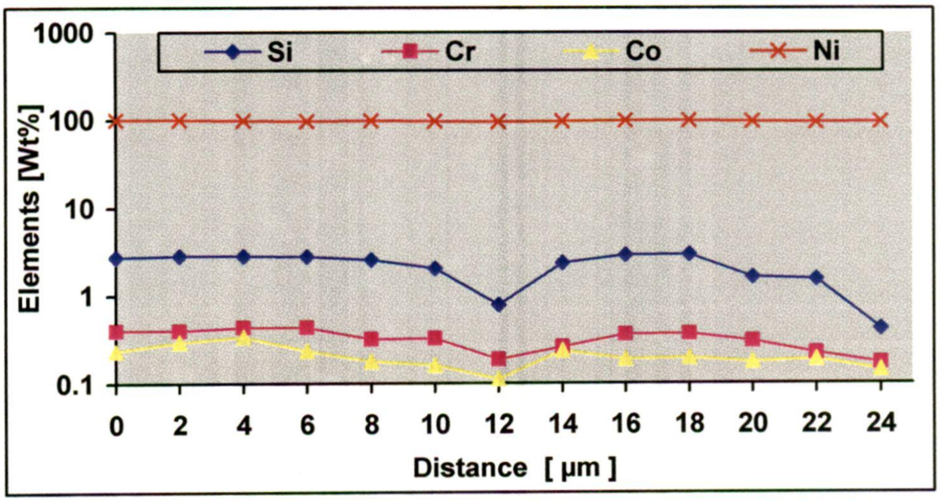


Fig. 4.3.EPMA analysis of unmodified powder coating across the phase.

4.1.2. Hardness and wear behavior

Normal hardness of the coating was measured in terms of Vickers scale (VHN). The hardness of coating in non heat treated condition is 195VHN and that after heat treatment is 228VHN. The hardness was improved after heat treatment. So we can expect less wear rates in heat treated coatings. Microhardness was improved after heat treatment.

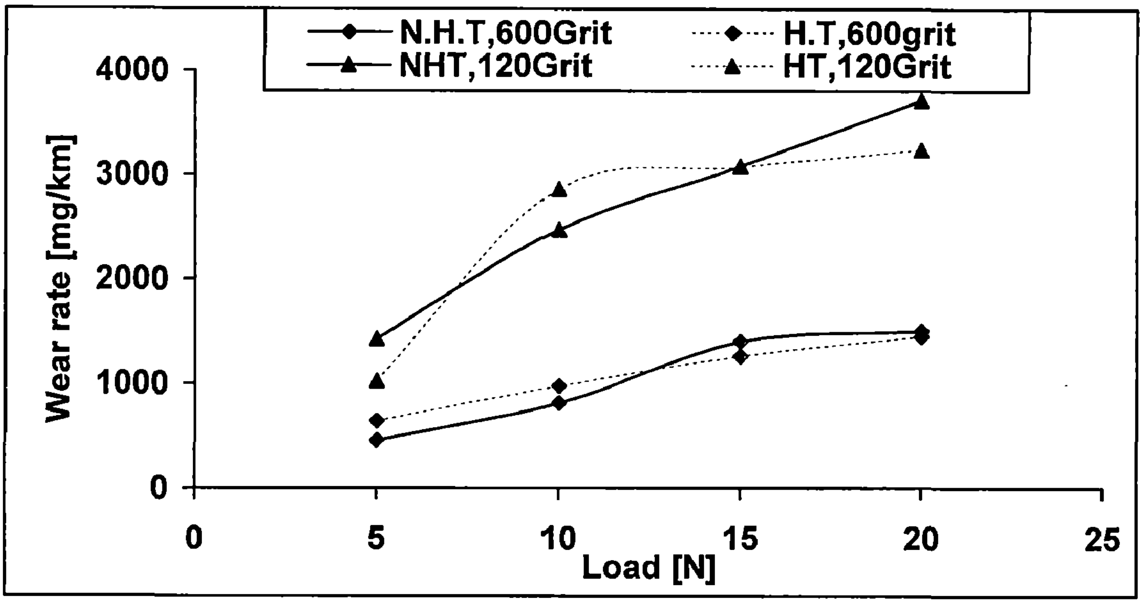


Fig. 4.4. Wear rate Vs normal load in unmodified Ni-Cr coatings.

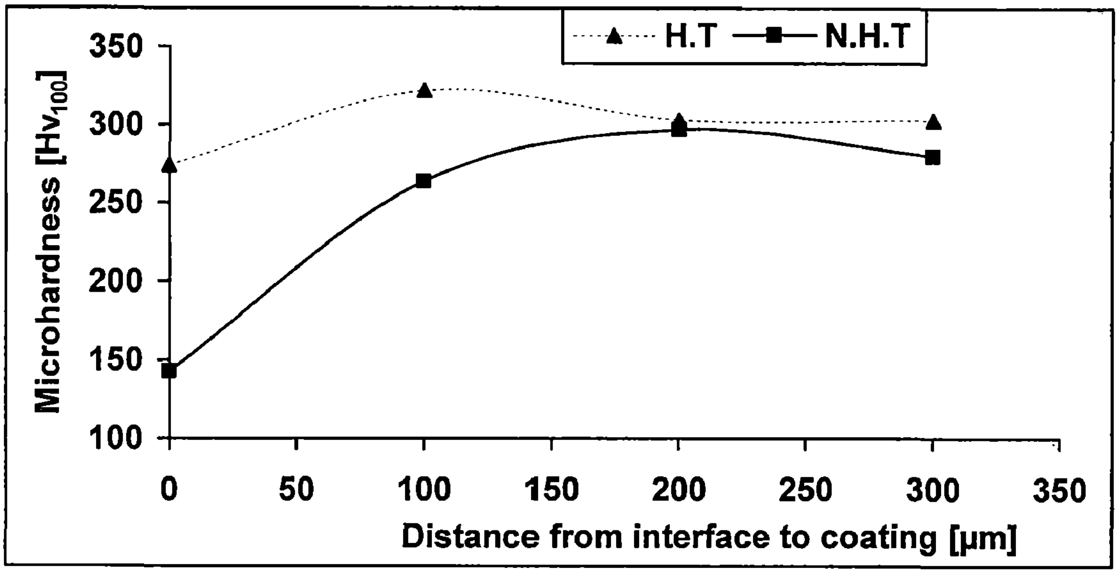


Fig. 4.5.Microhardness Vs distance from interface in unmodified Ni-Cr coatings.

Microhardness of unmodified powder coating Vs distance from interface is shown in Fig. 4.5. Microhardness in heat treated coating is more than of non heat treated coating.

Microhardness of the heat treated sample is 300Hv, and that of non heat treated sample is 280Hv. Wear behavior of the heat treated and non heat treated coatings against different abrasive mediums is shown in Fig. 4.4. Wear rate in case of heat treated coatings is less than the non heat treated coatings against both 120grit and a 600grit abrasive medium at lower and higher loads (Fig. 4.4). Wear rate of non heat treated sample is more against 120 grit abrasive medium at an applied load of 20N (3.7gr/km) and after heat treatment wear rate was reduced (3.2gr/km).

4.1.3. SEM Analysis

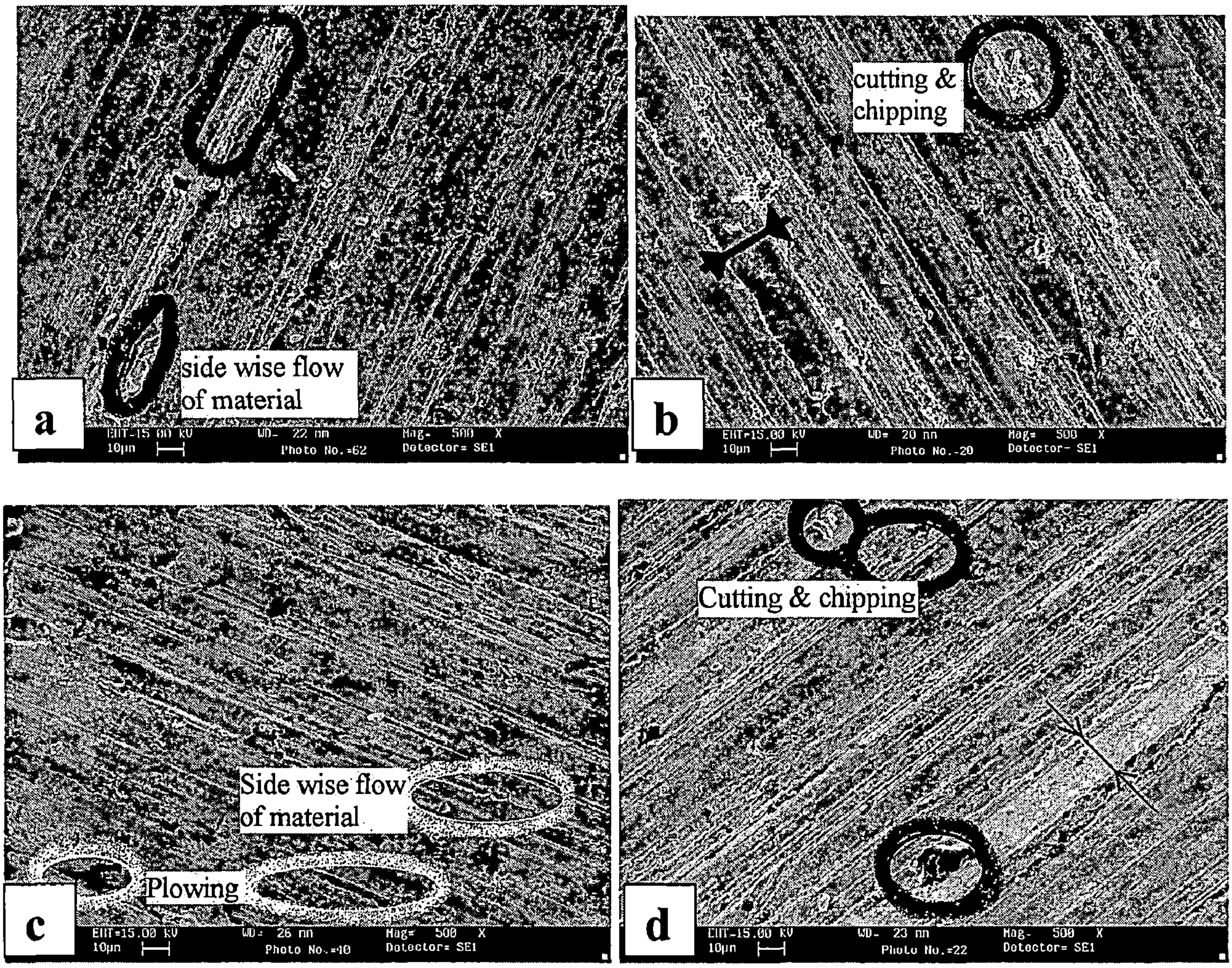


Fig.4.6. SEM photographs of the worn out unmodified powder coated samples against 120grit abrasive medium. a) Heat treated-5N load; b) Non heat treated-5N load; c) Heat treated-20N load; d) Non heat treated-20N load;

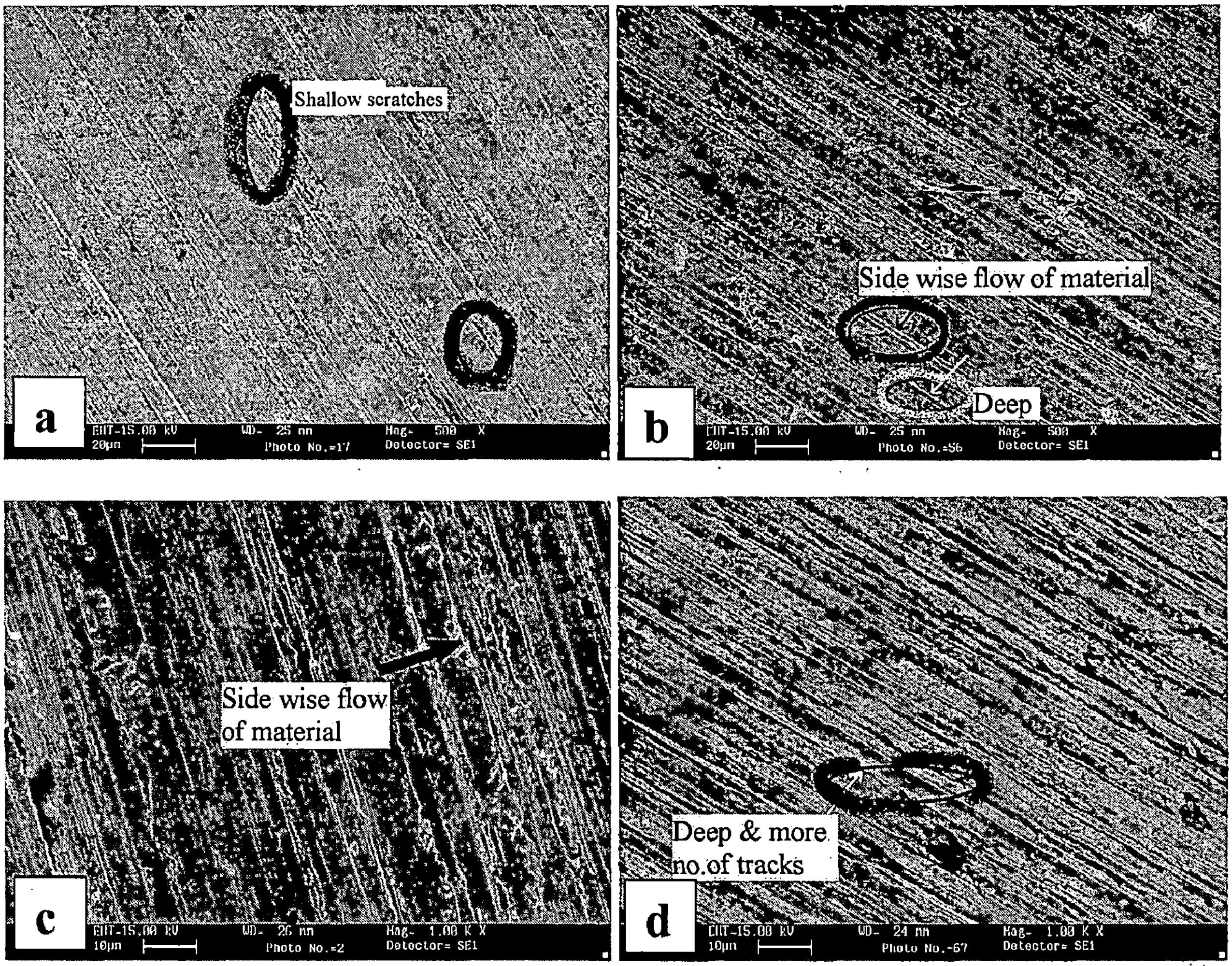


Fig.4.7. SEM photographs of the worn out unmodified powder coated samples against 600grit abrasive medium. a) Heat treated-5N load; b) Non heat treated-5N load; c) Heat treated-20N load; d) Non heat treated-20N load;

SEM images of the worn out samples of coatings in heat treated and non heat treated conditions against different abrasive surfaces and load conditions are shown in Fig. 4.6&4.7. Wear rate in heat treated coatings is less than the non heat treated coatings (Fig. 4.4) against 120 grit abrasive medium and 5N load. Wide and deeper wear tracks are visible in non heat treated coatings (Fig. 4.6 b & d). Plowing was taken place in heat treated coatings (Fig. 4.6 a & c). Wear results were supported by the SEM images (Fig. 4.6. a&b), where wear tracks are more in non heat treated coating (Fig. 4.6 a) and less in heat treated coating (Fig. 4.6 b). More the wear tracks more the wear rate, which means that the wear rate is less in heat treated coating and more in non heat treated coating. Similarly SEM images of the wear tested coatings against 120grit abrasive medium and 20N load were shown in Fig. 4.6 (c&d). Heat treated coating shows less wear tracks (Fig. 4.6c) than the non heat treated coating (Fig. 4.6d). As the wear test shows (Fig. 4.4), SEM images are also supporting the wear test data.

SEM images of the worn out samples in heat treated and non heat treated condition, against 600grit abrasive surfaces are shown in Fig. 4.7. The wear tracks formed against 600grit abrasive medium are narrow/shallow than the wear tracks formed against 120grit abrasive surface. Wear tracks formed under low loads (5N) in non heat treated coating (Fig. 4.7b) shows wedge type abrasive wear. And wear tracks formed in heat treated coating (Fig. 4.7a) shows plowing mechanism of material removal. The wear tracks formed in non heat treated coating under higher load (Fig. 4.7d) are more and deeper compared to heat treated coating (Fig. 4.7c). Deeper the wear tracks more wear rate and vice versa. These SEM images are evident for the wear results (Fig. 4.4).

4.2. MODIFIED POWDER COATINGS:

Chromium carbide (10--20%) is added to unmodified (Ni-Cr eutectic mixture) powder of 1004EN to get modified powder coatings. Metallurgical characterization such as microstructure study, microhardness test, normal Vickers hardness, EPMA and SEM analysis were studied to understand the wear behavior of these coatings.

4.2.1. Microstructure and EPMA

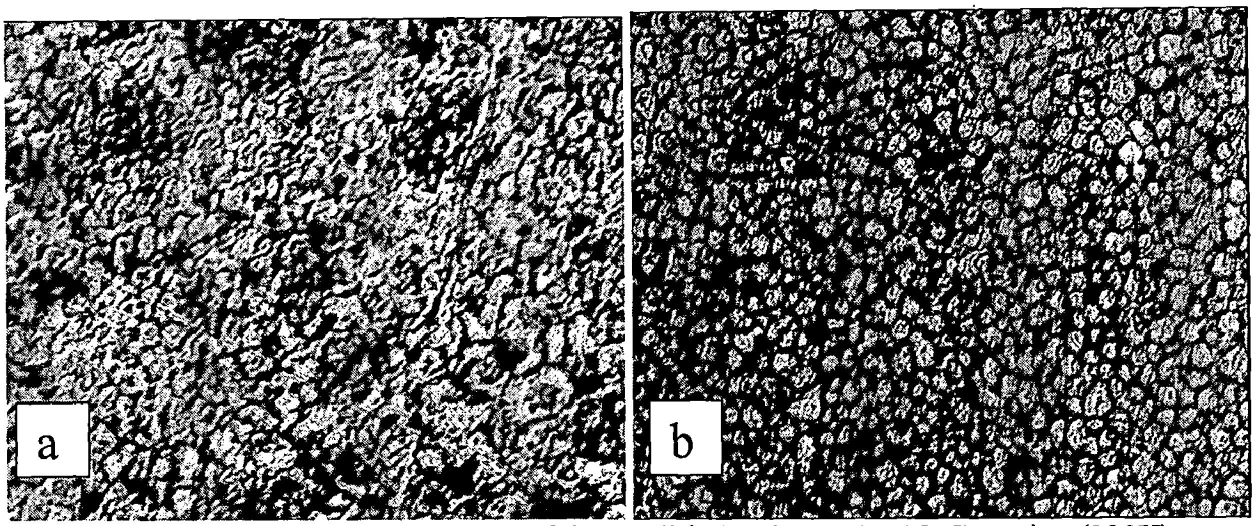


Fig. 4.8.Microstructures of the modified 1004EN-10%CrC coating.(320X)
a) Heat treated coating b) Non heat treated coating

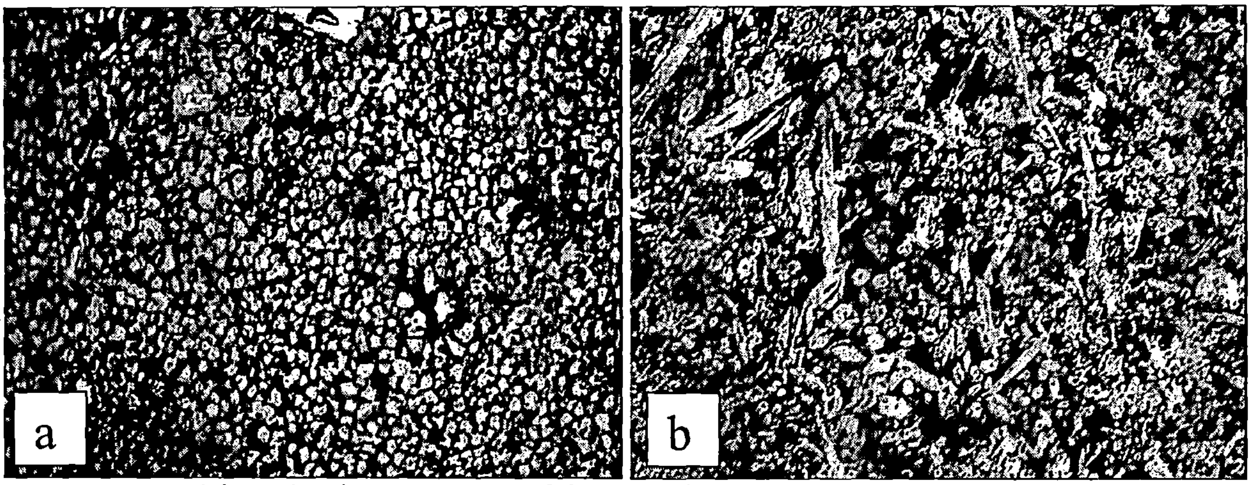


Fig. 4.9.Microstructures of the modified 1004EN-20%CrC coating.(320X)
a) Heat treated coating b) Non heat treated coating

Microstructures of the modified 1004EN-10%CrC powder coatings in non heat treated and heat treated condition are shown in Fig. (4.8). Grain size of carbide particle was increased after heat treatment (Fig. 4.8 a) in to the matrix. The microstructure of the non heat treated sample (Fig. B) shows the cellular grains. The microstructures of the modified 1004EN-20%CrC powder coating in non heat treat condition and after heat treatment are shown in Fig. (4.9 a&b). The carbide particles (white color) are visible in cellular and needle shape after heat treatment. But after heat treatment (Fig. 4.9a) they are not visible, probably because of precipitation during heat treatment at 800^0C.

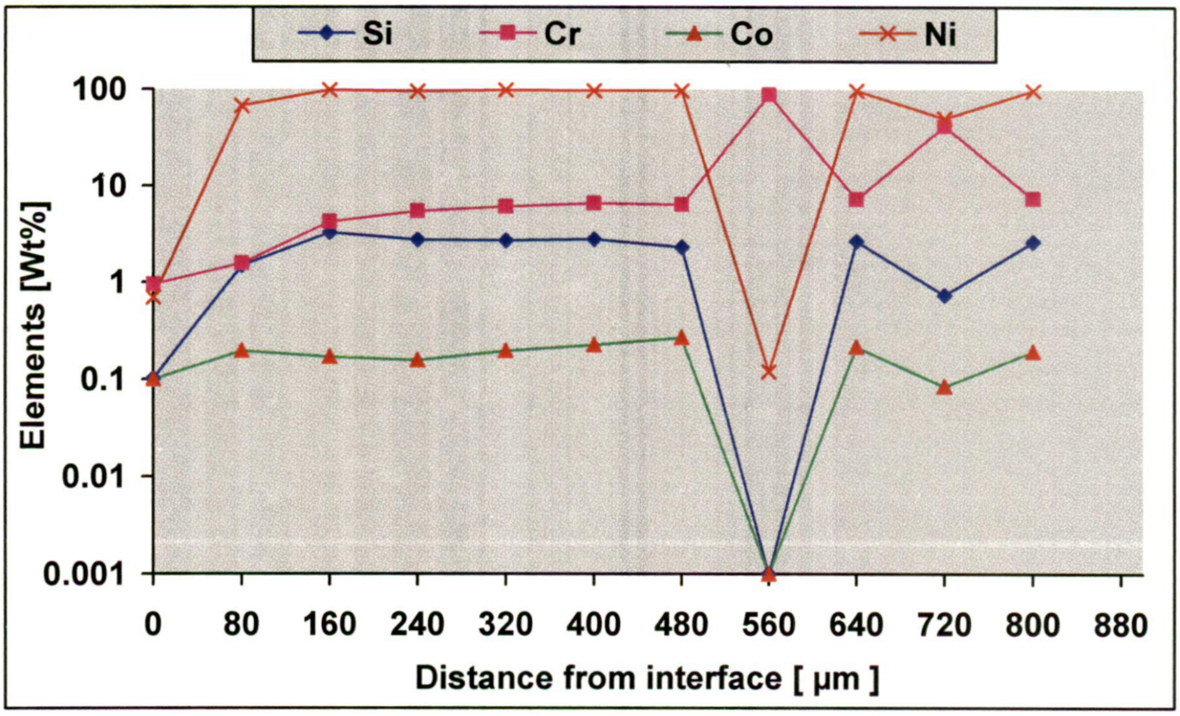


Fig. 4.10.EPMA analysis of modified powder coating across the coating.

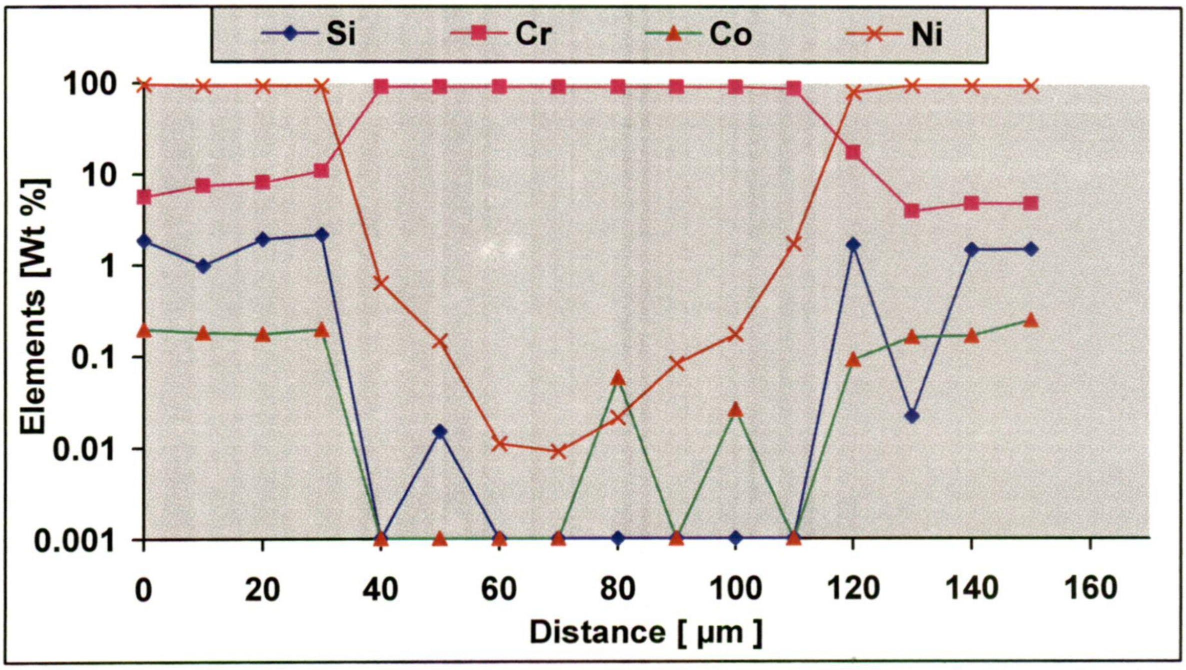


Fig. 4.11.EPMA analysis of modified powder coating across the carbide particle.

EPMA was carried out after heat treatment for modified powder 1004EN-20%CrC coating and results were shown in Fig. 4.10&4.11. The percentage of different elements across the coating from interface is shown in Fig. 4.10 and the presence of different elements across the white carbide particle, visible in microstructure (Fig. 4.9B), is shown in Fig. 4.11. Modified powder coating has mainly approximately 94-96%Ni and 4-5%-Cr, 2-3% Silicon, and Cobalt is less than 1%. And across the carbide particle 99.5%Chromium and remaining (Si, Co) elements are less than0.1%. So it can be inferred that Chromium is in the form of Chromium carbide, which is a hard phase.

4.2.2. Hardness

Normal hardness of the modified powder coatings are measured in-terms of Vickers hardness. Hardness of modified 1004EN-10%CrC coating, in non heat treated condition (286VHN) is more than that of heat treated coating (266VHN). Similarly hardness of modified 1004EN-20% powder coating, in non heat treated coating (578VHN) is more than that of heat treated coating (309VHN). Addition of carbide particles to the coating improved hardness, but heat treatment affect adversely in modified powder coatings.

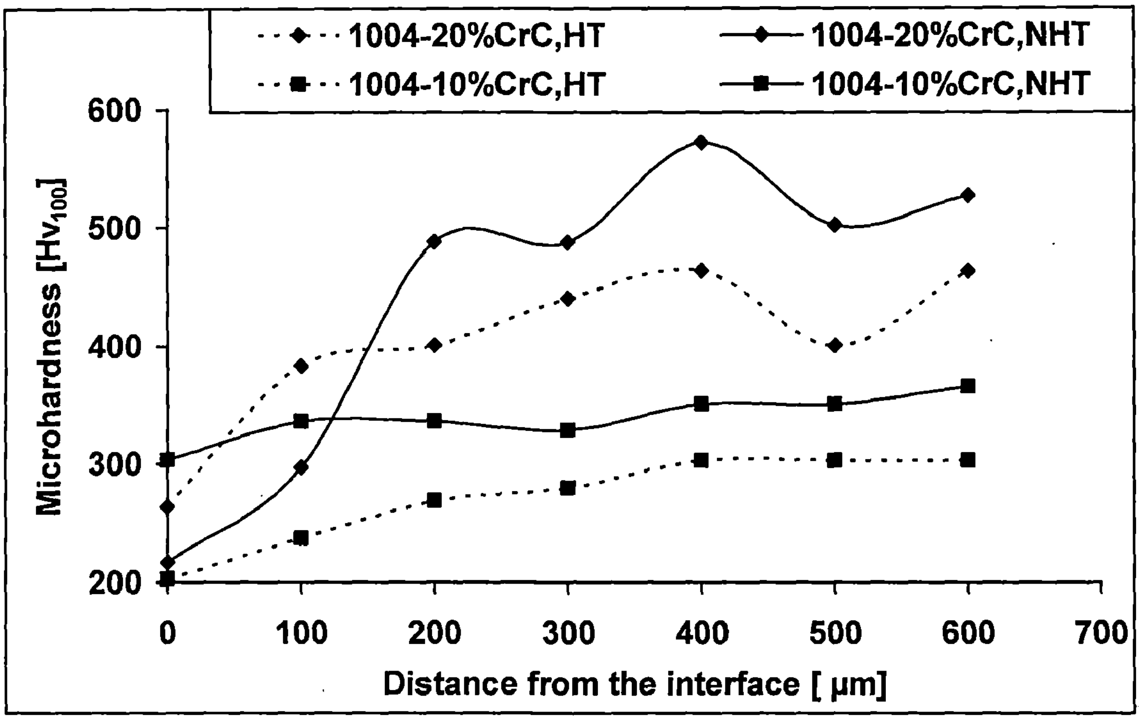


Fig. 4.12.Microhardness Vs distance from interface in modified powder coatings.

The microhardness of modified powder coatings in heat treated and non heat treated conditions are shown in Fig. 4.12. Microhardness is measured from the interface towards the coating for both heat treated and non heat treated coatings. In the heat treated coatings the microhardness was reduced than that of non heat treated coatings. Addition of the Chromium carbide increased the microhardness of the coatings compared to unmodified powder coatings (Fig. 4.13, 4.14). The addition of Chromium carbide improved microhardness in both non heat treated coatings (Fig. 4.13) and heat treated coatings (Fig. 4.14). Microhardness was high in case of modified powder 1004EN-20%CrC coating than modified powder 1004EN-10%CrC coating. That is more the amount of Chromium carbide is more microhardness.

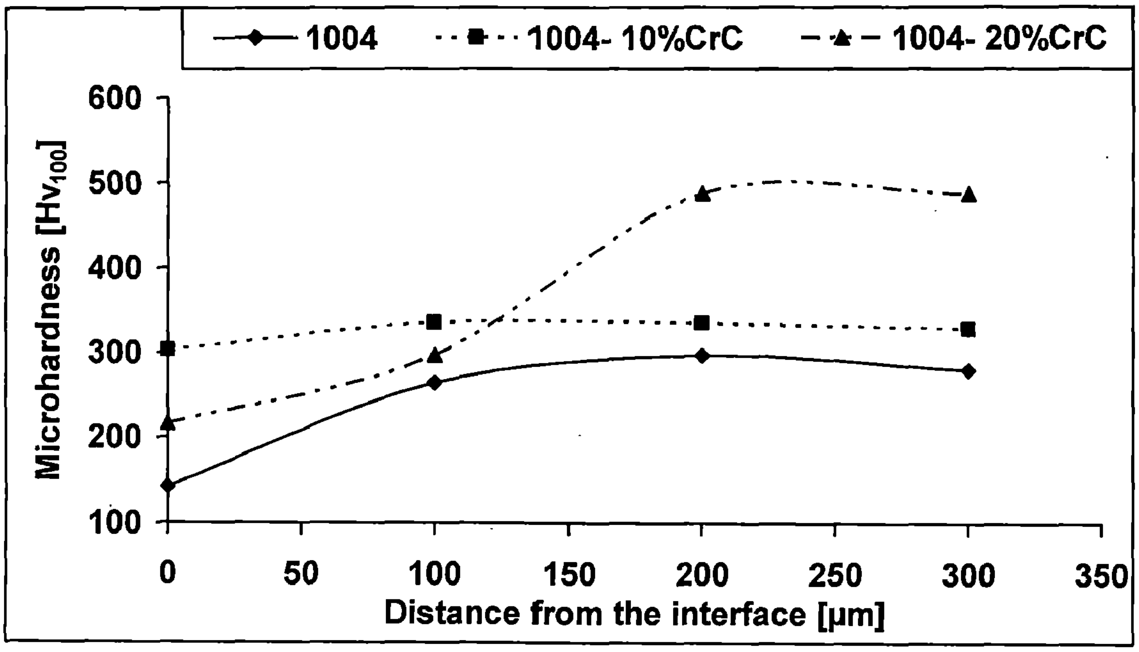


Fig. 4.13.Microhardness Vs distance from interface in Ni-Cr (1004EN) non heated coatings.

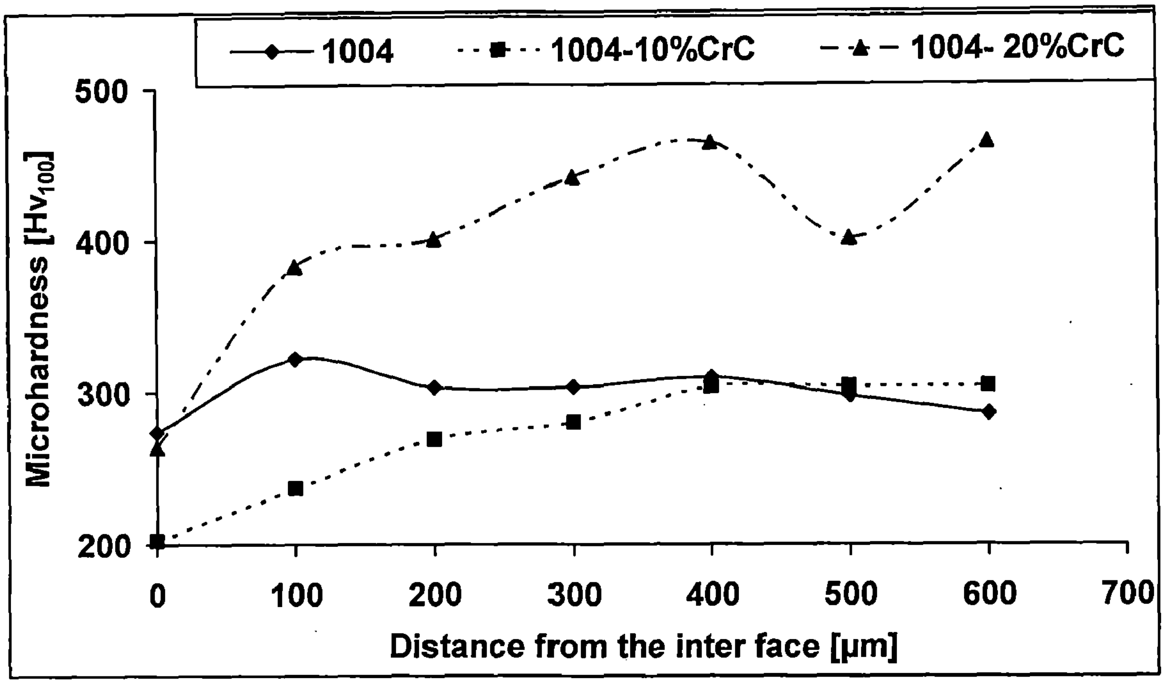


Fig. 4.14.Microhardness Vs distance from interface in Ni-Cr (1004EN) heat treated coatings.

4.2.3. Wear behavior

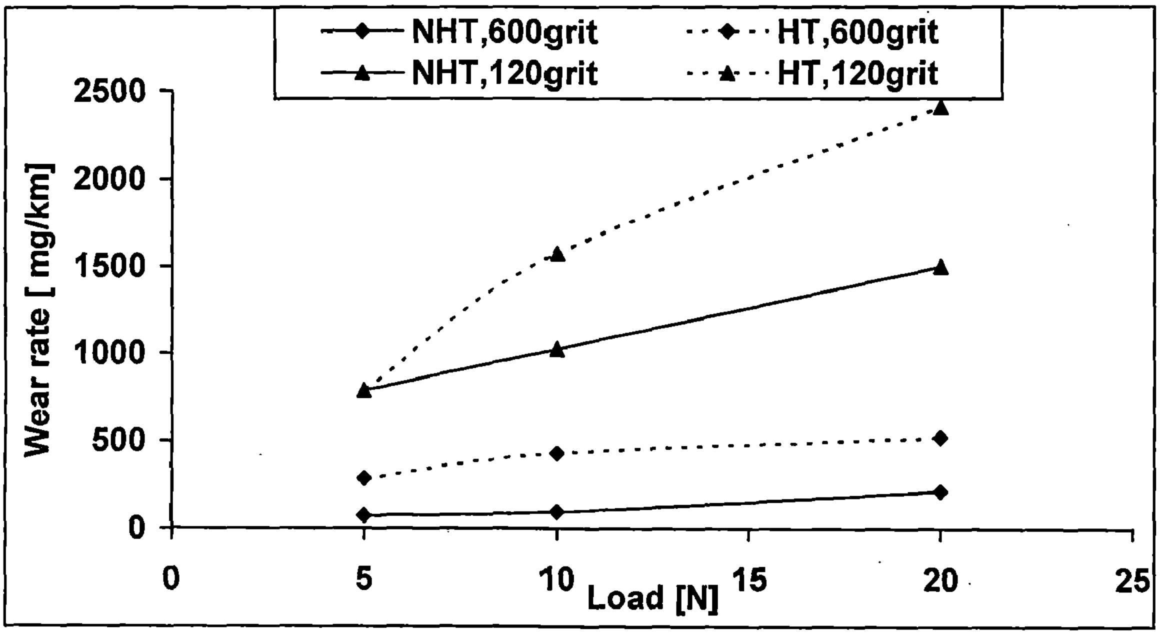


Fig. 4.15.Wear rate Vs normal load in modified powder 1004EN- 10%CrC coating.

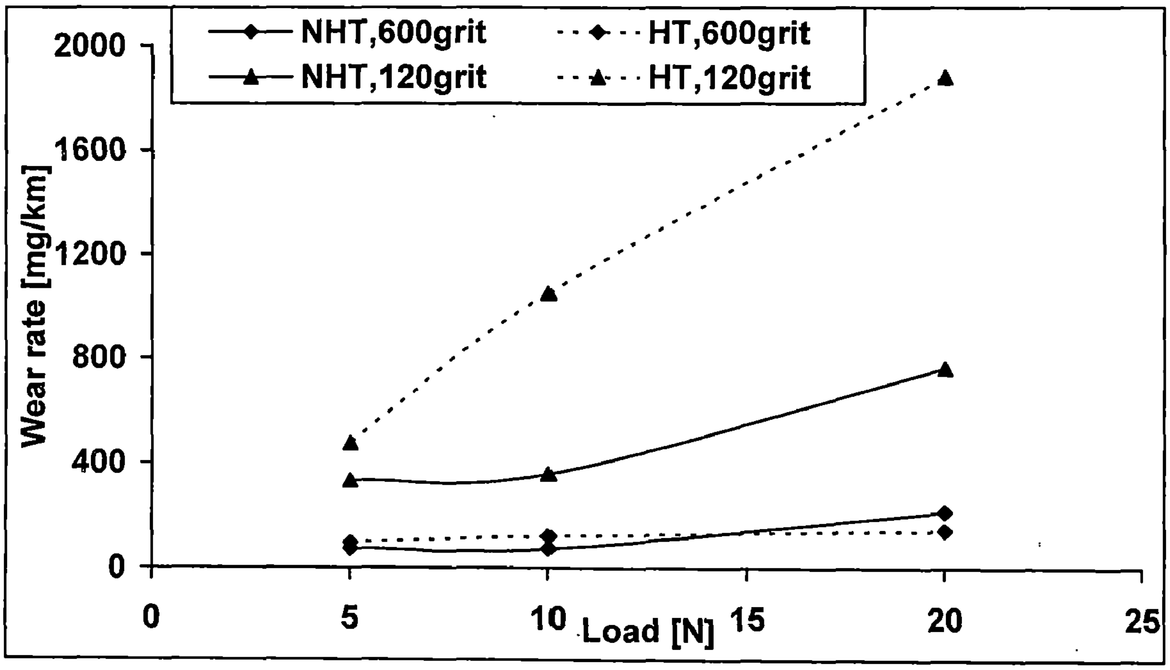


Fig. 4.16. Wear rate Vs normal load in modified powder1004EN- 20%CrC coating.

The wear behavior of the modified coatings in heat treated and non heat treated conditions are shown in Fig. 4.15&4.16. Modified powder coatings showed higher wear rate in heat treated condition compared to the non heat treated condition against both 600&120grit abrasive counter surface under all loads. Higher the microhardness is better the wear resistance. Wear results are in line of microhardness results. Particularly at higher loads against 120grit abrasive medium the wear rate is much higher in heat treated condition. As microhardness was improved after the addition of Chromium carbide particles (Fig. 4.13&4.14), the wear rates were reduced as shown in Fig. 4.17. Higher the Chromium carbide added, higher the microhardness and hence better wear resistance.

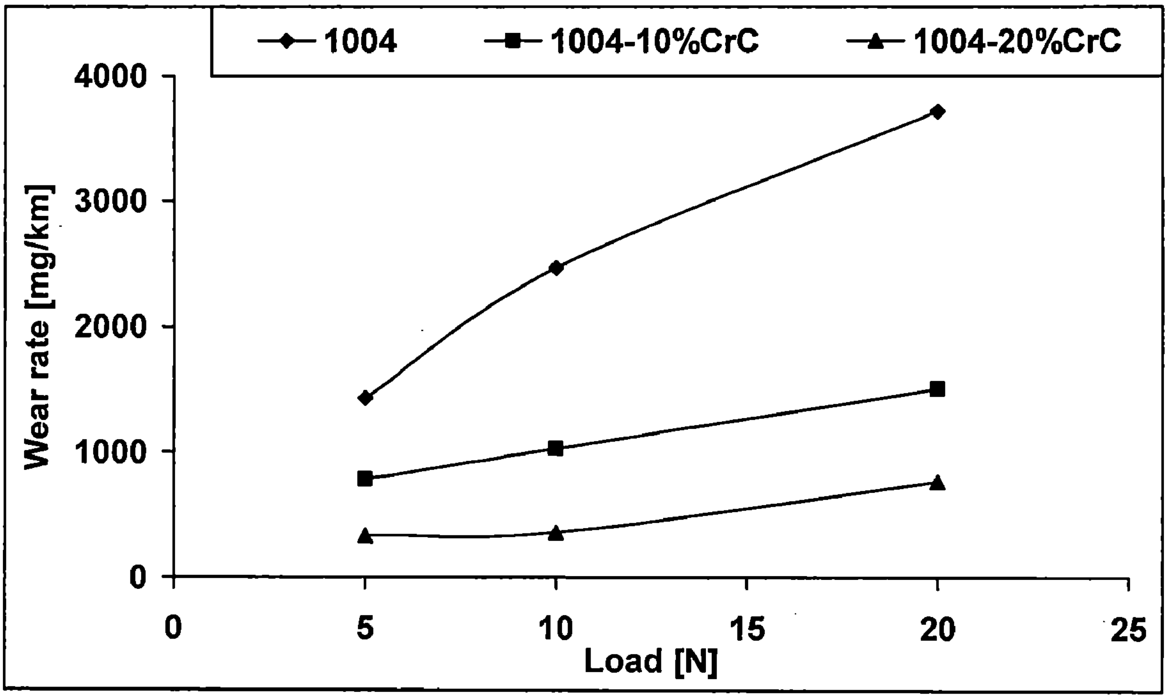


Fig. 4.17. Wear rate Vs applied load in non heat treated coatings against 120grit abrasive surface.

The addition of the Chromium carbide up to 20% improved the wear resistance of a minimum of 4-5 times than unmodified coatings and up to 10% addition improved 2-3 times that of unmodified coatings against 120grit abrasive counter surface in non heat treated condition. The same modified coatings show 7times better wear resistance against 600grit abrasive counter surface under above conditions. In case of heat treated modified coatings, wear resistance is double that of unmodified powder coatings against 120grit. In case of heat treated modified powder (1004EN-20%CrC) coatings, wear resistance is ten times that of unmodified powder coatings against 600grit abrasive surfaces.

4.2.4. SEM analysis

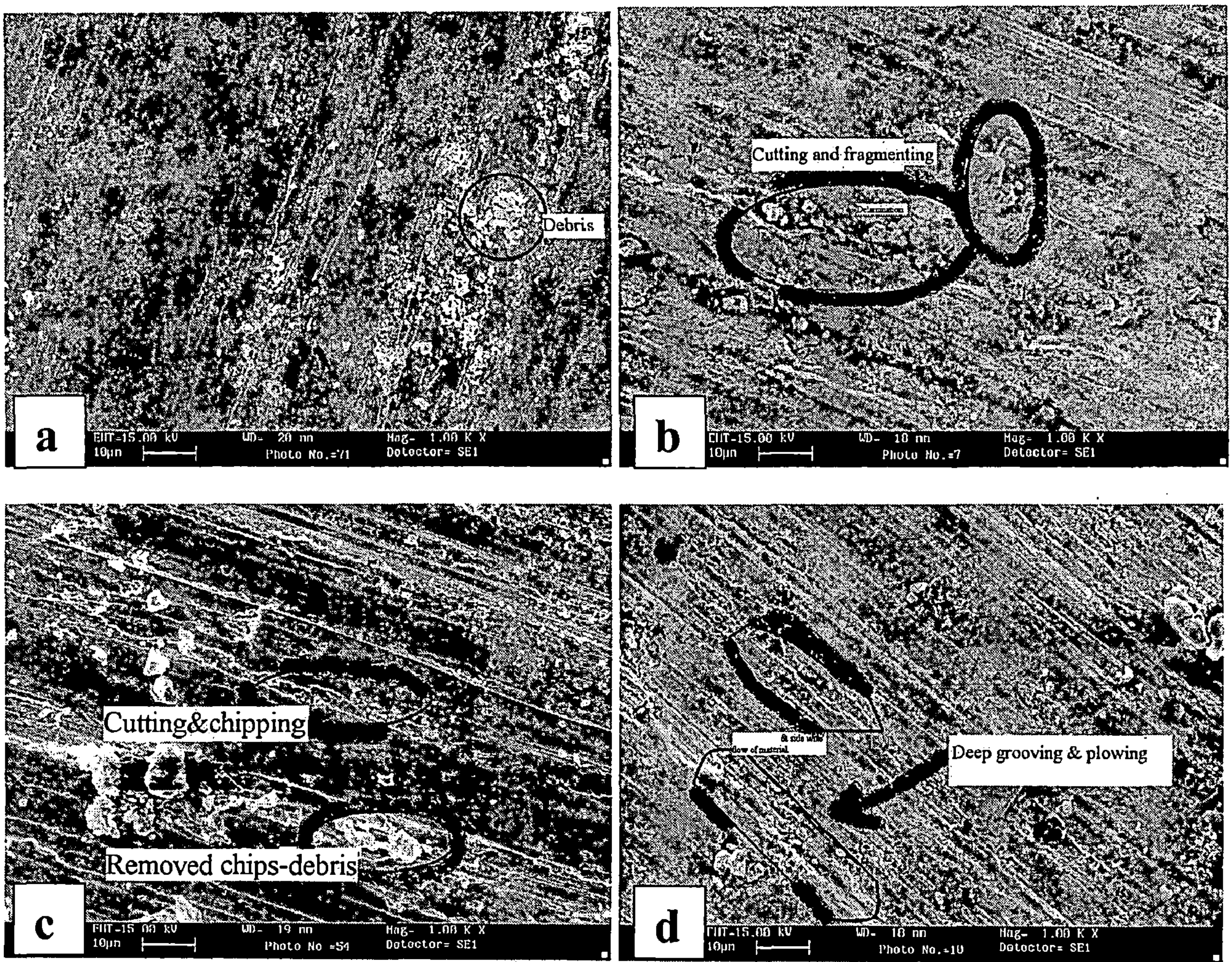


Fig. 4.18. SEM photos of the worn out surfaces of modified powder (1004EN-10%CrC) coatings tested against 120grit abrasive counter surface. a) Heat treated-5N; b) Non heat treated-5N; c) Heat treated-20N; d) Non heat treated-20N;

SEM images of the coating surfaces tested against 120grit abrasive surfaces were shown in Fig. 4.18. The worn out debris was visible in SEM images (Fig. 4.18 a & c). During wear test the material flowed side wise in non heat treated coatings (Fig. 4.18 b & d), whereas in heat treated coatings there is no side wise flow of material and huge amount of material was removed. That means delamination was taken place during wear in non heat treated coatings. Also higher micro-hardness in non heat treated coatings than heat treated coatings. So lower wear rates in non heat treated coating and higher wear rates after heat treatment (Fig. 4.15). So SEM images are supporting the wear results.

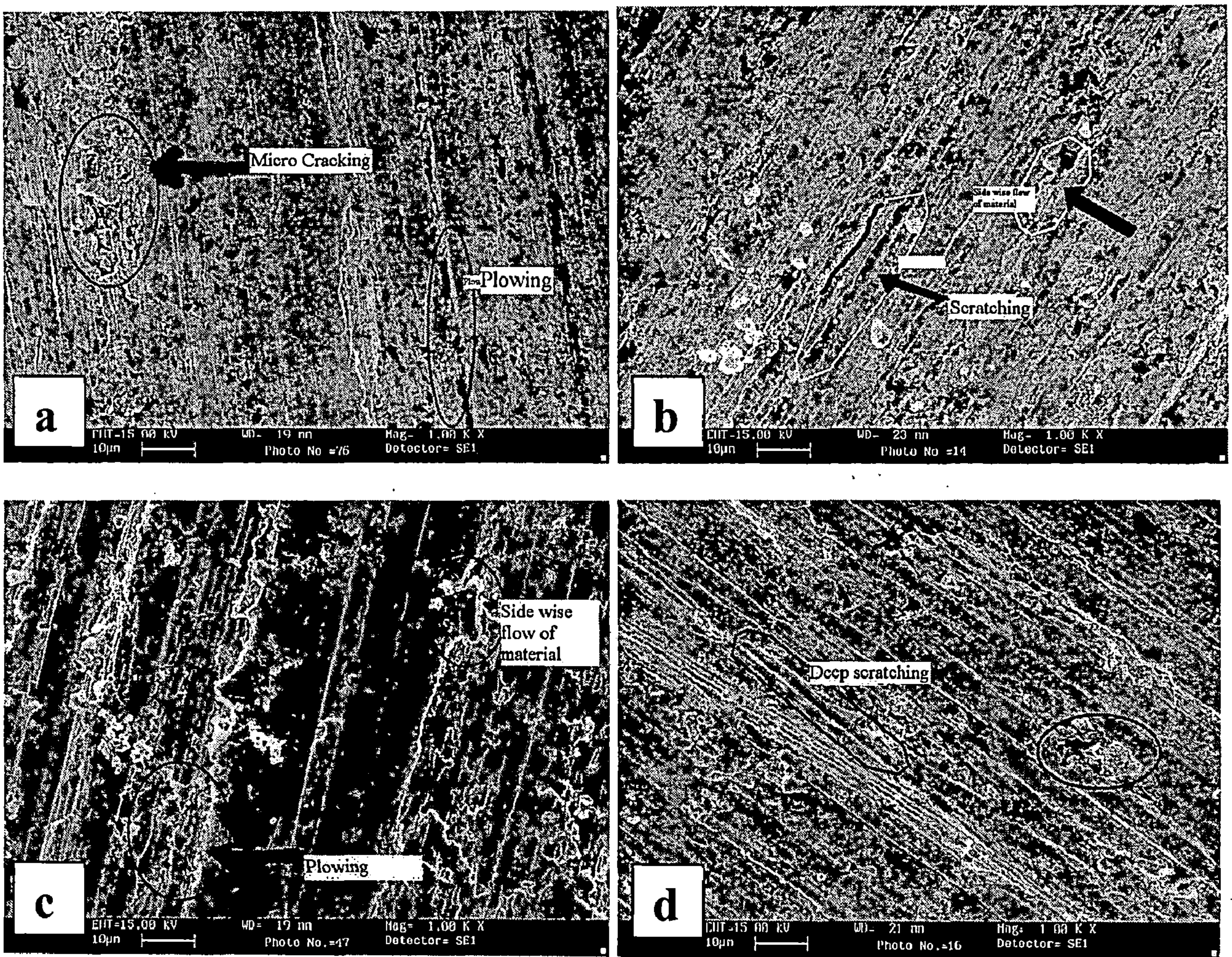


Fig. 4.19. SEM photos of the worn out surfaces of modified powder (1004EN-20%CrC) coatings tested against 120grit abrasive counter surface. a) Heat treated-5N; b) Non heat treated-5N; c) Heat treated-20N; d) Non heat treated-20N;

SEM images of the coating surfaces tested against 120grit abrasive surfaces were shown in Fig. 4.19.The removed material follows side wise and still adheres to the coating in non heat treated condition (Fig. 4.19 b & d). And the coating behaves like ductile, more resistance to removal of material. In case of heat treated coatings, clearly the material was removed and there is no side ways flows of material (Fig.: 4.19 a & c). At higher loads (20N), in non heat treated coatings the flaws of removed material were present side to the wear tracks (Fig. 4.19d). And these SEM images are evident for the wear results (Fig. 4.16).

4.3. Summary

- In modified powder coatings carbide particles are visible in microstructure in non heat treated coatings.
- In unmodified powder coatings nickel solid solution in cellular form present in the coating, which is easier to remove under wear. But in case of modified powder coatings carbide particles (cellular and needle shape particles) are present in the coating and so less rate of material removal under wear.
- Heat treatment improved normal hardness and Vickers hardness in unmodified Ni-Cr powder (1004EN) coatings and wear resistance improved after heat treatment. But in-case of modified powder coatings heat treatment reduced hardness and hence reduced wear resistance than unmodified powder coatings.
- Addition of Chromium carbide improved hardness in both heat treated and non heat treated coatings. Addition of CrC in 10-20% improved wear resistance significantly.
- Among all types of 1004EN coatings, coatings with 20% CrC in non heat treated condition showed best wear resistance.

Surfacing powder of Co and Ni based CrC (EWAC 1006EE) is used to develop coatings on mild steel substrate. And modified powder coatings are also developed by adding WC to 1006EE powder. Modification of powder is done by adding 10-20% WC. Modified 1006EE-10% powder contains 10% of added WC to 1006EE powder and similarly modified 1006EE-20% powder contains 20% of added WC to 1006EE powder.

5.1. UNMODIFIED POWDER COATINGS

Surfacing powder of type 1006EE is used to develop unmodified powder coatings. Mechanical and metallurgical characterization of Cobalt & Nickel based Chromium carbide powder (1006EE) coatings was done with the help of microstructure study, microhardness measurement, normal hardness measurement, EPMA, SEM analysis, and wear rate measurement to understand the wear behavior.

5.1.1. Microstructure

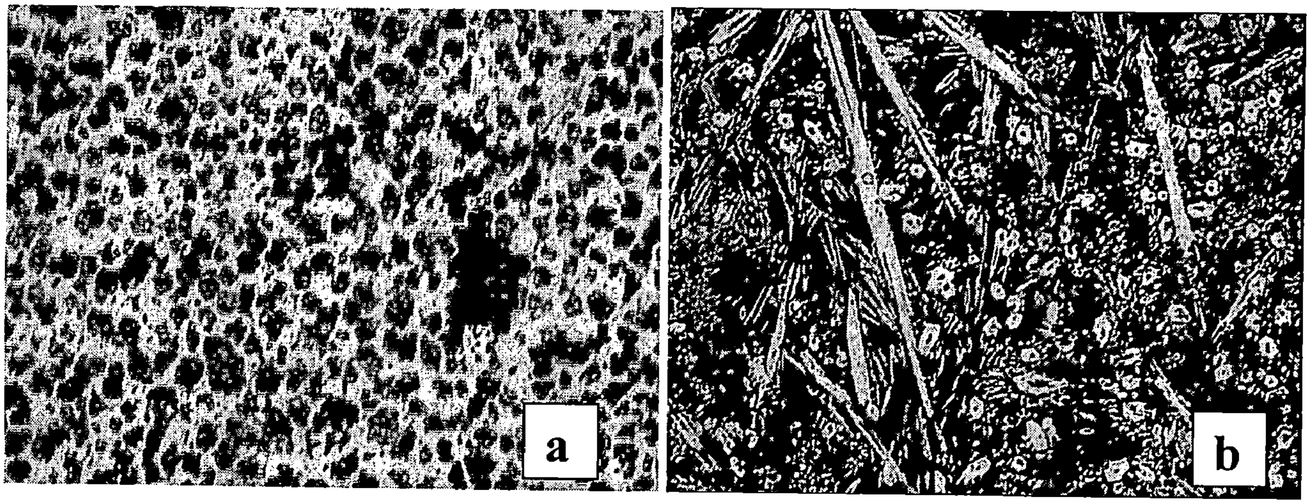


Fig. 5.1. Optical micro photographs of unmodified powder coatings.
a) Non heat treated coating (500X) b) Heat treated coating (320X)

Microstructures of the non heat treated coating (a) and heat treated coating (b) are shown in the Fig. 5.1, taken at 320X magnification. The microstructure in heat treated condition shows cuboid and needle shaped grains in the eutectic matrix which are formed due to the presence of carbides particles in surfacing powder used for the coating. Carbide particles are uniformly distributed in total eutectic matrix non heat treated condition (Fig. 5.1 a). After heat treatment carbide particles reached together and formed cuboid and needle shaped grains.

5.1.2. EPMA

EPMA analysis gives the weight percentage of the different elements present in the coating. EPMA results of unmodified powder coating (1006EE) after heat treatment are shown in Fig. 5.2&5.3. The major elements present in the coating matrix are Cobalt, Nickel, and Chromium, and a little amount of Tungsten. And these are more or less uniform through out the coating. The weight percentage of different elements present across the coating are 33-35%Ni, 30-45%Co, 12-16%Cr, 3-5%W and Silicon is less than 1% (Fig. 5.2). The major elements present in the carbide particle are Cr, Co, Ni, and W (Fig. 5.3). The weight percentage of different elements present in the carbide particle is as follows 66-67%Cr, 17-18%Co, 4%Ni, and 3%W (Fig. 5.3).

Across the entire coating the weight percentage of different elements are more or less uniform. But in the report at a point of 300μm from the interface the weight percentage of Chromium was increased and the remaining elements like Ni, Co, and Si was decreased. This is probably due to falling of testing point on a carbide particle. This will be clearer if we observe the EPMA result taken across the carbide particle (Fig. 5.3), looks as white particle in microstructure (Fig. 5.1b). The approximate percentage of different elements present at this carbide particle is 66%Cr, 18%Ni, 4%Co, and 3% W.

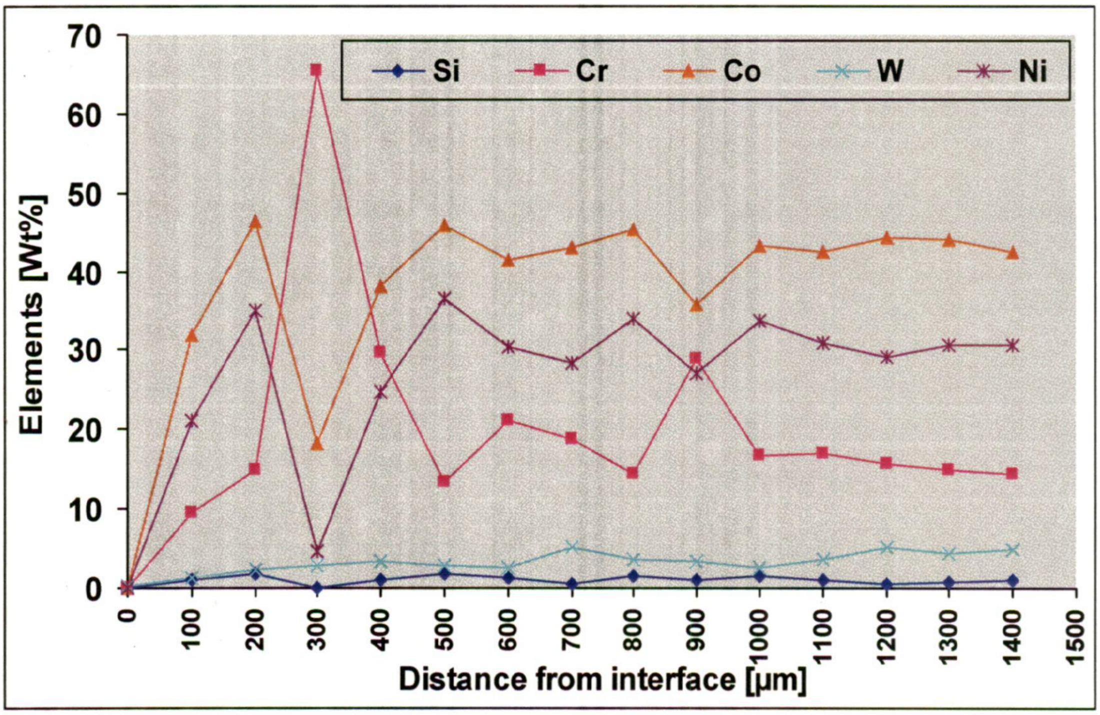


Fig.5.2. EPMA across unmodified powder (1006EE) coating from interface.

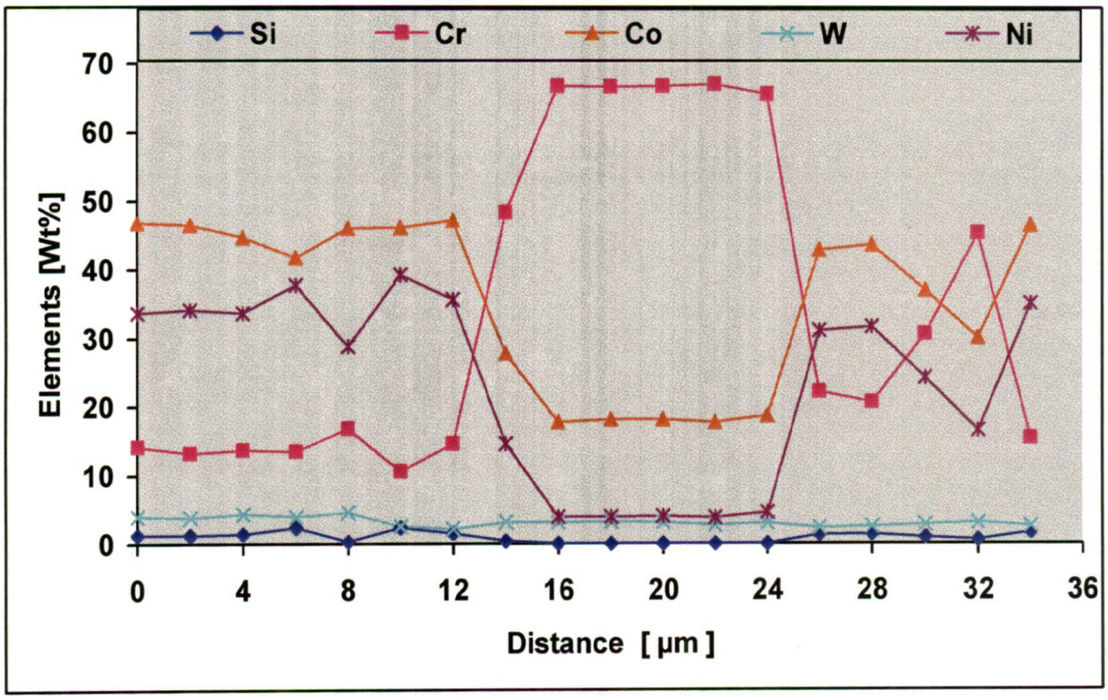


Fig.5.3. EPMA of unmodified powder (1006EE) coating across the carbide particle.

5.1.3. Hardness

Normal Vickers hardness was measured on the coating surface for both heat treated and non heat treated samples. The hardness is more after heat treatment (667VHN) than that of non heat treated condition (595VHN).

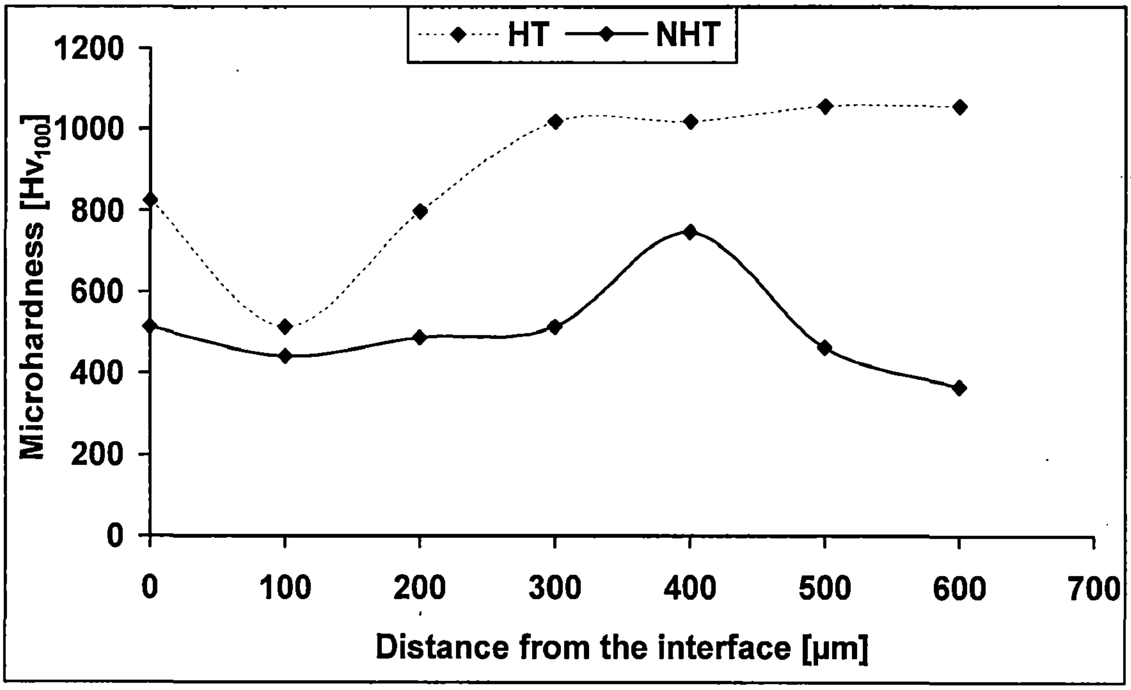


Fig. 5.4.Microhardness Vs distance from interface in unmodified powder coating.

Microhardness of unmodified powder (1006EE) coating as a function of distance from interface is shown in Fig. 5.4. The average microhardness of the heat treated sample (900Hv) is more than the non heat treated sample (500Hv). In case of non heat treated sample the microhardness is maximum (750Hv) at 400μm from the interface. But at the surface it is up to 330Hv only. This may be due to internal stresses developed in the coating. After heat treatment the microhardness is more uniform (1000Hv) from the top surface to the center of the coating. So heat treatment improved the microhardness.

5.1.4. Wear behavior

Wear rates of unmodified powder (1006EE) coatings in heat treated and non heat treated conditions, and against different abrasive surfaces is shown in Fig. 5.5. Heat treatment reduced the wear rate against both 120&600grit abrasive surfaces, at all applied loads. Reduction in wear rate is due to increase in microhardness of coating after heat treatment. More the micro hardness is better the wear resistance. Wear rate is also influenced by applied load, higher the applied load is higher the wear rate. Wear rate is reduced by 3times in case of heat treated sample against 600grit, under 20N applied load, compared to non heat treated sample (Fig. 5.5).

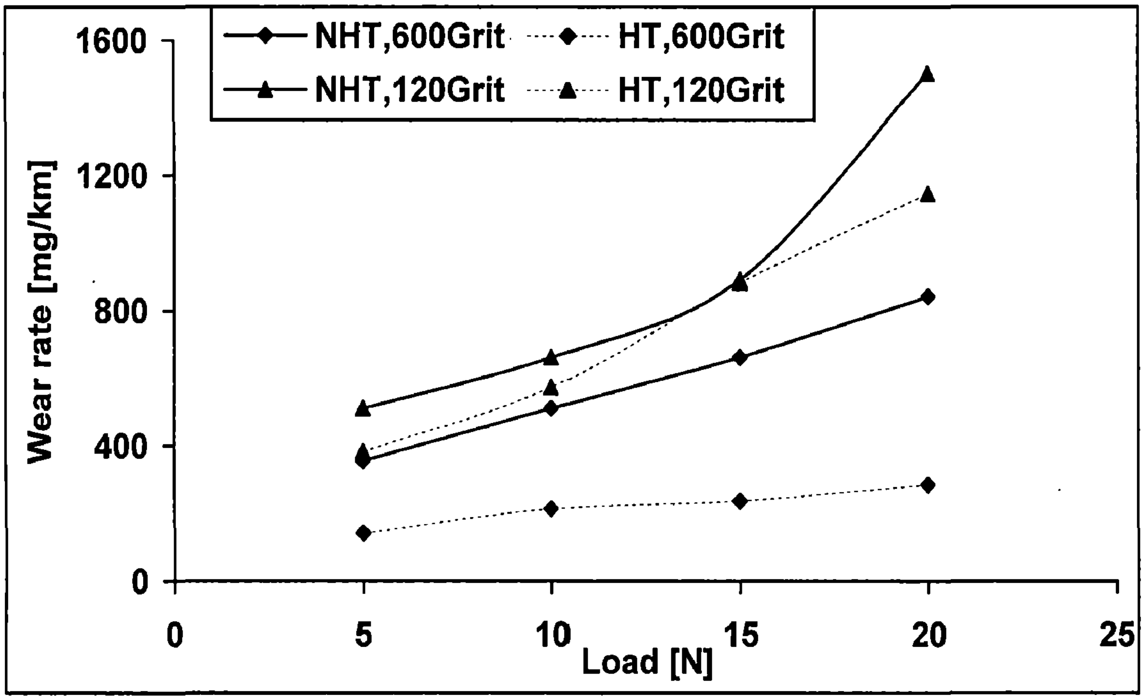


Fig.5.5. Wear rate Vs applied load in unmodified powder (1006EE) coating.

5.1.5. SEM Analysis:

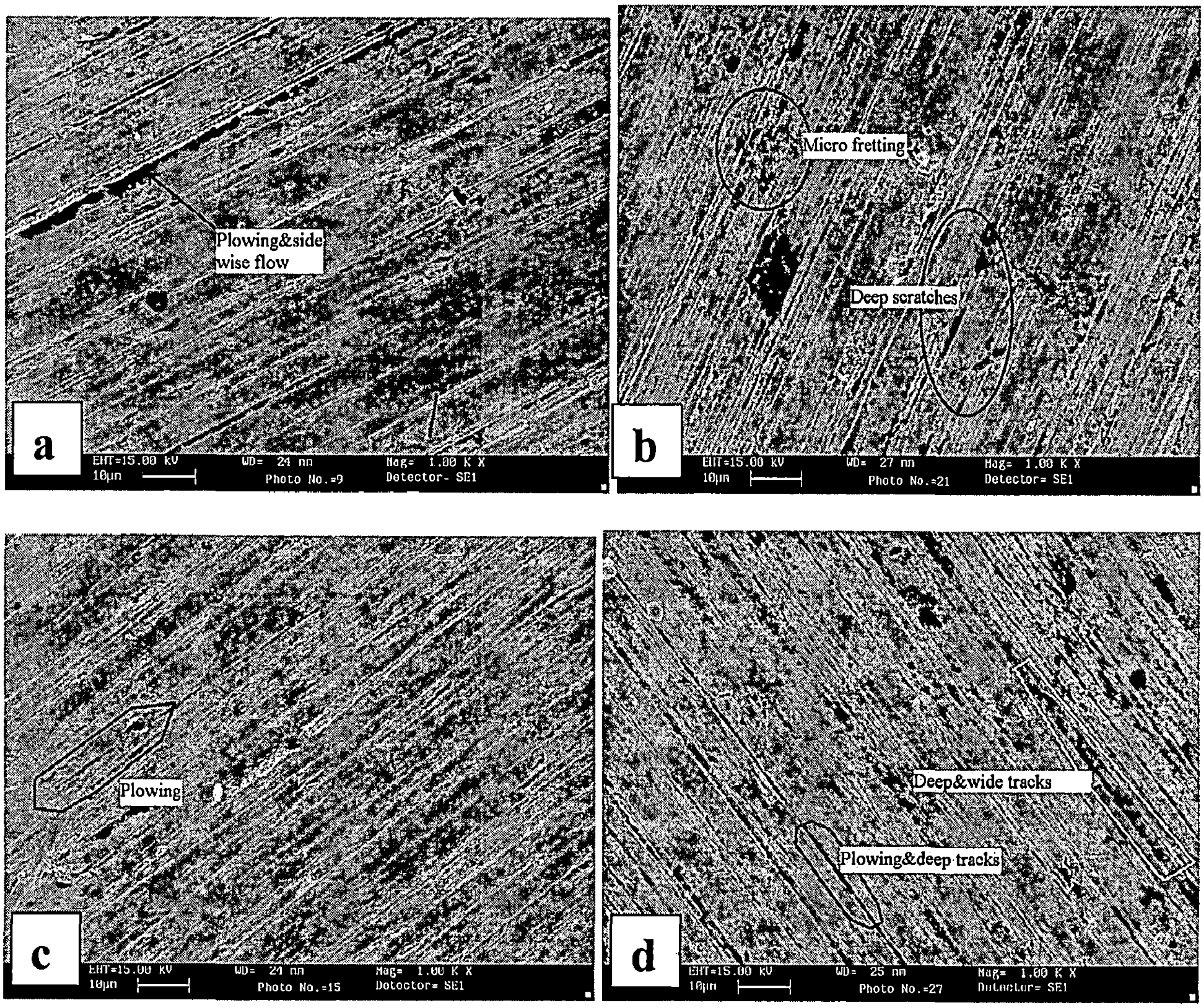


Fig. 5.6. SEM photographs of the worn out samples of unmodified powder coating samples against 600grit abrasive medium. a) Heat treated-5N load; b) Non heat treated-5N load; c) Heat treated-20N load; d) Non heat treated-20N load;

SEM images of the worn out samples in heat treated and non heat treated condition against 600grit abrasive surfaces and at different load conditions are shown in Fig. 5.6. We can observe less wear tracks in heat treated coatings (Fig. 5.6 a) than non heat treated coatings (Fig. 5.6 b) under low loads. Also we can see deeper and more wear tracks in non heat treated coating (Fig. 5.6 d) than in heat treated coating (Fig. 5.6 c) under higher loads (20N). Plowing taken place in heat treated samples (Fig. 5.6 a&c), which is a mild form of wear and deeper and wide scratches were formed in non heat treated coatings (Fig. 5.6 b&d). These SEM images are supporting the wear results (Fig. 5.5).

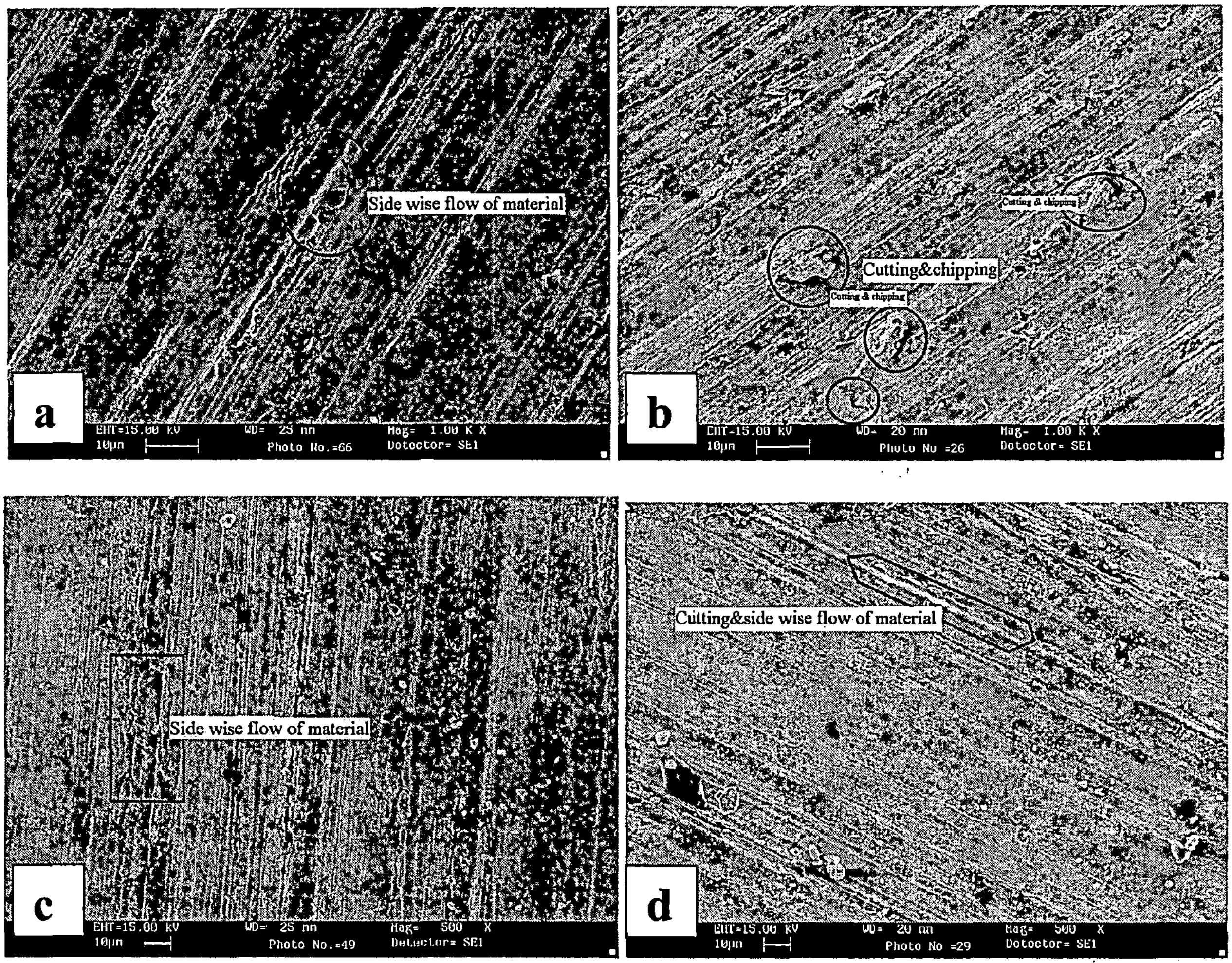


Fig. 5.7. SEM photographs of the worn out samples of unmodified powder coating samples against 120grit abrasive medium. a) Heat treated-5N load; b) Non heat treated-5N load; c) Heat treated-20N load; d) Non heat treated-20N load;

The samples tested against 120grit abrasive surfaces were also studied under SEM, and were shown in Fig. 5.7. The number of wear tracks formed in non heat treated coating (Fig. 5.7b) is more and deeper than in heat treated coating (Fig. 5.7 a) under lower loads (5N). Cutting/chipping mechanism is predominant in non heat treated coatings where as side wise flow of material is in heat treated coatings under low loads (5N). That means that wear rate in non heat treated coating (Fig. 5.7 b) is more than heat treated coating (Fig. 5.7a). Also at higher loads (20N) we can observe deeper wear tracks in non heat treated coating (Fig. 5.7 d) than heat treated coating (Fig. 5.7 c). So in all of the above 4 cases the SEM photos are supporting the wear results.

5.2. MODIFIED POWDER COATINGS

The powder of type 1006EE (Cobalt and Nickel eutectic mixture) was modified by adding Tungsten Carbide. Similar to the unmodified powder coatings, mechanical and metallurgical characterization was done by measuring microhardness, normal hardness, micro structure study, EPMA, SEM analysis.

5.2.1. Microstructure

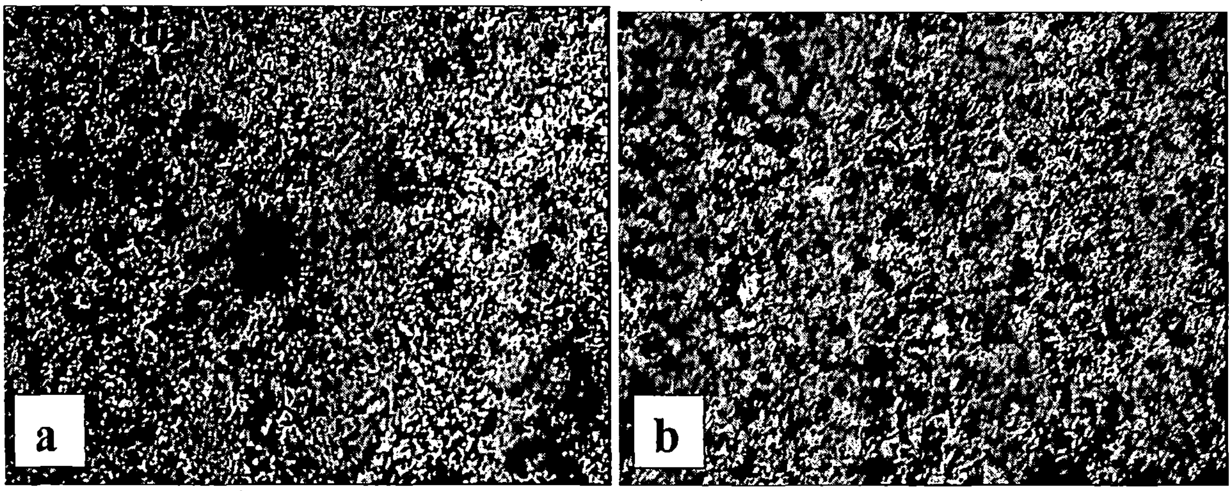


Fig. 5.8.Optical microphotographs of modified powder (1006EE-10%WC) coatings (320X).
a) Heat treated coating b) Non heat treated coating

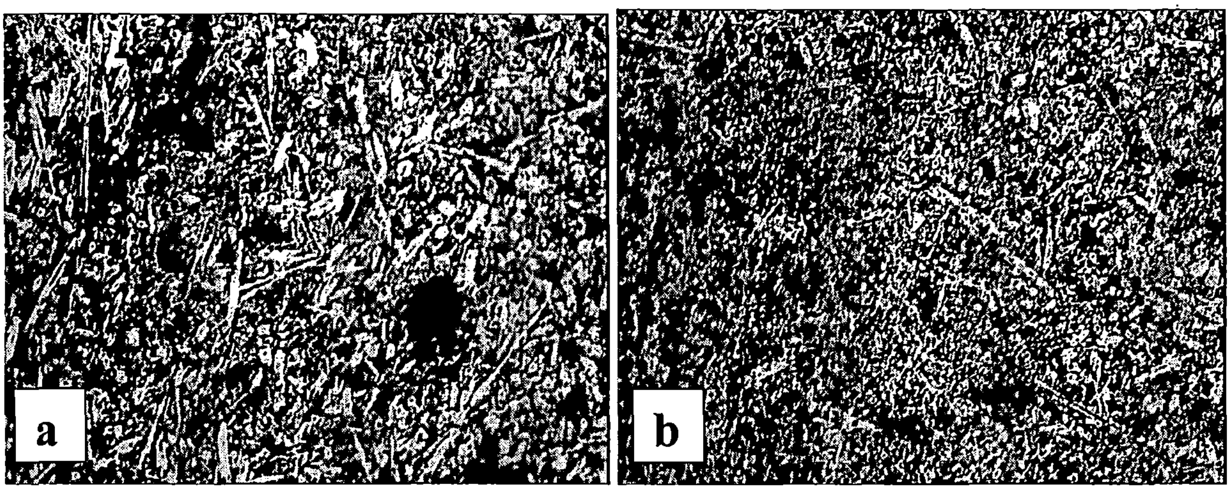


Fig. 5.9.Optical microphotographs of modified powder (1006EE-20%WC) coatings(320X).
a) Heat treated coating b) Non heat treated coating

Microstructures of the modified powder coatings, in heat treated condition and non heat treated condition, are shown in Fig. 5.8&5.9. The carbide particles of needle shape can be seen in (Fig. 5.9) the matrix of eutectic composed of Co, Ni, Cr, and W. The size of carbide particles increased after heat treatment.

5.2.2. EPMA

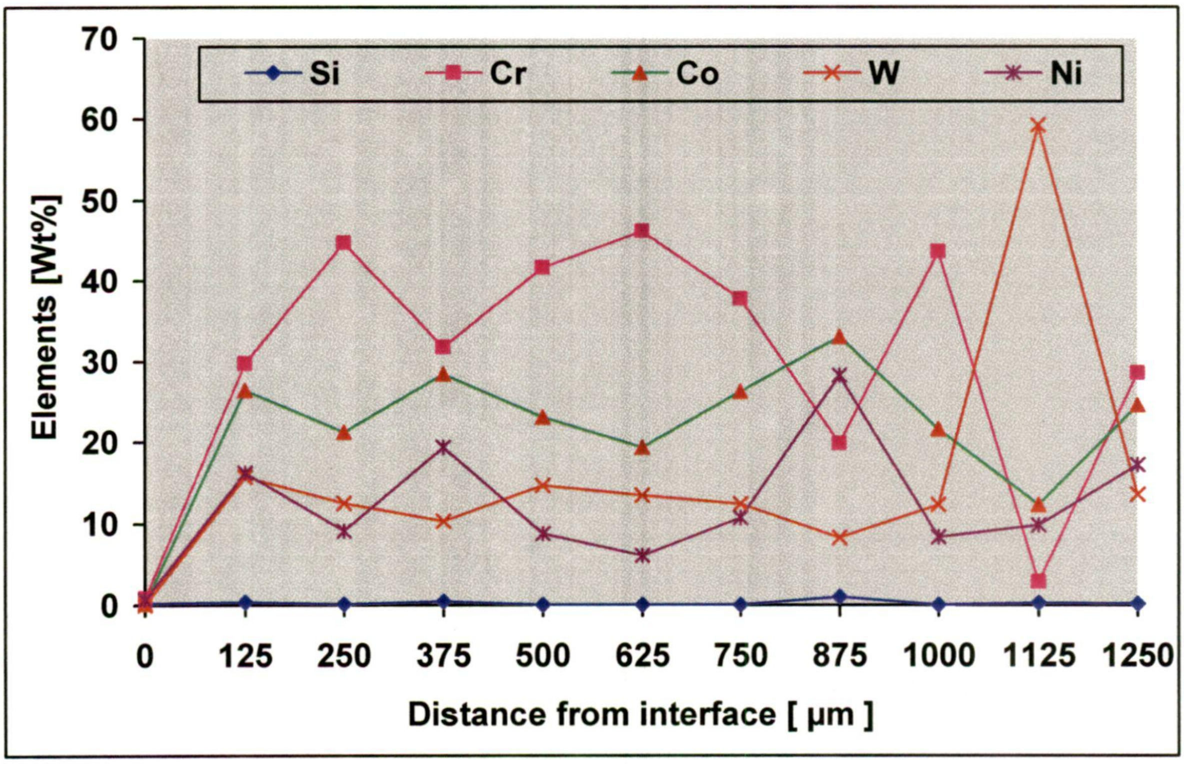


Fig. 5.10. EPMA across modified powder (1006EE-20%WC) coating from interface

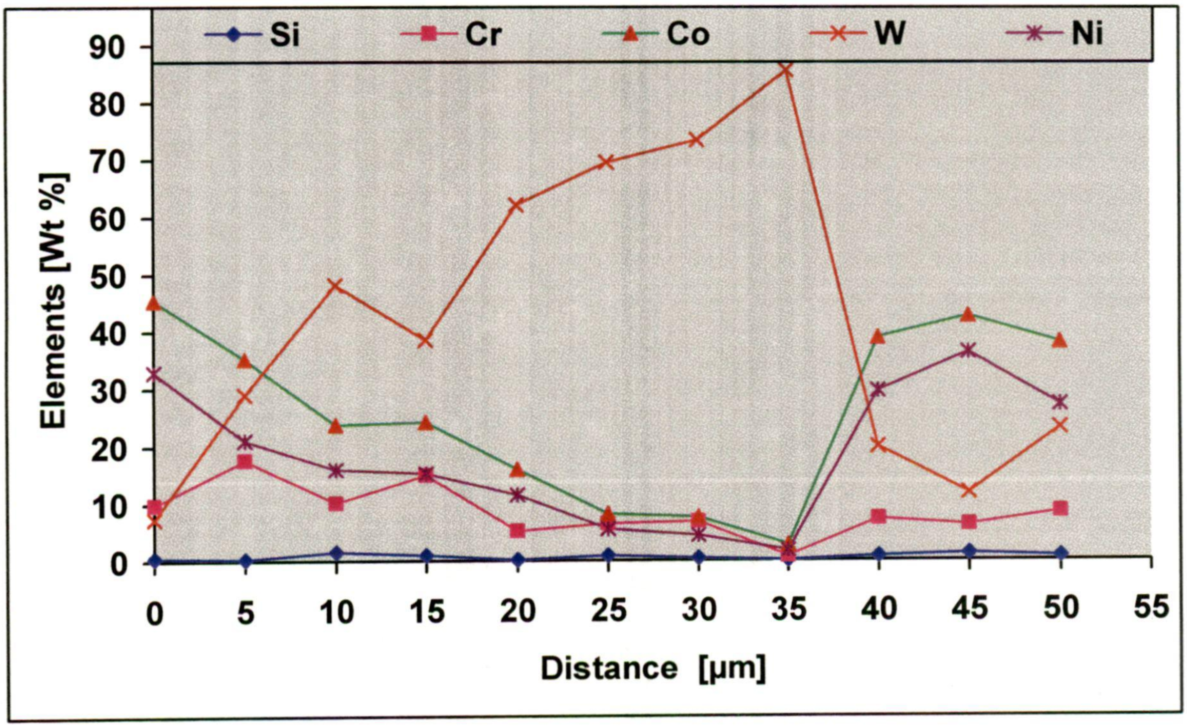


Fig.5.11. EPMA of the modified powder (1006EE-20%WC) coating across the carbide particle.

EPMA has been done for the modified powder (1006EE-20%WC) coating after heat treatment. EPMA of the sample, across the coating (Fig. 5.10) from interface and across the carbide particle (Fig. 5.11) has been done separately. Across the coating (Fig. 5.10) the average percentage of different elements was in a range of Ni-10%, W-15%, Co-20-25%, and Cr-30-45%. Analysis of carbide particle is shown in Fig. 5.11. Across the carbide particle the weight percentage of the Tungsten is high with percentage of other elements is very less. For example in Fig. 5.11, at a distance of 35μm the weight percentages of different elements are as follows: Nickel 1.7%, Tungsten 85.7%, Cobalt 2.8%, and Chromium 0.7% ; due to limitation of EPMA analyzer carbon count is not analyzed.

5.2.3. Hardness

Normal Vickers hardness of modified powder coatings was measured. In case of 1006EE-20%WC coating, Vickers hardness was 744VHN and that of heat treated coating was 663VHN. Hardness was reduced after heat treatment. But in case of 10% WC coating hardness was increased after heat treatment. Before heat treatment hardness is 346VHN and after heat treatment 550VHN.

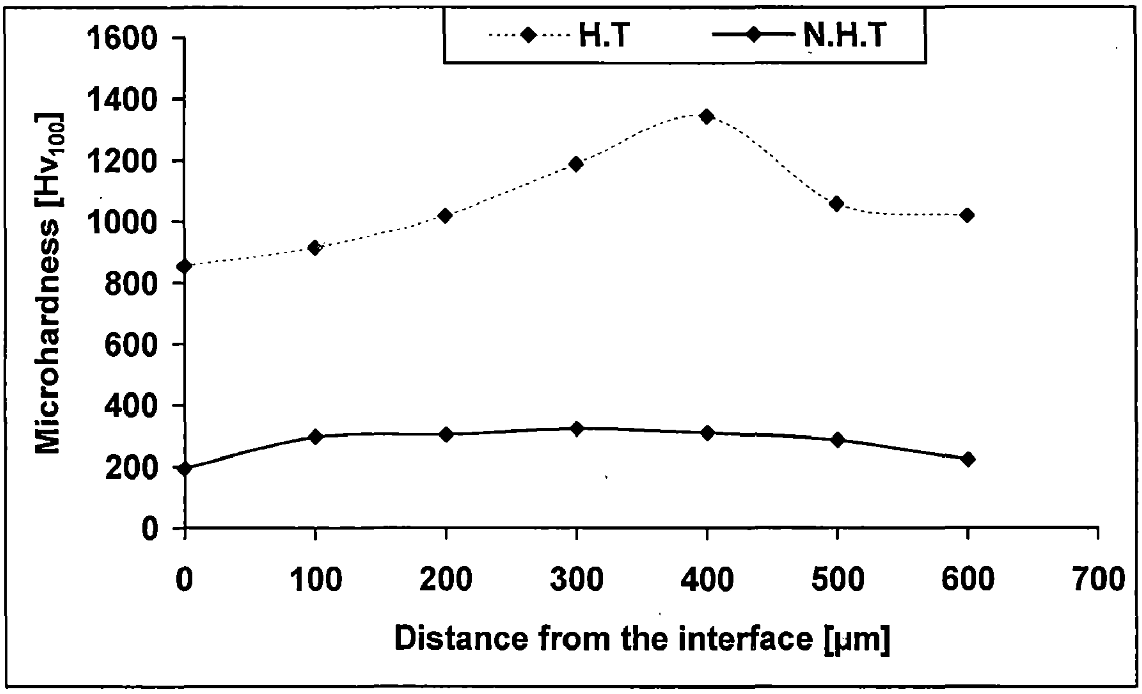


Fig. 5.12. Microhardness Vs distance from interface in modified coating (1006EE-10%WC).

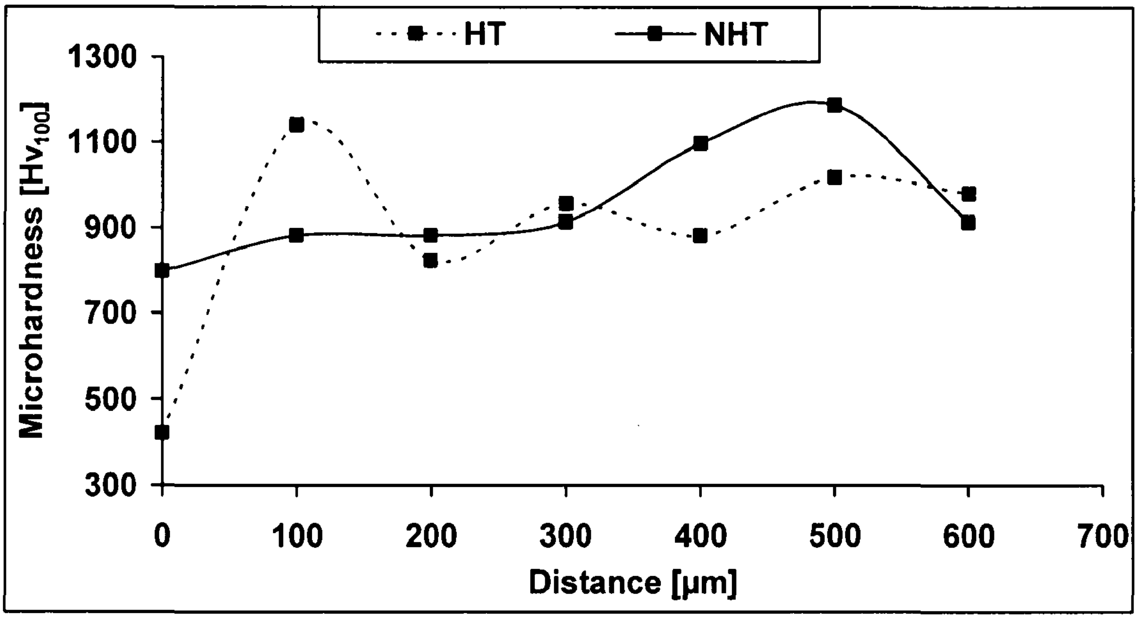


Fig. 5.13. Microhardness Vs distance from interface in modified coating (1006EE- 20%WC).

Addition of Tungsten carbide to 1006EE powder improved the microhardness both in heat treated and non heat treated coatings. Microhardness Vs distance from interface of base and coating was shown in Fig. 5.12&5.13. But the addition of the Tungsten carbide up to 10% to 1006EE powder reduced the microhardness than that of unmodified powder coating, in non heat treated condition. But after heat treatment the micro-hardness was improved and a little bit higher than the pure heat treated coating. The addition of Tungsten carbide up to 20%, improved microhardness in both heat treated and non heat treated conditions than the unmodified powder coating value. Heat treatment does not improved the microhardness in modified powder (1006EE-20%WC) coating and the microhardness is more or less same at the surface of the coating. Modified powder coatings showed higher microhardness (arround1000Hv) among the all type of modified and unmodified coatings.

5.2.4. Wear behavior

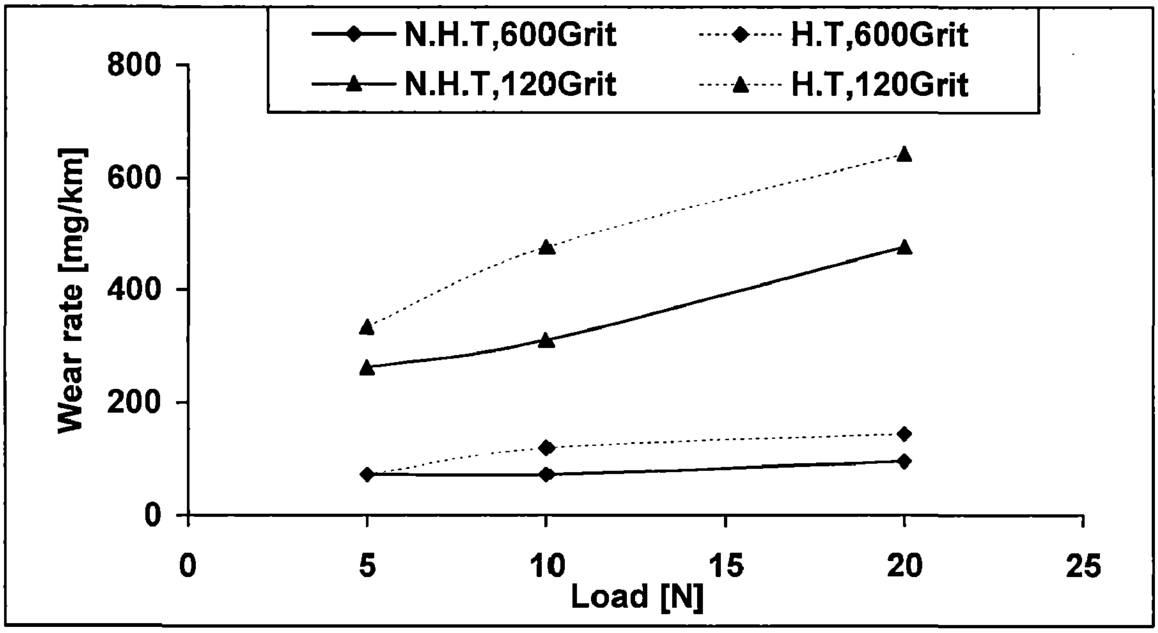


Fig. 5.14. Wear rate Vs applied load in modified powder (1006EE-10%WC) coating.

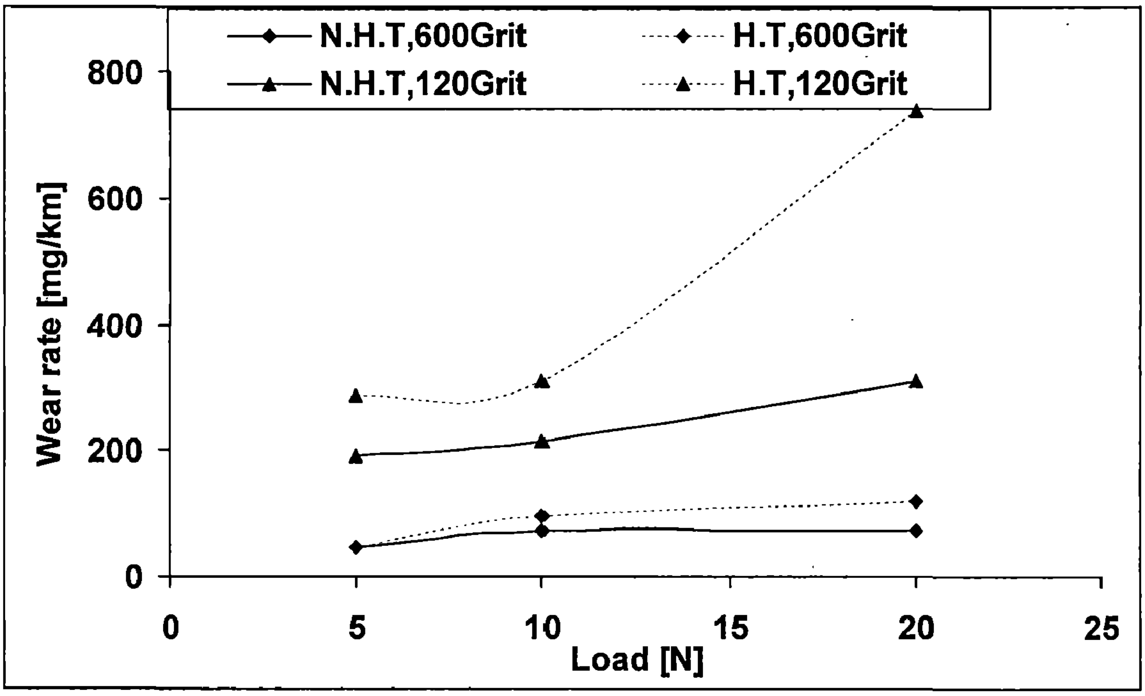


Fig. 5.15. Wear rate Vs applied load in modified powder (1006EE-20%WC) coating.

Wear rate Vs applied load in modified powder coatings are shown in Fig. 5.14&5.15. In both the modified powder coatings heat treatment showed adverse effect on wear rate, under all identical conditions. In case of modified powder coatings, non heat treated samples showed good wear resistance against all identical conditions than heat treated samples. In case of modified powder (1006EE-20%) coating, microhardness and normal Vickers hardness were reduced after heat treatment. This may be the reason for increased wear rates in modified coatings after heat treatment.

But the addition of Tungsten carbide to 1006EE powder has improved wear resistance of the modified powder coatings both in heat treated and non heat treated conditions. In non heat treated condition, there is much improvement in the wear resistance after the addition of the Tungsten carbide. Wear rates of non heat treated coating, against 120grit and 600grit abrasive surfaces were given in Fig. 5.16&5.17. In case of 600grit abrasive surface the wear rates in modified powder coatings are very less (9-12times) than unmodified powder coating. And in case of 120grit abrasive surface, wear rates are 3-5 time less than unmodified powder coatings. Addition of Tungsten carbide to Co-Ni-Cr powder improved wear resistance of coatings.

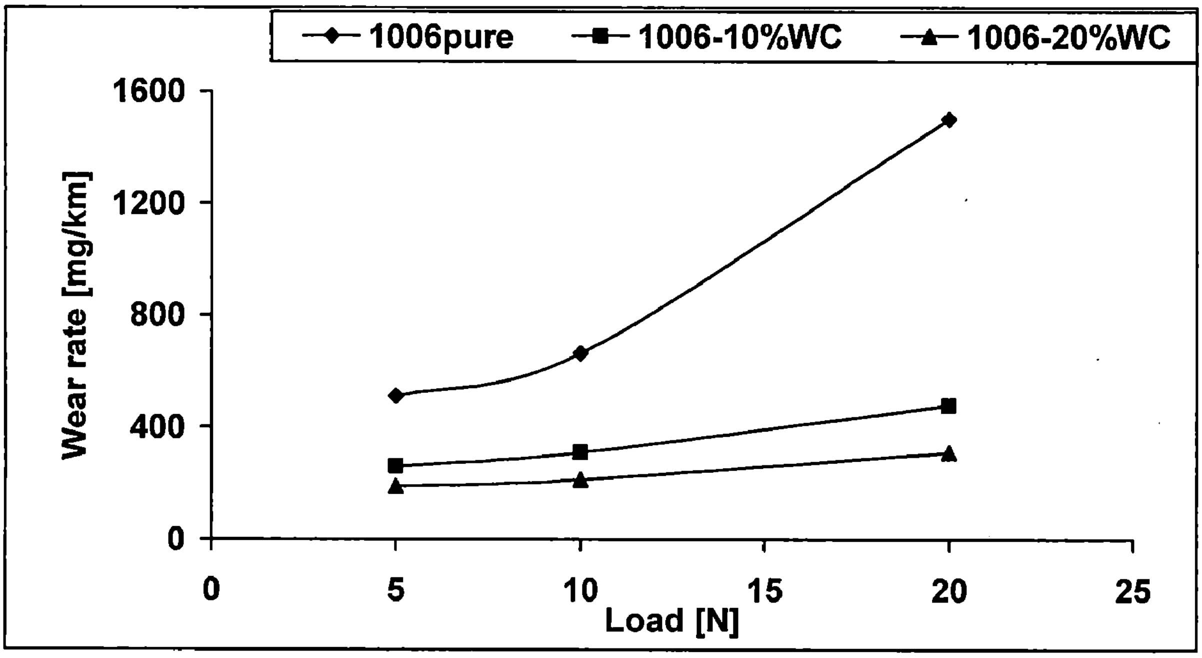


Fig. 5.16. Wear rate Vs applied load in non heat treated samples against 120grit abrasive.

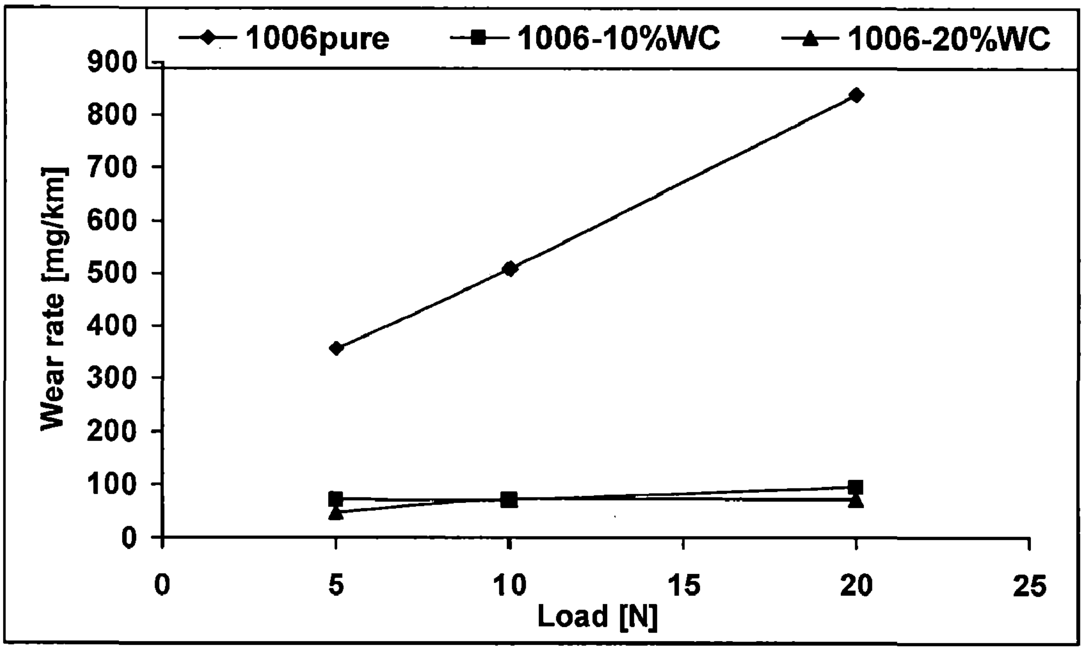


Fig. 5.17. Wear rate Vs applied load in non heat treated samples against 600grit abrasive.

5.2.5. SEM analysis

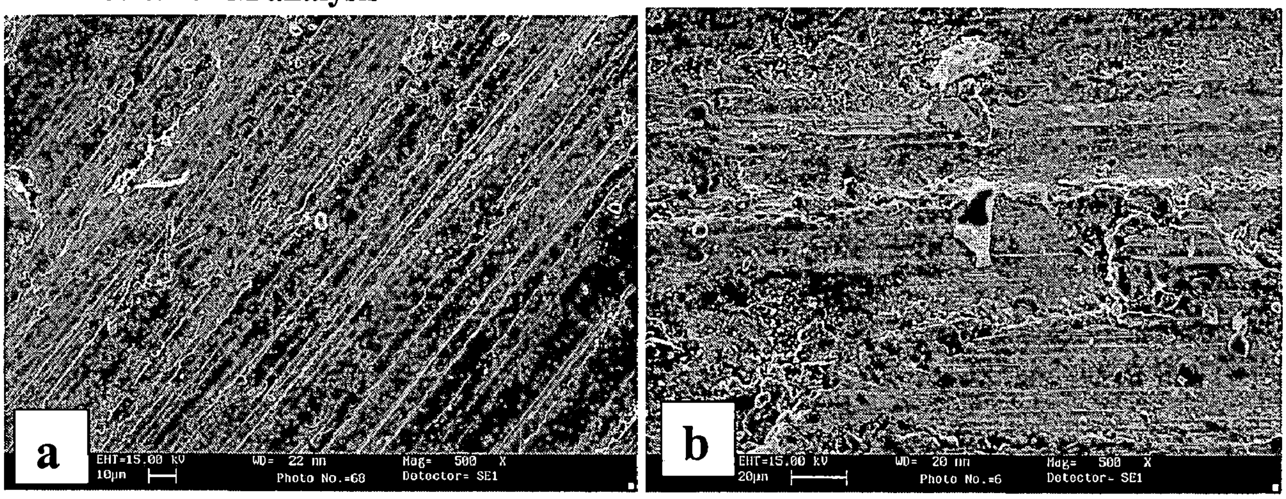


Fig. 5.18. SEM images of modified powder (1006EE-20%WC) coatings tested against 120grit abrasive surface under 5N load. a) Heat treated sample b) Non heat treated sample

SEM photos of worn out surfaces tested under 5N load and against 120grit abrasive surface of modified coatings (consists of 20%WC) were shown in Fig. 5.18. In non heat treated coating (Fig. 5.18 b) we can see that the material flows side wise and less wear tracks compared to heat treated coating (Fig. 5.18 a). So wear rates are less in non heat treated coating compared to heat treated coating (Fig. 5.15). These SEM photos are evident and proving the wear test data.

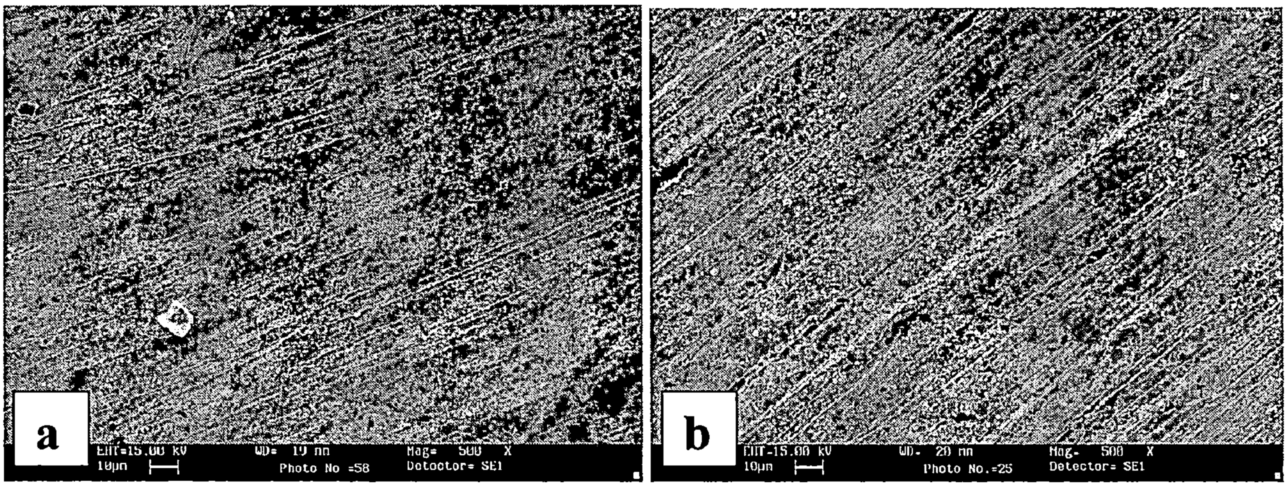


Fig. 5.19. SEM images of modified powder (1006EE-10%WC) coatings tested against 120grit abrasive surface under 5N load. a) Heat treated sample b) Non heat treated sample

SEM photos of worn out surfaces tested under 5N load and against 120grit abrasive surface of modified coatings (consists of 10%WC) were shown in Fig. 5.19. The micro-hardness and normal Vickers hardness are more incase of heat treated coating but wear rate was more in heat treated coating than non heat treated coating. Worn out surface of heat treated coating was shown in Fig. 5.19a, and of non heat treated coating was shown in Fig. B. In non heat treated coating (Fig. 5.19 b) there is side wise flow of material and we can see the debris present on the surface. But there is no side wise flow of material in heat treated coating (Fig. 5.19 a). So these SEM images are supporting the wear results (5.14).

5.3. Summary

- Heat treatment improved micro-hardness and reduced wear rates in Co& Ni based Chromium carbide coatings.
- Addition of Tungsten carbide to the surfacing powder improved microhardness.
- Addition of WC, improved wear resistance in both heat treated and non heat treated coatings.
- Among all modified coatings of 1006EE, the coating containing 20%WC showed higher wear resistance (9-12 times) than unmodified powder (1006EE) coating in non heat treated condition.
- That means modified powder (1006EE-20%WC) coating has shown best performance in non heat treated condition.

All three basic coatings and modified coatings were compared based on their wear rates under different conditions. The comparison is mainly based on their micro-hardness and wear rates under different conditions. These coating are also compared with respect to mild steel behavior under abrasive wear.

6.1. Hardness

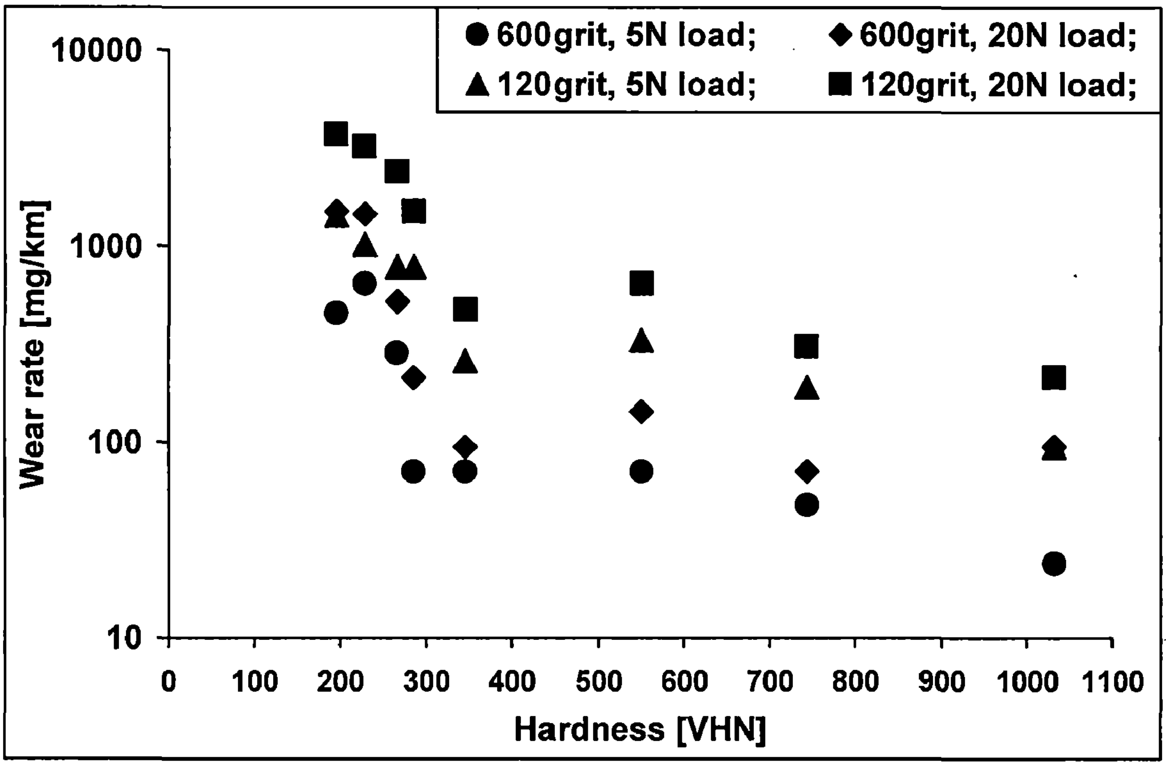


Fig. 6.1. Hardness Vs Wear rate under different conditions

The normal Vickers hardness Vs wear rate graph was shown in Fig. 6.1. Wear rate depends up on type of abrasive surface used and applied load (Fig. 6.1). Wear rate is less in 600grit abrasive surface and more in 120grit abrasive surface. As the applied load increased wear rate also increased (Fig. 6.1). At almost all points, as the hardness was increasing the wear rate was reduced. When hardness is less than a minimum amount (260VHN), wear rates are very much high. And when hardness is in between 260VHN to 300VHN, wear rates are in reducing and below than that of 300VHN hardness wear rates reduced an appreciable amount. This range of 260VHN to 300VHN is transition zone for wear rates, below whose wear rates are high and above which wear rates are reduced appreciable amount.

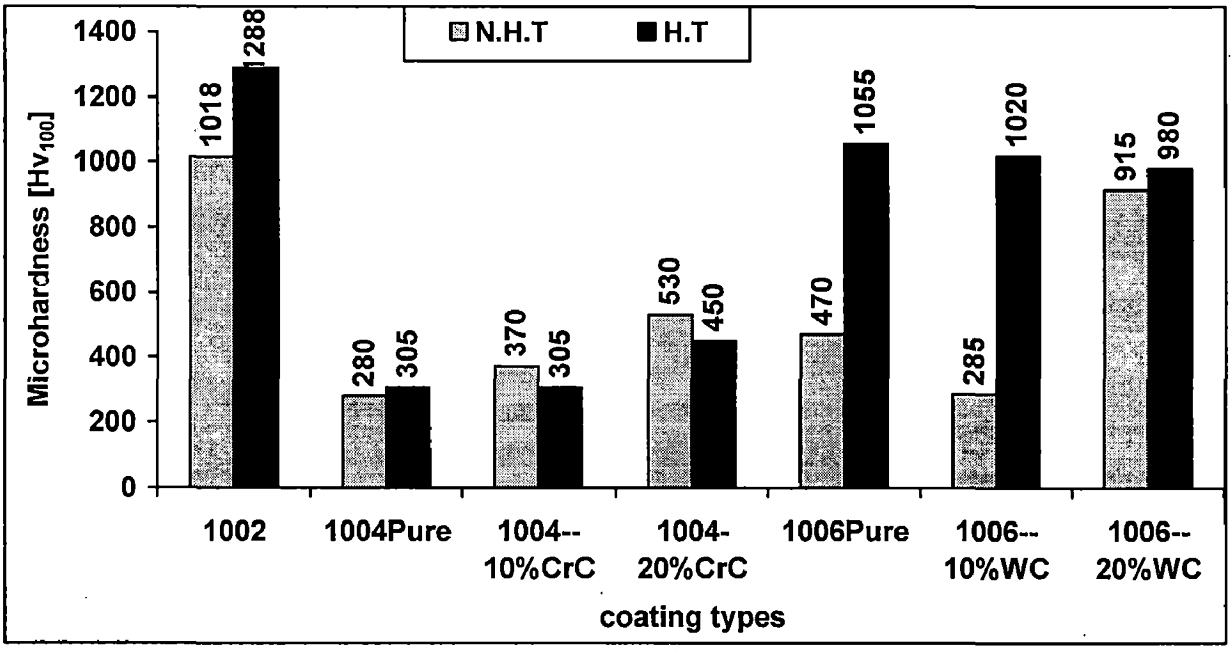


Fig. 6.2.Bar chart comparing micro hardness of different coatings.

Microhardness of different coatings in heat treated and non heat treated condition was compared by using bar chart as shown in Fig. 6.2. Microhardness improved after heat treatment in all the cases except in the modified powder coatings of 1004EN. Among all the coatings Ni based WC (1002ET) coating showed higher microhardness, in heat treated as well as in non heat treated condition. Ni eutectic (1004EN) coating and its modified coatings showed least microhardness and Co-Ni-Cr based WC (1006EE) coatings are in-between of other two coatings.

6.2. Wear behavior

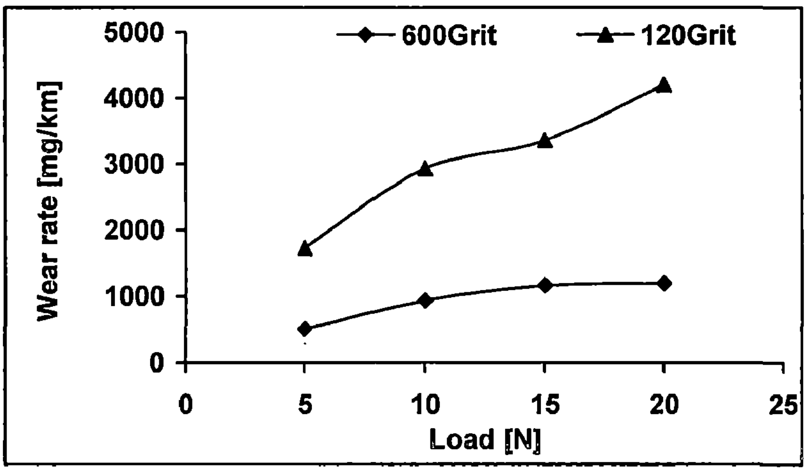


Fig. 6.3. Wear rate Vs applied load in mild steel.

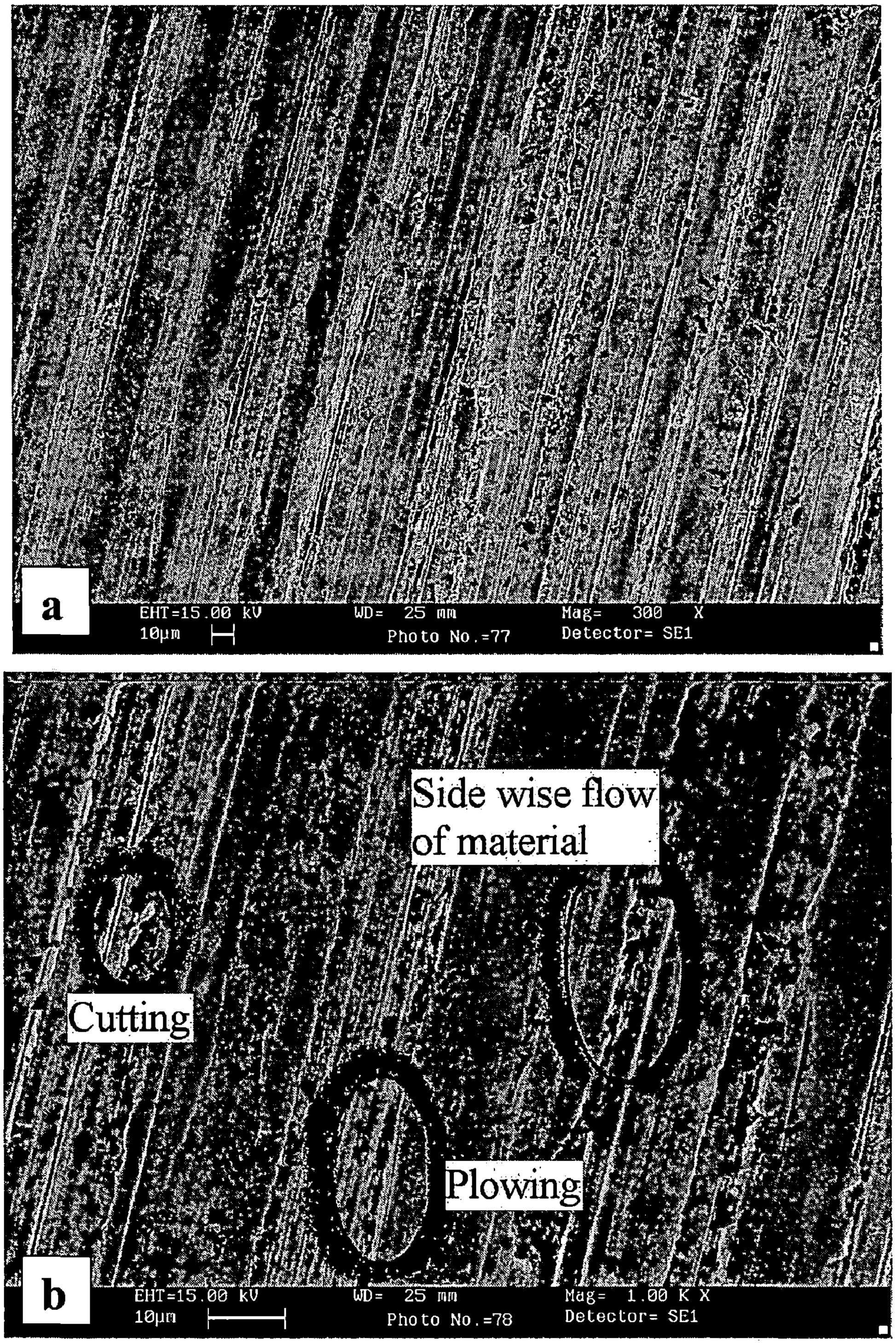


Fig. 6.4. SEM image of mild steel tested against 600grit abrasive surface under 20N load. (a) 300X, (b)1000X

SEM photo of mild steel surface tested under higher load (20N) against 600grit emery surface. Wear mechanism such as plowing, side wise flow of material, and cutting and chip formation can be seen in SEM image.

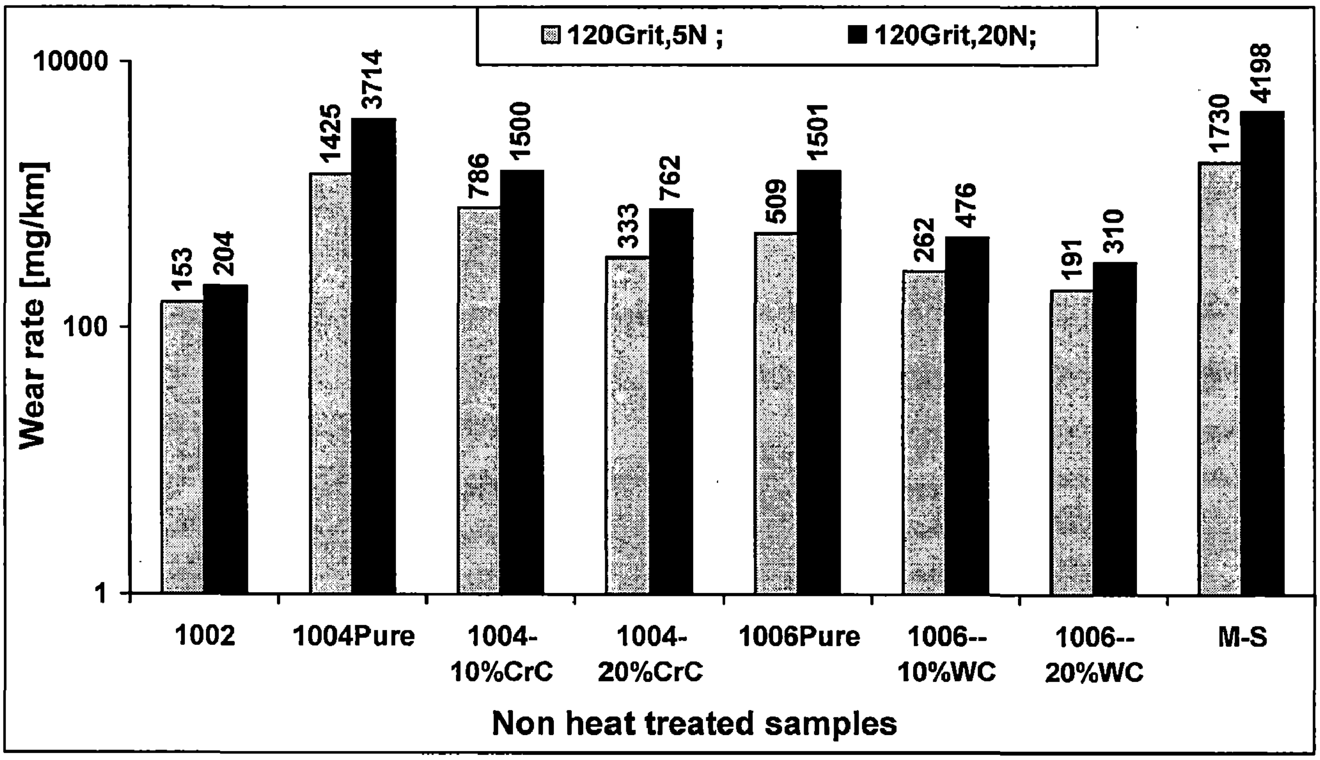


Fig. 6.5.Wear rates in different non heat treated coatings.

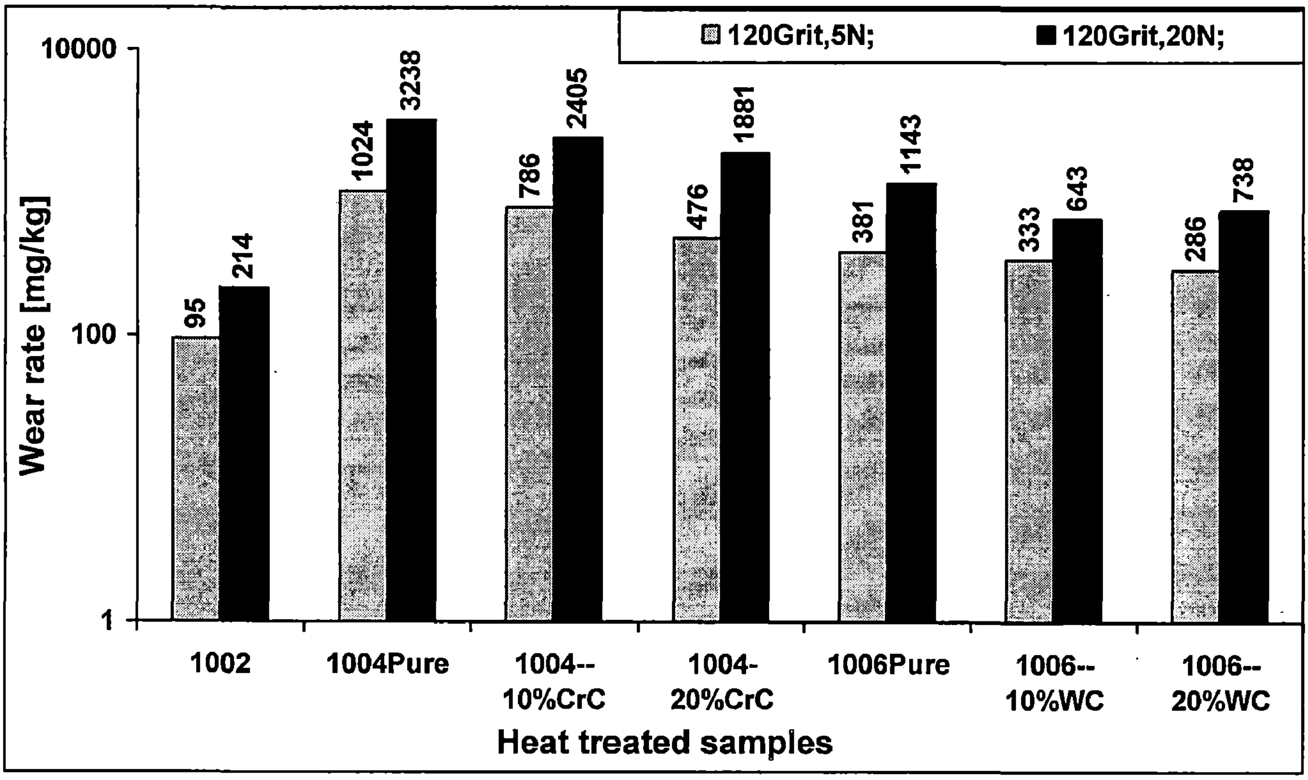


Fig.6.6.Wear rates in different heat treated coatings.

Wear rate of mild steel under different loads, and against different abrasive surfaces was shown in Fig. 6.3. Wear rate is more against 120grit abrasive medium under20N load and less in 600grit abrasive and 5N load (Fig. 6.3). Wear rate of all types of coating was compared with mild steel and showed as bar chart (Fig. 6.4& 6.5). Among all coatings and in all conditions, Ni based WC (1002ET) coatings showed best wear performance. Both heat treated and non heat treated coatings showed lower wear rates against 600grit abrasive medium at 5&20N applied load. Immediate next to this coating the modified powder coatings of Co-Ni-Cr based coatings (1006EE) showed better wear resistance.

With respect to mild steel, these coatings showed high wear resistance. Wear rate of mild steel under 600grit, 5N load condition is 509mg/km and at same conditions Ni based WC (1002ET) coating has a wear rate of 24mg/km. i.e. Ni based WC coating is approximately 21 times resistant than mild steel under wear. Similarly under 120grit & 20N load condition mild steel has a wear rate of 4198mg/km and Ni based WC (1002ET) coating has a wear rate of 214mg/km, i.e. Ni based WC coating is 19times resistant than mild steel under wear. Similarly under 600grit and 5N load condition Co-Ni-Cr (1006EE) coating is 3.6times more resistant, and the modified powder (1006EE-20%CrC) coating is 10time more resistant than the mild steel under wear. Wear ratios were calculated with respect to mild steel in non heat treated and heat treated condition and shown in Table: 6.1.

Wear ratio = (wear rate of mild steel) / (wear rate of coating)

	1002ET	1004EN	1004EN +10%CrC	1004EN +-20%CrC	1006EE	1006EE +10%WC	1006EE +20%WC
N.H.T	10 times	< 1 times	7.2 times	7.2 times	1.5 times	7.2 times	10.5 times
H.T	21 times	< 1 times	1.8 times	5.4 times	3.6 times	7.2 times	10.6 times

A) Against 600grit and 5N load

	1002ET	1004EN	1004EN +10%CrC	1004EN +-20%CrC	1006EE	1006EE +10%WC	1006EE +20%WC
N.H.T	20.6 times	1.1 times	2.8 times	5.5 times	2.8 times	8.8 times	13.5 times
H.T	19.6 times	1.3 times	1.8 times	2.2 times	3.7 times	6.5 times	5.7 times

B) Against 120grit and 20N load

Table: 6.1. Comparison of wear rates in different coatings with mild steel.

The data regarding to wear rates of different coatings in comparison with mild steel, N load & 600grit surface and 120grit & 20N load was shown in Table: 6.1.
The increasing order of wear resistance in differ coatings after heat treatment was as

- Mild steel and 1004EN pure coating
- (1004EN-10%CrC) modified coating
- (1004EN-20%CrC) modified coating
- 1006EE pure coating
- (1006EE-10% WC) modified coating
- (1006EE-20% WC) modified coating
- 1002 ET pure coating.

In non heat treated condition the increasing order of wear rate is similar to the above, with few changes, and it is in the follows order of

- Mild steel and 1004EN pure coating
- 1006EE pure coating
- (1004EN-10%CrC) modified coating
- (1004EN-20%CrC) modified coating
- (1006EE-10% WC) modified coating
- (1006EE-20% WC) modified coating
- 1002 ET pure coating.

In non heat treated condition, the behavior of modified powder (1006EE-20% WC) coating and 1002ET pure coating was different against different abrasive surfaces. Against 600grit and under 5&20N load, the Ni based WC (1002ET) coating showed lower wear rate. And against 120grit, the modified powder (1006EE-20%WC) coating showed lower wear rate compared to all other coatings. So it is evident that the wear rate was also influenced by the counter surface (i.e. counter surface roughness value and hardness).

Wear resistance is influenced by microhardness of the coating. Higher the microhardness is higher the wear resistance. Wear resistance of all these coatings was found better compared to mild steel. Wear resistance was found maximum for Ni based WC (1002ET) coatings (i.e. 19-20times more resistant than mild steel), and minimum for Ni eutectic alloy (1004EN) coating (i.e. 1 to2 times more resistant than mild steel), where Co & Ni based CrC (1006EE) coating showed 3 to 4times better wear resistance than the mild steel.

Addition of carbide particles to the original surfacing powders improved microhardness and increased wear resistance of coatings. Addition of WC to Co-Ni based CrC (1006EE) powder improved microhardness and improved wear resistance. The addition of WC 20% to 1006EE improved its wear resistance 10 to 13 times than mild steel, while unmodified powder coating is 3 to 3.5 time more resistant than mild steel. Addition of CrC up to 20% to Ni based eutectic alloy (1004EN) powder improved its wear resistance 5to7 times more than mild steel, while unmodified powder (1004EN) coating is 1to2 times more resistant than mild steel.

Heat treatment increased microhardness in all the coatings, except in modified powder coatings of Ni-Cr based Eutectic alloy (1004EN). These modified powder (1004EN-CrC) coatings showed least wear resistance after heat treatment. Ni based WC (1002ET) coating showed higher microhardness after heat treatment and improved wear resistance among all other coatings. Modified powder coatings of Co &Ni based CrC coatings (1006EE) showed reduced wear resistance after heat treatment compared to non heat treated condition. But all types of unmodified powder coatings (1002ET, 1004EN, 1006EE) showed improved microhardness after heat treatment, and hence increased wear resistance after heat treatment.

Wear mechanisms are different in each type of coating. Ductility is the major mode of wear in Ni based eutectic alloy (1004EN) and its modified coatings. Plowing and deep scratching, cutting and chipping are associated with wear in 1004EN coatings. Grooving, deep scratching, micro cracking and fragmenting are the wear mechanisms associated with 1002ET coatings. Cutting, micro cracking, scratching, and plowing are the major wear mechanisms visible in 1006EE coatings.

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