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Experimental set up for characterization of carbide-based materials in propulsion environment

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Abstract

This paper summarizes our achievements in the development of an experimental set up for the characterization of ultra-high-temperature carbide-based material ceramics under conditions representative of a propulsion environment. A segmented converging diverging (de Laval) nozzle was manufactured and tested in a lab-scaled hybrid rocket engine. The converging and diverging sections are manufactured from high temperature tolerant graphite. A straight section of the graphite nozzle throat was replaced with a low-eroding tantalum carbide composite. The experimental setup enables to carry test with different combination of solid propellants and gas oxidizers. The purpose of this study was to prepare and test, under typical operating conditions in the hybrid rocket engine, i.e. very high temperature, high oxygen partial pressure and total pressure, a nozzle throat insert of a Tantalum Carbide-based composite, fabricated with hot-pressing technique and characterized in terms of strength and fracture toughness, thermal shock, high temperature oxidation behavior.

Keywords: Ceramic materials; Propulsion environment; Hybrid rocket

1. Introduction

The thermal, chemical, and mechanical environments typical of aero-propulsion applications, such as those characteristic of combustion chambers or of high performance rocket nozzles introduce many problems from the point of view of materials. These typical environments are characterized by highly corrosive atmospheres that may also contain metal additives, with typical flame temperatures even higher than 3000 °C. Next generation propellants have become more energetic in order to impart a higher specific impulse to the system, resulting in higher temperatures and pressures that need to be contained. These propellants produce very hostile, abrasive environments; existing materials for boost throat applications have been shown to erode at unacceptable rates, leading to a loss in performance due to throat widening. Implementation of these propellants for boost and thrust applications requires the development of a new family of materials providing structural integrity, thermal protection, and low- or near-zero ablation rates above 3000 °C. Erosion resistant nozzles that can maintain dimensional stability during firing are required. The materials used for these applications include refractory metals, refractory-metal carbides, graphite, ceramics and fiber-reinforced plastics.[1,2] Certain classes of materials demonstrated superior performances under specific operating conditions but the choice depends on the specific application. For instance, fully densified refractory-metal nozzles generally are more resistant to erosion and thermal-stress cracking than the other materials. Graphite performs well with the least oxidizing propellant but generally is eroded severely. Some of the refractory-metal carbide nozzles show outstanding erosion resistance, comparable to that of the best refractory-metal materials, but generally suffer due to fractures induced by thermal stresses.

The interaction of environmental conditions together with the usual requirement that dimensional stability in the nozzle throat can be maintained makes the selection of suitable rocket nozzle materials extremely difficult. This work is focused on the development and testing of monolithic composites based on carbides

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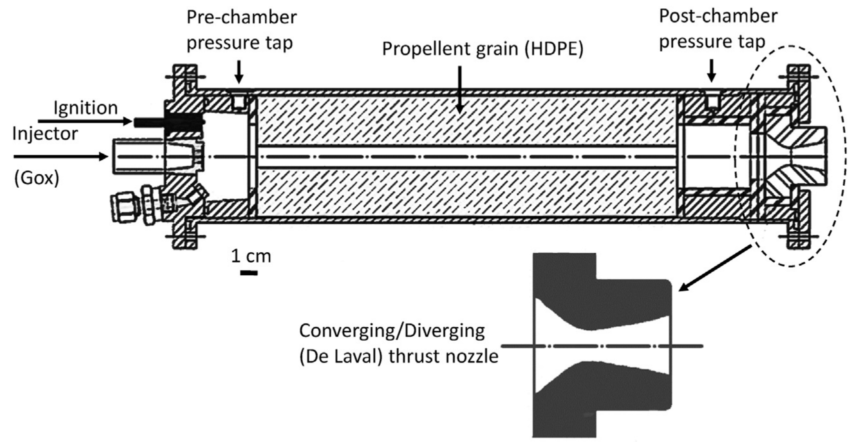

Fig. 1. Layout of the hybrid rocket engine. HDPE: High Density Polyethlene fuel, Gox: Gaseous Oxygen.

of early transition metals, in particular tantalum carbide.[3–5] Ultra High Temperature Ceramics (UHTC) such as Tantalum Carbide (TaC) and Hafnium Carbide (HfC) are very good potential candidate materials for use in propulsive systems. These compounds possess an excellent combination of properties including extremely high melting point (3950 °C and 3928 °C, respectively), high electrical and thermal conductivity, good thermal shock resistance and superior ablation resistance compared to C/C composites. For the above reasons they have been already identified as excellent candidates for aerospace applications, including also the possibility to develop thermal protection systems for hypersonic atmospheric re-entry conditions, together with other ultra-refractory ceramic composites.[6–10]

The long-term purpose of this program is to develop thermal-shock-resistant composite materials systems that could be reliably used with high-flame temperature rocket propellants for aerospace propulsion applications. In concurrent work,[11] hot pressing procedures were developed and materials physical properties have been characterized. Preliminary torch test have been also carried out at relatively high temperature in significant combustion environments to investigate the materials ablation behavior.

Typical conditions encountered in real propulsion applications, i.e. the combination of very high temperature, high oxygen partial pressure and total pressure, leading to extreme nozzle thermal and mechanical stresses, can be found in rocket engines. The purposes of the present study are to utilize a lab-scaled hybrid rocket engine available at the University of Naples to test candidate nozzle materials under the typical operating conditions of a rocket engine. Prior work has shown that graphite nozzle throats are eroded and consequently the physical properties, in particular the combustion chamber pressure and the thrust, change during operation.

Therefore, a segmented de Laval nozzle was designed and manufactured with a throat insert of the same ceramic composite material (TaC + 10 vol% $MoSi_2$) hot-pressed with the technique described elsewhere.[11] The nozzle with the ceramic insert was tested comparing the performance of a similar graphite nozzle throat insert.

2. Experimental

2.1. Device

The Aerospace Propulsion Laboratory of the University of Naples "Federico II" was set up primarily for the purpose of testing hybrid rocket engines.[12] Being equipped with a test bench and a general purpose acquisition system, it is extremely versatile as it is possible to easily adjust the experimental apparatus to several classes of tests, including evaluation of performances of propellants and combustion processes, testing of sub-components and/or complete power systems, nozzles, air intakes, catalytic systems, burners, ignition and cooling systems.[13–16]

The layout of the engine used in the present work, including injector, ignition, single-port cylindrical fuel grain, pre and post-combustion chambers, and expansion nozzle, is illustrated in Fig. 1. The facility was here utilized with the focus on test of low erosion thermal protection materials in thrust nozzles. In order to evaluate the performance of hybrid engines, the small size rocket engine shown in Fig. 1 is generally equipped with a De Laval nozzle, to accelerate hot exhaust producing thrust forces up to 200 N. The pressure in the pre and post-combustion chambers are in the range 5–20 bar. Each test has a duration that, according to the fuel (Hydroxyl-Terminated Polybutadiene, HTPB, Polyethylene, PE, High Density Polyethylene, HDPE, Paraffin Wax) and to the mass flow rate of the oxidizer (Oxygen or Nitrous Oxide) may be in the order of 20–30 s.[17,18]

The test bench consists of a supporting structure for the experimental motor where the thrust is measured by load cells, Tedea-Huntleigh model 1042 (range between 0 and 1000 N). A pressure line reaching a maximum value of 40 bar supplies the oxidizer. Before the injection, mass flow rate is evaluated through gas temperature and pressure measurements across a Venturi tube. The pressures in the pre-chamber and post-chamber (see Fig. 1) are measured by two capacitive transducers, Setramodel C206 (range between 0 and 35 bar: accuracy: ±0.13% Full Scale). The acquisition system is based on a software developed in Labview and National Instrument PXI