

Technology Challenges in Solid Energetic Materials for Micro Propulsion Applications

by Eugene Zakar

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1. Introduction/Background

Because of their simplicity, micro thrusters have received significant attention during the last few years, though early development in the microelectromechanical systems (MEMS) field began approximately 10 years ago. The principle applications of micro thrusters are for primary propulsion and attitude control of micro spacecraft, micro satellites (10 to 100 kg), nano satellites (1 to 10 kg), and pico satellites (0.1 to 1 kg). These small-scale satellites will require efficient propulsion systems that can approach and maneuver around objects in space orbit.

If we consider fundamental requirements for a rocket thruster, assuming that it is used for the attitude control of a 10 kg spacecraft with 1 m/s velocity increment to maneuver around an object in orbit, a total impulse thrust of 10 N-s would be needed. If each thruster produces an impulse thrust of 1mN-s, the total impulse thrust of 10 N-s can be produced by 10,000 thrusters. If each thruster occupies an area of 1 mm in diameter, a 100×100 array of thrusters (10,000 thrusters) requires an area of 10×10 cm, which can fit on the side of a satellite. Evolutionary improvements in material and assembly techniques are expected to significantly reduce the total array size requirements.

Technological efforts are converging from two directions: the miniaturization of conventional thrusters and the development of new solid energetic materials (EM) and device concepts. Most of the focus is on the use of MEMS technology and hybrid technology for reducing the cost, size, and system weight. MEMS technology allows subsystems (such as micro thrusters, switches, phased array antenna) to be reduced significantly in size and mass, and in the longer term will enable new classes of extremely small, intelligent, and relatively low-cost batch-produced pico satellites consisting primarily of bonded MEMS devices.

The conventional use of liquid EM for propellant systems is due to their high specific impulse, their ability to be restarted, and their high thrust-weight ratio. The main disadvantages of liquid EM propellants are the power consumption required for pumps and valves, and the necessary cooling system to keep the system from overheating. Additional concerns are the precautions of handling toxic liquids, storage, and leakage, making solid EM alternatively more reliable. The concept is based on the high rate combustion of one single propellant or explosive stored in a combustion chamber.

Some advantages of solid EM include:

- Propulsion systems of the same order of size as the other components of the vehicle
- No moving parts
- Vastly reduced number of components

- No storage tanks, fuel lines, or valves
- Ability to deliver thrust in very precise and predetermined amounts

Disadvantages, however, include the following:

- Single shot characteristic
- Relatively low specific impulse

The lack of restart ability in solid EM is compensated by the fabrication of arrays of thrusters. Each thruster could be addressed independently, or in other words, "digitally controlled micro thrusters."

- Each thruster is a chamber filled with an EM
- The heater element ignites the EM in the chamber
- The content bursts and the escaping gas delivers an impulse force
- Thousands of needle-sized thrusters can be fabricated using MEMS micromachining methods

Solid EM can be classified into different classes—propellants, explosives, and pyrotechnics (1). Propellants and pyrotechnics release their energy through relatively slow deflagration processes (combustion). Explosives release their energy in fast detonation processes. Explosives can be produced by mixing of oxidizer and fuel constituents (nitroglycerine), or by mixing oxidizer powders (ammonium nitrate or perchlorate) and fuel powders (sulfur or carbon) to produce a composite black powder. But these are slower reactions due to limited mass transport of the reactants.

So far, several types of EMs have been evaluated by several institutions; achievements are in the evolutionary stages, and systems are not reliable for micro propulsion applications. The greatest challenges are compatibility of EM and MEMS. A summary of the research and developments by the participating institutions is presented to illustrate the technology trends for micro propulsion applications.

2. Trends

TRW Corporation lead a Defense Advanced Research Projects Agency (DARPA)-sponsored a large project (US \$3.5M) in the late 1990s to investigate digital micro propulsion. The research was jointly conducted by TRW, the Aerospace Corporation, and the California Institute of Technology (2). They used a MEMS approach to assemble an array of chambers filled with a

propellant (figure 1). The researchers reported that the propellant did not achieve complete combustion within the confines of their chambers. Test data can be summarized as the following:

- Fuel is lead styphnate
- 1 m-s thrust duration
- 0.1 mN-s impulse
- 100 W of power

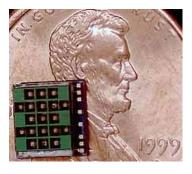


Figure 1. Micro thruster array fabricated jointly by
TRW and teammates Caltech and the Aerospace
Corp. under a contract from the DARPA.

Tanner Research Inc. demonstrated high specific impulse chemical micro thruster actuators (3) shown in figure 2. The institution helped gain a more thorough understanding of the fundamental relationship between thrust and nozzle dimensions. They determined that more propellant volume produced more impulse. Impulses were roughly proportional to volume, and the specific impulse (Isp) was intrinsic to the energetic material (black powder). Isp was found to be inversely proportional to the nozzle exit hole diameter.



Figure 2. Photograph of Tanner Research Inc. array of micro thrusters.

Performance and characteristics of the Tanner micro thruster:

- Fuel black powder
- Array metal/epoxy composite
- Spacing 10 mm (center to center)

- Thrust pulse 4 m-s, thrust ~10 N
- Max Isp record 140 s
- Firing voltage 10 V
- Total volume 800 mm³ (per thruster including epoxy unit cell)
- Impulse/volume 0.05 mN-s/mm³

The Berkeley Sensor and Actuator Center at the University of California conducted research on millimeter-scale solid propellant rockets, shown in figure 3, for one-time deployment of wireless sensor platforms, known as the Smart Dust program. The investigators found the thermal losses to the silicon sidewalls were too high to reliably maintain a burn (4). Successful combustion was demonstrated in cylindrical alumina ceramic combustion chambers with thermal conductivities five times lower than silicon and cross sections of 1–8 mm². Micro heaters required less than 0.5 W of power to ignite a propellant composed primarily of hydroxyl-terminated polybutadiene (HTPB) with ammonium perchlorate (AP) oxidizer. The researchers also added ten-junction thermopiles near the micro rocket flame that produced a maximum power of 20 µW in order to demonstrate energy harvesting to augment the Smart Dust power supply.

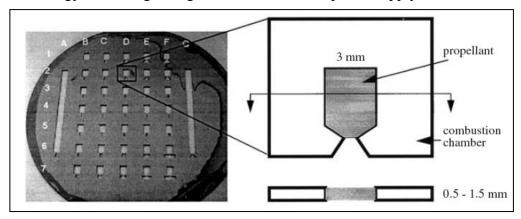


Figure 3. BSAC silicon wafer with DRIE combustion chambers (left), and chamber design (right).

Peformance and characteristics of the Berkeley micro thruster:

- Fuel is HTPB/AP
- Thrusts of 10⁻¹⁵ mN were measured for ceramic
- Specific Impulse 14 s
- Burn rate 1.7 mms⁻¹

Tohoku University of Japan, together with the Institute of Space & Astronautical Science (5), fabricated silicon nozzles (left) and pyrex cylinders with electrical igniters (right), shown in figure 4. The prototype device has an array of 10×10 solid propellant micro-rockets with a pitch

of 1.2 mm. Boron/potassium nitrate (NAB) propellant was selected over HTPB/AP and glycidyle azide polymer (GAP), because of its ignition capability at atmospheric pressure and low temperature. NAB is used with/without lead rhodanide/potassium chlorate/nitrocellulose (RK) to aid in the ignition. The impulse thrust was measured using a pendulum suspended in ambient air.

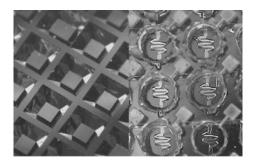


Figure 4. Silicon nozzles (left), and Pyrex cylinders with electrical igniters (right) from Tohoku University. Pyrex cylinders are each approximately 1 mm in diameter.

Performance and characteristics of the Tohoku micro thruster:

- Fuel is NAB
- Ignition = 4 W with RK, and 6 W without RK
- Impulse thrusts = 2×10^{-5} N-s to 3×10^{-4} N-s

Sandia National Laboratories investigated mesoscopic structure of EM and their response during ignition, growth, and detonation (6). Mesoscopic properties, such as particle size, porosity, heterogeneity and material interfaces, determine rates of gas transfer, heat conduction, void collapse, and frictional heating during ignition and growth. Pentaerythritol tetranitrate (PETN) films up to 40-µm thick were deposited on silicon substrates to investigate processing techniques and determine the resolution of film patterns.

Patterning of PETN using oxygen plasma etching proved to be too harsh an environment, causing solubility changes and physical eruptions of the film, likely due to thermal stresses in the film. An alternative patterning technique that has proved successful involves the use of a "lift-off" procedure in which, prior to the EM film deposition, a silicon dioxide masking layer with openings was deposited on the surface. The openings acted as anchor points during PETN blanket deposition that followed. When the hydrofluoric acid (HF) solution was finally introduced, the underlying silicon dioxide film dissolved, leaving only the PETN anchor points in place. Coarse PETN features were defined using this method, but finer features in this film thickness range were difficult to achieve due to the lack of a continuous mass of sufficient

material to support the remaining structures in the film. A representative patterned film is shown in figure 5.

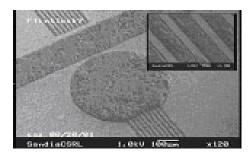


Figure 5. Arrays of micro patterned PETN explosive materials are being investigated by Sandia National Laboratories as on-chip sources of gas for actuation, locomotion, and acoustic signal generation. Center ball pattern is less than 0.5 mm in diameter.

The Laboratory for the Analysis and Architecture of Systems-National Center for Scientific Research (LAAS-CNRS) in France assembled solid propellant micro thruster arrays with reservoir dimension 1.5 mm across using Foturan or silicon materials (7). The nozzle and igniter parts were made by micro machining the silicon in figure 6. Ignition energy was very low—tens of mJ, depending on the ignition material. Two specific propellant mixtures have been optimized and used for the ignition process: a GAP mixed with AP and zirconium (Zr). When a GAP propellant is used in the reservoir, the ignition process works unreliably with the nozzle attached on top. A more sensitive and easily ignitable zirconium perchlorate potassium (ZPP) propellant has since been substituted with improved reliability (8).

First thrust characteristics tested were:

- Fuel is GAP-based propellant
- Impulse force of 6 mN-s
- Ignition Power 150 mW

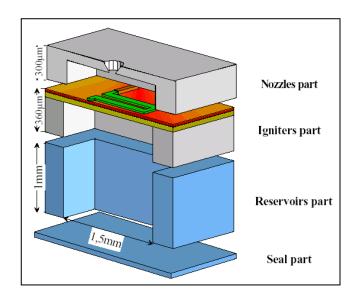


Figure 6. Schematic view of one single micro rocket by LAAS-CNRS Laboratory.

The Federal Polytechnic Institute of Lausanne Space Center (IPFL) at Lausanne, Switzerland, RUAG Aerospace (Emmen, Switzerland), and RUAG (Ammotec GmbH, Fürth, Germany) reported at a recent conference that the largest technological barriers to the development of solid propellant micro thruster arrays shown in figure 7 were reliable, low power ignition and complete combustion of the propellant within the micro cavities (9).

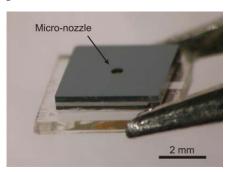


Figure 7. Photograph of a single solid propellant micro thruster by team IPFL Space Center, and RUAG.

The U.S. Army Research Laboratory (ARL) has developed high energetic reaction in porous silicon (10). The motivation was on-chip integration of energetic material and MEMS using existing processes on silicon wafers, shown in figure 8. The silicon was etched electrochemically in HF to create nano pores; those pores were filled with an oxidizer such as sodium perchlorate (NaClO₄), and a heated bridge wire created a self-sustaining energetic reaction between the silicon and the oxidizer. Using characterization techniques during energetic reaction and infrared absorption analysis after explosion, the principal investigators demonstrated an improved

understanding of the energetic behavior. The technical challenges are reliability and repeatability; low-power electronic initiation; and protection from the effects of aging.

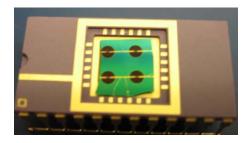


Figure 8. Photograph of a MEMS addressable 2x2 energetic device by ARL. Each circle pattern is approximately 2 mm in diameter.

3. Conclusions

Promising propulsion technologies have been developed by research groups involved with arrays of solid propellant micro-thrusters that are able to deliver small, controlled levels of thrust.

On the system-level there are issues with low ignition yield, and unsustained combustion in small dimensions (quenching); further, filling procedures are not being reproducible, and the propellants and igniters are not easily adapted to each other. Challenges related to small length scales, heat transfer, and surface tension in micro and nano EM need to be sorted out before the technology is ready for its intended applications.

MEMS technology has been identified by several institutions as key to the development of future micro propulsion systems.

4. References

- 1. Rossi, C.; Zhang, K.; Estève, D.; Alphonse, P.; Tailhades, P.; Vahlas, C. Nanoenergetic Materials for MEMS: A Review. *J. Microelectromechanical Systems* **Aug 2007**, *16* (4).
- 2. Lewis, D. H., Jr.; Janson, S. W.; Cohen, R. B.; Antonsson, E. K. Digital Micropropulsion. *Sensors and Actuators* **2000**, *A80* (2), 143–154.
- 3. Desai, A.; Fuchs, B. Micro (MEMS) and Nanotechnologies for Defense and Security. *Proc. of SPIE* **2007**, *6556*, 65561A.
- 4. Teasdale, D.; Milanovic, V.; Chang, P.; Pister, K.S.J. Microrockets for Smart Dust. *Smart Mater. Struct.* **2001**, *10*, 1145–1155.
- 5. Tanaka, Shuji; et al. MEMS-Based Solid Propellant Rocket Array Thruster with Electrical Feedthroughs. *Trans. Japan Soc. Aero. Space Sci.* **2003**, *46* (151), 47–51.
- 6. Tappan, A. S.; Long, G. T.; Renlund, A. M.; Kravitz, S. H. Microenergetic Materials-Microscale Energetic Material Processing and Testing. *presented at the 41st AIAA Aerospace Sciences Meeting and Exhibit*, Reno, NV, Jan. 6–9, 2003, Paper AIAA-2003-0242.
- 7. Larangot, B.; Conédéra, V.; Dubreuil, P.; Do Conto, T.; Rossi, C. Solid Propellant MicroThruster An Alternative Propulsion Device for Nanosatellite," *presented at Aerospace Energetics Equipment 2002*, Avignon (France), 12–14 Nov 2002.
- 8. Rossi, Carole; Briand, Danick; Dumonteuil, Maxime; Camps; Thierry; Phan, Phuong Quy^en; de Rooij, Nicolaas F. Matrix of 10×10 Addressed Solid Propellant Microthrusters: Review of the Technologies. *Sensors and Actuators* **2006**, *A 126* 241–252.
- 9. Briand, D.; Guillot, L.; Bley, U.; Danninger, S.; Gass, V.; de Rooij, N. F. Digital Micro-Thrusters with Simplified Architecture and Reliable Ignition and Combustion. *Proc. of 8th International Workshop on Micro and NanoTechnology for Power Generation and Energy Conversion Applications*, Sendai, Japan, November 9–12, 2008, pp. 157–160.
- Churaman, W.; Currano, L., Singh; A. Kumar; Rai, U. S.; Dubey, M.; Amirtharaj, P.; Ray, P.
 C. Understanding the High Energetic Behavior of Nano-energetic Porous Silicon,"
 Chemical Physics Letters October 2008, 464 (4–6).

List of Symbols, Abbreviations, and Acronyms

AP ammonium perchlorate

ARL U.S. Army Research Laboratory

DARPA Defense Advanced Research Projects Agency

EM energetic materials

GAP glycidyle azide polymer

HF hydrofluoric acid

HTPB hydroxyl-terminated polybutadiene

IPFL Federal Polytechnic Institute of Lausanne Space Center

LAAS-CNRS Laboratory for the Analysis and Architecture of Systems-National Center

for Scientific Research

MEMS microelectromechanical systems

NaClO₄ sodium perchlorate

NAB boron/potassium nitrate

PETN Pentaerythritol tetranitrate

RK lead rhodanide/potassium chlorate/nitrocellulose

Zr zirconium

ZPP zirconium perchlorate potassium

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