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Novel Zinc / Tungsten Carbide Nanocomposite as Bioabsorbable Implant

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Abstract

There is a lack of bioabsorbable materials with adequate mechanical strength suitable for implant applications that provide temporary support while tissue integrity is restored, especially for pediatric applications. Bioabsorbable metals have emerged as an attractive choice due to their combination of strength, ductility, and biocompatibility *in vivo.* Zinc has shown great promise as a bioabsorbable metal, but the weak mechanical properties of pure zinc limit its application as an implant material. This study investigates zinc-tungsten carbide (Zn-WC) nanocomposite as a novel material for bioabsorbable metallic implants. Ultrasound-assisted powder compaction was used to fabricate Zn-WC nanocomposites. This study includes the material characterization of microstructure, microhardness, and degradability. Results showed that tungsten carbide nanoparticles enhanced the mechanical properties of Zn, and maintained the favorable corrosion rate of pure Zn. These results encourage further investigation of Zn-WC nanocomposites for biomedical applications with the ultimate goal of creating safe and efficacious bioabsorbable metallic implants for many clinical applications.

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Declaration of Interest Statement

All authors in the author list declare that there is no conflict of interest.

Keywords

Nanocomposites; Zinc; Bioabsorbable implant

1. Introduction

Permanent implant materials are frequently used in clinical cases that only require their presence temporarily. These situations are less-than-ideal as the long-term presence of implants is associated with many complications, including infections [1], implant migration [2], altered tissue growth [3, 4], stress shielding [5], toxicity [6-8], and subsequent surgeries. Permanent implants are also unsuitable for the pediatric patient population due to their inability to accommodate growth [9-11], Conversely, bioabsorbable implant materials provide transient support that allows for the restoration of a tissue's physiological integrity followed by complete reabsorption of the implants. Most bioabsorbable implants are currently polymer-based [12]; however, their lower mechanical strength and viscoelastic behavior have limited their clinical use for load-bearing applications, such as bone staples, fixation plates, ACL screws, cardiovascular and nonvascular stents, and spinal fusion cages and clips [13, 14], Bioabsorbable metallic implants are an attractive alternative. Metals have a higher mechanical strength and toughness necessary for load-bearing applications and have a proven history of biocompatibility *in vivo.*

Magnesium- and iron-based alloys have been extensively studied as candidates for bioabsorbable metallic implants with some success [15], However, their drawbacks include the non-favorable corrosion rate and chronic inflammatory response[16, 17], Zinc (Zn) has recently generated interest as a suitable candidate for bioabsorbable metallic implants. As an essential element in basic biological functions, Zn is required for the proper function of numerous proteins that regulate the proliferation, differentiation, and apoptosis of cells, and is involved in nucleic acid metabolism, signal transduction, and gene expression [18], Therefore, living tissues have transport mechanisms that regulate Zn levels, which combat against toxic cellular levels [19, 20], Additionally, recent *in vivo* studies have shown that Zn implants demonstrate steady corrosion rates with no severe inflammation, platelet aggregation, thrombosis or intimal hyperplasia [21-23], Furthermore, Zn has a greater elongation to failure (60-80%) than magnesium (13%) and iron (18%) [22], This can positively influence the fatigue resistance and fracture toughness of the Zn- based implants. However, insufficient mechanical strength inhibits pure Zn from being used for load-bearing applications [24], Alloying can properly enhance the strength, but at the cost of other favorable properties, such as corrosion rate [25], ductility [26], and biocompatibility [27], Additionally, there are limits to the extent that the properties of metals can be improved through alloying.

Metals reinforced with nanoparticles have emerged as an important class of materials that offer significantly enhanced mechanical, thermo-physical, and electrical properties [28, 29], It is therefore believed that nanoparticles can also be added to Zn melts to significantly enhance Zn's mechanical properties. In this study, non-cytotoxic tungsten carbide (WC) nanoparticles [30] were incorporated into Zn as the nano-reinforcement through ultrasound-

assisted powder compaction. WC nanoparticles dispersion in Zn matrix was studied, as well as its impact on hardness. Furthermore, the degradation rate in simulated body fluid was studied, and the change in surface morphology after immersion testing was evaluated. Compared with conventional strengthening methods, e.g. alloying, which commonly has a highly increase corrosion rate [26], Zn matrix nanocomposite fabricated in this work provided high strength and had a corrosion rate similar to pure Zn.

2. Material and Methods

2.1 Fabrication and characterization of Zn-WC Nanocomposites

Ultrasound-assisted powder compaction was used to fabricate Zn-WC nanocomposites (Fig. 1). Tungsten carbide (WC) nanoparticles (200 nm average diameter) were well-mixed with Zn powders (50 pm average diameter) by mechanical shaking. The powder mixture was compacted in cylindrical stainless steel mold (inner diameter of 2 cm) by a hydraulic press machine for Zn- WC pellets. These pellets were melted with ultrasound processing (20 kHz) under an inert atmosphere (Ar) at 450°C for 30 minutes and allowed to cool to room temperature. The distribution and dispersion of WC nanoparticles, as well as the element composition, were characterized by scanning electron microscopy (SEM), transmission electron microscopy (TEM) and energy- dispersive X-ray spectroscopy (EDX).

2.2 Biodegradation studies of Zn-WC nanocomposite microwires

Zn-WC nanocomposite was cast into a borosilicate tube by vacuum and went through thermal fiber drawing for microwires [31] to represent bioabsorbable devices for biodegradation characterization. The immersion test (ASTM G31-72) was carried out, where samples were kept in Simulated Body Fluid (SBF) for 14 days at 37°C. The SBF used in this study had ionic concentrations equal to that of human blood plasma, as well as an equal pH value (Table 1). Inductively coupled plasma optical emission spectrometer (ICP-OES) and ICP- mass spectrometer (ICP-MS) were carried out for Zn and W ion concentration measurement in the SBF. The changes in the surface morphologies after immersion were studied by environmental scanning electron microscopy (ESEM). The analysis was conducted with a ZEISS Supra 40 Variable Pressure SEM (VP-SEM) equipped with a Thermo Noran System 6 energy-dispersive X-ray spectroscopy (EDS) system. Vickers Microhardness test was performed before and after the emersion test.

2.3 Statistical Analysis

The statistical significance of differences between groups during immersion testing was determined using one-way ANOVA followed by Tukey post-hoc analysis. The SPSS statistical software package 24.0 for Windows (IBM, Armonk, NY, USA) was used for statistical analysis. Significance was established by a value of $p < 0.05$. Data are expressed as mean ± standard deviation (SD).

3. Result and discussion

3.1 Zn-WC Nanocomposite Microstructure

The microstructure of Zn-WC nanocomposites was shown in Fig. 2a-b with different magnifications. The bright phases correspond to WC, and the dark-phase regions are Zn. WC nanoparticles are observed with homogeneous dispersion with no severe sintering (Fig. 2b), because the ultrasound processing and reasonably good wettability between WC and molten Zn enabled nanoparticles self-dispersion [32], TEM image further reveals the homogeneous dispersion of WC nanoparticles, shown in Fig. 2c. The darker phase indicates the microstructure of the single crystal WC, as confirmed by selected area electron diffraction. WC obtains a hexagonal crystalline structure, where (111) plane and (201) plane are presented with an angle of 27° as indicated in Fig. 2d.

3.2 Microhardness of Zn-WC Nanocomposites

Vickers microhardness was determined for both pure Zn and Zn-WC nanocomposites with different nano-reinforcement concentration. The Vickers microhardness for pure Zn was 40.6 HV and 46.7, 50.6, 54.2 and 60.1 HV for 2.5, 5, 7.5 and 10 vol.% of Zn-WC nanocomposites (indicated as Zn-10WC), respectively (Fig. 3a). 10 vol.% WC nanoparticles enhanced the microhardness by 48%. This is primarily due to the Orowan strengthening that nanoparticles block the dislocation motion. The Vickers microhardness was measured after 14 days of immersion in the simulated body fluid (SBF) solution to evaluate mechanical integrity. No statistically significant change was detected in Fig. 3b.

3.3 Biodegradation of Zn-WC microwires

The results from the immersion test show that the number of Zn ions released from the Zn-WC nanocomposites are statistically the same regardless of the volume fraction of WC nanoparticles and was similar to that of pure Zn micro-wires based on one-way ANOVA (Fig. 3c). These results suggest that Zn retains the favorable degradation rate with the addition of WC nanoparticles. The degradation rate of the Zn-WC micro-wires was linear during the 14-day study length. The average corrosion rate of each sample per day was 0.25 ~ 0.4, 0.33 ~ 0.43, 0.31 ~ 0.33 and 0.25 ~ 0.26 pg/mm^2/day at days 1, 3, 7 and 14, respectively, shown in Table 2. These levels of Zn ion release are likely to be well tolerated *in vivo.* The National Academy of Medicine has set the recommended daily intake value of Zn at 2-3 mg/day for infants up to 8-11 mg/day for adults [33], and normal serum and urine levels in adults have been reported as 1 pg/mL and 0.5 mg/g creatinine, respectively. Therefore, the toxic potential of the daily dose of Zn released from a Zn-based implant should be negligible[21, 34], Additionally, no detectable levels of tungsten were released from Zn-WC nanocomposite microwires after 14 days of static immersion in SBF as measured by ICP-MS with a lower quantifiable limit of 0.5 ppb (0.5 ng/mL). Tungsten has historically been considered an inert metal; however, there are concerns that accumulation of tungsten within the bone may alter the bone biology as well as result in higher exposure levels within the bone marrow, which contains part of the developing immune system [35], However, relatively high doses of tungsten (15-500 ppm) are used in such toxicity studies [35-37], Taken altogether, these results indicate that WC remains reactively stable with

minimal toxic potential *in vivo* [30] and its impact on the corrosion rate of Zn-based implants is negligible.

Fig. 3d and 3e show the surface morphologies of the Zn-WC nanocomposite micro-wires before and after soaking in SBF for 14 days, presenting that the surface immersed in the SBF for 14 days was similar to that before testing. A large amount of salt precipitation forms a layer to cover the sample's surface. According to EDS results, it is reasonable to assume that the layer of biodegradation products may contain ZnO, Zn(OH)2, Zn3(PC>4)2, and Ca3(PC>4)2 based on the composition and insolubility of by-products in water and SBF.

4. Conclusion

In summary, ultrasound-assisted powder compaction was used to successfully fabricate Zn-WC nanocomposites. Zn - 10 vol.% WC gained a 48% increase in hardness, which did not change after 14 days of biodegradation testing. Evaluation of the biodegradation showed that the WC nanoparticles did not impact the release rate of Zn ions, and no detectable levels of tungsten ions were released from any of the nanocomposites. These results suggest the novel Zn-WC nanocomposites retain the favorable biodegradation profile of pure Zn necessary for bioabsorbable metallic implant applications while enhancing the mechanical properties. Further material characterization is required, including fatigue testing, electrochemical biocorrosion analysis, and both *in vitro* and *in vivo* biocompatibility testing to verify the suitability of nanocomposites for bioabsorbable metallic implant applications.

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Highlights:

- Zinc/Tungsten Carbide matrix nanocomposite is manufactured via ultrasound-assisted powder compaction.
- The hardness of Zn/Tungsten Carbide is 48% higher than pure zinc.
- The corrosion rate of Zn/Tungsten Carbide is statistically the same as pure zinc, with not toxic tungsten ion leaching.
- Zn/Tungsten carbide can be applied to bioabsorbable metallic implant.

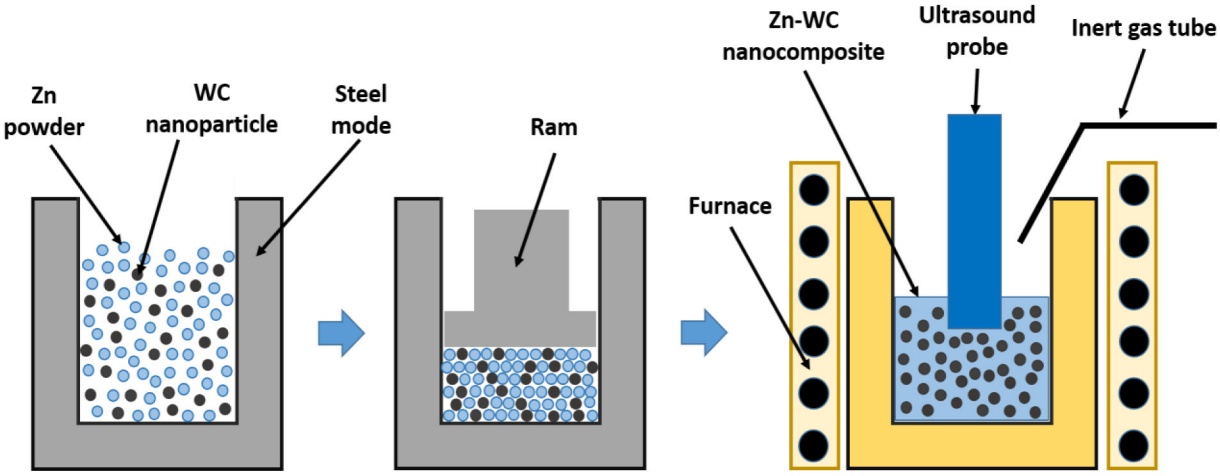

Figure 1.
Schematic of Zn-WC nanocomposite fabrication via ultrasound-assisted power compaction

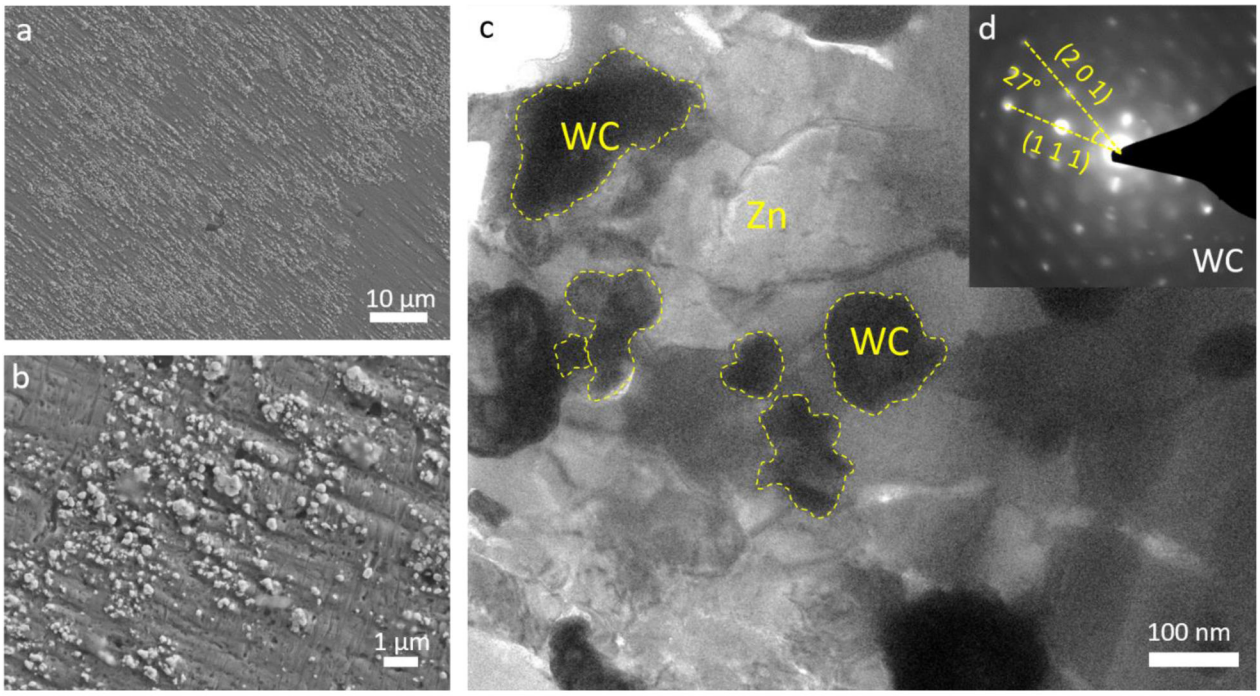

Figure 2.

(a) SEM images of WC nanoparticles homogeously dispersed in Zn matrix. (b) Magnified SEM image of dispersed WC nanoparticles of 150nm with no severe sintering. (c) TEM image of Zn-WC nanocomposite. (d) Diffraction patern of WC on the darker phase after fast Fourier tranformation.

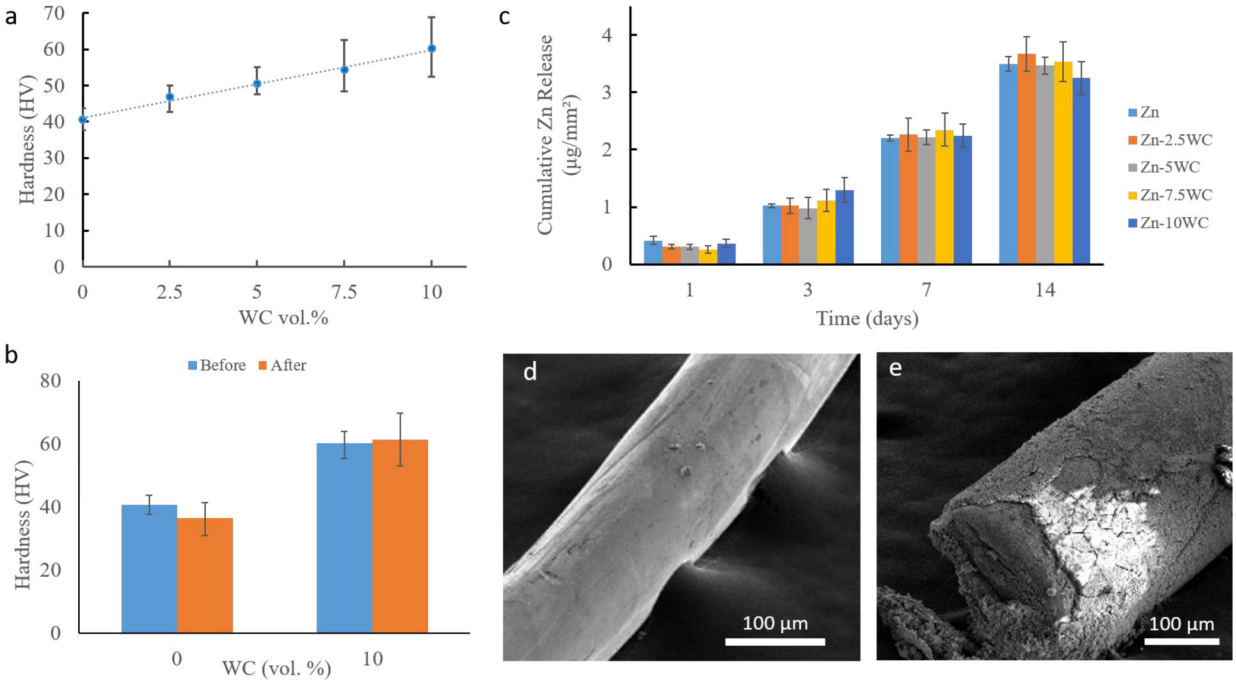

Figure 3.

Biodegradability of pure zinc and Zn-WC nanocomposite. (a) Vickers microhardness of Zn-WC nanocomposites vs. WC vol.% (b) Comparing the Vickers hardness of pure Zn and Zn-WC (10 vol. %) nanocomposites before and after 14 days of immersion in SBF. (c) Static immersion of Zn-WC micro-wires in SBF (n=3). No statistically significant difference ($p > 0.05$) was measured in Zn release between samples with increasing WC nanoparticle content based on one-way ANOVA. SEM images of Zn- WC nanocomposite micro-wire before (d) and after (e) immersion in SBF for 14 days.

Table 1.

Ion concentration and pH values of simulated body fluid versus human blood plasma

Ions	SBF (mM)	Human Blood Plasma (mM)
Na^+	142	142
K^+	5	5
Mg^{2+}	1.5	1.5
Ca^{2+}	2.5	2.5
Cl^-	147.8	103
HCO_3^-	4.2	27
HPO_4^{2-}	1	1
SO_4^{2-}	0.5	0.5
pH	7.4	7.4

Table 2.

Average cumulative Zn ion release at day 14 of the immersion test

	Zn	Zn-2.5WC	Zn-5WC	Zn-7.5WC	Zn-10WC
Zn ion release (gg/mm^2)	0.329±0.068	0.346±0.047	0.299±0.047	0.305±0.070	0.338±0.090
Significance level P	N/A	0.49	0.21	0.39	0.78