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Chapter 2

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Titanium carbide and its composites with aluminum oxide are synthesized by self-propagating high temperature synthesis (SHS).

2.1. Raw Materials

The raw materials used in this study were powders of titanium dioxide (98% purity, <1 μm), aluminum metal (99.5% purity, ≤93 μm) and graphite (~78 μm). Pure argon gas was also employed as an inert atmosphere.

2.2. Synthesis Procedure

2.2.1. Mixing

For producing titanium carbide/aluminum oxide composites the raw materials TiO_2, Al and C powders were mixed in the stoichiometric molar ratio of 3: 4: 3 respectively. In some experiments the ratio of Al was increased to produce $3TiC /2Al_2O_3 /xAl$ composites, where; $0 \leq x \leq 6$. The starting materials were blended in a slow rotating mill with alumina balls for 24 hrs using ethanol as a mixing medium to ensure high homogeneity. Ethanol is chosen because of its density (0.8 g/cm^3) is lower than that of all the mixed powders specially the graphite (2.2 g/cm^3) [119]. After completion of mixing process, the obtained slurry was dried at 105 °C over night.

2.2.2. Briquetting:

The mixed powders were then pressed into cylindrical shape of ~2.0 cm long, 1.2 cm diameter using a die steel where the pressure was applied from both sides of the sample by hydraulic press and was maintained for one minute to achieve most possible homogeneity and nearly complete distribution of the pressure throughout the compact mass.

The applied pressure was adjusted to produce green compacts from 53 to 55 % relative density.

2.2.3. Combustion synthesis reactor

A special reactor was used in order to carry out and monitor SHS reactions; Fig. 6 [120] visualizes of cylindrical body containing a glass window in order to observe the combustion process. Two movable electrodes are fastened on a cylindrical base carrying between them the heating element. The compact which is movable was put under the heating element. In some experiments, the cylindrical wire of coil surrounded the compact and acts as a heater was connected to AC power source in order to elevate the temperature of the compact using its internal resistance. The ignition was carried out using a thin graphite electrode one mm above the compact. The electricity was supplied from an AC power source (30-40 A and 17 V).

The temperature profiles as well as wave velocities were recorded using two thermocouples of type C (tungsten 5% rhenium against tungsten 26 % rhenium, Nilaco Corporation, Japan) of 0.2 mm thickness with bead size of approximately 0.4 mm. The thermocouples were inserted through very narrow holes drilled in the side of the compacted sample where they were positioned at 10 mm far from each other and were forced against the compact to ensure good contact, Fig. 7. Data acquisition card (NI-DAQ 6023 E series, National Instruments Co, USA) was used to acquire the output mV from the two W-Re thermocouples. The data acquisition system was configured to collect data at a rate of a sample each 11.1 millisecond for each channel. By recording the time involved for the reaction front to propagate from the upper thermocouple to the lower one and by knowing the distance between the two thermocouples the combustion wave velocities were calculated from the
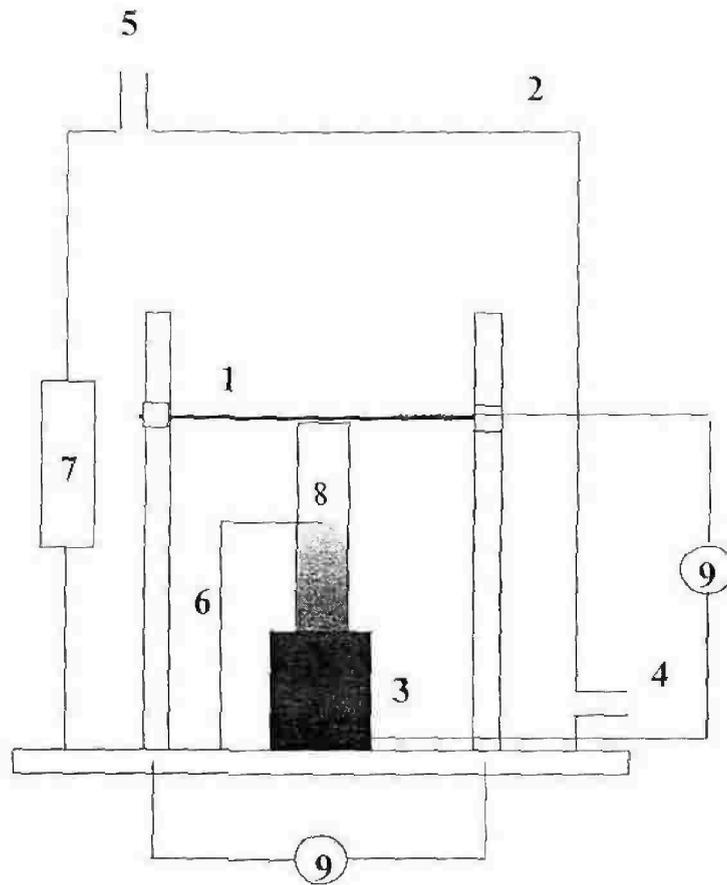
Fig. 6 SHS reactor:

1 Movable heating element

2 Cover

3 Movable graphite plate

4 Gas inlet

5 Gas outlet

6 Type C thermocouple

7 Window

8 Sample
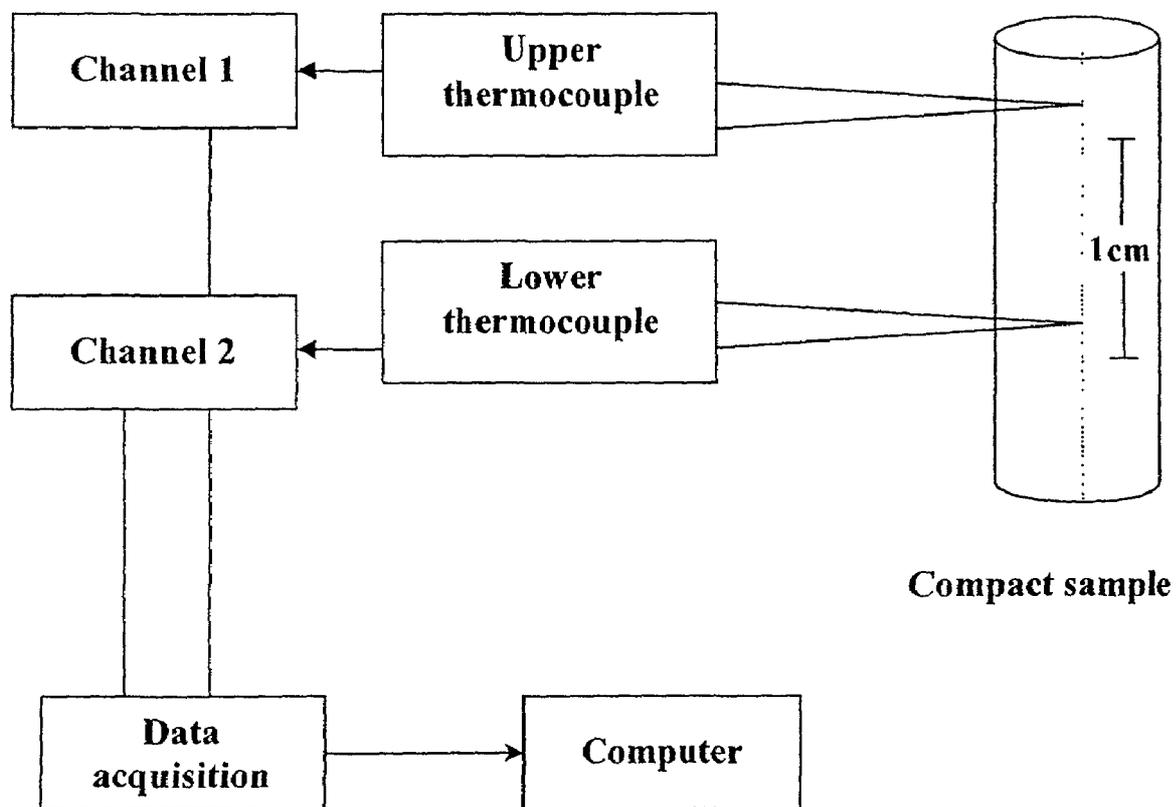
9 AC power supply

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Fig. 7 Schematic representation of SHS reaction monitoring

simple equation: u = r / t where, u is the wave velocity, r is the distance between the two thermocouples, and t is the time.

Some experiments were conducted at room temperature, in some other experiments, the initial temperature of the compacts was increased up to 600°C by using a Nickel Chrome coil surrounded the compact.

2.2.4. Synthesis

The compact was tightly fixed under the heating element and the two thermocouples were inserted firmly in their narrow holes. The reactor was then evacuated and backfilled with pure argon at one atmosphere. If required the initial temperature of the compact was raised as described before and the temperature of the ignition element was subsequently increased suddenly to its possible maximum one for several seconds using external power source. Once the ignition has been accomplished, the igniter was switched off while the wave continues its propagation to the bottom end of the compact. Switching of the igniter is very important step otherwise the excess energy input into the lower sample should be accompanied by fast unsteady propagation of the SHS wave. The product was removed from the reactor after cooling to the room temperature. Density and porosity of the obtained reaction products were measured by the water immersion technique (Archimedean method). Also, optical microscopic as well as scanning electron microscopic examinations were conducted to the products. The reaction products were then crushed in agate mortar and subjected to x-ray analysis to identify the different formed phases.

2.3. Characterization of the Products

2.3.1. X-ray diffraction analysis

Different phases of the synthesized products and sintered bodies were identified by X-ray diffraction analysis using Cu radiation (λ = 1.5418 A°). The XRD analysis was carried out with the aid of Philips

Diffractometer of type PW 1370 operated at 30 kV and 10 mA with scanning speed of $2°min^{-1}$. Phase identification of the examined samples was carried out using ASTM cards.

2.3.2. Metallographic examination

Microscopic examination of some of the combustion products was carried out in order to identify the structure of the different phase's present using optical microscope. The selected samples were impregnated with an epoxy resin. Then samples were cured at 70 °C in an oven over night. The cured samples were ground on various grades of 220-1000 mesh silicon carbide paper on a wet grinding machine. Finally, polishing was done on a slow moving disc with alumina paste (10 nm) on micro-polishing cloth. After polishing, the sample was washed with ethanol and investigated with the aid of metallurgical research microscope (model Metallux II Leitz).

2.3.3. Scanning electron microscope investigation

The surface morphology and the microstructure of the obtained products and the sintered bodies were observed using conventional scanning electron microscope ([SEM], JEOL JSM-5410 [EDX], JED-2140 at 20 kV and 0.02 mm step). The sample is coated with thin layer of gold.

2.3.4. Determination of density and porosity by Archimedean method

The sample was boiled in distilled water for two hours to insert the water inside the porous and was cooled to room temperature. Then, it was weighed in water, in air (as a wet sample) and after drying to constant weight at 105 °C. The bulk and apparent densities were calculated as follows:

Bulk density $= d_w \times w_1 / w_3-w_2$

Apparent density $= d_w \times w_1 / w_1-w_2$

Where, w_1 is the weight of the dry specimen in air

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, w_2 is the weight of the specimen in water and it equals to

$(w_2+w_c)-w_c$

, w_c is weight of the cage in water

, w_3 is the weight of the wet specimen in air

, d_w is the density of water

The open porosity of the specimen can be also calculated from the same weights as follows:

Open porosity = w_3-w_1/w_3-w_2

When theoretical density is known the relative density is deduced as follows:

Relative density = Bulk density × 100/Theoretical density

2.3.5 Oxidation behavior

The oxidations of some selected samples were investigated in a muffle furnace up to 800°C for three hours in air. The samples were separately put in platinum crucibles. When the required temperature of the furnace was achieved the compact was hanged in the bottom of the crucible and suspended at the hot zone of the furnace.