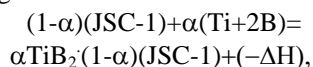


**NANOENERGETIC FABRICATION OF DENSE CERAMICS FOR LUNAR EXPLORATION PROGRAM.** K. S. Martirosyan<sup>1</sup> and D. Luss<sup>2</sup>, Department of Chemical and Biomolecular Engineering, University of Houston, 4800 Calhoun, Houston, Texas, 77204, (<sup>1</sup>kmartirossian@uh.edu, <sup>2</sup>dluss@uh.edu).

**Introduction:** As major space agencies from US, Europe, Russia, and China turn their exploration ambitions towards the Moon, the research and development of new technologies for lunar operations require meeting a fast pace schedule, reminiscent of the 1960's Apollo program [1]. Fabrication of structural and refractory materials from *in-situ* lunar resources (either metals, ceramics, or composite-based materials) will be essential for enabling extended human presence on the Moon.

Recently we have proposed a novel process for synthesis of lunar composites [2] that allows fabrication of dense ceramic composites with signify a minimal external energy input. This process uses the principles of *Self-propagating High-temperature Synthesis* (SHS) [3] that produces a controllable morphology of synthesized products while decreasing both power consumption and cost of high-temperature equipment. We used this process to produce dense ceramic composites from lunar soil simulant (JSC-1) [4], which is derived from volcanic ash of basaltic composition by the following chemical reactions:



where  $\alpha$  is a parameter that controls the exothermicity of the reaction. At  $\alpha=1$  the heat of the reaction,  $(-\Delta H)$  is  $-278.2$  kJ/mol and the adiabatic temperature rise  $T_{ad}=3063$  K. The temperature during the combustion synthesis of  $\text{TiB}_2$  diluted by JSC-1 (up to  $1500$  °C) may cause partial melting and sintering of the lunar soil powder. The combustion needs to be conducted in vacuum which is the *lunar environment*. In addition, the reaction time is short, order of seconds.

We describe here a study of the use of nanoenergetic system such as titanium and boron nano particles to fabricate dense ceramic materials for Lunar Exploration Program application.

**Nanoenergetic systems:** Nanostructured high-exothermic materials, referred to as *Nanoenergetic Materials* (NM) or *Metastable Intermolecular Composites* (MIC), release energy much faster than conventional energetic materials [5,6]. They have various potential military applications and are likely to become the next-generation explosive and propulsion materials. The size reduction of the reactant powders from micro- to nano-size generates a more intimate contact between the particles [6]. Since the heat of combustion is independent of particle size, nano-powders therefore suffer from poorer specific energy densities. This, in turn, improved the combustion kinetics compared to micron-sized particles and increases the reaction front

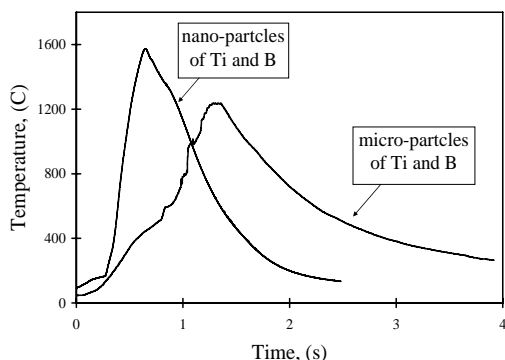
propagation velocity. Thus, using nanoscale precursors as the combustion source may be beneficial for fabrication of structural and refractory dense ceramics for various applications by using lunar regolith.

**Experimental:** The green mixture contained up to 60 wt. % of titanium and boron nanoparticles ( $\sim 60$ - $100$  nm) and non-combustible lunar soil simulant JSC-1 with particle size  $\sim 1$   $\mu\text{m}$ . All the reactants were thoroughly mixed by rotary milling in hexane. Dried mixture was pressed into cylindrical samples (diameter 12 mm and length 15 mm) with porosity  $\sim 35$  %. The combustion synthesis was conducted inside a cylindrical stainless steel vessel (70- mm i.d. and 60- mm length) fed by argon at a flow rate up to 100 cc/min. Local ignition of the reactant mixture by an electrically heating coil generated a propagating temperature front. An infrared-transparent sapphire window on top of the vessel enabled viewing and recording of the sample surface temperature at a frequency of 60 frames/s by an infrared camera (Merlin, Indigo Systems). The digital IR images determined the temperature-time history, shape and average velocity of the propagating combustion front. Image analysis software (ThermaGRAM) was used to determine the instantaneous and average velocity of the moving thermal front and temporal changes in the shape of the temperature front. The local combustion temperature ( $T_c$ ) inside the reactants mixture was measured by an S-type (Pt-Rh) thermocouple of about 0.1 mm diameter inserted in the center of sample. The thermocouple readings were recorded and processed by an Omega data acquisition board connected to a PC.

The composition and crystal structure of the products was determined by X-ray diffraction (Siemens D5000 diffractometer) with  $\text{Cu K}\alpha$  radiation ( $\lambda=1.54056$  Å). The morphological features and microprobe analysis were determined by scanning electron microscopy (SEM; JEOL JAX8600, Japan) of sample cross sections.

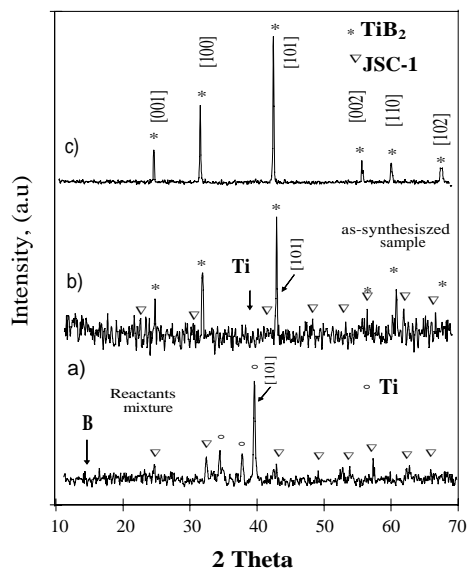
**Results and Discussion:** The ignition of the samples containing nano precursors was rather easy and a self-sustained combustion wave propagated through the sample with an average velocity of  $\sim 8$  mm/s, about twice faster than for combustion using micro Ti and B particles. To compare the maximum combustion temperature using either nano or micro reactants we prepared samples with same ratio of  $\text{TiB}_2/\text{JSC-1}$  but different Ti and B particles sizes. A typical temperature profiles for the nano and micro systems are shown in Fig. 1. The smaller (nano) size of Ti particles generated a higher combustion temperature

than using micro particle size. This enables to use a smaller amounts of Ti and B particles to generate self-sustaining reaction and fabricate matrix ceramics composites.



**Fig. 1.** The local temporal temperature generated during the combustion synthesis of micro and nanostructured  $\text{TiB}_2$  and (JSC-1) composites.

Fig. 2 shows the XRD patterns for three different samples: reactants mixture (before combustion), as-synthesized nano $\text{TiB}_2$ /(JSC-1) matrix composites and pure  $\text{TiB}_2$  obtained from titanium and boron nano powders. X-ray indicates that the combustion led to an almost complete conversion of Ti and boron to the  $\text{TiB}_2$  for the both cases ( $\text{TiB}_2$  diluted with JSC-1 and for pure  $\text{TiB}_2$ ).

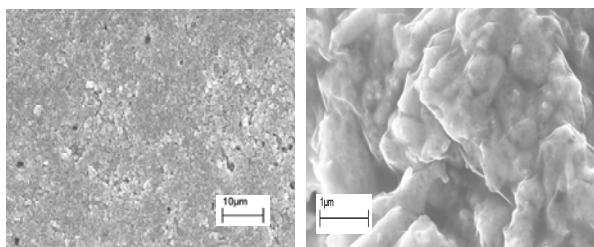


**Fig. 2.** X-ray powder diffraction patterns of (a) reactants mixture, (b) as-synthesized nano $\text{TiB}_2$ /(JSC-1) and (c) pure  $\text{TiB}_2$  composites.

The major reflection peaks in the XRD patterns  $2\theta=44.49^\circ$  and  $34.15^\circ$  are related to titanium diboride with hexagonal structure (space group  $P6/mmm$ ). The calculated lattice parameters are  $a=3.029 \text{ \AA}$ ,  $c=3.229 \text{ \AA}$  that is in a close agreement with the JCPDS diffraction data, (PDF card 85-2083).

Using conventional XRD it is difficult to determine the glass or amorphous phases of composites. A microprobe (EDX) analysis was conducted to determine the distribution of the elements in the products. The results for 60% $\text{TiB}_2$ /JSC-1 showed that the elements were distributed as following mass percent: Ti- 39.67 %; B-16.43%, Si- 11.65%, Al-3.89, Fe-1.73 %; Ca- 3.59%, Mg- 3.52% and oxygen-19.52 %.

The scanning electron micrograph of the combusted nano  $\text{TiB}_2$ /(JSC-1) sample (Fig. 3) shows microstructure with grains size  $\sim 1 \mu\text{m}$  indicating that the combustion temperature was insufficient for the complete formation of the ceramic structure with partial melting of JSC-1 components.



**Fig.3.** SEM micrograph of nano  $\text{TiB}_2$ /(JSC-1) sample after combustion with different magnifications.

Our study demonstrate the feasibility of producing dense ceramics composites from lunar soil simulant by using high exothermic nanoenergetic systems.

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**References:** [1] Sibille L. (2005) Lunar regolith simulant materials workshop, p.1, Marshall Space Flight Center. [2] Martirosyan K.S. and Luss D. (2006), *LPS XXVII*, Abstract #1896. [3] Merzhanov A.G. (2004), *J. Mater. Chem.*, 14, 1779-1786. [4] McKay D.S., Carter, J.L. Boles, W.W. Allen C.C. and Allton J.H. (1997) “JSC-1: A new Lunar Regolith Simulant”, *LPS XXIV*, Abstract #963. [5] Miziolek A. (2002) *AMPTIAC Newsletter, Spring*, 6, 1, 43. [6] Dlott D.D. (2006) *Mat. Sci. and Tech.*, 22, 4, 463-473.