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Laser Ignition and Combustion Properties of Composite Propellant Containing Nanometal Powders

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The effect of nanometal powders (nano-grain-size aluminum powders and nanograin size nickel powders) on the ignition delay time of ammonium perchlorate composite propellants has been examined in detail within a carbon dioxide laser ignition system. A comparison of different experimental techniques for combustion-related properties evaluation, including burning rates, deflagration heat, and ignition temperatures of the ammonium perchlorate propellants containing nanometal powders is presented. A thermogravimetric analyzer is employed to investigate the thermal behavior of nano-grain-size and general-grain-size aluminum powders heated in air. The content of active aluminum in the combustion residue was measured by means of the titration. The results show that, with the cooperating effect of nanonickel powders, the addition of nanoaluminum powders in propellants can greatly increase the burning rate, shorten the ignition delay time, and improve the combustion efficiency of aluminum in the combustion of propellant. The individual application of nanometal powders (nanoaluminum or -nickel powders) displayed an intermediate effect, whereas the general-grain-size nickel powders had the least effect. The effect of nano-grain-size Al is determined to be on the solid-phase ignition of the propellant. The effect of nano-grain-size Ni is determined to be on the gas-phase ignition of propellant initially and the solid-phase ignition subsequently.

Nomenclature

Al %	=	active aluminum content in combustion residue
$C_{\mathrm{KMnO_4}}$	=	labeled concentration $KMnO_4$
E_a	=	activation energy of ignition
g	=	gas
k	=	reaction rate constant
r	=	reaction rate
s	=	solid
T	=	temperature of measuring zone
t_i	=	ignition delay time
$V_{\mathrm{KMnO_4}}$	=	consumption volume of $KMnO_4$ in titrimetric process
W_{sample}	=	weight of combustion residue
$[Y]$	=	reactants' concentration

I. Introduction

NANOSTRUCTURE materials, defined as materials characterized by structural features less than 100 nm in average size, have exhibited high chemical reactivity associated with the ultrafine grain size of nanoparticles, which allows a number of metals to undergo reactions that were previously considered impossible. Therefore, there is a growing interest among scientists and engineers for approaching the synthesis and fabrication new types of propellants containing nanometal powders.[1–16]

In spite of the numerous studies on the beneficial effects of nanometal powders,[1–8,11–16] all of this research focuses mainly on the influence of the addition of nanoaluminum on the combustion properties of propellants. Little is known about the influence of other nanometal powders, and little is known about the ignition properties involved in nanometal powders in propellant. The ignition of solid propellants involves an array of intricate physiochemical processes under energetic stimuli. It is a subject similar to, but not the same as, combustion. The purpose of this paper is to discuss the influence of nanometal powders (nanoaluminum powders, and nanonickel powders) on the ignition properties of AP composite propellants at atmospheric and subatmospheric pressures. Some combustion-related properties evaluation, including burning rates, deflagration heat, and the ignition temperatures of AP composite propellants containing nanometal powders, are also presented.

II. Experiment

The basic formula of the samples studied consisted of HTPB/AP/Al, in which general-grain-size aluminum powders (G-Al) (13 μm) is 12%. The nano-grain-size aluminum powders (N-Al) (83 nm), nano-grain-size nickel powders (N-Ni) (68 nm), and general-grain-size nickel powders (G-Ni) (25 μm) were applied to compose four different formulas. The detailed formulations used in this study are shown in Table 1.

In Fig. 1, the experimental apparatus is shown schematically. A continuous-wave CO_2 laser with a wavelength of 10.6 μm was used. The sample ($4 \times 4 \times 3$ mm^3) was fixed on the strand in the combustion chamber. The initial temperature of the combustion chamber was adjusted to a setting temperature of 15°C. The output power of the laser was measured with a power meter just before each run. The level of incident heat flux onto the sample surface can be changed by changing the output of the laser. All heat fluxes were measured as incident radiation. The used ignition criterion in experimental studies is the first detection of a flame on the sample surface. The instant ignition was detected by a phototransistor, and the ignition delay time was defined as an interval between the start of irradiation and the time corresponding to the ignition.

To investigate the thermal behavior of aluminum particles in air, a Mettler TA2950 thermogravimetric analyzer was employed for thermogravimetry (TG) analysis at a scanning rate of 20°C/min in a dynamic air atmosphere. An almost constant sample mass of 2.0 ± 0.1 mg was used in all experiments. The flow rate of air in all TG analyzer experiments was 100 ml/min. TG–differential

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thermogravimetry (DTG) experiments on the two kinds of powdered aluminum with different particle sizes (10 μm and 90 nm) were carried out.

III. Results

A. Ignition Delay Time, Ignition Temperature at 1 Atmosphere and Deflagration Heat

The ignition delay time was measured with respect to two different incident laser energy fluxes (75.0 and 95.54 W/cm^2) at 1-atm air pressure. Table 2 lists the average value of the five experimental results. The propellant's deflagration heat was measured by the thermostatic oxygen bomb calorimeter in an N_2 atmosphere. At room temperature and 1-atm air pressure, the ignition temperatures of five samples were measured by thermocouple at the elevate-temperature rate of 10°C/min. The values of the experimental results are listed in Table 2.

It is generally observed that the ignition delay time of the same formulation decreases with the increase of transferring energy to the propellant. At the same rate of external energy, a comparison of the difference in ignition delay time between NM-1 and the other formulations has shown that the combined application of N-Al and N-Ni in NM-5 has the greatest influence on the ignition delay time among the formulations. Such influence has yielded a dramatic shift of the ignition delay time of NM-5 to a shorter time, 31 and 10% less than that of NM-1 at 75 and 95.54 W/cm^2, respectively. The individual application of nanometal powders (N-Al and N-Ni) in NM-2 and NM-4 displayed an intermediate effect, whereas the G-Ni in NM-3 had the least effect. The same tendency exists in the influence of metal powders on the ignition temperature of formulations shown in Table 2.

As compared with the basic formula NM-1, the deflagration heat of NM-3 and NM-4 has decreased 573.3 and 338.9 J/g, respectively, due to the relatively lower heating capacity of N-Ni (4088 J/g) compared with that of conventionally used Al (30,480 J/g) (Ref. 17).

As compared with NM-1, the deflagration heat of NM-2 with G-Al/N-Al = 1/1 increased 116.7 J/g due to N-Al's special active reaction ability. N-Al particles will be quickly heated to the surrounding gas temperatures and lead to sustained burning. As a consequence, the local heat point in the propellant can be formed easily and will strengthen the interaction between the oxidizer and aluminum powders. Thus, the addition of N-Al will lead to almost complete combustion of aluminum powder, which can further increase the propellant's thermal decomposition reaction heat.

A special case appeared in NM-5. As compared with NM-1, the deflagration heat of NM-5 with N-Al/G-Al = 1/1 and 2% N-Ni increased 118.5 J/g. It was almost the same as that of NM-2 without N-Ni. Note that the deflagration heat of NM-4 containing 2% N-Ni and without N-Al decreased 338.9 J/g. It is apparent that the combined effects of N-Al and N-Ni in the propellant can greatly improve the deflagration heat of AP/HTPB/Al propellants.

B. Ignition Delay Time at Subatmospheric Pressures

The ignition delay time was measured with respect to three different subatmospheric pressures (0.4, 0.2, and 0.1 atm) in the same incident laser energy flux 95.54 W/cm^2. The choice of high external energy fluxes in the experiment is taken into consideration as the probable cause of critical pressure at subatmospheric pressures.[18] In fact, nearly one-half of the samples cannot maintain self-sustained ignition at 0.1 atm. Thus, in Table 3, we only list the value of the ignition delay times at 0.4 and 0.2 atm, respectively.

The change of pressure has important affect on the measured ignition delay time. The decrease of pressure will increase the diffusion rate of the decomposition gas and reduce the collision frequency, as well as the chemical reactions rate.[18] The concentration of combustible mixtures in the gas phase is insufficient to produce the exothermic energy necessary for self-sustained ignition. The ignition will tend to be controlled by the gas-phase reactions, and because the reaction zone is moved away from the propellant surface,

Table 1 Formula of propellant samples

Sample	NM 1	NM 2	NM 3	NM 4	NM 5
HTPB	18	18	18	18	18
AP	70	70	70	70	70
G-Al	12	6	12	12	6
N-Al	0	6	0	0	6
G-Ni[a]	0	0	2	0	0
N-Ni[a]	0	0	0	2	2

[a] External addition.

Table 3 Ignition delay time at subatmospheric pressures (95.54 W/cm^2)

	Ignition delay time, s	
Propellant sample	0.4 atm	0.2 atm
NM-1	4.677 ± 0.014	6.272 ± 0.015
NM-2	4.593 ± 0.016	Partly ignited
NM-3	4.499 ± 0.015	5.520 ± 0.023
NM-4	4.411 ± 0.018	5.221 ± 0.020
NM-5	3.584 ± 0.010	3.826 ± 0.012

Table 2 Ignition temperature, burning rate, deflagration heat, and ignition delay time at different heat fluxes (1 atm) of propellants

	Ignition delay time, s			
Sample	75 W/cm^2	95.54 W/cm^2	Ignition temperature, °C	Deflagration heat, J/g
NM-1	1.345 ± 0.006	0.769 ± 0.006	317	6682.5
NM-2	0.981 ± 0.005	0.730 ± 0.008	310	6799.2
NM-3	1.383 ± 0.004	0.812 ± 0.005	318	6109.2
NM-4	1.246 ± 0.008	0.723 ± 0.003	309	6343.6
NM-5	0.930 ± 0.006	0.690 ± 0.004	293	6801.0

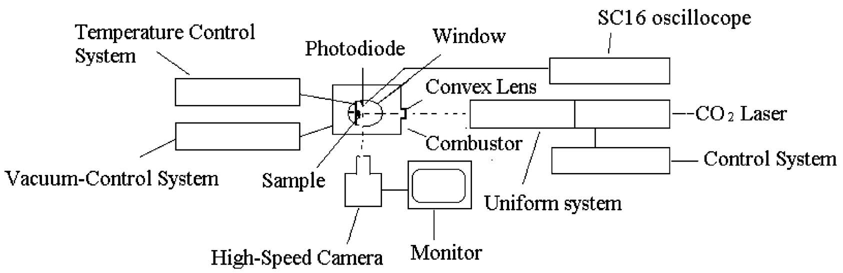


Fig. 1 Schematic of CO_2 laser ignition apparatus.

the heat feedback is then decreased to cause the generally observed longer ignition delay time. In our experiment, the different formulations will lead to the different ignition behavior at subatmospheric pressures.

The data indicate that, whether the pressure is high or low, sample NM-5's ignition delay time is the lowest. That is 23 and 39% less than that of NM-1 at 0.4 or 0.2 atm, respectively. The three other samples do not show a regular pattern as they did at 1 atm. Compared with that listed in Table 2, the subatmospheric pressure will result in more uncertainty for the ignition time test as shown in Table 3. The results suggest rather complicated ignition behavior of propellant containing nanometal powders at subatmospheric pressures. Such behavior needs more detailed and in-depth investigation.

C. Active Aluminum Content in Combustion Residues

The residue on the bottom of the chamber was collected and analyzed. Compared with other ingredients, the Al particles have a unique tendency to ignite less readily and accumulate on the burning surface, thus, forming relatively large agglomerates that burn relatively slowly after leaving the burning surface. For a larger-size agglomerate, it will take a longer time until burnout, and the Al combustion efficiency is expected to decrease. The active Al content in the combustion residue of the propellant is considered to be related to the combustion efficiency of Al in the propellant, and the general efficiency of the application of aluminum in the propellant formulation depends on the agglomeration intensity that can be characterized by the extent of aluminum conversion into oxide, that is, the mass content of free Al in condensed combustion products.

At present, the titrimetric method is commonly used to determine the metallic aluminum content in condensed combustion products. The chemical reaction of permanganatometric titrimetric method is described as follows.

Reaction 1:

$$3Fe_2(SO_4)_3 + 2Al = Al_2(SO_4)_3 + 6FeSO_4$$

Reaction 2:

$$2KMnO_4 + 10FeSO_4 + 8H_2SO_4 = 2MnSO_4 + K_2SO_4 + 5Fe_2(SO_4)_3 + 8H_2O$$

The resulting Fe^{2+} ions in reaction 1 are titrated by $KMnO_4$ in the acidic environment of H_2SO_4 via reaction 2. The point of equivalence is established visually with the appearance of pink coloring, indicating a surplus of $KMnO_4$. The equation to calculate the free Al in the combustion residue is shown in Eq. (1):

$$\mathrm{Al}\% = \frac{C_{\mathrm{KMnO_4}} \times V_{\mathrm{KMnO_4}} \times 26.98}{W_{\mathrm{sample}} \times 1000 \times 3} \times 100 \qquad (1)$$

The free Al in the combustion residue was calculated using Eq. (1) and is shown in Table 4. The burning rates of propellant at 6.84 MPa at a constant temperature of 20°C are also shown in Table 4. The burning rate of the propellant sample was not determined from the recorded images. Some trouble was encountered with the extreme brightness of the flame with formulations containing nanometal powders. Burning rates were then measured by the target line method. Measurements were performed with a strand burner pressurized with nitrogen. For the samples that burned unevenly, the rates were measured at multiple surface sites. The flame characteristics of the burning samples were observed in the combustion chamber, and the flame images are shown in Fig. 2.

The active Al content in the combustion residue was found to show a strong correlation with burning rate. As compared with the basic formula NM-1, the burning rates of all other formulas containing nanometal powders increased, and the active Al content in the combustion residues of the corresponding formulas containing nanometal powders decreased. A special case appears in sample NM-3 containing G-Ni, in which the burning rate and the corresponding active Al content all decreased as compared with that of NM-1. The reason for this special case lies in that the active Al content in NM-3 mainly decreases, not because of the increase of the Al's combustion efficiency, but because of the decrease of content of Al itself in the formulations.

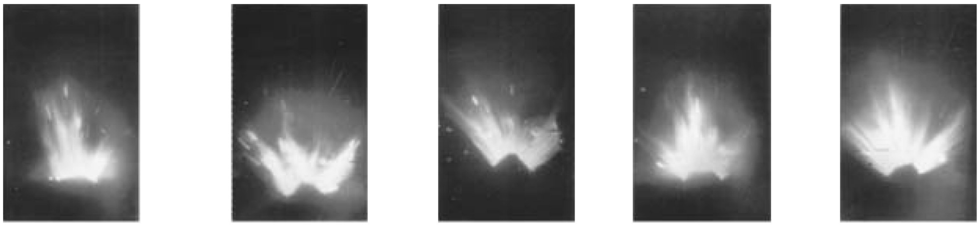
The burning rate enhancement is especially notable in NM-5. The individual application of nanometal powders N-Al and N-Ni in NM-2 and NM-4 displayed an intermediate effect, whereas the G-Ni in NM-3 had a decreased burning rate. Compared with NM-1, the burning rate of NM-5 has been increased by 23%, and such an increment (1.51 mm/s) is 260% larger than that of NM-2 (0.42 mm/s). The flame characteristics of the burning samples observed in the combustion chamber (Fig. 2) show that the combustion of NM-1 shows a large fraction of Al burning as very large agglomerates away from the surface. NM-1 has the largest amount of active Al in the condensed combustion products (Table 4), whereas sample NM-5 has the smallest agglomerates with a rather bright flame. The region above the surface was luminous, starting very close to the surface. NM-5 also has the smallest numeric value of active aluminum content in the combustion residue, 44% less than that of NM-1.

D. TG/DTG Results of Aluminum in Air

The TG and DTG results of aluminum powders are shown in Figs. 3 and 4, respectively. It is shown that aluminum particles in air have undergone a multstage process with the increase in temperature. The onset of the initial oxidation reactions of N-Al occurs at a threshold temperature of around 400°C. At around 628°C, the initial oxidation reactions show one sharp DTG peak with a conversion ratio at 10.1%. The second-stage reaction of N-Al, which results in a weight gain, has a threshold temperature of around 726°C. This is believed to be associated with the aluminum nitride formation process.[4–6] The TG trace of G-Al in air is different from that of N-Al powders. It has been shown that N-Al has an extremely rapid weight gain at around 628°C with a high degree of reaction ratio, which corresponds to vigorous heat release around its melting point (660°C for pure aluminum). G-Al has one small weight DTG peak

Table 4 Burning rates and active aluminum content in combustion residues

Propellant sample	Burning rate, mm/s (6.84 MPa)	Al%
NM-1	6.45 ± 0.09	1.54
NM-2	6.99 ± 0.08	1.02
NM-3	6.45 ± 0.12	1.09
NM-4	6.80 ± 0.12	0.97
NM-5	8.08 ± 0.06	0.85



NM-1 NM-2 NM-3 NM-4 NM-5

Fig. 2 Flame images of samples.

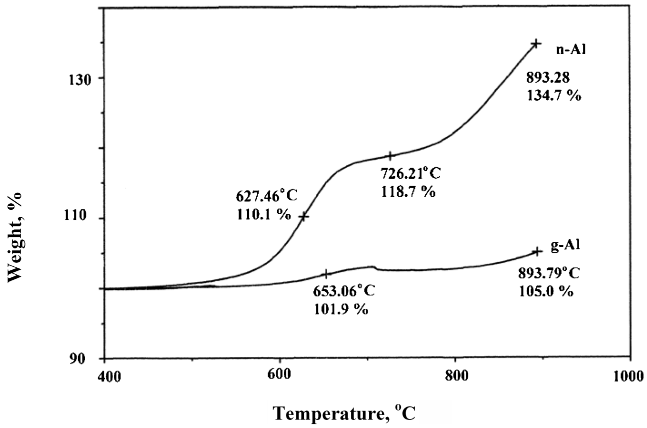


Fig. 3 TG curves of N-Al and G-Al: 0.1 MPa and 20°C/min.

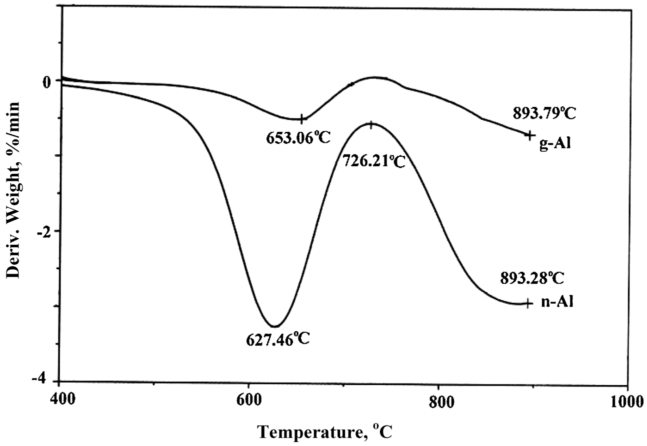


Fig. 4 DTG curves of N-Al and G-Al: 0.1 MPa and 20°C/min.

at around 653°C with a conversion ratio of only 2%, and the conversion ratio at 894°C is 5%, which is about 30% lower than that of N-Al. This difference could be due to the high reactivity of N-Al, which will lead to a lower reaction temperature and a higher degree of reaction ratio.

An oxidation study of metal powders in air provides an opportunity to measure the effects of particle size on some important physiochemical properties of metal powders, such as the ignition threshold energy. Because the surface oxidation–reduction reaction heat is approximately proportional to the surface area, when the aluminum particle size is smaller than a certain critical value, the aluminum particles (N-Al) will be rapidly heated before an appreciable deposit of Al_2O_3 on their surfaces occurs, leading to a progressive self-acceleration of heating due to the reactions between Al and the oxidizer. Such heat from self-acceleration released during the surface oxidation reaction will be enough to cause ignition at much lower temperatures than the melting point, and the N-Al will then tend to ignite as single particles in the combustion process of the propellant. However, in the present experiment, the G-Al powders are two orders of magnitude larger than the N-Al powders and have also showed rather inert reactivity. As shown earlier, such definitively different oxidative behavior will be one important factor in different ignition and combustion behaviors.

IV. Discussion

The ignition of a propellant is a complex phenomenon that involves many physicochemical processes. According to solid-phase theory, ignition is due to reaction in the solid phase at elevated temperatures.[19] In gas-phase theory, an exothermic gas-phase reaction, adjacent to the propellant surface, is considered responsible for the ignition.[19] With the existence of oxygen present in the environment, it is possible that the decomposed fuel from the propellant surface reacted with oxygen in the gas phase and supplemented the solid-phase reaction.

Further interpretation of the experimental data was made considering a bulk analysis of the ignition phenomena together with the solid-phase and gas-phase ignition theory. The bulk analysis of the ignition phenomena considers a first-order rate-controlling reaction with constant reactants and consumption during the preignition period. The reaction rate can, thus, be correlated by an Arrhenius equation,[20]

$$r = \frac{\mathrm{d}[Y]}{\mathrm{d}t} = k[Y] = A[Y]\exp\left(\frac{-E_a}{RT}\right) \tag{2}$$

Based on the simple assumption that the ignition delay time is inversely proportional to the reaction rate, the ignition delay time can be expressed by

$$t_i \propto (1/r) = \exp(E_a/RT)/A[Y] \tag{3}$$

Based on the expression of t_i shown in Eq. (3), the combined effects of N-Al and N-Ni will be discussed, in which N-Al and N-Ni improve the propellant's ignition and combustion characteristics.

A. Effect of N-Al

The approximate ignition threshold of single-particle aluminum will be proportional to the mass of particle. Because of the small mass of N-Al particles, the ignition threshold of N-Al should be orders of magnitude shorter than that of G-Al. As shown in Figs. 3 and 4, N-Al releases significant heat near 600°C, showing N-Al's rather active oxidation properties. During the process of irradiation, self-acceleration heat released during a surface oxidation reaction will be enough to cause the ignition of N-Al at much lower temperatures than the melting point. The influence of N-Al is then assumed to affect the solid-phase ignition of AP/HTPB/Al propellant. When the temperature of the solid phase increases by external heat flux, an exothermic reaction takes place between the oxidizer and the ignited N-Al in the solid phase. Such behavior will obviously promote the exothermic reaction that takes place between the oxidizer and the fuel in the solid phase, causing the temperature to reach a runaway condition. The ignition properties of the NM-2 and NM-5 containing N-Al are then improved.

B. Effect of N-Ni

The influence of N-Ni is assumed to affect the gas-phase ignition of propellant initially. The solid-phase ignition of propellant is promoted subsequently.

According to the gas phase-ignition theory, the propellant vapor evolves from the surface by external heat flux, diffuses into the surrounding gas, and reacts with it, generating heat. The reaction can take place between the vaporized fuel and oxidizer species, either vaporized or present in the test gas.

In the AP/HTPB/Al propellant, the vaporized fuel and oxidizer species are originated from HTPB and AP, respectively. Previous study showed that the decomposition of the fuel and its subsequent reaction with AP is the deciding factor in the ignition of AP/HTPB/Al propellant.[19] AP meltes before ignition, and reactions will occur in the melting layer formed on AP's surface. Liquid AP can then decompose to produce gaseous products. The gas-phase species formed during the decomposition process of AP will then react with each other and release a great deal of heat.[19]

Because N-Ni can promote the thermal decomposition of AP and decrease the ignition activation energy of AP (Ref. 10) N-Ni can then be regarded as an activated stimulation point in the propellant. Such promoting effects will lead to the more effective yield of chemical reactive gases such as Cl_2 and O_2. The reactants' concentration $[Y]$ in Eq. (3) will then increase, and the activation energy of ignition E_a will decrease simultaneously. Such promoting effects will result in the decrease of ignition delay time and the preignition of AP, and the exothermic reaction in the gas phase leads to the additional heat feedback to the surface, causing further evaporation and reaction that ultimately reaches a runaway condition.

At the same time, the more effective yield of the large amounts of oxidative gases can be produced to effectively oxidize aluminum powders and the C–H class of compounds, so that they will improve the propellants' solid ignition simultaneously. For example, Al will react with Cl_2, one gaseous products of AP's decomposition, and generate so much heat that Al melts and continues to burn[21]:

$$2\text{Al}(s) + 3\text{Cl}_2(g) \rightarrow 2\text{AlCl}_3(s) + 1408.4\ \text{kJ}$$

The influence of nanometal powders (N-Al and N-Ni) on the optical properties of energetic materials should also be considered. Ishiara et al.[22] have demonstrated that even a small amount of aluminum powders in a propellant increases the absorptivity significantly through multiple scattering, in spite of the intrinsically high bulk reflectivity of aluminum. Thus, small amounts of nanometal powders will increase the absorptivity of propellant and enhance the solid-phase ignition of propellant more significantly, as shown by the broadband absorptivity of nanometal powders due to their ultrafine grain size.

C. Combined Effect of N-Al and N-Ni

Obviously, the aforementioned properties of N-Al and N-Ni on the ignition properties of propellant will work together to influence the ignition ability of NM-5 with N-Al/G-Al = 1/1 containing 2% N-Ni. The ignition properties of the NM-5 containing N-Al are then improved dramatically as shown in Tables 2 and 3.

Returning to the mechanistic arguments on the burning behavior of NM-5, the bright near-surface flames seen when N-Al is used are an indication of enhanced Al burning. When the burning process of the propellant occurs, the agglomerate of Al requires a degree of particle accumulation (and close contact) before ignition and/or ejection from the surface. The majority of the N-Al can directly enter the gas phase to burn in single-particle form in the combustion process. Such near-combustion surface behavior will lead to a greater amount of heat-flux feedback from the burning of N-Al to the unburned surface and will increase the combustion behavior greatly. At the same time, the more effective yield of the large amounts of oxidative gases produced by the decomposition AP in the existence of N-Ni can also effectively benefit the combustion efficiency of many species and increase the intensity of the whole flame. All of these features will contribute to the combustion of NM-5.

V. Conclusions

The ignition process of AP/HTPB/Al propellant containing N-Al and N-Ni has been investigated. The ignition experiments were conducted in air atmosphere within a CO_2 laser ignition system. A comparison of different experimental techniques for combustion-related properties evaluation, including burning rates, deflagration heat, active Al content in the combustion residue, and ignition temperatures of the AP/HTPB/Al propellants is presented. The present experiment shows the following:

1) The influence of N-Al is shown to take place on the solid-phase ignition of propellant.

2) The influence of N-Ni is shown to take place on the gas-phase ignition of the propellant initially. The solid-phase ignition of propellant takes place subsequently.

3) With the combined effect of N-Ni powders, the addition of N-Al powders in propellants can greatly increase the burning rate, shorten the ignition delay time, and improve the combustion efficiency of Al in the combustion of propellant. The individual application of nanometal powders (N-Al or N-Ni powders) displayed an intermediate effect, whereas the Gr-Ni powders had the smallest effect.

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