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MICROSCALE COMBUSTION OF DDNP PARTICLES IN VACUUM ENVIRONMENT

Takahiro Okada[1], Yoshinori Yamada[2], Yoshiaki Mitarai[2], Tatsuya Ikuta[1]
Kunihito Nagayama[1] and Koji Takahashi[1]

[1]Dept. Aeronautics and Astronautics, Kyushu University, Fukuoka 819-0395, JAPAN
[2]Asahi Kasei Chemicals Corporation, Miyazaki 882-0854, JAPAN

Pressure dependence of solid-propellant combustion is explored by using DDNP (diazodinitrophenol) in a 100μm-order micro tank. Comparison study of the open and closed tanks in vacuum and atmospheric conditions is conducted. The reason of no-firing of open tank in vacuum environment is discussed. Lead styphnate is also compared with DDNP and DTA/TGA study gives their intrinsic sensitivity. The design direction of DDNP-based micro rocket is also proposed for the precise control of pico satellites.

1. INTRODUCTION

Solid propellant has unique advantages for power MEMS applications: leak-free storage, simple system without mixing, etc. MEMS rocket array is one of the most attractive devices and has been developed all over the world using many kinds of propellant [1-8]; GAP(Glycidyl Azide Polymer), lead styphnate, NAB(Boron/Potassium Nitrate), RK(lead rhodanide-based explosive), nitrocellulose, HMX, RDX, HTPB/AP/AL, PETN(Pentaerythritol Tetranitrate), ZPP(Zirconium Perchlorate Potassium), gun powder, etc. However, their microscopic characteristic is not fully understood though the choice of propellant is a key for the performance of micro rocket.

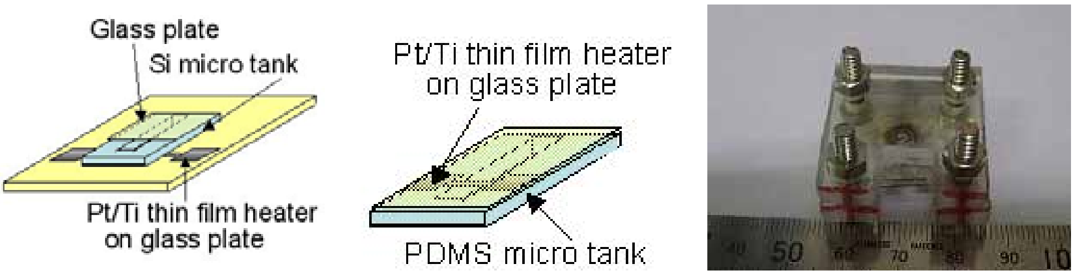
The recent continuous effort using MEMS technology has reached close to practical micro rocket array of 1mm-order bits by using the existing propellant. For the future, in order to apply this kind of MEMS rocket to the precise control of micro satellites, more miniaturization is required keeping high specific thrust and low ignition energy [6-7]. The most important issue for this miniaturization is sensitivity of solid propellant because increased surface/volume ratio results in quenching. However, too sensitive explosives are difficult to handle and cause unintentional firing. The authors recently proposed DDNP for power MEMS application because its lead-free constitution and generated gas volume as much as GAP are attractive. The theoretical ISP of DDNP is 69.5s, which is lower than GAP (97.9s) but higher than gun powder (57.1s). Our 200μm-order rocket successfully fired in atmospheric pressure by using DDNP and lead styphnate but not by nitrocellulose, HMX, RDX, and PETN [8]. Moreover our synthesis technology of rounded DDNP enables much easier handling than any other powder when charging it to the micro tank.

The present paper treats DDNP combustion in vacuum environment especially using real-size system. It is well known that the pressure dependence of solid rocket is so critical. Correspondingly the micro rocket design should be adjusted depending on the pressure condition but the freedom of micro rocket structure is so limited. Our priority is to understand the physics of pressure effect on propellant combustion. The difference between DDNP and lead styphnate is explained using differential thermal analysis (DTA) and thermo gravimetric analysis (TGA) and the design of 100μm-order micro rocket is discussed.

2. COMBUSTION TESTS

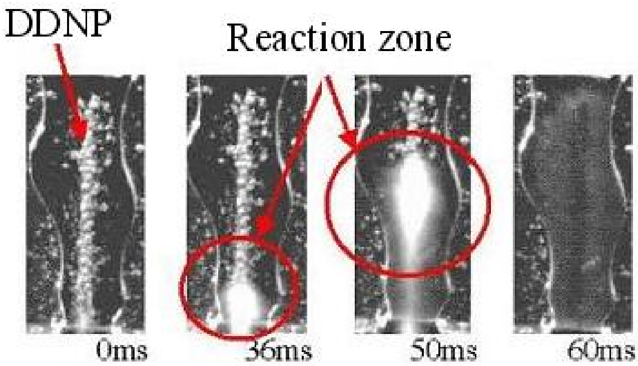
2.1 Experimental setup

Two kinds of chips are used for combustion tests in atmospheric (1atm) and vacuum (10Pa) environment. One is composed of a silicon wafer of 100μm thickness and two glass substrates as shown in Fig. 1(a), where a silicon wafer is etched through for micro tank of 100μm X 400μm X 2000μm and Pt/Ti thin film heater is deposited on a glass substrate and set close to the open end of tank. Spherical DDNP particles of 180μm diameter and lead styphnate powders are used here. Both are charged by hand in the 100μm depth tank. Consequently some DDNP particles are crashed and the remaining gap is bridged by epoxy adhesive.

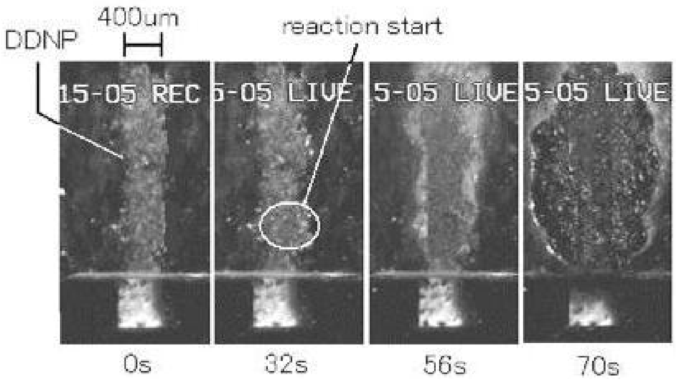


(a) (b) (c)

Fig. 1 Test chips (a), (b) and picture of jig (c)



(a) Atmospheric



(b) Vacuum

Fig. 2 Snapshots of DDNP in open-end tank ignited by 5W heater.

The other test chip of the same size is fabricated by using PDMS $[C_2H_6OSi]_n$ as shown in Fig. 1(b). Experiments are carried out by sandwitching the test chip between two acrylic plates as shown in Fig. 1(c) to seal the tank perfectly because tiny gap always remains in the Si-glass-adhesive structure. PDMS tank has enough flexibility to fit the DDNP particles.

The power of ignition heater is set 5W. It has to be noted that most of this power dissipates to the substrate because no membrane structure is applied here but this fundamental research does not concern it. From the combustion tests, burn rate and ignition delay are obtained by using high speed camera.

2.2 Results and discussion

Some snapshots of DDNP combustion in atmospheric and vacuum environment are shown in Fig. 2. It was found that DDNP is not burned and only melts in vacuum because the radiation is so weak. The melting stars after 32s, which is much longer than the ignition delay of 4-36ms in atmospheric pressure. All results including lead styphnate are listed in Table 1. In order to find the best way to improve DDNP reaction, spicular powder of about 30x80μm, more dried sample, and spicular powder plus $KClO_3$ were prepared and tested. However, all effort did not work well for ignition of this open-end system. It is concluded that powder shape, residual water, and oxidizer balance do not have the fatal effect.

The successful result of closed tank in vacuum is good news. Both its burn rate and ignition delay of DDNP are same as open in atmospheric. The major difference between open and closed tanks is the diffusion of the generated gases. Because the diffusion velocity depends on the mass of gases, DDNP is disadvantageous due to its lead-free constituent while metal-based propellants; ZPP, gun-powder, etc, are expected favorable for this problem. Other possible disadvantages of DDNP are discussed in the following chapter by comparison with lead styphnate.

3. COMPARISON WITH LEAD STYPHNATE

3.1 Characteristics

Table 2 shows representative characteristics of DDNP and lead styphnate. Because O_2 balance is not the critical issue as mentioned above and detonation velocity and calorific value are similar, the higher specific gravity might be critical for the total heat release from a constant-volume tank.

3.2 DTA/TGA analysis

The lower ignition temperature of DDNP should be preferable for thermal ignition but our combustion test results do not hold this sensitivity index. To investigate this contradiction, DTS/TGA analysis was carried out using about 0.3mg sample. Fig. 3 shows examples of data employing several heating rate, from which we obtained the activation energy of DDNP and lead styphnate. The activation energy is a characteristic of ease of starting reaction. In addition to the known data [9] of PETN and RDX, the obtained activation energy and ignition temperature are listed in Table 3.

We already reported that PETN and RDX do not ignite even in atmospheric condition differently from DDNP and lead styphnate. This reason is deducted from Table 3 that the very low ignition temperature or very low activation energy resulted in such results. However, the open end in vacuum environment is expected to cause fatal cooling due to gas expansion and the characteristics of low ignition temperature is degraded.

Table 1 Combustion test results of DDNP

Propellant/tank	Atmospheric	Vacuum
DDNP/Open	Burned	Melted
DDNP/Close	Burned	Burned
Lead styphnate /Open	Burned	Burned

Table 2 Kinetic parameters of DDNP and lead styphnate

	DDNP	Lead styphnate
Molecular formula	$C_6H_2N_4O_5$	$C_6H_3N_3O_5Pb$
O_2 balance	-60.9%	-22.2%
Detonation velocity	6600 [m/s] (ρ = 1.5 [g/cc])	5200 [m/s] (ρ = 2.9 [g/cc])
Specific gravity	1.63 [g/cc]	3.0 [g/cc]
Ignition point	180 [^oC]	275-280 [^oC]
Calorific value	1,089-1,706[J/g]	1,102-1,549[J/g]

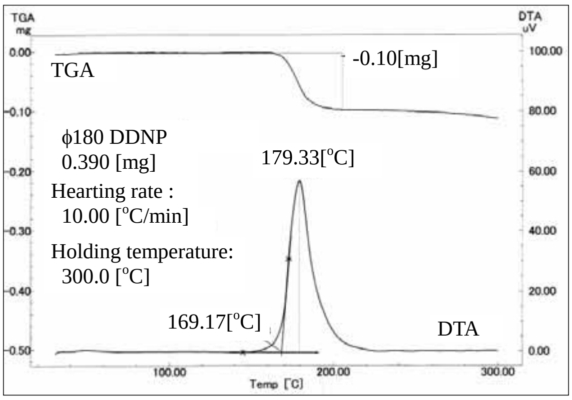


Fig. 3 DTA/TGA curves of DDNP

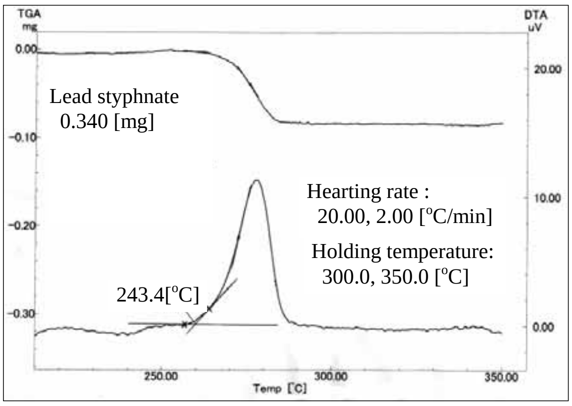


Fig. 4 DTA/TGA curves of lead styphnate

Table 3 Activation energy and ignition temperature

	Ea [KJ/mol]	T [^oC]
DDNP	250.5	180
Lead styphnate	136.6	275
PETN [9]	175	201-216
RDX [9]	144	231-251

4. CONCLUDING REMARKS

Microscale combustion of DDNP is tested in vacuum environment. Because the increased surface/volume ratio requires high sensitivity of propellant, the pressure reduction effects critically on the accomplishment of combustion. The reasons of no ignition in vacuum were discussed and it is concluded that diffusion velocity and activation energy are the critical factors. Because activation energy is intrinsic and temperature is difficult to keep higher than ignition point due to the gas expansion, the DDNP combustion system has to be closed in vacuum. If DDNP is used for micro rocket in space, the membrane structure, which is applied by the past-reported rocket arrays, is an indispensable part as far as a heater of small power is used. The membrane should be designed to break almost at the end of combustion. This kind of system can be realized by the current MEMS technique but the uniform charging of propellant is another key technique.

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