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REVIEW PAPER

APPLICATION AND PROPERTIES OF ALUMINUM IN ROCKET PROPELLANTS AND PYROTECHNICS

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Abstract

Aluminum is the third most abundant element in the Earth's crust. This metal is very reactive and is characterized by high heat of combustion. Aluminum is widely used in various technical fields such as: space technology, production of common use items, cars, airplanes, high energy materials. Aluminum in the shape of flakes or granulated with different particle size (nano- and micro-) is used in different mixtures. Aluminum powder is used as a component in rocket propellants, pyrotechnics, primary or secondary explosives. Aluminum as one of the components of solid rocket propellants acts as metallic fuel and the particle size of aluminum influences the utility properties of products. The particle size and aluminum content affect the viscosity of a rocket propellant mixture, burning time, ignition temperature, ignition delay time and specific impulse. This paper discusses the influence of modifications of the external layer of aluminum on the properties of aluminum combustion in rocket propellant. Aluminum acts as a fuel in pyrotechnic mixtures. The size of aluminum particles can affect parameters such as decomposition temperature and ignition temperature in pyrotechnic mixtures. One type of pyrotechnic mixtures consists of thermites. Introducing nano-sized aluminum to thermites creates mixtures with new properties called superthermites, nano-thermites or metastable intermolecular composites (MICs). Nano-thermites can possess different combustion characteristics than standard thermites. The firecrackers' sound volume depending on the used particle size of aluminum was also studied.

Keywords: thermite, particle size, specific impulse, ignition delay time.

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INTRODUCTION

Aluminum is the most abundant metal in the Earth's crust. A thin layer of Al oxide is formed on the surface of this metal. This superficial layer prevents further reaction with oxygen and other chemical agents, because it is impermeable and non-conductive (RAMASWAMY, KASTE 2005). Aluminum can be capped by various materials, e.g. oleic acid, glycidyl azide polymer – GAP (GROMOV et al. 2006, SIPPEL et al. 2013, GONG et al. 2014). This allows us to change reactivity of the materials or other properties (e.g. from hydrophilic to hydrophobic). Nano-Al is more reactive than micro-Al. Alex is a nano-Al powder (about 70-100 nm particle size), which is formed by the wire explosion method (SANDEN 1998).

Heterogeneous solid propellants are used in chemical rocket, booster and gas-generator engines. The basic components of propellants are: oxidizer, liquid fuel, burning rate modifiers and metal powders. Oxidizers are ammonium perchlorate (AP), ammonium nitrate (AN). Liquid fuels are hydroxyl-terminated polybutadiene (HTPB), polyurethane, carboxy-terminated polybutadiene. Nitramines such as octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX), 1,3,5-trinitroperhydro-1,3,5-triazine (RDX), 2,4,6,8,10,12-hexanitro-2,4,6,8,10,12-hexaazaisowurtzitane (CL-20) are added to increase the specific thrust in high-energy propellants. The specific thrust is defined as a thrust achieved from a single fuel flow rate in one second of engine work. The important parameters in rocket designing are the operation range and flight time.

Another field of the application of aluminum is pyrotechnics. Pyrotechnics are multicomponent mixtures containing fuel and oxidizer. Aluminum is widely used in military pyrotechnics: IR flares, gas-generators, thermite burners used for mine destruction, nano-thermite and fireworks (WHEATLEY 1999, VALLIAPPAN et al. 2005, AZHAGURAJAN et al. 2014). Al is used as one of the additives to IR flares, due to the presence of Al_2O_3 in combustion products of Al which is one of selective emitters (DANALI et al. 2010). Flares of this type comprise a pyrotechnic mixture based on Mg, Teflon and Viton combined with a mixture including Al, boron, hexamethylenetetramine, potassium nitrate (KNO_3) and AP (KOCH 2001).

For many years our Department of High Energetic Materials has conducted research on high energy mixtures containing metals. Studies on pyrotechnical mixtures for creating a burner for nonexplosive mine clearance were conducted (MAKSIMOWSKI et al. 2012, GOŁOFIT et al. 2015). The aim of the research was to develop cheap, easy to make pyrotechnical mixtures which would burn through a mine casing as well as the explosive contained within. Three compositions were proposed containing aluminum with different oxidizers: a mix of iron oxide with Teflon, gypsum and potassium perchlorate. The mass which was the cheapest and easiest to produce was composed of aluminum and gypsum. This mixture effectively burned through a casing, however, due to large amounts of solid residue being produced, it could not be used to clear mines effectively. The mixture of Al, potassium perchlorate and shellac proved to be the best, as it burned through a casing and burned the explosive within. In the study by Syczewski (1994) detonation parameters for the mixture of carbon tetrachloride, zinc oxide and Al are presented. Detonation velocity depends on the density of a mixture used. The authors measured detonation velocity for densities above 1800 kg m^{-3}. One of the important parameters characterizing heterogeneous propellants is the specific impulse. The amount of magnesium in fuels influences the specific impulse as shown by Zygmunt et al. (2014). In heterogenic fuels based on HTPB, the specific impulse decreases with the increased amount of polymer, whereas a reverse correlation was observed for GAP. The optimal amount of magnesium is between 20-45 wt% for the following oxidizers: polytetrafluoroethene (PTFE), potassium perchlorate and iron(III) oxide. Thermal decomposition of PTFE is dependent on the type of a metal used (Książczak et al. 2003). A double stage of mass loss was observed for a mixture of PTFE with iron. Iron does not change the kinetics of the decomposition process of PTFE. Thermal decomposition of a mixture of PTFE and silicone in 550-950 K range is a single-stage, mass loss, exothermic process. Addition of Si accelerates the decomposition rate of PTFE.

ALUMINUM IN ROCKET PROPELLANT

The granularity of aluminum influences the properties and preparation process of propellants. In the propellant production process, solid particles are mixed with liquid binders. The blended mass is poured into molds, where it is conditioned and cured at high temperature. A casting process determines the structure of propellants. Adverse rheological parameters may cause cracks and pores in a propellant. Propellant defects can lead to detonation of a propellant due to a rapid increase of the combustion area. Therefore, it is important to choose the viscosity of a mixture properly (Sutton, Biblarz 2010). The rheological properties of a mixture depend on the geometry and content of solid components (Jain et al. 2009). The viscosity of a mixture increases with the rising of a micro-Al particle size or the rising of the volume fraction of micro-Al (Arefinia, Shojaei 2006). The Newtonian dependence of viscosity was observed in a range up to 50% volume fraction of Al for a mixture of nano-Al dispersed in HTPB (Teipel, Förter-Barth 2001). The increase of viscosity was obtained by other methods, e.g. when ammonium perchlorate was covered by copolymers (Nandagopal et al. 2009).

A variety of methods were studied regarding the changes to parameters of aluminum combustion in propellants. Al particles were coated with another metal (Bocanegra et al. 2007), organic substances (Lewis et al. 2011) or different sizes of Al particles were used (Meda et al. 2007, Jayaraman et al.

2011). Combustion mechanisms of aluminum and its systems with oxidizers and binder were examined (Jayaraman et al. 2010). The agglomeration of aluminum particles on heated propellant was observed. As the result of heating an Al/HTPB sample the solid particles of nano-Al (50 nm) accumulated with molten aluminum and created clusters approximately 4 μm in size. The oxide coating of examined micro-Al was detached from Al particles in the heating process. No shift of aluminum particles was observed in cured propellant with toluene diisocyanate (TDI). Nano-Al samples prior to heating were uniformly dispersed in the binder and were not clustered, which was observed in post-heated samples. Large agglomeration of nano-Al particles on the propellant surface was observed.

The flame structure of AP/HTPB/15 μm-Al propellant was studied (Brewster, Mullen 2010). Over the pressure range of 0.41-1.52 Mpa, the aluminized laminates burned at lower burning rates than non-aluminized laminate. Al_2O_3 coated Al particles are protected from ignition by this layer. The temperature of approximately 2900 K was sufficient to melt the protective oxide coating and ignite aluminum. The ignition time and burning time were tested for systems containing Al with ammonium nitrate or ammonium chloride (Lewis et al. 2011). Nano-Al (20-40 nm), aluminum oxide nanoparticles (40-50 nm) and oleic-acid-passivated Al nanoparticles (approx. 30 nm) were used. The oleic-acid-passivated Al nanoparticles were more reactive than aluminum oxide nanoparticles. Al particles with 56 nm thicknesses of Ni coating were characterized by reduced ignition delay and burning time (Bocanegra et al. 2007). The ignition delay time for Al coated by Ni significantly decreased with the increasing of Ni mass fraction from 0 to 3%. Further increase in the amount of Ni did not change the ignition delay time. The burning time increased with the rising of Ni mass fraction from 0 to 5%. Above 5 wt%, the combustion time decreased up to about the same value as for uncoated Al.

Spherical agglomerates of aluminum connected with the gelled nitrocellulose (NC) can be an alternative to nano-Al (Wang et al. 2013). The achieved gelled microspheres of mean sizes 2, 3.1 and 11.1 μm contained 3, 6.5 and 10 wt% NC, respectively. With the increasing NC content, the burning time increased and the ignition delay time decreased. Al nanoparticles containing 10 wt% NC were characterized by shorter ignition time compared with Al nanoparticles.

The influence of Al size and binder nature on burning rates was studied (Meda et al. 2005). The size of Al particles was analyzed by X-ray photoelectron spectroscopy, X-ray diffraction and scanning electron microscopy. The propellant burning rate was higher for Al nanoparticles than micro-Al. The lower ignition temperature and shorter ignition delay time were obtained for Al particles of smaller dimension. It was observed that the burning rate under the pressure of 1, 3, 7 MPa was not linear with the increase in the surface area of Al powder. The effect of nano-Al on the plateau-burning of the propellant HTPB/AP under different pressures was examined (Jayaraman et al. 2009). It was shown that the non-aluminized 20 μm fine-AP/HTPB (60/40) matrix did not burn at low and intermediate pressures when cured by TDI. An uncured matrix burned at low pressures. An irregular burning at high pressure was observed for a propellant cured with isophorone diisocyanate. Matrices containing micro-Al or Al nanoparticles with fine AP particles burned in the entire test pressure range, irrespective of the curing agent. The burning rate decreased with the increase of an Al particle size. The highest burning rate was observed for a propellant containing Al nanoparticles, irrespective of the curing agent. The effect of the aluminum content from 10 to 18% on the burning rate of a propellant under different pressures was examined (Jayaraman et al. 2009). Linear dependences of the burning rate on the Al content were not observed. The effects of the Al content (from 0 to 20%) and particle size (spherical: 7.9 and 10 μm or irregular shape: 25 μm) on the burning rate of HTPB-based composite propellants were examined (Ozkar et al. 1996). It was demonstrated that both the burning rate and pressure exponent decreased with the increasing Al content of a propellant.

Aluminum in the combustion process is oxidized to aluminum oxides (γAl_2O_3, $\delta^*Al_2O_3$ and αAl_2O_3) (Fedotova et al. 2000, Meda et al. 2005, Galfetti et al. 2007). Condensed combustion residues γAl_2O_3 and $\delta^*Al_2O_3$ were observed in the combustion process at pressures 0.1 and 3 MPa for Al/HTPB/AP propellant containing 0.15 μm-Al (Galfetti et al. 2007). Unreacted aluminum was observed as a main product of the combustion process at 0.1 MPa pressure for propellant containing 30 μm size Al particles. The main combustion products at 3 MPa pressure were γAl_2O_3 and $\delta^*Al_2O_3$ obtained in amounts of 48.4 wt% and 40.0 wt%, respectively. The rest were aluminum metal and αAl_2O_3 (Meda et al. 2005, Galfetti et al. 2007). AlN (Fedotova et al. 2000) and Al-O-X systems, (where X could be carbon or nitrogen) (Ji, Shufen 1999) as combustion products were achieved from a propellant containing HMX, AP, Al and binder. These products were formed in the reaction of aluminum with HMX decomposition products – nitrogen oxides (NO_2 and N_2O).

The propellants based on Al/HTPB/AP were examined (Florczak, Witkowski 2006, Cerri et al. 2009, Florczak, Cholewiak 2011). The dependence of the specific impulse on the Al content is presented for an AP/HTPB/Al propellant in Figure 1. The specific impulse rises up to 18% of the Al content and reaches the value of 263.3 s (Ozkar et al. 1996). Further increasing Al content causes the decrease of the specific impulse value in the studied propellant. The value of the specific impulse equals 249.7 s for a propellant without aluminum.

Table 1 summarizes the specific impulse for different content and size of Al particles in an AP/Al/HTPB propellant (Cerri et al. 2009). Two different particle sizes of Al (18 μm and 100-200 nm) were added to the examined propellant. The specific impulse was calculated by the ICT-Thermodynamic code together with the ICT Termochemical Database (ICT-Fraunhofer Insti-

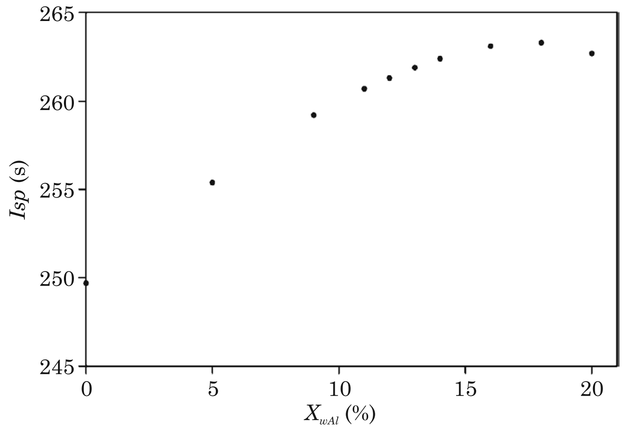

Fig. 1. The dependence of the specific impulse (*Isp*) on the aluminum content for an AP/Al/HTPB propellant (OZKAR et al. 1996)

Table 1

The specific impulse (*Isp*), content (x_{wAl}) and size of used aluminum in an AP/Al/HTPB propellant (CERRI et al. 2009)

Size of Al	x_{wAl}	*Isp* s^{-1}
Micro	0.06	249.3
Micro	0.12	247.3
Nano	0.06	256.3
Nano	0.12	254.6

tute for Chemical Technology). The specific impulse of cured propellant decreases with an increasing Al content, which is contrary to the authors (OZKAR et al. 1996). The increase of the specific impulse was obtained for a smaller particle size of aluminum.

The safety of use and storage of a rocket propellant are related to the propellant's properties. The impact sensitivity was determined for propellants containing Al and high-energy materials, such as CL-20 and RDX, which were used with a binder 3-(azidomethyl)-3-methyl oxetane/3,3-bis (azidomethyl)oxetane. The addition of aluminum decreases the value of impact sensitivity (~11 J) (LUMAN et al. 2007). The impact and friction sensitivity changes with the size and content of aluminum in an Al/HTPB/AP propellant (CERRI et al. 2009). The friction sensitivity increases with the ageing time. The propellant containing 12 wt% of nano-Al is characterized by the highest friction sensitivity after 20 days of ageing at 363 K compared to other samples. A propellant with 18 μm sizes particles of Al had the lowest friction sensitivity. There is a weaker dependence of the particle size of aluminum on the impact sensitivity.

ALUMINUM IN PYROTECHNIC MATERIALS

The particle size of aluminum and the influence of aluminum fineness on thermal behavior are considered in the studies of pyrotechnic mixtures. The addition of nano-Al, regardless of the metal particle sizes, does not affect the decomposition temperature of a mixture composed of Al, KNO_3 and sulfur. Nevertheless, the reduction of the particle size decreases enthalpy during the decomposition. The increase of the melting point of $KClO_3$ is caused by using larger Al particles (18 μm) and increasing the powder content in mixtures based on Al and $KClO_3$. The increase in the Al content in mixtures raises the temperature of their ignition. The increase of the Al content in a mixture causes an increase in the amount of Al_2O_3 in the reaction products. Thermal capacity of aluminum oxide is higher than that of other decomposition products and it is observed as broadening of the signal. It is noticeable as compared to mixtures with smaller Al content (Azhagurajan et al. 2011). Similar studies were performed for mixtures composed of Al with different particle sizes (5 and 18 μm) and AP. The increase of the decomposition temperature with an increasing Al content was observed regardless of the particle size (Pourmortazavi et al. 2006).

Metastable intermolecular composites (MICs) are pyrotechnic mixtures also called nano-thermites, or superthermites. The differences in terminology are due to various particle sizes of such mixtures and a faster reaction rate in comparison with standard thermites. Nano-thermite systems composed of Al and: CuO, MoO_3, Fe_2O_3, WO_3 were investigated (Danen, Martin 1993, Bazyn et al. 2007, Sarawadekar, Agrawal 2008, Piercey, Klapoetke 2010). Fluoropolymers such as PTFE, Viton A or poly(vinylidenefluoride) (PVDF) were also added to MICs (Dixon et al. 1998, Li et al. 2015, 2016). Fluoropolymers can act as oxidizer and reducer in the process of thermal decomposition. There are excellent oxidizer for metals and reducer for metal oxides. These polymers also improve mechanical properties of nano-thermites.

Spitzer et al. showed that WO_3/Al nano-thermite containing only nano -particles are highly reactive. The rate of combustion can reach up to 7.3 m s^{-1}, the ignition delay time was less than 2 ms (used CO_2 laser, λ=10.6 μm). Dried WO_3/Al has an extremely high sensitivity to friction stress (<4.9 N). This sensitivity threshold is significantly higher when nano-Al contain a small amount of adsorbed water. However, these nano-thermites are insensitive to impact (42.2 J) (Spitzer et al. 2010). Changing the size of molecules from micro- to nano- may significantly affected the temperature of thermite ignition. A mixture of MoO_3 based on the particle size of 100 nm and Al of 40 nm size ignites at temperature of 731 K. The temperature of a mixture composed of MoO_3 with the same size and Al of 10-14 μm size is much higher (1228 K) (Pantoya, Granier 2005). The composites consisting of CuO nano--tubes and particles of Al can be characterized by the combustion wave velocity of 1650 m s^{-1}. This value increases to 1900 m s^{-1} for composites consisting of nano-wires and particles of Al. The highest value equals 2400 m s^{-1} for self-organizing composites (Shende et al. 2008).

Studies on firecracker sound volume depending on the used particle size of Al were also performed. The results showed that reducing the particle size improves the efficiency of a mixture to produce sound. For 250 μm and 150 μm-Al, a flash was observed instead of a sound, and sound was produced for 63 μm-Al. The sound level was tested for Al+KNO_3 mixtures with 63-250 μm-Al. It was found that a change in the size of KNO_3 had much less influence on the sound level than changes in the particle size of Al. The generation of sound by this kind of mixture depends not only on its composition and particle size of ingredients but also on the particle shape, density and packing of the components (Azhagurajan, Selvakumar 2014).

CONCLUSION

Aluminum with different particle sizes is commonly used in rocket propellant and pyrotechnic mixtures. The particle size of Al and Al content affect many properties of products: viscosity, burning rate, ignition delay time, specific impulse or sensitivity to external stimuli. The viscosity of a mixture rises with the increasing size of micro-Al and the volume fraction of metal. Nano-Al, nano-Al_2O_3, oleic-acid-passivated nano-Al, Ni coating Al and spherical agglomerates of Al connected with gelled NC were described. The oleic--acid-passivated nano-Al were more reactive than nano-Al_2O_3. Ni coated Al were characterized by reduced both: ignition delay time and burning rate. The highest burning rate was observed for a rocket propellant containing nano-Al, irrespective of the curing agent. The linear dependences of the burning rate on the Al content were not observed. Al is oxidized to γAl_2O_3, $\delta^* Al_2O_3$ and αAl_2O_3 in the combustion process. The specific impulse increases with a smaller particle size of Al in a rocket propellant based on HTPB and AP.

The addition of nano-Al does not affect the decomposition temperature of a mixture composed of Al, KNO_3 and sulfur. The reduction of the Al particle size decreases enthalpy during decomposition. The increase of the Al content in mixtures raises the temperature of their ignition. The ignition temperature varied from 731 K to 1228 K depending on the Al particle size. Reducing the particle size improves efficiency in the production of sound by firecrackers.

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