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A COMPREHENSIVE REVIEW OF BLACK POWDER

Ronald A. Sasse'

January 1985

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A diligent attempt has been made to scrutinize black powder and the charcoal it contains as fully as possible, utilizing modern testing techniques and various analytical chemical procedures. Although the tests performed are well established, their application to define a porous propellant represents a new point of view for investigating structure. From S.E.M. microphotographs and compaction studies, it is suggested that the pressing action used to make black powder results in plastic flow that produces a conglomerate

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and cohesive mass containing a matrix of inter-connecting passageways. The degree of openness of black powder grains and, in particular, internal surface area, pore volume, internal free volume, and density were all found to be related to burn rate. Thermodynamic calculations were cited that relate computed theoretical values to experimentally determined quantities. Closed bomb combustion was compared to strand burn-rates and it was suggested that closed bomb data embrace more than one combustion mode and as such do not directly relate to bulk values. From this interrogation, chemical and physical properties were related to combustion phenomena. As work progressed it became evident that material produced by the Indiana Army Ammunition Plant will be, or has become, the most successfully characterized black powder produced in the United States.

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I. INTRODUCTION

Black powder was made in production mills in Europe as early as 1340 in Augsburg, 1344 in Spandau and 1348 in Legnica. The process, traceable to the early B.C. efforts of the Chinese, evolved as an "art form" having historical rather than scientific origins. The development of the craft has been reconstructed by various authors and excellent accounts have been given by Urbanski,[1] Marshall,[2] and Vieille.[3] The subject has been reviewed by Fedoroff and Sheffield,[4] who provide numerous references and a very recent work by Gray, Marsh and McLaren[5] reproduces detailed historical drawings of the early gun powder facilities.

In essence, black powder is made by grinding potassium nitrate, sulfur, and charcoal into a fine powder. This meal is transformed into a cohesive conglomerate mass by either forming a paste which is forced through and divided by a screen, or it is directly pressed into a cake. Both forms are subsequently broken into pieces of a particular size. The small lumps or grains of black powder are then coated with graphite to retard the absorption of moisture and to prevent the grains from adhering. This process has been standardized to some degree, in accordance with prevailing technology, but the operator adds water, adjusts temperature, selects grinding time, or changes compression pressure based on personal experience, which is guided by individual judgment of color, odor, or general perception of the condition of black powder as it undergoes its various transformations. Such judgments are exchanged from experienced operator to journeyman.

Although the production of black powder was as great as 10.6 million pounds[6] during World War I, the advent of smokeless powder diminished demand. This fact, together with the inherent dangers of production marked by occasional explosions, has resulted in the closing of most manufacturing facilities in North America. The only production plant supplying the military is the Belin Powder Works in Moosic, PA, which was built in 1911-12, embracing the technology of 1850. The method of manufacture is termed the "standard process" and incorporates a ball and wheel-mill to prepare the meal. The

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[1] *T. Urbanski, Chemistry and Technology of Explosives, Vol. 3, Pergamon Press, NY, pp. 322-346, 1967.*

[2] *A. Marshall, Explosives, 2nd. Ed., Vol. 1, Blakiston's Son and Co., Philadelphia, PA, 1917.*

[3] *Memorial Des Poudres Et Salpetres, Vol. 6, Chapter 2 by P. Vieille, Gauthier-Vallars Et Fils, Imprimeurs-Libraires, Paris, pp. 256-391, 1893.*

[4] *B.T. Fedoroff and O.E. Sheffield, Encyclopedia of Explosives and Related Items, Vol. 2, PATR 2700, Picatinny Arsenal, Dover, NJ, pp. B165-B178, 1962.*

[5] *E. Gray, H. Marsh, and M. McLaren, J. Material Science, Vol. 17, pp. 3385, December 1982.*

[6] *A.P. Vangelder and H. Schatter, History of the Explosive Industry in America, Columbia University Press, NY, 1927.*

plant was operated by E.I. DuPont de Nemours & Co., who sold the facility to GOEX Inc. in April of 1973. Because black powder still plays an important role in fuses and ignition devices, the U.S. Army Corps of Engineers started to construct a production plant in Charlestown, IN, in 1974. The installation was completed in 1978 and it was operated by ICI Americas Inc. One principal departure from standard practice was the inclusion of a jet-mill to grind material as developed by Kjell Lovold,[7] which has been used in Norway to manufacture black powder. Every effort was made to modernize the new black powder manufacturing process and to make production as safe as possible. These concepts were translated into hardware that resulted in the first production cycle completed in early 1983. In the planning stage, various studies were conducted to: (1) elucidate the ball and wheel-mill processes, (2) investigate new techniques and equipment, and (3) compare the ballistic properties of black powder originating from this and other countries. This work was reported in part by the Chromalloy Corp.,[8] Battelle Memorial Institute,[9] Olin Corp.,[10] and ICI Americas Inc.[11] Battelle published abstracts of 50 articles and 60 patents describing the wheel-mill process in fine detail and gave particle size distribution as a function of grinding time and moisture content for both the jet and wheel-mill processes. The accumulated knowledge was used as a foundation for the design of the Indiana Plant.

The performance of black powder, in contrast to its preparation, has been characterized in the classic papers of Noble and Abel.[12,13] Recently, Williams[14] summarized burning characteristics and discussed several foreign

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[7] *K. Lovold, U.S. Patent No. 3660546, "Process for the Preparation of Black Powder," 2 May 1972.*

[8] *Chromalloy Corp., "A Study of Modernized Techniques for the Manufacture of Black Powder," Propellex Chemical Division, Final Report No. DAI-23-072-501-ORD-P-43, Chromalloy Corp., Edwardsville, IL, January 1960.*

[9] *H.E. Carlton, B.B. Bohrer, and H. Nack, "Battelle Memorial Institute Final Report on Advisory Services on Conceptual Design and Development of New and Improved Processes for the Manufacture of Black Powder," Battelle Memorial Institute, Columbus, OH, October 1970.*

[10] *J.R. Plessinger and L.W. Braniff, "Final Report on Development of Improved Process for the Manufacture of Black Powder," RCS AMURE-109, Olin Corp., Indiana Army Ammunition Plant, Charlestown, IN, 31 December 1971.*

[11] *Indiana Ammunition Plant, "Black Powder Manufacturing Facility," Vols. 1 and 2, Indiana Army Ammunition Plant, Charlestown, IN, 1975.*

[12] *R.A. Noble and F.A. Abel, Phil. Trans. Roy. Soc., London, Series A, Vol. 165, pp. 49-155, 1875.*

[13] *R.A. Noble and F.A. Abel, Phil. Trans. Roy. Soc., London, Series A, Vol. 171, pp. 203-279, 1880.*

[14] *F.A. Williams, "The Role of Black Powder in Propelling Charges," Picatinny Arsenal Tech. Report No. 4770, Picatinny Arsenal, Dover, NJ, May 1975.*

references. Rose[15] studied the effects of various charcoals on performance and concluded that volatile content was an important parameter; he also wrote an excellent review article.[16] Kirshenbaum[17] concluded from differential thermal analysis, DTA, that even when volatiles were removed from various charcoal samples this treatment did not alter the order of ignition; thus, it was concluded that some property other than volatiles also affected ignition. He also reported that ignition was not a property of surface area, ash content, or sulfur content. Blackwood and Bowden[18] suggested sulfur reacts with volatiles as an initiation step, but Kirshenbaum showed all three components, potassium nitrate/sulfur/charcoal, must be present to induce the low temperature pre-ignition exotherm. Clearly, volatiles affect burning rate, and this is shown by Hintze[19] who recommends 82% carbon while Blackwood and Bowden suggest 70%. Neither work relates these values to the standard form of water or ash-free basis, and it is not clear as to the exact state of the starting material; therefore, these recommendations cannot be compared directly. However, both studies recommend a high volatile content. DuPont has maintained a standard of 75% carbon since the 1930's and this practice has been followed by GOEX.

In this discussion it is emphasized that the properties of charcoal are important, but the exact requirements to make a good ballistic product are elusive. Present practice is that charcoal be made from hardwood, currently maple being selected in the United States, and that it be of low, 5% or less, ash content. No American specification exists citing volatile content but current practice uses material of 20-30%. The current specification does not cite the type of wood, particulars relating to pyrolysis, nor any required physical property. One control on black powder is it must conform to the density range of 1.72 to 1.80 g/cm^3. The amounts of potassium nitrate/sulfur/charcoal are given, but compaction pressure and particle size are not. Such general criteria result in a large latitude of allowable variance in making black powder.

From the historical significance of black powder, it would appear that the manufacturing technology has been developed; however, one general observation is the same manufacturing procedure, using the same ingredients, results in lots exhibiting different burning rates. This has been the experience of GOEX and DuPont where fast and slow lots have been blended to achieve a particular result. Although some of the factors that affect burn rate are known, the lot-to-lot variations have not been identified. It was

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[15] *J.E. Rose, "Investigation of Black Powder and Charcoal," IHTR-433, Naval Ordnance Station, Indian Head, MD, September 1975.*

[16] *J.E. Rose, "Black Powder - A Modern Commentary," Proc. of the 10th Symposium on Explosives and Pyrotechnics, Franklin Research Institute, Philadelphia, PA, pp. 14-16, 1979.*

[17] *A.D. Kirshenbaum, Thermochimica Acta, Vol. 18, p. 113, 1977.*

[18] *J.D. Blackwood and F.P. Bowden, Proc. Roy. Soc., London, A213, p. 285, 1952.*

[19] *W. Hintze, Explosivstoffe, Vol. 2, p. 41, 1968.*

for these reasons that the Ballistic Research Laboratory (BRL) has initiated several black powder studies to elucidate the sensitive parameters that control combustion. From scanning electron microscopy (S.E.M.), it was inferred that structure was important, and in developing this hypothesis, it soon became clear that correlations exist between physical properties and combustion. This review will blend both past and current research, focusing on ingredients, physical structure, and combustion, in an attempt to replace some of the mysticism associated with black powder and black powder production.

II. CHARACTERIZATION OF BLACK POWDER

A. Ingredients

No comment is directed to sulfur or potassium nitrate; these chemicals propose no particular technological problem. However, charcoal, a naturally derived substance which contains approximately 35% "tar-like" constituents, varies from one source to another and from one lot to another. Such variance has been studied in detail for maple charcoal,[20] where ten samples from material supplied by one producer and used by the Indiana Black Powder Plant were analyzed. Hydrogen, carbon, nitrogen, sulfur, and oxygen were measured and the elemental composition of ash was determined. Results are given in Table 1 on a weight-percent basis. Physical properties were determined and include true density, volatiles, heat content, surface area, and pore volume. The extreme ranges of ten samples are summarized in Table I, whereas individual values for each analysis are given in Reference 20. Freedman[21] used averages of this data, where the empirical formula for charcoal was taken to be $C_{14.57}H_{7.17}O_{1.00}$ to compute thermodynamic properties; his results were the data base for closed bomb evaluations.[22] The empirical formula used to represent DuPont and GOEX charcoals was $C_{8.68}H_{4.96}O_{1.00}$.

An interesting observation is the low surface area of this charcoal, and thus, it is inferred that most of the pores must be plugged, for a completely carbonized material would have a surface area of about 1000 M^2/g.

Another disturbing aspect of this data is the range of carbon and volatile content where some values are at the extreme limits of what is believed to make good black powder. The data illustrates the problem of making a reproducible product using an ingredient the composition of which

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[20] *R.A. Sasse', "Characterization of Maple Charcoal Used to Make Black Powder," ARBRL-MR-0332 Ballistic Research Laboratory, USA-ARRADCOM, Aberdeen Proving Ground, MD, November 1983. ADA-136-513.*

[21] *E. Freedman, "The Thermodynamics of Real and Unreal Black Powder," Proc. of the 20th JANNAF Combustion Meeting, CPIA Publication No. 388, Vol. I, pp 511, October 1983.*

[22] *R.A. Sasse', H. Holmes, D. Hansen, W. Aungst, O. Doali, and R. Bowman, "Evaluation of Black Powder Produced by the Indiana Army Ammunition Plant," ARBRL-TR-in press, Ballistic Research Laboratory, USA-ARRADCOM, Aberdeen Proving Ground, MD, 1984.*

TABLE 1. CHARACTERIZATION OF MAPLE CHARCOAL

carbon	72-----81	SiO_2	16----43	enthalpy	11400-13000
hydrogen	2.9---3.5	CaO	28----44	Btu/lb	
nitrogen	0.30-0.48	Al_2O_3	12----14	surface area	2.2-15.7
sulfur	0.01-0.02	K_2O	8.4-12.9	M^2/g	
oxygen	12.6-15.4	Na_2O	1.0--1.8	pore volume	2---10
ash	2.5--10.4	Fe_2O_3	2.3--4.9	mm^3/g	
volatiles	22-----30	MgO	1.9--3.7	true density	1.44-1.56
		P_2O_5	1.2--2.1	g/cm^3	

Values are in weight percent unless otherwise noted.

cannot be closely controlled. To address this problem, research has been directed to identify pure organic compounds that could be substituted directly for charcoal.[23] Polyphenols, diacids, and phthaleins gave promising results. Testing must be conducted before such propellants can be properly considered as viable black powder substitutes to include: card gap, drop weight, and friction sensitivity.

B. External Structure of Grains of Black Powder

The size latitude of class one black powder is large and it is defined as material which will pass through a number four sieve (4.75 mm) and not a number eight sieve (2.36 mm). Even though lots of black powder met this criteria, some lots appear to be composed of different size grains from others, suggesting that a more quantitative measure be undertaken. Size distributions were determined[22] for DuPont 111-12, GOEX 75-44, and Indiana 1983 black powders and results are given in Table 2. These materials are referred to later without using lot identifiers. Each sample had a slightly different distribution, and in all cases, the function was not pronounced. This broad distribution should be taken into account in combustion modeling studies and will be addressed later.

Another characteristic of black powder is its graphite coat, and little information exists to describe this envelope or its function. One characteristic of the coating is to keep the grains from adhering to one

[23] *(a) S. Wise and R.A. Sasse', "Organic Substitutes for Charcoal in Black Powder Type Pyrotechnic Formulations," Proc. of the 19th JANNAF Combustion Meeting, CPIA Publication No. 366, Vol. II, pp. 305, October 1982.*

(b) S. Wise, R.A. Sasse', and H.E. Holmes, "Organic Substitutes for Charcoal in 'Black Powder' Type Pyrotechnic Formulations," ARBRL-TR-02569, Ballistic Research Laboratory, USA-ARRADCOM, Aberdeen Proving Ground, MD, July 1984.

TABLE 2. GRAIN SIZE DISTRIBUTION

Largest Size in mm	4.75	4.00	3.35	2.80	2.36	2.36
GOEX	0.31	9.48	26.69	22.25	28.46	11.70
DuPont	3.39	33.23	36.13	17.31	8.82	0.65
Indiana	2.24	22.20	29.80	25.58	18.02	1.53

another, and a second role is to prevent absorption of water. From high speed cinematography of a single burning grain, it appears that the coating acts as an inhibitor, and as such, it is a very important part of black powder. To show the influence of this coat, samples of black powder were obtained just before graphite was added to the glaze barrel; these are called green grains, and later the corresponding graphited material was also selected. The flame-spread rates of these two samples differed by a factor of two showing the retarding effect of the graphite coat. One attempt to examine this coating was undertaken in which but one DuPont grain was examined using S.E.M. techniques. Microphotographs were taken of the flat side of the hemisphere created by cleaving the grain in half, much like snapping a twig, such that the surfaces were not marred by a tool. From the pictures, the graphite coat was judged to be about five microns thick. Future work will attempt to correlate electric conductance of a black powder bed to graphite film thickness.

C. Internal Structure of Black Powder

S.E.M. microphotographs of class one black powder[24] were obtained by first cleaving the grain in half as described. Two samples from the jet and wheel-mill are given in Figures 1 and 2 at two different magnifications. Comparison of the figures shows the jet-mill produces a smaller and more uniform distribution of particles. In addition, the charcoal pores were not filled with either potassium nitrate or sulfur, a property that had been ascribed to the pressing action of the wheel-mill[3] by the concept termed "incorporation." From these and other such photographs, it was noted that the degree of openness of the grain increased as did its burning rate. A revealing feature of the photographs was the lack of crystalline sharp edges, and it became apparent that a dominant feature was plastic flow that created voids and porosity in the grain. This feature was quantified by determining internal surface area and pore volume. Brunauer, Emmett, and Teller, B.E.T., gas absorption techniques were used to measure surface area, and values of 0.5 to 1.0 M^2/g were obtained where the larger surface areas were proportional to

[24] *(a) R.A. Sasse', "The Influence of Physical Properties of Black Powder on Burning Rate," Seventh International Pyrotechnics Seminar, Vol. 2, ITT Research Institute, Chicago, IL, p. 536, July 1980.*

(b) R.A. Sasse', "The Influence of Physical Properties of Black Powder on Burning Rate," ARBRL-TR-02308, Ballistic Research Laboratory, USA-ARRADCOM, Aberdeen Proving Ground, MD, March 1981, (AD A100273).

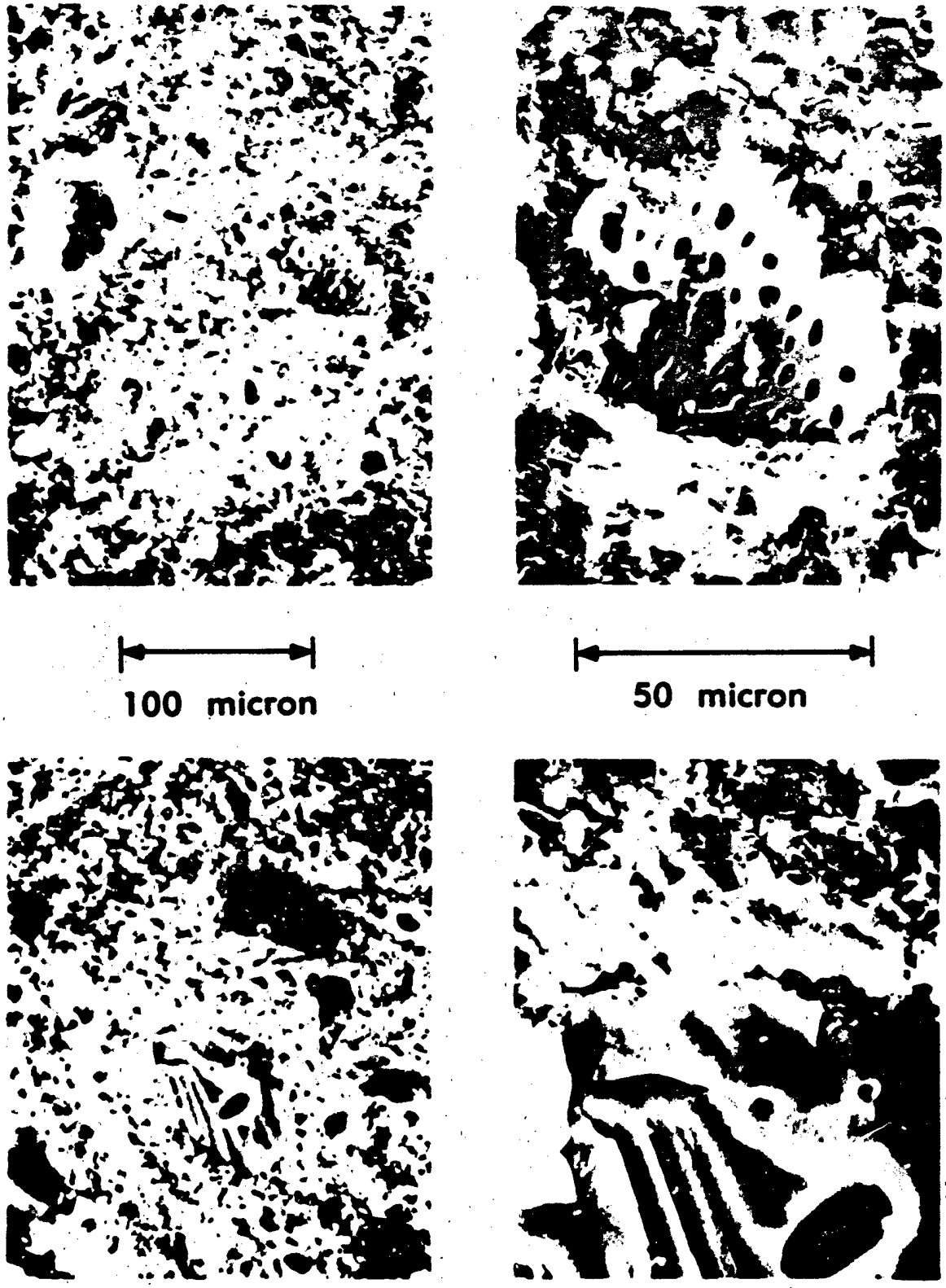


Figure 1. Interior of Black Powder Grain Produced by Wheel Mill Process

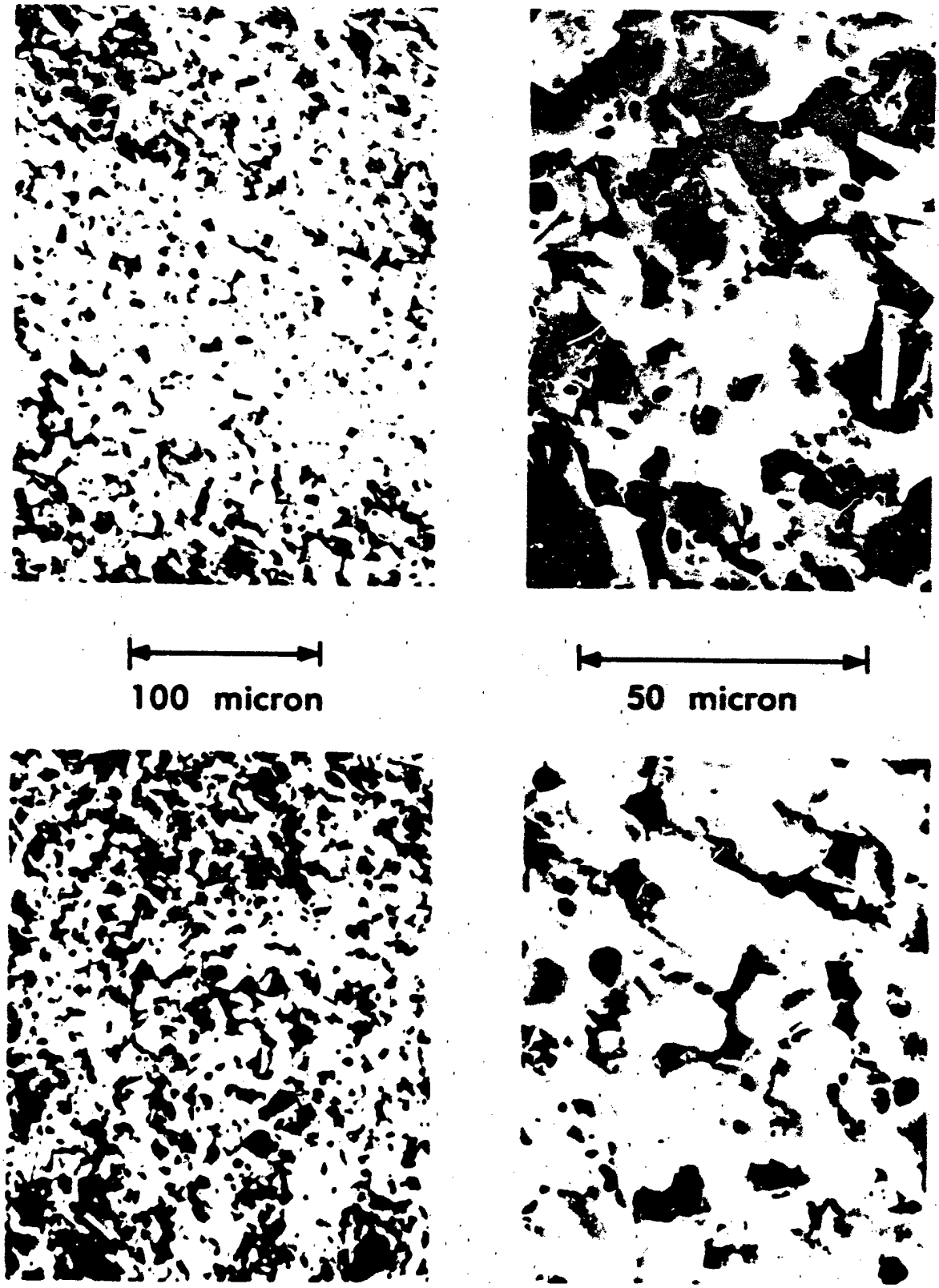


Figure 2. Interior of Black Powder Grain Produced by Jet-Mill Process

the faster burning material. The same result was found with pore volume as determined by mercury intrusion porosimetry, where values ranged between 0.01 to 0.04 cm^3/g for both whole and cleaved grains. Thus, the mercury penetrated the entire grain without hindrance from either the graphite coat or internal structure. In addition, the mercury intrusion data showed that passageways had mean diameters of about 0.1 micron at the narrowest cross-section.

Another method of determining the degree of openness of black powder is to determine internal free volume. This quantity can be calculated from the bulk and true densities where sample volumes are measured two different ways: bulk density is determined from the volume taken equal to the amount of mercury displaced by the sample, and true density is given by the amount of helium displaced. Since mercury does not enter the pores as does helium, these differences can be related to void space. Indiana, DuPont and GOEX black powders [22] had bulk densities of 1.75 to 1.79, true densities of 1.94 to 1.97, and internal free volumes were 4.10 to 5.75%. Also, the maximum theoretical density for this black powder would be 1.97 using the average true density of 1.45 for charcoal (see Table 1) and the density values for sulfur and potassium nitrate. The calculated value is almost equal to the measured values of true density.

All of these measurements support the conclusion that the compaction step used in making black powder induces local plastic flow producing a fused conglomerate and cohesive mass containing a matrix of interconnecting passageways and that the degree of openness of a black powder grain is directly related to burning rate. These concepts immediately suggest the question addressed in the next section, "How do we quantify the compaction process and apply this knowledge to make reproducible laboratory samples and reproducible production lots?"

III. COMPACTION OF BLACK POWDER MEAL

Black powder meal is pressed into a cake using various pressures and different moisture contents to achieve a particular grain density. This process was examined in detail[25] by pressing meal slowly in a material testing instrument, an Instron, and recording both the pressure and density of the sample. The small displacement rate of 0.254 mm/min was selected and a total load of 3,000 g/cm was applied slowly to samples of different moisture content. Results are given in Figure 3, where the early portion of the curve represents movement of material into a close packed geometry and the linear portion reflects plastic flow. Had the experiment been extended to higher

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[25] *(a) R.A. Sasse', "Strand Burn Rates to One Hundred Atmospheres," Eighth International Pyrotechnics Seminar, ITT Research Institute, Chicago, IL., p. 588, July 1982*

(b) R.A. Sasse', "Strand Burn Rates to One Hundred Atmospheres," Proc. of the 19th JANNAF Combustion Meeting, CPIA Publication No. 366, Vol. I, p. 13, October 1982.

(c) R.A. Sasse', "Strand Burn Rates to One Hundred Atmospheres," ARBRL-TR-02490, Ballistic Research Laboratory, USA-ARRADCOM, Aberdeen Proving Ground, MD, May 1983, (AD-A129-087).

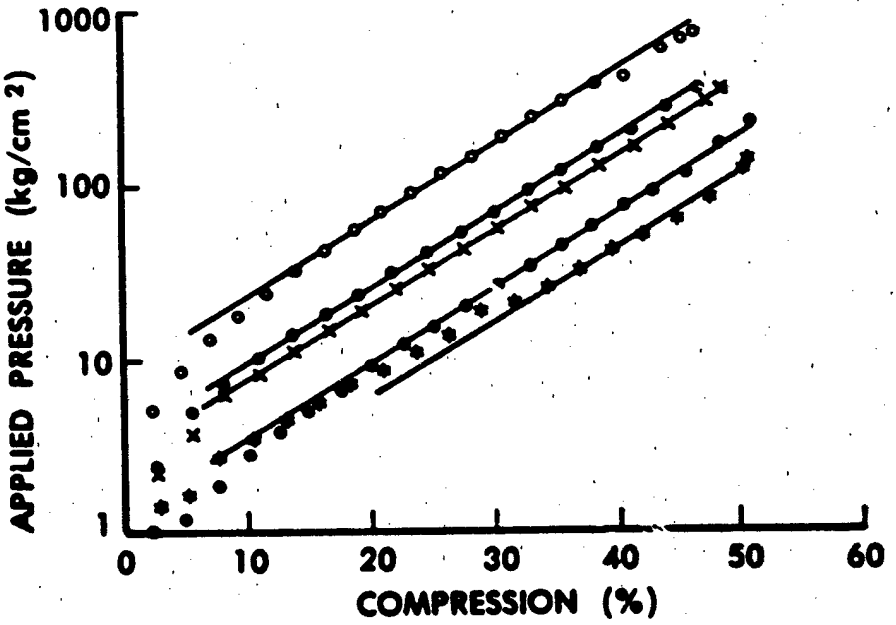


Figure 3. Effect of Pressure on Black Powder Meal: 0, 0%; ⊕, 1%; +, 2%; ⊕, 3%; and *, 4% Water

pressures, the curve would approach a vertical asymptote near values of 65% compaction. A direct relationship is shown between the logarithm of applied global pressure to density; this functionality was suspected to be the result of compressing bounded spheres. This idea is supported by Knudsen[26] who shows that porosity is a logarithmic function of the contact area between spheres. To evaluate these concepts and assess the effect of particle size, a collection of two different diameter copper spheres, 50 and 125 microns, were individually compressed. The resulting curves were logarithmic and could be superimposed one upon the other. Apparently, particle size does not influence grain density at particular compaction pressures and particular water contents. Also, these experiments indicate the logarithmic relationship is the result of plastic flow among a collection of spheres. This same relationship was even shown for the much larger class one grains where such results can be applied to the fabrication of fuse and time delay elements.

The application of physical testing to evaluate the compaction process has been successful, and the results can be applied to fabrication technology, dwell-time studies, and to the preparation of laboratory samples. Since plastic flow was found to be dominant, an equivalent process was adopted where samples were pressed in a constant volume die using a spacer to control piston travel; this avoided the difficulty of simultaneously controlling both pressure and water content. The technique offered the advantage of fabricating a series of samples having predictable densities by placing known weights of meal into a parallelepiped die forming "sticks" 4x5x20 mm. Due to a slight degree of elasticity, bulk densities were calculated from the final measured dimensions of the extruded samples. These samples were inhibited with a coat of cyanoacrylate-based glue for strand-burn rate studies.

The preceding sections presented the chemical and physical properties of black powder; subsequent data and discussion will pertain to combustion.

IV. ATMOSPHERIC STRAND BURN RATES

Inhibited black powder sticks of different densities were made from maple and oak charcoal meals that were ground in the Indiana pilot plant jet-mill. They were burned in air at atmospheric pressure and photographed by cinematography at 2000 frames per second.[25] The position history of the burning interface was plotted as a function of time and the least square slope was used to calculate burning rate. The deviation of the slope was taken as the precision of one measurement and is given as an error bar on the data points drawn in Figure 4. Later, it was found that some deviations among these measurements was traceable to a poor inhibitor coat, indicated in the movies by a growing crown forming at the burning interface. These instances could be identified and discarded. From the remaining subset, burn-rates of about one centimeter per second were obtained, and they are shown to be a linear function of density. Black powder containing maple charcoal burned about 20% faster than oak. One additional experiment was performed to compare the burning rates of two sticks of black powder that were made to equal densities where one was compressed damp and the other was pressed dry at a

[26] F.P. Knudsen, J. Am. Ceramic Soc., Vol. 42, No. 8, p. 376, August 1959.

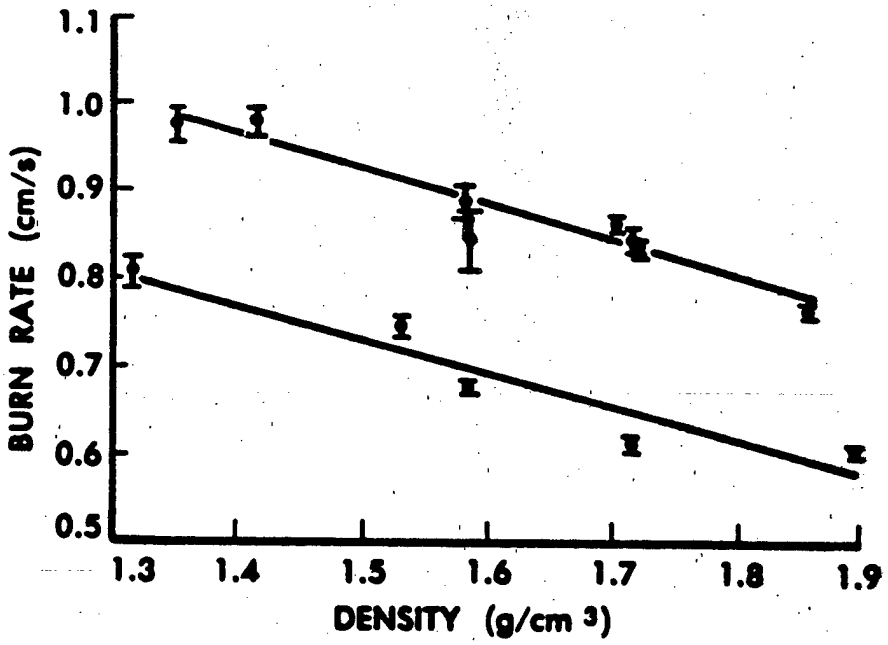


Figure 4. Strand Burn Rates at Atmospheric Pressure as a Function of Density. Lower Curve Oak and Upper Curve Maple Black Powders.

higher pressure. Both were dried and the two burning rates were found equal, and, in effect, the samples did not reflect their different respective preparation histories.

In a further attempt to evaluate class one black powder, Indiana graphited class, grains were ground, sieved, pressed into sticks, and strand-burn rates measured. A similar sample was made using Indiana green grains, and the two burn rates were similar, 0.89 and 0.91 cm/s. It was concluded that the small amount of graphite in one sample did not effect the result. GOEX and DuPont samples had equal but slightly higher values of 1.00 cm/s. In these measurements, original grain size or distribution of sizes is negated as is any effect related to the graphite coat or original density of the grains. Under these circumstances, burn rate is only proportional to chemical composition, original fine degree of grinding, and density of the stick. Thus, these measurements reflect an inherent burning quality. Advantages of the technique are that in photographing burning, one can be certain of the mode of combustion and insure that the inhibiting coat is adequate. For these reasons, the test appears to have merit and parallels the older DuPont and GOEX methods in which black powder is placed in a lead tube, and its diameter is reduced by drawing the sample through an extrusion bench.

Even though few samples were evaluated, this intrinsic combustion measurement ranks Indiana black powder slower than the others. This is somewhat surprising, for the Indiana meal was ground to a smaller particle size, ca 15 microns, than the other meals; hence its burning rate, now dependent on just particle size, chemical composition and density, should be the fastest. Such a result must be attributed to either a non-optimum volatile content in the charcoal or some other unidentified variable.

To obtain surface burn rates,[25] non-inhibited sticks were evaluated. The surface burned faster than the bulk material and thus a pyramid formed, the sides of which became steeper as combustion progressed. This resulted in four distinct gas plumes jutting outward and normal to the burning surface. The surface that had been adjacent to the movable piston burned three times faster, and the surface formed by the bottom of the die burned six times faster than the bulk burn rate.

One 16mm motion picture frame was enlarged and particle sizes were measured by Nathan Klein of the BRL using a Quantimet 720 ImageAnalyzer.[25c] The larger particles were found to be near perfect spheres having a size distribution that peaked at diameters of 225 to 300 microns; they are believed to be frozen droplets of unreacted material. A second distribution of small porous material of irregular shape and sharp edges was collected and examined by S.E.M. techniques. In this case diameters were between 0.4 to 1.0 micron, and these bodies are thought to be the result of condensation of reaction products.

V. HIGH PRESSURE STRAND BURN RATES

Inhibited maple stick samples were burned while being photographed in a windowed chamber of the design of Kubota.[27] Pairs of samples of different densities were evaluated[25] at several different nitrogen pressures to 100 atmospheres. To minimize the obscuration effects of smoke, the optical path inside the chamber was reduced to 8 mm by inserting two plastic spacers. Results are given in Figure 5. The data contain some scatter and are compared to the average response curve derived from several references summarized by Williams[14] using the values of Belyaev and Maznev,[28] Glaskova and Tereshkin[29] and Belyaev et al.[30] The excellent agreement between the Russian and present study is surprising considering the different charcoals and different preparation procedures employed. The burn rate, or more precisely, the regression rate function, r (cm/s), was determined for pressures, P (atm), between 3 to 100 atmospheres. The relationship:

$$r = 1.72\ P^{(0.164 \pm 0.017)} \qquad (1)$$

was obtained. The burn rate curve exhibited a sharp decrease in slope at pressures of a few atmospheres. The films were examined to see if a different combustion mode, such as deconsolidation, was associated with this transition and no change was recorded. The only physical difference noted was that at low pressures the cell had a carbonaceous deposit on the walls, whereas at high pressures, large frozen droplets were seen; however, films in either case looked much alike. In other experiments, where phenolphthalein was substituted for charcoal,[23b] its burn-rate curve also had a decrease in slope and was similar to those reported here. Since the melting point of the organic, 258 C, and that of potassium nitrate, 334 C, are both below the ignition temperature, ca 450 C, the substituted system could be a liquid-liquid reaction in contrast to the liquid-solid black powder system. This could imply that the "break" in the burn-rate curve could be due to nitrate chemistry as opposed to the nuances of charcoal.

VI. CLOSED BOMB EVALUATION

Closed-bomb techniques and subsequent data reduction have been a concern of several laboratories. To insure equivalent data processing, laboratories exchanged propellant samples and results are given in a JANNAF Combustion

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[27] *N. Kubota, T.J. Ohlemiller, L.H. Caveny, and M. Summerfield, "The Mechanism of Super-Rate Burning of Catalyzed Double Base Propellants," Report No. AMS 1087, Dept. of Aerospace and Mechanical Sciences, Princeton University, Princeton, NJ, March 1973.*

[28] *A.F. Belyaev and S.F. Maznev, "Dependence of Burning Rate of Smoke-Forming Powder on Pressure," Dokl. Akad. Nauk SSSR, Vol. 1, p. 887, 1960.*

[29] *A.P. Glaskova and I.A. Tereshkin, "Relation Between Pressure and Burning Velocity of Explosives," Zhur. Fiz. Khim., Vol. 35, pp. 1622-1628, 1961.*

[30] *A.F. Belyaev, A.I. Korotkov, A.K. Parferfenov, and A.A. Sulimov, "The Burning Rate of Some Explosive Substances and Mixtures at Very High Pressures," Zhur. Fiz. Khim., Vol 37, p. 150, 1963.*

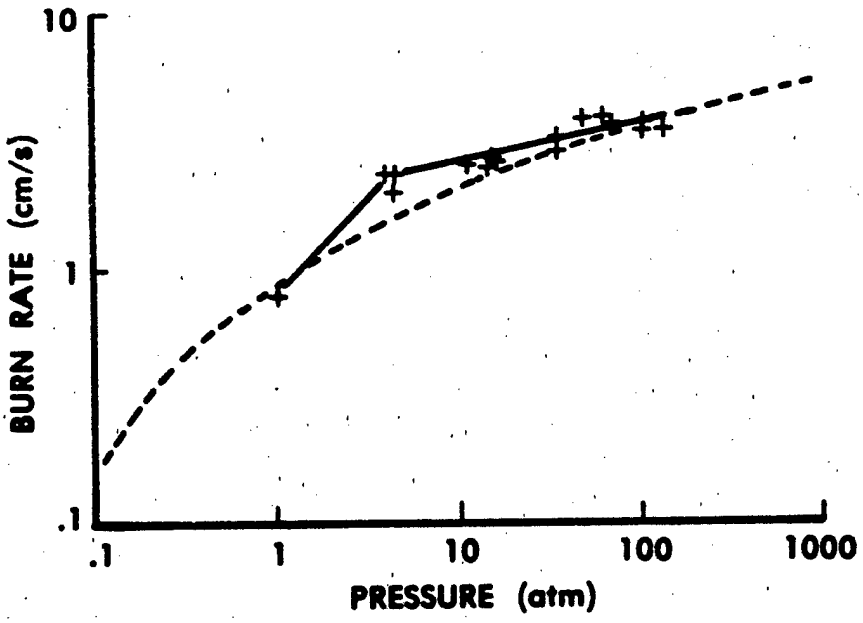


Figure 5. Strand Burn Rates at High Pressures; Dashed Line References 14, 28, 29, and 30.

Report.[31] Although black powder was not considered, this document does reflect closed-bomb evaluation methods and the description is a valuable reference tool. The calculated maximum pressure, flame temperature and other quantities have been predicted by various thermodynamic computer codes that require the chemical composition of the propellant. In the case of black powder, the composition of charcoal, as well as the other including ash ingredients, must be specified. Eli Freedman[21] performed such calculations for the Indiana, GOEX, and DuPont samples. For the first time, charcoal was represented by its elemental composition as opposed to only carbon as had been the custom. For the GOEX and DuPont samples, Freedman used the elemental composition of charcoal given by Rose[15] as "Roseville B" made by the Roseville Charcoal Co. of Zanesville, OH, and obtained the potassium nitrate/sulfur/charcoal concentrations from the data sheets supplied by Indiana. In a like manner, the chemical composition of the charcoal used at Indiana (also made by Roseville) was taken by averaging the values reported by Sasse'[20] and summarized in Table 1 of this chapter. Again, potassium nitrate/sulfur/ charcoal concentrations were obtained from Indiana data sheets. The computed results were compared[22] to closed bomb data where an 88 cm^3 bomb, at a loading density of 0.14 g/cm^3, was employed to produce peak pressures of 472 atmospheres. For one example, a flame temperature of 1765 C and an impetus of 95583 (FT-lb/lb) was calculated. To calculate burn-rate equations, thermodynamic quantities were used, and the grains were considered to be perfect spheres with a diameter chosen at the midpoint of the size range of the original screening, and a value of 0.14 inch, or 0.55 mm, was assumed. Closed bomb burning-rate equations were derived for four samples each of GOEX, DuPont, and Indiana black powders and they are given in Table 3. Equations were fitted to the data between 136 and 408 atm.

One major concern of this and other closed-bomb black powder experiments is the large value of the burning-rate exponent, 0.5 to 0.6, in the burning-rate equation, as contrasted to the supposedly equivalent value derived from strand burn-rate experiments where a smaller value of 0.164, Eq. 1, has been reported. Such differences have been previously observed in other work and they have been discussed, first by Rose[15] and later by White and Sasse',[32] The different values in exponents has to reflect a different burning mode in the closed bomb than is normally assumed, and hence, these burning rates should be considered as pseudo-burning rates.

In the strand-burning experiments, room temperature gas pre-pressurized the chamber and sample as contrasted to the closed bomb where hot combustion

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[31] *JANNAF Combustion Subcommittee, Burn Rate Measurements and Data Reduction Procedures Panel, "Round Robin Results of the Closed Bomb and Strand Burner," CPIA Publication No. 361, edited by A. Juhasz, July 1982.*

[32] *(a) K. White and R.A. Sasse, "Combustion and Flame Characteristics of Black Powder," Proc. of the 18th JANNAF Combustion Meeting, CPIA Publication No. 347, Vol. II, p. 253, October 1981.*

(b) K. White and R.A. Sasse, "Relationship of Combustion and Physical Properties of Black Powder," ARBRL-MR-03219, Ballistic Research Laboratory, USA-ARRADCOM, Aberdeen Proving Ground , MD, November 1982. (AD A122 264)

TABLE 3. COEFFICIENTS FOR THE BURN-RATE EQUATION AND QUICKNESS VALUES.

Sample Type	b cm/s	n	Peak Pressure atm	Average Quickness atm/s
COEX	0.253	0.671	454	44.48
	0.551	0.531	455	44.81
	0.287	0.660	454	44.65
	0.326	0.641	453	45.63
DuPont	0.561	0.493	469	38.41
	0.518	0.485	466	34.03
	0.457	0.503	462	32.05
	0.251	0.547	465	32.19
Indiana	0.179	0.661	459	29.14
	0.309	0.553	459	28.34
	0.328	0.542	463	28.27
	0.345	0.533	467	29.09

Burning rate, r(cm/s) is given by the function $r=bP^n$ where pressure, P, is in atmospheres.

gases had this effect. It was thought that this different temperature and pressure history might influence combustion to a degree that would change burning-rate exponents. Unpublished data of a single inhibited black powder cylinder, burned in the same closed bomb at pressures to 110 atm, had an exponent of 0.192, a value near 0.164 derived by cinematography from strand-burner experiments. From this relationship it was concluded that the difference in gas temperature and pressure history does not induce a different combustion mode and furthermore, deconsolidation or porous burning does not take place. Similar confirmation was obtained in high pressure steady-state rocket motor experiments. Therefore, the large burning-rate exponent must be an artifact of burning a collection of grains of black powder.

To explain this discrepancy, one must invoke mechanisms whereby surface area increases during combustion and two substantive suggestions have been offered. One is that grain break-up or fracture is the root cause for increasing surface area, and a second hypothesis is all black powder grains do not ignite at the instant of ignition. Either explanation could account for a high value of the exponent in the burning-rate equation. Another approach to this problem is to consider that the graphite coat acts as an inhibitor and combustion progresses from a single point ignition source that results in increasing surface area during the burn. High-speed movies of single burning grains suggest this effect. If this idea is mechanistically correct, then green grains (where surface burn-rates are greater by a factor of 5 than bulk values) should burn with smaller burn rate-exponents than graphited material. Closed-bomb experiments exerting a maximum pressure of 100 atm followed this pattern and the approach seemed promising; however, in the present work, and at pressures to 500 atm, green and graphited grains gave the same combustion curves. The contradiction of the two sets of experiments is unresolved and

the concept of single-point ignition is not supported even though this process may be operative. Work in this area should continue for this contradiction is the behavior of black powder.

In dealing with combustion of black powder, it is recognized that combustion rates are proportional to grain size and the size distribution should be known. These functions were measured for all samples and they are given in Table 2. Each sample had a slightly different distribution and in all cases the function was not sharp. Under these conditions one worries that the numerous small or large grains dominate the calculation and invalidate the assumption of "average radius." Since an average and particular radius was chosen for data evaluation, it seemed worthwhile to perform a mathematical sensitivity analysis using different radii. This was accomplished using one set of GOEX data and the various radii of the sieve sizes, four through eight, embracing the sub-sizes of class-one black powder. The burn-rate curves had similar exponents but were displaced one from another where the pre-exponent changed by a factor of 2.5. Clearly, the distribution function should be folded into the calculation, but the main point is grain size distribution relates to the pre-exponent and not to the burning-rate exponent.

VII. QUICKNESS VALUES

The derivative of the closed bomb pressure-time curve was formed as a function of the percent of maximum pressure achieved. Each point on the curve is a quickness value related to a particular corresponding pressure. Four such points at 25.0, 37.5, 50.0, and 62.5% of maximum pressure are customarily selected to represent quickness and the average of these four points is termed, by the propellant community, as average quickness.[31] Such values are given in Table 3. Since these values are extracted from the closed bomb data, comments concerning burning mode in that device also apply to the interpretation of quickness. In presentation of relative quickness values, test samples have been normalized to a reference standard run under exactly the same conditions. The quotient between these values renders a relative gasification rate that to some degree integrates and compensates for different burning modes, whatever they may be.

VIII. FLAMESPREAD RATES

Open air flamespread rates were obtained by measuring the time required to burn 16 g of class one black powder strung in a straight line 46 cm long. A recording TV camera was used to measure burn times. Examples are given by White, Holmes, and Kelso.[33] The technique was improved by placing the grains on a plastic strip and using a mirror to photograph the underside of the silhouetted burning string. This placed the black powder between the camera and the light. Flamespread rates of 63 cm/s were measured for DuPont, 74 cm/s for GOEX and 63 cm/s for Indiana samples.

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[33] *K. White, H.E. Holmes, and J.R. Kelso, "Effect of Black Powder Combustion on High and Low Pressure Igniter Systems," Proc. of the 16th Combustion Meeting, CPIA Publication No. 308, Vol. I, p. 477, September 1979.*

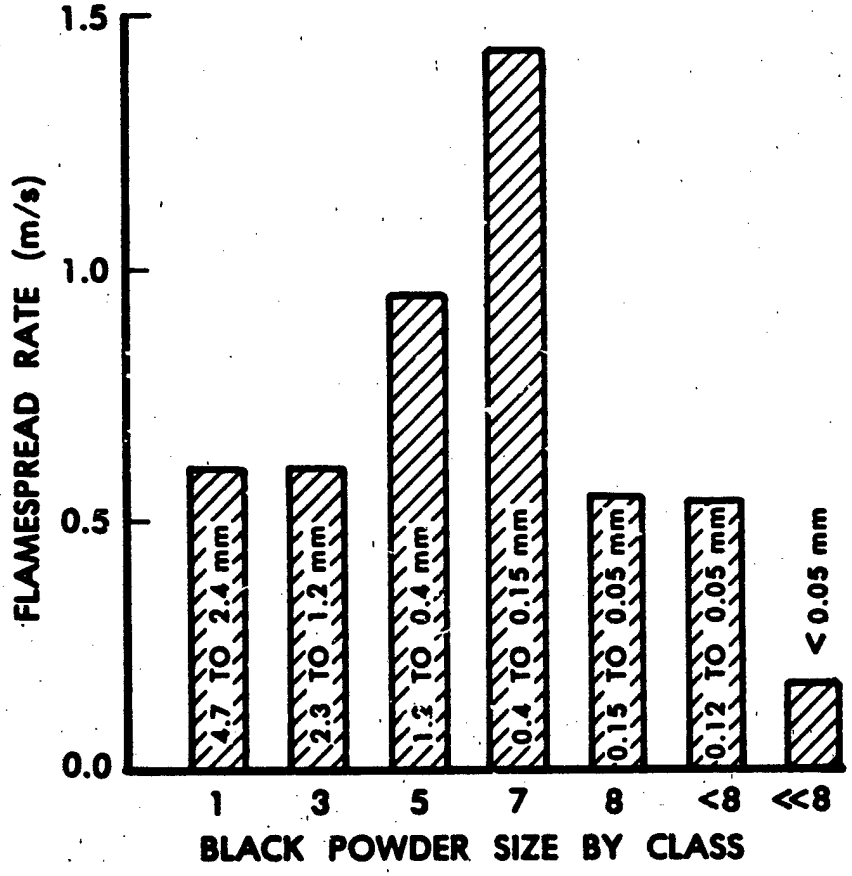


Figure 6. One Atmosphere Flamespread Rates for Various Classes of Black Powder

Kevin White[32a] extended these measurements to different classes of black powder and his results are shown in Figure 6. The very small grains had the slowest rates, for the burning grains emitted gas jets that blew light material out of the straight propellant line and retarded flame propagation. The fast flamespread rate noted was suggested to be the result of individual grains oscillating, causing the gas-particle jets to interact to a greater degree with adjacent surfaces. This concept was confirmed by gluing grains to a plastic sheet and then the flamespread rate dropped to 20 cm/s from the free value of 60 cm/s. When two sheets were placed in opposition, such that the gas-particle jets from one sheet sprayed on the other, then the flamespread rate increased to its free value of 60 cm/s.

A great deal of effort has been devoted to develop a flamespread "tester" where most of the work was performed by the Princeton Combustion Research Laboratories of NJ[34] and White, et al.[33] contributed to this subject. These results have been extended and summarized in a recent report.[22] The overall objective was to devise a relatively quick and functional test that could be exercised during the production of black powder such that results would be available before material was packaged. It was envisioned that this test would enable one to determine, in a timely manner, if a production cycle produced a ballistically acceptable product or would indicate if process or raw material changes were required. Most experiments measured black powders flamespread rate in a 19 cm tube with open slits and used soft igniters that first vented into a small plenum expansion chamber. This is the functional design of the Princeton Research Laboratory "Flamespread Tester." Other applications have used this tube as received with the holes plugged with a "wax-like" substance, and measurements have been made with an electric match or a brisant configuration using the M61 primer as found in the M28B2 ignition system of the 105 mm Howitzer. Since these experiments are very specific in nature, and strongly depend on the particular geometry and venting conditions employed, they will not be described here but are in Reference 22. In general, it was found that soft ignition using vented tubes resulted in pressure pulses of 34 atm, uniform combustion, and flamespread rates of 2000-3000 cm/s whereas plugged tubes and soft ignition gave pressures to 700 atm and rates to 10000 cm/s. In this latter case, non-uniform combustion was indicated by the rear pressure gauge, which, at times, recorded pressure pulses twice as large as normally recorded. Harsh ignition and plugged tubes resulted in fracturing the grains that led in some cases to plugged flow and slow propagation rates, 5000 cm/s, non-uniform combustion, and thermal excursions. It would appear that using soft igniters and either open air or semi-confined conditions result in reliable flame propagation rates.

IX. STRUCTURAL STRENGTH OF BLACK POWDER

In semi-confined plugged tubes and closed-bomb experiments, occurrence of grain fracture could, in itself, explain both extraordinary pressure pulses and enhanced gas generation rates. Such fracturing will be dependent on the

[34] *N.A. Messina, L.S. Ingram, M. Summerfield, and J.C. Allen, "Flamespread Propagation Rates of Various Black Powders Using the PCRL-Flamespread Tester," Seventh International Pyrotechnics Seminar, Vol. I, ITT Research Institute, Chicago, IL, p. 388, July 1980.*

intrinsic strength of the propellant. To assess this parameter, several 1.0 cm long black powder cylinders having diameters of 1.3 cm were prepared each of a different density. They were slowly compressed on a Materials Testing Machine until they shattered and that pressure is given as a function of density in Figure 7. Additionally, dynamic effects were obtained with a similar series of samples using a modified Drop Weight Testor where samples were mounted on top of a piezo-electric force gauge. A 2.0 kg weight was dropped on the cylinders from a 20 cm height and force was recorded as a function of time; displacement was also measured by an Optron Electro-Optical Displacement Follower using the technique described by Lieb.[35] The dynamic crushing strengths are also given in Figure 7, and they are slightly smaller than quasi steady state values where both functions are steep functions of density. By extrapolating the data, it is inferred that black powder must be at least as dense as 1.3 to form a cohesive mass. From helium density measurements of carbon, density of 1.45, and density values for sulfur and potassium nitrate black powder can be no more dense than 1.97. The military specification requires the higher values, and it is suspected that the specification was originally cited to achieve a minimum strength for black powder. From these dynamic experiments, stress-strain curves were derived that were nearly linear, indicating that black powder crushes by a brittle fracture mode, much like glass.

X. COMBUSTION TEMPERATURE

Although combustion rates and mode have been discussed, no comment has been directed to the heat released by various exothermic reactions. The temperature of the gas stream was measured by the sodium line reversal technique by Harris,[36] and he reported a value of 1549° ± 25 C for burning black powder in air at one atmosphere. From arc image experiments, Lanchits[37] found an ignition temperature of 469 ± 50 C for a DuPont sample which agrees well with Kirshenbaum's[38] value of 420 C obtained by differential thermal analysis. Another, but simpler, experiment was attempted, where a thin 0.127 mm diameter chromel-alumel thermocouple was placed in the center of meal just before being pressed into a stick. Even within the general limitations of thermocouple measurements, the burning of this composite should give some lower bound estimate for heat propagation within the stick. During combustion the indicated temperature rose to 480 C in 22 ms before the bead entered the gas stream and melted. Assuming the bead was twice the thickness of the wire, and a burning rate of 1.0 cm/s, heat penetrated about 220 microns into the

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[35] *R.J. Lieb and J.J. Rocchio, "Standardization of a Drop Weight Mechanical Properties Testor for Gun Propellants," ARBRL-TR-02516, Ballistic Research Laboratory, USA-ARRADCOM, Aberdeen Proving Ground, MD, July 1983, (AD A132-966).*

[36] *L.E. Harris, J.A. Lannon, R. Field, and D. Husted, J. of Ballistics, Vol. 1, p 353, 1977.*

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[38] *A.D. Kirshenbaum, J. of Ballistics, Vol. 1, p. 171, July 1978.*

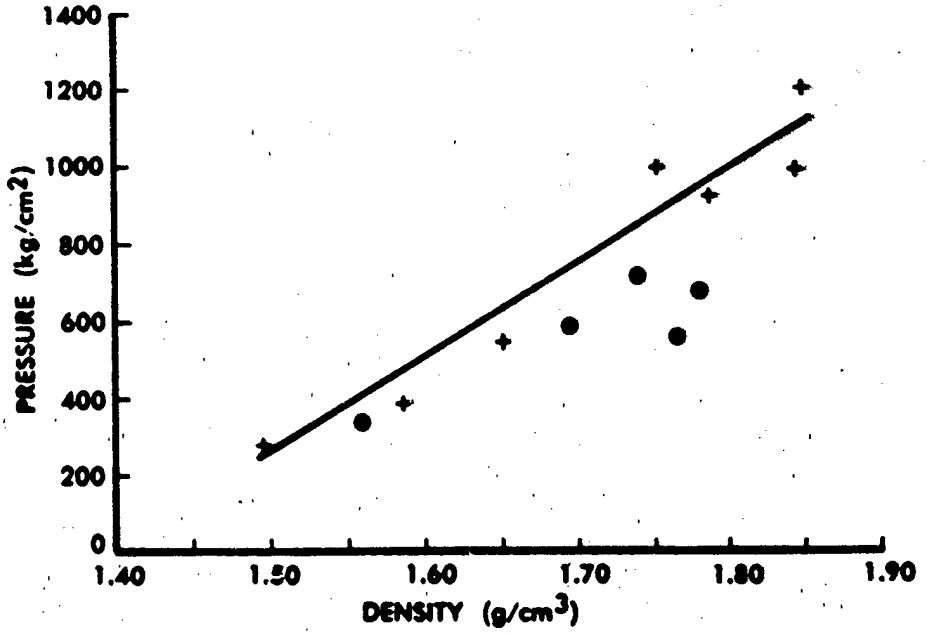


Figure 7. Strength of Black Powder; +, Static Measurements, and ●, Dynamic Measurements

grain. Clearly, the thermocouple was too thick, and the measurement was not corrected for heat losses, but one can conclude that the heated black powder shell is very thin.

XI. GENERAL COMMENTS

The several functional and physical tests, each of which relates to a particular combustion mode, have been presented. The relationships and properties discussed in this chapter each characterize black powder; however, complete characterization will only become finalized when this work is coupled to gun performance. This latter program is now in progress and is sponsored by an Engineering Study (ESP 1A-3-8428) funded by the office of DRSMC-LE(R). Firings of DuPont, GOEX, and Indiana black powders will be evaluated in relation to actual ballistic performance of igniting a propellant, and only from these results can we determine the adequacy or inadequacy of the black powder. It is hoped that from the merging of laboratory and performance data one or more of the laboratory tests can be identified to predict this quality.

XII. ACKNOWLEDGMENTS

To my friends Kevin White and Eli Freedman, I extend my gratitude for their personal and scientific support. I also dedicate this report to my daughter Chris.

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