

# Development of green propellants for future space applications

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## Abstract

Several years ago, toxic and carcinogenic hydrazine and its derivative propellants are commonly used in spacecraft propulsion. These propellants impose distinctive environmental challenges and consequential hazardous conditions. For the future applications, the increasing level of space activities and their applications required a substitution of hydrazine by low toxic green propulsion which becomes even more pronounced. In this review, promising green space propellants such as hydroxylammonium nitrate and ammonium dinitramide are reviewed and investigated for various space missions. In-depth system studies in relation to the aforementioned propulsion, the design for advanced green propulsion systems of the future is appreciated.

**Keywords** : green propellants, specific impulse, thermal decomposition, combustion, reaction control system.

## 1. Introduction

Space missions use neat hydrazine ( $N_2H_4$ ) and its various mixtures in monopropellant applications, mainly for orbit, attitude control of satellites and spacecraft propulsion subsystem. However, hydrazine as conventional monopropellant is considered extremely toxic due to its high vapor pressure of hydrazine and its high toxicity caused high storage and handling costs. Therefore, its possible substitution by *low toxic propellants* is the main goal to reduce the handling costs linked to this toxicity. Among possible hydrazine substitutes, aqueous energetic ionic liquids comprising an ionic oxidizer, an ionic or molecular fuel and water as solvent, are often proposed and investigated as new monopropellants.

### 1.1 Hydroxylammonium nitrate

Hydroxyl ammonium nitrate (HAN,  $NH_3OHNO_3$ ) is a promising substitute for hydrazine because of its low toxicity, high density, high specific impulse, and low freezing point. Thus, the substitution of toxic conventional

propellant by a *less toxic monopropellant* is desired<sup>(1),2)</sup> in order to investigate low toxic spacecraft propulsion subsystem with *green propellant*. In artillery guns, one of the significant advantages of using liquid propellants rather those solid propellants is the capacity to control the combustion by metering the propellants<sup>(3)</sup>. Extensive studies have been conducted to characterize the ignition and combustion behavior of HAN. Pressure effects on the burning rates as well as flame structures of HAN and HAN-based liquid propellants were investigated<sup>(4)-11)</sup>. The behavior of the propellant droplets was observed in terms of micro-explosion and gasification characteristics<sup>(12),13)</sup>, the phenomenon that occurred between the liquid and gas phase under high pressure and temperature environments<sup>(14),15)</sup>, and droplet combustion<sup>(16)</sup>. The decomposition products of HAN were detected by employing various diagnostic techniques<sup>(16)-20)</sup>. Chemical kinetic studies on HAN have been performed in order to determine reaction mechanisms and global kinetic rates. Reactions for dilute HAN in the presence of a large excess

of  $\text{HNO}_3$  were investigated<sup>21)–29)</sup>.  $\text{N}_2$  and  $\text{NO}$  formation reactions in HAN decomposition were also studied<sup>24),30),31)</sup>. However, the experiments were conducted with very low concentrations of HAN at low temperatures to maintain isothermal conditions. Based on the previous reaction studies<sup>21)–31)</sup> and experimental observations, two reaction mechanisms were proposed for fused HAN<sup>32)</sup> and concentrated HAN mixtures<sup>33)</sup>. However, the adiabatic temperature of several liquid propellants are very high and in order to lower these temperatures, a lot of mixtures were tested such as HAN with methanol and ethanol<sup>34)</sup>, HAN in the presence of triethanol ammonium nitrate (TEAN)<sup>35)</sup>, HAN with hydrazinium nitrate (HN)<sup>36)</sup> for reaction control systems and *green propellant* for low toxic spacecraft propulsion subsystem. Moreover, the U.S. army has developed XM45 (LGP1845) and XM46 (LGP1846) for gun applications, which are homogeneous mixtures of HAN, TEAN and water<sup>37)</sup>. In an attempt to develop a new rocket propellant, many formulations have been considered. The most recent application of *green propellant* was developed by Ball Aerospace and Technology corporation which it used AF-M315E, it demonstrates stable performance of a flight weight attitude control thruster for at least 10,000 total pulses<sup>38)–40)</sup>. The current formulation consists of HAN 73.6%, water, methanol as fuel and ammonium nitrate (AN) as additive to stabilize the mixture for long-term storage<sup>41)</sup>.

## 1.2 Ammonium dinitramide

In the late 1960s, Hamel and Olson<sup>42)</sup> synthesized the first dinitramide structures. These materials were covalently bonded alkyl N,N-dinitramides. The resulting dinitramides were unstable and decomposed at 75°C or less, making them unsuitable as propellant ingredients. Recently, a new class of inorganic oxidizers have been synthesized<sup>43)–47)</sup>. This class of oxidizers incorporates the newly discovered dinitramidate  $[\text{N}(\text{NO}_2)_2]^-$  anion. The dinitramide acid and its salts are thermally more stable than the original alkyl N,N-dinitramides discovered in the late 1960s. One promising oxidizer from this class of compounds is ammonium dinitramide; abbreviated as ADN,  $\text{N}_4\text{O}_4\text{H}_4$ , which has the structural formula  $\text{NH}_4\text{N}(\text{NO}_2)_2$ . ADN, like AN, undergoes residueless combustion. However, it burns more readily and predictably than AN. Therefore, it is much more attractive to use ADN as an ingredient in propellants than AN. Several solid propellant formulations of ADN in various binder systems have recently been accomplished<sup>48),49)</sup>. Although it is well known that dinitramides are energetic compounds, the thermal decomposition of dinitramides has not been studied extensively. ADN is a relatively new oxidizer which may be environmentally friendly because it does not lead to a halogen acid decomposition product. ADN decomposition has been discussed by many researchers<sup>50)–52)</sup>.

## 2. Materials and methods

The three different experiments carried out in this study can be distinguished by their heating method and initial

pressure conditions. Firstly, during the thermal analysis by DTA-TG, the propellant was treated under gradual increase of temperature at 1 atm. Secondly; DSC was performed at different initial pressures in order to follow the propellant behavior of HAN and ADN using this method. Finally, HAN monopropellant was injected into preheated catalytic bed.

### 2.1 Liquid propellants

HAN-based liquid monopropellant was prepared by Hosoya Corp. (Tokyo, Japan). The solution composition is HAN 74%, AN 4%,  $\text{H}_2\text{O}$  6%, MeOH 16%; (methanol denoted MeOH). AN and MeOH were added to HAN solution to decrease the freezing point and to control the burning rate of the monopropellant, respectively. ADN in crystalline state (purity 97%) was prepared in ISAS/JAXA, Japan. This material used to prepare liquid ADN and ADN-MeOH solutions by dissolution of ADN in pure water and then addition of desired MeOH quantity was performed. In fact, part of quantity of water was substituted by MeOH because water can affect the oxidation state of the active metal of catalysts.

### 2.2 Thermal decomposition and propellant combustion

#### 2.2.1 DTA-TG and DSC

The decomposition was followed by differential thermal analysis-thermal gravimetric (DTA-TG) and differential scanning calorimetry (DSC) apparatus coupled to mass spectrometer (MS) at different pressures. Known masses of catalyst were put into Al-cell holder followed by spray injection of liquid monopropellant. The mass and temperature were recorded versus time. The analyses by DTA-TG enable to obtain the following information: (i) evaporation of different solvents (endothermic peak); (ii) onset temperature of decomposition given by the inflexion point of the temperature curve; (iii) monopropellant concentration; (iv) efficiency of the catalyst (exothermic peak) and (v) detection of emitted products by MS.

#### 2.2.2 HAN-thruster

The HAN thruster 20 N was already described in previous papers<sup>53)–55)</sup>, in which the pressure sensor was placed in the combustion chamber (after catalytic bed). The upper part of HAN-based monopropellant tank was filled with  $\text{N}_2$  gas from a gas cylinder. The  $\text{N}_2$  pressure in the tank was fixed for HAN catalytic decomposition reaction in order to give an almost constant feeding rate of HAN. The monolithic catalyst was charged into the thruster. Then, the catalyst bed was preheated before test at 210–350°C. The preheating of catalyst was performed by flexible sheath heater with 88W as power and 35 VDC as power-supply voltage. When the electromagnetic valve was opened, HAN-based monopropellant was pressed by the  $\text{N}_2$  gas into the thruster. Ignition delay, catalyst bed temperatures, chamber pressures and feeding rate of HAN were recorded via Lab-view program. The opening time of the electromagnetic valve was varied from 1 to 5 s. The average mass flow for each thruster firing test is around  $12 \text{ g s}^{-1}$  and 1.2 MPa as initial pressure. After tests,

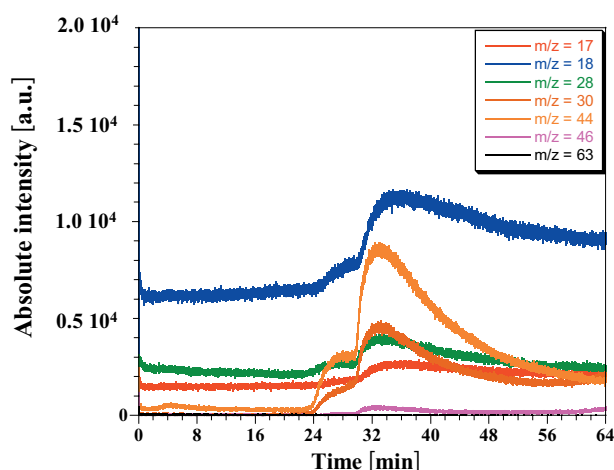


Figure 1 DSC/MS-online analysis of thermal decomposition of HAN-based monopropellant at  $P = 1$  MPa.

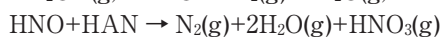
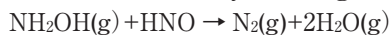
the catalytic bed was purged by  $N_2$  flow, which to remove the produced gases and to decrease temperatures.

### 3. Results and discussion

#### 3.1 HAN

HAN-based monopropellant has been thermally decomposed via DTA-TG apparatus without and with powder catalyst. In fact, the thermal decomposition was reported in our previous paper<sup>53</sup>. The thermal decomposition of HAN-based has been started around  $154^\circ\text{C}$  after an endothermic reaction which demonstrated by total evaporation of MeOH and water as additives. The thermal decomposition occurs in two steps: (i) highly exothermic reaction with a maximum at  $191^\circ\text{C}$ , which correspond to the high loss of HAN weight, while (ii) the second step was less exothermic and reach maximum at  $201^\circ\text{C}$ . In the case of catalytic process, the temperature profile was changed to a violent exothermic reaction of HAN-based in one step at low temperature onset<sup>53</sup>.

The previous obtained data were confirmed by DSC analysis. Thermal decomposition of 5 mg of HAN-based was carried out at 1 MPa as initial pressure and the formed gas were shown in Figure 1. It has been reported that  $N_2$  can be formed by following reactions<sup>18),26),56)</sup>:



Different gas such as NO,  $N_2\text{O}$ ,  $\text{NO}_2$ ,  $\text{NH}_3$ , HNO,  $\text{HNO}_2$  and other intermediates were reported and the reaction mechanisms were already discussed<sup>53),55)</sup>. From data obtained by DSC at  $P = 1$  MPa, HAN thermal decomposition was performed in two steps which can confirm the data obtained by DTA-TG<sup>53)</sup>. Further, the nature of obtained gas was similar to DTA-TG/MS data<sup>53)</sup> at  $P = 0.1$  MPa (atmospheric condition), which implicates that the reaction mechanism was independent of initial condition of pressure.

Otherwise, several HAN-thruster tests were performed in the presence of different catalytic shapes. At first, firing tests were carried out over preheated granular Shell 405 (36% Ir/ $\text{Al}_2\text{O}_3$ ) catalyst at  $210^\circ\text{C}$ <sup>55)</sup>. The thickness of catalytic layer was 1 mm and the size of granular shape

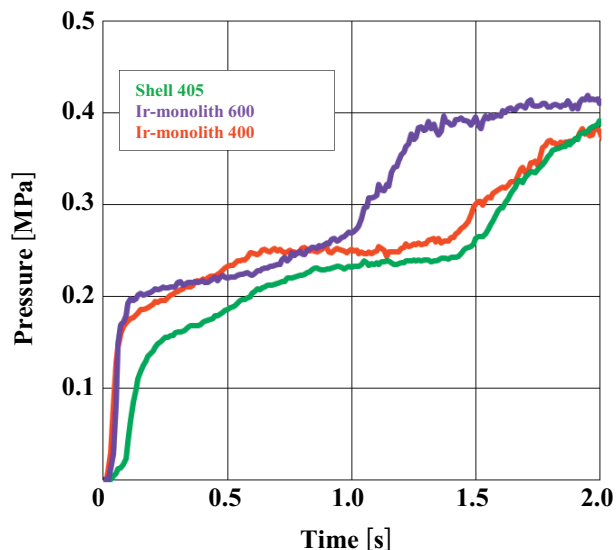


Figure 2 Burning tests of HAN-based liquid monopropellant over different catalyst shapes on 20 N thruster.

was  $25 \mu\text{m}$ . In deed, the reaction of HAN over granular surface was rapid and temperature achieved more than  $1400^\circ\text{C}$ . The efficient heat and mass transfer was observed in the granular catalyst bed, because the granular provided complex flow-paths for mixing. Therefore, for a given reaction volume, the reactivity of Shell 405 catalyst could be brought to the high residence time and high pressure drop. It should be noted that after propellant injection, the temperature of the combustion chamber decreased due to the stored HAN at ambient temperature. Then, the temperature of the system rapidly increased, in which the catalyst can suffer and its activity can be decreased or totally deactivated<sup>54)</sup>.

In order to limit the deactivation phenomena and to reduce the generated pressure drop during reaction tests, the shape of catalyst was changed to honeycomb with square channels.

Figure 2 illustrates the evolution of pressure as a function of time during the burning reaction processes of HAN-based. The burning reactions of HAN-based were conducted over monolith catalysts using 20 N thruster showed that the pressure slopes in the case of Ir-monolith catalysts at different number of channels (400 and 600) were higher against Shell 405 catalyst. In fact, the pressure slope in the case of Shell 405 was around  $1.5 \text{ MPa s}^{-1}$ , while this value was doubled to  $3.5 \text{ MPa s}^{-1}$  in case of 30 wt.% Ir-monolith catalysts. Moreover, the three curves showed second pressure slopes due to second reactions of gas phase products over catalysts. The second pressure slope has been started earlier (after 1 s of monopropellant injection) in the case of Ir monolith catalyst 600. Otherwise, slopes were delayed in the case of Shell 405 and monolith 400. The difference could be proved by the size of channels of catalyst surfaces; monolith 600 has small size channels compared to monolith 400. Indeed, the generated gas products have more contact or longer residence time within catalyst surface 600; that mean gaseous phase undergoes fast reaction with solid catalyst surface in this case. The pressure drop across the catalyst

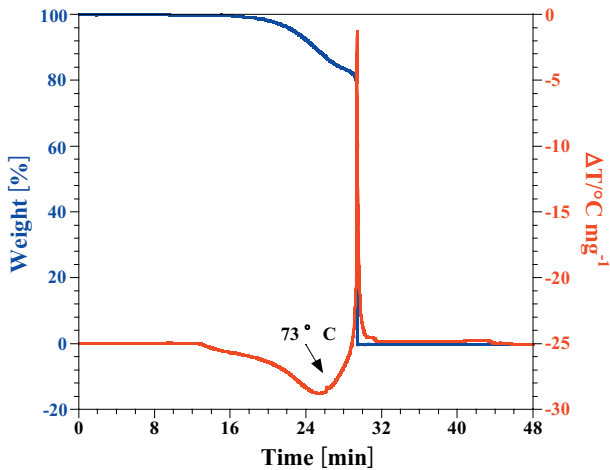


Figure 3 DTA-TG thermogram of ADN catalytic decomposition over CuO-based catalyst.

bed in the case of monolith catalyst was negligible as other advantage.

### 3.2 ADN

The thermal decomposition of ADN-based monopropellant has been performed through DTA-TG. The thermal decomposition was improved by adequate catalyst for ADN decomposition. Doped alumina by  $\text{La}_2\text{O}_3$  was used as catalyst support. Then, CuO active phase was deposited by wet impregnation. The activation of the catalyst was performed by calcination in air at  $800^\circ\text{C}$  for 8 h. ADN catalytic decomposition of 5  $\mu\text{l}$  of ADN-based, 8 mg of CuO-based catalyst and  $2^\circ\text{C min}^{-1}$  as heating rate are given in Figure 3. The thermogram profile was different from the thermal decomposition<sup>41</sup>. ADN-based decomposed rapidly in one step in an exothermic process and happened only after complete water vaporization of ADN. This decomposition started at low temperature. Before ADN decomposition, low mass loss accompanied by a small endothermic phenomenon was observed. This was mainly due to the slow evaporation of water in this temperature range. However, the catalytic decomposition reaction was less exothermic than the thermal decomposition; the low exothermicity was primarily due to the use of the released energy during the decomposition reaction in the process of water evaporation from the mixture. From these results, we can conclude that the CuO-based catalyst decomposed ADN-based at low temperature ( $T_{\text{dec}} \sim 73^\circ\text{C}$ ). In deed, the decomposition temperature was decreased in the catalytic process in comparison with the thermal process ( $T_{\text{dec}} \sim 156^\circ\text{C}$ )<sup>41</sup>, which  $T_{\text{dec}}$  represents the chemical decomposition of the substance. We concluded that the use of a catalyst to improve the ADN-based decomposition has been investigated. The DTA-TGA results have been previously presented; they show that the ADN decomposed in two exothermic steps after water evaporation. The disappearance of the second endothermic peak in the presence of the catalyst shows a good catalytic activity towards ADN decomposition. After that, MeOH was added to ADN-based in order to substitute water and stabilize the propellant. A comparison of the TGA profiles

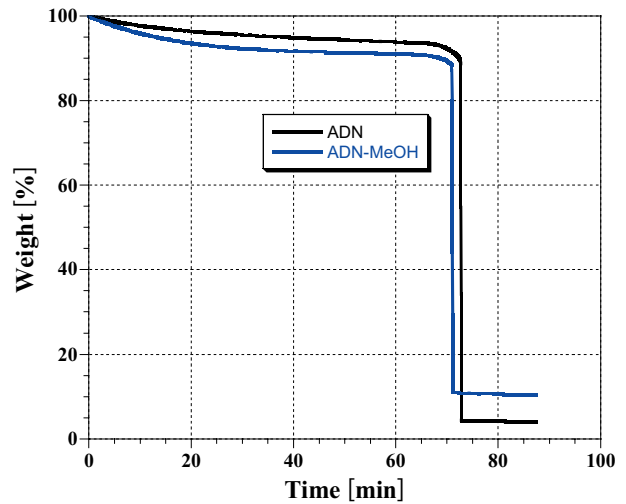


Figure 4 DTA-TG thermogram of ADN-MeOH catalytic decomposition over CuO-based catalyst.

Table 1 Comparison of the thermal and the catalytic decomposition process of ADN and ADN-MeOH based monopropellants.

Monopropellant	Process	Weight loss (%)	Onset temperature of decomposition ( $^\circ\text{C}$ )
ADN	Thermal	23	156
	Catalytic	16	73
ADN-MeOH	Thermal	43	158
	Catalytic	35	87

corresponding to the catalytic decomposition of ADN and ADN-MeOH mixtures with the CuO-based catalyst was followed by thermal analysis, as shown in Figure 4, and the numerical results are given in Table 1. We observed the strongest decrease of the decomposition temperature in the presence of the catalyst in the case of the ADN-based propellant containing methanol. The decomposition started before the complete vaporization of the solvent and a small percentage of the propellant remained in the porosity of the catalyst. This accounted for the slight weight decrease after decomposition. In Table 1, we present the weight loss after thermal and catalytic decomposition of ADN-MeOH with the decomposition points of each mixture. The agreement was generally good, showing that the solvent was removed, leading to ionic liquids for MeOH-containing mixture.

### 3.3 Application on space mission

#### 3.3.1 HAN-based monopropellant

Hydrazine monopropellant system has been the dominant propulsion technology for low total-impulse applications; however, expensive storage, handling, and disposal procedures are required to address the propellant toxicity and flammability hazards, which, though well established, continue to hinder efforts to reduce mission integration costs and schedule. Toward this objective, the NASA Space Technology Mission Directorate (STMD) has initiated the *Green Propellant* Infusion Mission (GPIM)



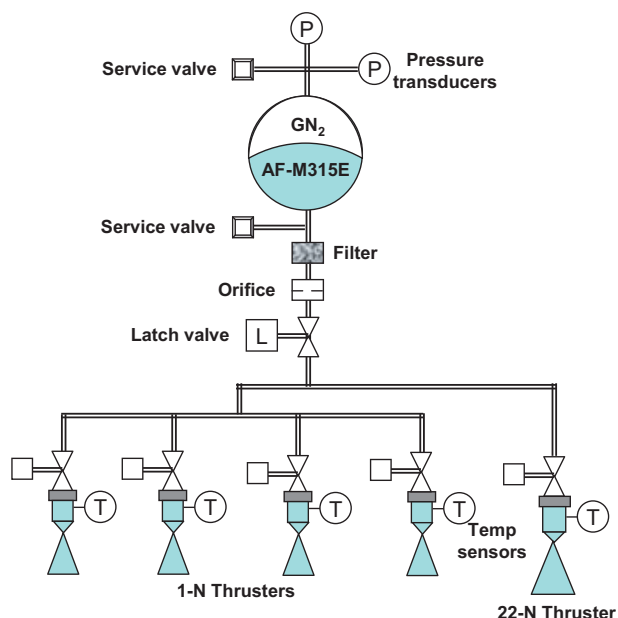


Figure 5 Propulsion system schematic<sup>56)</sup>.

program with the objective of completing the first on-orbit demonstration of a complete AF-M315E high-performance (+50% Isp compared to traditional hydrazine) *green propellant* propulsion system by the end of 2015<sup>57)</sup>.

Design considerations for the AF-M315E propulsion system are mostly similar to a traditional hydrazine system, with a few special considerations as shown in Figure 5. Principally, all system components must be compatible with AF-M315E, and therefore system component selections must strongly take compatibility into account, especially for longer duration missions. As the general schematic layout is identical to single string blow-down hydrazine systems commonly employed on small spacecraft, many of the same general design guidelines apply. The AF-M315E system however, is far less hazardous than a traditional hydrazine system when considering range safety requirements. The propellant is far less prone to leakage (due to higher viscosity), is non-toxic if leaked, and the thrusters cannot inadvertently fire without having first preheated catalyst beds<sup>56)</sup>.

### 3.3.2 ADN-based monopropellant

Two liquid monopropellants based on ADN were developed by Swedish ECAPS agency and FOI Corp.; and were denoted FLP-106 and LMP-103S; respectively. In Sweden, High Performance Green Propulsion (HPGP) was initiated by ECAPS space agency with the concept of development of storable liquid monopropellant blend i.e. a pre-mixed insensitive bipropellant which could be ignited by heat, catalysis or electrical devices. The requirements were to have at least 5% higher specific and 30% higher density impulse than hydrazine, significantly lower toxicity, non-carcinogenic, and being environmentally benign.

They both contain about 64% ADN and they are both considerably less toxic than hydrazine. Both propellants are compatible with most materials currently used in propulsion systems. None of them are sensitive to shock

initiation and they should, from this point of view, not be considered as hazard class 1.1 materials<sup>58),59)</sup>. The main difference between the propellants is that FLP-106 has a substantially lower vapor pressure and higher density. The low vapor pressure is due to the low volatile fuel used compared to the high volatile fuels used in LMP-103S. The composition and the boiling point of the components used in respective propellants are measured. FLP-106 also has somewhat higher performance than LMP-103S but at the expense of higher combustion temperature.

## 4. Conclusion

In this review, we have shed light on a selected number of green space propulsion systems. Findings from this study suggest that *green space propellants* are constantly gaining an augmented interest as alternative propellant candidates for future space applications. In Japan, several research studies were conducted and other research plan still in progress for future development of low toxic propulsion systems. HAN and ADN propellants showed acceptable combustion characteristics and their decomposition onset temperatures are higher than that of hydrazine, indicating that the both propellants are more stable. In fact, the both mentioned propellants can be compatible with different materials for storage issues. Moreover, the onset temperatures of catalytic decomposition of both propellants were decreased over prepared catalysts.

JAXA did similar concurrent effort by other worldwide space agencies such as NASA and ECAPS in order to develop a low hazardous and reduced cost alternative to traditional monopropellant systems using hydrazine, with the goal of meeting new requirements for small satellite missions. Recently, HAN 20 N-thruster chamber and corresponding advanced monolith catalysts were developed and considered as the most promising technology to replace the conventional hydrazine in Japan. In fact, HAN-based monopropellant mixture was selected to be used in "Innovative Satellite Technology" program launched by JAXA.

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