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REVIEW PAPER

Nitramine Double Base Propellants

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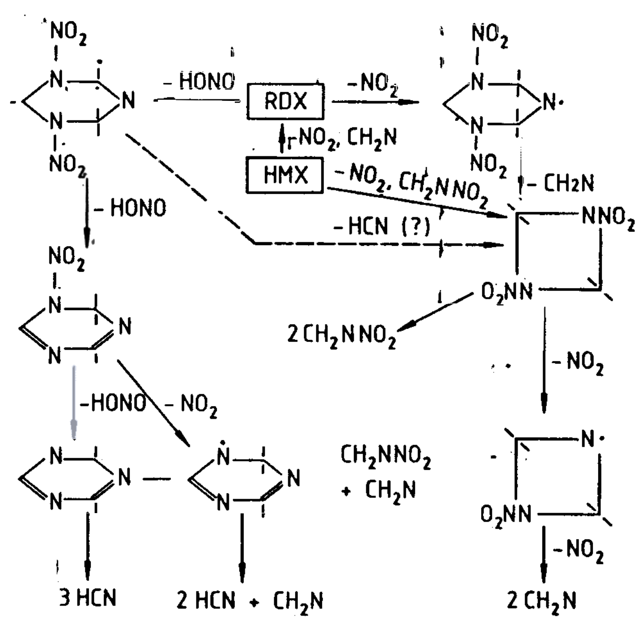
ABSTRACT

This review paper broadly covers the studies conducted on nitramine double base (DB) propellants, particularly in the field of formulation, evaluation, catalysis and combustion mechanism. Addition of RDX and HMX in double base matrix shows relatively low burn rates and high pressure index values. Further, the burn rate of this class of propellants enhances in the presence of energetic binders/plasticisers like glycidyl azide polymer. This paper also discusses the combustion mechanism of HMX/RDX-based DB propellants, especially in the presence of catalytic salts. As scanty data is available on extruded nitramine DB propellants, further work is needed in the field of formulation as well as evaluation with a view to generate exhaustive data.

1. INTRODUCTION

Nitramine double base (DB) solid propellants are advanced energetic propellants and produce high value of specific impulse (Isp). The addition of cyclic nitramines, such as RDX and HMX in the DB matrix (DB and CMDB), improves not only the energy output but also the thermal stability of the propellants. The major contribution in the energy output by this class of propellants is attributed to the high heat of combustion of RDX and HMX and formation of low molecular weight gaseous products during combustion. The heat of formation of HMX and RDX is about +17.9 k cal/mole and +14.7 k cal/mole, respectively and major decomposition species during their combustion are NO_2, N_2O, CO_2, HCN and H_2O, etc. However, the different routes predicted for their decomposition are given in Fig. 1.



CH_2N ----- $HCN + H$

CH_2NNO_2 ----- $N_2O + HCHO$

$2HONO$ ----- $NO + NO_2 + H_2O$

$HCHO$ ------ $NO + CO + CO_2 + H_2O$

Figure 1. Simplified decomposition scheme for HMX and RDX.

Further, the detailed studies of flame chemistry indicate that the thermal degradation of RDX and HMX produces more or less similar products as in the combustion of conventional DB propellants (in primary and secondary flame zones). Besides, this class of propellants shows low signature and low vulnerability to the spall of fragments[1]. However, major problems associated with these propellants are the low burn rates and high pressure index. Slow burning is related to the melting of cyclic nitramine (RDX/HMX) before active participation in the combustion reaction. The burning

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rate of nitramine CMDB propellants is found to be 2-15 mm/s at 70 kg/cm^2 and the pressure index between 0.35 and 0.60. However, the temperature coefficient lies between 0.15 and 0.35 per cent/^{o}C similar to conventional DB propellants. Furthermore, the burn rate of DB as well as CMDB propellants can be enhanced by the addition of energetic binders[2,3]. These propellant formulations are prepared by adopting well known techniques, viz., casting and extrusion techniques.

A review on nitramine solid propellants presented by Fifer[4] broadly covers the research work conducted on nitramine mono propellants. However, a few references are also related to important observations on ballistic performance of nitramine DB solid propellants. The main findings in this review are: (i) combustion mechanism of nitramine propellants is similar to the conventional DB propellants; (ii) there is no significant effect on burn rate by the addition of catalysts; (iii) nitramine DB propellants exhibit relatively superior thermal stability; and (iv) no concrete evidence has been found regarding *N-N* or *C-N* bond breaking. Apart from this, other two reviews on nitramines presented by Bogg and Cook[5,6] exclusively deal with the thermal behaviour of cyclic nitramines, particularly with reference to RDX and HMX. Keeping in view, the Fifer's review findings and scattered literature available on extruded nitramine DB propellants (EDB), an attempt has been made in this review paper to broadly cover all information related to this class of propellants with emphasis on their catalytic effect and combustion behaviour.

2. FORMULATIONS & EVALUATION

Nitramine propellants (EDB, CMDB, AP-CMDB) are prepared by well-known techniques viz., (i) slurry cast method[1]; (ii) slurry screw technique[7,8]; and (iii) extrusion technique as adopted in the manufacture of conventional EDB and CMDB propellants. In the slurry cast technique, cyclic nitramines (RDX/HMX) are incorporated along with other additives into pre-mixed casting liquid, whereas in the slurry screw technique it is added during NC/NG slurry mixing. But in the case of extrusion technique, RDX/HMX is incorporated during kneading of NC/NG mix with other ingredients[9]. The addition of RDX/HMX (more than 20 per cent) in DB propellant poses exudation problem in the propellant grain during long storage[10]. Some of the typical nitramine formulations based on EDB and AP-CMDB along with their important characteristics are summarised in Table 1.

The evaluated results on RDX-based EDB propellants containing RDX (2-10 pt) in the NC/NG matrix processed by solvent EDB technique show low burn rates and better insensitivity towards unintentional mechanical stimuli (friction and impact). However, thermal stability is found to be the same as that of conventional EDB propellants[11]. On the other hand, formulations containing RDX (30 per cent) in CMDB propellants have shown less impact and friction sensitivity than the AP-CMDB as well as PETN- CMDB propellants. Lowering in sensitivity is attributed to the oxygen balance of the composition[12]. However, the inclusion of nitramine increases the shock sensitivity appreciably as compared to the conventional ones.

Asthana, *et al*[13] have studied a composition containing RDX (12 per cent) in the CMDB propellant matrix along with different combustion instability suppression additives, such as aluminium (3 per cent), AP zirconium silicate (2-9 per cent) to assess their effectiveness for the elimination of combustion instability. Ballistic evaluation data indicated that the addition of aluminium (3 per cent) increased the burn rate by 7 per cent and eliminated combustion instability considerably owing to its melting and formation of a thin layer on regressive propellant surface. Inclusion of AP (9 per cent) in the nitramine CMDB increased the burn rate by 20 per cent but yields no change in combustion instability. However, a combination of AP & $ZrSiO_3$ in the proportion of 3:1 effectively removed the combustion instability.

Kubota N.[14] studied a nitramine DB propellant containing HMX (23 per cent) and lead stearate (3.2 parts) as catalyst and found that the burning behaviour was similar to that of the conventional DB propellants, but the burn rate was faster than that of the non-catalysed propellant.

The generated data on AP-CMDB propellant containing RDX (7.5-15 per cent), GAP (7.2 per cent), 2NDPA (0.8 per cent) shows that GAP reduces the impact and friction sensitivity considerably due to the interaction of GAP with an oxidiser or cyclic nitramine[15].

NENAs[16] (nitrato ethyl nitramines) and their azido derivatives have been found to be promising energetic plasticisers on account of their positive heat of formation and low molecular weight gaseous products. These are used along with RDX in DB propellants.

Table 1 : Typical compositions with their characteristics

Type of propellant	Formulation	Burning rate (mm/s)	Pressure (kg/cm^2)	Isp (s)	Thermal decomposition (°C)	Impact Ht. of 50% Explosive	Friction not exploding up to (kg)	Ref No.
EDB	NC/NG/DEP/SOA/HMX/PbSt 43/19/7/8/23	15-26	16-36					14
EDB	43/19/07/08/23/3.2	24	16-36					14
EDB	NC/NG/DBP/additives/RDX 51/35/5.5/8.5 /1-10pt.	4.4-9.0	35-90	226-229	197-194	23.1-27.5	14.4-16.0	11
CMDB	DB matrix/RDX (88/12)	8.6-10.6	50-70	209	204			13
CMDB	DB matrix/RDX/Al (85/12 /3)	9.2-10.7	50-70	212	193			13
CMDB	DB matrix/RDX/AP/$ZrSiO_3$ (80 / 12/6 /2)	9.8-11.5	50-70	211				
CMDB	DB matrix/RDX/AP/Al (79/12 /6/3)	10.4-12.5	50-70	221	190			13
CMDB	DB matrix/RDX/AP (79/ 12/9	10.4-11.7	50-70	219	192			13
CMDB	NC/NG/Stab./GAP/RDX/AP 60/32/0.8 /7.2/7.5/-					22.0	12.0	
CMDB	45/32/0.8/7.2/15.0/-					16.9	11.2	
CMDB	45/32/0.8/7.2/7.5 /7.5					17.4	8.4	15
CMDB	30/32/0.8/7.2/15/15					13.0	4.8	15

3. CATALYSIS

From Fifer's review[4] it is obvious that catalysts are not significantly effective in the nitramine monopropellants in the lower pressure region and thus a distinct correlation is not established during the combustion of the nitramine propellants. However, limited studies on nitramine DB propellants after incorporation of catalysts, such as lead and copper salts reveal that catalysts do have significant effect in enhancing the rate of reaction during combustion of nitramine DB propellants in the lower pressure range and also give plateau effect in higher pressure range, similar to conventional DB propellants.

Raman, *et al*[17] have found that the use of lead methylene disalicylate (LMDS), basic copper salicylate, cobaltic oxide and lead stannate increases the burn rate of RDX-based CMDB propellants by 10-16 per cent in the pressure region of 35-105 kg/cm^2. The addition of carbon black along with lead salts or metallic oxides further enhances the burn rate appreciably. However, the addition of fluorides of iron, lead and chromium have not shown any improvement in the burn rate. Borohydrides, although reported to catalyse nitramine propellants[18], are found to be incompatible and pose processing problems due to their hygroscopic nature, as reported by Asthana, *et al*[19].

The detailed study conducted on the burning behaviour of DB propellants containing HMX (23 per cent), and lead stearate (3.2 per cent) showed a plateau effect between the pressure region of 16-36 kg/cm^2 and the super rate burning at pressure less than 16 kg/cm^2 [14]

However, a systematic investigation, conducted by Asthana, *et al*[19] on the role of catalysts like organic lead and copper salts, metal oxides (*PbO*, Cu_2O) and their combinations with or without carbon black in AP-CMDB propellants, primarily containing DB matrix with AP, *Al* and RDX illustrates that most of the catalysts enhance the burn rate at 40-50 kg/cm^2. The combination of basic lead salicylate, cuprous oxide, and carbon black (3:1:1) shows best synergetic effect. This catalytic system indicates temperature sensitivity coefficient of the order of 0.3 per cent/°C and burn rate of 10.4 mm/s at 50 kg/cm^2, respectively.

Li Shang Wen[20] studied the influence of carbon black on burning rate and pressure index of high-heat RDX-CMDB propellant. The particle size of carbon black (24 to 30 nm), exhibits positive effect for the improvement of pressure index (n). This work entirely differed from Preckel[21] who suggested that in high-heat propellants aromatic lead salts having higher decomposing temperature more than 750 °C is necessary to achieve plateau.

Catalytic effect of basic copper chromite, copper chromite pyridine complex in the nitramine CMDB propellant was studied by Ma Xieqi and Sou Syuing[22] and observed that there is a tendency for the emergence of plateau and mesa combustion at low pressure (3 to 5 kg/cm^2).

The work of Joseph[23] on DB propellants, containing RDX/HMX (54-56 per cent) and lead stannate—as ballistic modifier along with lead beta resorcylate and carbon black has shown considerable reduction in pressure index (n) and temperature sensitivity coefficient $(\pi\ r)_p$ values, when fine HMX (20-25 micron) is used in the formulations. Further, a remarkable reduction in pressure index value has been observed when HMX is taken in bimodal form (180 and 25 micron) in the ratio of 75/25.

Kawasaki, *et al*[24] found the burn rate of 2-10 mm/s in the pressure range of 5-15 atm when EDNA (13 per cent) was used in DB matrix. Further study[25] of the flame structure of EDNA DB propellants by the same author led to the several conclusions; viz., (i) flame structure of EDNA/DB propellants was of two stage similar to that of DB base propellants exhibiting a dark zone between luminous flame and burning surface, (ii) these propellants showed mesa tendency, (iii) the thermochemical properties of EDNA were between NQ and HMX, (iv) the burning rate was found to be independent of concentration, and (v) slope break phenomenon was absent.

4. FLAME CHEMISTRY OF NITRAMINE DB PROPELLANTS

The combustion flame of nitramine DB propellants is similar to that of the conventional DB propellants. It mainly consists of foam, fizz, dark, and luminous zones respectively[26]. The combustion products found in successive zones are: (i) $HCHO$, NO_2, (ii) HCN, CO_2, $HCHO$, NO, H_2, (iii) CO, CO_2, HCN, NO, N_2O, and (iv) CO_2, H_2O, N_2, H_2 with a little amount of CO and NO. These products have been experimentally identified by means of DSC and shock tube technique[27]. A schematic diagram showing decomposition species in different zones is shown in Fig. 2.

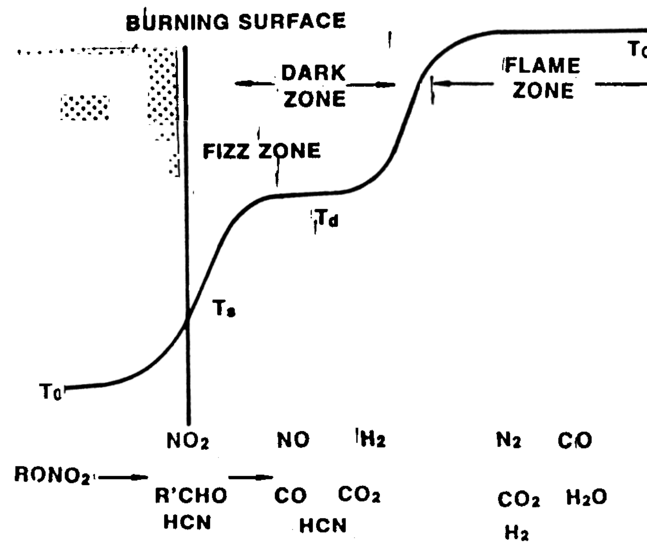

Figure 2. Schematic diagram of the flame structure (combustion wave) nitramine double base propellant.

5. COMBUSTION MODELLING

Taking into consideration the flame chemistry of nitramine-based DB propellants, two mathematical models have been proposed. Bizot, *et al*[28] have proposed a mathematical relation for calculation of burn rate, combustion species and temperature profiles in condensed and gas phases. The combustion products as a result of thermal degradation of HMX/RDX in these phases are assumed to be $HCHO$, HCN, NO_2 and N_2. The absence of dark zone in the nitramine combustion is attributed to the reduction of N_2O to N_2 occurring in gas phase very close to the burning surface. However, this reaction is very effective at higher temperature.

Another model exclusively based on the presence of dark zone region of nitramine propellant combustion has been suggested by Vanderhoff, *et al*[29]. This model assumes that dark zone occurs when the conversion of N_2O to N_2 through NO takes place after attaining temperature as a result of delayed thermal ignition. However, slight modification in the reaction from the earlier ones predicted by different coworkers as shown below has indicated 25 per cent enhancement in the reaction rate.

Earlier equations

$HNO + NO = N_2O + OH$

$CO_2 + N = NO + CO$

Modified

$HNO + NO = N_2O + OH$

$CO_2 + N + N = N_2 + CO + O$

Taking into consideration the above modified equations the predicted time (t_m) from this model is 3.6 ms for DB propellants which is well in agreement with the experimental value of 3.2 ms. However, the predicted values of tm for nitramine DB propellants are found to be lower than the experimental values. Beside, a correlation has been suggested for cylindrical type of propellant burning in cigarrette fashion, between dark zone length and time duration, i.e.,

$t_m = L_d/v$ and $V = \dot{r} \times \rho_s/\rho_g$

where

v = Flow velocity of gases(m/s)

$(t)m$ = Maximum time (s)

Ld = Length of the dark zone in cm.

$\dot{r}$ = Burn rate of the propellant

ρ_s = Solid density

ρ_g = Gas density

A mathematical model proposed by Melius[30,31,32] suggests that cyclic nitramines (RDX/HMX) form dominantly HCN in non-luminous flame zone which gets converted into N_2, CO, CO_2, etc in the luminous flame zone subsequently. Further, by employing this model he has calculated the length of non-luminous and luminous flame zones as 20 and 80 micron respectively.

Kubota[33] has proposed a mathematical combustion model based on heat feedback process in the combustion wave and the chemical reaction on the burning surface and gas phase for nitramine DB, CMDB and azidopolymer propellants.

A correlation has been found out between burning rate of the propellant and temprature sensitivity.

$$\sigma_p = [\delta \ln \dot{r}_b/\delta T_o]_p \qquad (1)$$

where $\dot{r}_b$ = Burn rate m/s

T_o = Initial propellant temperature (°K)

σ_p = Temperature sensitivity at constant pressure

$$r_b = \alpha_s \times \varphi/\psi \qquad (2)$$

where

α_s = Thermal diffusivity at burning surface (cm^2/s)

φ = Gas phase temperature gredient ($dt/dx)_g$

ψ= Condensed phase temperature (K)

and

$$\alpha_s = \lambda g / C_p \times \rho_p \qquad (3)$$

where

λ_g = Thermal conductivity in gas (kW/mK)

ρ_p = Density of propellant (kg/m^3)

$$\psi = T_s - T_o - Q_s/C_p \qquad (4)$$

where T_s = Temperatute of burning surface (°K).

T_o = Initial propellant temperature (°K)

Q_s = Heat of reaction of burning surface (cal/g)

C_p = Specific heat (kJ/kg.k)

Now

$$\sigma_p = \quad + \psi \qquad (5)$$

where

= Temperature sensitivity of gas phase

ψ = Temperature sensitivity of condensed phase

$$\psi = \frac{1 - (\frac{\delta T_s}{\delta T_o})_p}{T_s - T_o\ (Q_s/C_p)} \qquad (6)$$

Where

$$(\delta T_s/\delta T_o)_p = \frac{\sigma_p R T^2 s}{Es}$$

The burn rate of the propellant at 243 and 343 °K are determined. Temperature sensitivity parameters such as φ, T_s, and Q_s, are determined from thermal profile and ψ from Eqn (6). Temperature sensitivity of gas phase is calculated from Eqn (5). Experimental data obtained with nitramine propellant is in good agreement with predicted values. Besides, it also reveals that the temprature sensitivity at constant pressure is dependent 60 per cent on gas phase temperature sensitivity and 40 per cent condensed phase temperature sensitivity.

6. COMBUSTION MECHANISM

The burning process of the propellant is largely dependent on the propellant composition. The propellant generally produces heat and high tempdrature gases by the phenomenon of combustion. The heat feedback from hot gases raises the temperature of unburnt propellant surface to its decomposition temperature. As a result, the unburnt portion gasifies and produces heat by exothermic chemical reaction which is ultimately responsible for the successive heat feedback process, occuring continuously to sustain a steady state burning.

The flame structure analysis of nitramine DB propellant based on the quenched propellant microscopic analysis has clearly indicated that the crystalline RDX/HMX particles mixed with DB matrix melt first and decompose subsequently into gaseous products at the burning surface of the propellant. The decomposed RDX gas is then diffused into the decomposed gas of DB matrix just above the burning surface (fizz zone); and thus a homogeneous gas mixture produces a luminous flame in luminous zone above the burning surface. Besides, it is also found that stand-off distance[26] is decreased as compared to that in conventional DB propellants owing to fast heat feedback transfer from luminous zone to propellant surface.

To understand the combustion mechanism of nitramine CMDB propellant (catalysed and non-catalysed), Kubota[14,34] measured the temperature profiles in different zones and found that catalysed propellants exhibited super rate burning in lower pressure region and subsequently plateau effect in the higher pressure region. This is attributed to inhibiting reactions of gaseous products with *Pb* in fizz zone. In addition, other findings are: (i) appreciable reduction in the stand-off distance in catalysed propellant (ii) insignificant effect of particle size of nitramine for both catalysed and noncatalysed propellants, (iii) decrease in the burn rate with increase in concentration of nitramine, and (iv) similarity of flame structure for both propellants. Another systematic study carried out by Zhao, *et al*[35] on the combustion characteristics of RDX/CMDB, AP-CMDB propellants including thermal studies over a wide range of pressure has indicated: (i) increase of RDX/HMX content in the basic propellant composition decreases the burn rate at lower pressure range and increases at higher pressure range, (ii) there is a significant effect of particle size of RDX in higher pressure region, and (iii) incorporation of nitroguanidine in nitramine CMDB propellant improves the physical structure and thermal behaviour thereby eliminating pressure exponent shift considerably.

On the other hand thermal studies conducted by means of DSC and SEM reveal that the pressure exponent shifts are related to the decomposition of RDX and binders, their physical structure variation, change in burning surface and chemical activation variation of N_2O with pressure.

7. CONCLUSION

This review clearly indicates that the studies on extruded DB propellants containing nitramine appears to be limited and the scope for further work in respect of processing, evaluation and ballistic performance exists. Although, a number of papers appeared in the literature exclusively dealt with processing, burning behaviour, sensitivity, etc. for nitramine CMDB propellants, the field is still open for further studies on the assessment of shelf life, ballistic characteristics and mechanical properties essentially needed for their application in rockets/missiles.

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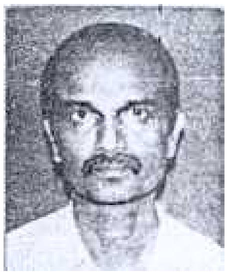

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