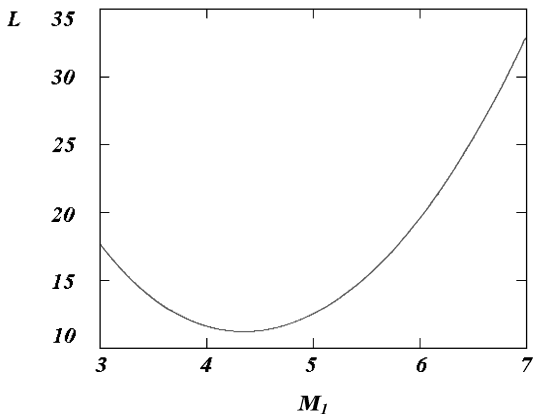


**Fig. 4 Deceleration length vs transition Mach number in an MHD + GDP combination. (The initial and final Mach numbers are $M_0 = 7$ and $M = 3$.)**

the Mach number is reduced from $M_1$ to its final value of $M = 3$. The combined value of the energy input by GDP and the Lorentz force work used for flow deceleration by MHD is kept constant and equal to that in Fig. 3. Length $L$ is the total normalized length over which the MHD and GDP actions are applied. The $L$ vs $M_1$ dependence features a minimum, and this minimum value is significantly smaller than the deceleration length of the single MHD or GDP action using the same total input power (see Fig. 3). The calculations we have carried out for a range of initial Mach numbers showed that, for a given inlet length, the Mach number can be reduced by the combined action to a value which is up to three times smaller than that for the single action of GDP or MHD, with the same input energy. A reduction in the Mach number can lead to a decrease in the total pressure loss and increased specific impulse.[6] Therefore, the presented study demonstrates that the simultaneous application of GDP together with MHD can enhance scramjet performance.

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# Studies on Advanced CL-20-Based Composite Modified Double-Base Propellants


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## I. Introduction

PROPELLANTS and explosives are the obligatory energy delivering systems of weapons. The development of propellants and explosives technology is focused on the enhancement of the lethality of weapon systems and improvement of the range of missiles. Research in this direction brought into focus the concept of high-energy dense materials (HEDMs). Hexanitro hexaazaisowurtzitane (HNIW or CL-20),[1] the most powerful HEDM of today, has emerged as a viable high velocity of detonation alternative to cyclo trimethylene trinitramine (RDX) and cyclo tetramethylene tetranitramine (HMX). The HEDMs such as CL-20 have also evoked interest as a powerful replacement for ammonium perchlorate (AP) for realizing the eco-friendly high-performance, $I_{sp}$, propellants for futuristic missiles and space missions. Potential applications of CL-20 include boost propulsion of strategic missiles or space launchers as well as high-lethality warheads for SMART and light weapons.[2]

Golfier et al.[3] reported that CL-20 propellants offer 7% superior $I_{sp}$ (251 s) compared to corresponding RDX-based formulations. Weiser et al.[4] found that the CL-20/glycidylazide polymer-(GAP) propellants exhibit burning rates twice those of HMX/GAP propellants. Attempts have been made to ballistically modify CL-20 formulations, but specific information about the modifiers is not available.[3]

The present study was undertaken to evaluate CL-20 as the HEDM component of composite modified double base (CMDB) propellant. Because the technology of CL-20-based systems is in a transitional state, the data generated during this study are envisaged to provide inputs for weapon designers. Slurry cast CL-20 incorporated CMDB propellant containing 17.5% aluminium (Al) was selected in view of the optimum $I_{sp}$ level of $\sim 265$ s obtained by theoretical calculation using NASA CEC71 program (Table 1). The effect of ballistic modifiers on the burning rates of CL-20 propellants was assessed. Copper chromite (CC) found effective during earlier work[5] in RDX-based compositions, which like CL-20 belongs to the nitramine class, was selected in this work. The effect of $Fe_2O_3$ was also assessed. Bis-dinitropropylformal/acetal (BDNPF/A) and low molecular weight GAP were evaluated as coplasticizers with NG as a substitute for the inert phthalate plasticizer with the aim of augmenting the ballistics. The NG-free trimethylolethanetrinitrate-triethyleneglycoldinitrate (TMETN-TEGDN) plasticized propellant was also investigated to achieve low vulnerability. The thermal studies were undertaken to gain insight into the chemical processes occurring in the condensed-phase during combustion.

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## II. Experimental

### Formulations

The CL-20 used for this study was synthesized and characterized by the authors along the lines of the method reported in Ref. 6. CMDB formulations comprising 25% spheroidal nitrocellulose (SNC) [nitrocellulose (NC), 90%; nitroglycerin (NG), 7%; and N–N' diphenyl diethyl urea, 3%] of 25–45 $\mu$m particle size, 35% desensitized NG [NG, 78%; diethyl phthalate (DEP), 20%; and 2–nitro diphenyl amine (2NDPA), 2%], 17.5% Al ($15 \pm 1$ $\mu$ particle size) and 22.5% CL-20 ($30 \pm 2$ $\mu$ particle size) were processed by slurry cast technique described elsewhere.[5,7] In the ballistically modified compositions, 2 parts of the ballistic modifiers were added to over 100 parts of the composition (by weight). Energetic plasticizers BDNPF/A and low molecular weight GAP synthesized[8,9] in the laboratory were added as replacement of DEP, whereas TMETN + TEGDN (2:1) combination was added as a substitute for desensitized NG itself.

### Strand Burning Rate

Strand burning rates of the propellants were determined in the pressure range of 2–9 MPa by employing an acoustic emission technique.[10] The methodology involved combustion of the strand (ignited by means of a nichrome wire) of dimensions $100 \times 6 \times 6$ mm in the nitrogen-pressurized steel bomb. The acoustic signals generated due to perturbations caused by the propellant deflagration were unidirectionally transmitted through the water medium to a piezoelectric transducer (200 kHz) in conjunction with an X–Y chart recorder. The burning rates were computed from the time that was recorded for the combustion of a preselected length of the strand. Five experiments were conducted at each pressure for each sample. The standard deviation was of the order of 0.2%.

### Sensitivity

Sensitivity of the propellant compositions to impact stimuli was determined applying the fall hammer method (2-kg drop weight) as per the Bruceton staircase approach and results are given in the terms of statistically obtained 50% probability of explosion ($h_{50}$). Friction sensitivity was measured on Julius Peter apparatus by standard methodology.

### Thermal Analysis

Thermal analysis of propellant was carried out on the differential scanning calorimeter (DSC)(Pyris-7) and thermogravimetric analyzer (TGA) (Mettler Toledo Model 8551) at the heating rate of 10°C/min under $N_2$ atmosphere (sample mass of ~10 mg). The gaseous products of decomposition were analyzed by Fourier transform infrared (FTIR) (Bruker make equinox 55) hyphenated with TG.

**Table 1 Theoretical performance parameters of CL-20 based CMDB propellants**

| Sr. No. | Composition | $I_{sp}$,s | $T_f$, K | Mw |
|---|---|---|---|---|
| 1 | CL-20:40 | 252 | 3155 | 25.9 |
| 2 | CL-20:30, Al:10 | 262 | 3510 | 28.5 |
| 3 | CL-20:27.5, Al:12.5 | 263 | 3596 | 29.2 |
| 4 | CL-20:25, Al:15 | 265 | 3675 | 29.9 |
| 5 | CL-20:22.5, Al:17.5 | 266 | 3717 | 30.5 |
| 5 | CL-20:20, Al:20 | 264 | 3692 | 31.3 |

## III. Results and Discussions

### Burning Rates

The strand burning rate experiments conducted in the pressure range of 2–9 MPa, revealed that the aluminized CL-20 based CMDB propellant did not exhibit stable combustion up to 3 MPa chamber pressure. It exhibited the burning rates of the order of 6.5–9.7 mm/s in the pressure region of 5–9 MPa (Table 2). These trends suggest that the combustion behavior of CL-20 CMDB propellants is superior to that of the RDX–CMDB system.[5] The addition of $Fe_2O_3$ and CC led to 9–40% enhancement of the burning rates of the propellant, as well as extending the low pressure combustion limit (LPCL) to 2 MPa. The CC was relatively more effective and offered burning rates of the order of 4.3–13.6 mm/s in the pressure range of 2–9 MPa.

In the next set of experiments, nonenergetic plasticizer (DEP) was replaced by the energetic ones, namely, BDNPF/A and GAP in CC modified propellant. This led to a gain in $I_{sp}$ of 5–6 s, as well as a 45–170% enhancement of the burning rates. The GAP plasticized CL-20-based aluminized CMDB formulation gave relatively higher burning rates, and the values obtained (9.5–37 mm/s) are close to those reported by Golfier[3] for the CL-20-GAP propellant. The high-energy potential of the GAP plasticized formulation was also brought out by its remarkably higher calorie value (1582 cal/g) than that of the corresponding DEP plasticized composition (1255 cal/g). The NG-free TMETN+TEGDN composition offered relatively lower burning rates than those of the DEP desensitized NG-based composition (Table 2), whereas their energetics, $I_{sp}$, were comparable.

### Sensitivity

The aluminized CL-20-based CMDB formulation gave $h_{50}$ of 29 cm and was friction insensitive up to 19.2 kg. These results demonstrate that the CL-20 formulations are less vulnerable to mechanical stimuli, particularly in terms of friction sensitivity, compared to modern AP–CMDB systems.[11] This may be attributed to the negative oxygen balance of CL-20 in contrast to the positive oxygen balance of AP. Incorporation of the ballistic modifiers led to a marginal increase in the friction sensitivity. The addition of GAP and BDNPF/A as replacement for DEP led to further increase in the impact and friction sensitivity, whereas the substitution of the DEP desensitized NG by TMETN + TEGDN combination rendered them marginally less vulnerable (Table 2). These trends may be attributed to superior oxygen balance of the former (from −51 to −121%) compared to that of the DEP (−194%) and the lower oxygen balance of TMETN + TEGDN system (−45%) than that of the DEP desensitized NG (−36%).

### Thermal Analysis

The DSC results for the CL-20 incorporated Al–CMDB propellant gave two exotherms with $T_{max}$ of 203 and 253°C, which are close to those obtained for double base matrix (199°C) and CL-20 (243°C) individually. The $\Delta H$ associated with the exotherms were 941 and 209 J/g, respectively. The ballistic modifier incorporated aluminized formulations also exhibited two-stage decomposition with similar $T_{max}$ and $\Delta H$. The incorporation of BDNPF/A and GAP led to increase in heat output by ~400–550 J/g during the first stage of decomposition. In the case of the TMETN + TEGDN plasticized formulation, the two stages of decomposition were not distinguishable. However, the final decomposition temperature was

**Table 2 Sensitivity and burning rates results of CL-20 based CMDB propellants**

| | Sensitivity | | Burning rates, mm/s, at pressure, MPa | | | | | |
|---|---|---|---|---|---|---|---|---|
| Composition | Impact $h_{50}$ | Friction, kg | 2 | 3 | 5 | 7 | 9 | $n$, (2–9 MPa) |
| Base[a] | 29 | 19.2 | Ext | Ext | 6.5 | 8.3 | 9.7 | 0.68 |
| $Fe_2O_3$ | 26 | 14.4 | 4.2 | 5.0 | 7.1 | 10.6 | 13.5 | 0.79 |
| CC, 2 parts | 26 | 16 | 4.3 | 5.7 | 9.0 | 11.5 | 13.6 | 0.78 |
| BDNPF/A desensitized NG | 24 | 9.6 | 6.2 | 8.9 | 12.1 | 16.8 | 21.3 | 0.79 |
| GAP desensitized NG | 22 | 12.0 | 9.5 | 12.9 | 17.8 | 21.5 | 37 | 0.81 |
| TMETN : TEGDN (2 : 1) | 32 | 16.0 | 3.2 | 4.2 | 7.2 | 9.5 | 12.1 | 0.90 |

[a]Base composition (%): SNC, 25; desensitized NG, 35; CL-20, 22.5; and Al, 17.5.

**Table 3 DSC and TG thermal analysis results of CL-20 propellant formulations**

| | DSC | | | TGA | | |
|---|---|---|---|---|---|---|
| Formulation | $T_i$ | $T_m$ | $T_f$ | $-\Delta H$, J/g | Temperature, °C | Weight loss, % |
| Base[a] | 165 | 203 | 238 | 941 | 162–210 | 56 |
| | 238 | 253 | 263 | 209 | 247–249 | 15 |
| $Fe_2O_3$ | 153 | 204 | 242 | 958 | 158–207 | 51 |
| | 242 | 254 | 261 | 145 | 247–249 | 18 |
| CC, 2 | 164 | 204 | 241 | 865 | 122–217 | 55 |
| | 241 | 253 | 259 | 253 | 244–252 | 16 |
| parts | | | | | | |
| BDNPF/A | 142 | 203 | 241 | 1244 | 167–213 | 58 |
| | 241 | 253 | 261 | 202 | 246–248 | 18 |
| desensitized NG | | | | | | |
| GAP | 155 | 205 | 242 | 1396 | 153–210 | 50 |
| | 242 | 253 | 262 | 200 | 243–244 | 20 |
| desensitized NG | | | | | | |
| TMETN:TEGDN | 148 | 207 | 260 | 1508 | 169–207 | 49 |
| (2:1) | | | | | 243–244 | 23 |

[a]Base composition (%): SNC, 25; desensitized NG, 35; CL-20, 22.5; and Al, 17.5.

close to that of the second exotherm obtained in the case of other compositions.

In dynamic TG, CL-20 exhibited a 57% weight loss in the temperature range of 214–306°C followed by a gradual decomposition of the residue accompanied by the 39% weight loss in the temperature region of 306–506°C. The CL-20-based aluminised CMDB propellant underwent 56 and 16% of weight loss in the temperature ranges of 160–210 and 247–249°C, respectively. The weight loss in the first stage appears to correspond to the decomposition of the double base matrix and in the second stage to that of the CL-20, which supports the inferences drawn from the DSC pattern. The ballistically modified formulations exhibited more or less similar weight loss patterns. The replacement of DEP by energetic plasticizers and that of the DEP desensitized NG by TMETN + TEGDN combination in the CC modified aluminized formulation did not have much effect on the weight loss pattern (Table 3).

It is well known that $Fe_2O_3$ catalyses the gas-phase redox reactions of the decomposition products of propellants, whereas lead salts facilitate the formation of C-nuclei on the surface and catalyze the exothermic reactions of $NO_2$/NO with aldehydic fragments resulting from the combustion of the propellant, leading to the occurrence of part of the catalyzed exothermic reduction of NO to $N_2$ near the deflagrating surface rather than away from it in the luminous zone.[12–15] The CC is known to undergo exothermic redox reactions at ∼700°C and catalyse the decomposition of both the oxidizer as well as the binder in condensed and gas phases. Similar processes are also expected to play a role in the ballistic modifier incorporated CL-20-based propellants and are expected to cause efficient heat feedback to the deflagrating propellant surface, leading to high burning rates.

That there was almost no change in the $\Delta H$ recorded in the DSC of the ballistically modified composition despite the remarkable burning rate enhancement brings out that the exothermic processes in the condensed-phase/near gas-phase region compensate for the heat losses associated with heating of the catalyst to the temperature essential for them to exert a catalytic effect, and the site of their catalytic action is mainly the gas phase.

Pendant azide group of GAP is known to undergo exothermic cleavage in the sub-surface region,[16] which is reflected in an increase in the $\Delta H$ of the GAP plasticized CL-20 formulation and is manifested in its higher burning rates compared to those for the corresponding DEP plasticized formulation. The BDNPF/A also appears to promote oxidative reactions in the condensed-phase/near-surface gas-phase reactions due to the presence of oxygen-rich $NO_2$ group, albeit to a lesser extent than GAP. As regards the TMETN/TEGDN-based system, NC plasticized by it appears to undergo sluggish decomposition (in the initial stage) leading to relatively lower burning rates than those of even DEP desensitized NG-based propellant as reported by Kubota[17] in case of the double base systems.

The FTIR of decomposition gases evolved during TG of CL-20 brought out the formation of C≡N and –N=O as well as –C=O containing species. This is in line with the findings of Patil and Brill.[18,19] They established the formation of HCN, $NO_2$, and NO (in secondary reaction) on the basis of T-jump FTIR spectroscopy. The decomposition products of the double base matrix exhibited absorption bands corresponding to –C=O and –N=O containing species. The CL-20 incorporated aluminized CMDB formulation evolved gaseous products similar to those observed on decomposition of both CL-20 and double base matrix without absorption band corresponding to –CN containing species. This observation needs explanation. There is a possibility of –CN containing species getting adsorbed in the amphoteric Al in the condensed phase or a disfavor of the formation of –CN moieties in the presence of Al. The ballistically modified formulations exhibited similar trends. The incorporation of BDNPF/A did not have much effect on the decomposition products of the aluminized composition. However, the gaseous decomposition products of the GAP plasticized formulation exhibited the bands corresponding to –CN containing species, which may be due to the contribution from its (GAP) decomposition itself. The GAP is reported[20] to evolve C≡N containing species in abundance on combustion. The TMETN + TEGDN plasticized composition also exhibited similar decomposition product profile.

## IV. Conclusions

The CL-20-based aluminized CMDB propellants offer burning rates superior to the RDX incorporated propellants and are less vulnerable to friction stimuli than the AP–CMDB formulations. CC was found to be a relatively superior burning rate modifier than ferric oxide ($Fe_2O_3$) in terms of the optimized combination of burning rates and LPCL. The addition of low molecular weight GAP and BDNPF/A as coplasticizers of NG in place of DEP led to a remarkable increase in the burning rates and $I_{sp}$. The TMETN/TEGDN based CL-20 propellant was found less vulnerable, albeit with lower burning rates. The thermal analysis results bring out that the major part of the decomposition of the CL-20 and double base matrix takes place in discrete steps. The DSC patterns suggest that the site of action of the catalysts is in the gas phase, whereas GAP/BDNPF/A based compositions enhance burning rates due to increased energy release in the condensed phase.

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