

# The evaluation of W/ZrC composite fabricated through reaction sintering of two precursors: Conventional $\text{ZrO}_2/\text{WC}$ and novel $\text{ZrSiO}_4/\text{WC}$

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## ABSTRACT

In this study the W–ZrC composites fabricated by in situ reaction sintering of two precursors were compared, 1-The conventional WC and  $\text{ZrO}_2$  which are ball milled with established molar ratio of 3–1 for 12 hours, gelcasted to form a green body and then undergo a pressure less sintering cycle, 2-A new and innovative way in which for the first time  $\text{ZrSiO}_4$  was used instead of  $\text{ZrO}_2$ , and by testing different molar ratio between WC and  $\text{ZrSiO}_4$  it was understood that the optimum ratio is 3–1 once again. Furthermore the starting  $\text{ZrO}_2$  and  $\text{ZrSiO}_4$  powder were selected in nano size and it was understood that by using nano powders the amount of unreacted and unwanted phase reduce, the reaction progress and the mechanical proprieties improve. Although the reaction sintered WC/ $\text{ZrO}_2$  possess better properties, regarding the cost considerations, reaction sintering of WC/ $\text{ZrSiO}_4$  is a much cheaper process.

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## 1. Introduction

Composites comprised of a refractory metal as matrix and carbides as the reinforcement have interesting combination of chemical, thermal and mechanical properties. For instance W and ZrC often coupled to make composite because ZrC is compatible with tungsten from several points of view [1]. Some of the properties of W and ZrC which make them suitable to make composite is presented in Table 1. Besides they exhibit little mutual solid solubility at high temperature (less than 7 mol % at 2800 °C) and do not react to form other compounds [2–4].

ZrC/W composite possess higher wear resistance, creep resistance and hardness rather than pure refractory metals and shows better fracture toughness ( $K_{IC}$ ), which demonstrates higher resistance than pure carbides. As a result this composite is used in many industries like aerospace, aircraft, automotive, energy, material processing and defense industry. Also is used in many applications such as rocket nozzles, leading edges, combustion chambers and exhaust flaps [1,2].

Three ways of production have been reported for fabrication of W/ZrC composites. 1-DCP (Displacive compensation of porosity), a porous WC preforms will be exposed to molten  $\text{Zr}_2\text{Cu}$  at 1200–1300 °C and ambient pressure, then the  $\text{Zr}_2\text{Cu}$  liquid infiltrate into it and ZrC and W will form in conclusion [1–5]. 2-Hot press, the elemental ZrC and W powders are mixed and then will be subjected to heat and pressure at the same time [6–15]. 3-Reaction sintering,  $\text{ZrO}_2$  and WC powders in designed volume or mass fraction are subjected to high temperature and after that will be formed into a green body

which then is heated in an inert gas atmosphere to produce ZrC and W [16,17]. Also in a new way, deployed by the authors, the  $\text{ZrSiO}_4$  has been used instead of  $\text{ZrO}_2$  to make the composite through reaction sintering, because  $\text{ZrSiO}_4$  is more abundant and much cheaper than  $\text{ZrO}_2$ , so by using  $\text{ZrSiO}_4$  the cost of production decrease considerably.

In this study at first the W–ZrC composite is fabricated by reaction sintering of micron and nano WC/ $\text{ZrO}_2$ , and then fabricated through reaction sintering of  $\text{ZrSiO}_4/\text{WC}$ , in which the powders are blended with different molar ratio to understand the optimum ratio and are selected in micron and nano size to see the differences between the resultant composites. The volume fraction of different phases in the composites is approximately estimated by the ratio of the integrated area of the peaks and the total XRD patterns to see the efficiency of process [19]. The density are determined using Archimedes method, according to ASTM B311 [20] standard method. The hardness is evaluated in accordance with ASTM E10 [21]. Moreover the bending strength of the composite has been measured by bending test according to ASTM C1161 [22] and the Modulus of Elasticity is measured as well, by ASTM C1419 [23] test standard method. Also by secondary electron microscopy (SEM) and back scattered electron (BSE) the morphology of samples are studied.

## 2. Experimental

To produce W–ZrC composite through reaction sintering, a preform, conventionally made from the mixture of  $\text{ZrO}_2$  and WC is sintered, but here for the first time  $\text{ZrSiO}_4$  was used instead of  $\text{ZrO}_2$  to fabricate the composite. The preform can be made by any conventional ceramic forming method, like normal mechanical pressing or isostatic pressing, however for the first time a low-toxic gel casting system is used to form

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**Table 1**  
Properties of W and ZrC [2–4].

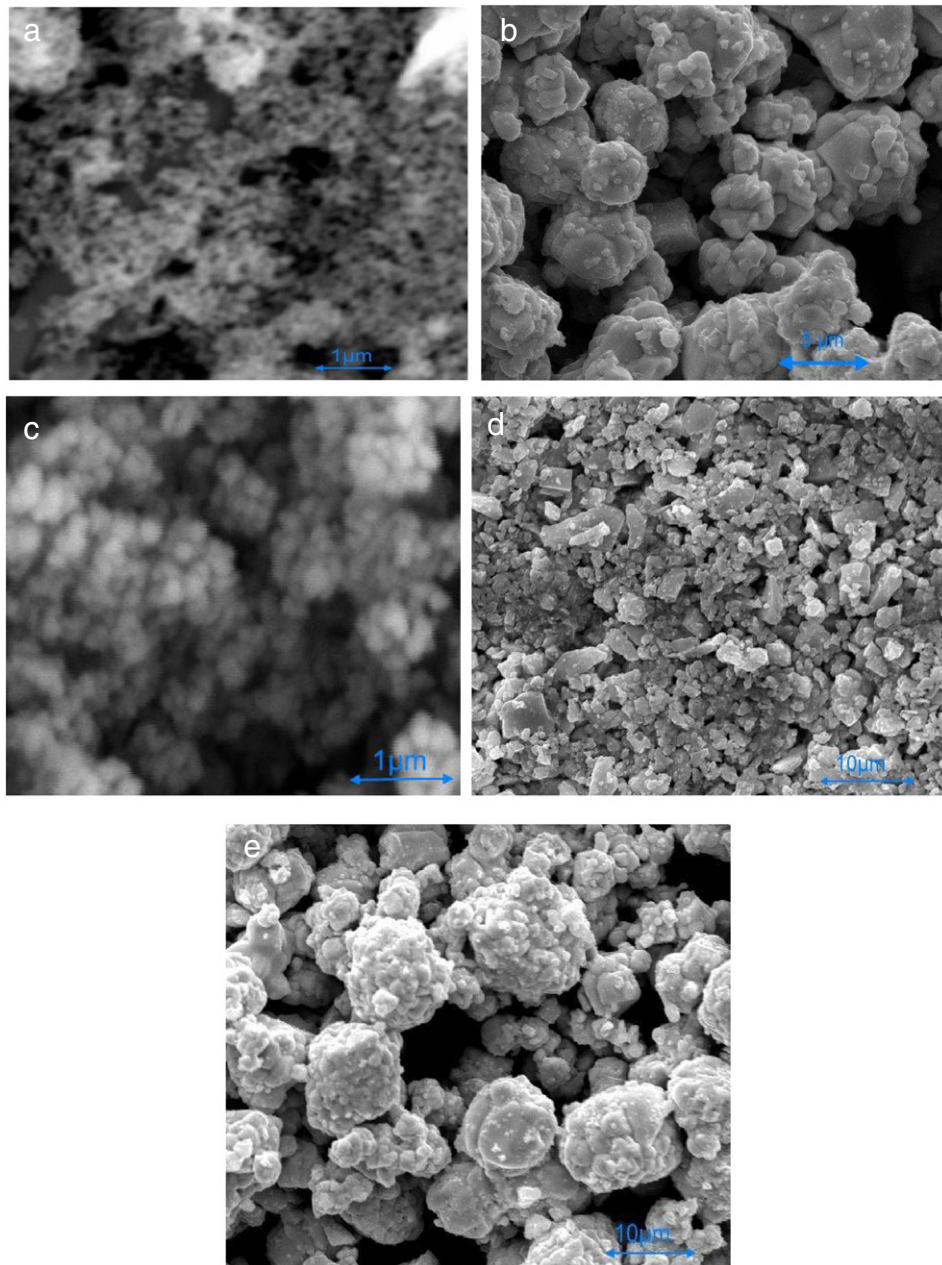
	Melting point (°C)	Linear expansion coefficient (/°C)	Thermal conductivity (W/m-k)	Density (g/cm <sup>3</sup> )
W	3422	$4.5 \times 10^{-6}$	105	19.3
ZrC	3420	$4 \times 10^{-6}$	40	6.63

the green body made from WC/ZrO<sub>2</sub> or WC/ZrSiO<sub>4</sub>, to evaluate the process and use the results for next studies. As a consequence the process is studied for two different precursors.

### 2.1. Reaction sintering of WC and ZrO<sub>2</sub>

ZrO<sub>2</sub> and WC powder are mixed at presence of a binder so that a precursor is formed. According to the results obtained in a study [18]

the optimum ratio between WC and ZrO<sub>2</sub> powder is 3–1, so to produce a work piece weighing 150 g with molar ratio of 3–1, 126gr WC and 26gr ZrO<sub>2</sub> powders are mixed, however regarding the desired proportion of W to ZrC in composite this ratio may be different. Mixing is conducted in alcohol media and then after 6 hours mixing, the resulting mixture is dried in an oven for 12 hours. Finally this mixture is gelcasted to form a green body, in which sodium alginate (C<sub>6</sub>H<sub>7</sub>NaO<sub>6</sub>), a natural polymer is used as monomer [24–26]. Hence at first 19.41 wt% of ceramic powder (here is 150 g WC/ZrO<sub>2</sub>) distilled water and 0.34 wt% of ceramic powder, sodium alginate are mixed for at least half an hour. After this period 0.11 wt% of ceramic powder, hexametaphosphate ((NaPO<sub>3</sub>)<sub>6</sub>) is added as collator and mixing continues for more 10 minutes. Then 0.21 wt% of ceramic powder, diphosphatecalcium (Ca<sub>3</sub> (PO<sub>4</sub>)<sub>2</sub>) is added to the mixture and mixing continues for another 10 minutes. Finally 0.19 wt% of ceramic powder, ammonium citrate ((NH<sub>4</sub>)<sub>3</sub>C<sub>6</sub>H<sub>5</sub>O<sub>7</sub>) is added as dispersant in the same way and mixing continues 10 minutes. At this stage the WC and ZrSiO<sub>4</sub> powders are added to the slurry slowly and finally the initiator,



**Fig. 1.** The morphology and particle size of a) nano ZrO<sub>2</sub>, b) micron ZrO<sub>2</sub> c) nano ZrSiO<sub>4</sub>, d) micron ZrSiO<sub>4</sub> and e) WC powder.

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