COMBUSTION SYNTHESIS OF CERAMIC COMPOSITES FROM LUNAR SOIL SIMULANT.

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Introduction: The extended human presence on the Moon will enable astronauts to develop new technologies and harness the Moon's abundant resources to allow manned exploration of more challenging environments [1]. Various approaches to establish permanent outposts on the Moon are being considered by major space agencies and construction companies from USA, Europe, Russia and China. Construction materials for such applications must be dense and strong, should be produced mostly from indigenous sources and able to protect crew members and equipment from the effects of radiation, meteoroid bombardment, and temperature extremes. Proposed materials for planetary outpost construction include concrete (cement or sulfur-based), sintered regolith, cast basalt ceramics, ice and Fe or Ti metals [2]. Construction of even simple infrastructures on planetary surfaces can greatly help to optimize operations and research activities, as well as the development of new missions through *in-situ* resource utilization. Lunar regolith is an easily accessible resource that consists mainly of silicon dioxide (47.3 wt %), aluminum oxide (17.8 wt %), iron oxide (10.5 wt %), calcium oxide (11.4 wt %), magnesium oxide (9.6 wt %) and others [3]. The lunar soil is very fine sand with particle size of ~100 μm [4].

The proposed method for synthesizing of lunar construction materials is calcination of a compacted lunar soil in a microwave furnace at temperature of up to 1500 °C for periods of about 2 h [5, 6]. An attractive advantage of this approach compared to others is the ability to utilize lunar soil to produce large, strong, crack-free sintered bricks. However, this process requires high energy consumption and use of complicated high-temperature equipments. In addition, long time calcination of lunar soil generates large particle agglomerates and hence reduces the degree of microstructural homogeneity lowering material mechanical properties. The process we propose overcomes some of the major disadvantages of that process.

Novel approach: In this work, we are demonstrating the novel process for rapid production of dense ceramic composites from lunar soil simulant by using Self-propagating High-temperature Synthesis (SHS). SHS also referred to as combustion synthesis provided controllable morphology of synthesized products and decreased both power consumption and cost of high-temperature equipment. SHS has been successfully applied to the synthesis of a large variety of refractory and advanced ceramics. [7, 8]. In SHS the reactants to products conversion is accompanied by a large heat release 10^{12}-10^{14} W/m^3. Under such conditions, a high-temperature combustion wave propagates through the reactants mixture, without requiring any additional energy supply. High temperatures, up to 4000 K, are reached at the combustion front, which propagates at a velocity of up to 25 cm/s. SHS processes are characterized by very high temperature gradients (up to 10^5 K/cm), and short reaction time (order of seconds). As a result, chemical conversion takes place under non-equilibrium conditions which favor the formation of metastable phases and unusual microstructures not attainable with conventional calcination in furnaces. Moreover, the volatile impurities adsorbed on reactant powders are removed during the high-temperature reaction, allowing for the synthesis of high purity materials. Major advantages of this process are the very low power consumption and use of simple equipment.

Experimental: Our propose process for production of dense ceramic composites from lunar soil simulant (JSC-1) consists the following chemical reactions:

$$\begin{aligned}&(1-\alpha)(\mathrm{JSC\text{-}1}) + \alpha(\mathrm{Ti} + 2\mathrm{B}) \rightarrow \\ &\quad \alpha \mathrm{TiB_2} \cdot (1-\alpha)(\mathrm{JSC\text{-}1}) + (-\Delta H) \qquad (1)\end{aligned}$$

where α is a parameter that controls the exothermicity of the reaction; (-ΔH) is a heat of the reaction, at $\alpha=1 \Rightarrow (-\Delta H)=-278.2$ kJ/mol; JSC-1 is derived from volcanic ash of basaltic composition. The simulant's chemical composition, mineralogy, particle size distribution, specific gravity, angle of internal friction, and cohesion have been characterized and fall within the ranges of lunar soil samples [9].

The powders of titanium, boron (Aldrich 99+ %) and (JSC-1) were thoroughly mixed by ball milling of about 1 h. The mixture was pressed to obtained cylindrical sample (height 2 cm, diameter 1.2 cm) with initial relative densities ~0.7. The sample was placed inside a steel cylindrical vessel having an infrared-transparent sapphire window. The sample was locally ignited in vacuum by an electrically heated coil. An S-type (Pt-Rh) thermocouple of about 0.1 mm diameter was installed in the center of sample and was measured the inner local temperature. An infrared camera (Merlin, MW18-Indigo Systems, 60 frame/s) was used to determine the surface 2-D temperature distribution, front shape, and average combustion front velocity. The composition and crystal structure of the products was determined by X-ray diffraction (Siemens D5000 diffractometer). The product morphology were observed with optical and a scanning electron microscopy SEM, (JEOL, JAX8600).

Results and Discussion: The evolution and stability of self- propagating temperature waves in solid state reactions are dictated by the thermodynamic and kinetic properties of the combustion process. The estimation of the adiabatic temperature (T_{ad}) of the reaction (1) by minimizing the thermodynamic free energy (assuming that JSC-1 consist only SiO_2) shows that at $\alpha > 0.25$ self-sustaining processes can occur. The maximum adiabatic temperature in system (1) achieved at $\alpha=1$ with $T_{ad}=2892$ K.

Typical IR thermal images of the surface temperature of sample during the combustion synthesis of TiB_2-60wt.%(JSC-1) composite are shown in Figure 1. They show that the moving temperature front propagated layer-by-layer mode with an average velocity of about 4 mm/s. The temperature front propagated trough sample about 5 s. Thermocouple measurement revealed that the maximum combustion temperature at the center of the sample was about 1400 °C. The temperature rises drastically in a short time with the average rate of temperature rise of ~1000 °C/s. After combustion the sample were cooled down to room temperature.

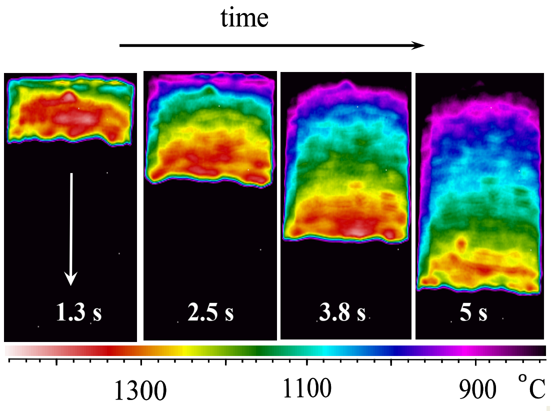

Fig. 1. (color) IR thermal images obtained during the combustion synthesis of TiB_2-60%(JSC-1) composite. The combustion front propagated from top to bottom.

The XRD patterns of the synthesized TiB_2-60wt.% (JSC-1) composites indicate that the combustion led to an almost complete conversion of Ti and boron to the product. The biggest diffraction picks were observed at $2\theta=44.46°$ and 34.15° corresponding to interplanar spacing of 2.036 Å (101) and 2.623 Å (100) of TiB_2. The presence of the flat pattern background and numerous sharp diffraction picks indicate that the product is mainly crystalline. The scanning electron micrograph of the sample (Fig. 2) show microstructure with fairly small grains and open porosity indicating that the combustion temperature was insufficient for the complete formation of the product structure with grain and pore size less than 100 μm. The Vickers hardness (under a 300 g indentation load) of the synthesized composites was around 500 kgf/mm^2.

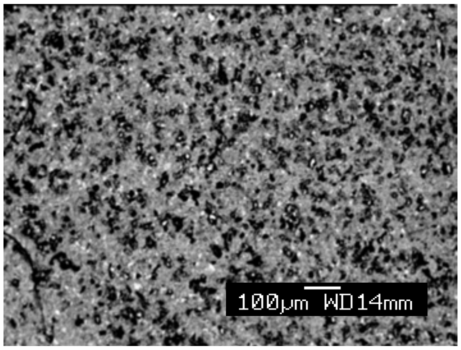

Fig. 2. Scanning electron micrograph of TiB_2-60wt.% (JSC-1) composite.

The results of our experiments on the combustion of TiB_2/(JSC-1) composites lead to the following conclusions concerning the potential synthesis of “lunar bricks” (i) The combustion temperature is sufficiently high to cause partial melting and sintering of the lunar soil powder; (ii) The combustion can be readily ignited by an electrically heated coil; (iii) The reaction time is very short (order of seconds); (iv) The combustion product is solid and hard, scratched glass; (v) The specific combustion needs to be conducted in vacuum which is the lunar environment.

Acknowledgments: We wish to acknowledge the financial support of this research by Texas Center for Superconductivity and Advanced Materials, and the Materials Research Science and Engineering Center at the University of Houston.

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