



In-Depth Chemistry in Plasma-Exposed M30 and JA2 Gun Propellants

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Abstract

JA2 and M30 recovered from interrupted ETC-closed bomb, interrupted ETC small-scale chamber, and open-air experiments were subjected to chemical and microscopic analysis. Evidence of subsurface reaction in both M30 and JA2 has been discovered using a desorption-gas chromatography-mass spectroscopy (D-GC-MS) method to detect low levels of NO in the propellant. It appears that for M30, profiles for radiation-induced denitration of nitrate esters are consistent with Beer's law, and that effects occur as deep as 0.5 mm into the exposed surface. Radiation-induced denitration in JA2 has been detected as deep as 0.75 mm from the exposed surface, but profiles are not consistent with Beer's law. It is suspected that denitration at and below the exposed JA2 surface occurs mostly in locations where large graphite particles are found. Rough estimates of graphite particle temperature when exposed to plasma radiation are in excess of those required for denitration to occur. Microscopic examination of plasma-exposed propellant indicates several features (pits, gouges, blisters, wormholes, etc.) that increase the surface area of the propellant and can contribute to an increased burning rate.

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1. Background

There are a number of advantages to using plasma ignition for gun propulsion, including short, reproducible ignition (Katulka and Dyvik 1996), significantly reduced temperature sensitivity (Dyvik and Katulka 1995; Marinos 1995; Perelmutter et al. 1996), and the ability to ignite charges with very high loading density (White et al. 1995). In addition, it has been observed that propellants burned in closed bombs with plasma ignition exhibit mass generation rates that are significantly higher than those observed with conventional ignition (Woodley and Fuller 1996; Del Güercio 1997). To exploit this technology to its maximum, the interactions between plasma and propellant must be clearly understood. Toward this end, the U.S. Army Research Laboratory (ARL) has undertaken the study of these interactions. Several reports on the chemical characterization and morphological characterization of plasma-exposed propellants have appeared over the past few years. In one of the first such studies, Lieb and Gillich (1994) studied morphological changes in JA2 propellant recovered from a 30-mm electrothermal-chemical (ETC) gun. It was reported that exposed surfaces were pitted and showed evidence of both brittle fracture and plastic flow, and that regions of the propellant showed evidence of augmented mass generation as compared to samples exposed to a conventional ignition system. Lieb postulated that mechanisms to explain observed morphological changes include erosive burning, fracture-generated tunneling, and in-depth burning. Blowout pressures were typically larger than those found by Kaste et al. (1998a, 1998b, 1999) and Kinkennon et al. (2000) to be of use for studying plasma effects, i.e., 95 MPa vs. 35 MPa. Kaste et al. (1998b) examined seven different propellant formulations, including a double base (M9), a modified double base (JA2), a LOVA (HELP-1), and nitramine-filled thermoplastic elastomers (TPEs), all recovered from experiments where the propellant was exposed to plasma in an "open air" configuration. In that study, evidence for the denitration of nitrate esters in the M9 and JA2, the hydrolysis of the cellulosic binder in the LOVA, and a reduction in the level of nitramines in the TPEs was found. In a separate experiment involving shielded JA2 samples, FTIR microscopy revealed carbonyl functional groups, again indicating that denitration of the nitrate esters had occurred when JA2 was unshielded and shielded by aluminum, but not when it was shielded by Mylar (to shield from UV radiation and convective heating). No evidence of plasticizer depletion was observed in the samples recovered from either of these experiments.

Kaste et al. (1998a) reported on the analysis of M30 and JA2 from interrupted closed bomb experiments with plasma and conventional ignition sources. It was found that denitration at the surface of JA2 occurred at blowout pressures of 75 and 100 MPa, but not at 35 MPa, suggesting that the reaction was a result of burning, not of

exposure to plasma. For both plasma and conventional ignition of M30 at blowout pressures of 75 and 100 MPa, IR bands for nitroguanidine (NQ) decreased relative to other bands in M30's IR spectrum. This is consistent with results reported by Schroeder et al. (1994) and indicates that with normal burning, NQ is depleted at a faster rate than is nitrocellulose. No evidence of plasticizer depletion was detected in any of the samples examined.

Kaste et al. (1999) again analyzed M30 samples recovered from interrupted closed bomb experiments, but this time first notched all of the grains in the propellant charge to indicate their position relative to the igniter so that their original position could be identified when recovered after the experiment. By doing so, grain damage relative to the position in the original charge could be determined, including determining where any unrecovered grains were located relative to the igniter. Scanning electron microscopy (SEM) was used to analyze the perforation surface of polyethylene (PE) plasma ignited samples of M30, and indicated that NQ crystals had been stripped from the surface. Very little evidence of chemical modification of the propellant was found. As in previous studies, no evidence of plasticizer depletion was found for either M30 or JA2 by conventional or plasma ignition. Evidence of aldehyde formation (indicating that denitration had occurred) was found for M30 extinguished at up to 80 MPa. As was found for previous plasma and conventional ignition studies, NQ level decreased as regression increased. X-ray fluorescence (XRF) analysis revealed the presence of Fe (from the closed bomb hardware) and Cu (from the plasma igniter) in the perforations of plasma-ignited M30. Fe was not found in the perforations of conventionally ignited M30 (Cu would not be expected).

Kinkennon et al. (2000) made a complete SEM characterization of conventionally and Mylar plasma-ignited M30. Composite SEM photos of the exterior surface, the end surface, and the center perforation surface were reported. For this study, the grains in the charge were labeled with respect to their position relative to the ignition source, as was done by Kaste et al. (1999). The SEM photos of the plasma-ignited samples revealed a significant amount of damage to the grains. The amount and type of damage sustained varied, depending on the location of the grain in the original charge. The study confirmed that plasma ignition results in a large increase in surface area of the propellant grains, but did not confirm that the surface area increase was sufficient to account for previously reported burn rate enhancements for M30 (Del Güercio 1997).

The original intention for the current study was to apply the strategy used in the Kinkennon et al. (2000) study to JA2. It was expected that the most significant results to be obtained were the morphological changes produced in the JA2 due to plasma exposure. While these changes were found and are partially documented here, it was also found that in-depth chemical changes had occurred in the JA2 recovered from interrupted closed bomb experiments. The study was then expanded to include samples recovered from interrupted small-chamber

experiments and open-air experiments (Beyer 2000), and it was found that the in-depth chemistry had occurred in these samples as well. The focus of this report is on the documentation of this in-depth chemistry and the speculation as to its significance with respect to plasma-propellant interactions.

2. Experimental

2.1 Plasma Exposure

ETC-closed bomb experiments were performed using nickel wire/Mylar capillary plasma, a blowout disk that ruptured at 35 MPa, and a foam-lined collection chamber as described by Kinkennon et al. (2000). Small-scale chamber and open-air experiments were performed using an aluminum wire/polyethylene plasma as described by Beyer (2000).

2.2 Microscopy

SEM was performed with a JEOL Model JSM-820 instrument. Samples were sputtered with gold prior to examination.

2.3 Sample Preparation for D-GC-MS Analysis

Samples were exposed to either conventional or plasma ignition sources as described by Beyer (2000). Plasma exposure was either through the center of the grain for cylindrical samples, or "head on" for sheet samples. The propellant was either sliced by hand using a razor blade (M30) or microtomed (JA2) to give cross-sectional slices with a thickness of approximately 200 μm . The ends of each slice were trimmed to yield a rectangular sample. The width of each rectangle depended on the particular sample. Lengthwise strips were cut from the rectangular slice. Masses of each strip relative to the total mass of the slice were used to estimate the thickness of each strip. In all cases, slices of samples were analyzed starting with the side exposed to the ignition source and proceeding into the bulk of the sample.

2.4 Desorption-Gas Chromatography-Mass Spectroscopy (D-GC-MS)

Analysis of NO levels was performed by means of D-GC-MS. Desorption was achieved via a CDS Model 2000 Pyroprobe (coil type) connected through a heated interface chamber to the splitless injector of a Hewlett Packard GC-FTIR/MS system (Model 5890 GC, Model 5970 MSD, and Model 5965 IRD with narrow band MCT detector). The GC Column used was a J&W Scientific capillary column (0.32 mm \times 25 m; 3-m DB-17HT film). The injector temperature was 200 $^{\circ}\text{C}$; the Pyroprobe

interface temperature was 175 °C. The GC oven temperature program was as follows: 50 °C isothermal for 1 min; 50–250 °C at 40 °C/min; and 250 °C isothermal for 4 min. The Pyroprobe was programmed to give a 20-s desorption pulse at 175 °C. Propellant strips were held within the coil of the Pyroprobe by first placing them in a quartz tube containing a small plug of glass wool and then inserting the entire tube into the coil.

NO levels in propellant samples were determined via Hewlett Packard ChemStation software by first extracting the selected ion chromatogram (SIC) for $m/z = 30$, the molecular weight of NO, from the total ion chromatogram (TIC). An example of a TIC and SIC is given in Figure 1. Next, a ratio of the heights of the peaks for neat NO (2.5-min peak) and the NO generated when nitroglycerine (NG) and/or diethylene glycol dinitrate (DEGDN) plasticizers are fragmented at the mass spectrometric detector (5.2-min peak) were obtained. Prior to doing this, NG and DEGDN peak areas were examined to determine that they remained constant across the diameter of propellant samples and could serve as an internal standard.

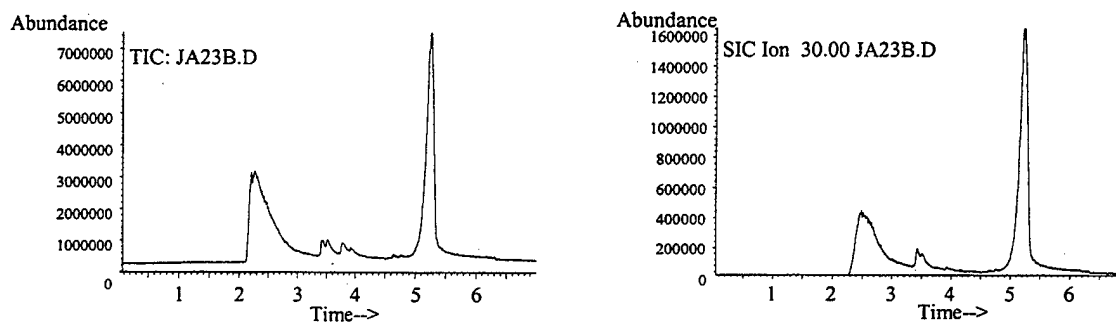


Figure 1. Typical TIC and SIC for D-GC-MS analysis of JA2.

3. Results and Discussion

3.1 Microscopy

Figure 2 shows a composite photo of an entire M30 grain that had been directly exposed to a plasma ignition source in a closed bomb (Kinkennon et al. 2000). The grain has pits, gouges, chasms, and cracks in the surface. The sides of the grains closest to the plasma had the greatest amount of damage, but even surfaces of grains facing the outer wall of the bomb had small pits. The perforations (not shown) contained gouges and abnormally burned regions (wormholes) that extended into the web.

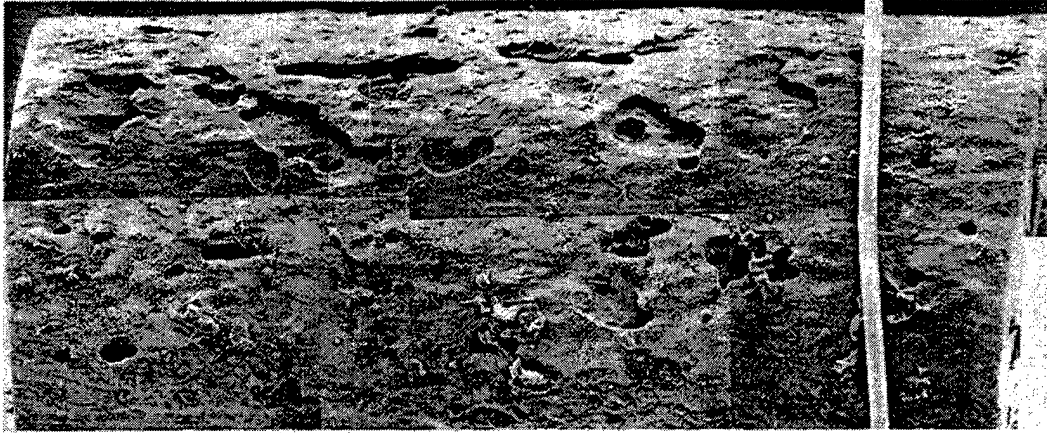


Figure 2. SEM photo of M30 grain exposed to plasma ignition source (Kinkennon et al. 2000).

The SEM photos of JA2 in Figures 3 and 4 show a different kind of response to the plasma. The photos in Figure 3 are from the ETC-closed bomb experiment and show an almost “lacy” texture on the surface closest to the plasma, while the photos in Figure 4 show blisters that form on the surface of JA2 when exposed to the plasma in an open-air configuration. The cross section of the blistered surface, shown in Figure 4(b), reveals a subsurface pocket. A high magnification photo taken downstream of the area that experienced direct exposure to the plasma shows evidence of pitting. These features were not observed in the photos of virgin propellant (not shown).

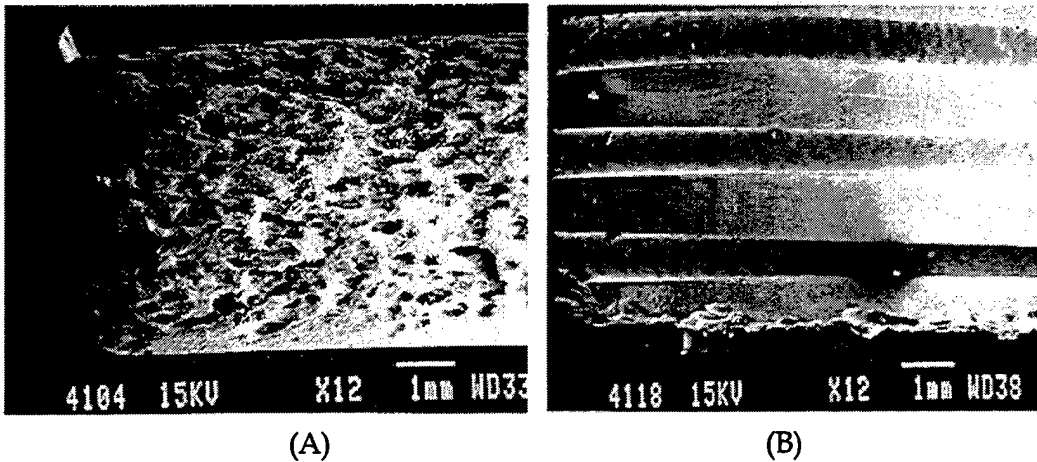


Figure 3. SEM photos of a 7-perf grain of JA2 recovered from an interrupted ETC-closed bomb experiment (35-MPa blowout). (A) Exterior surface of grain, located directly adjacent to plasma source, and (B) cross-sectioned grain showing three “striped” perforations and a section of web below the exposed surface (bottom of photo).

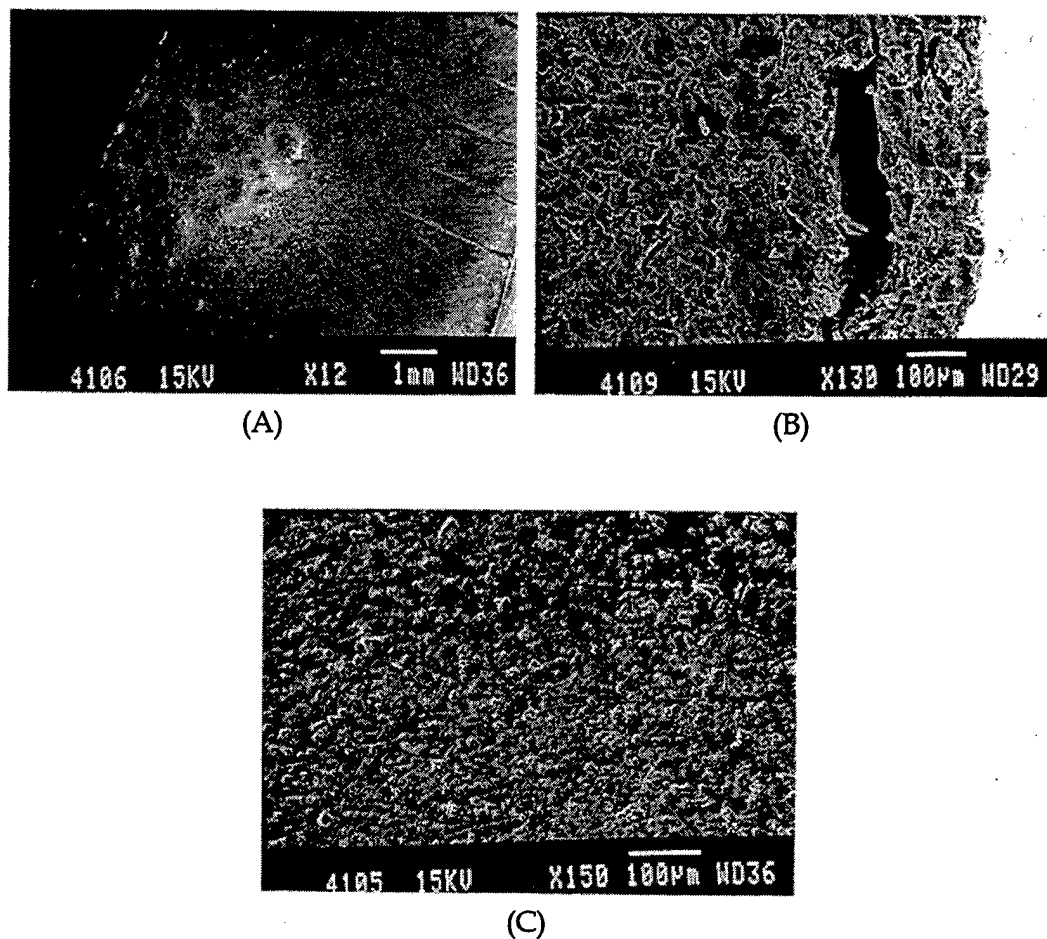


Figure 4. SEM photos of a 2.5-mm-thick JA2 sheet exposed to a plasma ignition source in an open-air configuration. (A) 12X photo of exposed surface (approximately one-third of the sample has been cut away); (B) 130X photo of cross section of left edge of sample in Figure 4(a); and (C) 150X photo of section of propellant near the top right-hand corner of Figure 4(a).

3.2 P-GC-MS

For all samples, regardless of the ignition source used, it was determined that plasticizer concentrations remained constant across the diameter of the sample (data not shown). This is consistent with high-performance liquid chromatography (HPLC) results reported for M30 and JA2 in a previous study (Kaste et al. 1999). It was observed, however, that the levels of NO in the propellant varied with distance from the exposed surface. Because of tailing and overlapping of the chromatographic peak for NO, it was difficult to quantify by integration of the peak. Peak height was therefore used instead for this purpose and was normalized to the plasticizer peak. Results for the "NO ratio" for plasma-exposed M30 and JA2 suggest in-depth reaction leading to the generation of NO. Figure 5 shows profiles

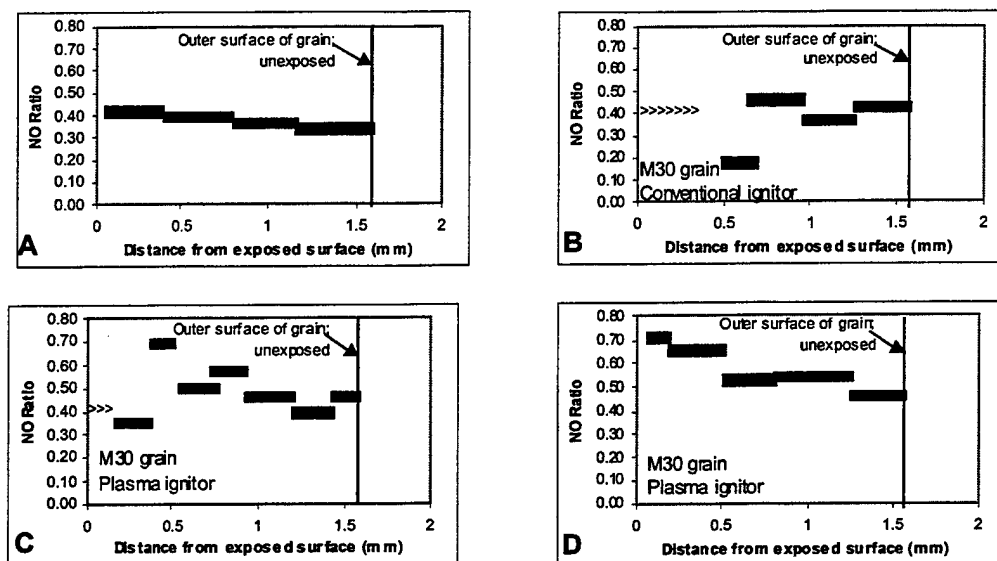


Figure 5. NO ratios for M30 propellant grains. Nominal thickness is 1.55 mm. Filled boxes represent NO level in slice whose thickness is length of the box; ">>>>" indicates regression from the surface.

for NO ratios for M30 exposed to conventional and plasma ignition sources. Figures 6 and 7 show the same for JA2 sheet and grains, respectively. In each of the figures, the vertical bars represent the level of NO in a slice whose thickness is the length of the given bar. Repeated "greater-than" signs (">>>>") indicate how far the exposed surface has regressed as a result of the exposure, and in some cases, of subsequent sustained combustion.

The data for M30 in Figure 5 indicates that the level of NO is nearly constant in unexposed material (Plot A). The level for M30 exposed to a conventional ignition source (Plot B) is approximately the same as for unexposed material, except for the first slice, which is significantly lower. This lower value is the result of char at the surface (presumably black powder residue) that contributes to the sample's mass and thickness, but not to its energetic material or NO level. Data for plasma-exposed M30 (shown in Plots C and D) indicate the presence of elevated NO levels at or directly below the exposed surface. The section of the sample used to generate the data in Plot C regressed more than did the section used to generate the data in Plot D, leaving a small amount of residue that does not contribute to its NO content. However, the level in the second slice is nearly twice that of the baseline, unexposed material. The same is true for the first two slices shown in Plot D; little regression occurred in this area. From these plots, it is estimated that NO-level enhancement occurs as deep as 0.5 mm into the surface, and it is concluded that profiles for radiation-induced in-depth chemistry are consistent with Beer's law (i.e., the effect is greatest at the surface and decreases as the distance from the exposed surface increases).

Figure 6 gives the data for JA2 sheet exposed "head-on" to plasma. Plot A is for unexposed JA2 sheet and serves as a baseline for comparison. Plot B gives data obtained from a sample that was approximately 7 mm away from the area of direct exposure. The sample was covered with residue, but to the naked eye was not otherwise different in appearance than unexposed propellant. Examination by SEM (see Figure 4c) indicates that there was some damage to the sample even downstream from the area of direct exposure to plasma. The data for this area suggests an NO-level enhancement at a depth of 1-1.5 mm. Plots C and D are for two different areas of the section that received direct exposure to the plasma and was obviously blistered and somewhat pitted (as the sample shown in Figure 4a). The data for these areas suggest NO-level enhancement at a depth of 0-0.75 mm.

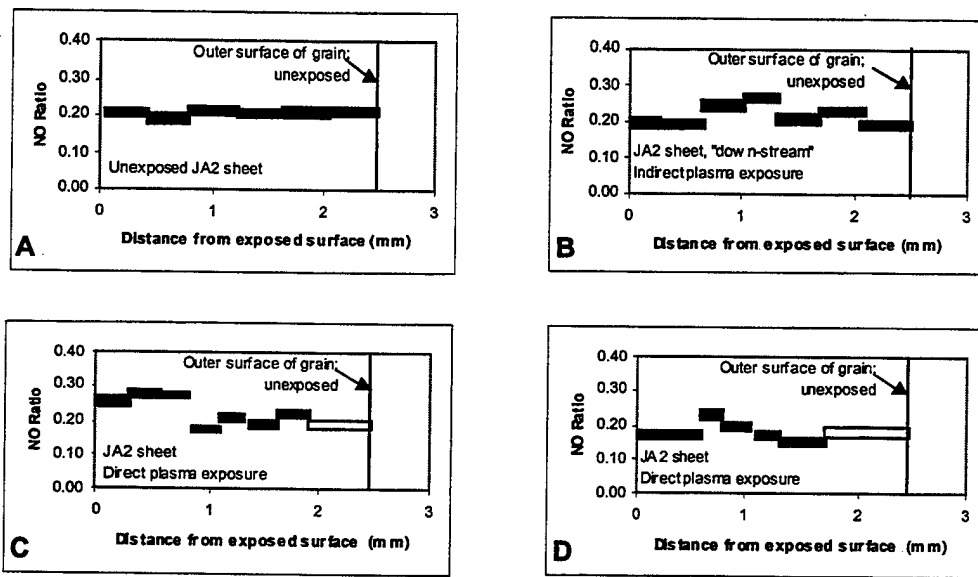


Figure 6. NO ratios for JA2 propellant sheet. Nominal sample thickness is 2.50 mm. Filled boxes represent NO level in slice whose thickness is the length of the box. Unfilled boxes represent slices for which data was not measured; nominal NO level is assumed.

Figure 7 gives data for JA2 grains exposed to both conventional and plasma ignition sources (micrographs not shown). Neither sample shows any evidence of NO-level enhancement. The plasma-exposed sample actually shows a decreased NO level at the exposed surface. As in the case of the conventionally ignited M30, this is likely due to the fact that the surface regressed significantly and left a residue that contributes to mass and thickness of the sample, but not to its NO level.

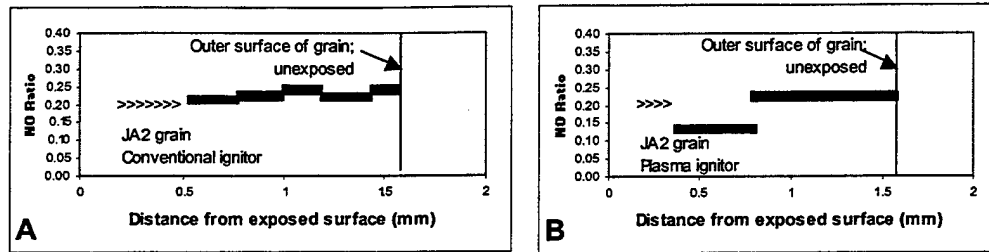


Figure 7. NO ratios for JA2 propellant grains. Nominal sample thickness is 1.55 mm. Filled boxes represent NO level in slice whose thickness is the length of the box; ">>>" indicates regression from surface.

The "spottiness" of NO enhancement for plasma-exposed JA2, as opposed to that for plasma-exposed M30, suggests that something other than absorption of radiation by nitrate esters is the dominant factor in the NO production. It is proposed that graphite is this factor, and that upon exposure to plasma, blisters form from gas forming due to degradation of nitrate esters surrounding large graphite particles. As can be seen in Figure 8, which shows a photo of virgin JA2 (2.5 mm thick) taken by (back lit) optical microscopy, there is a distribution of particle sizes of graphite in the propellant. In the particular sample examined, particle sizes as large as 40 μm were observed. MIL-SPEC graphite is in the form of platelets (Worrell 2000), so estimates of particle size are actually estimates of particle diameter, and assume the platelets are positioned parallel to the plane of observation. Estimation of temperature increases possible assuming black body absorption by the graphite (given the specific heat, density of graphite, and radius of a graphite particle or agglomeration and given the heat flux produced by the plasma and heat loss terms) are well in excess of the temperatures necessary for denitration of the nitrate esters immediately surrounding the graphite particle (Miller 2000). The importance of graphite in the absorption of plasma radiation by JA2 propellant has been reported previously by German researchers (Voronov et al. 2000; Kappan and Bauder 2000) and was speculated to result in in-depth decomposition of nitrate ester groups, but was not associated with the "spotty" degradation of plasma-exposed JA2, nor was any chemical analysis performed to confirm in-depth chemical (as opposed to purely morphological) changes.

It is suspected that the significance of enhanced NO levels is related to the ability of the radiative and/or conductive components of the plasma to affect the propellant in-depth and somehow "prime" it for decomposition, perhaps by an increase in temperature, an increase in porosity or microcracking (to facilitate flame-spreading), a change in optical properties, or by otherwise generating material that is more readily combusted. The M30 appears to be affected at a depth on the order of 0.5 mm, whereas the JA2 appears to be affected to a depth of 0.75 mm. In hindsight, the observed enhancement of NO levels is not very surprising given the blisters observed on the surface of JA2 propellant (see Figure 4a). It is proposed that in-depth reaction results in the generation of decomposition gasses that can cause an



Figure 8. Optical microscope photo of a 2.5-mm-thick sheet of virgin JA2.

upwelling of propellant. Depending on the degree of in-depth reaction, the upwelling may result in just a blister, or may burst and leave pits and protrusions at the exposed surface. The consequence of this is an increase in surface area, which in turn will result in an increased burning rate. Chemical analysis of M9 propellant, which contains no graphite but is more energetic than JA2, was not performed because the propellant did not survive the plasma-exposure experiment. It is proposed (Beyer 2000) that the M9 decomposes very rapidly when exposed to the plasma and actually “blows to bits” while still in the small-scale chamber. It is possible that this results from in-depth absorption of radiation that in turn results in in-depth decomposition/gasification which results in the failure in mechanical integrity. The fact that the profiles for in-depth radiation effects in nitrate-ester-rich M30 are consistent with Beer’s law, while those for similarly nitrate ester-rich JA2 are not, is proposed to be due to the multiple internal reflections experienced by radiation at the interfaces of NQ and nitrate ester binder (i.e., scattering increases the path length of the photons and therefore its probability of being absorbed).

4. Conclusions

- A microscopic examination of plasma-exposed M30 and JA2 has been performed. The examination indicates that M30 and JA2 respond very differently to the plasma.

- In spite of the differences in *how* M30 and JA2 are damaged, it is clear that both experience an increase in surface area as a result of plasma exposure, and this surface area increase can contribute to a concomitant increase in burning rate.
 - Some effects of radiation have been elucidated. For JA2, it appears that absorption of radiation by graphite is the dominant phenomenon, while for M30, absorption of scattered radiation appears to be dominant.
 - For both M30 and JA2, radiation effects are limited to top 1 mm. This is likely not true for M9 and graphite-free JA2. RDX-filled TPE (non-nitrate ester) were examined, but results (not reported here) were inconclusive.
-

5. Future Work

- Characterize graphite-free, plasma-exposed JA2.
- Modify D-GC-MS method to detect in-depth chemistry in non-nitrate ester propellants.
- Study effect of absorption of UV radiation by propellant ingredients and potential additives.
- Continue to investigate effects for filled/unfilled propellants.
- Continue work to understand why plasma-induced damage in M30 is so irregular and what initiates pitting.

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13. ABSTRACT (Maximum 200 words) JA2 and M30 recovered from interrupted ETC-closed bomb, interrupted ETC small-scale chamber, and open-air experiments were subjected to chemical and microscopic analysis. Evidence of subsurface reaction in both M30 and JA2 has been discovered using a desorption-gas chromatography-mass spectroscopy (D-GC-MS) method to detect low levels of NO in the propellant. It appears that for M30, profiles for radiation-induced denitration of nitrate esters are consistent with Beer's law, and that effects occur as deep as 0.5 mm into the exposed surface. Radiation-induced denitration in JA2 has been detected as deep as 0.75 mm from the exposed surface, but profiles are not consistent with Beer's law. It is suspected that denitration at and below the exposed JA2 surface occurs mostly in locations where large graphite particles are found. Rough estimates of graphite particle temperature when exposed to plasma radiation are in excess of those required for denitration to occur. Microscopic examination of plasma-exposed propellant indicates several features (pits, gouges, blisters, wormholes, etc.) that increase the surface area of the propellant and can contribute to an increased burning rate.				
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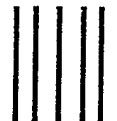
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