WASC 2190

ERDE Research
Summaries
donated by
Mr. W. Smith

WASC 2190/001

ERDE

THE EXPLOSIVES RESEARCH AND DEVELOPMENT ESTABLISHMENT OF THE PROCUREMENT EXECUTIVE, MINISTRY OF DEFENCE: ITS HISTORY AND CURRENT ACTIVITIES

The Establishment occupies the site of the former Royal Gunpowder Factory, established in 1787 on the premises of a gunpowder mill then in the possession of the Walton family. Under the direction of Sir William Congreve and his successors production and quality of black powder were greatly improved. The US Ordnance Manual of 1862 records that no one makes better powder than the Waltham Mills. Manufacture of guncotton was begun in 1872 and of cordite in 1891. Later the Factory undertook the manufacture of more advanced explosives and propellants, culminating in the production of RDX at the beginning of the Second World War - the Factory was the only source of this explosive in the UK for some years. The Branch structure below has evolved from the time when the Establishment assumed its present name in 1948, by which date much of the work of the former Research Department at Woolwich Arsenal had been transferred to Waltham Abbey.

Explosives Branch	Development of explosive compositions, explosives performance, development of initiating explosives and assessment of sensitiveness and hazard associated with the use of explosives and propellants
General Chemistry Branch	Chemical analysis by classical and instrumental methods (including spectroscopy and gas and liquid chromatography), thermal analysis, compatibility of explosives and propellants with materials of construction, X-ray crystallography (including computer analysis of data), development of electronic and mechanical instrumentation, glassworking, mathematical, computing, library and information services
Non-Metallic Materials Branch	Development and small-scale production of plastics and rubbers for use in association with explosives and propellants and in other exacting environments; simulated and natural weathering of materials (conducted in association with a tropical exposure site in Queensland, Australia), fabrication of composites using asbestos, carbon fibre, glass fibre and 'whisker' reinforcement, automated testing of plastics for creep, flexural and tensile properties over a wide temperature range, synthesis of high-performance polymers and instrumental study of polymers by infra-red nuclear magnetic resonance, ultraviolet and visible spectroscopy, vapour phase and gel permeation chromatography and thermal analysis methods.
Process Research Branch	Synthesis of organic compounds (low molecular weight and polymeric) for use in explosives and propellants, development of chemical engineering processes for explosives and propellants ingredients to full production scale if needed. pilot plants for the manufacture of new or otherwise inaccessible materials, study of thermophysical properties of solids liquids and gases over wide temperature and pressure ranges and of heat transfer
Propellants 1 Branch	Development of propellants for guns, rockets and other uses from nitrocellulose and nitroglycerine bases, ballistic assessment, calorimetric and combustion studies on such propellants
Propellants 2 Branch	Development of plastic propellant with poly-iso-butylene and polyisoprene binders as used in UK and foreign meteorological rockets, development of propellants using rubbery binders derived from telomers ('liquid rubbers'), ballistic assessment of propellants for rockets, rheology of propellants and study of adhesion, adhesives and sealants

EXPLOSIVES RESEARCH AND DEVELOPMENT ESTABLISHMENT
WALTHAM ABBEY, ESSEX EN9 1BP LEA VALLEY 713030

Packaged Power from Solid Propellants

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Packaged Power from Solid Propellants

General

Instantly ready, programmed power can be obtained from gas-operated devices by the use of packaged solid propellant charges. The performance is built in during manufacture by selecting a propellant with a suitable burning rate and using the correct geometrical form for the charge. Most of our work is for application in the Defence field but some of it is relevant to upper stage space motors and Civil activities such as signal and line-throwing rockets. We do not actually develop the weapons — this is the responsibility of other establishments and firms. It is our job to formulate and develop propellants for all three Services, and to devise better propellants that can be manufactured safely and accurately. A simple example will indicate the nature of our work. Nitroglycerine is an ingredient of one type of propellant. On its own, it is such a violent explosive that 1 oz. (28 g) would blow a hole in a 9-inch (22.9 cm.) brick wall. When suitably tamed as a propellant, the same quantity will propel a 10 lb. (4.5 kg) mortar bomb safely for 1000 yards (900 m). All batches of propellant must behave similarly when new and also after some years of storage in the tropics. Most of our work is Security Classified and so is not publicised but *all British weapons* contain significant contributions from us no matter which firm is responsible for the weapon development.

The introduction to exhibit A1 indicates the range of weapons and devices in which propellants are used. They can be classified on the basis of operation times:-

10 to 100 milliseconds	Guns, mortars, revolvers, shoulder-fired rockets, ejection devices for bombs or "black boxes".
A second or two	Boosts for guided missiles and aircraft rockets.
Five seconds to a minute	Guided missile sustainers, engine starters, space applications.
A minute or more	Liquid ejection charges.

ERDE has experienced personnel and a well established expertise for work on propellants for all these fields of work.

Service weapons have been used in the past throughout the world and must operate safely and successfully after exposure to full sunlight (say 70°C) or at —40°C on earth or at even lower temperatures on aircraft at high altitude. The latter also present additional problems in that aerodynamic heating can arise for short periods and result in temperatures well over 100°C.

Types of Propellants

ERDE works on two main types of propellant which are briefly described below.

Double-Base or Colloidal Propellants

These contain nitrocellulose and nitroglycerine (or other nitric ester) with various additives to give the required burning properties. They are used when minimum SMOKE is a requirement. *The Solvent Process* is used when small granules are required for short burning times in guns and small arms. Rocket charges are made by either *Solventless Extrusion* from hydraulic presses, the size of which limits charge diameter or by the *Cast Double Base* (*CDB*) *Process* which is suitable for making very large charges. Some compositions may be made by either process, both of which are well established in the production factories. As loose charges, these propellants have good low temperature performance but are slightly low in energy. Recently this has been remedied by the successful development at ERDE of a process for bonding a special type of soft CDB to the motor wall and the development of higher energy compositions containing ammonium perchlorate.

Composite Propellants

Special exhibits A5-8 deal with these propellants.
They contain ammonium perchlorate with an inert binder which acts as a fuel; aluminium is often added to increase the energy level.

Plastic Propellant

was invented in the U.K. It uses a high molecular weight liquid polymer as binder and a high content of filler, giving a high energy propellant. It is readily processed and bonded to the motor wall but its physical nature gives limitations in size and low temperature performance. Propellants made with *rubbery* binders do not have these limitations and can be used over a wide range of temperature but they are slightly lower in energy.
Propellants do not need air for burning and consequently are ready for instant operation. The surface decomposition products react in a fraction of a millisecond to give gases such as carbon dioxide, water, carbon monoxide, nitrogen and hydrogen together with aluminium oxide and hydrogen chloride from aluminised composite propellants. Flame temperatures range from 1500°C to 3000°C and the hot gases produced expel projectiles from guns or propel rockets when they escape through nozzles as high pressure jets. One kilogram of a typical high energy propellant under suitable operating conditions can supply a force of 250 kg for one second, and on a weight basis

this is at least twenty times more effective than the performance from modern jet aircraft engines.

Facilities at ERDE

Our facilities for developing and processing experimental propellants are unique in that all types can be assessed in a single establishment. Our processing plant ranges from the laboratory scale of a kilogram or so up to full scale production plant capable of dealing with a thousand kilograms or so. Basic parameters are established from material manufactured on the kilogram scale by precise measurements in our well-equipped laboratories. Full-scale plant is used to manufacture large quantities of experimental propellants which are supplied to the Weapon Development Establishments to expedite new weapon developments.

We also use our full-scale plant to provide the Royal Ordnance Factories with quality control procedures for ballistic control, improved safety devices for production (such as fire drenching devices) and also to specify purity standards for new ingredients.

Propellant charges must be free from flaws and we are equipped for radiographic and ultrasonic inspection, which we have pioneered. (The ERDE Mk.III Flaw Detector has been sponsored commercially and sets have been purchased by Australia, Canada, India, Germany and France.)

We are responsible for ensuring that all our propellants have an acceptable Service life under world-wide conditions. Advance information during the development phase is essential and ERDE has facilities for conducting accelerated trials and thermal cycling tests so that good storage capability is built into weapons at the design stage.

Applied Research

Improved propellants and associated materials are required as the sophistication of weapons increases. Current items include the following:-

1 Faster or slower burning rates, to give optimum weapon aerodynamics—requires a knowledge of catalysis and the mechanism of combustion.

2 Greater energy content to improve weapon range or reduce weight—requires computer work on compositions and special safety consideration as hazards frequently increase with energy level.

3 Wider temperature range of operation to simplify Service handling and for higher speed aircraft—requires information on mechanical properties at high and low rates of strain and on polymers.

4 Reduction of smoke and flash to increase range of visually guided weapons—requires a knowledge of fundamental thermodynamics, pyrolysis of materials, and the physics of smoke formation.

5 Longer Service life to minimise replacement costs—requires work on gas evolution and permeability, polymer deterioration, plasticiser migration, etc.

6 High temperature resistant compositions for high speed flight applications—requires assessment of high temperature binders and special "cook-off" testing to ensure safety.

7 Reduced temperature coefficients to enable increased performance to be achieved from an existing device or to reduce inert weights—requires ballistic modifiers and a knowledge of burning under erosive conditions.

8 Adhesive systems for bonding propellants to the motor wall to enable lightweight cases to be used—requires expertise in adhesion over a wider temperature range than normal and also storage work to confirm permanence of bonding.

9 Reduction of erosion in high velocity guns—requires special extrusions and special compositions.

10 Combustible cartridge cases for new guns—requires a knowledge of paper felting technology and physico-chemical properties of composite paper-nitrocellulose materials.

Weapon Applications

ERDE makes important contributions to the propulsion systems for all weapons. We are involved in a complete spectrum of weapon development which ranges from the design assessment stage to the problems which arise with propellants in prolonged Service use.

The most important single recent application of our propellants has been in the Martin Baker Aircraft Rocket Ejection Seat. The marrying of superb engineering with a first class propellant has resulted in an Ejection Seat for which large export orders have been received from many countries.

Our propellants are also being used for the latest weapon developments for all three Services such as Blowpipe (by Shorts) Atlas (by BAC), Knebworth (by RARDE) and Giant Viper (by MEXE), and also in other classified weapon developments. The basic ERDE case-bonding process for CDB has been applied by the motor designers in the new Naval Seaslug 2 and Sea-dart.

Older weapons which are in or near to Service use are also based upon ERDE propellants and include the new aircraft weapon Red Top, the 2-inch aircraft rocket and the first generation Guided Missiles such as Thunderbird, Bloodhound, Seaslug, Firestreak and Malkara (an Australian development).

A recent development which appears to us to be a "growth area" is combustible cartridge cases for use in guns, which are being exploited in two new guns on the Classified list. The ERDE process gives a product greatly superior to and cheaper than any other combustible case available elsewhere.

All Service weapons receive comprehensive testing for Service acceptance and ERDE has been involved in assessment and consultative work for Seacat, Vigilant, Swingfire, Rapier and foreign weapons purchased by the Services. We have a sound knowledge of foreign propellant technology.

ERDE is also closely associated with propellant aspects of Martel, the current joint Anglo-French development, and has been involved in special bi-lateral propellant projects with Belgium, Holland and Norway.

Space and Meteorological Applications

These are shown in some detail in exhibit A5-8. The rockets for the very limited U.K. space programme, such as Skylark, and the meteorological sounding rockets, Skua, and Petrel use our plastic propellants.

Civil Activity

The new Coastguard rocket uses one of our extruded propellants. Recently we have extended the service that we give on propellants to Private Industry. Signal and line-throwing rockets, being manufactured by Pains-Wessex, use our plastic propellant and development is also proceeding with Schermuly Ltd.

Propellants are essentially plastics and some further exploitation of our expertise, which ranges from rheology to combustion and ultrasonic inspection may be possible in this field. The standards and philosophy of excellence achieved for Defence work must be applicable to high quality commercial products.

Enquiries should be addressed to:

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Superintendent, Propellants I Branch
or
Mr. P. R. Freeman
Superintendent, Propellants 2 Branch
ERDE
Waltham Abbey, Essex
Tel: Waltham Cross 23688
(Extn. 319 or 414)

Procurement Executive, Ministry of Defence

Development and Applications of Composite Rocket Propellants

ERDE

Development and Applications of Composite Rocket Propellants

What is a Composite Propellant?

Composite Propellants are chemical mixtures containing oxidising and fuel components; a well-known example is gunpowder. As most schoolboys know, gunpowder is a granular mixture of potassium nitrate, an oxidising component, with carbon and sulphur, which are fuel components. When ignited, even out of contact with air, gunpowder burns vigorously because the potassium nitrate supplies the oxygen required for burning the carbon and sulphur. This combustion liberates a considerable quantity of heat and a considerable quantity of gas. If it takes place in a confined space, the result is the generation of hot gas at high pressure from which useful work can be obtained. In a rocket motor, propulsive power is derived from the gas by allowing it to escape through a nozzle as a high velocity jet.

Although gunpowder is a suitable propulsive filling for small low-performance rockets, such as those used for celebrating the 5th of November, much more powerful composite propellants have been developed for military rocket motors. These more modern composite propellants are now finding increasing application in rocket motors used for civil purposes.

Case-Bonding of Rocket Charges

There are two main types of composite propellant used in British rocket motors; plastic propellant, which is putty-like, and rubbery propellant, similar in appearance to the rubber used for making car tyres. Both types are used in the form of a single block, or charge, which exactly fits the rocket motor case and is tightly bonded to the internal wall of the case. The charge usually has a central hole, or conduit, which provides an exposed surface at which burning can take place. One of the objects of case-bonding is that the propellant charge protects the motor case from the hot combustion gases. This allows a lightweight case to be used, reducing the overall weight of the motor and increasing its performance.

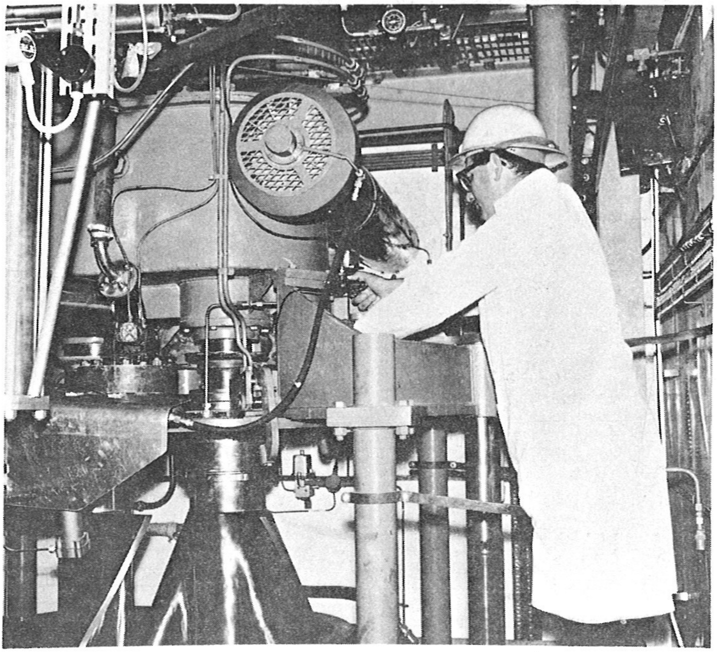
Military rockets are designed for use in any part of the world and consequently they are required to function satisfactorily over a wide range of temperature. An even wider range may be required for some particular purpose, as with rockets carried externally on aircraft at high altitude. All materials expand or contract to some extent when subjected to a change in temperature and it is an unfortunate fact that the expansion or contraction of a rocket motor case is usually very much less than that of the propellant charge it contains. As a result, when there is an increase in temperature, the motor case squeezes the charge, and when there is a decrease, the charge tends to shrink away from the case. A rigid propellant material would be broken into pieces by the squeezing action or would become detached from the case at low temperatures. Either eventuality would be disastrous since the propellant burning surface would be increased to such an extent that the extra gas generated on ignition would be sufficient to burst the case. It is for this reason that composite propellants are formulated so as to have plastic or rubbery properties. This enables them to be easily deformed by the case without rupture or loss of adhesion, so that considerable temperature changes are possible without any bad effect.

Several Thousand Different Compositions

The oxidising component for both plastic and rubbery composite propellants is usually ammonium perchlorate, a very powerful oxidiser which nevertheless is safe to handle. Plastic compositions are made by mixing the powdered oxidant with a viscous organic liquid fuel, such as polyisobutylene, so as to form a stiff paste or putty. Other ingredients are used to aid the mixing process, to control the burning rate and physical properties, and to increase the energy content. The propellant is loaded into motor cases by a simple pressure moulding technique.
Rubbery propellants are also made by mixing the powdered oxidant and other ingredients with an organic liquid fuel but, in this case, a less viscous liquid which subsequently can be converted to a soft rubber by means of a chemical reaction is used. The propellant mix is produced in the form of a slurry which can be poured or cast into the rocket motor case and then converted to solid rubber by the application of heat.

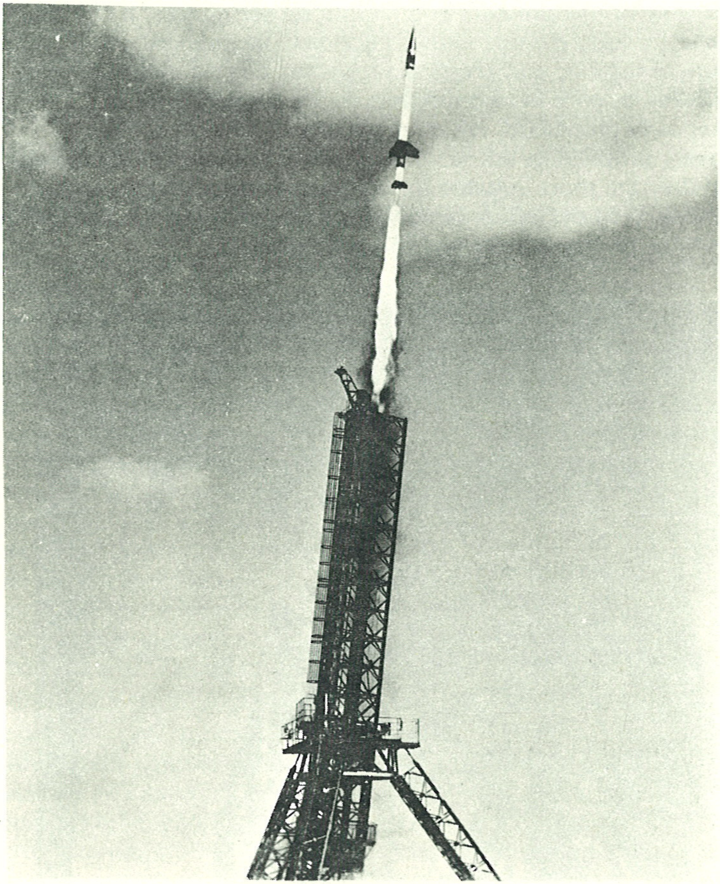
In general, each rocket motor application requires a propellant with certain specific characteristics and the rocket motor designer will state his needs in terms of ballistic requirements (burning time, chamber pressure, total impulse) and mechanical behaviour requirements (dependent on size and type of charge, usage temperature range, acceleration on projection). It is then up to the propellant chemist to produce a composition which will meet the various requirements. This is done largely on the basis of knowledge previously obtained from the systematic study of the effects of variations in composition and manufacturing method. Many experimental compositions have been made for this purpose, particularly in the case of plastic propellant which has been under continuous research and development for about twenty-five years. During this period several thousand completely different compositions have been made and the characteristics of each have been assessed by extensive laboratory measurements, static firing in small rocket motors, storage trials, and so on. The scope and limitations of this type of rocket filling are, therefore, fairly well established.

Plastic propellant is a British invention which offers many advantages over other rocket propellants. It is powerful, cheap and easy to manufacture and fill into rocket motors, it can be stored for long periods without appreciable change, and a wide range of ballistic and physical properties can be obtained. It is, however, inferior in two respects to the rubbery type of composite propellant, which originated in the USA. These rubbery propellants are more resistant to extremes of temperature and they are more suitable for use in very large rocket motors. Development of both types of composite propellant is therefore desirable to cover every possible requirement.



Facilities at ERDE

Many requirements for the formulation and development of new composite rocket propellant compositions come to the ERDE at Waltham Abbey since this is the only place in the United Kingdom where such work is carried out. The Establishment is equipped with all the facilities needed for the development of any composition. Initial investigations are carried out in laboratories and with small-scale manufacturing plant and this is followed by sufficient large-scale manufacture to establish exact manufacturing and quality control procedures. Final large-scale production is usually the responsibility of the Royal Ordnance Factories, but problems or difficulties which arise during manufacture are referred back to the ERDE.



Some of the Applications

The success of this work is indicated by the use of composite propellant charges in a large variety of British rocket motors. These range from the tiny Imp VIII, less than 3½ inches long and containing half-an-ounce of propellant, to the Raven motor, over 17 feet long and containing almost a ton of propellant. Composite propellant rockets are used as boost motors for the latest versions of the Thunderbird and Bloodhound guided weapons, they are used for other military weapons under development which are still security classified, for the Skua and Petrel meteorological rockets produced by Bristol-Aerojet Ltd., for the highly successful Skylark upper atmosphere research vehicle, for small line-throwing rockets, parachute distress rockets, and the Minotaur long-range illuminating rockets produced by Pains-Wessex Ltd., and for rockets of various sizes used for the propulsion of a high speed sledge at Pendine which is used for testing the resistance of delicate equipment to high accelerations. If a British satellite launcher is developed it will, in all probability, have a composite propellant rocket motor for the upper stage.

Enquiries should be addressed to:

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P2/1

Development and Applications of Composite Rocket Propellants

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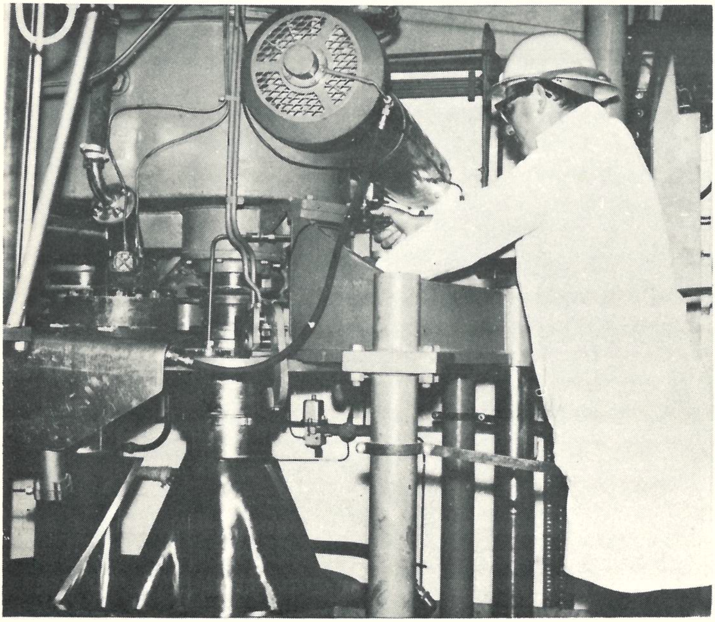

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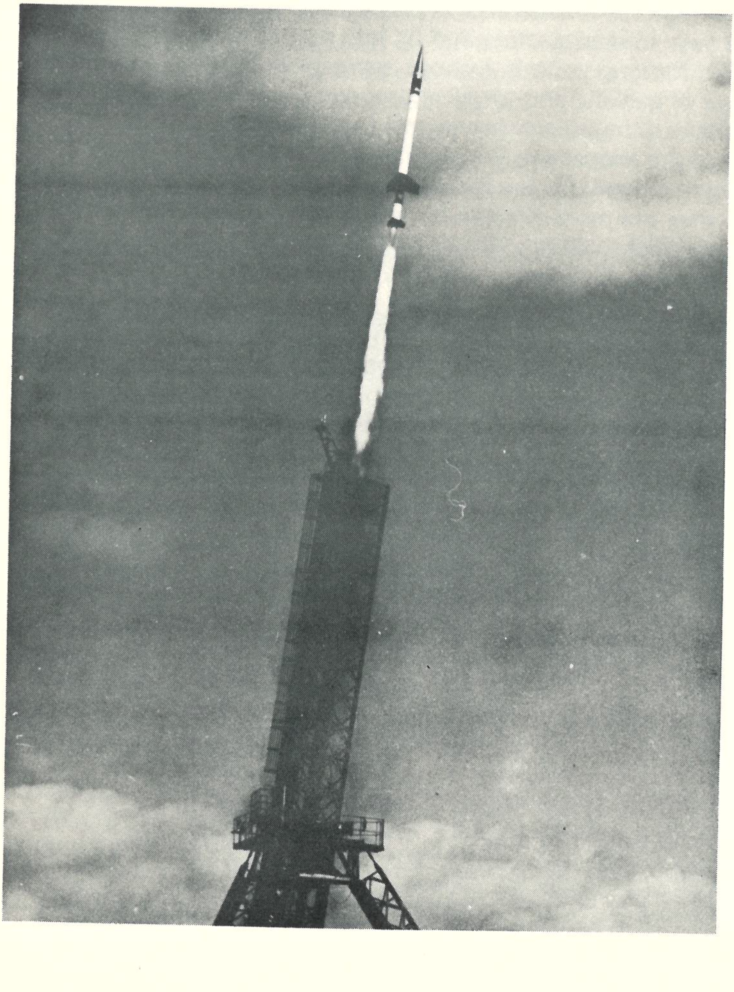

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Mr. P. R. Freeman
Superintendent Propellants 2 Branch
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Tel: Waltham Cross 23688 (Extn. 414)

Procurement Executive, Ministry of Defence

Polymer Development and Applications

ERDE

Polymer Development and Applications

The work of the Polymer Development and Applications Group is divided into two main sections: evaluation of new rubbers, thermoplastics and thermosetting resins; and special investigations and 'trouble shooting'.

Evaluation of New Rubbers and Plastics

Considerable attention is, and has been, paid to the evaluation of new and improved rubbers and plastics. These include processing studies on modern commercial machinery, optimisation of moulding conditions and moulding of end items, curing and vulcanisation, and assessment of their ageing characteristics under a wide range of conditions likely to be met with in use. Polymers are normally subjected to accelerated ageing under hot/wet and hot/dry conditions, in contact with explosives and propellants, and protracted immersion in petrol. The results obtained in the accelerated trials are compared with results obtained on samples exposed to outdoor ageing at Waltham Abbey and in Queensland, Australia.

In order to optimise the moulding conditions for rubbers and plastics, a number of special purpose moulds have been designed in the group, to produce the wide range of test specimens required for a full assessment of the engineering properties of these materials.

Special investigations and 'Trouble Shooting'

'Trouble shooting' investigations are frequently made into the causes of failures in military and civil use. These investigations, and the background investigations into the processing, moulding and ageing characteristics of rubbers and plastics allow the Polymer Development and Applications Group to offer unbiased advice to designers and users on the choice of material for a particular application. Examples of some failures in service are shown.

As a result of some of the investigations, the development of new materials has to be undertaken to provide a satisfactory solution to the problem. Typical of such a problem was the development of static electricity on the solid rubber tyres of tracked vehicles. It was suggested that a high electrically conducting rubber might provide a solution, and a range of new, highly conducting rubber compounds were developed at E.R.D.E. The best of these also proved to have excellent wear characteristics and low heat build-up, and assessment of one of these compounds, L7/70, in commercially produced solid tyres is in progress.

Currently work is going on on the effects of moulding machine variables on the properties of two classes of material, glass-filled thermoplastics and filled thermosetting resins. Such work is of particular interest to specifications. A further investigation is involved in the satisfactory extrusion of a 'composite' plastic inhibitor tube using mixtures of cellulose acetate and modified polyoxymethylene.

Equipment

The group is equipped with a range of modern small scale and commercial processing equipment, including rubber mills, plunger and reciprocating screw injection moulding machines, equipment for injection moulding of rubbers and thermosets, hydraulic presses, and equipment for transfer moulding of rubbers and thermosetting resins.

In addition, the group has a wide range of standard testing equipment, which enables a full assessment of the physical properties of the materials used and produced in the group to be made.

The expertise of the group is available to users and designers, both military and civil, for consultation, and special investigations and moulding of end items can be undertaken.

Enquiries should be made to:

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NM/6

Polymer Development and Applications

Mintech
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Polymer Development and Applications

The work of the Polymer Development and Applications Group is divided into three main sections, work on rubber-proofed fabrics; evaluation of new rubbers, thermoplastics and thermosetting resins; and special investigations and 'trouble shooting'.

Proofed Fabrics

Work on proofed fabrics has been chiefly directed towards the development of large flexible storage containers for liquids, particularly petrol and oil. These large structures contain stuck or stitched joints in the proofed fabric skin, and many investigations have been made into the design and strength of such joints, particularly for the Dracone flexible barge and large pillow tanks.
Currently, the preparation of standard samples of rubber-proofed nylon fabrics under closely controlled conditions is being undertaken, in order to obtain basic engineering data on these materials. This is in an attempt to elucidate some of the failures which have occurred in Hovercraft skirts. Also under investigation are high strength proofed fabric tapes for use in aircraft arrester gear. Samples of experimental tapes with nylon or steel reinforcement are being shown.

Evaluation of New Rubbers and Plastics

Considerable attention is, and has been, paid to the evaluation of new and improved rubbers and plastics. These include processing studies on modern commercial machinery, optimisation of moulding conditions and moulding of end items, curing and vulcanisation, and assessment of their ageing characteristics under a wide range of conditions likely to be met with in use. Polymers are normally subjected to accelerated ageing under hot/wet and hot/dry conditions, in contact with explosives and propellants, and protracted immersion in petrol. The results obtained in the accelerated trials are compared with results obtained on samples exposed to outdoor ageing at Waltham Abbey and in Queensland, Australia.

In order to optimise the moulding conditions for rubbers and plastics, a number of special purpose moulds have been designed in the group, to produce the wide range of test specimens required for a full assessment of the engineering properties of these materials. The production of low-cost moulds in metal-filled thermosetting resins, either for prototypes or for limited production runs, is currently under investigation. Examples of the moulds and mouldings produced from them are shown.

Special investigations and 'Trouble Shooting'

'Trouble shooting' investigations are frequently made into the causes of failures in military and civil use. These investigations, and the background investigations into the processing, moulding and ageing characteristics of rubbers and plastics allow the Polymer Development and Applications Group to offer unbiased advice to designers and users on the choice of material for a particular application. Examples of some failures in service are shown.

As a result of some of the investigations, the development of new materials has to be undertaken to provide a satisfactory solution to the problem. Typical of such a problem was the development of static electricity on the solid rubber tyres of tracked vehicles. It was suggested that a high electrically conducting rubber might provide a solution, and a range of new, highly conducting rubber compounds were developed at E.R.D.E. The best of these also proved to have excellent wear characteristics and low heat build-up, and assessment of one of these compounds, L7/70, in commercially produced solid tyres is in progress. Samples of some of these new conducting rubbers are on show.

Currently, work is in hand to produce low-cost fibre reinforced thermoplastics possessing good engineering properties such as high strength and rigidity with low weight. Examples of a range of thermoplastics containing fibrous fillers, particularly asbestos, as reinforcement are on display. The work has formed the basis of a British Patent application.

Equipment

The group is equipped with a range of modern small scale and commercial processing equipment, including rubber mills, plunger and reciprocating screw injection moulding machines, equipment for injection moulding of rubbers, hydraulic presses, and equipment for transfer moulding of rubbers and thermosetting resins. Most of the equipment will be demonstrated.

In addition, the group has a wide range of standard testing equipment, which enables a full assessment of the physical properties of the materials used and produced in the group to be made.

The expertise of the group is available to users and designers, both military and civil, for consultation, and special investigations and moulding of end items can be undertaken.

Enquiries should be made to:

Dr. B. L. Hollingsworth, Polymer Development and Applications Group, Materials 1 Branch, E.R.D.E., Waltham Abbey, Essex
Waltham Cross 23688, Extension 451

WASC 2190 / 005

Mintech ERDE

BALLISTIC CONTROL OF ROCKET CORDITE

Traditionally, chemical analysis and dimensions were used as the main controls on the uniformity of cordites. When ballistic catalysts are not used, the burning rate, calorimetric value and density run parallel to composition. Calorimetric value is a very good overall check on composition, whilst density is a good quick test that nothing is far wrong, either chemically or due to porosity.

Chemical methods, though still essential to confirm long-term stability and compatibility, and frequently required to clear up other problems, are no longer sufficient for modern rocket propellants. This is due partly to the close ballistic tolerances called for by rocket designers and partly to the use of ballistic catalysts whose amount and activity can both greatly alter the burning rate. The chemistry can then still be within the accepted limits whilst the burning characteristics are unacceptable.

ASSESSMENT

ERDE has developed a very good self-contained automated recording equipment for the static firing of small rockets, using standard strain gauges to measure pressure, and including electronic integration of varying pressure-time and thrust-time functions, and reliable gating of the rapidly rising and falling pressure transient, to give a direct read-out of the time of burning[1]. The strand burner simultaneously firing three one-eighth inch cordite strands in a pre-pressurized chamber, and timing the burning of a 5-inch length of lightly inhibited propellant, is even simpler.

These techniques are used for all solid propellants at ERDE, small rockets (2-inch 'K' rounds) being generally preferred to strands for cast double-base and composite propellants on grounds of accuracy. Although strand rates run a few per cent low, correlation with rockets is adequate for their routine use in production control of cordites.

PROCESS CONTROL PROCEDURE

A choice is made of some convenient factor, or factors, that can be varied readily in factory production to control the burning rate, with little or no alteration in other properties, including:

1 The amount of ballistic catalyst (AU, BU, PU cordites, some composite propellants)

1 Rocket Propulsion Technology, Vol 1, 167-184. (Exhibit No 35)

2 The activity of the catalyst, which is controlled by blending a number of batches to give a specified ballistic level (VU cordite).

3 The amount of a cool ingredient (EU, TU cordites). This is not used in cases where specific impulse is critical.

4 The oxidizer grist size (particularly in composite propellants).

5 Small adjustments by varying processing conditions (solventless cordite rolling, or proportions of rework).

6 Combinations of these methods.

A very thorough study was made of a Service cordite a few years ago, to harmonize the ballistics from manufacture at ERDE and the two production factories. There were differences in processing equipment, in the purity of the factory water supply and in the allowances made for loss of catalyst at the wet-mix stage (based on chemical analysis). These resulted in ballistic differences and a comprehensive investigation showed that:

Steam injection of Additive Y solution increases depth of mesa, as compared with simple mixing.

Increase in amount of Additive Y increases rate slightly and depth of mesa considerably.

Increase in amount of Additive X increases rate considerably and depth of mesa slightly.

Hard water (mainly calcium bicarbonate in solution) worsens the mesa slightly.

Different sources of paper for making nitrocellulose sometimes affected ballistics slightly.

Re-use of wet-mix back water containing traces of particular compounds considerably worsens both the reproducibility of the burning rate, and the mesa depth.

It turned out to be uneconomic to install new plant to eliminate all these factory processing differences, so some latitude was allowed in the amounts of X and Y, at each factory, so that the ballistics were standardized. A running chart of ballistics, calorimetric value, chemical analysis and tensile data was instituted, working to within-factory limits which were set about two-thirds as wide as the specification limits. Provided a regular flow of production has been running, this has worked very well, and is also now used for other propellants.

NEW PROBLEMS OF BALLISTIC CONTROL

Some platonized propellants are quite sensitive to the nitrogen level in the nitrocellulose.

Any undue hold-up (over about two weeks) between mixing the ingredients and drying and rolling the cordite paste to form gelatinized sheet may cause small changes in burning rate due to slow hydrolysis of catalysts.

The grist size of fine powders is simply checked by measuring their specific surface as determined by the resistance they give to the passage of air, and a Fisher sub-sieve sizer is shown. Besides oxidants such as ammonium perchlorate, catalysts are also checked. The very finest particulate catalysts, such as carbon blacks present problems; nitrogen adsorption etc. has been measured but in practice it is found preferable to measure the direct ballistic effect and then blend up large batches to minimize the inspection needed (method 2) followed by minor adjustment of amount (method 1) as necessary.

Enquiries should be addressed to:

Dr C.G. Lawson
Propellants 1 Branch
(Ext 545)

A/4/1

Mintech ERDE

NON-DESTRUCTIVE TESTING OF SOLID PROPELLANTS

INTRODUCTION

Modern rockets are designed with lightweight cases for a high overall performance, and it is not feasible to allow factors of safety large enough to tolerate any badly flawed charges. Hence, it is essential to be able to detect such flaws [1]. Non-destructive testing is also required at ERDE to investigate the quality of charges made by experimental manufacturing techniques, and to study any defects that might develop in Service usage. The latter is generally simulated by standardized rough treatment, temperature cycling and hot storage.

Visual inspection is used wherever possible, but most modern propellants are opaque, and internal defects cannot be seen. The three methods mainly used at ERDE are radiography, charge deformation under vacuum (for air inclusions in plastic propellant), and ultrasonics. Application of the two latter techniques to propellants originated at ERDE.

The defects occuring in different solid propellants depend upon the various methods of manufacture and may include inhomogeneity, internal cavities, cracks, porosity, capillaries and lack of bonding. Acceptance standards are set up for each kind of flaw, depending on the nature of the rocket and its usage.

RADIOGRAPHY

RARDE has used radiography (X-ray photography) for propellants since about 1930 and, from their technology, ERDE procedures have been developed since 1950. The ERDE equipment is limited to 400 kV, suitable for propellant charges up to about a foot in diameter.

In capable hands it is an excellant tool, especially useful when diagnosing the causes of charge failures. Its general limitations such as the need for protection from radiation hazards, cost, and delay time for processing - with hold-up in inspection runs, are well known. Propellant radiography requires high sensitivity techniques (e.g. small X-ray source, large film-focus distance, fine-grain film with thin lead screens), involving highly skilled viewers whose output is limited by fatigue.

⇨

EXPLOSIVES RESEARCH AND DEVELOPMENT ESTABLISHMENT
WALTHAM ABBEY, ESSEX
WALTHAM CROSS 23688

Assessments made at ERDE in recent years to improve or cheapen techniques include:

1 Use of cobalt 60 source - found too slow and/or to give poor resolution

2 Photographic paper in place of film - poor resolution

3 Electronic image intensifier - poor resolution and tube costly

4 Xero-radiography - good quality for smaller charges but no cheaper than conventional radiography. Special selenium-coated plates are costly, easily damaged and rather small.

The radiography section gives a comprehensive service to the whole Establishment, though most of the work is devoted to double-base propellants. An automatic Refrema film processing unit was introduced in 1966 and enables films to be viewed only ten or fifteen minutes after exposure, besides saving labour.

ULTRASONIC INSPECTION

This method uses a beam of inaudible high-frequency sound waves which, owing to their short wave length, travel almost in straight lines, like light. They can be used to locate flaws, either by reflection of the radiation from the flaw, or by reduction of transmission through it. The transducers used for generating and detecting the radiation are discs composed of oriented piezo-electric crystals such as barium titanate or lead zirconate, with their flat faces plated with metal. An electric voltage between these faces is suddenly discharged, leading to a small change in dimensions of the disc, which vibrates at its natural frequency and emits a short damped train of ultrasonic waves. Pressure fluctuations generate a tiny oscillatory voltage between the metallized faces of the receiving transducers, which can be amplified and fed to a receiver. High frequencies ($2 - 5 \times 10^6$ cycles/second) give good resolution and are used in reflection sets to detect flaws in metal or absence of bonding, but they are rapidly attenuated in propellant and plastics. The ERDE-designed through-transmission set for plastics operates at a frequency of 370,000 cycles/second, with 60 pulses/second.

ERDE ULTRASONIC FLAW DETECTOR

The first design was developed between 1952 and 1955[2], made commercially and adopted by the Inspectorates and Ordnance Factories for inspecting cordite rocket charges. It has also been exported to the Commonwealth and NATO countries.

The propellant charge is immersed on rollers in water, the acoustic coupling medium, and the transducers in watertight housings traversed on either side of it. The received signal viewed on an oscilloscope is distorted by reflections and 'hash' which, however, do not affect the first half-pulse ⇨

travelling directly through the propellant. An electronic 'gate' is used to select this first signal, which is amplified and converted to a continuous DC current fed to a pen recorder. Any flaw deflects the pen, whose position on the chart can be correlated with the position in the charge that has caused it. The newer Mark III equipment is capable of inspecting propellant charges up to eighteen inches thick and is both rapid and cheap for production inspection. Inspection standards have been correlated with flaw sizes and current investigations cover precision work to quantify the ultrasonic and ballistic effect of density variations of the order of one per cent.

REFERENCES

1 Rocket Propulsion Technology, Vol 1, 269-279

2 British Plastics, July 1956, 262-264

Enquiries should be addressed to:

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WASC 2190/007

Mintech ERDE

FACILITIES AVAILABLE AT BALLISTIC ASSESSMENT SECTION

CALORIMETRY

Facilities are available for measuring heats of combustion of organic compounds containing any, or all of the following elements, carbon, hydrogen, nitrogen and oxygen, using an isothermal bomb calorimeter at 25°C. Temperature is measured by means of a platinium resistance thermometer in conjunction with a Smith's No. 3 Difference Bridge, to an ultimate accuracy of 0.0003°C in the working range of the apparatus. The precision apparatus, which is of a similar design to that used at the National Physical Laboratory, Teddington, is capable of giving results to an accuracy of the order of 0.01%.

Other equipment is available for the measurement of total heat evolution of pyrotechnic and propellant formulations.

PRESSURE RECORDING AND MEASUREMENT

Two semi-portable, self-contained, analogue pressure recording systems are available for 'on-site' measurements.

SYSTEM 1

Maximum Range of Pressure:	0-15,000 lb/in^2 (0-100 MN/m^2)
Transducer:	Quartz piezo gauge
Frequency Response:	Flat response within band 0-10 Kc/s
Recording Time Interval Range:	0.005s-1s
Accuracy:	2%
Record Dimensions:	18" x 6"

Facilities for synchronizing external events

SYSTEM 2

Maximum Range of Pressure:	0-15,000 lb/in^2 (0-100 MN/m^2)
Transducer:	Strain gauged type
Frequency Response:	Flat response within band 0-100 c/s
Accuracy:	2%
Record Dimensions:	Width 50 mm, length, as convenient

Permanent installations are also available for the recording of pressure and rate of change of pressure from explosions in closed systems at pressures up to 4.5×10^4 lb/in^2 (310 MN/m^2) and rates of change of pressure up to 4.5×10^7 lb/in^2/s (3.1×10^5 MN/m^2).

Enquiries should be addressed to:

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G/35/1

Mintech ERDE

COMBUSTIBLE CARTRIDGE CASES

Combustible cartridge cases have been under development for a number of years, since they offer several advantages over the traditional brass case, such as:

1 an appreciable saving in weight and of strategic raw material imports,

2 a reduction in the amount of toxic gas in a tank, normally brought in by the empty brass case,

3 no empty case disposal problem,

4 a saving in the cost of manufacture.

The combustible cartridge case must give adequate protection to the propellant charge and ignition system during its Service life, and be compatible with them so that the ballistic stability of the charge is not affected adversely. It must not absorb water readily. At the same time, it must be burnt completely before the projectile leaves the muzzle of the gun, and leave no debris in the gun.

One method of manufacture which has received wide support consists of several stages:

(a) a very dilute aqueous slurry of nitrocellulose and kraft fibres is felted on to a former of the required shape by a vacuum technique,

(b) the wet felt is removed from the former and bag-moulded to remove much of the water and to press it to size,

(c) after drying, the preform is impregnated with a resin solution, and again bag pressed to remove surplus resin solution and to give the required external contour,

(d) the solvent is removed by stoving and the combustible case is trimmed to the required length.

This process has a number of unattractive features, mainly associated with the resin impregnation stage, and the resin is expensive and has to be imported into the UK. These cases readily absorb water, and the relatively open structure of the material makes it difficult to apply a water-proof coating without its penetrating into the material and retarding burning.

A simplified process has been devised at ERDE whereby a water dispersable binder is used to improve the water resistance and strength of a mixed nitrocellulose/kraft fibre structure. This binder is made in the UK and is available from a number of manufacturers at a cost about one third that of the imported resin. It also permits the preform material to be moulded by the application of heat and pressure.

This process only involves felting from a water slurry of all the ingredients, either with male or female felting formers and pressing the nearly dry preforms for about a minute at 130°C under a pressure of 2.07 MN/m^2 (300 psi).

The burning properties for a nitrocellulose/kraft ratio of 2/1 can be varied over a twenty fold range by binder contents between 0 and 16 per cent. Parallel changes which occur in tensile strength are noted below:

Binder content per cent	Tensile Strength MN/m^2	Tensile Strength psi
0	3.5	510
4	9.5	1380
8	15.0	2180
12	21.5	3120
16	31.1	4510

Water absorption for such material containing about 14 per cent binder is about 4 per cent after immersion for 24 hours but this can be reduced to about ½ per cent by coating with a suitable lacquer.

Patent protection for the ERDE process has been applied for in Belgium, Canada, France, Italy, Germany, and the USA, in addition to the UK.

Our work is associated exclusively with ammunition usage but we have a sound background on felting technology and the process has industrial possibilities.

Enquiries should be addressed to:

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Mintech ERDE

THE COMBUSTION OF SOLID PROPELLANTS

The chemical energy stored in solid propellants is released by a process of combustion in which the initially solid reactants are converted, by a very complex sequence of chemical reactions proceeding at very high temperatures, to simple gaseous products. The development of sophisticated solid propulsion systems demands an understanding of the energy-releasing processes and research to this end is being actively pursued in all the leading countries.

Over the years ERDE has established recognized expertise in several areas: combustion and decomposition of systems containing nitrocellulose and liquid nitric esters; ballistic modification of these systems; the burning of ammonium perchlorate, a most important oxidiser in composite propellants; the combustion of aluminium in propellants and in the fundamental mechanisms involved in all these processes.

Some of the problems and complexities of propellant combustion in rocket motors can be studied to advantage at atmospheric pressure and below; other problems are peculiar to combustion at rocket-operating pressures and require high pressure laboratory techniques. One or two practical problems may be readily demonstrated at ordinary atmospheric pressure without recourse to rocket motor firings.

The rate of energy release is a function of the surface area of propellant exposed and may be illustrated by the very high burning rates which develop on ignition of dry guncotton or propellant 'crumble', compared with the relatively slow rates of properly-consolidated propellant. Very real hazards in the manufacture of solid propellants relate to the handling of finely divided energetic materials and propellant porosity or cracking presents dangers in usage. The phenomenon of the spread of flame over the propellant surface is not well understood.

Efficient usage of propellants requires that the energy-releasing reactions are complete before the products issue from the rocket nozzle. Two well-identified situations where this is not so are when double-base compositions are used at low operating pressures (say below 300-400 psig) and when aluminium powder is used as a high energy fuel without proper consideration of burning conditions. In the first case the inefficiency is related to the fact that in double-base systems energy is released in two distinct stages, and at low pressures only the first controls the rate of burning of the composition. If the pressure is too low, the second stage may not be complete inside the chamber. With progressively higher pressures both stages become more rapid and merge together. ERDE research into ballistic modification of double-base systems is concerned with the possibility of controlling the ⇨

partition of energy release in the two stages. The phenomenon is readily demonstrated at atmospheric pressure where the separation of the two stages is very large.

The addition of aluminium powder and certain other light metals to both double-base and composite systems can theoretically give a very significant increase in specific impulse. However, many problems are associated with the efficient practical usage of such metals. Research at ERDE has shown that one of the most important phenomenon in metal combustion is the natural growth of molten metal particles on the burning propellant surface. If conditions permit growth to large globules severe combustion inefficiency can arise. Propellant burning rate is one of many parameters which influence metal agglomeration and may be readily demonstrated at atmospheric pressure.

In the combustion of real propellant systems all the individual features are related to each other and to their environment and continued research is necessary if a wider and more positive understanding and control of these complex systems is to be achieved.

Enquiries should be addressed to:

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A/2/1

Explosives Research and Development Establishment

Waltham Abbey, Essex

USING SOLID PROPELLANTS

Solid propellants are used to power a wide range of devices. Rockets and guns are well known examples but there are many other applications. Here are a few.

The release of stores from aircraft uses an unlock/thrust mechanism which is instantly ready, fast, precise and reliable.

Ship-borne damage control uses a method for "nailing" steel plates together which is mobile and adaptable.

Aircraft emergency flotation equipment and escape slides use a source of cool gas, a preprogrammed quantity of which is available on demand.

Liquid expulsion systems ranging from the controlled slow delivery required for liquid-fuelled rockets to the instant high volume discharge of fire extinguishers.

Fluidic control uses a compact supply of cool, clean, gas.

What are Solid Propellants?

Solid propellants contain fuel, oxidiser and other additives and are designed to give controlled burning with good reproducibility even after 20 years storage. They are safe to handle, store and use; obviously precautions must be taken to avoid accidental ignition and HM Inspector of Explosives* has a code of practice which is mandatory.

Why should I use solid propellants?

Action time:	Milliseconds to a minute or more. Power output may be as high as 40 megawatts.
Programmable:	Generation of gas or energy can be designed to vary to give a boost/sustain mode of operation.
Compactness:	Hot gas more energetic than cold. Good power to weight ratio.
Reliability:	Consistent performance over a wide temperature range. Long shelf life with minimal maintenance.

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*Health & Safety Executive, Chief Inspector of Factories, Baynards House, No 1 Chepstow Place, London W2 4TF

How do I select a Propellant?

ERDE will advise on the choice of propellant for your application. In choosing they will wish to know how it will be used. Here is a sample of the questions which may be important.

Time	Is functioning time important? Must the propellant burn throughout this time? What is the residence time of propellant gases in the device?
Pressure	What pressure of gas is required? Must the propellant be burned at this pressure?
Temperature	What is the maximum gas temperature than can be tolerated during functioning? Over what temperature range must the device function reliably? At what temperature (or over what range) will the unit be stored?
Efficiency	Are there any weight or volume restrictions?
Physical Properties	Will the propellant be subjected to any unusual stress?
Gas Composition	Is it important to avoid condensation, corrosion, toxicity? Is smoke or debris in the product gases undesirable?

How do I design using Solid Propellants?

Typically solid propellants can be made to burn efficiently at pressures of about 3 MPa (30 bar, 400 psi) and upwards. Usually they burn faster at higher pressures. In some propellants however, this behaviour is reversed over certain pressure regions in the range 3 - 30 MPa where it can be used as another performance control mechanism. As propellants burn in layers parallel to the original surface, the surface area is an important aspect of design. The charge shape and the pressure will thus determine the burning time. Devices exist having burning times ranging from a few milliseconds to a minute or more.

Outlines of simplified methods of arriving at an approximate design are attached. Some degree of empiricism may be needed however, especially where requirements dictate departures from established practice.

Advice on ignition and cartridge design will not normally fall within the field of ERDE's experience, but ERDE will be happy to redirect design engineers to the appropriate sources of information.

The standards and philosophy of excellence achieved for Defence work are of value for high quality commercial products. ERDE possesses a unique capability to design, manufacture and test the complete range of solid propellants.

For further information write to

Dr S W Bell
Superintendent, Propellants 1 Branch
Explosives Research and
Development Establishment
Waltham Abbey
Essex

Telephone: Lea Valley 713030, Ext 319

ROCKET DEVICES

A rocket motor using a solid propellant usually maintains a constant burning surface area during operation, requires an igniter and a pressure chamber fitted with a nozzle. Examples of civil use of rockets include line throwing devices, signal rockets and meteorological rockets.

Data for Some Typical Solid Rocket Propellants

Operating Pressure MPa (psi)	8.5 (1233)	10 (1450)	20 (2900)	25 (3626)
Restriction ratio K_n	320	200	400	250
Burning rate at +20°C $mm\ s^{-1}$ ($in\ s^{-1}$)	12.5 (0.49)	21 (0.83)	24 (0.94)	40 (1.57)
Density $kg\ m^{-3}$	1580	1640	1600	1640
Specific Impulse ms^{-1} 6.9 to 0.1 MPa	2150	2340	2210	2360
Pressure increase with temperature π_K - % $°C^{-1}$	0.16	0.16	0.07	0.15

The final velocity V of a rocket with a missile mass of M and a propellant mass m with specific impulse I is given approximately by:

$$V = I\left(\frac{m}{m/2 + M}\right) \text{ and the range by } \frac{V^2}{2g}$$

Example

What weight of propellant is required to project a mass of 1 kg a distance of 1 km assuming a specific impulse of 2200 ms^{-1}? (For a range of 1 km $V = 140\ ms^{-1}$)

$$m = \frac{2\ VM}{2I - V} = \frac{280}{4260} = 65.7\text{ g } (2.32\text{ oz})$$

PISTON ACTUATOR DEVICES

Typical gun propellant gas properties

Force Constant nRT (moles of gas × gas constant × temperature)	1000 kJ/kg
Gas temperature	3000K (5432°F)
Operating Pressure	300 MPa (19.42 tons/in^2)

Example

How much propellant is needed to give the equivalent force of a 7 kg sledgehammer falling a height of 2 m?

Energy = mgS = 7 × 9.81 × 2 = 137 joules (101 ft lb)

Delivered force = 500 kJ/kg (assuming 50% efficiency)

Weight for 137 joules = $\frac{137}{500}$ g = 0.27 g (0.0095 oz)

INFLATION DEVICES

Examples of inflation devices include flotation collars, aircraft escape chutes and car crash bags. Comparison with compressed gas is given below.

Typical Properties	Propellant	Carbon Dioxide
Volume of gas produced per kg of solid at ambient	0.84 m^3 (29.4 ft^3)	0.51 m^3 (17.9 ft^3)
Volume of gas produced / Volume of generator	1300	450
Time to complete inflation of 0.46 m^3 (16 ft^3)	milliseconds	seconds
Gas temperature	*1400K (3000°F)	Less than ambient
Operating Pressure of cartridge	**5.2 MPa (750 psi)	5.2 MPa (750 psi)
Gas composition	Carbon dioxide 21 Carbon monoxide 32 Hydrogen 17 Water 18 Nitrogen 12	Carbon dioxide 100

*Gases may be further cooled by passing through a bed of pellets or alternatively entraining air.

**Pressure may be varied to suit operating conditions.

Example

How much propellant is needed to inflate a car crash bag with a volume of 0.0028 m^3 (0.1 ft^3) to a pressure of 0.15 MPa (22 psi) and can this be achieved in less than 15 milliseconds? (NB several small bags used in each car.)

1 kg of propellant gives 0.85 m^3 of gas at 0.1 MPa

Therefore $\frac{0.0028}{0.85} \times \frac{0.15}{0.1}$ kg propellant needed = 0.005 kg or 5.0 g (0.18 oz)

Rate of burning = 25.4 mm s^{-1} Burning thickness for 15 milliseconds = 0.76 mm.

Since propellant can be made 0.76 mm thick the operating time can be met.

The Stability of Nitrate Esters

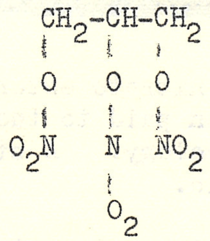
Although several investigations have been made of nitrate ester hydrolysis in ethanolic alakli solutions, little attention has been paid to the behaviour of those esters which are important in propellant technology. A study has now been made of the twenty-two nitrates listed in the Table.

Hydrolysis rates were measured in 90% (v/v) ethanol by titrating the unreacted hydroxide ion with standard sulphuric acid, using phenol red as the indicator. Three types of kinetic behaviour were found: (i), the observed specific rate remained constant, within experimental error, throughout the reaction (methyl, n-heptyl nitrates; ethylene glycol mononitrate); (ii), the second order specific rate increased as the reaction proceeded, due to reaction between hydroxide and the products first formed (ethylene glycol dinitrate, metriol trinitrate, pentaerythritol tetranitrate); (iii), the specific rate decreased as the reaction proceeded. The reason for the latter behaviour is not clear; it was found in only three cases, for triethylene glycol mono- and dinitrates and for diethylene glycol dinitrate, and may reflect the presence of a small amount of faster reacting impurity.

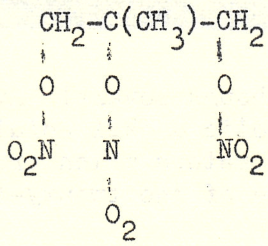
The measure of the stability of an ester is the initial specific rate for the hydrolysis, k_1: subsequent decompositions are important only in determining the overall rate of hydroxyl ion consumption and the final nature of the products. Accordingly, all rate constants were extrapolated to zero time, and the values obtained are listed in the Table. It can be seen that stability increases as the carbon chain length increases, and is further enhanced by the introduction of an α-methyl group. Alkyl substitution in the β-position has a smaller, but similar, effect. This is not the case when nitro, nitrate, or hydroxyl groups are in the β-position: the rate of hydrolysis of propane 1,2-diol dinitrate is 1500 times greater than that of the n-propyl ester. Stability can thus be related to the inductive effect of the substituent and the resulting induced charge on the α-carbon; electron attracting groups decrease stability. Steric effects are more difficult to assess, but replacement of hydrogen by a methyl group decreases hydrolysis rates:

$CH_3-CH_2-CH_2-ONO_2$	and	$CH_3-CH(CH_3)-CH_2-ONO_2$
2.9		1.6

or



and



24,000 69

<u>Table</u>

<u>Initial second order rate constants for hydrolysis[a]</u>

Nitrate	$10^5 k_i$ 30°C	$10^5 k_i$ 60°C		$10^5 k_i$ 30°C	$10^5 k_i$ 60°C
methyl		112	TEGDN		340
2-nitroethyl	1050		1,2-PDN	54	4,650
n-butyl		2.9	1,3-BDN	20	
n-heptyl		2.6	2,3-BDN	2.9	66
Iso-butyl		1.6	metriol TN	69	
EGMN	168	2700	NG	24,000	
EGDN	160		1,3GDN	4,500	66,200
1,3-PDN	58.7	1300	1-GMN	2,150	51,000
1,4-BDN		16.0	2-GMN	920	20,500
1,5-PDN		12.5	NIBGTN	128,000	
DEGDN		340	PETN	34	2,700

[a] $M^{-1} s.^{-1}$; measured at 30±0.1° or 60±0.05°. $[NaOH]_o$ 0.150 - 0.080 *M*, $[RONO_2]_o$ 0.035 - 0.010 *M*.

MN mononitrate, DN dinitrate, TN trinitrate
T(D)EG tri-(di-)ethylene glycol
1,2-(1,3-)P 1,2-(1,3-)propane diol
1,5-P 1,5-pentane diol
B butane diol
NG nitroglycerine
G glycerol
NIBG nitro*iso*butyl glycerol
PETN pentaerythritol tetranite

SPECTROSCOPY SECTION

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INSTRUMENTAL ANALYSIS

While it is unusual for one instrumental method alone to give the complete answer to a problem, the careful use of a number of techniques together may supply the required information. To this end, to support the work of other sections in the branch and the work of the Establishment as a whole, the Spectroscopy Section brings together ultraviolet and infrared spectroscopy, mass spectrometry, and electroanalysis. Besides carrying out routine measurements on a service basis, a number of research problems have been initiated and carried to a successful conclusion within the group itself, and work has also been undertaken for a number of universities. Equipment available includes microcells for very small volumes of sample, accessories for time-rate studies in the ultraviolet and infrared regions, and for attenuated total reflectance measurements. Infrared spectra can be run at temperatures up to 120°C. One important function of the section is to act as a reference library for infrared and mass spectrometry: authenticated spectra of individual compounds or groups of compounds can be supplied on request.

ULTRAVIOLET AND INFRARED SPECTROSCOPY

Ultraviolet and infrared rays, although invisible to the eye, are absorbed by materials in much the same way as visible light. The infrared frequencies that are absorbed by a molecule depend on the type of atoms and their particular arrangement. Just as each kind of molecule is different, so each substance has a different infrared spectrum which can be used to identify or 'fingerprint' unknown compounds. The absorption of ultraviolet radiation is a little less specific, but still serves to indicate the class of compound to which the unknown belongs. While u.v. spectroscopy is limited to liquids or to soluble solids, infrared can be applied to gases, liquids, and solids, even to the investigation of surface coatings on paper or plastic sheets. Both methods can be used for quantitative analysis of mixtures.

Some recent examples of work are the examination of asbestos fibres, brushing thinners, adhesives, and irradiated resins; the analysis of chlorosilane mixtures; the estimation of antioxidants.

MASS SPECTROMETRY

Mass spectrometry differs from ultraviolet or infrared spectroscopy in that it is a destructive method: although only very small samples are required, these are subjected to bombardment with high energy electrons, fragmented, and the ion fragments separated in a magnetic field according to their mass

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to charge ratio. Any material can be examined, as long as its vapour pressure (if necessary, under mild heating) is reasonably high.

Mass spectrometry will give three different types of information: it can be used to identify and estimate components of a mixture, it can be used to establish the molecular formula and possibly the structure of a pure unknown, and it can be used to locate isotopically labelled sites in compounds. Recent projects have been the study of gases taken from different zones of flames, the analysis of fractions trapped from gas chromatograph columns, molecular weight determination of styrene polymers, and estimation of nitrogen-15 labelling in N-nitroso p-methyl aniline.

ELECTROANALYSIS

If the electronic content of a compound can be altered, either increased or decreased, it becomes possible to analyse its solutions by measuring the effect of an electric current. The process is called electroanalysis, and it may either involve plating out on a platinium electrode or drop of mercury, or measuring electrical current or voltage as a function of time. Determinations of styrene monomer in polystyrene resins, cadmium and titanium in steels and aluminium, phenols in waste water, and unsaturation in natural rubbers are some of the possibilities.

Enquiries should be addressed to:

Dr R. T. M. Fraser
Analysis and
Ingredients Branch
(Ext 490)

F/30/1

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Fibre-Filled Thermoplastics

Thermoplastics are a cheap easily fabricated class of materials. However they have insufficient strength and rigidity for many applications. Fibrous fillers, in particular graded asbestos fibre which is cheap, impart increased strength and rigidity to thermoplastics thus making these materials more suitable for engineering applications and for use in building.

Normal asbestos consists of fibrous material of many sizes with varying quantities of rock and dust. Grading removes the rock and fine material leaving an asbestos with a large length/diameter ratio. It is this large L/D ratio which gives the graded asbestos superior reinforcing properties.

Examples of the effect of incorporating graded asbestos into thermoplastics are given below.

Plastic	Specific Gravity	Tensile Strength psi	Flexural Modulus 10^6 psi	Specific Modulus 10^6 psi
Polypropylene	0.91	4,400	0.15	0.17
+40% asbestos	1.29	6,800	0.77	0.60
Toughened polystyrene	1.04	3,500	0.27	0.26
+30% asbestos	1.27	9,500	1.60	1.26
ABS	1.04	5,300	0.22	0.21
+30% asbestos	1.27	13,900	1.13	0.89
Nylon 6	1.14	11,000	0.34	0.30
+30% asbestos	1.38	19,000	1.09	0.79

The results quoted are for non-surface treated asbestos fibre. Glass fibres are normally surface treated to improve the properties of glass-filled thermoplastics. Research is at present in progress on the treatment of asbestos fibres and it is hoped a similar improvement in properties can be obtained.

Oriented Reinforced Thermosets

Asbestos, chopped carbon fibre, and whiskers are used in these composites, with epoxy or phenol formaldehyde as the base resin. Best mechanical properties are attained at the high volume packings gained by orienting the fibres, which should preferably be straight and stiff. Most pressings have been of particular chrysotile asbestos about 2 mm. long and 50 microns diameter.

Fibre in Composite	Fibre Content vol per cent	Specific Gravity	Flexural Strength psi	Tensile Modulus 10^6 psi
Asbestos (alginate)	61	1.99	116,000	14.4
Asbestos (aligned sheet)	54	1.95	103,000	11.0
Chopped carbon	64	1.65	90,000	22.5
Silicon nitride whiskers	39	1.87	111,000	13.0
Silicon carbide whiskers	39	1.9	220,000	25

There are two wet processes for aligning the fibres, after cleaning and sieving into lengths. In one they are extruded in a filament of alginic acid carrier which is wound into a mat (Exhibit 13), and then burned-off to remove the alginic acid. In the other they are extruded in viscous suspension direct on to a moving filter and form an aligned sheet, which can be handled (Exhibit 13). The mat or sheet is then built up to the shape required, sheets can be crossed or laid in the direction strength is required, resin is added and the article moulded in a heated press or autoclave to the finished form. Up to 1000 psi pressure is necessary to get a high enough packing density of fibre. The sheeting process is also being developed to produce continuous rolls of tape and composite filament which can be used for filament-winding.

Applications can include rocket motor components such as cases, exhaust tubes and nozzles, and airframe structures, where light weight but high strength, high stiffness and perhaps ablation resistance are required; and some examples are shown. Asbestos gives better stiffness and fatigue strength than fibre-glass, and is expected to justify itself in these applications. If the full properties of silicon carbide whiskers can be developed in resin composites, they would give the highest properties, at correspondingly greater cost, for the most exacting applications.

Whiskers in Light Alloys

Reinforced Metal or Reinforced Plastic?

Fibre reinforced plastics have recently made outstanding advances as readily fabricated materials with excellent but highly directional properties. However, structures are seldom stressed uniquely in one direction and designers are accustomed and equipped to use metallic materials which have virtually isotropic properties. By elaborate laboratory methods, research workers have already attained remarkable properties from simple metal matrices reinforced with whiskers. Now, using near-conventional metal forming techniques and tools, aluminium alloys reinforced with whiskers of silicon carbide show good prospects of development into hard-wearing, highly efficient materials.

Here the fibre is boosting an already efficient host matrix. Typically, a system of 18% of oriented whiskers in the alloy, which produces twice its normal stiffness in one direction, could only be replaced in overall stiffness by a distribution of some 42% of longer whiskers in a resin. For unidirectional properties alone, the position is reversed and the lighter reinforced plastic should be better, weight for weight.

Fabrication

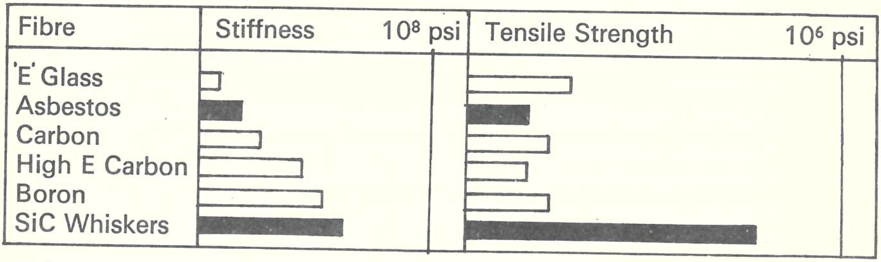
A number of fabrication routes and host metals have been studied, from which has emerged the present "pressure-casting" process. For carbide in aluminium this produces void-free composites with the fibres intact and completely stable. Billets and shapes have been made in this way and are subsequently hot worked and machined as necessary. For a balance in the composite of good elastic behaviour, reasonable impact strength and good high temperature performance, one of the "Concorde" alloys, RR 58, has been selected for development.

Various shapes, components and properties of this and other reinforced metals are displayed on the stand.

What Fibres and What For?

Higher strength—higher stiffness (more *usable* strength for the designer)—higher working temperature. These are all combined in lightweight materials of good and sometimes outstanding toughness (resistance to cracking). Degree of improvement naturally depends on the amount and quality of the fibre used, and its distribution in the matrix or base material. Very high values are possible by unidirectional arrangement, when the maximum proportion of fibre may be packed in.

Which are the best fibres to use?—Any fibre used must be of low density, otherwise much of the advantage is lost, and fortunately the strongest fibres are among the lightest.



Asbestos, the cheapest fibre, gives a good engineering balance of stiffness and strength. The best experimental fibres, silicon carbide whiskers, are the subject of an intensive research project aimed at large scale, cheap production (Exhibit 24). This involves chemistry and physics of crystal growth at high temperatures and a good deal of novel engineering. The raw materials used are cheap, and when consistent nucleation of fibrous crystals is achieved, this fibre should be available in large quantities.

Both asbestos and SiC whiskers are discontinuous fibres. This is no handicap in random composites and leads to ready shaping of the product. Certain critical structures require fully aligned fibres and techniques have been developed for doing this (Exhibit 13).

The load is passed from one fibre to another through the surrounding metal or plastic and through the surface of each fibre. To function efficiently therefore, the fibres must be long in relation to their thickness and, in practice, the longest fibres are used in thermosetting resins, and shorter fibres in light alloys and thermoplastics. The separation into grades is made quickly on special plant. (Exhibit 16 and 25).

Thus with the best fibres, suitably graded and aligned where necessary, the stage is set for making the best composites.

Enquiries should be addressed to:

Superintendents of Materials 1 or 2
or Superintendent of Chemical Engineering
ERDE
Waltham Abbey, Essex
Tel: Waltham Cross 23688
(Extn. 390, 403 or 374)

Rubbers and Plastics

Chemistry
Physics
Applications
Tropical Exposure

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Polymer Chemistry

Rubbers and plastics are liable to undergo slow changes which may eventually make the finished article useless. These are due to the individual or combined effects of, for example, heat, light, solvents or fuels. The main aim of the Polymer Chemistry Section is to disentangle these effects by laboratory studies and by correlation of these with the results from exposures at the Joint Tropical Research Unit.

Once the breakdown route is understood, it is frequently possible to modify the polymer to stop the process, or to suggest an alternative material which is more resistant to the particular environment. Attempts are therefore being made to prepare polymers with pre-determined properties by controlling precisely their chemical structures. In addition, specific reactive groups are being introduced into some polymers to try to improve their adhesion to reinforcing fillers such as asbestos.

The group is well equipped for the analysis and characterisation of rubbers and plastics e.g. infrared, ultraviolet and nuclear magnetic resonance spectroscopy, gas chromatography, osmometry and light scattering, and microscope examination. All of these are available for extramural use.

Information from Dr. D. H. Richards (extension 239)

Polymer Applications and Development

Plastics and rubbers are being used to an ever increasing extent in military equipment. The Applications and Development Section acts as advisory centre to the Army Department Design Establishments for this purpose and to provide trouble shooting for ad hoc problems as they arise.

The section is well equipped with processing equipment and as new materials become available from the manufacturer they are studied extensively for their properties, both mechanical and in processing, and also for general ageing and weathering behaviour.

Considerable progress is being made in the introduction of asbestos and other fibres into plastics in order to improve their mechanical properties. For example the breaking strengths can be increased from 100 to 200% and the flex modulus by up to 500% by the correct incorporation of about 30% by weight of asbestos.

Proofed fabrics are another topic of interest and much work has been done on flexible fuel tanks with this material.

Though the efforts of the section have been for defence needs, the experience gained is of immediate applicability to civil use and enquiries of this nature are already being dealt with.

Information from Dr. B. L. Hollingsworth
(extension 451)

Polymer Physics

Cheapness and ease of fabrication make plastics and rubbers of great interest as potential engineering materials. However for their use in load bearing structures it is most important to characterize their mechanical behaviour since this differs in many ways from that of metals. For example, it is necessary to know the various moduli of elasticity and ultimate strengths as a function of the *rate* of load application. Creep properties differ from those of metals. The materials can also show differences depending on the temperature at which they are being used.

The Polymer Physics Section is equipped to measure these properties over wide ranges. New instruments whose design has been dictated by cheapness and simplicity have also been devised to extend the measurements. All of these facilities are available for civil use.

In addition, theoretical studies are being made to relate the basic properties of the polymers to their molecular structures.

Information from Dr. J. Roberts (extension 212)

The chemistry, physics, and applications sections together with the Joint Tropical Research cover a wide range of experience of plastics and rubbers and have excellent experimental facilities. All of these are available for civil use and enquiries are welcome.

Joint Tropical Research Unit

Hitherto, it has been impossible to reproduce exactly in the laboratory the deterioration of plastics and rubbers which occurs under normal use. It is therefore always necessary to supplement laboratory work with outdoor exposure trials. Since the disruptive effects of temperature, sunlight and atmospheric oxygen are very much more pronounced in the Tropics, Australia and the U.K. set up in 1962 a jointly operated unit at Innisfail, N. Queensland.

The unit has four exposure sites for samples, viz:

	Mean Temperature	Mean Humidity	Daily Mean Sunshine
(i) Hot, wet, jungle (ii) Hot, wet, open (iii) Marine	74°F	about 85	7 hours
(iv) Hot, dry, desert	78°F	39	9-10 hours

After exposure, samples are examined and tested initially, in the J.T.R.U. laboratory before return to U.K. or Australia for more detailed study.

Hitherto, only materials for military use have been examined but the facilities are now available for suitable civilian trials and a number of these have commenced.

U.K. trials are administered by Materials 1 and information can be obtained from Mr. D. J. Evans (extension 239).

Apply to the section concerned or else directly to :-

Dr R. L. Williams, Superintendent Materials 1 Branch,
E.R.D.E., Waltham Abbey, Essex.
Tel. Waltham Cross 23688 (Ext. 390)