

Mintech ERDE

NEW FIBRE-FILLED THERMOPLASTICS

Thermoplastics are a cheap, easily fabricated class of materials. However, for many of the more exacting applications they have insufficient strength and rigidity. This has been partly overcome by the rapid growth of the use of glass-filled thermoplastics, particularly in the past five years. Currently, a range of thermoplastics containing fibrous fillers as alternatives to glass-fibre are under investigation, to increase the field of utilisation of fibre-filled thermoplastics.

In this laboratory, the incorporation of graded asbestos fibres, silicon nitride and silicon carbide 'whiskers', potassium titanate 'whiskers', and chopped carbon fibre into a wide range of thermoplastics, from polyethylene to nylon, has been in hand since 1966. Graded asbestos-fibre reinforced thermoplastics have shown the greatest promise, and as the fibre is cheap and readily available these materials should prove competitors to the comparable glass-filled grades.

The results have shown that the length to diameter ratio of the fibres is of importance in determining the degree of reinforcement obtained. Another factor is the ability of the resin to wet the fibre and the associated resin to fibre bond strength.

Collected below are some of the tensile and flexural results for commonly used materials filled with graded asbestos fibre. Tensile strengths up to 24,000 p.s.i. and moduli up to 1.6×10^6 p.s.i. have been obtained with materials capable of being processed on conventional screw injection moulding machines. Higher moduli up to 6×10^6 p.s.i. can be obtained with styrene-based materials but these composites are difficult to mould except by the use of ram injection moulding machines or by compression moulding.

Polymer	S.G. g/cc	Tensile Strength p.s.i.	Flexural Modulus p.s.i. x 10^6	Price of Polymer Pence/lb
Polypropylene	0.91	4,400	0.15	39
+ 40% asbestos	1.24	6,800	0.77	
Toughened polystyrene	1.04	3,500	0.27	19
+ 40% asbestos	1.36	9,500	2.02	
ABS	1.04	5,300	0.22	40
+ 30% asbestos	1.27	13,900	1.13	
Polycarbonate	1.22	9,500	0.25	72
+ 30% asbestos	1.42	16,000	1.46	
Nylon 6	1.14	11,000	0.3	72
+ 30% asbestos	1.48	19,000	1.0	
Nylon 66	1.12	11,000	0.3	72
+ 30% asbestos	1.33	19,000	1.3	
glass-filled	1.33	18-24,000	0.8-1.2	78

On display are asbestos filled nylon 6, nylon 66, polypropylene, toughened polystyrene and ABS together with some of the newer commercial fibre-filled materials.

Requests for samples and for further information should be addressed to:

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D/21/2

IMPROVED CONDUCTING RUBBERS

Rubbers (and plastics) are generally regarded as electrical insulators, but can be converted into conductors by the addition of suitable fillers such as special carbon blacks.

The problem of development of electrostatic charges on rubbers and plastics has become more acute in recent years due to the widespread use of these materials in conveyor belts in coal mines and the paper industry, tyres in the car industry, etc.

At the request of another Establishment, the examination of commercially available anti-static and conducting rubbers used in solid tyres, and the preparation of improved conducting rubbers, was undertaken.

A natural rubber compound containing 70 parts per hundred of rubber of acetylene black has been used as a standard for comparison with other rubbers containing the same loading of acetylene black. At a contact pressure of 15 p.s.i., the following comparative electrical resistances were obtained:

Commercial Rubber 1 (standard solid tyre rubber)	> 10 ⁶	ohms
Medium nitrile rubber	530	"
Hypalon	360	"
Commercial Rubber 2 (commercial conducting rubber)	90	"
Ethylene/propylene/diene terpolymer	35	"
Neoprene	30	"
Epichlorhydrin rubber	14	"
Butyl	13	"
E.R.D.E. natural rubber standard L7/70	6	"
Acrylate	4	"
cis-1,4-poly Butadiene	3	"
cis-poly Isoprene	2.5	"
Fluorinated rubber	0.6	"
poly Sulphide	0.5	"

Later work with two special furnace blacks gave lower resistivities than acetylene black with similar loadings.

The incorporation of fibrous fillers such as carbon wool and aluminium staple gave rubbers of poor conductivity, a natural rubber compound containing 100 p.p.h.r. aluminium staple having a resistance of greater than 10⁶ ohms.

As part of the programme, the effect of repeated flexing under compression or under tension has been studied. Even after flexing for the equivalent of running a tyre for 1,000 miles, the resistance of L7/70 was still well within the conducting range. The development of static charges during the impact of steel on conducting rubber, conducting rubber on steel, and

conducting rubber on conducting rubber has also been examined. A small charge is developed on impact, and a larger charge is developed on separation of the impacted surfaces. The size of the charge developed on separation is a function of the area of contact and the speed of separation. Whilst quite large charges can be developed at each separation, a further impact causes leakage, and when using a rubber of high conductivity, the building up of charge does not appear possible.

Dynamic Properties

The abrasion resistance and heat build-up on flexing of the E.R.D.E. conducting rubber L7/70 has been measured using the Akron abrader and the Goodrich flexometer. The results have been compared with the two commercial rubbers, and also with a standard natural rubber pneumatic tyre tread compound. The results obtained were:

	Natural rubber tyre tread compound	Commercial Rubber 1	Commercial Rubber 2	E.R.D.E. L7/70
<u>Abrasion loss</u>				
c.c./1,000 revs. 15° angle of slip.	0.20	0.37	0.29	0.20
c.c./1,000 revs. 25° angle of slip.	0.78	1.23	1.26	1.10
<u>Heat build up °C</u>				
240 p.s.i. 0.25" stroke	66	87	98	49

The results show that the E.R.D.E. developed conducting rubber L7/70 has better abrasion resistance and a lower heat build-up than either of the commercially available solid tyre compounds, and is comparable in properties with standard pneumatic tyre tread compound. From this, and from the work on electrical conductivity, it appears that L7/70 will make satisfactory electrically conducting solid tyres, and a full scale evaluation in vehicle solid tyres is in hand.

Further enquiries should be addressed to:

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D/21/3

Mintech ERDE

WHISKERS FOR STRENGTH

The limits of improvement for the conventional engineering metals are already in sight. Composite materials, that is materials composed of non-metallic fibres bonded together with a resin or a metal, offer a way past this limitation because of the exceptional mechanical properties of certain thin fibres.

THE NEED FOR HIGH STIFFNESS AS WELL AS HIGH STRENGTH

The most familiar of these fibrous composites is 'fibreglass' which, for its weight, has outstanding tensile strength and excellent toughness, and which has already become popular for many applications in aircraft, boats and cars. However, the Young's Modulus of fibreglass is low, owing to the relatively low elastic modulus of glass itself and this limits the fields of structural application for fibreglass, especially for high-efficiency light-weight structures such as occur in aircraft. This is because the design of many important structures is controlled as much by the allowable deflections under load, as by the fracture strength and, moreover, compression members such as columns and panels generally collapse by buckling elastically. Furthermore, engineering involving large strains presents other formidable difficulties.

All the conventional metals and their alloys have very much the same specific strength and stiffness (i. e. strength or Young's Modulus divided by density) at room temperature, and so for these reasons there have been over the past few years widespread attempts to produce reinforcing fibres of higher Young's Modulus than glass. There are a number of ways in which this can be achieved but all of them involve preparing fibres from rather intractable solids, frequently those which are covalently bonded.

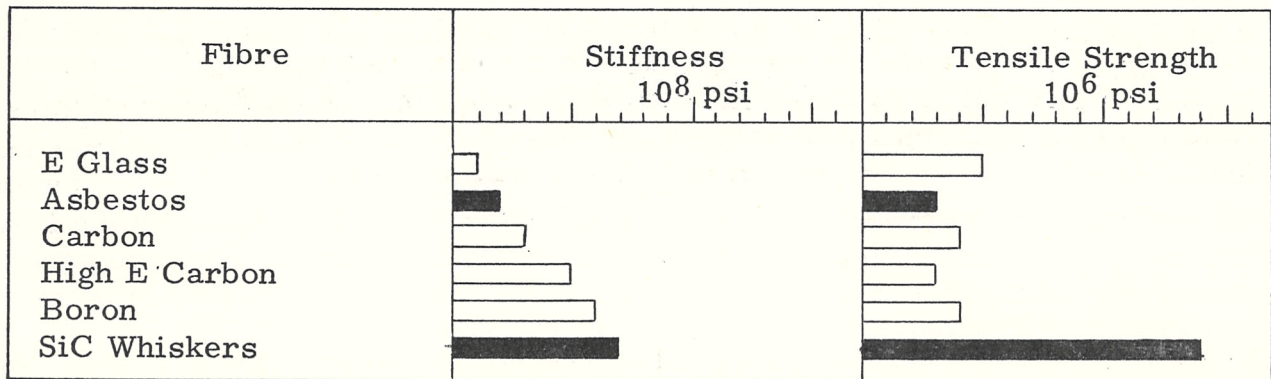
Successful continuous or partly continuous fibres have been produced elsewhere from graphitic carbon and from boron. These fibres show excellent stiffness although their tensile strength, as yet, is not outstanding. While for some purposes it is useful to have continuous reinforcing fibre, it is in many cases the practice, as in the case of glass fibres, to chop the filament into short lengths before incorporating it in a composite - so that the advantage of continuity may be illusory.

WHISKERS COMBINE HIGH STRENGTH WITH HIGH YOUNG'S MODULI

It is possible to grow whiskers (thin needle crystals) of most of the stiffest of the ceramic substances and these whiskers, though relatively short, combine



high Young's Modulus with the highest of all tensile strengths, indeed the strength of some whiskers may approximate to the theoretical value. As compared with continuous fibres, there is necessarily some loss of efficiency associated with getting the loads in and out of the ends of a short fibre such as a whisker, but this loss may be more than offset by the fact that a whisker may have from four to eight times the tensile strength of a continuous fibre. The high strength of these whiskers is due primarily to their exceptionally smooth surfaces which are also very hard and therefore difficult to damage. These whiskers retain their strength well and are found to be much more difficult to weaken by surface abrasion in processing than, say, glass fibres.



THE COST OF PRODUCING WHISKERS

To attain the highest strengths and stiffnesses it is necessary, however, to make whiskers from very intractable substances such as silicon carbide or silicon nitride. In the past such whiskers had only been produced in tiny quantities as laboratory curiosities. What we have been doing at ERDE is to develop the production of such whiskers upon a kilogram scale and at a reasonable cost. To this end the large experimental furnaces (2000 litres capacity) known as bran-tubs (E24 Building L. 168) have been designed and built. Although far from the optimum commercial design, these furnaces do produce useful quantities of good quality whiskers at a cost which is acceptable at this stage for experimental purposes. With the knowledge which we now have it is possible to improve and cheapen the production of whiskers very substantially.

The earlier experiments were made with silicon nitride (Si_3N_4) and whiskers of good quality can be made of this compound for around £100 per pound. Silicon carbide, (SiC) though technically more difficult to grow, provides whiskers of considerably better mechanical properties and promises to be much cheaper to produce. A process has been invented and is now in use for the production of silicon carbide whiskers in similar plant. From the known growth rates and the known cost of operating such plant it appears that the cost in the near future of SiC whisker wool should be between £5 and £10 per pound, while shorter whiskers, suitable perhaps for reinforcing thermoplastics and metals, may cost between 10/0 and £1 per pound because



of the higher bulk density at which they grow and the reduced time required to grow them. The chemical thermodynamics of the whisker forming reaction are now well understood and our current research covers two factors which strongly influence the final cost of whiskers. These are the controllable nucleation of whisker growth and the most efficient usage of a given reaction zone.

REFINING THE WHISKERS

With the present degree of control over growth, the whiskers are produced as a tangled mass of wool, containing some 50% of non-fibrous residue. To render them effective for reinforcement they are first cleaned and graded in water. Equipment consists of a single dispersion stage, a hydrocyclone network designed for removal of residues and for diameter grading, followed by rotary screen machines developed for length grading. A recent advance in rotary screen machines now enables whiskers to be treated at 5 lb/hr per foot of track, making production refining operations feasible at a few shillings per pound. This equipment may be seen in operation (Exhibit E 25).

WHISKERS COMPOSITES

Refined whiskers are readily aligned as needed for the production of mat, tape and cloth (Exhibit C15) and incorporated in plastics or metals. There are two main areas, reinforced light alloys and reinforced thermosetting plastics. The latter offer much the same stiffness as those based on boron or carbon fibres, but with inherently more fibre strength in hand for further development. Reinforcement of light alloys is accomplished by a novel pressure-casting process, based on the proven stability of silicon carbide whiskers in molten aluminium alloys. Void-free high modulus metal composites are thus obtained and these may be hot worked or machined to shape. Details are given in Exhibit E25 and also on the joint composites display (Exhibit C13).

Enquiries should be addressed to

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E/24-26/1



Mintech ERDE

CRYSTALLOGRAPHIC SECTION

The methods of chemical crystallography are applied to the problems of the Establishment. The scope of the problems studied and the chief methods employed in their solution may be outlined as follows:

	(Characterization of chemical compounds)	
X-ray diffraction	(Identification of chemical compounds) Optical methods,
applied to single	(Phase analysis of mixtures) mostly using
crystals and	(Polymorphism) the polarizing
powders	(Crystal orientation) microscope
	(Crystal structure analysis)

These methods are currently applied in work on initiatory explosives, explosives chemistry, propellants and whisker crystals for use in high-strength materials. The problems are generally of a chemical nature, and concerned with the search for useful materials, the establishment of conditions of preparation and the study of degradation in use. Much of the work on whiskers is concerned with the determination of crystal orientation, since the physical properties of crystals vary with direction.

Equipment available in the Section includes three X-ray generators, one being a high-power rotating anode set, X-ray powder cameras, goniometers (Weissenberg, oscillating crystal and optical), an X-ray powder diffractometer and polarizing microscope with heating stage; the requisite ancillary facilities for preparation and treatment of specimens by chemical and physical means, including the measurement of crystal density, are also provided. An automatic four-circle diffractometer is used for collection of the data required for studies of the atomic and molecular structure of crystals.

Much of the work on phase analysis and identification is accomplished by the use of X-ray powder methods, but single crystal studies are used in problems involving characterization, polymorphism and crystal orientation. The solution of some problems involves a determination of the detailed atomic arrangement in a crystal, in order to understand the chemistry involved, and in these cases also X-ray single crystal methods are generally employed.

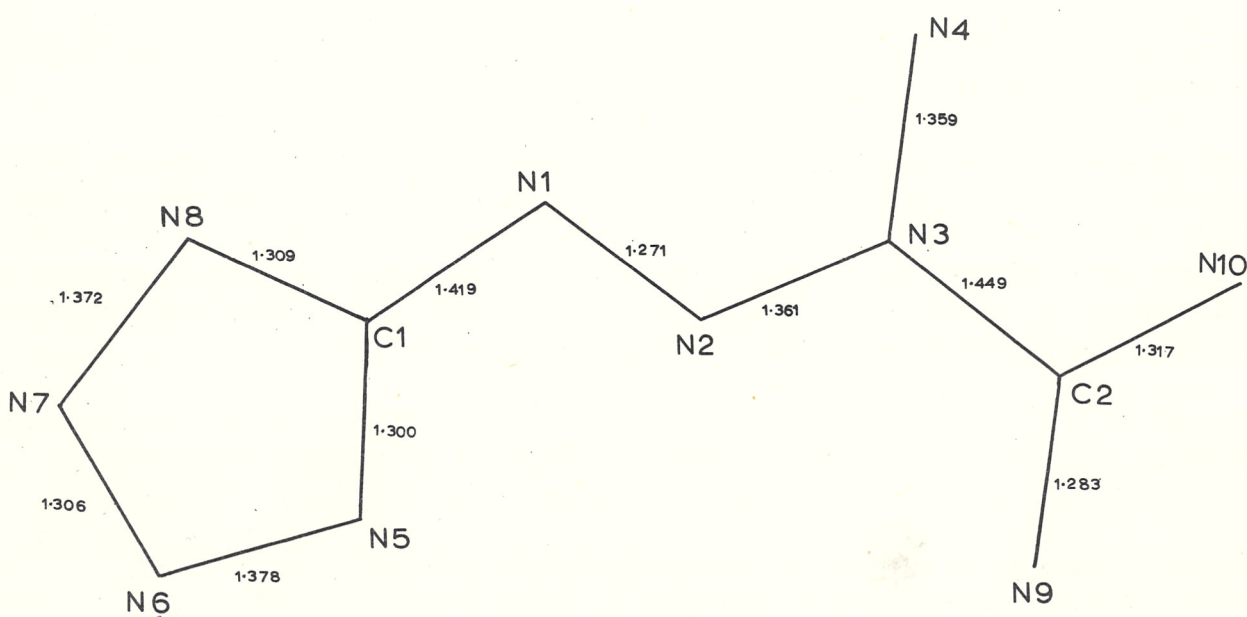
Most of the problems presented to the section originate within the Establishment, but some work is carried out for outside bodies, e. g. other government establishments and commercial firms engaged on government contracts.

A typical example of the use of crystallographic methods in support of Establishment programmes is provided by work in connection with the



preparation of new and improved initiatory explosives. Lead salts, of e. g. polynitrophenols, are used for this purpose, and it is necessary to prepare pure and well-defined chemical individuals in order to secure the reproducible explosive performance which is desired. Arbitrarily chosen preparative conditions are liable to result in mixtures of uncertain chemical and physical properties, and without knowledge of what phases are present it is often not possible correctly to interpret the results of chemical analyses. Correlation of the results of crystallographic examination with those of chemical analysis of products enables suitable conditions of preparation for the desired compounds to be established; tests of explosive functioning and other properties then enable a choice to be made of the most suitable compound for the particular application. The foregoing description of the process involved is somewhat idealized, but illustrates the principles used to guide operations. In the case of one such system which was investigated, 14 phases were identified, comprising anhydrous and hydrated forms of the normal salt, basic compounds and polymorphs of these.

In the field of crystal structure analysis, work is in progress on the primary explosive tetrazene (tetracene), $C_2H_8N_{10}O$; it exists in two polymorphic forms, and there has been continuing uncertainty regarding its chemical structure. It forms a hydrobromide $C_2H_7N_{10}Br$, whose crystal structure has been solved and refined by 3-D methods; the results for the organic ion may be represented as follows:



Dimensions in Angstroms



This result is not in agreement with previously accepted structures for tetrazene. The crystal structures of the two polymorphs of tetrazene itself are at present under investigation; it is expected that the results will show the relationships between these two forms and will reveal the situation and role of the 'water' molecule in tetrazene. As a preliminary to this stage of the investigation, it was necessary to evolve a procedure for the growth of suitable crystals of the centrosymmetrical A-form, free from intergrowths of the non-centrosymmetrical B-form.

Enquiries should be addressed to:

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F/31/1




Mintech ERDE

THE ADVANCED EXPLOSIVES RESEARCH SECTION: REMOTELY CONTROLLED PROCESSING FACILITY

The Advanced Explosives Research Section was formed in 1964 specifically to pioneer new explosive systems (both solid propellant and high explosive) of increased performance, and to develop equipment and techniques suitable for working with unfamiliar and possibly hazardous materials. Initial emphasis is on high explosives for use in sophisticated weapon warheads, but work will later move towards development of solid propellants suitable for specialized guided weapons applications, and for use in extending the range and/or payload of satellite launchers or similar vehicles. It must be remembered that exploration of new explosives is a long and often uncertain operation; if improved compositions are to be incorporated into future designs (either warheads or rocket motors) then the explosives chemist must be several years ahead of the designer. Otherwise by the time the requirement becomes firm it is too late to meet it! Equally, development of facilities and techniques must be ahead of the explosives programme. This associated work on equipment and techniques has its own intrinsic value; experience gained here will be available to all those concerned with bringing greater safety to explosives operations.

The explosives programme is concerned with composite explosives, in which discrete particles of oxidizers and fuels are suspended in binders of widely differing properties. Such composites have important advantages over homogeneous explosives in the search for high performance. The physical separation of oxidizers and fuels makes it possible to compound high energy compositions without the need to pack the energy into single molecules, which would, in consequence, become unstable and hypersensitive. At the same time it becomes possible to control the rates of energy release, both in combustion and detonation, by variation of the physical as well as the chemical characteristics of the ingredients.

The highest performance systems presently available all derive their performance from the energetic reaction between aluminium and ammonium perchlorate, and there is little prospect of further improvement using accepted well-tried materials. In recent years, however, many relatively unfamiliar replacements have been put forward for oxidizers, fuels and binders, which do offer higher performance at a cost. Part of this cost is that all possess characteristics which make them difficult or hazardous to process. These characteristics range from inherent sensitiveness, to gross incompatibility with other candidate materials and trace impurities, or with moisture or minor contaminants which may arise from equipment or from the atmosphere. In time, such foibles can be understood and controlled, but it is desirable to be 

able to assess explosive and ballistic properties relatively quickly, before entering upon a prolonged and detailed examination of all the factors which might influence sensitiveness and stability.

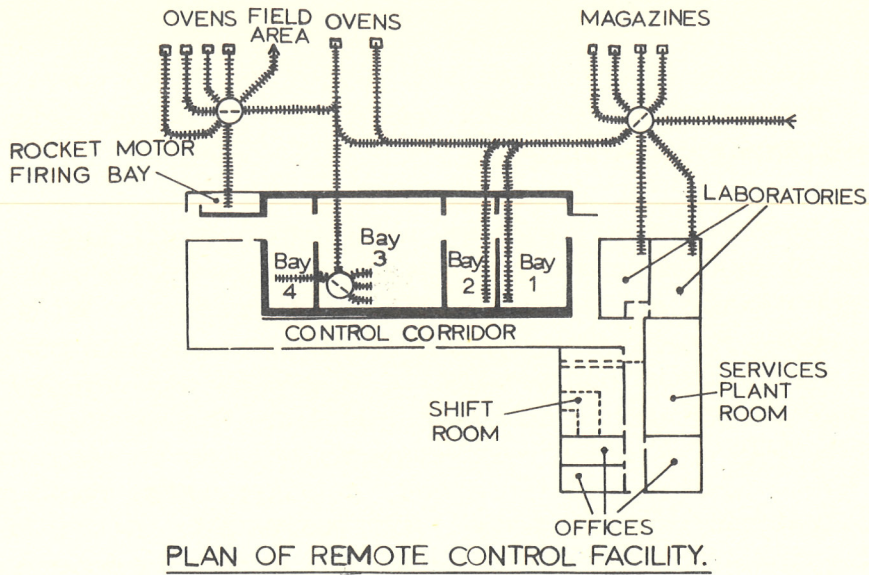
It has therefore been necessary to erect a facility in which high quality charges needed for assessment can be produced in complete safety, regardless of any waywardness which the materials may exhibit. With the Advanced Explosives Remote Processing Facility we are in a position to do this for a wide range of new compounds, in quantities equivalent to 15 lbs of TNT. Here ingredients can be stored in isolation, transported to processing areas, and accurately dispensed, and compositions can be vacuum mixed and moulded, heat-cured in isolated ovens, and inspected, radiographed, assembled into test rounds, and fired. The entire sequence is remotely controlled and operators are fully protected and separated from the explosives at all stages. All process operations can be carried out under controlled atmospheres, and samples taken from the process line can be brought into armoured work-cupboards in air-conditioned laboratories where small scale compatibility, sensitiveness and process control work can be carried out.

The facility (see diagram) is built around a massive reinforced concrete emplacement, in which explosives operations are carried out. Control, laboratory, and accommodation areas are wrapped around the stronger front side of this emplacement, and magazines, curing ovens, and test firing sites are situated beyond a concrete apron at the rear. Transfer of hazardous materials between storage, processing and test points is by a 5-inch gauge electric railway. Services include plenum heating, dry air (35 RH and 2RH), compressed air, vacuum and emergency power generation. Manipulators used at various stages include simple tong, master-slave extended reach, and robot types, but dispensing, mixing and filling are carried out by a fully-mechanized remotely-operated unit (see diagram) designed and developed within the Establishment, and housed in a controlled-atmosphere chamber. This will process a wide range of viscous slurries, and incorporates many features designed for the safe handling of sensitive ingredients and mixes.

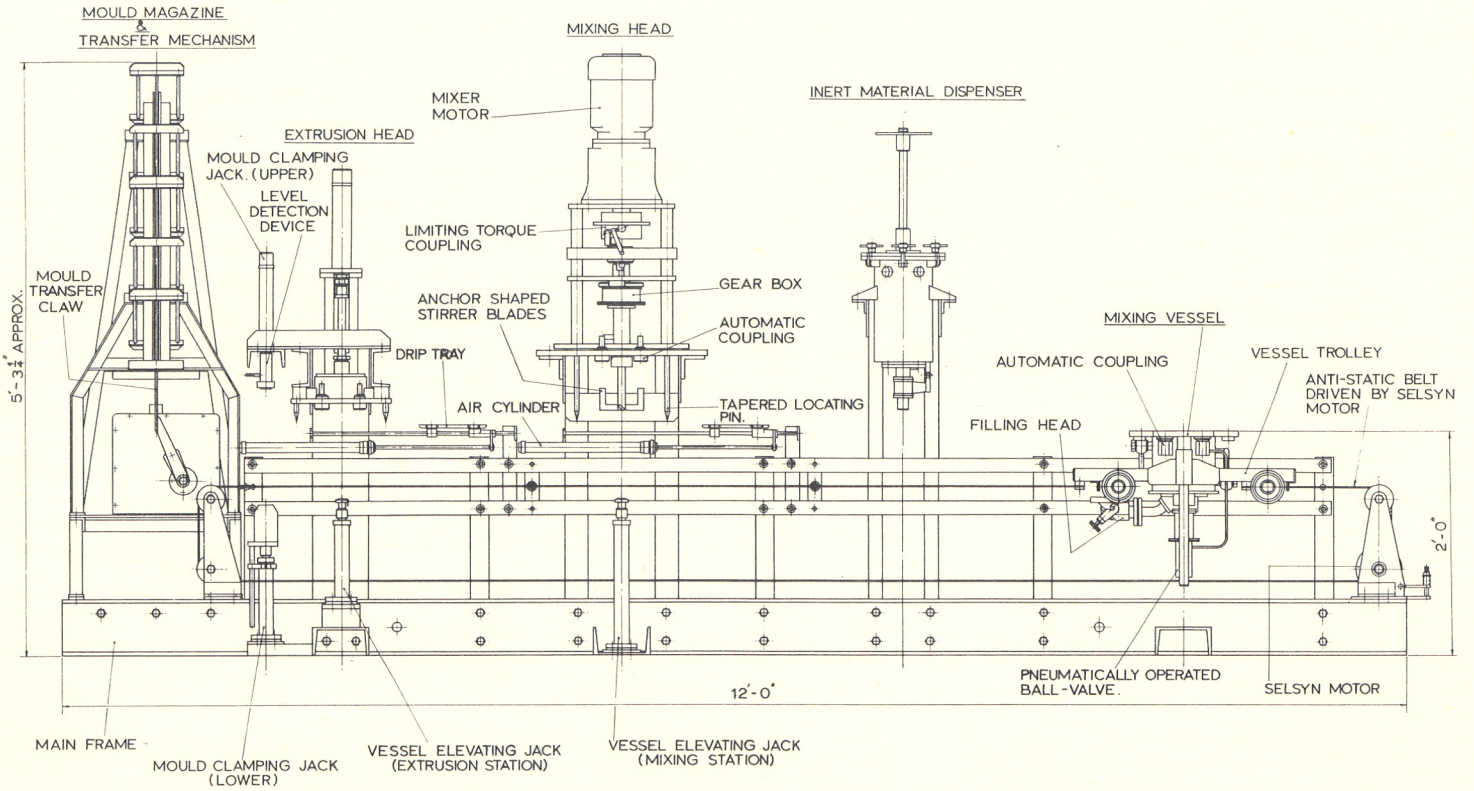
Enquiries should be addressed to:

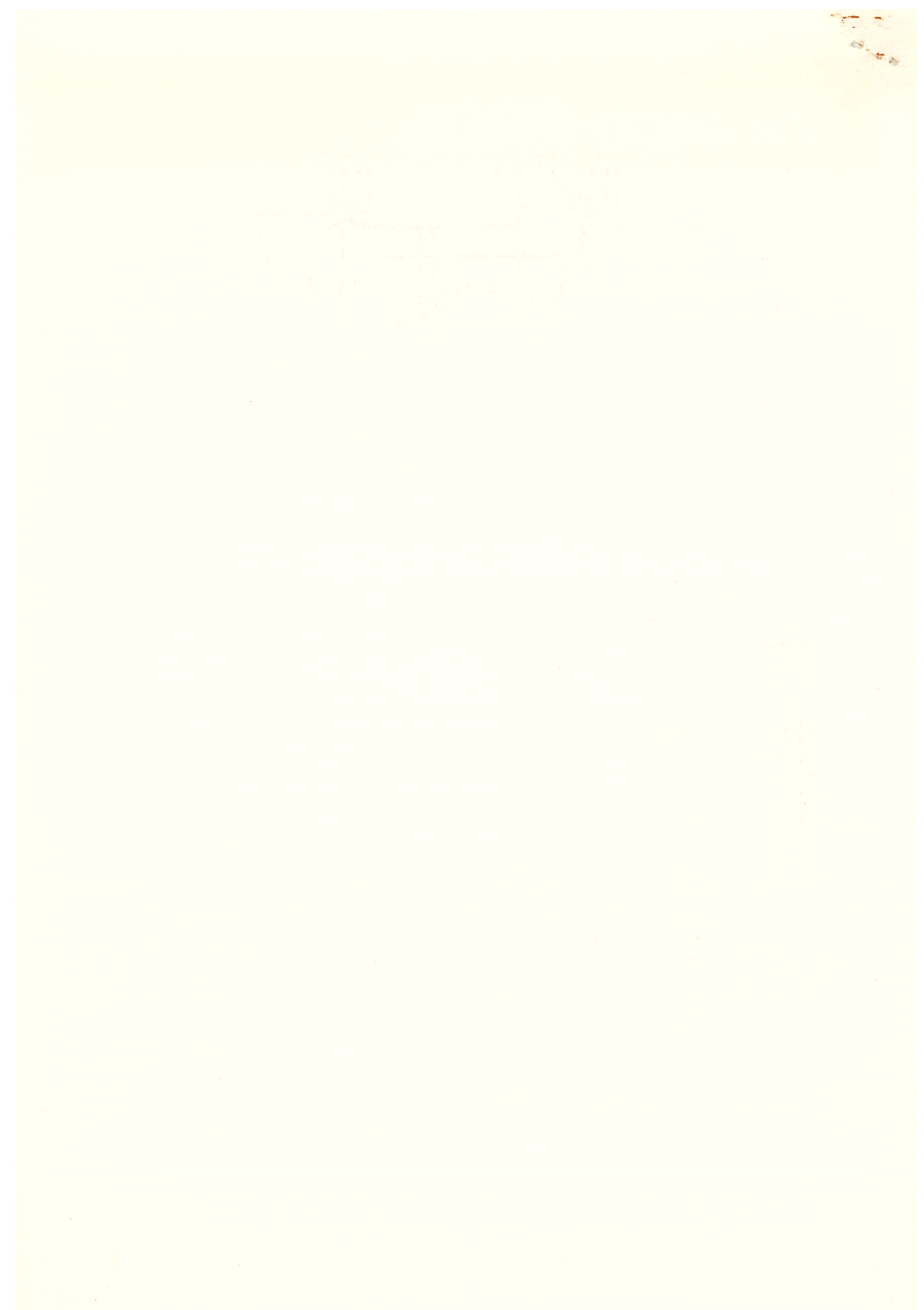
Dr R. Campbell
Explosives Branch
(Ext 571)

C/18/1



E.R.D.E. 24/M/65





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RESEARCH AND DEVELOPMENT OF INITIATING EXPLOSIVES

Initiators or primary explosives are so called because they are in the first stage of an explosive train. They must respond unflinchingly to the stimulus of a very small amount of energy and yet must be safe to manufacture and load and be sufficiently stable and compatible to meet service and storage conditions.

WHY RESEARCH?


These requirements severely limit the choice in a class which can be described as chemical freaks. The few well-known initiators have disadvantages; from time to time they are reported either to fail or to explode inadvertently. Thus the traditional primary explosive, mercury fulminate, has good initiating properties but poor thermal stability so that it becomes unreliable, and eventually inert, after storage in hot climates; it is also chemically incompatible with aluminium. Its replacement is now almost complete but only by a number of newer explosives.

Much of the research is aimed at finding initiators with longer service life and better compatibility to eliminate troublesome and expensive replacement programmes. Modern requirements include stores to withstand both very low and very high temperatures and severe mechanical stress and the need to meet the sensitivity and other characteristics of more sophisticated designs and devices.

There is also the ever-present need to maintain and if possible increase the margin of safety at all stages of manufacture and in use; for example, by substituting an explosive less sensitive mechanically in an electrical device. Accidents are costly, yet the failure of an initiator even on one single occasion could waste much money and effort expended upon a weapon and the means of getting it to its target.

Research on sensitive explosives has the wider objective of relating chemical composition and physical properties, in which crystallization plays an important part. Some of these compounds, such as lead azide, are chemically simple but their crystalline properties are complex and the reason for their exceptional explosive properties is still not understood.

STANDARDIZATION OF EQUIPMENT AND PROCESSES

The ERDE Initiator Section has facilities, which are believed to be unique, for a 'pipeline' scheme of development from the first preparation of a new initiator 

in minute quantities up to the full manufacturing scale. An essential feature is the standardization of versatile equipment on both semi-technical and production scales. Thus production-type samples can be provided for assessment trials which are much more costly than the explosive. The new process and material can be demonstrated at ERDE and reproduced quickly in any one of the standardized units installed in many research establishments and factories in this country and overseas.

Another advantage is that a potential customer for a licence to manufacture can confirm that the process is in operation on a production scale and that any problems which might arise (such as the supply and specification of an ingredient) could be investigated at ERDE under the right conditions.

The success of this scheme has been dependent on three main provisions:

- 1 The availability of the specially designed buildings and equipment
- 2 Staff with experience of both laboratory research and factory development
- 3 Active collaboration with other sections in ERDE concerned with ingredients, compatibility, crystallography, sensitiveness and safety.

THE RD1300 SERIES

Standardization is identified with a number in the RD1300 series. This became necessary when it was appreciated that the conventional chemical name was inadequate to describe the special characteristics of the composition even as a single substance. Thus under 'lead styphnate' there are several RD1300 compositions which are different either chemically or physically from the commercial explosive as described in the literature. These various modifications have been developed for such diverse applications as fuseheads, bridgewire electric igniters, conducting composition devices, primers and percussion caps and fuze delays.

Another example of a modification is RD1357 which is B type tetrazene in free-flowing granular form (UK Patent No 985,293); it is used as a sensitizing explosive and is in production in Pakistan with licence agreements in Belgium and Sweden.

The RD 1300 series includes new initiators which have been put into production for the first time with a wide range of applications from explosively-operated switches through detonators and fuzes to cable-cutters in release mechanisms, in fact wherever the convenient potted power of a small quantity of explosive can be put to work.

CRYSTALS

A common problem with primary explosives is that when prepared by normal chemical methods the particle size and shape makes them unsuitable for technical use with difficult and possibly dangerous processing and handling. For example silver azide could not be utilized until such difficulties were overcome.



Special crystallization techniques have been developed with novel nucleation methods and the use of additives to modify either without diluting the explosive, or to phlegmatize it to make it safe to handle. Mechanical mixtures have been replaced by incorporating an insoluble component within the explosive crystals, new polymorphic modifications have been discovered and the phenomenon of spontaneous explosion during crystal growth has been studied.

Finally, the possible application of novel crystallization techniques to substances other than explosives (e. g. urea) may be noted.

Enquiries should be addressed to :

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B/9/1

Mintech ERDE

EXPLOSIVES HAZARD ASSESSMENT

This exhibit illustrates some of the aspects of the work of the Sensitiveness Section, which deals with a wide range of problems connected with the preparation, storage and handling of explosive and other hazardous materials.

One of the principal responsibilities of the Section is the preparation of official Safety Certificates, this being a mandatory requirement before the manufacture of a new explosive composition in Royal Ordnance Factories may proceed. Conditions for handling and storage are laid down on the basis of the data contained in the Certificate. Methods for the tests stipulated for the Certificates are rigorously specified and members of the Section have taken an active part in the drafting of procedures. All of the following tests are carried out at ERDE, which is the sole authority for certificates issued by the Ministry of Technology, and for tests (3) - (6) for certificates issued by the Ministry of Defence.

1 ROTTER IMPACT TEST

A small specified quantity of the explosive is struck by a falling weight. The criterion of a fire is the production of a specified volume of gas as measured in a gas burette. Sensitiveness is essentially a statistical phenomenon, and the result of a series of impact experiments with the material under test and with a standard explosive at differing heights of fall are analysed to give a Relative Median Height or 'Figure of Insensitiveness'.

2 MALLET FRICTION TEST

A small quantity of the explosive is placed upon an anvil, and struck a glancing blow by a mallet held in the operator's hand. A boxwood mallet is used with anvils of softwood, hardwood or York stone, and a mild steel mallet is used with anvils of mild steel, aluminium bronze and naval brass. Although reasonably consistent results are obtained by different operators, it is hoped in due course to replace this test by means of a friction machine currently under development.

3 TEMPERATURE OF IGNITION

A small specified quantity is heated at 5°C per minute and the temperature of ignition is noted.

4 EASE OF IGNITION

A specified quantity is placed in a tube and subjected to the burst of flame and sparks emitted from a piece of Bickford fuse. The nature of the response (non-ignition, burning or explosion) is recorded.

5 TRAIN TEST

An unconfined train of material is ignited at one end by a naked flame and observations are made of the liability of the sample to continue to burn and how vigorously it does so.

6 ELECTRIC SPARK TEST

Specified quantities of the explosive are subjected to sparks of energy 4.5 joules, 0.45 joules and 0.045 joules successively until a run of 50 trials at the same energy gives no ignition. These materials ignited with the lowest energy i. e. 0.045 joules are likely to need stringent safety precautions and so receive further testing (see leaflet on 'Electrostatic Hazards').

Extra tests are available for assessing the impact and friction sensitiveness of especially sensitive materials.

All of the statutory tests are carried out on relatively small quantities of material, usually in powdered form, and it is realised that not all the risks liable to be encountered in practice with explosives in the mass are covered. This has led to the development of a wide range of further tests, most of which involve larger quantities of explosive. These include:

- (a) The various versions of the Gap Test, in which the material under test is initiated by a standard donor charge, the output of which is attenuated by a gap of brass shim or cardboard of variable thickness. The nature of the response (non-initiation, combustion or detonation) is determined by the use of either a witness plate behind the charge under test, or flag gauges. The results are analysed statistically as in the Rotter impact test.
- (b) Sealed vessel tests in which the material is heated or ignited under heavy confinement. The nature of the response, combustion or detonation, is assessed from the state of the remains of the vessel, and is used for determining the safety distance category.
- (c) A drop test in which a spigot is forced through the thin metal lid of a container filled with the explosive.
- (d) An oblique impact test in which a charge is dropped at an angle on to a rough surface.
- (e) A 'snatch friction' test in which a frictional force is rapidly applied to a small charge. This test differs from the mallet friction test in that friction occurs only between one rough surface and one surface of the explosive charge. (Tests (d) and (e) were originally developed at AWRE).
- (f) A bonfire test in which the explosive contained in a wooden box is enveloped in flames from a 'standard bonfire'.



- (g) The handling of liquid explosives introduces an additional hazard i. e. the adiabatic compression by pressure impulse of bubbles present in the liquid, leading to ignition of the vapour contained therein. Two tests are available which aim to reproduce this situation.

Enquiries should be addressed to:

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B/10/1

Mintech ERDE

TIME-RESOLVED SPECTROSCOPY OF TRANSIENT EVENTS

The combination of spectrograph, photomultipliers, oscilloscopes and camera permits time- and spectrally-resolved photometry of transient events. In such an apparatus in use at ERDE, several exit slits in the focal curve of a spectrograph transmit narrow wavebands of visible and near infra-red radiation on to end window photomultipliers types EMI 9558 and Mullard 150 CVP. The electrical outputs from the photomultipliers are fed via coaxial cables into Tektronix oscilloscopes types 551 and 555 fitted with type C19 cameras. Care is taken that the photomultipliers are operated under conditions which preclude inter-dynode space-charge effects, so that deflection of the CRO trace is a linear function of radiation intensity. The spectral locations of the exit slits are determined by calibrations with an iron arc. Photometry is made quantitative in terms of spectral radiation intensity by calibrating the apparatus with a tungsten ribbon lamp which has itself been calibrated at the National Physical Laboratory. The time bases of the oscilloscopes yield the time history of the phenomena studied. The synchronization of the camera shutters, the CRO time bases and the transient event to be studied is achieved by the use of suitable delay circuits.

We have used this technique to measure temperatures both in condensed phase detonations and in gaseous shock waves. Anticipating the results, there are some detonation and strong shock waves where the temperature and pressure are high enough for the front to radiate as a grey or even a black body. Radiation intensity measurements made at two or more wavelengths may thus be used to solve Planck's equation to give temperature and emissivity.

$$I_{\lambda} = C_1 \epsilon \lambda^{-5} \left[\left(\exp \frac{C_2}{\lambda T} \right) - 1 \right]^{-1}$$

DETONATIONS

As an example, our experiments on the liquid explosive nitromethane showed that its detonation front radiates as a black body at a temperature of about 3460°K. The high emissivity may be due in part to the presence of free carbon in the detonation products and it is intended to elucidate this point by experiments with carbon-free explosives.

A knowledge of detonation temperature is important in testing equations of state which are valid under the detonation conditions of 3000-4000°K and 200-400 kilobars. It enables an assessment to be made of the relative contributions of thermal and repulsion energies to the internal energy of the system. Under detonation conditions the repulsion energy may represent as much as 50 per cent of the total energy.

INTENSE SHOCKS IN RARE GASES

We have used the same apparatus to investigate the radiation emitted by argon and other rare gases when strongly shocked by explosives. The high luminosity (millions of candlepower) of such shock waves is frequently utilized in high speed photography. Our experiments have shown that strongly shocked rare gases emit black body radiation; in the case of argon, (initially at 1 atm 20°C) the temperature was about 21000°K when the shock was driven by the explosive RDX/TNT 60/40. Under these conditions the gas was about 75% ionized. The calculated shock pressure was 1000 atm and the calculated temperature based on hydrodynamic and thermodynamic considerations was 19880°K.

Work is at present in progress on the determination of detonation temperature of solid explosives. As most solid explosives are opaque the detonation wave may be observed only at the instant it emerges from the surface of the explosive. It is hoped that some insight will be gained into the kinetics and physics of the detonation process in new high-energy composite explosives.

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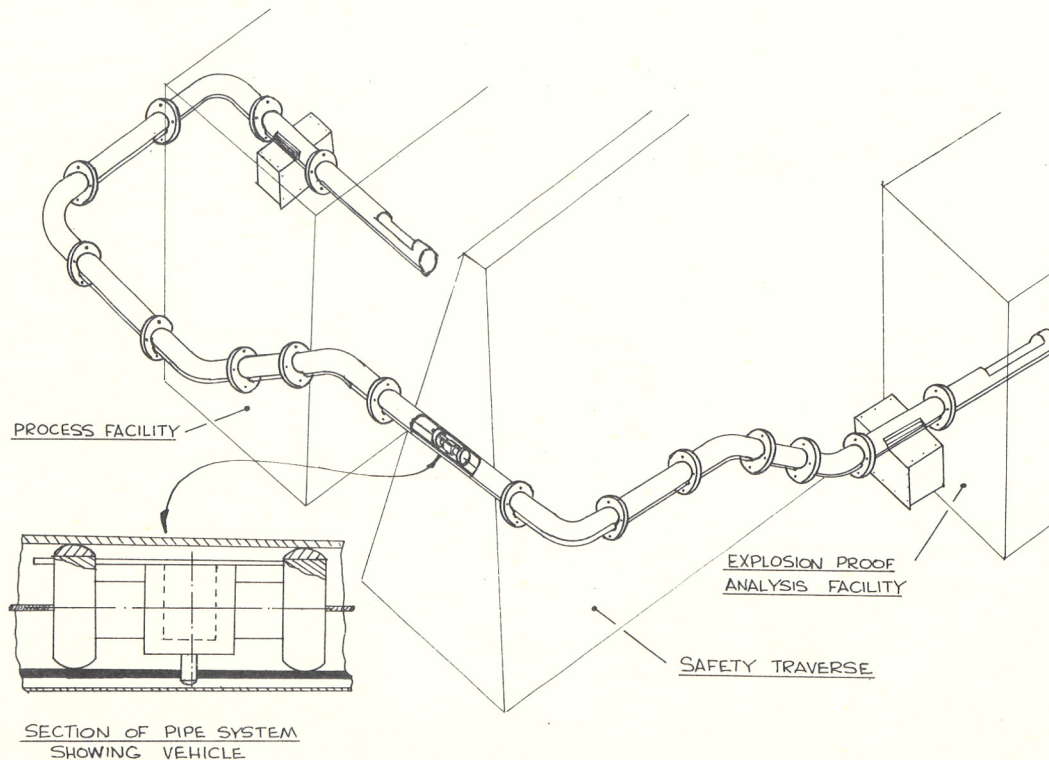
Mintech ERDE

REMOTELY CONTROLLED SAMPLING TRANSPORT SYSTEM

A prototype transport system has been devised to carry liquid or solid explosive samples by remote control from the process building to an explosion proof analysis facility.

The design uses a modified 3" internal diameter UPVC pipe through which passes a transport vehicle (mole) with a volume capacity of 5 cc. The mole is an articulated design to enable it to negotiate bends and irregularities in the pipe. The sketch below shows the basic design features. The centre section of the wall can be fitted with a compatible plastic liner and the end sections are rimmed with PTFE to reduce friction. A special leaf-proof valve is fitted to the centre section which is alternately opened and closed at the limits of travel of the mole, at the process bay and analysis facility. The mole is pulled in either direction by wire winding on to drums and its position is controlled automatically from the control room.

The system has been designed with a number of standard, easily assembled units, so that the system can be dismantled and reassembled and/or modified with the minimum of effort.



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Mintech ERDE

REMOTE CONTROLLED PROCESSING OF HAZARDOUS CHEMICALS

Research and development work in ERDE frequently requires poundage quantities of chemicals which are not available from the trade and may never have been made in more than gram quantities in the laboratory. Often the chemicals or intermediates in their preparation pose a formidable explosion, fire or toxicity hazard. In addition the reagents involved may be very powerful acids, alkalis, oxidizing or reducing agents of a highly corrosive nature and capable of producing violent reactions. Attempts to make poundage quantities of such materials by manhandling could obviously be very hazardous. Remote control is therefore advisable and enables these preparations to be carried out without any danger to the operating personnel.

The laboratory preparative method has to be critically examined and generally modified to make it suitable for remote controlled processing and/or remote control techniques developed to suit the process, keeping intrinsic safety in mind. Scale-up data has to be determined to enable heat and mass transfer, gas evolution and reaction control calculations to be made. Hazard appraisal, corrosion, compatibility, desensitization, storage and waste disposal studies are also necessary. With this information the poundage scale plant is designed for remote control and operational procedures drawn up to ensure smooth and troublefree production.

There are two facilities in the Chemical Engineering Section both being within mounds and with remote control operation. The essential difference between the two is that the building for this facility is designed to withstand an explosion equivalent to 5 lb of TNT without structural damage. The other facility has exceptionally good fume extraction and is now used principally where the major risk is toxic.

This facility, built of reinforced concrete, consists of two cells separated by a services room. The control room is outside the earth mound and vision of the plant is possible by binoculars through mirrors or by closed circuit television. A video-recorder is being developed by our Electronics Section working on a closed loop so that the last two minutes of operation is always on record. Thus it will be possible to analyse the sequence of events leading to an incident. When the facility is completed it will be possible to transfer material from the process cells to magazines or to the adjacent high energy explosive processing unit by small gauge railway, also remotely controlled. A transport system has been designed to convey samples from the cells to an explosion cupboard next to the control room. Analyses on these samples will be carried out from the control room by direct vision through a laminated safety glass window. 

Each process cell is fitted with an extraction fan and all electrical fittings are flameproof. Equipments for the remotely operated plants have been built almost entirely from glass, stainless steel, polythene and PTFE to overcome corrosion difficulties. Glass vessels also have the advantage that liquid levels, frothing, etc. can be easily observed thus reducing the instrumentation required. The general principle adopted is that the plant shall be static and the chemicals transferred from stage to stage. This avoids the need for bearings with the possibility of accidents due to friction. Reagents are kept below reactor level to avoid accidental spillage to the reactor and whenever possible chemicals are transferred by air blowing or suction. The use of valves in process streams is not desirable and is kept to a minimum. For safety reasons, all controls such as valves in service lines are operated by compressed air. The use of compressed air from the same main supply has the advantage that all systems can be arranged to give failure to safety. Operations are cut down to a minimum and the simplicity thus achieved is itself conducive to safety.

One of the difficulties for this scale of operation is that commercial equipment for remote control is generally too big and is not made to deal with the wide range of corrosive and explosive hazards that we encounter. It has been necessary to design small pneumatically controlled valves of glass and PTFE, special glass and polythene covered metal stirrers, glass air eductors, etc. Equipment is being built in units which can be easily joined together to enable chemicals using various unit processes to be produced with minimum delay. To date such operations as reactions with controlled feeds and temperatures, washing, filtration, liquid/liquid separation, distillation and drying have been carried out remotely. It is interesting to note that once the facility has been perfected it provides the easiest way of making chemicals quickly yet with maximum safety whether there is any obvious danger or not. This provides extra security against the unforeseen hazard.

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THE AIR BLAST FROM HIGHLY ASYMMETRIC EXPLOSIONS

The blast wave from an explosion in a fluid medium initially at equilibrium depends upon parameters descriptive of the medium and of the explosion. For the spherically symmetric explosion, the classical 'energy hypothesis' according to which the explosion is sufficiently characterized by the total energy release, E , together with the assumption that the medium behaves as an ideal gas, enables properties of the wave to be expressed as functions of a dimensionless variable

$$\frac{1}{r} \left(\frac{E}{p_0} \right)^{\frac{1}{3}}$$

p_0 being the undisturbed pressure and r the distance to the observer at O . For increasing r , the functions tend to linearity, corresponding to the approach to acoustic conditions. Scaling factors for the pressure and time dimensions are then simply

$$\frac{a}{r} \left(E p_0^2 \right)^{\frac{1}{3}} \quad \text{and} \quad \frac{b}{c} \left(\frac{E}{p_0} \right)^{\frac{1}{3}}$$

respectively, a and b being dimensionless constants and c the sound speed.

Consider now a linear distribution of explosive and imagine it divided lengthwise into elementary charges. We postulate that the energy hypothesis may be applied to each of these elementary charges individually. Dimensional analysis shews that under such an assumption the explosive is sufficiently characterized by $\sigma(r)$, the linear density of energy release. In this way the classical energy hypothesis is extended to linear distributions of explosive. The dimensional analysis shews, furthermore, that the scaling factors appropriate to such linear distributions are, in the acoustic limit,

$$\frac{a}{r} \left(\sigma p_0 \right)^{\frac{1}{2}} \quad \text{and} \quad \frac{b}{c} \left(\frac{\sigma}{p_0} \right)^{\frac{1}{2}}$$

for pressure and time respectively. These may be compared with the corresponding expressions for the spherically symmetric case in paragraph 1 above.

The extended energy hypothesis implies that for each element of linear charge distribution the volume density of energy release, e , and the element diameter, d , may be varied without affecting the form $p(t)$ of the resultant blast wave, provided that the variations are such as to keep the linear density, σ , constant; i. e. provided that $\delta(d^2 e) = 0$.

If we consider now an arbitrary 3-dimensional distribution of explosive and choose some convenient axis passing through O and through or near the charge distribution, then it may be seen that an isometric projection of the charge onto this axis satisfies the condition $\delta(d^2 e) = 0$, so that $\sigma(r)$ is invariant ◻

under the projection. In other words, to the extent that the extended hypothesis is valid, the arbitrary 3-dimensional distribution may be replaced by an equivalent linear distribution along the line-of-sight.

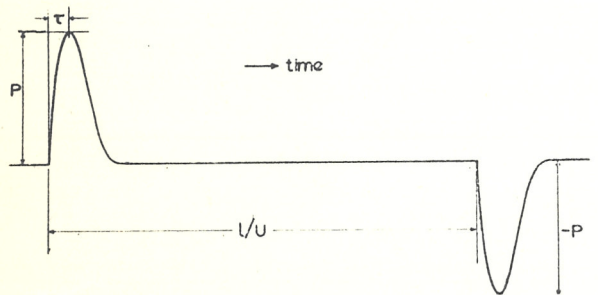
Each of the elementary charges comprising the linear distribution is assumed to generate a pressure wavelet having the following properties:

- 1 Independence of other wavelets
- 2 Spherical symmetry
- 3 A waveform whose shape is $(1-x)e^{-x}U(x)$
- 4 An acoustic overpressure/distance law
- 5 Pressure and time scaling factors given in paragraph 2 above

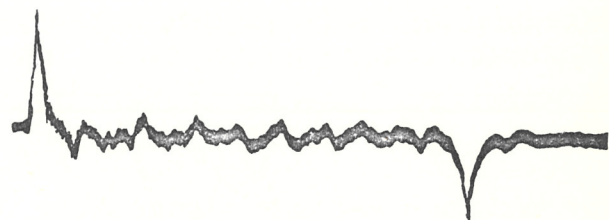
The resultant approximation to the far field blast wave from a distribution of equivalent linear energy release density $\sigma(r)$ is then given by:

$$p(t) = \int_{r_1}^{r_2} \frac{ap_0}{br} \left\{ 1 - \frac{ct - r}{b\sqrt{\sigma(r)/p_0}} \right\} \exp \left\{ - \frac{ct - r}{b\sqrt{\sigma(r)/p_0}} \right\} U(ct - r) dr$$

The solution for $\sigma = \text{constant}$ is shown below, compared with an experimentally determined waveform. The inverse problem of determining $\sigma(r)$ to obtain a specified $p(t)$ has also been solved (within the assumptions stated above).



Calculated pressure waveform from straight linear charge of uniform explosive linear density.



Experimental pressure waveform from straight linear charge of uniform explosive linear density, length 60 m.

REFERENCES: Hawkins S. J. and Hicks J. A. ERDE Report in preparation.

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HIGH SPEED PHOTOGRAPHY

Modern high speed cameras are essential tools in present day explosives research.


In ERDE wide use is made of both framing and streak cameras to study explosive behaviour both qualitatively and quantitatively.

Framing cameras may be divided into two distinct classes - those using 16 mm movie film giving picture rates up to about 18,000 per second, and those using larger format (35 mm) movie film and giving framing rates from about 100,000 to over 4 million per second. Examples of the first type used in ERDE are the Fastax and the Hycam, of which the Hycam Model K2001 is presently used in the Explosives Branch. This is a camera of the optical compensation type, and will give picture rates of 100-9000 per second in the full-frame mode and 200-18000 per second in the half-frame mode.

Such framing rates are appropriate in the study of flames and low order deflagrations as well as in the analysis of mechanical failure of apparatus under development. On display are series of photographs taken from 16 mm film, showing some of the applications in which the Hycam camera has been recently used.

For the study of extreme speed explosive phenomena, such as shock and detonation waves, ultra high speed cameras using the larger 35 mm format are necessary. The framing camera presently used is the Beckman and Whitley Model 189. This is of the Miller type in which the film is stationary and the light image is swept over it by a turbine-driven steel alloy mirror. Optical shuttering provides a series of 25 frames at rates of between 10^5 and 4×10^6 per second depending on the type of turbine in use plus the fact that the camera is a non-continuous access model.

These extreme speeds give rise to certain synchronization problems. Commercial hot wire type detonators were found to have too variable a time delay and a programme of work was initiated to design a detonator that would have a maximum variability of plus or minus one frame at the maximum framing rate of the 5000 rps turbine. The result was a high voltage spark initiated detonator containing a mixture of 98/2 PETN/aluminium powder with an operating time of 13.9 μ sec and a standard deviation of 1.1 μ secs. This has proved to be extremely reliable in many hundreds of firings.

To photograph non-luminous events such as non-reactive shock waves very intense front lighting must be employed. Standard electronic flash techniques 

cannot be used because of the limited intensity and normally destructive nature of the event. Recourse has to be made to the now standard technique of argon flash bombs. These are made within the Section and can be so constructed as to give the intensity and duration required to illuminate the event.

The main field of work in which the framing camera has been used in recent years is the study of shock and detonation waves in explosives. Mention may be made of the work on the explosive initiation of RDX crystals and on the transition from shock to detonation wave in RDX/TNT. Colour prints on display show an interesting sequence of framing camera photographs taken during this latter study.

For quantitative measurements of detonation and shock-wave velocities the high-speed streak camera is indispensable. This, unlike the framing cameras, does not produce a series of discrete pictures but rather a space/time curve of the event photographed. It is basically similar to the framing camera already described except that there is no optical shuttering action, the light image, of slit form, being swept by a rotating steel mirror directly over a length of 35 mm film. Thus, knowing the writing speed and demagnification of the optics, velocity measurements can be readily calculated. The camera that is in use here is the Beckman and Whitley Model 339B with a maximum writing rate of 8.5 mm per microsecond. This camera is far too large and immovable to be shown but photographs and typical records are included in the display.

To complete the exhibit there is a small historical display of 16 mm high speed cameras ranging from the Zeiss Zeitlupe of 1936 to the present day Hycam.

REFERENCES:

Special Detonators for Photographic Use: E. L. Kendrew and R. M. H. Wyatt. Seventh International Congress on High Speed Photography, Zurich, September 1965.

The Explosive Initiation of a Single Crystal of Cyclotrimethylene Trinitramine: G. K. Adams, J. Holden, E. G. Whitbread. XXXIst International Congress of Industrial Chemistry, Liege, September 1958.

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COMPOSITE PROPELLANTS

A composite propellant is a mixture of chemicals containing both oxidizing and fuel components. Gunpowder is a well-known example although modern composite rocket propellants differ from gunpowder in that they are coherent rather than granular materials. These modern propellants contain an oxidizing agent such as ammonium perchlorate and a fuel which also acts as a binding agent for the solid ingredients. When the mixture is ignited in a rocket motor, high pressure gas is produced which escapes through a nozzle and thus impels the rocket forward by reaction.

Two principal types of composite rocket propellant are in use or under development in the UK. These are known as plastic propellant and rubbery propellant, respectively. Both are used in the form of a single block or charge which fits exactly the rocket motor case and is tightly bonded to it. Burning takes place either at the end or in a central hole. Both types of propellant have the merit that because of their plastic or rubbery nature they retain their bond to the casing in spite of differential thermal expansion. Bonding to the case is important because it protects the metal from the hot gases and enables a light-weight motor to be used thus increasing the overall performance.

PLASTIC PROPELLANT

Plastic propellant is a British invention which has been developed at ERDE. It is a stiff putty-like material which uses polyisobutene as the binder and is easily made in a paste mixing machine. It is then deaerated and pressed into the rocket motor case by hydraulic pressure. It is cheap, about 15/0 per pound, is safe to handle, is chemically stable, and offers a wide range of burning rates.

A typical composition comprises ammonium perchlorate (77 wt per cent), aluminium powder (10 wt per cent), polyisobutene (11 wt per cent), wetting agents such as ethyl oleate, and a burning rate catalyst. A material of this composition has a specific impulse of about 245 seconds, a burning rate of 1.0 inches per second, and is usable over a temperature range of about -20°C to +50°C. By adjusting the components of the propellant, the burning rate may be varied within a range of about 0.1 to 1.5 inches per second.

Plastic propellant is not suitable for very large rocket motors exceeding about 36 inches in diameter or for applications where very low temperatures are encountered. Research is therefore being carried out also on the rubbery type of composite propellant.



RUBBERY PROPELLANTS

Rubbery propellants are formed by mixing a curable carboxyl-terminated polybutadiene (CTPB) binder with dried powdered oxidant at 60°C together with imine and epoxy curing agents. The slurry formed is filled into a motor casing and cured for several days at 60°C.

CTPB propellants originated some years ago in the United States and it is only recently that work on these materials has commenced in the United Kingdom. CTPB propellants have excellent rubbery properties which are retained at very low temperatures and since they have a high specific impulse they are suitable for high performance motors required to operate over a wide temperature range without a limitation on charge size.

A considerable variety of ballistic and physical properties can be obtained by changes to the composition of the propellant mixture. The range of burning rates so far obtained is less than with plastic propellant but future research is likely to produce considerable improvement.

TESTING FACILITIES AT ERDE

Many requirements for the formulation and development of new composite rocket propellant compositions come to ERDE since this is the only United Kingdom Establishment where such work is carried out. Initial investigations are carried out on small scale equipment. Results are then applied to large-scale manufacture in order to establish reliable manufacturing procedures, and methods of quality control.

During the development process an extensive range of testing has to be carried out. Typical tests which have been selected for demonstration include the determination of physical properties at low temperatures, the determination of specific surface of powders, a test to simulate differential expansion and contraction on storage and the determination of internal ballistics using a small 2 inch rocket motor usually called a K-round. Actual static firings of K-rounds will be shown together with the trace of pressure and thrust produced for a very slow burning propellant at low pressure. More energetic materials may be heard undergoing test in the reinforced concrete proofstand nearby.

COMMERCIAL USES

It is not surprising that some of the developments in the propellant field should find application in such items as signal rockets and line-throwing rockets. By courtesy of Messrs. Pains-Wessex of Salisbury a display of small commercial rocket motors which use plastic propellant has been arranged. The use of plastic propellant has enabled some 50 per cent improvement in performance to be obtained without significantly increasing the cost.

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Mintech ERDE

ELASTIC PYROTECHNICS - A NEW APPROACH TO AN OLD ART

Fireworks or pyrotechnics have been in use for centuries for purposes such as terrifying the enemy in war and celebrating the resultant victory. In modern warfare, pyrotechnics provide means of signalling with light or smoke, illuminating targets and guiding missiles, igniting rockets, setting time intervals (delays) and so on. The conditions under which they do so become more varied and exacting as shells fly faster, aircraft and rockets higher.

Pyrotechnic compositions consist of something to burn - fuel - and something to burn it - oxidant - chosen to suit the application. For example, for bright light, magnesium powder is included; for coloured light, a metallic salt oxidant, giving the flame this colour; slow-burning compositions are produced by using additives, a mild oxidant or an organic fuel.

Conventional pyrotechnic compositions are usually mixtures of powders which are pressed into containers often under high loads in heavy presses. Even so, many pressed charges crack fairly easily under shock or vibration giving rise to inconveniently rapid burning; they are also susceptible to damage by moisture and hazardous to mix and process.

Elastic pyrotechnics developed at ERDE can overcome these drawbacks in many cases. The constituents are bound together and protected by a rubbery matrix to form a robust, resilient charge; consolidation is carried out by hand-pressure in simple moulds, or extrusions can be made; 'loose' granular mixtures are non-segregating, in contrast to uncompressed powder mixtures. Charges are so little affected by moisture that they will function after a period of immersion in water.

Compositions are made by adding the oxidants in some instances, the fuels in others, to a liquid polymer, together with curing agents; mixing until the powders are wetted completely; then adding the other constituents in portions, mixing until a homogeneous mass is produced. In this way, fuel and oxidant particles are always separated by a film of liquid during mixing and the risk of an accidental ignition is small. The uncured composition, which may range in consistency from a coherent to a crumbly paste, can be kept in a refrigerator for several months and cured in about 16 hours at 70°C.



Compositions have been developed for the following applications:

priming in rolled sheet form to replace gunpowder primed cambric and
extruded cords, bare or encased in lead tubing;

coloured signal, rocket tracking and illuminating flares;

screening smoke;

rocket igniters.

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Mintech ERDE

Chemical Engineering Section

The Alignment Processes for Fibres and Whiskers

To obtain the maximum properties from fibres or whiskers it is necessary to pack the maximum amount into a composite. This is achieved by alignment. The problem of obtaining a usable aligned material resolves itself into:

- 1) dispersion in a carrier
- 2) alignment
- 3) removal of the carrier

Our basic technique for alignment is the use of viscous drag in extrusion through an orifice or slit and our processes are both based on this.

Alginate Process

A dilute dispersion of fibre or whisker in a viscous aqueous solution of ammonium alginate in water is extruded under slight pressure through a bath of dilute hydrochloric acid at 4-5 feet per second. A typical mixture contains 2% fibre and 3% alginate. The acid is hydrochloric. On contact with the acid the ammonium alginate gels instantaneously to alginate and "freezes" the aligned fibres. The thread is wound on to a reel and then cut off, washed and dried on a tray. The alginate is removed by controlled combustion leaving the aligned fibres.

The Glycerine Process

In this process the viscous carrier is glycerine and the alignment is achieved by extrusion through a slit. In this case the carrier is removed by suction from a filter bed. The slit moves over the bed with a velocity slightly in excess of the extrusion. This ensures that there is no misalignment as a result of surface flow. The excess glycerine is washed off with a water spray and the sheet of aligned fibres is dried for use. The glycerine is recycled. This process is operated both batch and continuous.

With the two processes we are able to produce aligned material from:

- 1) asbestos fibres
- 2) chopped carbon fibres
- 3) glass fibres
- 4) mixed fibres
- 5) whiskers such as silicon carbide

We produce material in a number of forms for maximum adaptability in use. Examples are:

1. Composite sheet for bars, tubes, and generally shapes produced in heated presses for high performance structural applications.
2. Thin Composite Sheet (5 to 10 thousands of an inch) for honeycomb structures where weight is important e.g. aircraft skins, rocket casings etc.
3. Thin Narrow Continuous Composite Strip for winding techniques to give high strength composites without the need for presses or autoclaves. For use in making tubes, tanks and other regular shaped objects for general use.

We have made composites from our aligned materials with epoxy, phenolic and polyester resins. We have found it best to apply the resin to the aligned material in dilute solution. The solvent is evaporated off to give a "prepreg" which, by suitable choice of resin, can be stored for several weeks. The composite is finally cured under pressure and temperature in a leaky mould. Best results are obtained when only a very slight excess of resin is present.

Basic Properties of Composites

Material	Flexural Strength x 10^3 p.s.i.	Flexural Modulus x 10^6 p.s.i.	Spec. Gravity
Asbestos/resin	up to 125	up to 14.5	1.8 to 2
Carbon (type 1)/resin	up to 100	up to 25	1.6
Silicon carbide/resin	200 - 300*	30 to 40	1.9 - 2

*Silicon carbide is in the early stages of development and only a fraction of its potential has yet been realised.

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PROCESS STUDIES


CONSTANT RATE EVAPORATIVE FEEDER

This has been developed in ERDE to enable the decomposition of methyl nitrite (b. pt. -12°C) to be studied but it has general application as a constant rate feeder. A constant evaporation rate without a large volume of liquid requires a large heating surface area to volume ratio, good heat transfer, stable boiling conditions and a constant temperature heat source. These conditions are met by use of a narrow bore coil which is one leg of a circulatory system. By inducing boiling at the base of the coil the mean fluid density is reduced and the vapour-liquid mixture rises to be replaced by liquid from the reservoir. As the fluid rises up the coil the gas and liquid reach equilibrium and the pressure is virtually independent of the liquid level in the reservoir. A constant rate feed can therefore be taken off through a suitable orifice. To enable the system to function smoothly it is essential to initiate boiling at the right point. This is achieved by the use of a small reed vibrator in the liquid at the bottom of the coil.

HARTRIDGE-ROUGHTON MIXER

Certain propellant ingredients are prepared by precipitation from solutions of electrolytes. Their ballistic properties are greatly influenced by the mixing conditions. The preparation of these materials can be studied in a rapid mixer of the Hartridge-Roughton design. The two streams to be mixed flow continuously and at high velocity through tangential jets into a tiny mixing chamber. The combined stream, partially mixed, leaves the mixing chamber through a tube in which mixing is quickly completed by the turbulent swirling motion. Nearly complete mixing can be achieved in one millisecond. This approaches the ideal precipitator in which the rate of mixing is rapid compared with the rate of nucleation. Thus the effect of reaction parameters such as concentration, pH and recycle can be studied without the complicating mixing factor. Also, the rate of precipitation can be measured by using light sensitive probes at intervals along the observation tube. This technique can be used to study other aspects of precipitate quality. Results can be applied to plant scale production.

MOLECULAR STILL

In the preparation of chemicals for propellants and general research, high purity is often required. As the chemicals are frequently heat sensitive, low temperature distillation with low residence time is essential. The 'molecular' still is really a short path, high vacuum distillation unit with a wiped wall, 

thin film heating section and is produced by Edwards High Vacuum Ltd. The material being heated at any one time is about 5 ml and the duration of heating about 20 seconds, the distillation rate varying from 200 to 800 ml/hr depending on the conditions. Operating pressures down to 10^{-3} mm Hg can be achieved. We have modified the unit by making the heating section of electrical conducting glass which has the twin advantages of low heat capacity and full vision. The consequent rapid response facilitates control and makes it easy to avoid overheating. Anything abnormal such as gum or residue formation can be observed and given attention at an early stage.

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C/15/1

Mintech ERDE

CRYSTALLIZATION STUDIES

Crystal size and shape play an important part in the manufacture and behaviour of propellants and explosives. Materials such as ammonium perchlorate and RDX are required in sizes ranging from 5 to 500 micrometers, and in a variety of size distributions and shapes, according to the particular application.

Most of the materials used are crystallized from solution and it has been necessary to study the mechanisms involved in this process in order to establish a more precise control over the operation.

Much research on crystallization in the past, because of the complexity, has been carried out on the laboratory scale except in isolated circumstances. This forms an excellent background but does not contribute much knowledge on the variables encountered in larger crystallizing plant, and consequently the approach to crystallization in industry, by necessity, has been largely empirical.

Studies in the Chemical Engineering Branch have been directed towards bridging the gap between the academic and the practical approaches, by investigating the significance of the variables encountered in a variety of crystallizing equipment. The majority of the work has used ammonium perchlorate because this is readily available in a pure state and in large quantities, and forms no hydrated solid phases.

A study has been made of an Oslo type classifying crystallizer which has provided useful information. More recently a programme has been undertaken using a computer to solve a set of complex implicit equations which has been produced to predict the product size distribution from a Kestner recirculating evaporative crystallizer.

These showed that nucleation in solution is a difficult problem and a study of this process has been done in two crystallizing systems. In one, a cooled agitated batch vessel, the closest possible control over such variables as the rate of cooling has been achieved in order to obtain reproducible results. The equipment is able to give repeatable crystallizations and it is possible to follow supersaturation changes and crystal size variations at the same time, the latter by a photo-extinction method.

The second is a cooled agitated continuous feed vessel, in which the agitation conditions can be accurately measured, together with close temperature control. By combining the information from these experiments with other



information on the rate of crystal growth obtained in the laboratory, it is possible to build up a clear picture of the mechanisms of crystallization in a large crystallizer. Similar considerations often apply wherever crystalline materials are produced, and the subject is of importance in industry generally.

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C/15/2

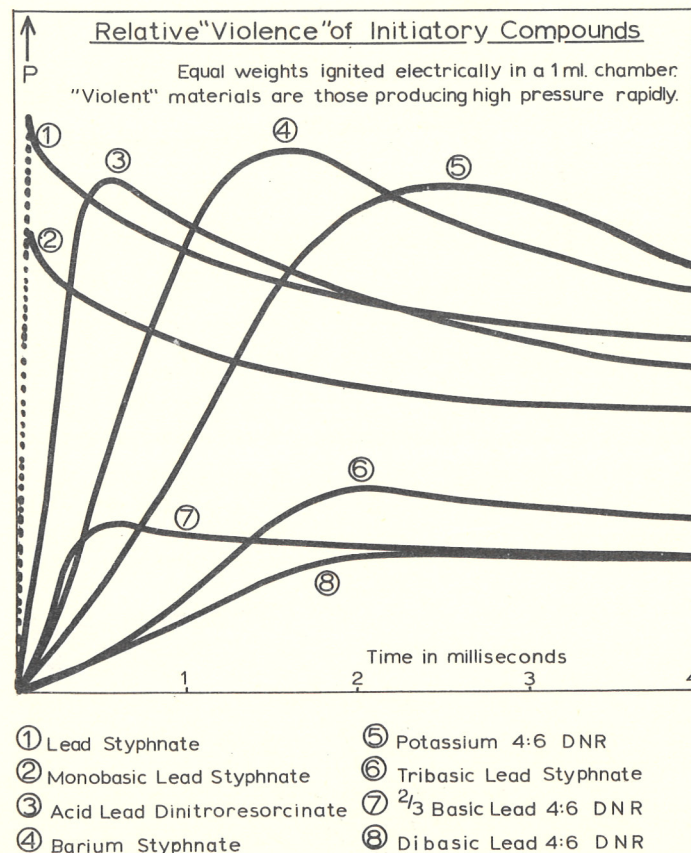
Mintech ERDE

USE OF PRESSURE/TIME STUDIES IN INITIATOR DEVELOPMENT

Igniting a small quantity of an initiating explosive or a device based on it in a small volume and observing the variation of pressure has been used for the following purposes:

- 1 assessment of the 'violence' or disruptive effect of the material;
- 2 matching a pressure/time profile of desired shape;
- 3 determining the effect of exposure to an adverse environment.

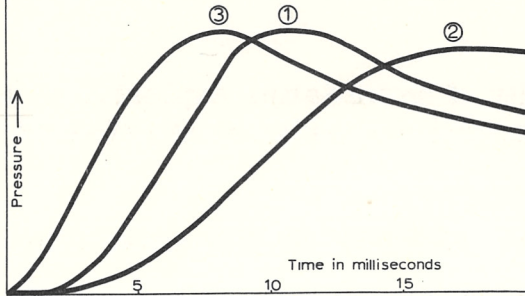
The equipment consists of a firing chamber into which are fitted a quartz crystal transducer and the device (a capsule or a fusehead) under test. Arrangements can be made to heat or cool the chamber or to fire into a large chamber at low pressure. The pressure change is displayed on an oscilloscope and the trace photographed.



High Temperature Fuseheads I

The type R matches the pressure profile of the type E but does not deteriorate at high temperatures.

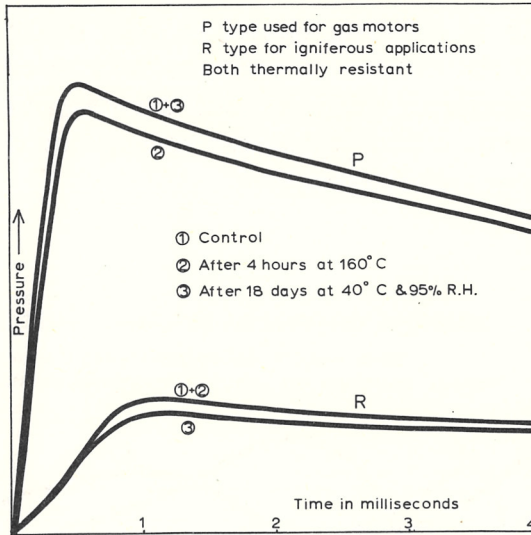
- ① E type fusehead
- ② Ditto after 24 hours at 90°C
- ③ R type fusehead: curve is virtually unchanged after 6 hours at 160°C



High Temperature Fuseheads II

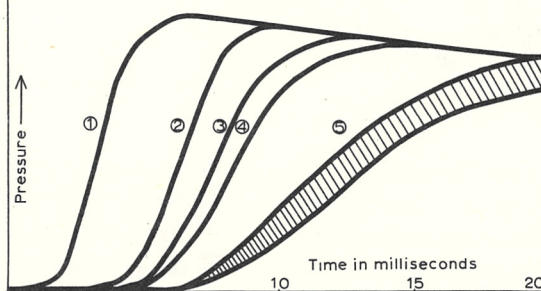
P type used for gas motors
R type for igniferous applications
Both thermally resistant

- ① Control
- ② After 4 hours at 160°C
- ③ After 18 days at 40°C & 95% R.H.



Change in output of standard igniferous fuseheads with time and temperature

- ① Control
- ② After 3 years storage
- ③ After 10 days at 60°C
- ④ After 14 days at 60°C
- ⑤ After 10 years storage (very variable)



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EQUIPMENT DEVELOPMENT

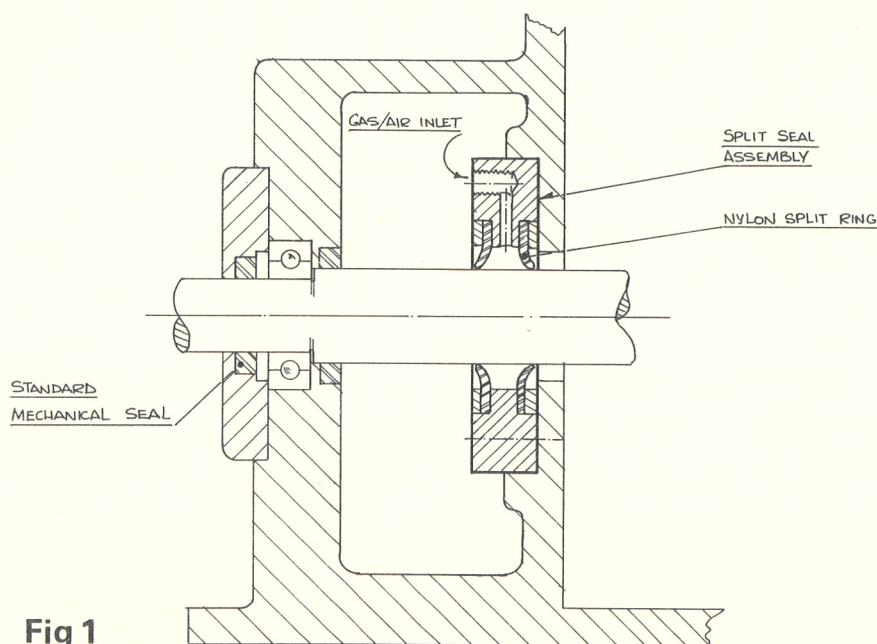
AN IMPROVED DUST PROOF SHAFT SEAL FOR EXPLOSIVES MACHINERY

Over the past decade incidents have occurred with explosive processing machinery which may have been caused by explosive material having entered drive shaft bearings. It is normally possible to use a shaft seal to prevent loss of material from the vessel and to prevent 'foreign' material entering the bearings, but some difficulty has been experienced providing a satisfactory seal for explosive machinery.

The main drawback to the standard types of shaft seal is that the mechanical friction can be very high and difficult to control, because of this it was decided to investigate a new design of pressurized lip seal.

The design considered most practical was a pressurized double lip seal shown in fig 1 made from a compatible plastic material. The mode of operation is that of air or inert gas passing into the centre section and being exhausted through the annulus between the rotating shaft and seal lips. There is therefore gas/air flow outwards from the seal which acts as a dust free barrier between the explosive material and the bearing assembly.

Tests have been conducted using a split seal of this design and they have shown that it could have practical uses in the explosives processing and allied fields.



AIR BEARING FOR A POWDER GRINDING MILL

At present the grinding of powders is carried out in a KEK mill, which uses a rotating pin disc spinning at up to 20,000 revs/min against a similar fixed pin disc.

However, for our particular uses there are certain difficulties with the standard machine. When grinding explosive powders a dangerous situation can arise if the bearing lubricant becomes contaminated with the product. In addition, conventional bearings of the ball and roller type have a limited life at these high speeds.

To overcome these difficulties, an air bearing has been designed, see fig 2, to replace the bearing/shaft assembly and is now being developed to improve its load bearing characteristics. The existing shaft is driven by means of a flat belt from a Kopp variator, and as belt tensions are high, a heavy journal load is placed on the mill shaft. Replacement of the flat belt by a direct drive is being examined. The practicability of using high speed electric motors or air turbines is being studied.

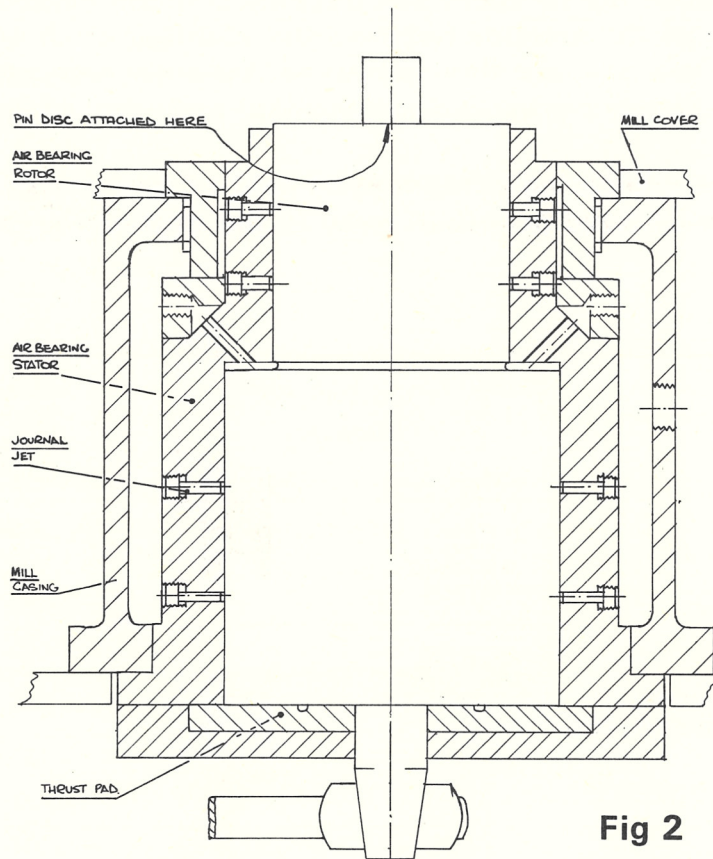


Fig 2

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Mintech ERDE

PROCESS STUDIES


CONSTANT RATE EVAPORATIVE FEEDER

This has been developed in ERDE to enable the decomposition of methyl nitrite (b. pt. -12°C) to be studied but it has general application as a constant rate feeder. A constant evaporation rate without a large volume of liquid requires a large heating surface area to volume ratio, good heat transfer, stable boiling conditions and a constant temperature heat source. These conditions are met by use of a narrow bore coil which is one leg of a circulatory system. By inducing boiling at the base of the coil the mean fluid density is reduced and the vapour-liquid mixture rises to be replaced by liquid from the reservoir. As the fluid rises up the coil the gas and liquid reach equilibrium and the pressure is virtually independent of the liquid level in the reservoir. A constant rate feed can therefore be taken off through a suitable orifice. To enable the system to function smoothly it is essential to initiate boiling at the right point. This is achieved by the use of a small reed vibrator in the liquid at the bottom of the coil.

HARTRIDGE-ROUGHTON MIXER

Certain propellant ingredients are prepared by precipitation from solutions of electrolytes. Their ballistic properties are greatly influenced by the mixing conditions. The preparation of these materials can be studied in a rapid mixer of the Hartridge-Roughton design. The two streams to be mixed flow continuously and at high velocity through tangential jets into a tiny mixing chamber. The combined stream, partially mixed, leaves the mixing chamber through a tube in which mixing is quickly completed by the turbulent swirling motion. Nearly complete mixing can be achieved in one millisecond. This approaches the ideal precipitator in which the rate of mixing is rapid compared with the rate of nucleation. Thus the effect of reaction parameters such as concentration, pH and recycle can be studied without the complicating mixing factor. Also, the rate of precipitation can be measured by using light sensitive probes at intervals along the observation tube. This technique can be used to study other aspects of precipitate quality. Results can be applied to plant scale production.

MOLECULAR STILL

In the preparation of chemicals for propellants and general research, high purity is often required. As the chemicals are frequently heat sensitive, low temperature distillation with low residence time is essential. The 'molecular' still is really a short path, high vacuum distillation unit with a wiped wall, 

thin film heating section and is produced by Edwards High Vacuum Ltd. The material being heated at any one time is about 5 ml and the duration of heating about 20 seconds, the distillation rate varying from 200 to 800 ml/hr depending on the conditions. Operating pressures down to 10^{-3} mm Hg can be achieved. We have modified the unit by making the heating section of electrical conducting glass which has the twin advantages of low heat capacity and full vision. The consequent rapid response facilitates control and makes it easy to avoid overheating. Anything abnormal such as gum or residue formation can be observed and given attention at an early stage.

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C/15/1

Mintech ERDE

THE MANUFACTURE OF RUBBERY COMPOSITE PROPELLANTS

Rubbery propellants are made by mixing together various powdered solids and liquids. The result is a viscous slurry which can be readily cast into a rocket case, and then cured by heating for several days at about 60°C.

A typical composition contains ammonium perchlorate (oxidizer), aluminium (high energy fuel), a solid burning-rate modifier, a liquid polymer (e. g. carboxyl-terminated polybutadiene, CTPB), a liquid curing agent, a cure catalyst and a plasticizer. The liquid polymer and the curing agent react to form the rubber matrix. The solids content is about 85 per cent by weight, of which the major portion is perchlorate.

The N550 mixing and filling plant, erected in 1962, was designed by ERDE. At present adapted for the filling of several small motors from a single mix, it could equally well be used for large motors requiring several mixes.

The central feature is a Baker Perkins stainless steel vertical mixer, capacity 300 kg, specially designed to handle viscous propellant slurries efficiently and safely. The two equal-speed, semi helical mixing blades rotate in fixed bearings. The water-jacketted vessel, which is of figure eight cross-section, is raised and lowered hydraulically. Blade-to-vessel clearance is 6 mm.

Mixing or discharging may be carried out in an inert atmosphere or under a vacuum of 5 mm of mercury. An interesting feature is the use of a labyrinth fluid seal to protect the blade shaft carbon seal from dust within the mixer, which could present a hazard. The fluid used is CTPB, and vacuum differential is catered for by suitable balance pipes.

Power drive is by a 25 hp electric motor through a Vulcan Sinclair fluid coupling and a Carter hydraulic infinitely variable speed gear, giving a blade rotation of 0 - 32 rev/minute. These are located in the adjacent bay.

All ingredients, except ammonium perchlorate, are normally added by hand loading, although facilities exist for remotely controlled additions of liquids and slurries. Ammonium perchlorate is added by remote control from a vibrated hopper, through a tubular vibro feeder and a vacuum-type butterfly valve in the lid of the mixer. The hopper is suspended from a tension load cell connected to an accurate weight indicator in the control room, which gives a check on rate and completeness of addition.



The mix falls into an evacuated transfer vessel. The vessel is then lightly pressurized (up to 50 lb/sq in), causing the slurry to flow into the motor case which is fitted with a teflon-coated mandrel to give the required conduit shape. The motor assembly is then disconnected, inverted and some of the filling tools are removed. The propellant level is adjusted by hand, the motor is sealed and then transferred to the curing oven. Finally, the assembly is dismantled and the mandrel is removed.

All mixing and filling operations are remotely controlled from the reinforced concrete building to the south of the main entrance. Closed circuit television and an intercommunication system are installed. All electrical equipment within the processing bay is dust-tight, because of the use of fine ammonium perchlorate. Services are located outside the bay wherever possible.

The layout, all equipment and the methods of operation have been designed for maximum safety. Numerous interlocking devices and spring-loaded switches contribute to safety of operations. In the unlikely event of mechanical failure causing ignition at the mixing stage, an infra-red detector in the lid of the vessel operates a rocket-powered drencher containing 40 gallons of water. At the same time the mixer motor and vacuum circuits are interrupted. Should the system fail to operate automatically, it can be actuated manually at the control panel.

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A/5/1

Mintech ERDE

PLASTIC PROPELLANT ROCKETS FOR SPACE AND METEOROLOGICAL RESEARCH

This exhibit demonstrates two rocket systems used for civil rather than military purposes. Both use rocket motors designed by RPE, based on plastic propellant compositions developed by ERDE.

The SKYLARK Upper Atmosphere Research Rocket, developed by RAE, Space Department, will carry a payload of 200 kg to an altitude of 150 to 300 km. It has been successfully used for the study of X-radiation from the sun and from outer space, and for the investigation of upper atmospheric currents and micro-meteorite distribution.

The main propulsion unit is the RAVEN rocket motor. This is 43 cm in diameter, has a charge weight of 1000 kg and a total impulse of 193,000 kg sec. It is the largest solid propellant rocket motor in production in the UK.

The performance and accuracy of the system may be increased by the addition of a boost motor; two are available, the CUCKOO and the GOLDFINCH. This choice of boost motors gives considerable flexibility to the system.

The three motors require propellants with widely differing ballistic properties and the plastic propellant system is particularly suitable because of the wide range of burning rates that can be obtained. The RAVEN motor requires a very low burning rate, 3.90 mm/sec at the operating pressure. This is difficult or impossible to obtain with any other type of propellant but can be achieved with a plastic propellant composition by the use of ammonium picrate and oxamide. The boost motors require a high burning rate which can readily be obtainable with plastic propellants by using finely ground oxidant and combustion catalysts.

The SKUA and PETREL meteorological rockets, developed by Bristol Aerojet Ltd., carry smaller payloads, the maximum altitudes are 100 km and 150 km, respectively. These rockets are cheap and efficient for two main reasons. A simple, high-performance, case-bonded, cigarette-burning charge is used in the sustainer motor, and the boost system is recoverable.

The design of the charge used in the sustainer motor would be impossible with any type of propellant other than plastic. The difference between the thermal expansion on contraction of the case and the cigarette-burning charge produces considerable deformation of the propellant surface and no other type of propellant would withstand such deformation without rupture. The high burning rate, 37.5 mm/sec, is easily achieved by the use of very finely-ground oxidant and copper chromate catalyst.



Both the SKYLARK and the SKUA rocket systems have been in use for several years and, by March 1968, 175 SKYLARKS and 204 SKUAS had been successfully projected. Both rockets have been exported.

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A/6/1


Mintech ERDE

ELECTROSTATIC HAZARDS

Electrostatic charges are produced whenever separation of surfaces occurs, and the higher the electrical resistivity of the materials involved the larger are the charges observed. A discharge to a conductor at a lower potential, or to earth, may take place directly from one of the surfaces, or more usually via an induced charge on an adjacent insulated conductor. This discharge may ignite an explosive material or solvent vapour/air mixture in its path, with disastrous results.

In estimating the electrostatic risk in a process, information is needed on the magnitude of charge likely to be generated, and on the level of energy required for ignition of the explosive or flammable mixture. Two sources of energy are personnel who become charged as a result of their movements, and containers which become charged as a result of material being poured into them, or as a result of their contents being withdrawn. In this country the maximum energy that a person can acquire is reckoned to be 0.02 joules, i. e. the energy as a result of charging the capacitance of a human being, taken as $400 \mu\mu\text{F}$, to a potential of 10 kilovolts. This figure of 0.02 joules provides a convenient level for subdivision of precaution, and the chief concern is with the handling of primary explosives, solvent/air mixtures and electrically initiated stores, since these can be ignited with energies less than this value.

In the case of primary explosives the energies for ignition by electrostatic spark are determined using an apparatus with either two steel electrodes, or one steel and one conducting rubber electrode. The first arrangement covers the occasions when sparks discharge from one metal body to another, and the second arrangement simulates the type of discharge from the finger of a charged person. This alternative method of test was developed at ERDE, following studies of the discharges from charged persons. With the two steel electrodes, tests with the two capacitances are sufficient to determine the threshold ignition energy. However with the conducting base electrode, it is necessary to cover a wider range of capacitance, leading to the determination of a minimum ignition energy and a minimum capacitance for ignition. The energy values obtained for most primary explosives are 1-100 microjoules. Those for solvent vapour/air mixtures range from 10 microjoules upwards.

Potentials attained by personnel as a result of walking and other movements are proportional to the leakage resistance to earth, and to avoid high values it is essential to reduce this resistance to less than 1 megohm for handling primary explosives. This is achieved by the use of footwear and flooring made sufficiently conducting by the incorporation of carbon black. However 

even if the operative's body is adequately earthed it is possible for significant charging to occur on outer clothing if this should become insulated from the body.

Powders and liquids of high electrical resistivity are readily charged on pouring from one container to another. This charge can be reduced to a safer level by increasing the conductivity of the powder or liquid, or by raising the relative humidity of the surrounding atmosphere to 65 per cent. Relative humidity and temperature have been found to be interdependent, enabling the hazard under conditions in less temperate climates to be assessed.

The electrostatic charging of containers and packaging for detonators and other electrically initiated stores is dependent on factors similar to those considered above. The replacement of natural products by synthetic materials has increased the magnitude of the charge attained on handling the container, and on withdrawal of its contents, since a much higher leakage resistance is presented - generally greater than 10^{10} ohms. Larger areas of contact and subsequent separation give rise to larger charges and hence potentially greater hazards. The charging of fabrics, and porous material in particular, is affected by their surface condition. The surface leakage resistance of most of these materials is reduced by raising the relative humidity.

These factors should be taken into account in designing the electrically initiated store and its container. If it is not possible to prevent electrostatic charges reaching the igniter, or the latter cannot be made sufficiently insensitive, the best solution is to reduce the electrical resistance of the container and packaging. This is generally achieved by incorporation of carbon black.

ERDE is the main adviser on electrostatic hazards in connection with flooring materials, footwear, clothing, container and packaging materials, and textiles to the Ministries of Defence and Technology and the Ordnance Board (Electrical/Explosives Hazards Committee) and advice is given at enquiries into accidents where it is thought that electrostatic factors are involved.

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B/10/2