



# Tribological and corrosion behavior of $(100-x)(\text{Fe}_{70}\text{Ni}_{30})-(x)\text{ZrO}_2$ composites synthesized by powder metallurgy



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## HIGHLIGHTS

- $(100-x)(\text{Fe}_{70}\text{Ni}_{30})-(x)\text{ZrO}_2$  composites were synthesized via powder metallurgy route.
- $\gamma-(\text{Fe,Ni})$  and  $\alpha-(\text{Fe,Ni})$  phase formation occurred in  $\text{Fe}_{70}\text{Ni}_{30}$  metal matrix.
- 10 wt%  $\text{ZrO}_2$  particles dispersion in metal matrix showed the maximum wear resistance and corrosion resistance.

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## ABSTRACT

The aim of the present work is focused on the effect of  $\text{ZrO}_2$  addition on the tribological and electrochemical behavior of  $(\text{Fe,Ni})$  matrix based composites.  $(100-x)(\text{Fe}_{70}\text{Ni}_{30})-(x)\text{ZrO}_2$  composites were prepared using powder metallurgy route, where  $x$  is 2.5, 5, 10 and 15 wt%. The formation of taenite  $\gamma-(\text{Fe,Ni})$  and kamacite  $\alpha-(\text{Fe,Ni})$  phases in the matrix was evident from x-ray diffraction (XRD).  $\text{ZrO}_2$  particles have not participated in any intermediate phase formation and found to be dispersed in matrix as a unreacted phase. Corrosion test of prepared composites was conducted in 3.5% NaCl aqueous solution (simulated sea water) at room temperature. The pin on disc tribological tests has shown a strong impact of reinforcing hard  $\text{ZrO}_2$  particles in improving the wear resistance whereas the presence of both taenite and  $\text{ZrO}_2$  phases has contributed in improving the corrosion resistance. Increasing the fraction of  $\text{ZrO}_2$  reinforcement particles up to 10 wt% improves both the wear resistance and corrosion resistance. Beyond this limit, property degradation was evident. It is due to the high contact of ceramic to ceramic particles and weak metal/reinforcement interface at higher reinforcement content, resulting in brittle behavior. Electrochemical impedance spectroscopy (EIS) studies indicate the adsorption/diffusion phenomenon at the interfaces which is responsible for localized corrosion. Microstructural and surface roughness test before and after the tests were performed using scanning electron microscope (SEM) and profilometer studies.

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## 1. Introduction

In mechanical engineering, innovative methods with the rigorous research made it possible to implement new designs using advanced material systems for development of new types of machinery for new and diversified applications. Various kinds of metals are used for producing different machineries. Iron and its alloys are known for their high strength and high melting temperature and are widely used for tribological and load bearing applications [1,2]. Metal matrix composites (MMC's) have been found

as special candidates serving the above purpose. These composites consist of at least two constituents, which are physically and chemically distinct in nature. One phase is suitably distributed into another to achieve the combined properties of both the phases in the final composite. The addition of reinforcement phase strengthens the matrix by providing it support. Fiber reinforcement to develop MMCs has been focused so far. But fibers as reinforcement phase are costly and they also improve the properties in a particular direction. Particles as a dispersion or reinforcement phase are less costly and they can be easily added and distributed throughout the matrix by selecting the appropriate route [3]. Ceramic particles reinforced metal matrix composites are potential candidates for structural, automobile, aviation and transportation applications. The properties and applications of these composites

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can easily be altered by varying the process parameters such as type and amount of reinforcement as well as the matrix phase, synthesis route and processing temperature [4]. Ceramic particles (ex. TiC, SiC,  $\text{Al}_2\text{O}_3$ ,  $\text{ZrO}_2$ , etc.) are hard in nature and they improve the structural, mechanical and electrochemical behavior of these composites when added in some metal matrix [5–7]. Different routes such as stir casting [8,9], plasma synthesis [10], hot pressing [11] etc are well-known routes for the production of metal matrix composite, depending upon the compositional selection. Powder metallurgy is a very reliable route for the fabrication of high melting temperature materials. It is also advantageous due the low cost, better homogeneity, unlimited compositional choices and controlled porosity [12,13].

There are a number of studies based on the composites taking light weight metals (Al, Ti, Cu etc) as matrix due to their low density and high corrosion resistance [14,15]. But, they lack in strength and hardness thus cannot be used for heavy duty applications. Iron is a reliable candidate, commonly used in structural and tribological applications from several decades due to its better physical properties such as density, strength and high melting temperature than most of the other common metals. From the previous literature, iron, nickel and their alloys are the consistent materials for replacing the low strength metals used as a matrix [16]. Therefore, focus towards the development and study of iron based composites should be concerned. Very few studies are reported on iron matrix composite. Gupta et al. [17–20] have reported the effect of  $\text{Al}_2\text{O}_3$  content on the mechanical and corrosion behavior of Fe- $\text{Al}_2\text{O}_3$  composites. Jha et al. [21] have studied the phase and mechanical behavior of Fe- $\text{ZrO}_2$  composite using powder metallurgy route.

Fe-Ni alloys are mostly studied for magnetic and high temperature applications and  $\text{ZrO}_2$  is known as an inert, hard and refractory material with a very high melting temperature as compared to most of the other ceramic materials. But, to the best of our knowledge, there is a lack of systematic studies on mechanical and corrosion behavior of  $\text{ZrO}_2$  reinforced Fe-Ni matrix based metal composites. Therefore, the present work is an effort towards developing a high strength composite with high wear resistance and corrosion resistance characteristics for heavy duty applications. Chloride anion is the most common aggressive ion found in many industrial and natural environments. Iron-rich phase in  $\text{Fe}_{70}\text{Ni}_{30}$  alloy system is susceptible to localized corrosion in chloride containing solutions therefore  $\text{ZrO}_2$  reinforcement has been done in  $\text{Fe}_{70}\text{Ni}_{30}$  alloy composition to retard the attack of chloride ions. The composites were processed through powder metallurgy route. The effect of reinforcement concentration on the corrosion behavior in simulated sea water environment (3.5 wt% NaCl solution) is also focused along with the wear study in present work.

## 2. Experimental

### 2.1. Material synthesis

$(100-x)(\text{Fe}_{70}\text{Ni}_{30})-(x)\text{ZrO}_2$  composites were prepared by using powder metallurgy route. Here, x represents the fraction of reinforcement phase ( $\text{ZrO}_2$ ) which was varied as 2.5, 5, 10 and 15 wt% whereas  $\text{Fe}_{70}\text{Ni}_{30}$  is the iron-nickel matrix containing 30% Ni. The supplier and characteristic of each powder are shown in Table 1. All the powders were weighed accurately in stoichiometric ratio using the electronic balance. The powders along with 1 wt% binder (dextrine) were mixed vigorously in a sealed container for dispersing the reinforcement phase uniformly. The powder mixture was pressed into cylindrical shapes ( $d = 12.9$  mm,  $h = 14.5$  mm) using a high carbon steel die with the help of uniaxial hydraulic press at a load 120 kN. The prepared specimens were sintered in argon atmosphere using an atmosphere controlled alumina tube

**Table 1**

The characteristics and company of the powders used for synthesis.

Powder	Supplier	Powder characteristics
Fe	Loba Chemie	250–300 $\mu\text{m}$ , 99.5% purity
Ni	Loba Chemie	200 mesh, 99.5% purity
$\text{ZrO}_2$	Loba Chemie	11–39 $\mu\text{m}$ , 99.8% purity

furnace. In the first step, the temperature was raised to 500 °C with a heating rate of 5 °C/min and held for 1 h to remove the binder, followed by sintering at 1150 °C/3 h. Density measurement of sintered specimens was carried out using Archimedes method.

### 2.2. Characterization

#### 2.2.1. Phase and wear characterizations

Phase determination of specimens was done by using X-ray diffraction (XRD) with the help of Mini Flex II Desktop X-ray diffractometer with  $\text{Cu-K}\alpha_1$  radiation and Ni-filter.

Wear test was done using a pin on disk wear testing machine taking 4 m/sec sliding speed of pin with total 14.4 km distance travelled. All the tests were conducted at three different loads 10, 20 and 30 N respectively. Wear loss in the specimens was measured using an analytical balance of  $\pm 0.0001$  g precision after the test at different loads. Wear rate calculation was done by dividing the total volume loss by total distance travelled.

Microstructure study of the specimen surface after wear test was recorded by using Evo 18 Zeiss Scanning Electron Microscope (SEM).

#### 2.2.2. Corrosion test

**2.2.2.1. Coupon preparation.** 3 mm thick disc of the prepared specimen was taken and abraded using different grade emery papers and diamond paste. They were then properly cleaned by ultrasonication in acetone medium for 10 min  $1 \times 1 \text{ cm}^2$  surface area of the dried specimen was exposed for the corrosion test whereas remaining surface was completely coated by using protective nail paint.

**2.2.2.2. Electrolytic solution.** Electrolytic solution of NaCl was prepared by dissolving 3.5 wt% NaCl in distilled water. 150 ml volume of the electrolyte was taken for each experiment.

**2.2.2.3. Electrochemical measurements.** Corrosion study of the specimens in 3.5 wt% NaCl solution was performed on Biologic electrochemical cell with three electrodes connected to Biologic Instrument Potentiostat/Galvanostat. The prepared composites were used as working electrodes whereas platinum electrode and standard Ag-AgCl were used as auxiliary and reference electrode, respectively.

**2.2.2.4. Potentiodynamic polarization measurements.** Before starting measurements, the electrodes were kept in electrolytic solution for around one hour to get stabilize and to obtain open circuit potential (OCP). Potentiodynamic polarization curves were recorded with respect to OCP in the range  $-250$  to  $+250$  mV at a scan rate of 0.5 mV/s. Corrosion current density ( $I_{\text{corr}}$ ) and Corrosion potential ( $E_{\text{corr}}$ ) were calculated by extrapolating the cathodic and anodic linear segments of the Tafel plots to the intersection point. Protection efficiency has been obtained by using respective  $I_{\text{corr}}$  values.

**2.2.2.5. Electrochemical impedance spectroscopy (EIS).** Electrochemical impedance spectroscopy (EIS) measurements were

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