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Experimental investigation on Surface characterization of Nickel coated High Speed Steel using Scanning Electron Microscopy and Energy Dispersive Spectroscopy

[1]Sk. Shahanaz Mubeena.[2] P.Anudeep
[1]Assistant professor, Department of Mechanical Engineering, Guru Nanak Institute of Technology,Hyderabad.
[2]Assistant professor, Department of Mechanical Engineering, TKR College Of Engineering & Technology in Meer pet, Hyderabad.

ABSTRACT
Scanning Electron Microscope (SEM) is one of the advent in the field of nano surface characterization tools. It is mainly aimed for morphological analysis but can also be used for elemental analysis in conjunction with Energy Dispersive Spectroscopy (EDS). Even though some experimental investigation has been carried using SEM for morphological studies, but still there is some work to be carried for morphological as well as elemental investigation in combination with EDS on coated samples like High Speed Steel (HSS) with Nickel. An HSS sample of 2cm X 1cm X 1mm is chosen and coated with Nickel in the order of 300 µm. With the variation of magnification factor, the distribution of nano particles with in the samples of the constituents has been estimated at a constant acceleration voltage of 15KV. The elemental composition and surface structure plays a major role in the mechanical behavior of the materials. Therefore, the present paper has been focused on experimental investigation on morphological and elemental analysis of Nickel coated HSS at various accelerating currents. The elements analyzed during morphological studies are Carbon, oxygen and Nickel etc. The studies of coated samples have been carried using SEM by the secondary electron imaging (SEI) or backscattered electron imaging (BEI) mode and elemental analysis by Energy Dispersive Spectroscopy (EDS).
Keywords: SEM, EDS, Coated samples, Elemental analysis.

I. Introduction

Scanning electron microscopy uses a focused high energy electron beam to image the surface of a variety of samples and collect information on morphology and elemental composition. A scanning electron microscope is a highly versatile tool and can be used to study biological specimens, geological materials, Nano particles, circuit boards, and many other sample types.Scanning electron microscopy (SEM) and x-ray microanalysis can produce magnified images and in situ chemical information from virtually any type of specimen. The two instruments generally operate in a high vacuum and a very dry environment in order to produce the high energy beam of electrons needed for imaging and analysis [1]. The use of Scanning Electron Microscopy / Energy Dispersive X-Ray Spectroscopy (SEM/EDS) in the analysis of failure related issues of printed circuit boards (PCBs), assemblies (PCAs), and electronic components (BGA, capacitors, resistors, inductors, connectors, diodes, oscillators, transformers, IC, etc.) is a well-established and accepted protocol.

II. Literature Survey

A controllable manner is required to achieve the reliability and reproducibility for producing the Ni-based components. Differential thermal analysis (DTA), energy dispersive X – ray analysis (EDXA), X-ray diffraction (XRD), scanning electron microscopy (SEM) and Transmission Electron Microscopy (TEM) have been used for micro structural characterization of slowly cooled Nickel–based hard facing alloys [6]. The presence of boride and carbide dispersions within their microstructures makes the nickel based alloys exhibit excellent resistance to abrasive wear [7].Silicon is added to increase the self fluxing properties and lower the melting point of nickel, the base metal. However, it reduces the tensile strength of the alloy [8]. the resistive transition curves and the corresponding scanning electron microscopy (SEM) pictures of YBa_2 Cu_3 $O_{7-\delta}$ (YBCO) films obtained by pulsed laser deposition (PLD) have been analyzed to better characterize the films and refine the deposition process [9].A Field Emission Scanning Electron Microscope (FESEM) operated with high emission current (50µA) was used to increase the number of BSE obtainable from imaged specimens have X-rays can be generated by any atom in the sample that is

sufficiently excited by the incoming beam [10]. The effect of Ni and NiO coatings on Fe-22Cr during oxidation at 1173K in 1% H_2O was examined with respect to scale microstructure 11]. The mechanical properties of Kenaf / Polyester and the effects of the modification on fiber were analyzed by using the SEM [12]. The micro structural studies are carried out with field emission SEM on the uncoated and coated graphite particles [13]. The alloying and phase formation in Ni-Hf samples with 0.2, 2, and 5 at.% Hf were studied by X-ray diffraction (XRD) technique and SEM [14]. SEM imaging showed an increase in grain size of manganese oxide nano particles coated on ceramic membranes with increasing number of coating layers [15]. The composition of the mild steel substrate, consisting of 0.37 wt.% C, 0.28 wt.% Si, 0.66 wt.% Mn and 98.69 wt.% Fe substrate was determined using energy dispersive X-ray analysis (EDAX) [16].

III. Experimental Investigation

3.1 Experimental set up:

The experimental set up consists of a Scanning Electron Microscope / Energy Dispersive Spectroscope for both morphological and elemental analysis. The SEM allows for visual observation of an area of interest and EDS can be used to obtain semi-quantitative elemental results about very specific locations within the area of interest.

The Scanning Electron Microscope used for morphological studies of the sample is of Hitachi SU1510 VP model that combines the high-performance electron optics of the S-3400N and S-3700N with an ultra-compact design. The width of the body of SEM is 55cm. and is equipped with a monitor and PC controls systems for displaying the microstructures. Figure-1 shows the experimental set up of SEM / EDS with monitor and displaying system. The Figure-1 shows an overview of the SEM with the three detectors viz secondary electron detector, back scattered electron detector and X-ray detector or Energy Dispersive X-ray Spectroscopy (EDS). Specifically, the SEM column and chamber can be observed in the center of the image with the secondary electron and backscatter detectors found attached to the left side of the chamber and the X-ray detector attached to the right side of the chamber.The samples of size 153 mm diameter and 60 mm height can be conveniently studied using the set up. The set up can be used for deposits and wear Debris Analysis, Particle Sizing and Characterization,, Failure Analysis, Contaminant Analysis and Metallurgical Studies The main components of SEM are an electron gun, focusing lenses, stage or specimen holder, and X-ray detectors. The electron gun has a heated tungsten filament to provide the source of electrons.

3.2 Secondary Electrons

The secondary electron detector is primarily used to observe surface structure(s) associated with the specimen. This detector converts the electrons reflected by the specimen surface into a signal that can

Figure-1: Overview of the SEM/EDS Unit

be displayed as an image on a monitor. Subsequently these images can be captured as a photograph, if desired. SEM images, as well as any "captured" photographs, are grayscale in appearance as opposed to color because the electrons being detected are actually beyond the light spectrum.

3.3 Energy Dispersive X-Ray Spectroscopy (EDS)

The term X-ray detector is a general term for the type of detector used to perform Energy Dispersive X-Ray Spectroscopy (EDS). The X-ray detector, or more specifically, the EDS technique is used to qualitatively and most of the time "semi-quantitatively" determine the elemental composition of an area of interest which was visually identified and observed using the secondary electron and backscatter detectors mentioned above.As the electron beam from the SEM itself strikes the specimen surface, the electrons within the atoms of this area of interest are elevated to an excited state. When the electrons in these atoms then return to their ground state, a characteristic x-ray is emitted. These x-rays are then gathered by the X-ray detector and converted into "useful" information. An image can, as described above, be generated but more importantly, these x-rays emitted from the specimen

give information as to the elemental composition of the area. As a result, the EDS technique can detect elements from carbon (C) to uranium (U) in quantities as low as 1.0 wt%. In combination with the SEM itself, the specific area of analysis for a given specimen of interest can be adjusted simply based on the magnification at which the specimen is being observed.

IV. Sample preparation & Methodology

4.1 Sample preparation:

The Nickel coated samples are obtained by Nickel electroplating where the deposition of Nickel takes place on HSS sample. For this purpose first, the HSS sample of size 2 cm X 1 cm X 1mm are produced from raw bulk material obtained by cold rolling using sheet metal cutting process. The samples to be coated must be clean and free of dirt, corrosion, and defects before plating can begin. Thus the HSS sample is smoothly polished using grinding. To clean and protect the part during the plating process, a combination of heat treating and masking have been used. Once the HSS sample has been prepared, it is immersed into an electrolyte solution and is used as the cathode. The nickel anode is dissolved into the electrolyte in form of nickel ions. The ions travel through the solution and deposit on the cathode.

4.2 Methodology of Image capturing

The Nickel coated HSS specimen is placed within the vacuum chamber located at the bottom of the SEM column. Initial adjustments like setting the accelerating voltage, Emission current and working distance are made before the sample is placed in the chamber. The filament current knob is turned down and the accelerating voltage is set at 15 Kv and the range of working distance is put between 12mm to1.5nm. the range Magnification of images captured is between 10x to 100x. The Emission current of electrons and the current of filament are 168 µA 85µA respectively. The Gun bias voltage is set at "0" volts & the phase current is at 50 µA.

The methodology of capturing the image in SEM begins with turning ON the chamber scope to verify if there is any other material is present or not in the specimen chamber. The computer and monitoring system are switched ON for image analysis.

The stage control dialog window is opened and the stage is then moved to the specimen exchange position The specimen is loaded in the specimen chamber by wearing clean gloves with face mask. The vacuum control panel is activated by switching over it. Then the conditions mentioned above for working distance, operation mode and detector mode are set for image capturing. Adjustments for brightness, contrast and focus to get a good image are made in magnification mode. The required image at the location of interest is shown on the Monitor after adjusting the beam current. The magnification from 10x to 100x is used to for bright, contrast and quality image. After getting the desired image on the monitor, it is captured by clicking the capture.

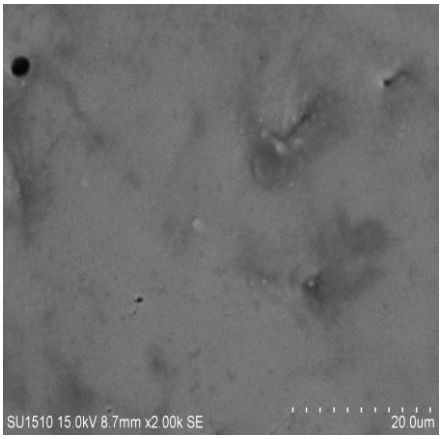
4.3 Quantitative analysis by EDS:

The EDS setup consists of four primary components: the beam source; the X-ray detector; the pulse processor; and the analyzer. The EDS measurement relies on the investigation of the sample through interactions between electromagnetic radiation and sample by analyzing the X-rays emitted. In the present work, a beam of X-rays is focused into the sample being studied to stimulate the emission of characteristic X-rays.The number and energy of the X-rays emitted from the specimen is measured by an energy-dispersive spectrometer. The elemental composition of the specimen is measured by the difference in energy between the two shells of emitted energetic levels of X-rays.

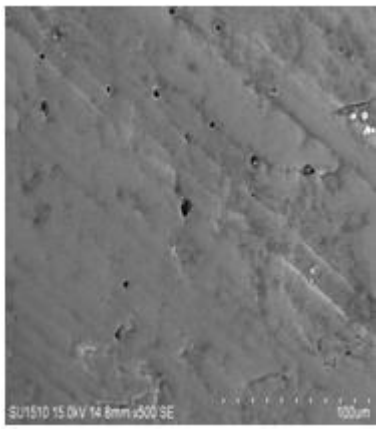
V. Results and Discussions:

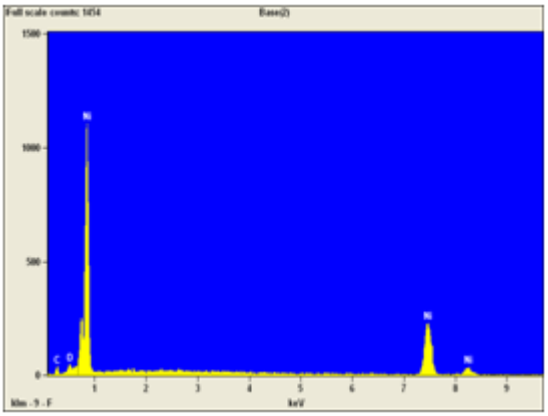
5.1 Morphological and Elemental analysis of Nickel coated samples:

The morphological and elemental analysis has been carried on High Speed Steel samples coated with Nickel and Cadmium. In order to obtain further micro structural and morphological information on the Ni and Cadmium coated samples, secondary electron images, at magnifications of 10, 20, 50, 100 and 500 were carried out. Some of the SEM secondary electron images of Nickel coated samples at 20, 50 and 100x magnification are shown in Figures-2(a), 2(b) and 2(c) respectively.



Figures-2(a): SEM images at 20x Figures-2(b): SEM images at 50x





Element Line	Net Counts	Net Counts Error	Weight %	Atom %
C	170	+/- 16	5.74	22.16
O	219	+/- 27	1.59	4.62
Ni	3945	+/- 110	92.67	73.22
Ni	8946	+/- 127	---	---
Total			100.00	100.00

Figures-2(c): SEM images at 100x

From the figures it can be observed that when HSS samples are coated with Nickel with increased magnification factor, the structure reveals, it is almost in pure lamellar and which is an indication of improved performance for wear and corrosion resistance. Further, the structures also indicate the imperfections present in the raw samples even though they are coated with Nickel that implies further densification of coating is required. Therefore, coatings of thickness of 500µm were laid over the samples in order to have high packing density and improved crystal structure.

The energy dispersive spectroscope of Nickel coated HSS samples are shown in Figure-1x. The figure shows the number of elements in the composite coating along with the net counts, % weight and % atoms. It can be observed that the maximum counts are for the Nickel in the coated sample and is around 1200 at an energy level of 1 KeV. Similarly, the counts of Carbon and Oxygen at various energy levels can also be seen in Figure-3.

Figure-3: EDS of Nickel coated samples

The display system consists of higher and lower peaks due to variation in voltage levels. Higher voltage causes higher peaks that correspond to larger energies and lower peaks for lower energies. The EDS spectrum consists of overlapping peaks (e.g., C. O and Ni) due to X-rays generated by any atom in the sample that is excited by the incoming beam.

The elemental analysis of the coated samples are clealy shown in Table-1. From the table it can be observed that the carbon element is having net counts as 170 and its total weight is 5.74% and the percentage of atoms is 22.16. Similarly the Oxygen & Nickel elements are having net counts of 219 and 3945 respectively. The weight of Oxygen and Nickel is 1.59% and 92.67 in the total weight percentage. Further, the percentage of atoms of Oxygen and Nickel in the composite coating is 4.62% and 73.22 respectively.

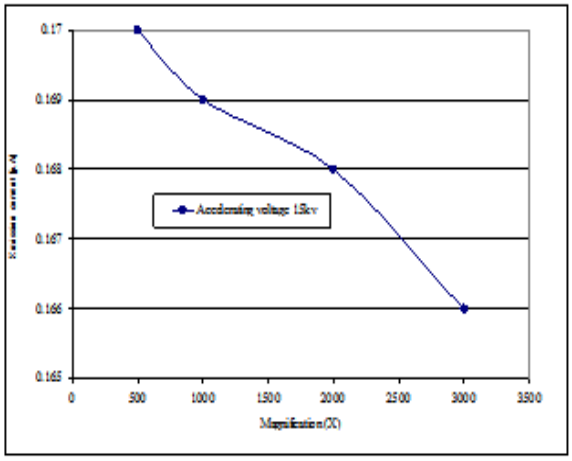
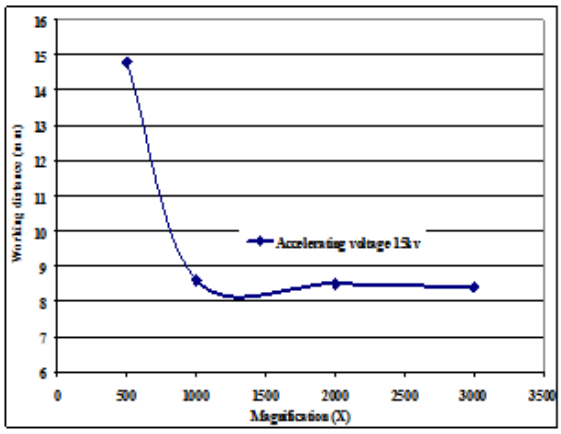


Table-1: Elemental composition



5.2 Operational Variables

The operating variables in the present investigation are (i) Emission Current vs. Magnification (ii) Working distance Vs. Magnification and (iii) Emission current vs. working distance

Magnification vs. Emission Current

Magnification in an SEM can be controlled over a range of up to 6 orders of magnitude from about 10 to 500,000 times. In an SEM, the magnification results from the ratio of the dimensions of the raster on the specimen and the raster on the display device. With fixed size of display screen, the higher magnification results due to reduction of the raster size on the specimen, and vice versa. Magnification is therefore controlled by the current supplied to the x, y scanning coils, or the voltage supplied to the x, y deflector plates. Thus, the variation of emission current with magnification is as shown in Figure-4 at a constant accelerating voltage of 15 KV. It can be observed from the Figure that the Emission current is falling with increase in Magnification and the quality of the image is also superior regarding the crystal structure as shown in Figure-2(c)

Magnification vs. working distance

The working distance is one of the operation variables that strongly affect depth of field. Further, the depth of field is related to the resolution which is a primary function of magnification. As shown in Figure-5, the working distance needed is very high when the magnification required is less. By reducing the working distance further, the resolution of the image along with magnification can be increased. It is also shown in the Figure; the magnification has been increased up to 2000x by reducing the working distance to 8.5 mm approximately. By limiting thethe working distance to 8.5 mm the magnification has been still increased to 3000x for fine crystal structure by SEM and elemental analysis with EDS.

Figure-4: Magnification vs. Emission current
Figure-5: Magnification vs. Working distance:
Emission current vs. working distance:

For getting higher magnification values, the working distance has been reduced from 15 mm to 8.5 mm. The reduction working distance has not only influences the magnification but also the emission current. As shown in Figure-6, when the working distance is reduced from ≈15mm to 8.5 mm, the emission current has been reduced to 0.169 µA. It is found that the emission current has come down to 0.166 µA at the constant working distance of ≈ 8.6mm. This trend indicates, the magnification of the image obtained is a function of not only working distance but also the emission current

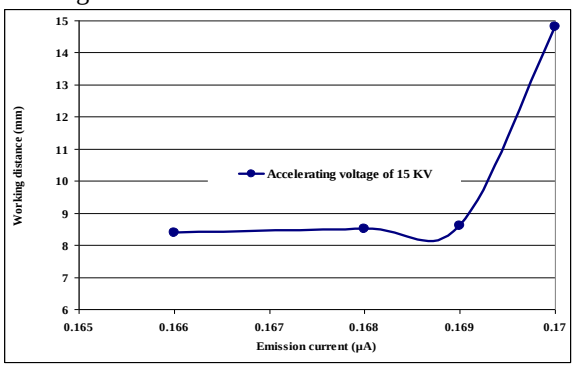


Figure-6: Emission current vs. Working Distance

VI. Conclusions:

The characterization of materials has revolutionized the use of sophisticated instruments like SEM, Atomic Force Microscope and other Energy particle based techniques viz EDS. In the present paper, an experimental investigation has been carried on structural characterization and elemental analysis of Nickel coated HSS samples due to their potential applications in the area of wear resistance, corrosion resistance, temperature resistance and other petro-chemical and metal

working industry. The coated surfaces play a major role in surface modification by changing the tribological properties due to the reduction particle size. When the particle is reduced to nano scale the coated samples behave superiorly, e.g. the mechanical and optical properties are very much enhanced by micrometer and nano meter scale coatings. In the present investigation, the Nickel coated HSS samples has been characterized for their crystal structure with the operating variables like accelerating voltage, emission current and magnification. Further, the elemental composition and % of the atoms with their net count has been analysed by EDS. The results obeys all the basic laws that can be applicable to electro-chemistry of nano scale structures.

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