COMBUSTION OF AP-H$_2$ AS PERSPECTIVE SYSTEM FOR PROPULSION

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ABSTRACT

Further progress in rocket propulsion is not possible without creation of new combustive systems and also without development of specific technique for the combustion process. System containing solid oxidizer (for example, ammonium perchlorate - AP) and gaseous fuel (H$_2$, CH$_4$, C$_2$H$_6$ or its mixtures) is one of the promising combustive systems. It is necessary to note that there are not enough investigations on combustion of such systems. However, such research is of great interest for development of rocket propulsion and propellant combustion chemistry. Results of these investigations possibly will be useful from the point of view of creation of new types of rocket engines. Separate storage of gaseous (or liquid) fuel and subsequent its introduction in combustion chamber with solid oxidizer is perspective direction for development of rocket engines. The case, when atmosphere contains one of the propellant ingredients in gaseous state, which can be used in engine, is of especial interest. However, space vehicle can contain the gaseous fuel itself. Traditionally one of the propellant components is oxidizer, and another - fuel. Interaction of these components in combustion chamber allows to operate and control a propulsion process.

This paper is devoted to experimental investigation of AP (solid oxidizer) - H$_2$ (gaseous fuel) flame structure at 0.1 MPa. Such system have been investigated early with the purpose of the creation of hydrogen-breathing combustion engine in the Jupiter atmosphere. But spectroscopic method did not allow to investigating the flame structure and analyzing of the main combustion products, which can be used to test combustion model. In our work flame structure was studied by molecular-beam mass-spectrometry (MBMS) and by microthermocouple method. Strand of AP (5 or 8 mm in diameter and 5 or 8 mm in height) was set on the ceramic needle connected with scanning mechanism, which can move burning strand to the probe. System based on CAMAC was used for obtaining mass-spectrometric data. The strand was ignited by metal wire heated by electrical current. Results of the conducted investigations can be used to test combustion models and to provide a new database for understanding of combustion chemistry in nontraditional propulsion system.

Key Words: ammonium perchlorate, hydrogen, flame structure

NOMENCLATURE

AP	- ammonium perchlorate
d	- diameter
t	- time
TOFMS	- time-of-flight mass-spectrometer
QMS	- quadruple mass-spectrometers
CAMAC	- computer-aided measurement and control
PC	- personal computer
NTP	- normal temperature and pressure
L_0	- initial distance between the surface of AP strand and thermocouple
L	- distance from the burning surface

INTRODUCTION

There are a lot of papers devoted to the thermal decomposition and combustion of ammonium perchlorate (AP). The main reason of it relates with that ammonium perchlorate is commonly used oxidizer in solid propellants. Its practical value accounts for interest of researchers. Recently new oxidizers such as ammonium dinitramide and hydrazinium nitroformate were synthesized, and propellants on their basis were created.[1,2] These propellants have better ballistic characteristics and can replace AP-based propellants. Besides, AP propellants are not ecologically safe. However new scientific discoveries, for example, revealing of hydrogen in atmosphere of Jupiter[3] gave new incentive for investigation of AP combustion. These investigations are connected with creation of hybrid rocket motors on the basis of solid oxidizer (AP) and gaseous fuel (H_2).[4] For development of H_2-breathing engines it is necessary to investigate many characteristics of combustion of AP-H_2 system. One of the most important characteristics necessary for creation of combustion model is flame structure, i.e. profiles of species concentration and temperature in flame. Agreement between experimental results and results of modeling calculations is an important criterion of validity of combustion model.

There are not many papers devoted to the investigation of combustion of AP in hydrogen.[5,6] In paper[5] the dependence of change of diameter (d) of spherical particle of AP on time (t) of burning for different gas flows and pressures were investigated. It was shown that at atmospheric pressure current diameter of burning particle effects on type of this dependence: $d^3 \sim t$ (Ref[5]) and $d^2 \sim t$ (Ref[6]) in the range of particle diameter from 6 to 3 mm and from 3 to 1.6 mm correspondingly. Investigation of flame structure of AP-H_2 by method of filtered photographs[6] allowed to determine concentration profiles of H_2O, ClO and OH in arbitrary units. Data on flame structure of preheated AP at pressure of 0.06 MPa (Ref[7]) was used by authors of paper Ref[6] as boundary conditions for calculation in one-dimensional approach of AP-H_2 structure of laminar flame using CHEMKIN code and three-phase combustion model.[8] But assumption that surface temperature of AP is equal to 1127 °C was unreal.[6] Probably the coarse resolution of "filtered photographs" method did not allow to detect thin zone of chemical reactions adjacent to AP burning surface. More real model of AP-H_2 combustion is presented in Ref[9]. In Ref[9] the temperature of AP burning surface 527 °C is close to experimental data (500 °C).[7] It is necessary to note that application of experimental data of Ref[6] as boundary condition is not proved. The main reason of that is investigation of combustion of strand with small diameter, and as a result increasing the temperature of strand. This fact affects on flame structure and may produce the additional inaccuracy in development of combustion model. So the investigation of steady state combustion of AP strand with enough large diameter when heat feedback does not change the temperature inside the strand have a great interest. Unfortunately, there are not enough experimental data for validation of present combustion model.

The objective of this research was further investigation of combustion mechanism of AP-H_2 system by studying flame structure at atmospheric pressure, including temperature and concentration profiles of species in flame as a function of distance from the AP surface. This system has a great interest and allows to obtain additional information about AP combustion mechanism, which can be used for further development of combustion model of solid AP-based rocket propellants.

EXPERIMENTAL

The investigation of flame structure of AP (solid oxidizer) in atmosphere of H_2 (gaseous fuel) was conducted using molecular beam mass spectrometry, microthermocouples and video recording. Investigation of structure of AP flame in hydrogen, i.e. profiles of species concentration and temperature in flame, was conducted using two methods. During the temperature measurements strand did not move with respect to the thermocouple. After ignition of strand the burning surface moved away from thermocouple with velocity equal to burning rate. In the case of mass spectrometric measurements of profiles of species concentration, burning strand moved toward the probe with the velocity exceeding burning rate. The probe continuously sampled gaseous products from all the flame zones up to the burning surface. Therefore in this case the distance between the probe and burning surface decreased during experiment. Set-up for measurement of concentration profiles of species in flame of solid propellants is shown in Fig. 1. Earlier this setup was successfully used for the investigation of flame structure of azidopolymer, sandwiches and composite solid propellants.[10,11] The sonic probe for sampling of gaseous products and molecular beam for consecutive delivering of sample into the ion sources of time-of-flight and quadruple mass-spectrometers were used in this set-up. In this paper only the time-of-flight mass-spectrometer was used. Cylindrical strands of AP with density of 1.9 g/cm^3 was 5 or 8 mm in diameter ($\varnothing$) and 5 or 8 mm height (h) correspondingly. Temperature profiles in AP-H_2 flame were obtained using Π-shaped WRe(5%)-WRe(20%) thermocouples, made from wire with diameter of 50 or 100 microns. The thermocouple's shoulders with length of 5 - 7 mm was located along the lateral surface of strand, and the junction of thermocouple was above the middle part of the lateral surface of strand. The inflexibility of material of the thermocouple provided the immobility of it relatively the strand. The wires of thermocouple on the distance of ~15 mm from junction were inserted into two-channel ceramic tube and were fasten to it using Ceramobond.[12] Location of strand in the case of mass
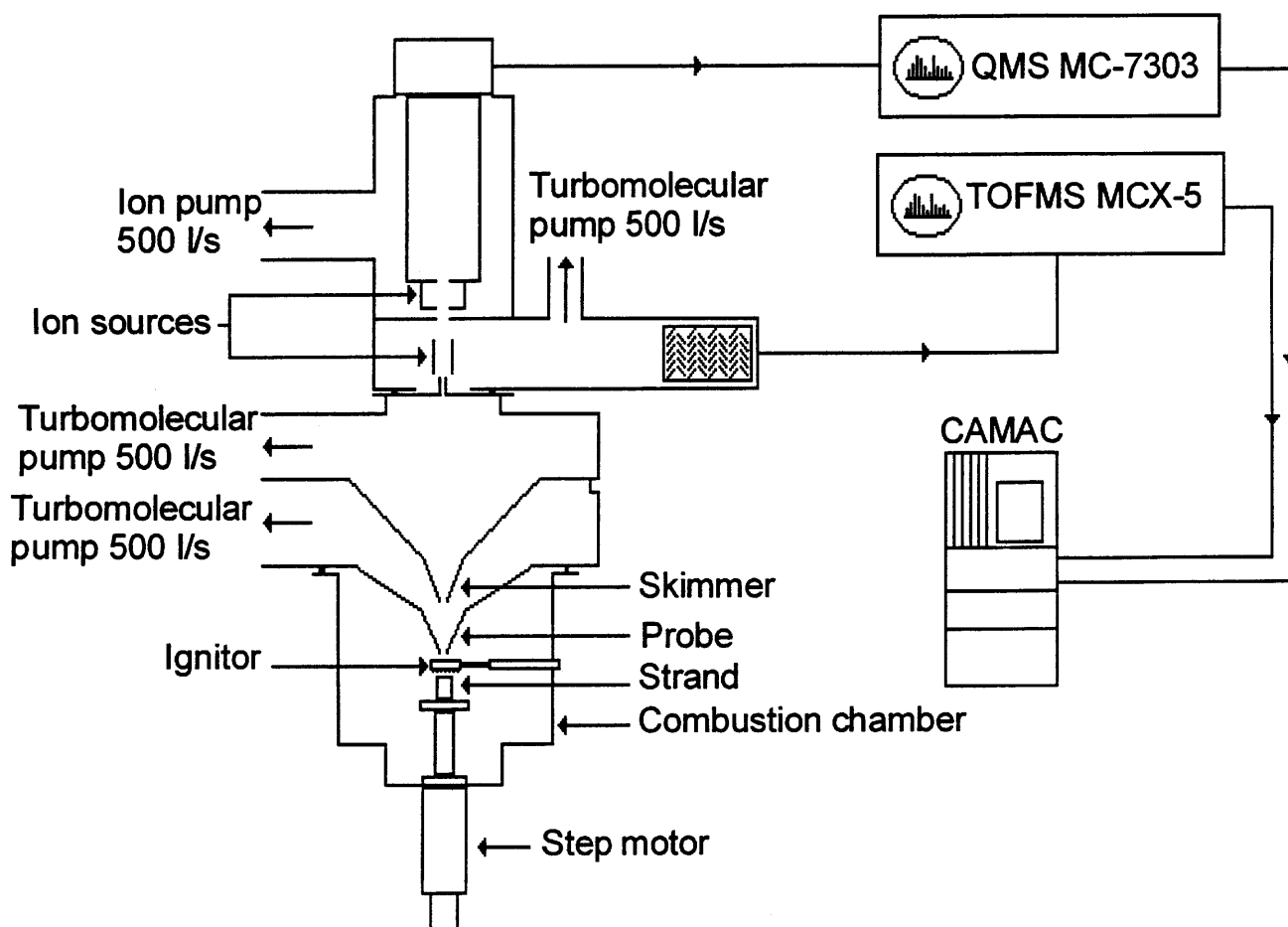

Figure 1. Set-up for investigation of flame structure of solid propellants.

spectrometric (a) and thermocouple (b) measurements is shown in Fig. 2. The distance between burning surface and the probe (or thermocouple) was determined using video recording. A video camera Panasonic NV-M3000EN was used to visualize the combustion process. Temperature profiles were obtained during the burning of strands in flow system. In this case strands were situated in the center of quartz tube of diameter 20 mm at the distance of 40 mm downstream the gas inlet. The hole with depth close to strand radius was made in the middle part of the lateral surface of AP strand. Then strand was fixed on a ceramic needle with diameter of ~ 1 mm, covered by small amount of glue. Protective layer did not cover the lateral surface of strand in order to allow flame propagation along the surface. At first, experiments were conducted in flow of pure hydrogen with volume flow of 60 cm^3/s (NTP). Strand was ignited using electrically heated metal spiral located under the strand at the distance of ~ 1 mm or in contact with strand. Ignition of strands was observed in both cases of location of the spiral. The life time of thermocouples (from the beginning of heating till the destruction of thermocouple) with diameter of 50 or 100 microns, covered by protective layer of Ceramobond with thickness of 30 microns, was equal only 0.4-1.2 s during the burning of AP strand in flow of pure hydrogen. During the heating of spiral an ignition of strand in contact point was observed, then flame propagated all over the strand surface. Initially cylindrical shape of strand became almost spherical during the combustion. The maximal temperature, measured by WRe thermocouple protected from chemical and catalytic interaction with combustion products of AP, was equal to 1800°C and probably was not the final temperature. It is known that at this temperature the melting of Ceramobond takes place. This led to violation of protective layer and as a result to destruction of the thermocouple.
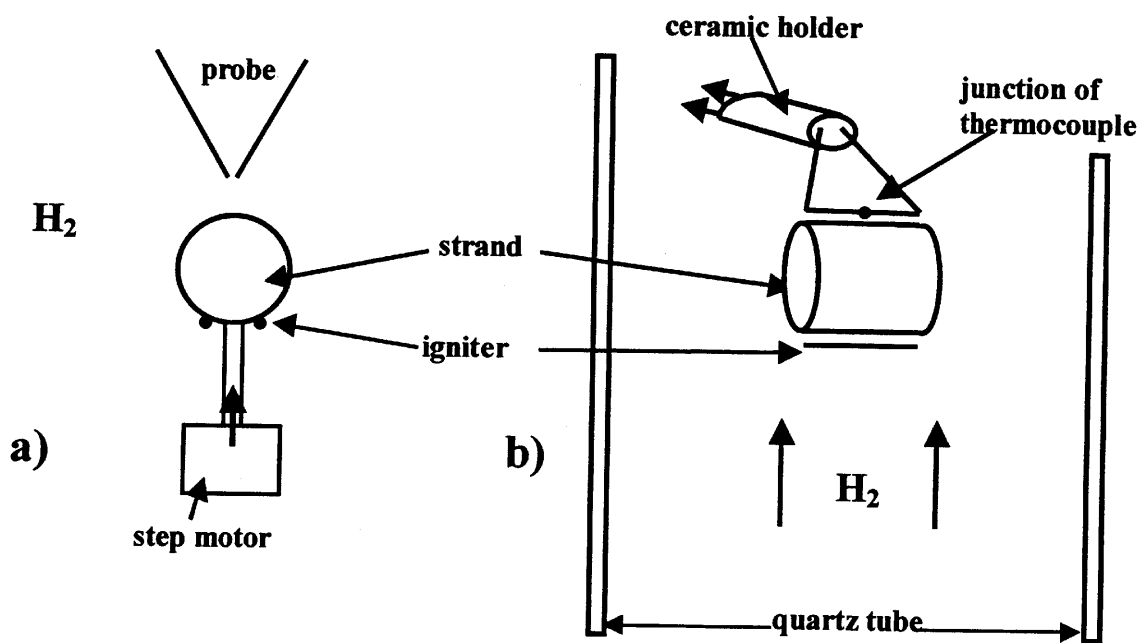

Figure 2. Location of strand in the case of mass spectrometric (a) and thermocouple (b) measurements.

Combustion products were sampled using aluminum probe, external surface of which was covered by Al$_2$O$_3$ using method of electrolysis. Presence of large amounts of hydrogen chloride and water vapor (strong corrosion of ignition spiral in preliminary experiments indicates this) at enough high temperature may lead to destruction of sampling system and as a result to emergency situation with mass spectrometric set-up. In order to prevent destruction of the aluminum probe and decrease of final temperature of combustion products subsequent experiments were conducted in flow of gas mixture H$_2$/He (50/50 by vol%) with lower concentration of hydrogen. This allowed also to conduct temperature measurements. Total flow of gas mixture was equal to 41.6 cm^3/s (NTP). Premixed gas mixture was delivered to the combustion chamber with volume of ~ 1.5 l. The gas flow was controlled using gas flowmeter. The gas was delivered to the combustion chamber from the direction of observation window. This prevented condensation of water vapor on the observation window and allowed to record the combustion process on videotape. The initial distance

between strand and the probe was ~ 4 mm. After inflammation of strand and formation of stable luminous flame allover the strand surface, the stepper motor moved the strand to the probe with velocity exceeding the burning rate of AP. Especial measurements of the burning rate as a function of strand diameter were not conducted. But the local burning rate of AP strands with diameter of 8 mm in the flow of H_2/He (50/50 by vol%) at atmospheric pressure was equal to ~ 0.24 mm/s in the moment when strand diameter decreased from 5.6 to 5 mm. Correspondingly, the burning rate of AP strands with diameter of 5 mm in the flow of 100% H_2 was equal to ~ 0.53 mm/s in the moment when strand diameter decreased from 4.4 to 3.1 mm. The burning rate is determined as change of stand radius during the time. The characteristic value of AP burning rate in hydrogen obtained in Ref[6] in the same unit is close to our data and it was changed in the range of 0.32 mm/s to 0.59 mm/s during the decreasing of strand diameter from 3 to 1.6 mm. During combustion of AP strand in hydrogen a decrease of strand diameter accompanied by increase of strand temperature occurs, and as a result burning rate increases.

The measurement of intensities of mass peaks of combustion products and thermocouple's signal was conducted using equipment on the basis of CAMAC and PC. The accuracy of temperature measurements was ±5%. The accuracy of measurement of distance from the burning surface using video recording was ±0.05 mm. The accuracy of determination of composition of combustion products (in mole fractions) was ±10%.

RESULTS AND DISCUSSIONS

The combustion of AP in the flow of pure H_2 and with addition of He occurred as the combustion of spherical particle. Burning rate of AP strand depended on particle diameter and H_2 concentration. Time dependence of temperature near the AP burning surface for investigated cases of strand ignition and combustion are shown in Fig. 3. Thermocouple was in contact with strand surface before the ignition. Temperature profiles were obtained when the thermocouple moved from the burning surface with the burning rate of AP. In the case of inflammation and further combustion of AP strands (∅5 mm) in pure H_2 (curve 1, Fig.3) temperature of 1800 °C in gas phase was reached for ~ 1 s. So flame zone width was
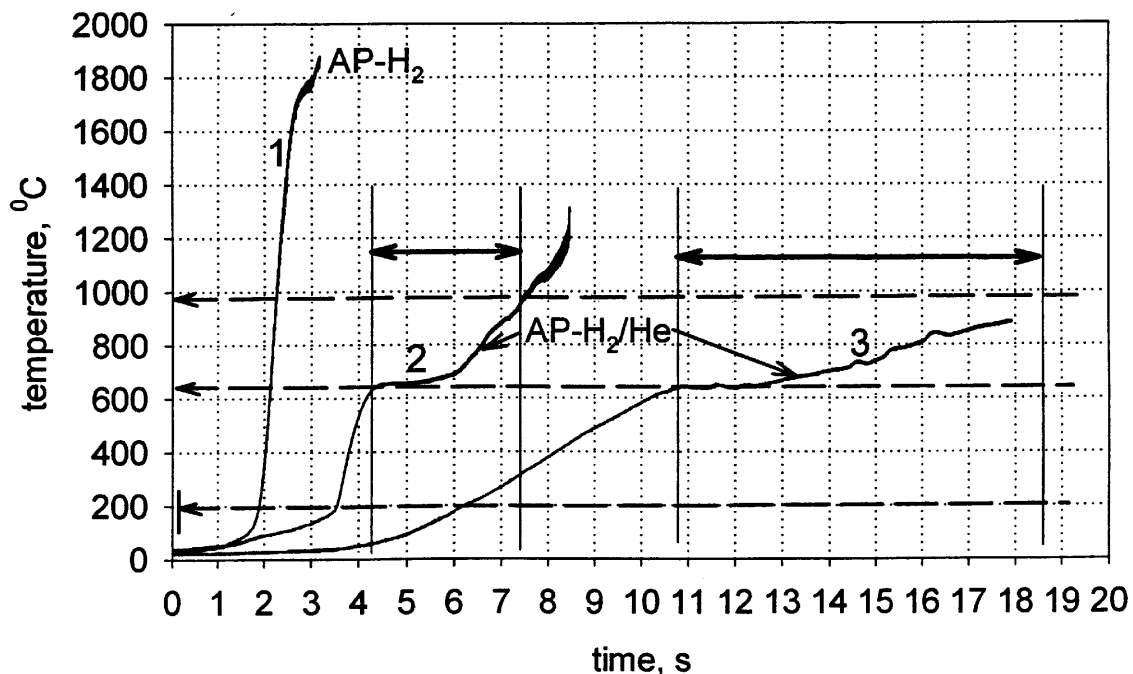

Figure 3. Time dependence of temperature near the AP strand surface for different concentrations of hydrogen in gas mixture and for different strand diameters (curve 1 - 100%H_2, ∅5 mm; curve 2 - H_2/He(50/50 by vol%), ∅5 mm; curve 3 - H_2/He(50/50 by vol%), ∅8 mm).

estimated as less than 0.25 mm. The decrease of H_2 concentration by two times for strands with diameter 5 and 8 mm allowed to extend ignition process in time. There are several regions in the temperature profiles 2 and 3 on Fig.3: increase of temperature near the strand surface from 20°C to ~ 200°C caused by heating using electrically heated spiral; monotonous growth of temperature from 200°C to ~ 650°C; following temperature growth up to 950-1000°C, when thermocouple lost contact with strand surface. If the heating of strand was ceased before temperature had reached 200°C, then ignition process stopped. If this temperature was reached, then ignition process did not depend on heat flow from igniter, and flame propagated allover the strand surface. The decrease of strand diameter was not observed during the fast surface propagation of flame. The inductive time with duration of 1-2 s was observed, when temperature near the strand surface reached ~ 650°C. During this time a flame appeared in different sites above the strand surface. The further increase of temperature to ~ 1000°C relates with formation of stationary flame allover the surface and beginning of strand burning surface moving. It can be assumed that AP surface temperature during combustion in mixture of hydrogen and helium is equal ~ 650°C and exceed that of preheated AP on 150 degrees.[7] On the one hand this difference can be associated with insufficient resolution of the thermocouple and existence of large thermal gradient near the AP burning surface. On the other hand the presence of hydrogen near the burning surface and possible heterogeneous reaction of hydrogen and AP on it surface can increase AP surface temperature in system AP-H_2 as distinct from preheated AP combustion in inert atmosphere.

Temperature profile in AP-H_2/He flame far from burning surface is presented in Fig. 4. It was obtained in different experiments with using thermocouples unmovable relatively the strand. Strand surface moved away from the thermocouple with velocity equal to the burning rate. Initial distance between the surface of AP strand ($\varnothing$ 8 mm) and thermocouple was equal to 0.5 and 1 mm in different experiments. Flame temperature at a distance of 0.5 mm was equal ~ 1000°C and slightly changed at the distance of 0.7 mm from AP surface. Then temperature increased and at a distance of 1.7 mm reached maximum value of ~ 1750°C and after this began to decrease on the distance 2.1 mm. So, at a distance of ~ 0.7 mm from AP strand surface the zone of chemical reactions with width of ~ 1 mm was detected. The temperature increased by ~ 750 degrees in this zone. It is necessary to note that maximum value of temperature in flame of AP-H_2 system is achieved at ~1.7 mm from the burning surface of AP (Fig. 4). This distance coincides with results of calculations presented in Ref[6]. However values of maximum temperature differ by 450 degrees (~2200 °C (Ref[6]), ~1750 °C (this paper)). Lower value of temperature in experiment probably relates to lower concentration of hydrogen (in our paper hydrogen was diluted by helium by 50%).

Profiles of intensities of mass peaks of combustion products in AP-H_2/He flame at atmospheric pressure as a function of time are presented in Fig. 5. It is known that mass spectra of different species can have coinciding masses as result of fragmentation. In Fig. 5 profiles of intensities of masses for individual species are presented in view of the contributions of fragmentary ions from other species. Diameter of AP strands was equal to 5 mm, and the overall time of experiment was ~ 8 s. This time is from the moment of appearance of mass peaks 44(N_2O) and 18(H_2O) till touching of the probe by AP strand surface. Initial distance between the strand and probe was equal to 4 mm. It did not change during the time of formation of steady state flame around the strand from ~2 to ~7 s (Fig.5), which was equal to ~ 5 s.
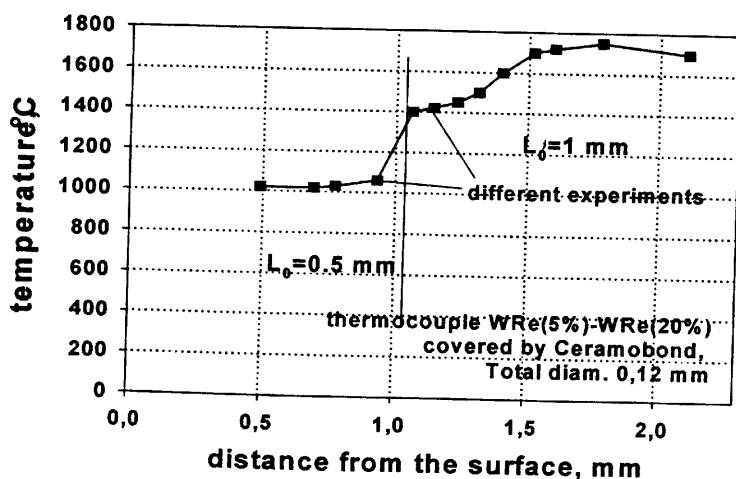

Figure 4. Temperature profile in AP-H_2/He (50/50 by vol%) flame at 0.1 MPa (diameter of AP strand 8 mm).
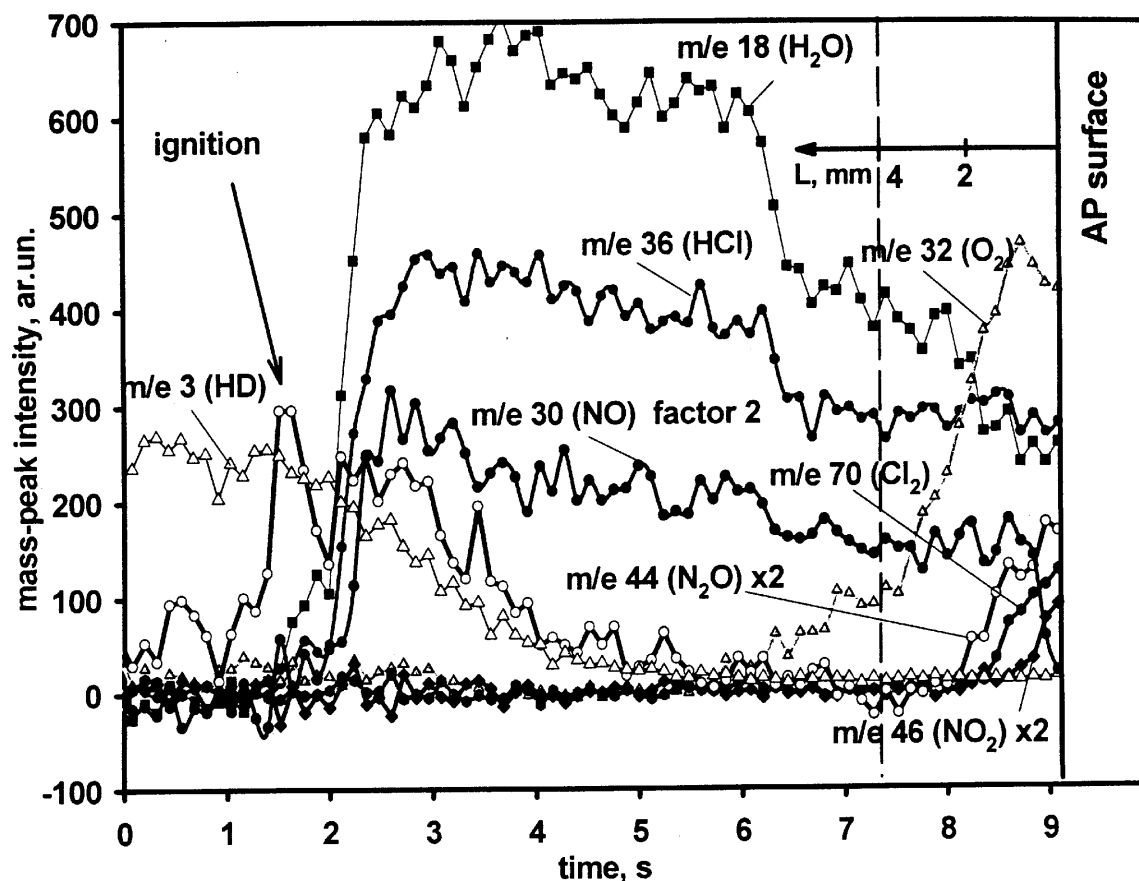
Figure 5. Profiles of intensities of mass peaks of combustion products in AP-H₂/He flame at atmospheric pressure as a function of time and distance from AP burning surface.

This time correlates with time of reaching of the temperature about 1000°C near the AP burning surface at temperature measurements (curve 2, Fig.3) using strands 5 mm in diameter. Dotted line on Fig.5 shows the time when strand began moved toward the probe with the speed (~2.4 mm/s) exceeding AP burning rate. Dependence of mass peaks intensities (18, 36, 30, 44 and 3) in the range of time from ~2 to ~7 s conducted with the ignition and combustion processes. In this case the probe sampling took place on the distance 4 mm from the strand, so these intensities of mass peaks correspond to the final product composition. Decreasing of mass peaks intensities shown on Fig.5 on time 6.5 s is a result of temperature increasing in the place of sampling during the flame propagation. The following mass peaks was detected during the experiment: 17(NH₃), 18(H₂O), 30(NO), 32(O₂), 36(HCl), 44(N₂O), 46(NO₂) and 70(Cl₂). Mass peaks 51 and 52(ClOH), 67(ClO₂), 83 and 100(HClO₄) was not detected. Intensities of mass peaks 28 (N₂) and 2 (H₂) were not measured, because values of these intensities were much higher than intensities of main combustion products and exceeded the dynamical range of system of data acquisition. It relates with technical characteristics of ion pump, which was used for pumping-out of ion source chamber of mass-spectrometer. Concentration profile of H₂ was measured using intensity of peak with m/e 3 (HD).

Profiles of mass peaks intensities or proportional to that concentration of combustion products near the AP strand surface obtained during the scanning of flame is of especial interest. It was determined that in zone adjacent to the AP surface at a distance (L, mm) of ~ 1 mm from burning surface an increase of concentration of O₂ (m/e 32) and NO (m/e 30), partial consumption of Cl₂ (m/e 70) and N₂O (m/e 44), complete consumption of NO₂ (m/e 46) take place. Complete consumption of Cl₂ and N₂O, slight increase of concentration of HCl, further consumption of O₂ and formation of H₂O at a distance from 1 to 2 mm take place. Ammonia was not observed near the surface of AP during the combustion in hydrogen atmosphere. In Ref[7] was shown that near the burning surface exists thin zone of chemical reactions including NH₃, ClOH, HClO₄ and ClO₂ which are complete consumed on distance about 100 microns from burning surface at pressure 0.06 MPa. The width of zone will decrease to about 50 microns at 0.1 MPa. So, it is real to suppose that system AP-H₂ has the same zone at 0.1 MPa too. The temperature increases to ~1000°C in this zone. However, the resolution of the thermocouple and the probe much exceeds the width of supposed zone. So this zone was not detected.

Unfortunately, in literature there are no mass spectrometric experimental data on flame structure of AP-H_2 system. The only (known to us) paper[6] contains solely emission intensity distributions (in arbitrary units) of H_2O, ClO and OH, which are in qualitative agreement with results of calculations[6]. The composition of combustion products of AP in the flow of hydrogen/helium near the AP surface and at the distance of 4 mm is presented in Table 1. The element balance near the surface is in the satisfactorily agreement (~15%) with initial balance and corresponds to the pure AP, i.e. it can be assumed that concentration of N_2 near the surface is small. The composition of combustion products of AP in inert atmosphere at the end of thin zone adjacent to the burning surface at 1100 °C is presented in Table 1 too.[7] Experimental data of Ref.[7] is close to obtained data of system AP-H_2 near the AP surface. It is noteworthy that mole fraction of N_2 in composition of combustion product is equal to 0.03, and this coincides with our estimations.[7] The main products of combustion AP in hydrogen atmosphere are H_2O, HCl, O_2, NO_2, N_2O and Cl_2. With experimental accuracy the concentrations NH_3 near the surface are equal to zero and coincide with data of Ref[7]. This fact justifies our supposition about existence thin zone in AP-H_2 flame.

Results of calculations[6] do not agree with experimental data Ref[7] and data of this paper on composition of combustion products of AP at the same temperature (~ 1100°C). It was obtained in our experiment and in Ref[7] that N_2O is present at temperature ~1100 °C and NH_3 is absent. The model gives opposite results. Besides, as distance from the burning surface increases the formation of significant amount of NO, which does not change in high-temperature zone, occurs (Fig. 5). Such behavior coincides qualitatively with results of model[6] but differs quantitatively (see Table 1). These facts point out that the combustion model proposed for AP-H_2 system should be corrected.

Table 1
Composition of combustion products of AP-H_2/He system at 01 MPa (in mole fraction)

	T, °C	H_2O	HCl	O_2	NO_2	N_2O	NH_3	N_2	NO	Cl_2	N:H:Cl:O (0.25:1:0.25:1)
Near the AP surface[a]	1000	0.44	0.11	0.16	0.16	0.06	0	NM	0	0.05	0.28:0.99:0.21:1.14
L=0.2 mm Ref.[7]	1100	0.47	0.16	0.16	0.07	0.03	0	0.03	0.05	0.03	0.24:1.10:0.22:1.01
Near the AP surface, calc. Ref.[6]	1127	0.31	0.15	0.23	0.09	0	0.05	0	0.03	0.05	0.24:0.99:0.25:0.98[b]
L=4 mm[a]	NM	0.62	0.12	0.04	0	0	0	NM	0.20	0	0.20:1.36:0.12:0.9
L~1.5 mm, calc. Ref.[6]	2200	0.52	0.15	0.03	0	0	0	0.05	0.05	0	0.15:1.33:0.15:0.67[c]

* - this paper
** - taking into account radical NH (mole fraction ~ 0.07)
*** - taking into account radical OH (mole fraction ~ 0.04) and H_2 (mole fraction ~ 0.05)
NM - not measured

At a distance of 4 mm, where concentration of O_2 is equal to 25% from initial concentration, the element balance by H differs from initial by 40%. This mainly relates to the reaction between O_2 (product of AP gasification) and ambient H_2 and as a result temperature in flame significantly increases.

CONCLUSIONS

Results of conducted investigations can be used for testing of combustion model and provide the new base information for understanding of combustion chemistry of nontraditional propulsion system.

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DISCUSSION

Question/Comment:

Reviewer No. 1

I had difficulty understanding some of the results as shown, for instance, in Fig 5 which plots the concentration as a function of time and distance from the AP in a two dimensional plot. As time increases, the O_2 concentration increased. Due to combustion, one would expect it to decrease, unless it was being liberated from $HClO_4$ fragments, in which case the decrease due to combustion would be delayed. Table 1, however, shows O_2 decreasing with distance from the surface. I think a little more analysis would be appropriate, and certainly more enlightening.

Author's Reply:

In Fig. 5 axes of time and distance from burning surface have opposite directions, i.e. concentration of O_2 increases in time, but the distance to the burning surface decreases. Mass peak with m/e 32, present in flame, does not relate to $HClO_4$, as it is absent in mass-spectrum of this species. In mass spectrum of $HClO_4$ (at 70 eV) present following mass peaks: m/e 100 ($HClO_4$), m/e 83 (ClO_3), m/e 67 (ClO_2), m/e 52 (HClO) u 51 (ClO).

Questions/Comments:

Reviewer No. 2

1. *The structure of $AP-H_2$ flame is still unclear. The authors should point out modifications in their experimental setup to improve our knowledge of $AP-H_2$ Combustion.*
2. *The strong effect of strand diameter observed should be given more attention and better explanation.*

Author's Reply:

1. *This paper is devoted to investigate the flame structure of a new interesting configuration ($AP-H_2$ system) using the method of molecular beam mass spectrometry (MBMS) developed in our laboratory. The set-up was not modified specially for conducting of these experiments. The width of narrow zone near AP surface was estimated around ~ 60 microns. Therefore, further progress in investigation of $AP-H_2$ flame by MBMS method can be connected with using probe with orifice ten times less than the width of the zone, i.e. ~ 6 microns. Unfortunately, the accessible diameter of probe orifice is only ~ 10 microns. Producing probes even with such orifices meets considerable technical difficulties.*
2. *During combustion of AP strand in hydrogen, a decrease of strand diameter accompanied by increase of strand temperature occurs and as a result burning rate increases. Temperature, measured inside the strand (in the center of the spherical strand of AP), was equal to 220 °C at ignition and then increased up to 900 °C [Saburo Yuasa, Satoshi Yushina, Tomohiro Uchida, Noriko Shiraishi, "Ignition and Combustion of Ammonium Perchlorate in a Hydrogen Atmosphere". Proceeding of the Combustion Institute, Vol. 28, 2000, pp.863-870.], that resulted in increase of burning rate by several times. Another factor influencing the burning rate of AP in atmosphere containing hydrogen is concentration of H_2. Increase of H_2 concentration leads to increase of final temperature of diffusion flame and to approach of zone of maximum flame temperature to the strand surface. The latter leads to increase of heat feedback from gas phase to the burning surface. It also results in increase of the burning rate.*