# Enhancement of Tribological properties of Mild Steel using ZrO2 Nano Particles

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*Abstract* - **Now-a-days, steel has become an important part of our life due to its extensive applications in automotive, household appliances, business machine and heavy construction such as marine and chemical industries. Mild steel is selected for construction because of its mechanical properties and machine-ability at a low price, while at the same time; they have to be resisted against corrosion phenomena. For this study, ZrO2 nano particles were synthesized by solution combustion process and size was found to be 45 nm from X-ray diffraction. Plate sample (10x10x3 mm) of mild steel was used as a substrate and after Polishing to about Ra 20 µm using Al2O3 slurry; the samples were first cleaned with acetone and then ultrasonically cleaned in ethanol, then heat-treated at different temperatures like 400˚C and 500˚C in a muffle furnace. After heat treatment the samples are coated by applying ZrO2 nano particles with an airbrush device. Coated samples are tested for surface roughness, X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM).** **Now-a-days, steel has become an important part of our life due to its extensive applications in automotive, household appliances, business machine and heavy construction such as marine and chemical industries. Mild steel is selected for construction because of its mechanical properties and machine-ability at a low price, while at the same time; they have to be resisted against corrosion phenomena. For this study, ZrO2 nano particles were synthesized by solution combustion process and size was found to be 45 nm from X-ray diffraction. Plate sample (10x10x3 mm) of mild steel was used as a substrate and after Polishing to about Ra 20 µm using Al2O3 slurry; the samples were first cleaned with acetone and then ultrasonically cleaned in ethanol, then heat-treated at different temperatures like 400˚C and 500˚C in a muffle furnace. After heat treatment the samples are coated by applying ZrO2 nano particles with an airbrush device. Coated samples are tested for surface roughness, X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM).**

*keywords* - **ZrO2, Surface Roughness, Airbrush device, XRD, SEM & EDX**

## 1. INTRODUCTION

In present-days automobile vehicles have more inner components parts in the system. Those parts are more hard-wearing and more heat-resistant. The auto engine wastes a lot of fuel and to create a population because of incomplete gas combustion. Now nanotechnology and nano materials are likely to play a significant role in sparkplugs. Since nano materials are strongest, harder and resist wear and erosion, they are currently being considered for the use in sparkplug. The interest for nano structured ceramic materials which are synthesized in dimensions smaller than 100 nm has been growing in the last decades. The interest has been stimulated by the large variety of applications in industries such as fabrication of dense ceramics, sensors, batteries, capacitors, corrosion-resistant coatings, thermal barrier coatings, solid electrolytes for fuel cells, catalysts, cosmetics, health, automotive, bioengineering, optoelectronics, computers, and electronics etc. This paper investigated about Microstructure, phase structure, oxygen content and micro hardness of the coated material and heat-treated at different temperatures like 400˚C and 500˚C in a muffle furnace. Coated samples were characterized by XRD, SEM and EDX.

## 2. EXPERIMENTAL DETAILS

### 2.1 MATERIAL USED:

- $Zr(NO_3)_4$
- Ascorbic acid $C_6H_8O_6$
- Mild steel plate (10 x 10 x 3 mm

### 2.2 SYNTHESIS OF $ZRO_2$ NANO PARTICLES USING SOLUTION COMBUSTION PROCESS

$ZrO_2$ is prepared by solution combustion process 1:1.69 ratio amounts of zirconium nitrate and ascorbic acid is taken into a beaker and stirred it for 30 minutes on a magnetic stirrer and places it on a hot plate (~1000°C) [7].

$$Zr(NO_3)_4 + C_6H_8O_6 \rightarrow ZrO_2 + 4H_2O + 6CO_2\uparrow + 2N_2\uparrow$$

heating rapidly the solution containing the redox mixture boils, frothing, smoldering, flaming, fumes and catches fire and burns with an incandescent flame to yield $ZrO_2$ with the evolution of large amount of gases like carbon dioxide, hydrogen oxide in the form of flames and the procedure had shown in the Figure 1 & Figure 2.


*Figure 1: Sequences of procedure for preparation of $ZrO_2$ nano particles by the solution combustion process*

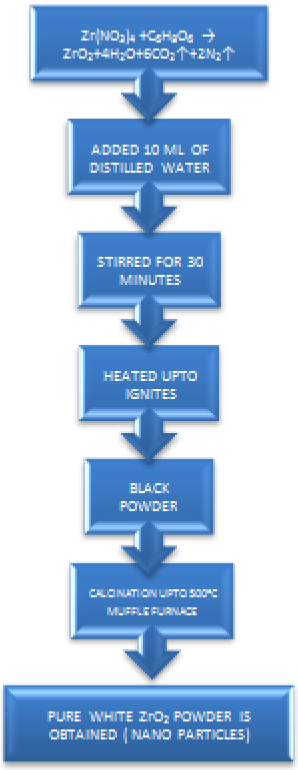


*Figure 2: The above flow chart shows Sequence of procedure of solution combustion process*

**2.3 OPERATIONS**

**2.3.1 SPECIMENS PREPARATION*:*** To perform the experimental test the mild steel specimen is prepared by the following steps

**2.3.2 CUTTING:** Experimental analysis and Surface condition of a specimen is considered as an important factor & it is necessary to prepare a uniform surface. Square specimens were obtained as a final specimen shape. 10*10*3 mm.

**2.3.3 POLISHING*:*** The specimens were polished using polish cloth and alpha-alumina 1 µm and 0.5 µm, and then washed with distilled water. The polished specimens were dried and tested for material composition by using EDX.

**2.4 COATING TECHNIQUE BY AIRBRUSH:**

An airbrush of model NX-A&NX-B NEW YORK was used for coating $ZrO_2$ nanoparticles on mild steel at different temperatures as shown in Figure 3. Prepared nanoparticles are taken into the airbrush reservoir, hose connector is connected to a compressor and the airbrush uses a pressure of 2 to 2.4 bars. As the force applied on the trigger a stream of fast-moving air through a venture, which creates a local reduction air pressure, that allows the particles to pull up from an interconnected reservoir at normal atmospheric pressure. The high velocity of the air carries the $ZrO_2$ nanoparticles and impinges on the mild steel.

*Figure 3: Air Brush Gun*

***Figure 4: Coating Process***

This technique was achieved using the setup system which consists of

- An electrical heater was used to heat the specimens to about $500^0$C which Measured by Thermocouple.
- Fine powder of $ZrO_2$ is taken into a reservoir of airbrush and the nano powder is sprayed on the specimen surface about 5cm above the specimen which has shown in figure 4.

**2.5 SCANNING ELECTRON MICROSCOPY:** The grain size, shape and surface properties like morphology were observed by the SEM (HITACHI S3400NS) machine at different magnifications. The SEM images of $ZrO_2$ nano particles are prepared by solution combustion process.

**2.6 ENERGY DISPERSIVE X-RAY SPECTROSCOPY:** The EDXS of $ZrO_2$ nano particles was done by the SEM (HITACHI S3400NS) machine. It reveals the composition present in the sample.

**2.7 SURFACE ROUGHNESS:** After heat treatment the samples at 400˚ C the surface roughness of the base metals are tested by using SJ-310 Portable Surface Roughness Tester. And after spraying $ZrO_2$ nano particles on the sample at 500˚ C , again the samples are tested for surface roughness as shown in Table 1

| S.NO | Base Metal | After Heat treated at 400˚C | After Spraying $ZrO_2$ nano particles at 500˚C |
|---|---|---|---|
| 1 | 1.355 | 1.044 | 1.1855 |
| 2 | 1.764 | 1.649 | 1.992 |
| Avg. | 3.119 | 2.693 | 3.0475 |

***Table 1: surface roughness values before and after coating $ZrO_2$ nano particles***

**3. RESULTS AND DISCUSSIONS**:

**3.1 X-RAY DIFFRACTION:** In the XRD pattern of the ZrO2 nanoparticles, the peaks are observed at 30.577, 35.151, 40.777, 50.354, 59.501 and 63.211 (h k l) values of the peaks are (0 1 2), (1 0 4), (1 1 0), (1 1 3), (1 1 6) and (3 0 0)respectively. These results coincide with JCPDS card number 82-1468, and it shows that the $ZrO_2$ nanoparticle consists of a Spherical shaped structure. The average crystalline size is measured using Debye-Scherer's formula [5].

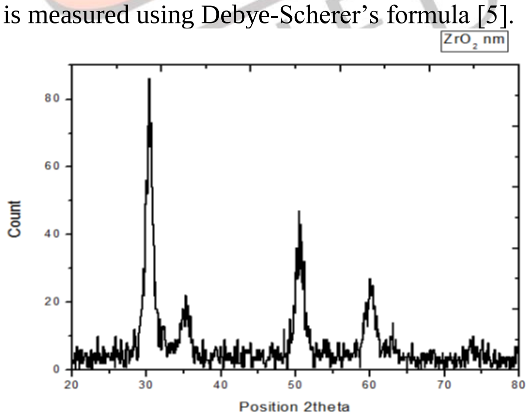


**Figure 5:** ***XRD pattern of $ZrO_2$ nanoparticles***

According to Debye-Scherer's equation:

$$D = \frac{0.9\lambda}{\beta\cos\theta} nm$$

Where D – Average size of the particle [nm]

$\lambda$ --Wavelength of the radiation [A°]

θ –Diffraction angle [degree]

B – Full width half maximum (FWHM) of the peak [radians]

From the above formula obtained average crystalline size is 45 nm. The lattice parameter a = b = 4.7589 A°, c = 12.9919 A°.

**3.2 SCANNING ELECTRON MICROSCOPY:** SEM images of $ZrO_2$ nanoparticles and the grain size, shape and surface properties like morphology were observed by using SEM with different magnifications. The SEM images of $ZrO_2$ nanoparticles show respectable morphology and the Grain size of $ZrO_2$ nanoparticles was nearly 153.2 nm as shown in Figure 6.

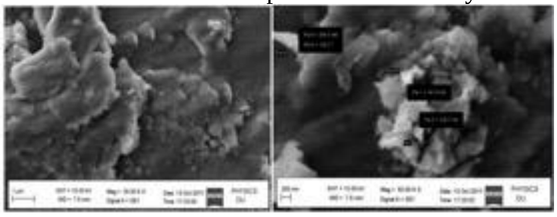

**Figure 6:** ***SEM image of a nano particle***

**3.3 EDX OF $ZrO_2$ NANO PARTICLES:** The EDX of the sample was done by the SEM (HITACHI S3400NS) machine. The Energy-dispersive X-ray spectroscopy reveals that the required phase has present. Zirconium (Zr) and oxygen (O) Elements are present in the sample as shown in Figure 7.

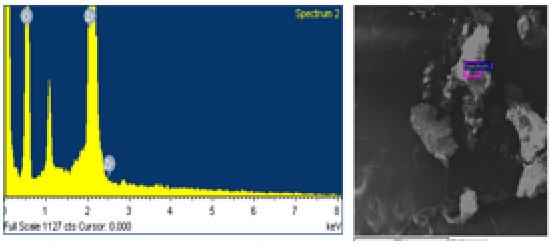


***Figure 7: EDX image of $ZrO_2$ nanoparticle***

| S.NO | Elements | Weight% | Atomic% |
|---|---|---|---|
| *1* | *Zr* | *63.75* | *23.57* |
| *2* | *O* | *36.25* | *76.43* |

***Table 2: Composition of $ZrO_2$ nanoparticle***

**3.4 SEM IMAGES OF THE BASE METAL:**

The grain size, shape and surface properties like morphology were observed by SEM images. It shows coarse grain structure and size were nearly 240 nm as shown in Figure 8 with different magnifications.

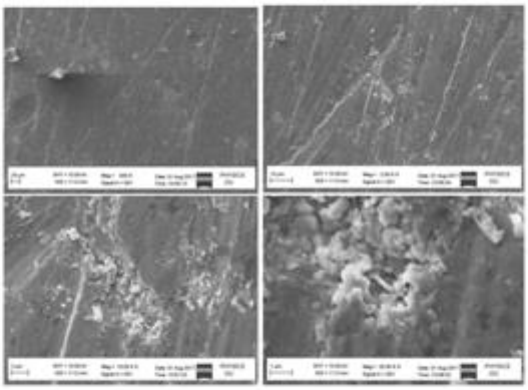

***Figure 8: SEM Images of Mild Steel***

**3.5 EDX OF BASE METAL:** The EDX of the sample was done by the SEM (HITACHI S3400NS) machine. The Energy-dispersive X-ray spectroscopy reveals that the required phase has present. Carbon (C), oxygen (O) and iron (Fe) Elements are present in the sample as shown in Figure 9 and the composition of the sample was clearly shown in Table 3.

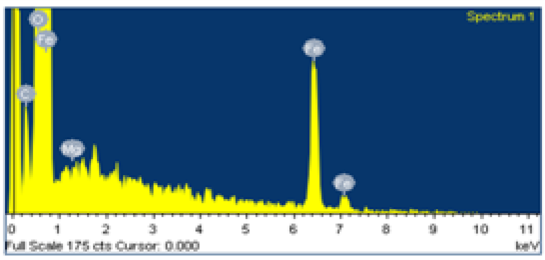


***Figure 9: EDX Images of Mild Steel***

| S.No | Base Material | Weight% |
|---|---|---|
| 1 | Carbon | 5.45 |
| 2 | Oxygen | 19.97 |
| 3 | Magnesium | 0.04 |
| 4 | Iron | 74.54 |
| 5 | Total | 100 |

***Table 3: Composition of a mild steel specimen***

### 3.6 SEM IMAGES OF COATED $ZRO_2$ NANO-PARTICLES ON MILD STEEL:

SEM images of coated $ZrO_2$ nanoparticles on mild steel at 500˚ C as shown in Figure 10. The grain size, shape and surface properties like morphology were observed by the SEM with different magnifications. Coated nanoparticles on mild steel at 500˚ C, shows respectable morphology when compared with a base metal of the sample and grain size of coated sample 500˚ C were nearly 234 nm.

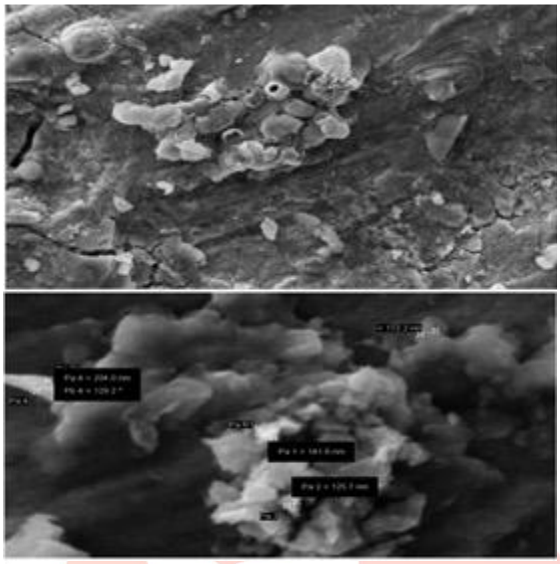

***Figure 10: SEM images of a coated specimen***

### 3.7 EDX OF COATED SAMPLE:

The EDX of the sample was done by the SEM (HITACHI S3400NS) machine. The Energy-dispersive X-ray spectroscopy reveals that the required phase has present. Carbon (C), oxygen (O), iron (Fe) and Zirconium (Zr) Elements are present in the sample as shown in Figure 11 and the composition was clearly shown in Table 4.

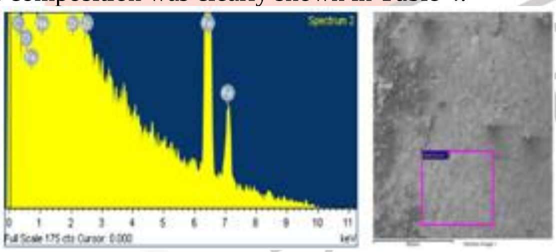

***Figure 11: EDX of the coated sample***

| S.NO | Elements | Weight% |
|---|---|---|
| 1 | C | 0.94 |
| 2 | O | 25.08 |
| 3 | Na | 1.83 |
| 4 | Fe | 56.63 |
| 5 | Zr | 15.52 |
| Total | | 100 |

***Table 4: Composition of coated specimen***

### 3.8 SURFACE ROUGHNESS*:*

Initially the surface roughness of the base metal is high when compared with a heat-treated sample at 400˚C due to the heat treatment the surface roughness of the sample is smooth. Surface roughness is high after deposition of $ZrO_2$ nanoparticles on mild steel at 400˚C, due to high bond generated between $ZrO_2$ and mild steel during the cold spraying process when compared with a heat-treated sample at 500˚C. Surface roughness is high after deposition of $ZrO_2$ nanoparticles on mild steel at 500˚C, due to high bond generated between $ZrO_2$ and mild steel during the cold spraying process when compared with Base Metal.

## 4. CONCLUSIONS

$ZrO_2$ nanoparticles are synthesized by the solution combustion process, the average crystalline sizes were found to be 45 nm. Compare to base metal the hardness of the coated metal is more. Surface roughness is high after deposition of $ZrO_2$ nanoparticles on mild steel at 500˚C, due to high bond generated between $ZrO_2$ and mild steel during the cold spraying process when compared with a heat-treated sample at 500˚C. Surface roughness is high after deposition of $ZrO_2$ nanoparticles on mild steel at 500˚C, due to high bond generated between $ZrO_2$ and mild steel during the cold spraying process when compared with Base Metal. The hardness of the coated sample at 500˚C is high when compared with base metal due to the deposition of $ZrO_2$ nanoparticles on mild steel. When compared with base metal the coated sample at 500˚C shows high hardness because of maximum deposition of $ZrO_2$ nanoparticles on mild steel. The hardness is high after coating nanoparticles on mild steel at 500˚C due to high friction generated which results in a good reaction between $ZrO_2$ nanoparticles and Mild steel.

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