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A Short History
of Gunpowder and
the role of Charcoal
in its manufacture.

Review

A short history of gunpowder and the role of charcoal in its manufacture

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Deflagrations caused by interactions of charcoal with potassium nitrate were discovered in 9th-century China. This led quickly to the development of primitive fuzes and ballistics. Roger Bacon, a 13th-century Franciscan monk experimented in England with gunpowder. The 18th-century saw the manufacture of charcoal by the cylinder method and the development of Waltham Abbey as the centre for gunpowder production which peaked to about 20 000 barrels per year at the time of the Battle of Waterloo. Methods of gunpowder manufacture changed little over the centuries until recent times. Whereas sulphur and nitre are reproducible ingredients, a mystery enshrined optimum specifications for gunpowder charcoal. Within recent years the application of modern analytical techniques has removed some of this mystery and anatomical features and physical properties have been characterized.

1. The discovery of gunpowder

Gunpowder is an explosive mixture of potassium nitrate (75%), charcoal (15%) and sulphur (10%). Early explosives (protogunpowder) contained similar ingredients but in varying proportions.

Protogunpowder was discovered in China in the 9th-century, when potassium nitrate was used as a flux in smelting metals [1]. For purification, the potassium nitrate was re-crystallized from boiling water. One alchemist accidentally dropped charcoal from the fire into the potassium nitrate and the mixture deflagrated, and others were warned not to mix potassium nitrate (nitre or saltpetre) and charcoal as the reaction "sing'd beards and burnt down buildings".

The discovery of this explosive mixture led to the rapid development of protogunpowder for use in warfare for fuzes, simple bombs and for grenades which were hurled by trebuchets, or large catapults. The first formula for protogunpowder appeared in China in 1044. At this time the mixture was used widely in flame throwers which were made from hollow bamboo or paper tubes, packed

with the explosive mixture and lit with a fuze (Fig. 1). Similar devices were used by the Chinese pirates up until the 19th-century.

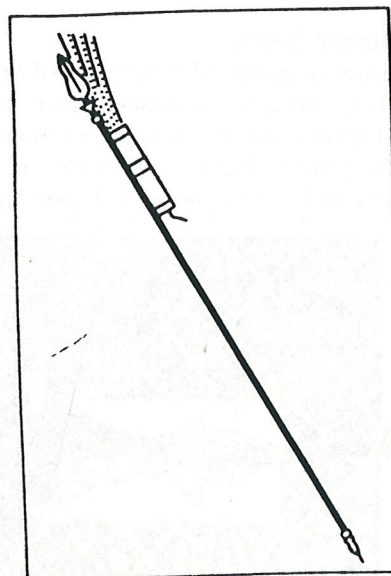


Figure 1 Diagram of an early Chinese flame thrower [1].



Figure 2 Roger Bacon and a pupil.

In 1230 the proportion of potassium nitrate in the mixture was increased and the propellant properties of the mixture were discovered. This led to the use of eruptors which were large diameter bamboo tubes packed with gunpowder and projectiles such as chain shot. Metal barrelled cannon were developed from these in 1290. In China it took three centuries from the discovery of gunpowder to develop from the simple flame thrower to the cannon.

2. Roger Bacon

The use of gunpowder then spread through Arab lands to Europe and through Spain and Germany to England where the name of Roger Bacon, a 13th-century Franciscan monk, is commonly linked with the discovery of gunpowder (Fig. 2).

Bacon, who was a learned scholar and carried out scientific work in many disciplines, did not discover gunpowder but probably learnt of its existence from an Arab trader. He experimented with gunpowder by varying the proportions of the constituents and noting the effect on the mixture's explosive properties. Bacon was also the first to make reference to the type of charcoal used, recommending the use of young hazel twigs to make charcoal for gunpowder [2].

Roger Bacon, although fascinated by gunpowder, realized the deadly implications should the formula become widely known so he wrote it in the form of a cipher. This cipher was written in 1267 but was not published then because of opposition from his Abbot.

In 1618 the gunpowder formula was published

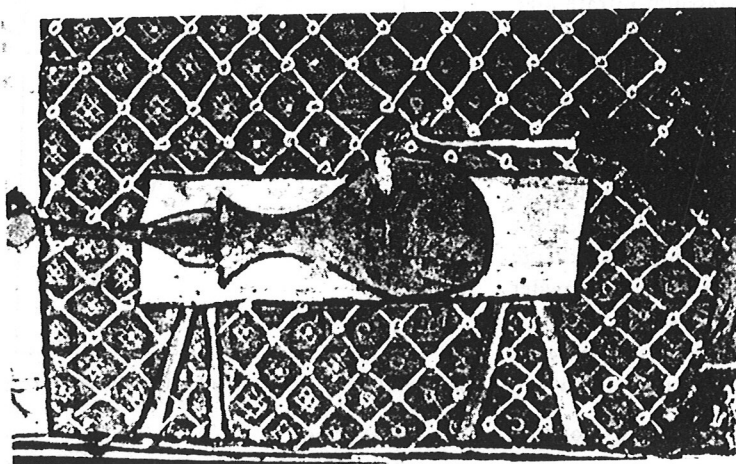


Figure 3 The Milemete gun [4].



Figure 4 Devils teaching monks how to make gunpowder (16th century).

but it was not until 1904 that it was deciphered by Hime by which time the gunpowder formula was widely known [3]. The formula given in the cipher was 7 parts saltpetre, 5 parts of hazel charcoal and 5 parts sulphur. Bacon discovered that powder with high nitrate concentrations was more explosive than mixtures used previously.

3. Gunpowder in Europe

By 1326 the discovery of the cannon had spread to Europe. The first illustration of a cannon is shown in a manuscript by Walter de Milemete in 1326 [4]. The cannon, known as the Milemete gun and shown in Fig. 3, was vase shaped and loaded with gunpowder and an arrow-shaped projectile.



Figure 5 Illustration of charcoal manufacture in the 18th century [7].



Figure 6 Illustration of charcoal manufacture in the 18th century [7].

The gunpowder used was the high nitrate variety and the propellant properties of the mixture were exploited. This weapon must have been inaccurate but probably proved effective at close range.

The arrival of the cannon led to the development of rifled barrelled guns. Gunpowder was now needed to be an even burning mixture and so more attention was given to the composition. Methods of purifying potassium nitrate and sulphur were well known, charcoal being the only variable component.

The first record of gunpowder being used by English troops was in 1346 at the Battle of Crecy.

Legend recounts that the gunpowder may have been manufactured by the monks at Waltham Abbey in Essex. (Waltham Abbey was destined to become the "home" of gunpowder in England.) Gunpowder manufacture was regarded at that time as one of the black arts and people believed that devils taught the monks how to make gunpowder (Fig. 4).

By the 16th century gunpowder was widely used in warfare but there were complaints about the price and the quality of powder obtainable in England. It was suggested to the Government that they build powder mills to meet demands for gunpowder in England, [5].



Figure 7 Photograph of Essex charcoal burner and his hut [8].

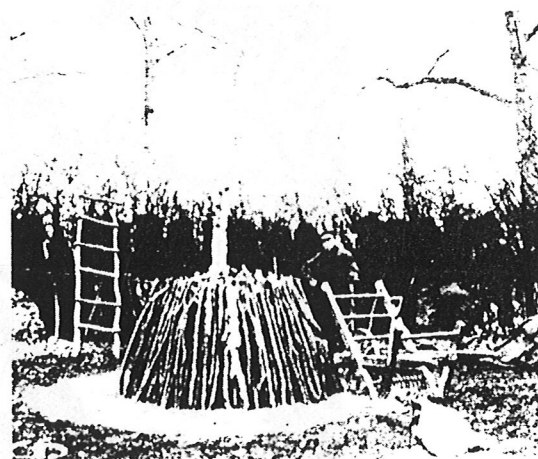


Figure 8 Photograph showing wood stacked around hearth prior to burning [8].

4. Gunpowder manufacture

In 17th century England methods of charcoal manufacture were investigated and Sir James Hope in 1646 described the process [6]. There was no mention of the type of wood used but small even pieces of wood were stacked, covered with turf and then charred. This method, illustrated in Figs. 5 and 6, was still in use in the early 20th century [7]. In Figs. 7 and 8, a charcoal burner in 1907 is shown. By comparing Figs 5 and 8 it can be seen that similar methods of charcoal manufacture were used for over three centuries in England [8].

In 1756 there were many recipes available for gunpowder. The proportions of the three ingredients varied slightly according to the type of gun with which they were to be used. All were high nitrate powders with approximately 75% potassium nitrate.

In 1759, under pressure, the Government eventually purchased powder mills which were previously established mills situated in Faversham, Kent. Powder produced here by the Government was said to be more costly and not as powerful as commercially available powders so the mills were under threat of closure. In 1783 Major William Congreve made successful representations to support the argument that they could be run as a viable proposition. Under Congreve's control the mills worked efficiently. He investigated methods of ensuring that the performance of gunpowder was reproducible. The main problem again was

with the charcoal, that is, from which woods and how it was produced.

In 1786 Bishop Watson was asked by the Government to suggest a more reproducible way of charring wood. Watson thought that making charcoal by distilling wood in cylindrical closed vessels would produce a better charcoal [9]. This method was viewed with scepticism by those who believed charcoal should be made by the method it had been made for centuries. The new innovation of using closed cylindrical vessels was tried and Congreve manufactured gunpowder from Bishop Watson's charcoal. Gunpowder made from the "cylinder" charcoal was found to be more powerful than previous gunpowder. Even though the tests proved that cylinder charcoal produced a superior gunpowder, this method of charcoal production was not introduced until 1794.

Congreve improved the efficiency of the mills at Faversham and the quality of the gunpowder produced. In 1787 the Government purchased the mills at Waltham Abbey which also came under Congreve's control. An early illustration of the water-powered mills is shown in Fig. 9. In 1788 the formula for gunpowder manufactured at Waltham Abbey was 75% potassium nitrate, 15% charcoal and 10% sulphur. These are the proportions which are used at the present time. Between 1789 and 1810 Congreve's mills produced 407 408 barrels of gunpowder, each weighing 100 lb, at a saving to the Government of £288 357 6s 0½d [5].



Figure 9 Illustration of Waltham Abbey Powder Mills as owned by John Walton 1735.



Figure 10 Photograph of Waltham Abbey in 1890 showing canals and small boats for gunpowder transport on the internal waterways.

In 1805 Congreve's son, also a William Congreve, developed the Congreve rocket used widely in warfare by naval and land forces.

In 1810 experiments were carried out to improve the uniformity of quality of batches of gunpowder. In 1812 Congreve was made a baronet after proving that Waltham Abbey gunpowder was not only cheaper but was more powerful than commercial powders.

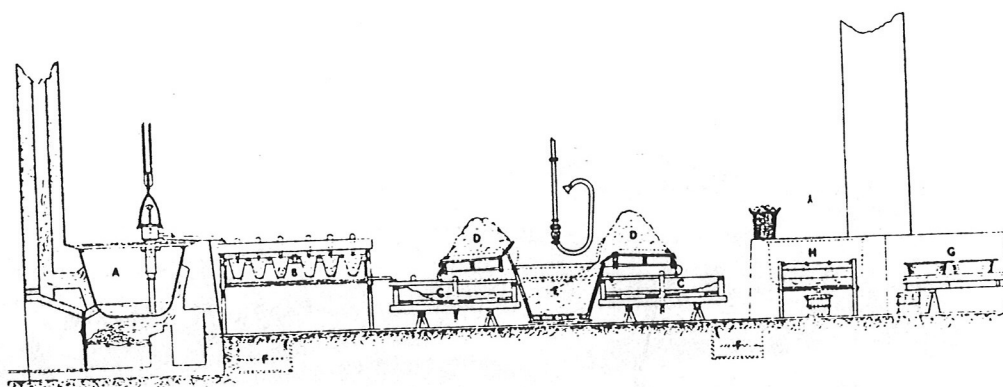
This was the time of the Napoleonic wars. In 1810 Waltham Abbey alone produced 20 000 barrels of gunpowder a year but after the Battle of Waterloo this was reduced to 4000 barrels in 1815, 1000 barrels in 1816 and less in the following years. As demand fell the Waltham Abbey mills were retained by the Government but all others were sold back to private owners. A view of the Waltham Abbey Powder Mills is shown in Fig. 10.

5. Methods of gunpowder production

By 1822 gunpowder production processes had been perfected and these and similar processes were used up to the present century. Throughout the 19th century many handbooks were published dealing with the manufacture and proof of gunpowder. One of the earliest was written by Frederick Drayson in 1830 [10]. Drayson gives a detailed account of the preparation of the three ingredients. The potassium nitrate, imported from India, was already partially refined and was known as "grough saltpetre". It was purified by boiling 2 tons saltpetre in 270 gallons water. The solution was boiled, cleared of scum, and the salt recrystallized. The material was stirred whilst crystallizing so that the saltpetre produced was in the form of fine flour. There was a 20% loss in this process due to impurities (Figs. 11 and 12).

The wood was charred at Faversham by the cylinder method recommended by Bishop Watson [9]. Before charring, the wood was stripped of bark and knots and cut to a length of 3 ft 4 in. About 580 lb wood was put into each cylinder, three cylinders being filled at a time. These were made of cast iron and are illustrated in Figs. 13 and 14. The cylinders were placed in the furnace and heated for 8 hours after which the furnace was shut down and the charcoal left overnight in the cylinders to cool before it was exposed to air.

One set of three cylinders was capable of making enough charcoal to produce 2500 barrels of gunpowder per annum. The building at Faversham contained four sets and was therefore capable of



A. Boiler. B. Filtering apparatus. C. Cooler. D. Drainer.
E. Washing Vat. F. Liquor Tank. G. Evaporating pans. H. Filter

Scale about $\frac{1}{2}$

Figure 11 Diagram of saltpetre refining apparatus at Waltham Abbey [14].

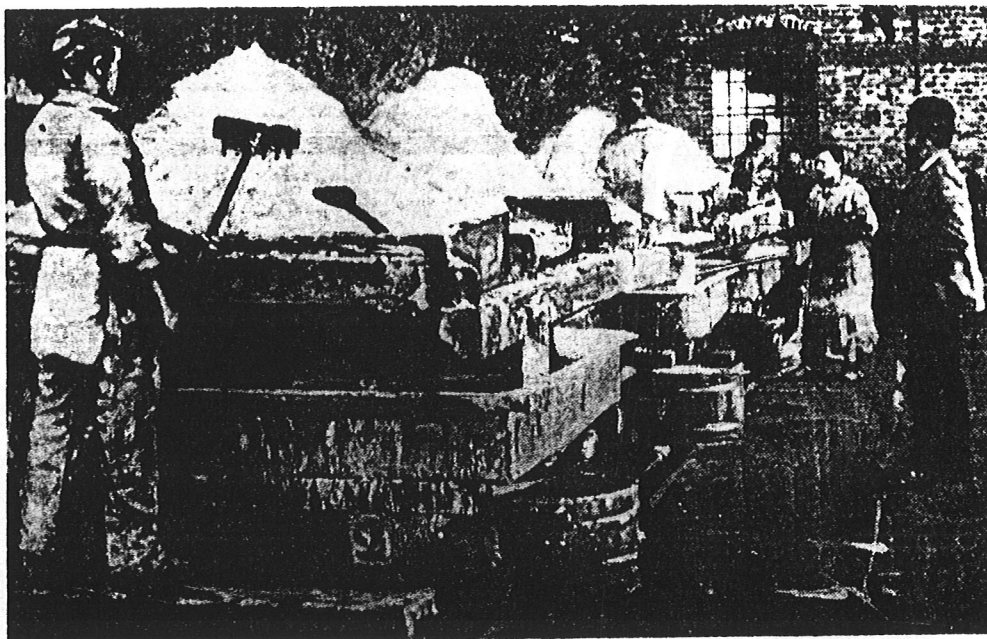


Figure 12 Photograph of the saltpetre refinery, Waltham Abbey [13].

producing enough charcoal for 10 000 barrels of gunpowder.

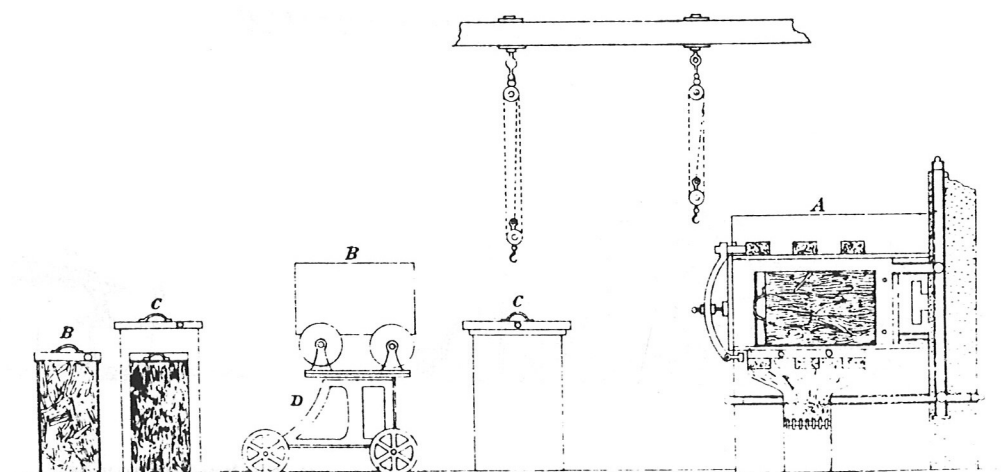
The wood used to make charcoal was the alder, willow or dogwood, much of which was grown on the plantations at Waltham Abbey when a cylinder house was built there at a later date.

Sulphur was refined by a process of distillation

by heating 6 cwt of grough sulphur in a cast iron pot in a furnace. The initial yellow fumes were led to the sublimation dome and the subsequent brown fumes to receiving pots. When partially cooled the sulphur was ladled by hand into wooden tubs (Figs. 15 and 16).

Baddeley in 1857 also describes the ingredients

APPARATUS FOR BURNING CHARCOAL.



A. Retort, showing pipes for conducting gases to the furnace
B. Slip for holding wood. C. Cooler. D. Carriage for Slip

Figure 13 Diagram of the cylinder method of charcoal production [12].

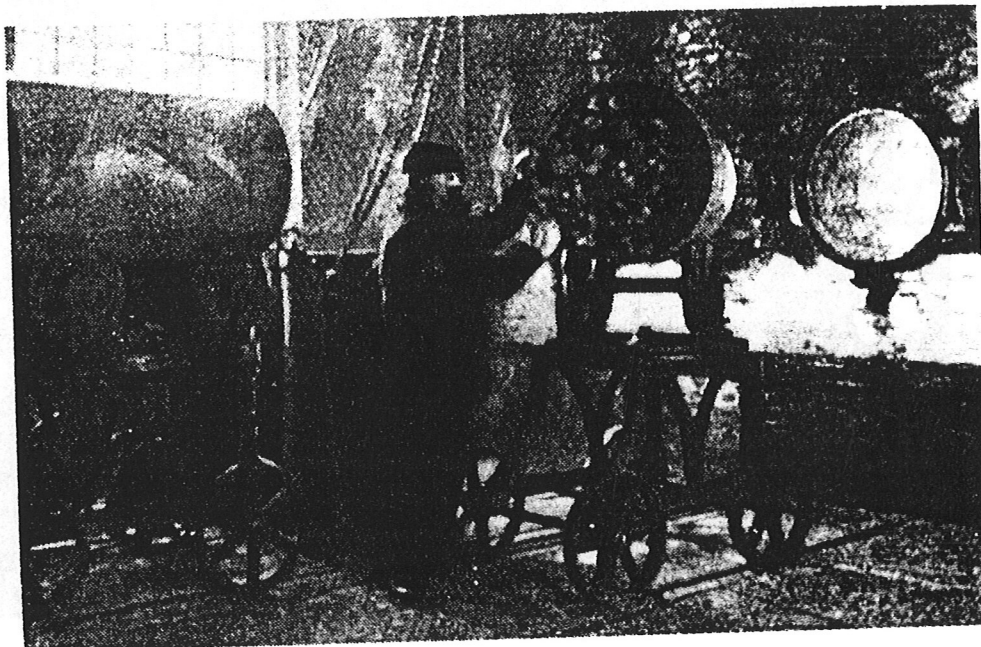


Figure 14 Photograph of charcoal manufacture [13].

of the gunpowder in detail [11]. He noted that alder, willow and black dogwood were used for good gunpowder charcoal. Wardell's handbook (1888) includes some excellent diagrams of the gunpowder production process [12].

One of the more general descriptions of gunpowder manufacture was published by Fitzgerald in 1895 [13]. The best description of gunpowder manufacture is in F. M. Smith's Handbook on gun-

powder published in 1870 [14]. This article described the preparation of all ingredients and the manufacture of gunpowder and other explosives. This process described in 1870 had already been used for 200 years and was in common use until the middle of this century.

The ingredients were prepared as described previously. All ingredients were ground individually and then mixed in the required proportions. The

SULPHUR REFINING APPARATUS.

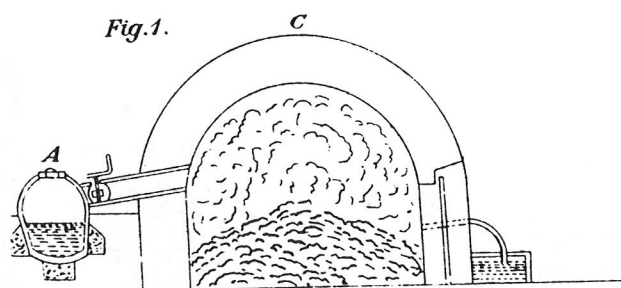


Fig. 1.

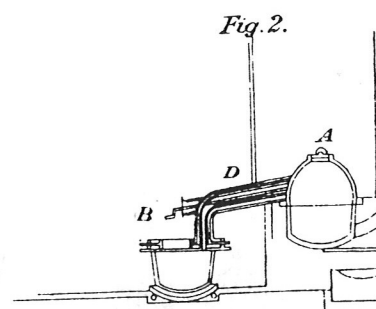


Fig. 2.

A. Melting Pot.
B. Receiving Pot.

C. Subliming Chamber.
D. Cold Water Jacket

The Subliming Chamber & Receiving Pot are placed at right angles to one another in the refinery.

Figure 15 Diagram showing the sulphur refining apparatus [12].



Figure 16 Photograph showing sulphur refining [13].

mixture was loaded in 60 lb sacks and taken to the incorporating mill. The incorporating mill was the first of the danger buildings as the mixture was highly inflammable at this stage. The steam-powered incorporating mills were built in sets of six, whereas the earlier water-powered mills were built in pairs (Figs. 17 and 18). As all walls except those dividing the mills were built of canvas on a light metal framework and the roofs were wooden these offered little or no resistance to explosions thereby reducing the damage to the machinery. Above the mills was a flash board which in the event of an explosion would be violently displaced and tanks of water would empty onto the bed of that mill and all the others nearby to reduce risk of fire.

The incorporating mills consisted of a circular stone or iron bed and two revolving wheels, or runners, of about 4 tons each. The mixture was put on

to the bed of the mill and wooden ploughs were fixed to the central shaft so that all the powder was kept under the runners (Figs. 19 and 20).

Owing to the danger involved in this process, all tools used were made from non-sparking materials. The mill operator, wearing protective heavy-leather clothing, would start the mill from outside (Fig. 21). After milling for 3 hours or more the mixture formed mill cake, which was removed from the mill and taken to the breaking-down house where it was broken down to meal powder by gun-metal rollers (Fig. 22).

The meal powder was then taken to the press house. As this was one of the most dangerous stages, the press house was separated from the men's retiring room by a massive stone, brick and earth wall (Fig. 23). In the press a number of copper plates were fixed in a rack and 750 lb of powder was placed between them. The plates were then

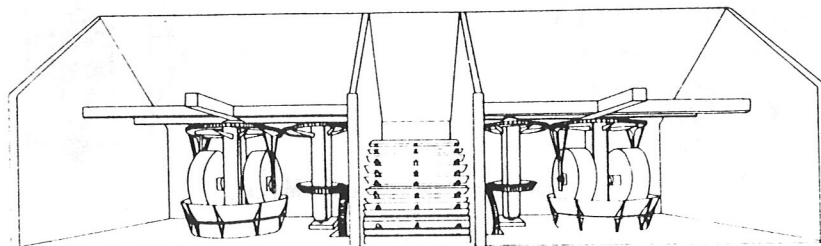


Figure 17 Diagram showing a pair of water-driven incorporating mills [10].

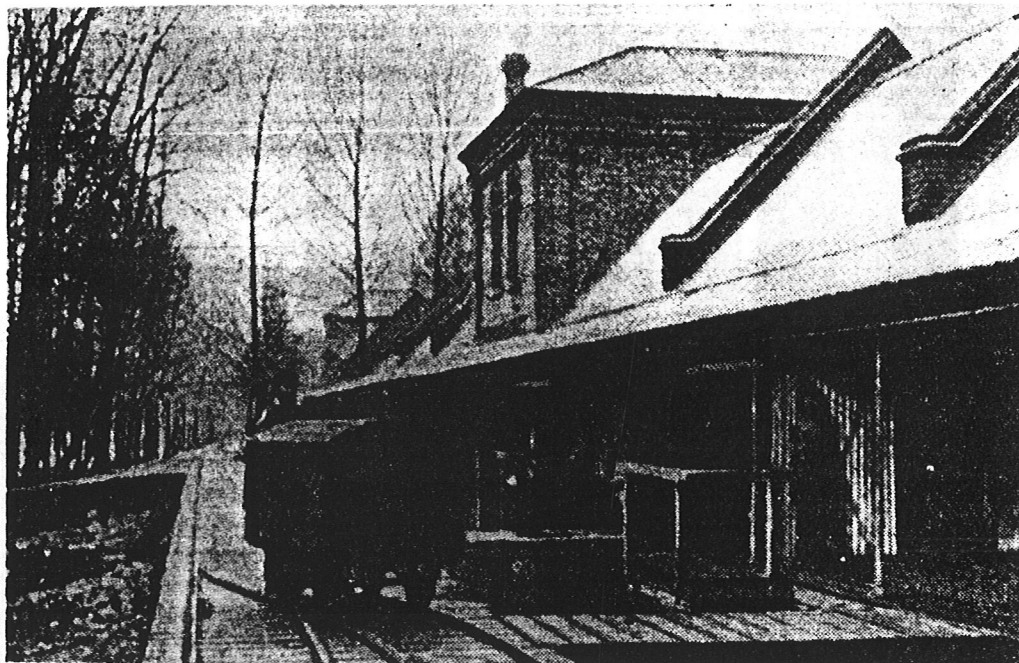


Figure 18 Photograph showing the exterior of the steam powered incorporating mills [13].

compressed at between 63 and 500 tons for $\frac{1}{2}$ hour (Fig. 24). After this period the press cake, as the gunpowder was then called, was removed. Deep wells were situated outside all the danger buildings so that badly burned men could leap into them.

The press cake was reduced to the required sized grains by a series of graduated toothed rollers

separated by sieving screens, this process being called "coming" (Figs. 25 and 26). Dust was removed from the powder by passing it through revolving cylindrical reels made of silk (Fig. 27). Some powders were glazed with graphite (Figs. 28 and 29).

After drying, the powder was then packed into

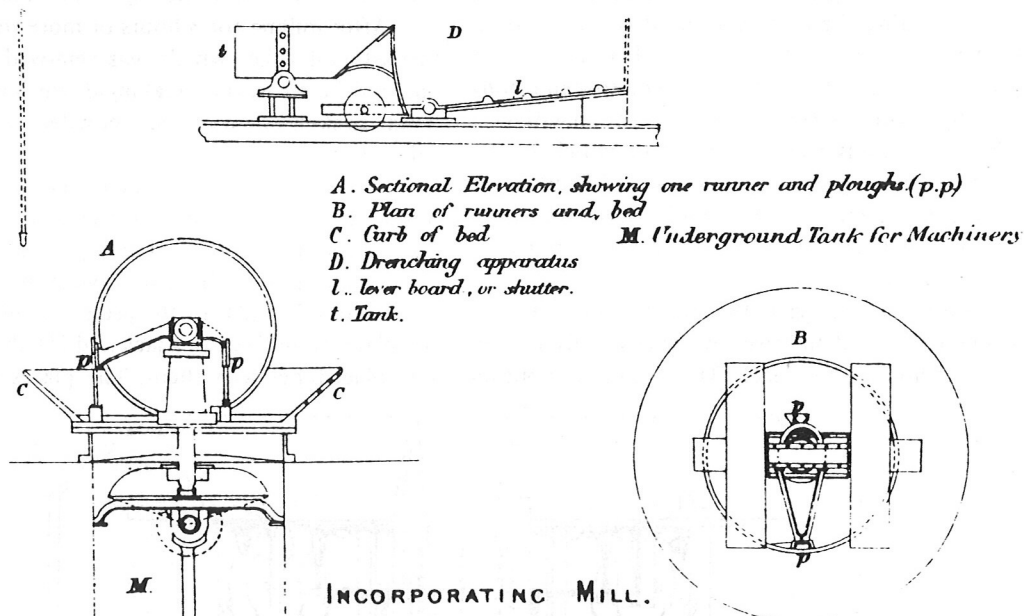


Figure 19 Diagram of an incorporating mill [12].

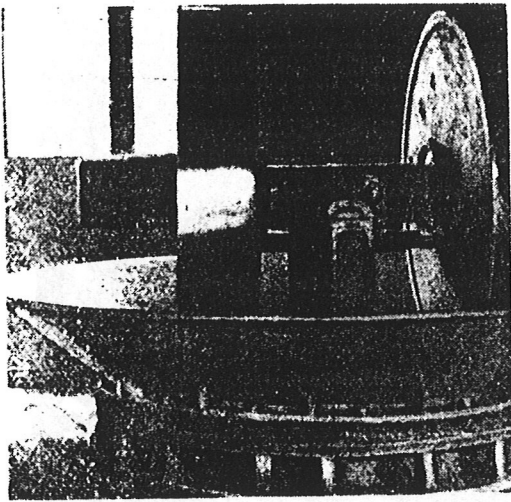


Figure 20 Photograph of the interior of an incorporating mill [13].

barrels and transported by means of powder barges (Fig. 30). Strict precautions were taken with the powder barges and no one was allowed to walk over a bridge while they passed underneath in case grit fell onto the decks.

This process of gunpowder manufacture was carried out at the Royal Gunpowder Factory, Waltham Abbey until the last of the powder mills was destroyed by enemy action in 1941.



Figure 21 Workers in protective clothing starting incorporating mills [13].

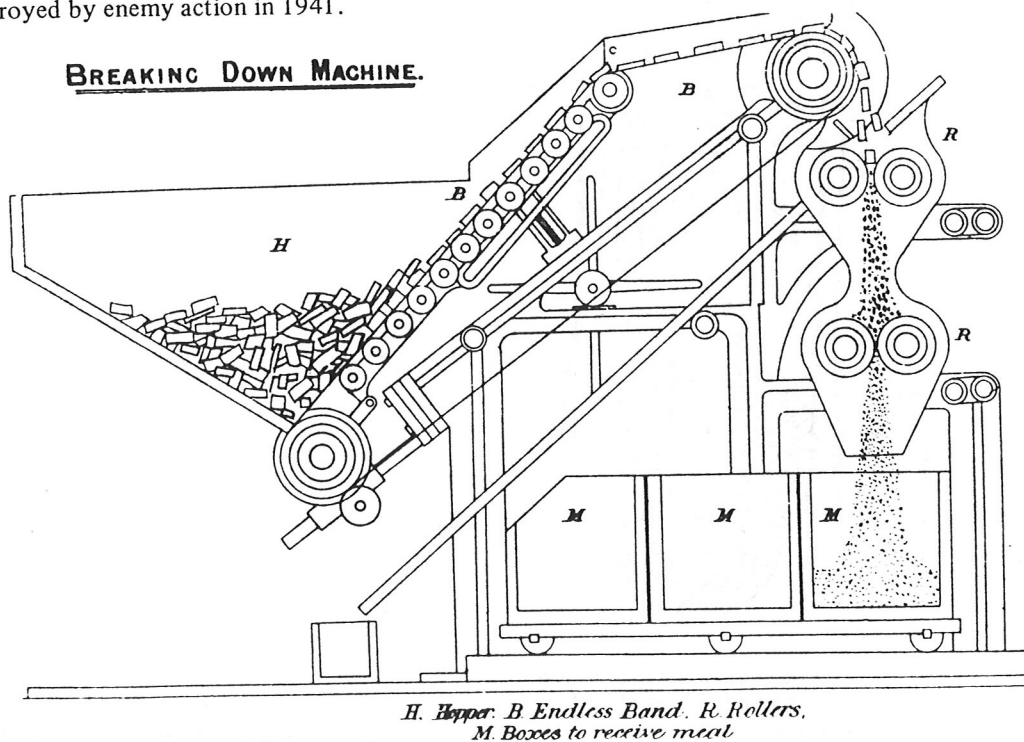


Figure 22 Diagram of machine for breaking down mill cake [12].

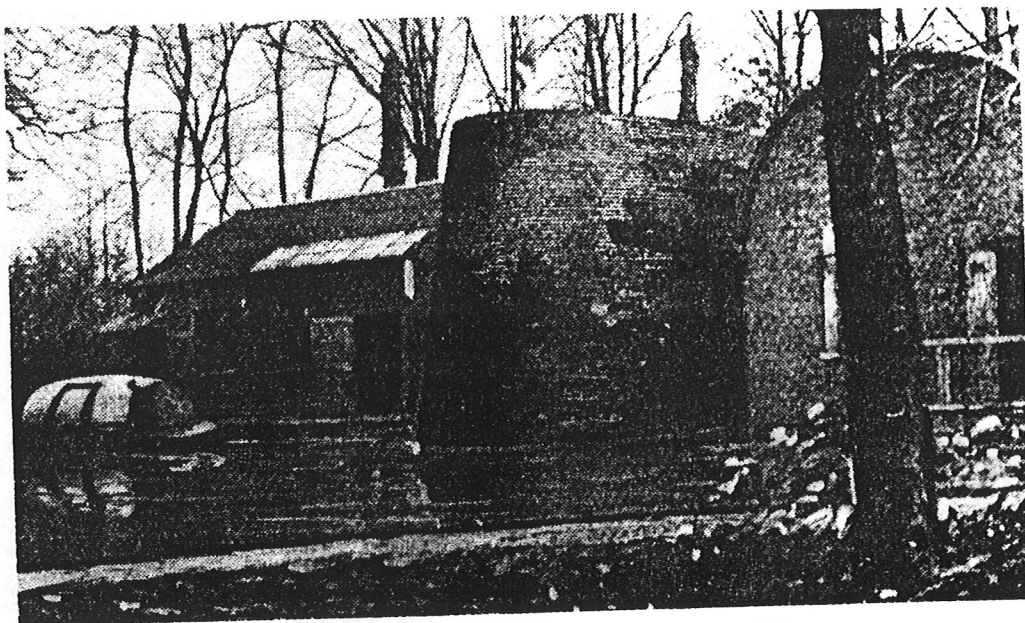
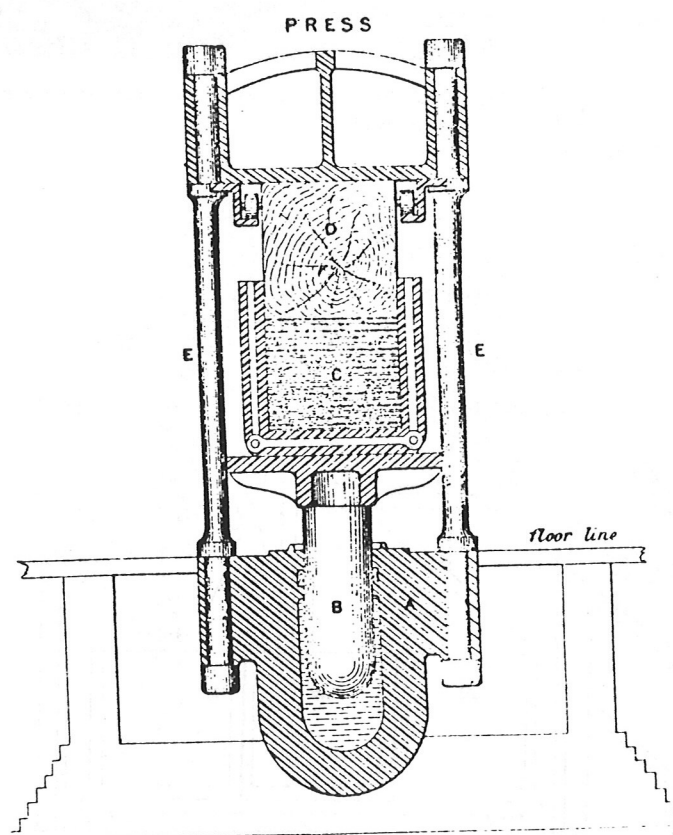


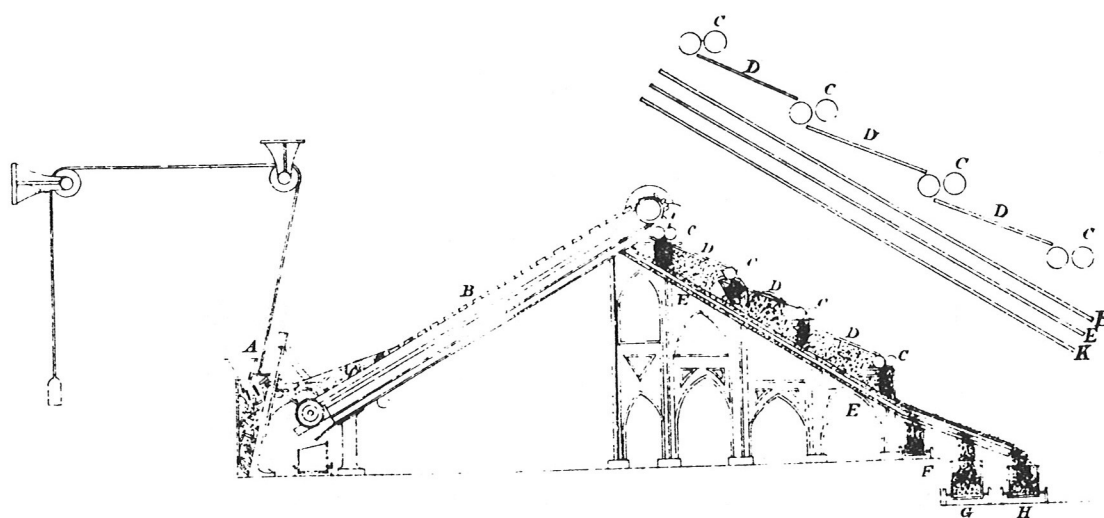
Figure 23 Photograph of the press house, protective barrier and mens retiring room [13].



ELEVATION AND SECTION
SHOWING PRESS IN ACTION
A Cylinder B Ram C Press bar
D Overhead block E E Standard

Scale $\frac{1}{2}$

Figure 24 Diagram of hydraulic press [14].



A. Hopper with raising arrangement.
 B. Endless band
 CCCC. Four pairs of rollers
 DDD. Short screens.

EE. Long Screens
 F. Box for dust
 G. Box for grain
 H. Box for chucks
 K. Bottom board

Figure 25 Diagram of a granulating or corning machine [12].

In the 19th century there were numerous references to charcoal in gunpowder. The common types of wood used were alder, willow and dogwood but many powders were made from other chars such as cocoa powder which was made from straw.

6. Properties of gunpowder

Blackwood and Bowden [15], investigated factors influencing the initiation, burning and thermal decomposition of gunpowder. They showed that ignition developed from the formation of local hot spots in the gunpowder. Hot spots were thought to

be formed by the adiabatic compression on impact of pockets of air between the grains or by crushing of grit particles. The addition of a high melting point grit of comparable size to the gunpowder grains increased the sensitivity to impact.

Gunpowder was found to be insensitive to friction even when grit was added. Gunpowder was also not ignited by a spark. With a pulsed laser gunpowder could be ignited but failed to burn. A hot wire was the only successful method of ignition which had the necessary thermal capacity to sustain the heat for long enough to cause the gunpowder to burn [15].

In their studies of burning, Blackwood and Bowden [15] showed the effects of pressure upon propagation rate. At atmospheric pressure (100 kPa) the burning front in 2 mm sized grains of gunpowder propagated at approximately 0.6 m sec^{-1} . At low pressures (20 kPa) the gunpowder could be ignited but failed to propagate. As the pressure was increased, burning rate and propagation increased rapidly, such that at 200 kPa the propagation rate was 7.6 m sec^{-1} .

Detailed examination of burning gunpowder showed that a liquid layer was formed on the surface of the gunpowder grains and that propagation was caused by fine sprays of hot molten salts impinging on neighbouring grains. In coarse-grain gunpowders the propagation rate exceeds the

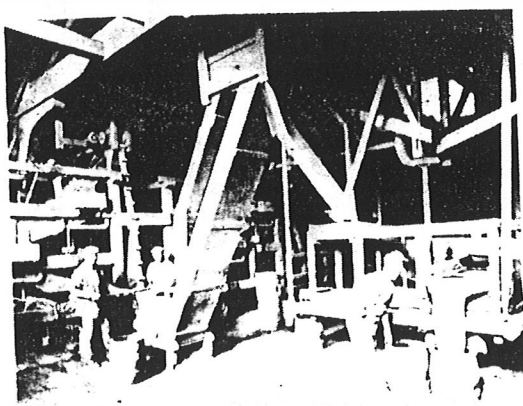
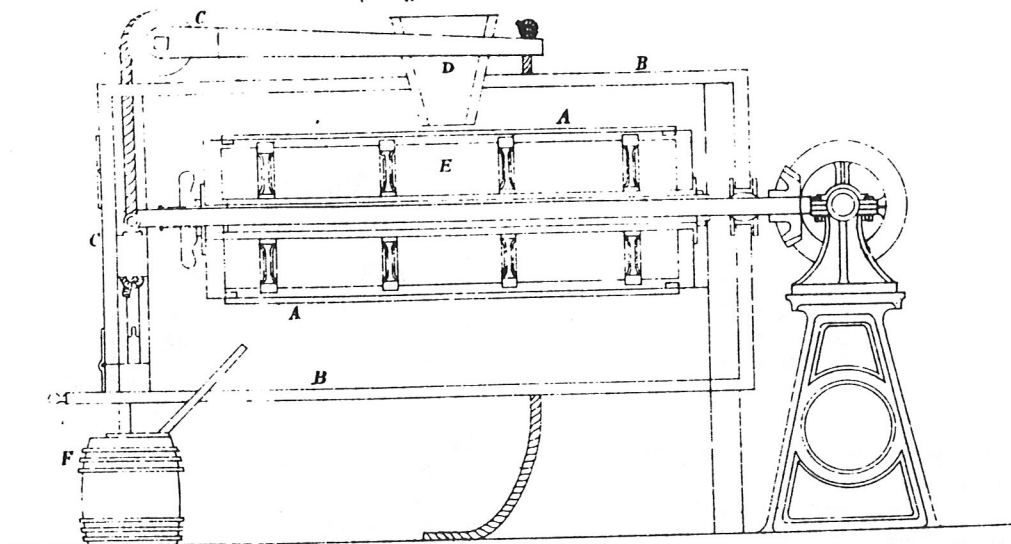


Figure 26 Photograph of the corning house at Waltham Abbey, 1890.

HORIZONTAL DUSTING & FINISHING REEL. (Longitudinal Section.)

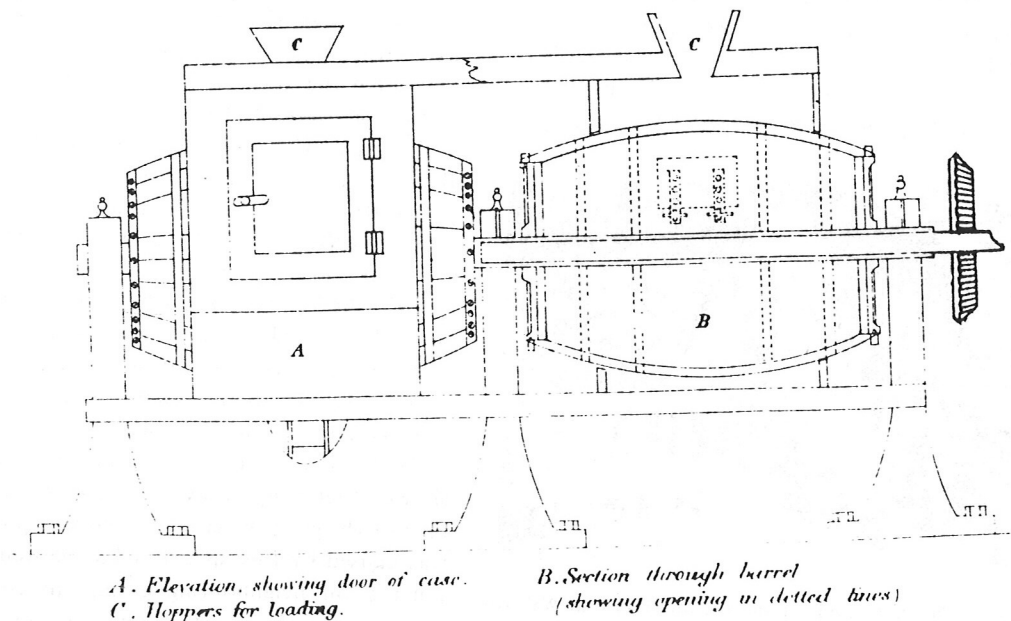


- | | |
|--|--------------------------------|
| A. Cylindrical reel | D. Hopper for loading |
| B. Reel Case | E. Opening in reel for loading |
| C. Apparatus for lowering one end for unloading. | F. Barrel for unloading into. |

Figure 27 Diagram of a horizontal dusting and finishing reel, 1870.

burning rate and grains ahead of the burning front are ignited by the molten salts. In fine-grain gunpowders compression of grains inhibits these sprays of molten salts and the propagation and burning rates are equal.

Blackwood and Bowden [15] investigated the role of charcoal in gunpowder. They mixed charcoal with sulphur and with potassium nitrate separately and did the same with graphite. The burning charcoal mixtures evolved sulphur di-



A. Elevation, showing door of case.
C. Hoppers for loading.

B. Section through barrel
(showing opening in dotted lines)

Figure 28 Diagram of glazing barrels [12].

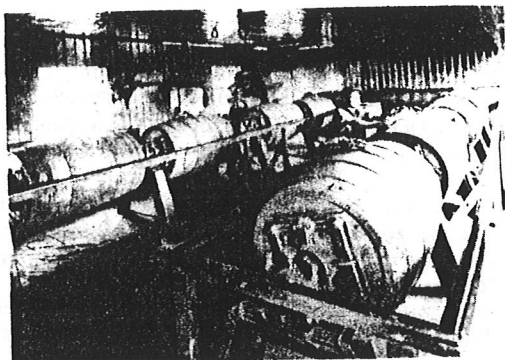


Figure 29 Photograph of glazing barrels, 1890.

oxide and nitric oxide and nitrogen dioxide, respectively. The graphite mixtures evolved little or no gas.

Blackwood and Bowden [15] believed that "oxyhydrocarbons", which they report could be extracted from the charcoals using acetone, were responsible for these reactions although the exact chemistry of these oxyhydrocarbons was not given.

Williams [16] investigated burning and flame-

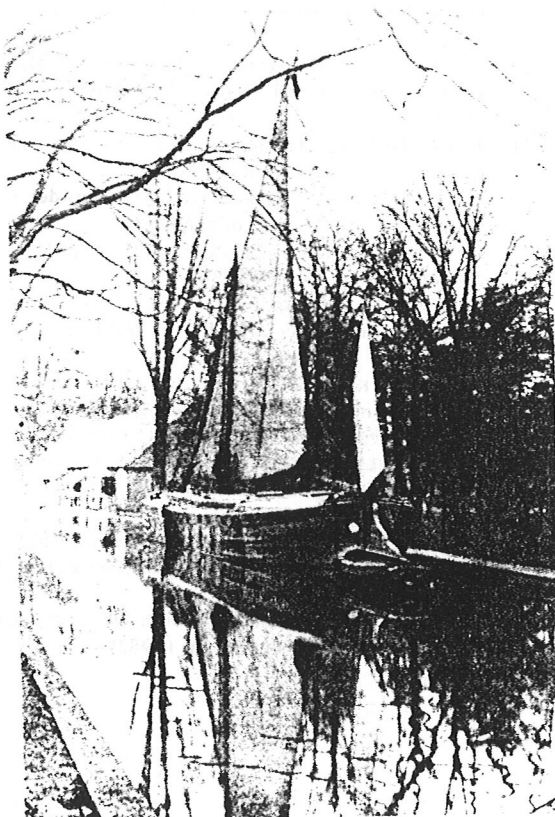


Figure 30 Photograph of powder barge which transported gunpowder from Waltham Abbey to Woolwich [13].

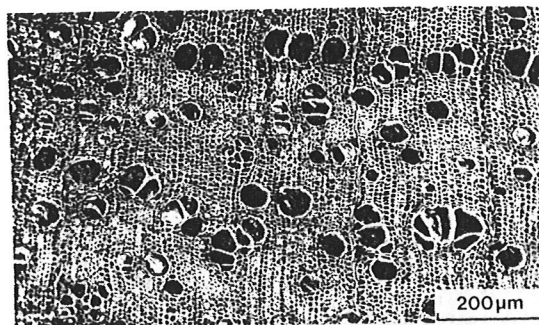


Figure 31 Transverse section of alder buckthorn charcoal [18].

spread in gunpowder and confirmed some of Blackwood and Bowden's [15] observations that flames spread through the porous matrix of the grains and that propagation was by jets of molten salts and some burning carbon fragments. He confirmed the previous results using spark and pulsed laser ignition.

Kirshenbaum [17] investigated the effect of different types of carbon on ignition temperatures of gunpowder by using charcoal, channel black and furnace black. It was claimed that volatile matter and ash content did not affect ignition temperature so perhaps the oxyhydrocarbons are not important. With furnace blacks the ignition temperature decreased with increasing surface area. With channel blacks and charcoals no similar correlation was found. Although Kirschenbaum [17] disagreed with Blackwood and Bowden, who believed extractable materials in the charcoal were responsible for the performance in gunpowder, he was not able to suggest an alternative theory.

7. Recent studies

Work in the Northern Carbon Research Laboratories started in 1979 following the closure of the Nobel's Explosives Company gunpowder plant at Ardeer when production of gunpowder was trans-

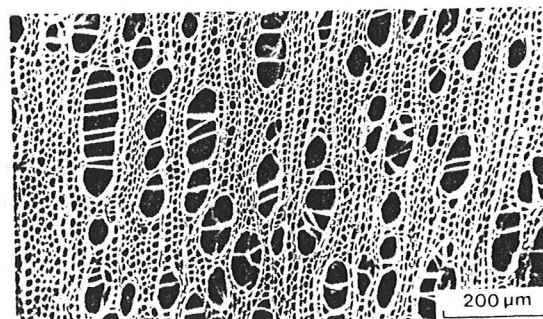


Figure 32 Transverse section of alder charcoal [18].

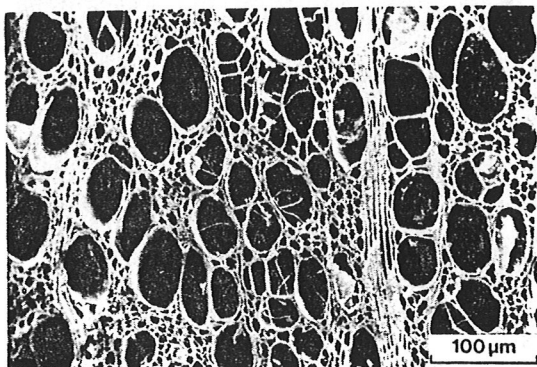


Figure 33 Transverse section of beech charcoal [18].

ferred to a German company. Although the gunpowder was manufactured using equipment moved from Ardeer, expertise had been lost and the German gunpowder proved unsatisfactory. As the main problem appeared to be the reproducibility of the charcoal, the aim of this study was to characterize the physical and chemical properties of the charcoals and replace them with an easily manufactured synthetic material [18].

The charcoals, supplied by Ministry of Defence, were identified by examining cut specimens using scanning electron microscopy. The three types of charcoal were identified as hardwoods, being alder buckthorn (*Frangula alnus*) known earlier as "dogwood" or "black dogwood", alder (*Alnus gultinosa*) and beech (*Fagus sylvatica*). Micrographs of the three hardwood charcoals are shown in Figs. 31 to 33.

Alder buckthorn is used in gunpowder for evenly burning fuzes whilst alder is used in most commercial powders and beech is used where precise burning is not required. Alder buckthorn charcoal was found to have the lowest spontaneous ignition temperature and highest porosity (as measured by mercury porosimetry) of the three charcoals and could be the most suitable charcoal for gunpowder because of the ease of ignition. The evenly distributed porosity of the charcoal may promote burning of gunpowder.

Acknowledgements

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References

1. J. NEEDHAM, "The Guns of Khaifeng-fu", *Times Literary Supplement*, 11 January, 1980, pp. 39-42.
2. J. R. PARTINGTON, "A History of Greek Fire and Gunpowder" (W. Heffer and Sons, Cambridge, 1960).
3. R. BACON, "Epistola de Secretis Operibus Artis et Naturae et de Nullitate Magiae" (Johannis Dee, Hamburg, 1618). Held at the British Library Reference Division, Great Russell St, London.
4. W. de MILEMETE, "De Notabilitabus, Sapientis, et Prudentia" (1326). Held at the library of Christ Church college, Oxford.
5. M. McLAREN, "The Explosives Research and Development Establishment, Its Historical Background", *J. Naval Sci.* 1 (1975) 176.
6. J. HOPE, "The Diary of Sir James Hope, 21 January-1 October, 1646" edited by P. Marshall, *Miscellany of the Scottish History Society* IX (1958).
7. D. DIDEROT, "L'Encyclopedie on Dictionnaire Raisonne des Sciences, des Art et des Metiers" (Paris, 1763). Held at the British Library Reference Division, Great Russell St, London.
8. W. PAGE and J. H. RAND (eds), "The Victoria History of the County of Essex", Vol. 2 (Constable, London, 1907).
9. W. H. SIMMONS, "A Short History of the Royal Gunpowder Factory at Waltham Abbey" (Issued by Controllor of Royal Ordnance Factories, London, 1963).
10. F. DRAYSON, "A Treatise on Gunpowder", Manuscript, Public Record Office, PRO Supply 5/762 (1830).
11. F. BADDELEY, "The Manufacture of Gunpowder" (H.M.S.O., Waltham Abbey, 1830).
12. W. H. WARDELL, "Handbook of Gunpowder and Gunotton" (H.M.S.O., London, 1888).
13. W. G. FITZGERALD, "How Explosives are Made", *Strand Magazine* IX (1895) 307.
14. F. M. SMITH, "A Handbook of the Manufacture and Proof of Gunpowder" (H.M.S.O., Waltham Abbey, 1870).
15. J. D. BLACKWOOD and F. P. BOWDEN, "The Initiation, Burning and Thermal Decomposition of Gunpowder", *Proc. Roy. Soc. (A)* 213 (1952) 285.
16. F. A. WILLIAMS, "Observations on Burning and Flame Spreading in Blackpowder", *AIAA J.* 14 (1976) 637.
17. A. D. KIRSCHENBAUM, "Effect of Different Energy of Black Powder", *Thermochemica Acta.* 18 (1977) 113.
18. E. GRAY, Ph.D. Thesis, School of Chemistry, University of Newcastle upon Tyne (1982).

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