Open Journal of Composite Materials, 2019, 9, 57-71
http://www.scirp.org/journal/ojcm
ISSN Online: 2164-5655
ISSN Print: 2164-5612

Corrosion and Wear Behavior of Nano-Zirconium (Zr) Coated Commercial Grade Cast Iron by Sol-Gel and Plasma Spray Process

Joel Hemanth[1*], Sara Sowjanya Steevenson[2]

[1]School of Engineering, Presidency University, Bangalore, India
[2]Visveswaraya Technological University (VTU), Belgaum, India
Email: *joelhemanth@hotmail.com

How to cite this paper: Hemanth, J. and Steevenson, S.S. (2019) Corrosion and Wear Behavior of Nano-Zirconium (Zr) Coated Commercial Grade Cast Iron by Sol-Gel and Plasma Spray Process. *Open Journal of Composite Materials*, **9**, 57-71.
https://doi.org/10.4236/ojcm.2019.92003

Received: January 4, 2019
Accepted: March 9, 2019
Published: March 12, 2019

Abstract

Hard facing with Nickel/cobalt based alloys for steel substrates are widely used for high temperature and pressure applications in chemically reactive environments due to their good corrosion and wear resistance properties. In the present research, the ceramic material, *i.e.*, zirconium, is coated on a hypoeutectic cast iron substrate to improve its corrosion and wear resistance. The substrate was coated with zirconium by sol-gel process as well as by the plasma spray process for comparison purpose. Results of the research indicated that the successful deposition of zirconium on the cast iron substrate by sol-gel deposition technique had improved both corrosion and wear resistance of cast iron. SEM analysis revealed that the coating was denser without any internal cracks indicating the soundness of deposition. Also, sol-gel process of coating indicated better wear resistance as compared with plasma spray coated cast iron. Thus, zirconium coating on the substrate has made cast iron sound (without any surface defects) along with excellent corrosion and wear resistance properties. This has made cast iron suitable for structural and automotive applications.

Keywords

Sol-Gel, Coating, Wear, Corrosion, Zirconium, Cast Iron

1. Introduction

Cast irons are widely used in various industries and automobiles particularly for engine cylinder, cylinder head, hydraulic valve bodies etc. due to their high machinability, good mechanical properties, good damping and wear resistance properties. When exposed to an aqueous corrosive medium the passivation layer is removed and hence the valve is often subjected to pitting corrosion or localized corrosion. Since cast iron is the most commonly used material for valve bodies, machine bases are often exposed to corrosive environment and subjected to corrosion [1]. Various coating materials are available to resist corrosion and wear and one such material is a ceramic. Ceramics have high chemical stability and high resistance to corrosion, wear and oxidation. Therefore, ceramic coating provided increases the wear and corrosion resistance of cast iron. One such ceramic is zirconium which has good mechanical properties, chemical stability, excellent thermal properties and wear and corrosion resistance, and hence it is selected as the material for coating of cast iron in the present research. The coefficients of thermal expansion (CTE) of zirconium and cast iron are $12 - 14 \times 10^{-6}\ K^{-1}$ and $13 \times 10^{-6}\ K^{-1}$ respectively [2]. Since zirconium is in thermal match with cast iron, it is used as a protective coating to resist wear and corrosion and also as a thermal barrier coating on valve bodies and machine bases. Several techniques are used for coating of zirconium such as chemical vapor deposition, physical vapor deposition, electro deposition, spray pyrolysis, plasma spray and sol-gel processes [3]. Also research is being conducted on gas phase deposition process of zirconium on titanium dioxide to form hybrid coatings [4]. Thus, the present research is mainly focused on improving the wear and corrosion resistance of commercial grade hypo-eutectic cast iron by nano coating of zirconium by sol-gel and plasma arc spray techniques wherein plasma arc spray is used for comparison purpose. Past research revealed that sol-gel process of ZrO_2 coating was already used on mild steel, stainless steel, brass etc. [5] [6]. Sol-gel method is used to form hard and protective layer on the substrate by the nano zirconium particles dispersed in a sol. In sol-gel method, numbers of monomers are converted into a colloidal solution that acts as the precursor for fabrication of metal oxides. Generally, sol is transformed to a gel in a diphasic system containing both liquid and solid phase [7]. Finally the liquid phase is removed by the drying process, which is followed by densification of the metal oxide. The rate at which solvent can be removed is determined by porosity or volume fraction by the dispersed molecules in the gel. The quality of the sol can be improved by addition of polymers like poly vinyl alcohol, poly vinyl butyral etc. [8]. Al_2O_3 used in sol-gel for coatings have been done on Fe-Cr alloys too by researchers [9]. However, the past research indicated that sol-gel method of coating seemed to improve the interfacial wettability of the coating and the substrate [10]. Investigations that have been carried out so for with zirconium as the coating material indicate that higher distribution is found at the edges and negligible in the interior regions. Research have also indicated that out of all ceramic materials, zirconium is the best for corrosion and wear resistance even under high temperature oxide environments [11] [12]. Few researchers have also found that thermal treatment is also necessary for poly-condensation to increase the mechanical properties through sintering at high temperatures. This helps in densification of grain growth of the deposited particles. Heat treatment also plays an important role in surface morphology of the coated materials [13]. Sol-gel process has many advantages like better control of chemical composition, ease of nano coating on cast iron, cheaper process and equipment's, adaptability for different functional group of materials. Note that zirconium exists in three structural phase's namely tetragonal, mono clinic and cubic and it can be stabilized by adding stabilizers in various weight percentages [14].

In sol-gel technique, different precursors like zirconium oxychloride octahydrate, zirconium acetate hydroxide, zirconium n-propoxide etc. are used to provide coating on metals. Research also reveals that bioactive hydroxyapatite coatings are formed by micro plasma spraying method on surgical steels [15]. It is observed that plasma spray process exhibits better wear resistance than other coating process since it forms nano crack arresting zones which harden the surface coating [16]. The residual strain developed in plasma spray process is less than hard facing technique and it can be controlled using temperature manipulation during cooling and varying air pressure [17]. Materials like aluminum alloys are used in high pressure application which leads to surface cracks and high pressure erosion; different coating techniques have been investigated so that it can operate in the environment of high pressure [18]. The coating strength of zirconium can be increased by adding an undercoat of Ni-Cr alloy which increases the thermal shock resistance. The powder for plasma spray process can be prepared from sol-gel route and stabilize using 8% weight yttrium by using zirconium oxychloride as precursor. A detailed literature survey indicated that for the preparation of sol-gel zirconium, n-propoxide was commonly used as the precursor for coating process and less research on zirconium oxychloride octahydrate as the precursor for the preparation of Zr sol-gel. Therefore the present research is focused on zirconium oxychloride octahydrate precursor. The parameters for plasma spray coating method were chosen from the literature available [19] and tests were conducted for comparison purpose.

Studies on ceramic material such as molybdenum were also studied by few researchers for selecting the suitable precursors that reduced oxidation level since the zirconium is an unstable molecule that reacts with oxygen forming zirconium oxides. Jin *et al.* [20] described the microstructure and mechanical properties of the electro-thermal explosion of directionally sprayed molybdenum coatings were determined by means of SEM, XRD and micro hardness tests respectively. Experiments have indicated that the dominant wear mechanism of EEDS coatings is slight furrow compared with that of HVAS coatings which is serious furrow and delaminating. Zhu *et al.* [21] had demonstrated by his research that molybdenum nitride coatings were synthesized by ion-beam-assisted-deposition (IBAD). Rhushikesh *et al.* [22] gave a new approach for synthesis of high quality faceted microcrystalline coatings of molybdenum (Mo), tungsten (W) and their carbides and composites. These studies were carried out using hot filament chemical vapor deposition (HF-CVD) method; results indicated that the oxide molecules reach the substrate which is kept beneath the filament assembly. Secondly, molecular hydrogen gets dissociated into atomic hydrogen and subsequently reaches the substrate to react with oxide molecules, leading to the precipitation of a pure metal. This method can also be used in situ process to convert metallic coatings into their carbides and/or composites. Laribi *et al.* [23] [24] from his research showed the relationship between, the deposition and post-deposition conditions on the wear resistance of molybdenum and Cr-Ni-Mn alloyed steel coatings thermally sprayed on a 35CrMo4 steel substrate. The results indicate that there was an improvement in the adhesion on the substrate that decreased the friction coefficient against a hardened 100Cr6 steel ball. He also pointed out form his research that surface treatments and coatings are the practical approach used to extend the lifetime of components and structures especially if the surface is the most solicited part of the engineering component. The author also proved the tribological resistance through sliding wear and impact fatigue resistance.

2. Experimental Method

2.1. Specimen for Testing

Commercial grade hypo eutectic cast iron (Carbon Equivalent CE = 4.22, chemical composition as shown in **Table 1** was obtained from Fen fey Metallurgical's, Bangalore, INDIA) specimen is usually subjected to hard-facing where the temperature is raised to its critical temperature to form the coating by the process of diffusion, dilution and penetration of the coating material. Hard facing and coating technique reduces residual stress in the cast iron substrate which causes metallurgical structural changes and distortion of the substrate and results in increased mechanical properties and corrosion resistance. Therefore, to form hydrophobic and nano coating on cast iron that has a melting point 1340˚C, without affecting the properties, specimen with dimension 5 mm × 5 mm × 5 mm was selected for sol-gel coating and 60 mm × 20 mm × 5 mm for plasma spray coating. Zirconium has a melting temperature of 1855˚C and has a good thermal match with cast iron is an added advantage for the coating. Coating is formed by sol-gel process at the sintering temperature of the substrate.

2.2. Preparation of Sol-Gel

In this research, zirconium oxychloride octahydrate ($ZrOcl_2.8H_2O$) salt is used as a precursor to form zirconium dioxide coating on the specimen. Zirconium oxychloride octahydrate is added to oxalic acid, and stirred using a magnetic stirrer for 4 hours so that it forms zirconium oxalate, a white transparent sol. Poly vinyl alcohol was used as a surfactant to control the viscosity, maintain the size and growth of the nano particle in the sol. The pH of the solution was maintained at 5.5 - 7. The process was controlled by the addition of nitric acid to the sol. After the solution is prepared, it is left for ageing for about 24 hours so that the following reaction occurs.

$$\mathrm{ZrOcl_2 \cdot 8H_2O + (COOH)_2 \cdot 2H_2O \rightarrow ZrO(COO)_2 + 2Hcl + 10H_2O}$$

Table 1. Chemical composition of cast iron tested.

Element	C	Si	Mn	P	Ni	S	Fe
wt.% Composition	3.42	2.4	0.41	0.08	1.5	0.04	Balance
(Carbon Equivalent CE = 4.22, Hardness 180 BHN)							

Thus, zirconium oxychloride octahydrate reacts with oxalic acid to form zirconium oxalate in the form of a gel. All the chemicals used in the present investigation are obtained from ACE Rasayanam, Kerala, India.

Finally, the precipitated sol is left for ageing for 24 hours before it is used for dip coating. The dip coated cast iron specimen in this sol was sintered up to 600˚C so that it forms zirconium dioxide by the process of decomposition. The zirconium oxide particles deposited can be characterized using UV spectroscopy technique and the surface morphology was examined using SEM.

2.3. Fabrication of Zirconium Dioxide Coated Cast Iron Substrate by Sol-Gel Technique

Cast iron specimen of dimension 5 mm × 5 mm × 5 mm were chosen for coating was finely ground in an emery sheet of grade 120 - 1200 grit size. Then the specimen was treated with concentrated nitric acid to get the desired surface roughness for effective bonding of coating on the surface. Before dip coating, the sol-gel was centrifuged for half an hour and finally the specimen was dipped in the sol-gel for half an hour and withdrawn at a rate of 4 cm/min followed by drying at 200˚C for 15 minutes and thus process was repeated thrice for better deposition. Finally the specimen was kept in a muffle furnace and sintered in stages from 300˚C to 600˚C with constant temperature rise of 100˚C for every half an hour and the reaction occurred was:

$$\mathrm{ZrO}\left(\mathrm{COO}\right)_2 \rightarrow \mathrm{ZrO}_2 + \mathrm{CO} + \mathrm{CO}_2$$

It is observed from SEM and EDS analysis that a coasting thickness of about 500 nm can be obtained by the sol-gel, process.

2.4. Fabrication of Zirconium Dioxide Coated Cast Iron Substrate by Plasma Spray Process

Another method used in the present research for zirconium coating is the plasma spray process in which molten zirconium is sprayed on the cast iron substrate. In micro plasma spraying method the porosity level is also reduced and better surface morphology was observed.

Cast iron specimen of dimension 60 mm × 20 mm × 5 mm was polished using emery sheet of grit size 120 - 1200. This specimen was coated with zirconium dioxide using plasma spray process which is an alternative to EB-PVD and APS process. The powder used in plasma spray process was less than 25 μm along with low powder feed rate [25]. It has been taken care that the bond strength required for coating, its modulus and Poisson's ratio were also carefully considered since it is directly affected by the coating parameters [26]. The nozzle pressure of argon gas for this process was set to 100 - 120 psi, flow rate of argon gas was set to 80 - 90 lpm, nozzle pressure of hydrogen gas was 50 psi, and flow rate of hydrogen gas to 15 - 18 lpm, current and voltage was set to 500 amps and 65 - 70 volts. The powder feed of zirconium dioxide was 40 - 50 gms/min and the spray distance was set to 2 - 3 inch. With these parameters it is possible to fabricate a zirconium dioxide coating of 100 micron thickness on the specimen which is confirmed through SEM and EDS analysis. Both the coatings (sol-gel was plasma) was done at Hindustan Metallurgical's, Bangalore, India.

2.5. Testing of Nano Zr Coated Cast Iron Substrates

X-Ray Diffraction (XRD), Dynamic Light Scattering (DLS), SEM and Energy Dispersive X-ray Spectrography (EDS) analysis were carried out to study the presence of deposition, particle size of deposition, distribution of the deposition and thickness of deposition. UV spectroscopy was also used to confirm the presence nano particles (make of XRD and DLS equipment's are by: Olympus scientific solutions, USA).

The specimens for micro structural studies were polished according to metallurgical standards and fine polishing was done using alumina powder and diamond paste, the procedure described elsewhere [27]. The specimens were etched with 3% Nital to evolve grain boundaries. Micro-structural studies were done using Olympus optical metallurgical microscope [28].

Dry wear tests were conducted according to the standard procedure using computerized pin-on-disc wear testing machine (by Ducom Instruments, India) and SEM (Jeol make) analysis was made to study the wear mechanism [29]. The specimen is a pin of size 6 mm in diameter and 25 mm long were used where as the disc is of alloy steel having hardness of HRC 62. Dry wear tests were conducted at a sliding distance of 1000 m with a velocity of 2.5 m/ sec and load of 2 kg.

Corrosion test was conducted using electrochemical potentio-dynamic polarization corrosion method (ASTM G 59-97, 2009 standard by Precision instruments, Bangalore, INDIA) to analyze the mechanism of corrosion [30].

3. Results and Discussion

SEM and EDS analysis shows that around 500 nm coating can be obtained through sol-gel process and 100 microns by the plasma coating. Therefore sol-gel coated cast iron specimens were further tested for their wear and corrosion properties since nano coating of Zr particle is possible by this process. Hence much of the results and discussion part is based on sol-gel coated cast iron substrate.

3.1. Sol-Gel Characterization

Sol-gel was prepared using the procedure discussed in Section 2.2 wherein the zirconium oxy chloride octahydrate reacts with oxalic acid to form zirconium oxalate. This when heated to a temperature above 600˚C, due to decomposition zirconium oxalate, zirconium dioxide is formed. The powder formed was characterized using UV spectroscopy to confirm the presence of zirconium dioxide. **Figure 1** shows the wavelength of the particle was found to be in the range of 187 nm to 200 nm. This critical size range under certain chemical conditions, allows particles grow to form colloids, which are affected both by sedimentation and force of gravity. The formation of more open continuous network of low density and high performance ceramic components occurred in 2 and 3 dimensions. This forms metal oxide that involves in connecting the metal centers with oxo (M-O-M) or hydroxo (M-OH-M) bridges, therefore generating metal-oxo or metal-hydroxo polymers in solution.

3.2. XRD Characterization of Sol-Gel

X-Ray diffraction analysis for the cast iron specimen is presented in **Figure 2**. XRD analysis was carried out after sintering and post heat treating the sol at 600˚C in CVD chamber. The diffraction peak at $\theta = 31.69˚$ confirms the presence of medium dispersed zirconium (m-zirconium) on the cast iron substrate. The XRD reflections at Bragg's angle 2θ value of $2\theta = 10˚$ - 80 representing (111), (200) and (202) planes for Zr (JCPDS 06-0696). The less intensity of Zr peaks was due to the size (10 - 45 μm) of the coating material. The graph reveals the presence of zirconium from peaks $29˚ \le 2\theta \le 32˚$, $45˚ \le 2\theta \le 54˚$ and $62˚ \le 2\theta \le 70˚$. The XRD pattern indicated the phase structure of zirconium synthesized by sol-gel route to be of tetragonal and monoclinic structures.

3.3. Dynamic Light Scattering (DLS) Characterization of Sol-Gel

Dynamic Light Scattering (DLS) analysis was also used to analyze the particle size of zirconium dispersed in the sol-gel. With a scattering angle set to 90˚, viscosity of the dispersion medium to 0.895 MPa/sec and the temperature to 25˚C, a count rate of the test 1642 kCPS was obtained. The test results as depicted in **Figure 3** shows the particle size of dispersed zirconium are in the range of 200 nm to 400 nm.

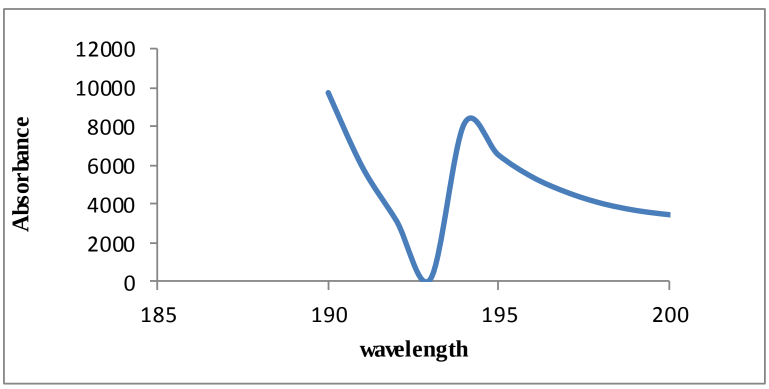

Figure 1. Plot of UV spectroscopy.

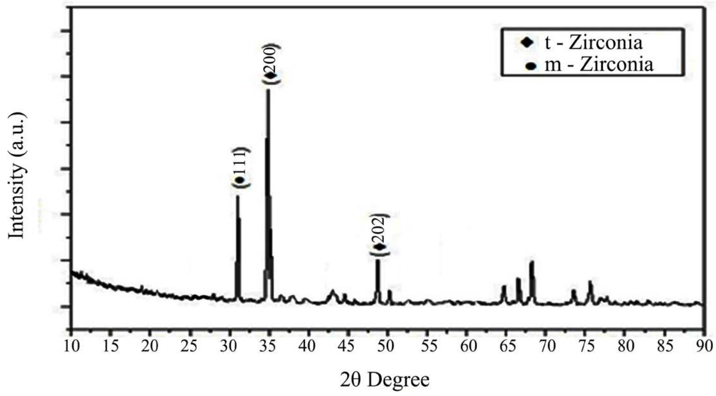

Figure 2. XRD graph (ZrO_2 sintered at 600˚C).

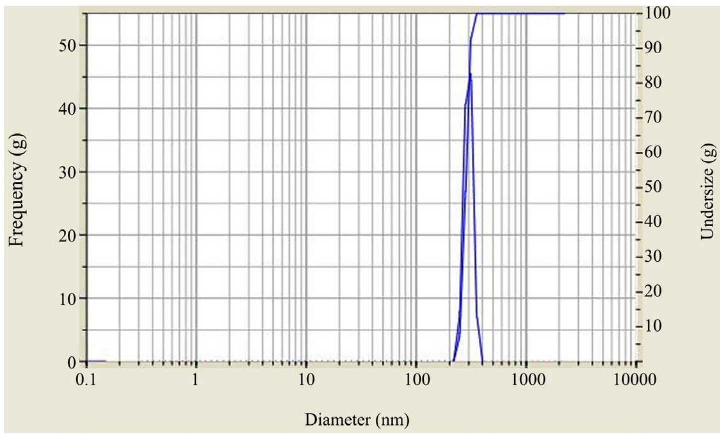

Figure 3. DLS graph showing particle size of synthesized zirconium in sol-gel.

3.4. SEM and Energy Dispersive X-Ray Spectrography (EDS) Characterization

SEM and EDS analysis were used to study the sol-gel Zr coating applied on cast iron substrate specimen. SEM image of Zr coated specimen is shown in Figure 4 and EDS image is shown in Figure 5. SEM studies revealed that there is huge uneven distribution of the nanoparticles due to oxidation formed in the CVD chamber during post heat treatment processes. It is also observed that the coating formed using sol-gel specimen is uniform indicating the formation of hard spots due to deposition of the carbon originated from the base material (cast iron substrate) and reactant materials. EDS analysis however revealed an average Zr particle size ranging from 674.7 nm to 6.321 μm. Therefore, it is validated that zirconium forms a fully dispersed coating on the base metal substrate. Finally, the sustainability of the coating is validated through corrosion and wear testing of the coated substrate as discussed in the following sections.

3.5. Influence of Friction Force on Sol-Gel Coated Cast Iron and SEM Analysis of the Worn Surface

Wear tests were conducted on specimens of cast iron coated with zirconium by sol-gel and plasma spray process using computerized pin-on disc wear testing machine. A coating of thickness 100 microns for plasma spray specimens and 540 nm for sol-gel specimens was tested. From the wear test, friction force plots were obtained for both sol-gel and plasma coated cast iron substrate. It is observed form the wear test plots as shown in Figure 6 and Figure 7 that, up to 70 sec of operation, the friction force of sol-gel coated specimen was lower than that of plasma spray coated specimen indicating resistance to wear and after 70 seconds of operation, the plasma coated specimen showed the lower frictional force. Form the results it is concluded that for longer duration of wear testing with the application of the load, sol-gel coating processes a better wear resistance compared to plasma spray coating processes.

Microstructure of coated cast iron tested and SEM photograph of the worn surface of the same are shown in Figure 8 and Figure 9 respectively. Microstructure of cast iron tested (with sol-gel coating) has ferrite in pearlitic matrix having a hardness value of 180 BHN. According to SEM analysis of coated and uncoated cast iron specimen, the wear mechanism is suggested by two modes. Mode-1 refers to adhesive wear (severe wear) and is predominant for the uncoated cast iron and Mode-2 which is essentially an abrasive wear (mild wear) which is observed in the case of coated cast iron as result of hard ceramic coating particles embedded on the surface of cast iron. The worn surface also revealed the loose fragments on the surface indicating that the substrate is coated.

SEM examination also reveals that the worn surface of the coated cast iron consists of both hardened and deformation layers. The structure of the hardened layer consists mainly of the fragmented ceramic (Zr) phase (Figure 9). However, the depth of the hardened layer in the cast iron is markedly increased by increasing the thickness of coating. SEM studies also revealed that because of limited plastic deformation in the mild wear regime of coated cast iron, the fragmentation of the ceramic particles on the surface appears as brittle fracture and in the case of un-coated cast iron; the plastic flow is due to deformation of ferrite present in cast iron.

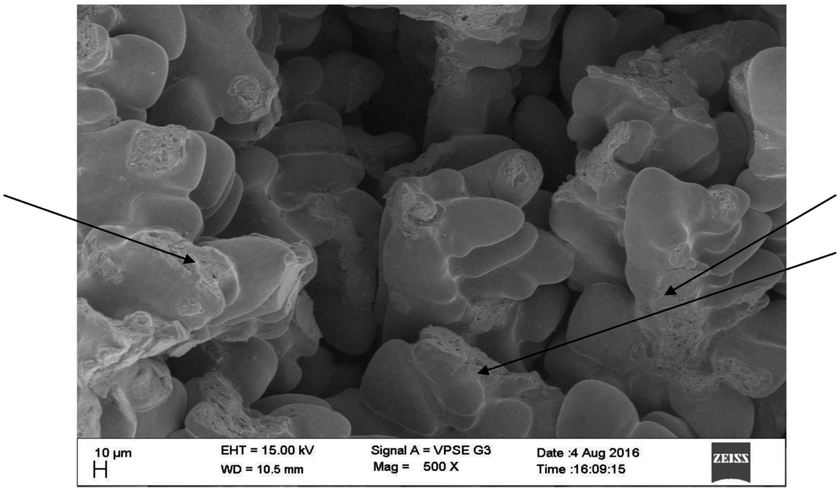

Figure 4. SEM photograph of sol-gel coated cast iron specimen (arrows indicate the presence of sol-gel).

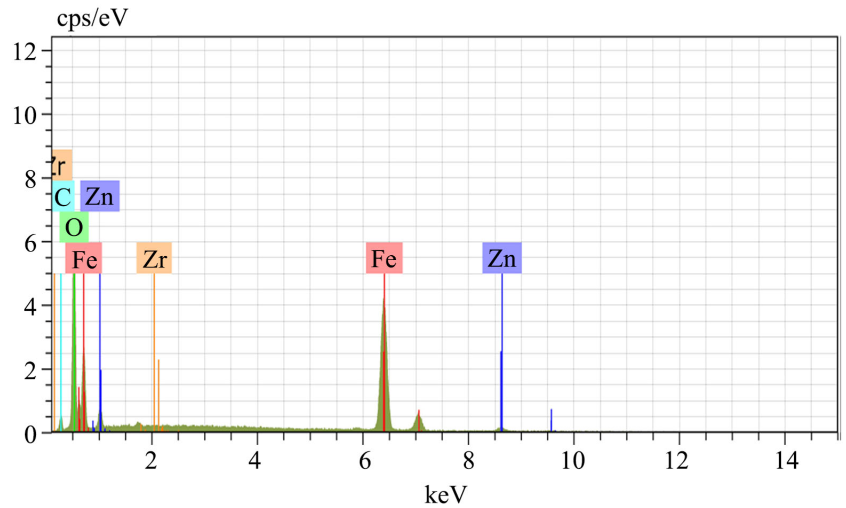

Figure 5. EDS graph of sol-gel coaated cast iron specimen.

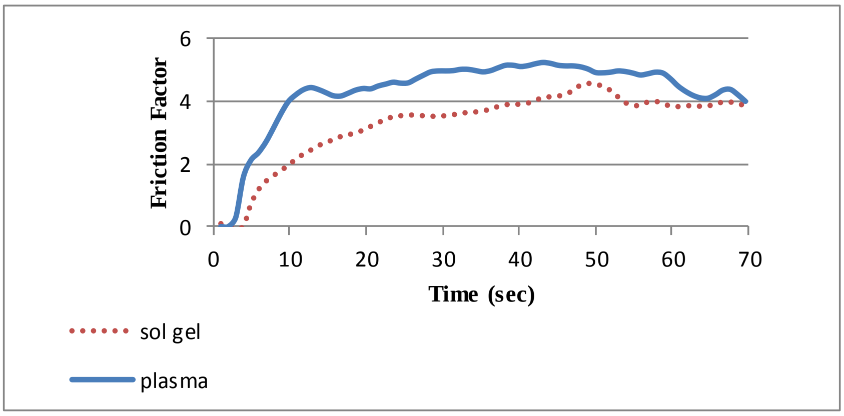

Figure 6. Friction factor of sol-gel and plasma spray coated cast iron specimen (testing time: up to 70 sec).

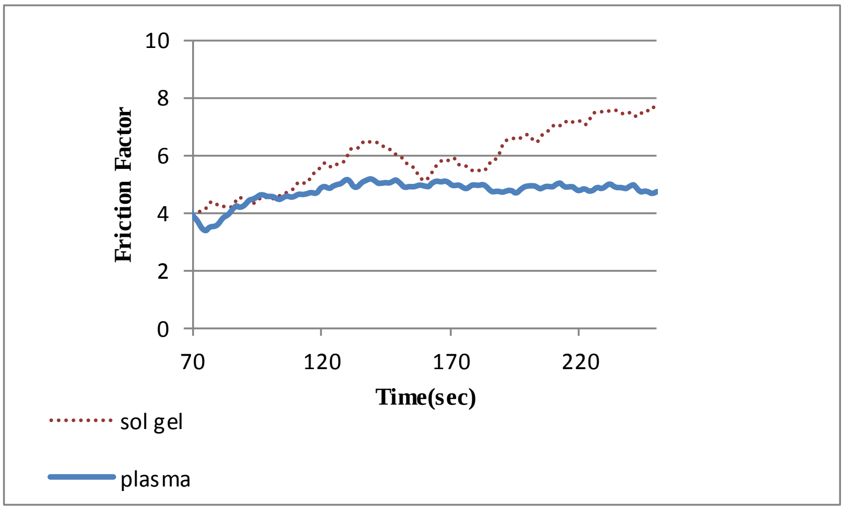

Figure 7. Friction factor of sol-gel and plasma spray coated cast iron specimen (testing time: after 70 sec).

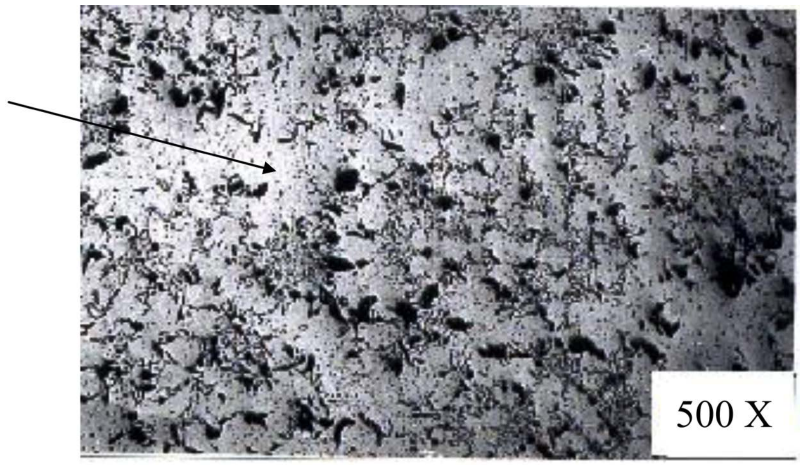

Figure 8. Microstructure of sol-gel coated cast iron. (whitish patch indicated by the arrow shows the presence of the sol-gel).

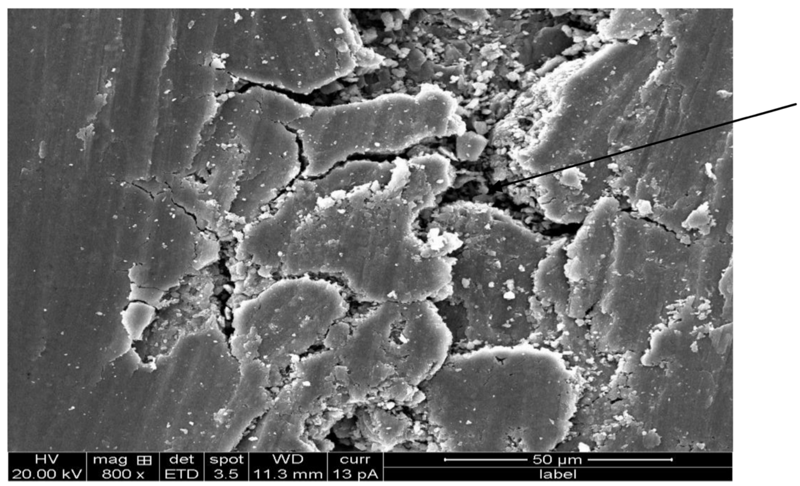

Figure 9. SEM photograph of worn surface of sol-gel coated cast iron (arrow indicates the cracking of the sol-gel because of wear).

3.6. SEM Analysis of the Corroded Surface

It is observed through the necked eye that the electrodes underwent sporadic pitting after the test. The SEM photographs of the corroded electrode surface of coated and uncoated cast iron after polarization in 3.5% normal NaCl (electrolyte used which is almost equal to sea water normality) with a pH value of 6 are shown in Figure 10 and Figure 11 respectively. It is clear from the SEM photographs that the cast iron without coating is the most susceptible to corrosion as compared against the coated specimens. The greatest difference between the corrosion rates of coated and uncoated cast iron is seen after many hours of corrosion testing. It can be seen from the SEM photomicrographs (Figure 10 and Figure 11) that the uncoated cast iron containing ferrite in pearlitic matrix has experienced the severest corrosion, as indicated by the large swollen blisters with shallow pitting on the surface. These blisters were found to peel off easily and contained a network of pits. These blisters in the form of layer were removed by immersion the specimen in Clark's solution and after light polishing, small cavities were found in the metal surface. Corrosion of cast iron coated with Zr is seen to suffer some small pitting attack on the surface which is only the localized attack on the surface (Figure 11). To the naked eye, coated cast iron surface were seen to be bright and shiny, and free of any visible form of corrosion damage, whereas the uncoated cast iron were observed to contain some patches. However, it is observed from SEM photomicrographs (Figure 10 and Figure 11) that the electrodes underwent sporadic pitting in the case of uncoated cast iron as compared with the coated cast iron.

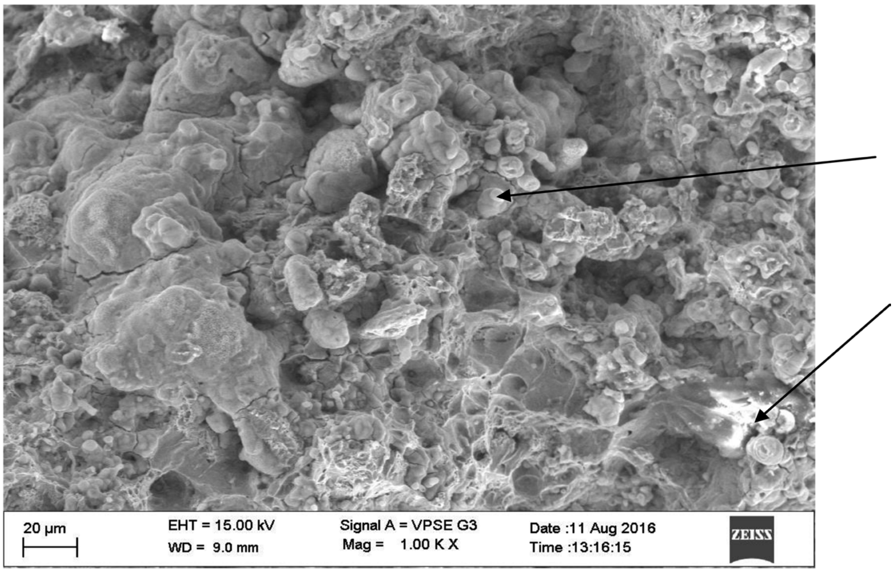

Figure 10. SEM photograph of uncoated cast iron after corrosion testing (arrow shows the corroded region).

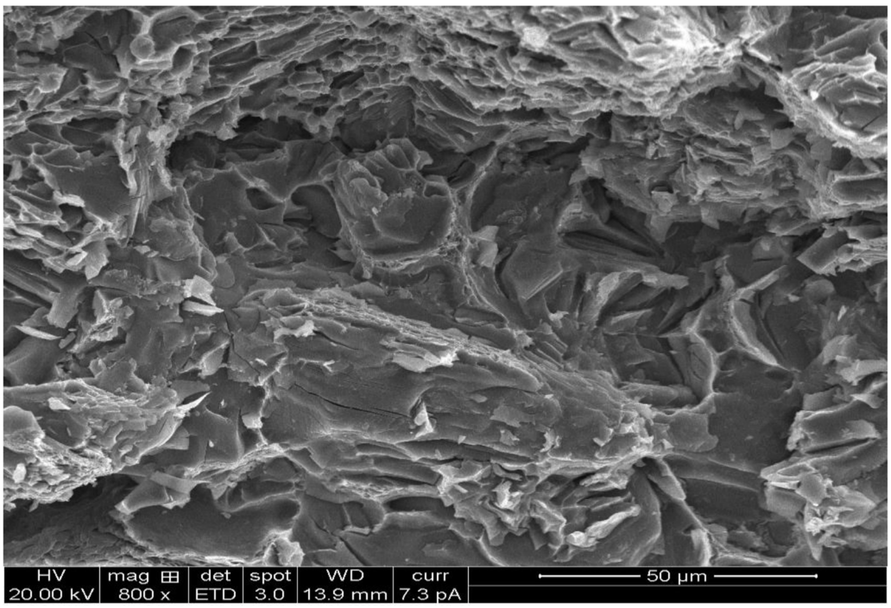

Figure 11. SEM photograph of sol-gel coated cast iron after corrosion testing.

4. Conclusions

Results of the research reveal the following:

Formation of nano Zr particles (size: 187 to 200 nm) by sol-gel process and the presence of these particles in sol-gel was confirmed through UV spectrography and XRD analysis. Particle size of deposition (size: 200 to 400 nm) and distribution of nano particles on the substrate were confirmed through DLS and SEM analysis. SEM analysis indicated the successful deposition of zirconium on the cast iron substrate by sol-gel deposition technique. SEM image also revealed that the coating was denser without any internal cracks indicating the soundness of deposition. The XRD pattern indicated the phase structure of zirconium synthesized by sol-gel route to be of tetragonal and monoclinic structures.

Wear test results of the coated samples subjected to wear testing indicate that the coating provided has good wear resistance under the force of friction. Therefore, it is concluded that material removal rate of cast iron can be decreased by having a dense packed ceramic coating over the substrate. SEM analysis of the worn surface indicates that the uncoated cast iron has undergone severe wear such as the adhesive wear whereas the coated cast iron has undergone mild wear because of the hard ceramic coating of the particles. Also, sol-gel process of coating indicates better wear resistance (in terms of friction force) as compared with plasma spray coated cast iron.

Corrosion test results indicate that uncoated cast iron specimens have undergone pitting action indicating severe corrosion attack as compared with the coated cast iron which shows only the localized mild corrosion without any pitting action.

Thus, zirconium coating by the sol-gel process on the cast iron substrate makes it sound (without any surface defects), has improved the properties (wear and corrosion resistance) of cast iron and makes it suitable for structural and automotive applications.

This research can also be aimed at other type of cast iron (alloyed) with other types of coating that suit that particular cast iron.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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