Research Article

Effect of Sintering Mechanism on the Properties of ZrO_2 Reinforced Fe Metal Matrix Nanocomposite

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The present paper reports phase, microstructure, and compressive strength of ZrO_2 reinforced Fe Metal Matrix Nanocomposites (MMNCs) synthesized by powder metallurgy (P/M) technique. High purity grade iron metal powder was mixed with varying percentage of zirconium dioxide (5–30 wt%), compacted, and sintered in argon atmosphere in the temperature range of 900–1100°C for 1 to 3 hours. X-ray diffraction (XRD) analysis of specimens was done in order to study the phases present and scanning electron microscopy was carried out to determine the morphology and grain size of the various constituents. XRD result shows the presence of Fe, ZrO_2 , and $\text{Zr}_6\text{Fe}_3\text{O}$ phase. $\text{Zr}_6\text{Fe}_3\text{O}$ phase forms due to reactive sintering and is not reported earlier by researchers throughout the globe. SEM results showed the presence of dense microstructure with the presence of Fe, ZrO_2 , and some nanosize $\text{Zr}_6\text{Fe}_3\text{O}$ phases.

1. Introduction

Metal Matrix Nanocomposites (MMNCs) have gained a significant attention in the recent past due to their excellent and high performance characteristics, thereby providing simultaneous service to the industries as well as towards research activities [1]. Addition of hard ceramic reinforcement in the ductile metallic matrix leads to improvement in electrical, structural, and mechanical properties [2, 3]. There are several routes which are put forth by the researcher throughout the globe for the fabrication and production of quality MMNC products [4–6]. Few of them are stir casting, powder metallurgy, physical vapor deposition (PVD), chemical vapor deposition (CVD), liquid infiltration techniques, and so forth [7]. Amongst them, powder metallurgy route is used to develop better MMNC products with improved overall homogeneity [8]. Apart from the processing technique, another important factor which plays a vital role is the size and morphology of the powder particles [9, 10]. Literature reports large number of investigations on phase and microstructure of aluminum,

magnesium, and copper as the matrix phase material but unfortunately no systematic attempt has been made to study phase, microstructure, electrical and mechanical properties of iron based composites [11].

Rahimian et al. [8] reported Al- Al_2O_3 composites having average particle size of alumina in size range of 3, 12, and 48 μm processed via P/M techniques, that is, by sintering in the temperature range of 500–600°C for 30–90 min. It was found that at higher sintering temperatures a denser structure is formed due to higher diffusion rates. They also explained the effect of sintering time on the microstructure and showed that the dependence of the diffusion on time may be given by the relation

$$r = 2.4\sqrt{Dt}, \quad (1)$$

where r is radial distance, D is the diffusion coefficient, and t is the sintering time. It can be seen that the atomic displacement is proportional to the square root of time which in turn is responsible for the atomic diffusion leading to grain

TABLE 1: Nomenclature of Fe-ZrO₂ composite specimens.

Sl. number	Sintering temperature (°C)	Sintering time (h)	Specimen code 5% ZrO ₂	Specimen code 10% ZrO ₂	Specimen code 20% ZrO ₂	Specimen code 30% ZrO ₂
1	900	1	5ZrFe900(1)	10ZrFe900(1)	20ZrFe900(1)	30ZrFe900(1)
2	1000	1	5ZrFe1000(1)	10ZrFe1000(1)	20ZrFe1000(1)	30ZrFe1000(1)
3	1100	1	5ZrFe1100(1)	10ZrFe1100(1)	20ZrFe1100(1)	30ZrFe1100(1)
4	900	2	5ZrFe900(2)	10ZrFe900(2)	20ZrFe900(2)	30ZrFe900(2)
5	1000	2	5ZrFe1000(2)	10ZrFe1000(2)	20ZrFe1000(2)	30ZrFe1000(2)
6	1100	2	5ZrFe1100(2)	10ZrFe1100(2)	20ZrFe1100(2)	30ZrFe1100(2)
7	900	3	5ZrFe900(3)	10ZrFe900(3)	20ZrFe900(3)	30ZrFe900(3)
8	1000	3	5ZrFe1000(3)	10ZrFe1000(3)	20ZrFe1000(3)	30ZrFe1000(3)
9	1100	3	5ZrFe1100(3)	10ZrFe1100(3)	20ZrFe1100(3)	30ZrFe1100(3)

coarsening. Chua et al. [12] have carried out investigation on Mg₉Al_{10.7}Zn_{0.15}Mn (wt%) reinforced with 10 vol% SiC particulates having particle size varying from 15 to 50 μ m. These were sintered at 400°C for 30 min followed by extrusion. SEM micrographs of as extruded polished composites reinforced with different sizes of SiC in the transverse direction revealed that although some agglomeration of SiC particles could be observed, the overall distribution generally appeared to be reasonably homogeneous.

Moustafa et al. [13] reported the copper matrix reinforced with either Ni-coated or uncoated SiC and Al₂O₃ particulate composites which were processed by means of the powder metallurgy route. Reinforcing particles of SiC and Al₂O₃ were coated with a thin layer of nickel by electroless method. Coated or uncoated reinforcement particles of either SiC or Al₂O₃ were added to copper metal powders with nominal loading of 20 wt.% and mixed in a mechanical mixer. Each mixture of the investigated powders was cold-compacted at 600 MPa and sintered at 900°C in hydrogen atmosphere. SEM results showed that almost no detectable porosity and very good adhesion between particles and Cu-matrix are observed in Cu-coated Al₂O₃ composite. In the case of Cu-uncoated Al₂O₃ composite, porosity between Al₂O₃ particles and Cu-matrix is observed indicating poor adhesion.

In our research group, exhaustive investigations on Fe-Al₂O₃ and Fe-ZrO₂ metal matrix composite showed that there is formation of nanosize iron aluminate (FeAl₂O₄) and nanosize iron zirconium oxide (Zr₆Fe₃O) phases as a result of reactive sintering phenomenon. Formation of nano-iron aluminate and nano-iron zirconium oxide phase improves the hardness, wear, deformation, and corrosion properties of the composites significantly. Formation of these nanophases and their characteristics depend on sintering temperature and time [14–19]. Literature studies revealed that there are few reports on using zirconium dioxide (ZrO₂) as the reinforcement material in iron matrix [20]. Fe-ZrO₂ nanocomposites find applications in heavy duty components like railway wagon wheels and so forth where pure iron cannot give the superior structural and mechanical properties [21].

Therefore, on the basis of previous investigations carried out by researchers worldwide, present investigations on the phase, microstructural characteristics, and compressive strength of the Fe-ZrO₂ (5–30 wt%) based Metal Matrix

Nanocomposites processed via powder metallurgy technique have been carried out. The results of these investigations are reported in this paper.

2. Experimental Details

Electrolytic iron (Fe) metal powder having 99.5% purity and particle size in the range 250–300 mesh (49–58 μ m) and active zirconium dioxide (ZrO₂) having monoclinic structure with particle size range of 0.8–7 μ m and mean size of $1.31 \pm 0.92 \mu$ m were used as starting materials. Composites selected for investigation contain 5, 10, 20, and 30 wt% zirconium dioxide (ZrO₂), respectively. Mixed powders were ball milled dry, using zirconia balls as the grinding and mixing media, keeping powder-to-ball ratio of 1 : 2 [22, 23]. Mixed powders were dry-compacted using a hydraulic press in a cylindrical shaped die under a constant load of 46 MPa. Green compacts of diameter 13 mm and height 20 mm were obtained after pressing. Green compacts were sintered in an argon atmosphere in the temperatures range of 900°C to 1100°C for 1 to 3 hours. After sintering, the surface of the specimen was polished. A nomenclature, for example, 5ZrFe900(1), is given to each specimen. Here, 5 denotes weight percentage of zirconium dioxide, Zr denotes zirconium dioxide, Fe denotes iron, 900 denotes the sintering temperature, and 1 denotes time of sintering in hr. In this manner there were four Fe-ZrO₂ systems (5, 10, 20, and 30 wt% ZrO₂) and in each system there were 9 specimens, sintered for 9 different temperature (900, 1000, and 1100°C) and time (1, 2, and 3 hours) schedules, respectively. Therefore, 36 specimens were prepared for this particular investigation. Table 1 illustrates the detailed nomenclature of Fe-ZrO₂ nanocomposites specimens. Figure 1 shows some of the synthesized nanocomposites specimens.

Phase determination was done by powder X-ray diffraction (XRD) using Rigaku Desktop Miniflex II X-Ray diffractometer employing Cu-K α radiation and Ni-filter. *Microstructure* was studied using Inspect S-50, FP 2017/12 scanning electron microscope. Cylindrical samples of 12 mm diameter and 15 mm thickness were used for SEM studies. The specimens were polished using various grades of emery paper (1/0, 2/0, 3/0, and 4/0) and finally polished with diamond

FIGURE 1: Few of the synthesized Fe-5% ZrO_2 nanocomposite specimens.

FIGURE 2: XRD pattern of (a) 5ZrFe900(1), (b) 5ZrFe1000(1), and (c) 5ZrFe1100(1), respectively.

paste, followed by pure HCl etching for 20 seconds. *Compressive strength* was determined using an Instron Universal Testing Machine (UTM). Prior to the compression test, cross-sectional area and height of the samples were measured. Dry compression test was done on the specimen up to a load of 4500 Kg, after which it was unable to bear the load. Load was applied on the samples gradually with crosshead moving at a speed of 0.05 cm/min. Load versus deformation was recorded with the help of a chart recorder.

3. Results and Discussion

3.1. X-Ray Diffraction. Figure 2 shows XRD patterns of specimens (a) 5ZrFe900(1), (b) 5ZrFe1000(1), and (c) 5ZrFe1100(1), respectively. Thus, Figure 2 shows effect of sintering temperature at a fixed sintering time and fixed ZrO_2 (5 wt%) concentration on the development of different phases. Specimens 5ZrFe900(1), 5ZrFe1000(1), and 5ZrFe1100(1) showed presence of iron (Fe), zirconium

FIGURE 3: XRD pattern of (a) 30ZrFe900(1), (b) 30ZrFe1000(1), and (c) 30ZrFe1100(1), respectively.

dioxide (ZrO_2), and iron zirconium oxide ($\text{Zr}_6\text{Fe}_3\text{O}$) phases, respectively. It was found that $\text{Zr}_6\text{Fe}_3\text{O}$ phase concentration increases as we increase the sintering temperature from 900°C to 1100°C . In a similar manner Figure 3 shows XRD pattern of specimens (a) 30ZrFe900(1), (b) 30ZrFe1000(1), and (c) 30ZrFe1100(1), respectively. As per previous observation, specimens 30ZrFe900(1), 30ZrFe1000(1), and 30ZrFe1100(1) also showed the presence of iron (Fe), zirconium dioxide (ZrO_2), and iron zirconium oxide ($\text{Zr}_6\text{Fe}_3\text{O}$) phases, respectively. It was seen from the XRD plots that the amount of iron zirconium oxide phase increases with an increase in the concentration of ZrO_2 . Higher ZrO_2 concentration in these nanocomposites specimens leads to higher formation of $\text{Zr}_6\text{Fe}_3\text{O}$ phase. The specimens with 30% ZrO_2 have higher concentration of $\text{Zr}_6\text{Fe}_3\text{O}$ phase in comparison to specimens with 5% ZrO_2 .

Representative XRD patterns of specimens with different percentage of ZrO_2 , sintered at a temperature of 1100°C for 1 h of time interval, are shown in Figure 4: (a) 5ZrFe1100(1), (b) 10ZrFe1100(1), (c) 20ZrFe1100(1), and (d) 30ZrFe1100(1). XRD results were matched with JCPDS data file in order to reveal the various phases present in the nanocomposites specimens. Specimen 5ZrFe1100(1) showed the presence of iron (Fe), zirconium dioxide (ZrO_2), and iron zirconium oxide ($\text{Zr}_6\text{Fe}_3\text{O}$). The present specimen showed high intensity peak

FIGURE 4: XRD pattern of (a) 5ZrFe100(1), (b) 10ZrFe100(1), (c) 20ZrFe100(1), and (d) 30ZrFe100(1), respectively.

of Fe, some smaller peaks of ZrO_2 , and a single peak of Zr_6Fe_3O .

In a similar manner specimens 10ZrFe100(1), 20ZrFe100(1), and 30ZrFe100(1) also showed the presence of (Fe), zirconium dioxide (ZrO_2), and iron zirconium oxide (Zr_6Fe_3O). Amount of Zr_6Fe_3O phase increases as we increase the amount of zirconium dioxide in the nanocomposites specimens. The intensity of iron zirconium oxide phase was the lowest in specimen 5ZrFe100(1) and was the highest in specimen 30ZrFe100(1). It was found from the results that the iron zirconium oxide phase formation took place as a result of reactive sintering between iron and zirconium dioxide particles. The average crystallite size (D) of Zr_6Fe_3O was found out from the Scherrer formula given by equation

$$D = \frac{0.9\lambda}{\beta \cos \theta}, \quad (2)$$

where λ is the wavelength of X-ray, β is the full width at half maxima, and θ is the angle of diffraction. From the XRD peak corresponding to hkl value of (311) of Zr_6Fe_3O , it was found

that the average crystallite size lies within the size range of 42–62 nm, respectively.

Density and hardness values of the various Fe- ZrO_2 Metal Matrix Nanocomposites have been described in our earlier publication [17]. Average green densities for 5%, 10%, 20%, and 30% ZrO_2 specimens were found to be 4.98, 4.96, 4.79, and 4.77 gm/cc, respectively. Maximum sintered density as a function of sintering temperature and time for 5%, 10%, 20%, and 30% ZrO_2 specimens was found to be 5.74, 5.64, 5.76, and 5.71 gm/cc. Variation in density values depends upon the formation of iron zirconium oxide (Zr_6Fe_3O) phase and the densifying mechanism. At lower sintering temperature and time, Zr_6Fe_3O phase formation is small and there is sintering between Fe metal particles. The corresponding density values are higher. In specimens with higher ZrO_2 content, there is more amount of Zr_6Fe_3O phase formation due to reactive sintering followed by densification, which also leads to increase in density. Hardness of the specimen as a function of composition, sintering temperature, and time lies in the range of 41–78 HRH. Hardness of specimens sintered at 1100°C is relatively higher. But it decreases with increasing sintering time. The hardness of 30% ZrO_2 specimens is however very much higher than the other specimens with lower ZrO_2 concentration [17].

3.2. Microstructure. Figure 5 shows the scanning electron micrograph of specimen 5ZrFe100(1) at (a) 1000x, (b) 5000x, (c) 10000x, and (d) 25000x magnification, respectively. Figure 5(a) shows the electron micrograph of specimen at 1000x which shows highly dense composite structure with the presence of negligible amount of intragranular and intergranular porosities. Figure 5(b) shows the micrograph of the same specimen at 5000x which shows homogenized particle distribution of Fe, ZrO_2 , and Zr_6Fe_3O phases. The black grains are of iron followed by white grains of zirconium dioxide and grey grains of iron zirconium oxide, respectively. The same micrograph when viewed at 10000x (Figure 5(c)) shows presence of some micron size particles of all the three phases. The particles of Fe are of size 2–4 μm , those of ZrO_2 are of size 3–5 μm , and those of Zr_6Fe_3O are of size 1–3 μm . Figure 5(d) shows micrograph of the same specimen at 25000x, and it shows nanosize grains of Zr_6Fe_3O . The size of these particles lies in the range of 50–500 nm, respectively.

Figure 6 shows the scanning electron micrograph of specimen 10ZrFe100(1) at (a) 1000x, (b) 5000x, (c) 10000x, and (d) 25000x magnification, respectively. Figure 6(a) shows a more denser microstructure of the composite specimens in comparison to the specimen 5ZrFe100(1). Figure 6(b) shows the micrograph of the same specimen at 5000x. The micrograph shows homogenous distribution of particles with the presence of some large and some smaller size particles of Fe, ZrO_2 , and Zr_6Fe_3O . The overall size of the particles can be determined by Figure 6(c). It shows iron particles of size 2–5 μm , zirconium dioxide of particle size 1–2 μm , and iron zirconium oxide of particle size lying in the range of submicron size to few nanometer sizes. Figure 6(d) shows the scanning electron micrograph of the same specimen at

FIGURE 5: SEM of specimen 5ZrFe100(1) at (a) 1000x, (b) 5000x, (c) 10000x, and (d) 25000x magnifications, respectively.

FIGURE 6: SEM of specimen 10ZrFe100(1) at (a) 1000x, (b) 5000x, (c) 10000x, and (d) 25000x magnifications, respectively.

FIGURE 7: SEM of specimen 30ZrFe100(1) at (a) 1000x, (b) 5000x, (c) 10000x, and (d) 25000x magnifications, respectively.

25000x. This micrograph shows nanosize particle of Zr_6Fe_3O lying in the range of 80–400 nm, respectively.

Figure 7 shows the SEM of specimen 30ZrFe100(1) at (a) 1000x, (b) 5000x, (c) 10000x, and (d) 25000x magnification, respectively. Figure 7(a) shows a denser microstructure in comparison to specimens 5ZrFe100(1) and 10ZrFe100(1) with the presence of no porosity. This increase in densification can be attributed to the presence of more amount of ZrO_2 and in turn the iron zirconium oxide phase formation. Figure 7(b) shows micrograph of the same specimen at 5000x revealing uniform distribution of different phases present in composite specimens. Figure 7(c) shows the grains of iron zirconium oxide which are of size 1–2 μm . Figure 7(d) shows the micrograph of the same specimen at 25,000x which shows nanosize grains of iron zirconium oxide phase.

It can be concluded from the above results that for 5% and 10% of ZrO_2 reinforcement particles of constituent phases appear in a distinct and separate manner. From dense microstructure of all the specimens and microcracking in 30% ZrO_2 reinforced specimen, it can be concluded that sintering is taking place by the formation of liquid phase. Iron zirconium oxide (Zr_6Fe_3O) phase may be helping in sintering as liquid phase and crystallizing in nanoform, respectively. In the present liquid phase sintering iron zirconium oxide is forming a major network due to reaction mechanism. As liquid phase sintering progresses, it leads to filling of iron

zirconium oxide particles in the open and channel pores of the specimens. Thus, this liquid phase sintering leads to improvement in grain growth and thus improves the compressive strength.

3.3. Compressive Strength. Stress versus strain plots of specimens 5ZrFe100(2) and 10ZrFe100(2) are shown in Figures 8 and 9, respectively. Stress versus strain plots can be divided into three regions. The initial first region, which extends up to around 98.07 MPa, shows that slope of stress versus strain curve is large. The region may be elastic and there is no bulging in the specimen. In the second region the specimen shows a bulging effect due to enhanced plastic deformation. The slope of stress versus strain curve decreases. This region extends up to a strain of approximately 0.4. When the dislocation movement is pinned by the presence of nano-iron zirconate grains or micrometer size zirconia grains, the rate of plastic deformation decreases. These specimens become hard and the slope of stress versus strain curve increases. From Figures 8 and 9, it can be concluded that the presence of nanosize iron zirconium oxide ceramic reinforcement along with ZrO_2 gives a strengthening mechanism to the specimens. There was only a bulging effect on the periphery of the specimens and there was no crack generation or failure in any of the specimens. From the stress versus strain plots average yield strength and compression modulus of specimens

FIGURE 8: Stress versus strain plot of specimen 5ZrFe100(2).

FIGURE 9: Stress versus strain plot of specimen 10ZrFe100(2).

5ZrFe100(2) and 10ZrFe100(2) was determined, which was found to lie within the range of 220.65 MPa–269.68 MPa and 550.74 MPa–628.80 MPa for specimens 5ZrFe100(2) and 10ZrFe100(2), respectively. It was also seen that for specimen 10ZrFe100(2) the value of yield strength and compression modulus was higher in comparison to that of the specimen 5ZrFe100(2).

4. Conclusions

A systematic study on phase and microstructure of Fe-ZrO₂ Metal Matrix Nanocomposites prepared by powder metallurgy technique has been reported in the present paper. The experimental results have been discussed critically and the following important conclusions have been drawn:

- (i) Iron zirconium oxide phase formation takes place due to reactive sintering between iron and zirconia particles.

- (ii) Formation of iron zirconium oxide phase depends on the sintering temperature and time.
- (iii) SEM result shows the dense phase microstructure with the presence of nanosize particles of iron zirconium oxide.
- (iv) Liquid phase sintering takes place in the specimen with 30% ZrO₂ due to which the iron zirconium oxide particles get filled in the open as well as in the channel pores.
- (v) During compression the hardening of composite takes place due to pinning of dislocation movement in ductile iron matrix by nanosize ceramic reinforcement. Yield strength and compression modulus values improve for specimens with higher percentage of ZrO₂.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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