"THE DEVELOPMENT OF INNOVATIVE PRODUCTS AND MANUFACTURING PROCESSES UTILISING FIRE RESISTANT MATERIALS"

26 SEPTEMBER 2001

EXECUTIVE SUMMARY

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1. INTRODUCTION

The sponsoring company for this Engineering Doctorate is a manufacturer of passive fire protection materials with about 80 employees. Its primary products are specialist coatings that provide protection from the heat of a fire. Since it is essentially a chemical blending company its core technical skills have been in the chemical formulation of these products. The company employed the Research Engineer (R.E.) to help find new applications for its materials and widen its product range. It identified that domestic Liquid Petroleum Gas cylinders are potentially dangerous because they may explode when exposed to the heat of a fire. It determined to develop an enclosure to protect them from fire for 30 minutes using its fire resistant coatings.

This project was conceived as a simple diversification, transferring existing company technology to a new market area. However, LPG cylinders are very sensitive to fire and require highly effective insulation from heat to make them safe. To meet the company specification, the R.E. developed a new composite fire insulation eight times as effective as the original solution, by combining one of the company's products with other materials. The effectiveness of the insulation and the enclosure design were proven by completely engulfing it in flame from a burning pool of aviation fuel. A series of these tests showed that the R.E.'s design reliably keeps LPG cylinders cool and safe for more than 30 minutes. This experimental work was assessed by the Loss Prevention Council who have based a new fire test standard on it.

During the development process it became apparent that the capabilities required to manufacture an engineered product are quite different from those needed for the creation of new coatings and materials. This new product required a revolutionary advance in the company's approach to design, test methods and production technology. The R.E. has developed a new method of fire testing to provide heat flow data for use in computer models. This is now used to reduce the number of prototypes needed for testing and so compress development time. In order to manufacture the new product the R.E. has had to develop new ways of processing the company's materials. These new moulding techniques have delivered substantial improvements in finish and reduction in material wastage. The capability of the company to produce complex shapes from materials that are difficult to process has been greatly enhanced.

Although the Gas Safe product itself has not yet achieved commercial success, the Engineering Doctorate programme has made a positive contribution to the company. In the last two years a number of interesting new projects have been undertaken that would have been impossible without the new engineering approach and production techniques. These are now beginning to show a return and several new products based on this work are under development.
2. GUIDE TO THE PORTFOLIO

2.1 LIST OF SUBMITTED DOCUMENTS

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Figure 1: List of submitted work in chronological order.
2.2 READING THE PORTFOLIO

It is suggested that the Executive Summary is read completely before the individual submissions are examined. This document should then be re-read in sections corresponding to the relevant submissions. This section provides a guide to which submissions correspond to each part of the Executive Summary and the best order in which to read them. It also describes each of the submissions briefly and explains any changes relating to their content that have occurred since they were written.

The Development of the Gas Safe

Section 3 of the Executive Summary provides the necessary background information about the sponsoring company and the environment in which this Engineering Doctorate took place.

**The Gas Safe Patent: 1, 4, {2}**.

Submissions 1 and 4, are the Gas Safe Patent Application and the granted patent and so are quite similar. However, the original application was written before the R.E. had much input to the project. The granted application reflects the improvements to the Gas Safe and changes to the patent made by the R.E. in the first two years of the project.

Submission 2 was a patent application that was dropped during the examination process. Although it has some significance to new methods and projects discussed in sections 5 and 6, it should not be read in detail.

**Development of a Fire Resistant Enclosure by Iterative Fire Testing: 8**.

Submission 8 describes the lengthy process of the development and testing of the prototype Gas Safes. The detail in this submission is necessary to make apparent the difficulties in developing a relatively complex fire protection product.

**The Gas Safe Pool Fire Test: 3, 5**.

Submission 3 describes the first Gas Safe Pool Fire Test and Submission 5 includes additional information reflecting an improved understanding of the results of the test. This test, and several subsequent tests, provide the evidence that the Gas Safe concept and the designs developed by the R.E. met the specified performance in real fire conditions.

This essentially completes the story of the development of the Gas Safe to meet the company's specification. It is suggested that section 4 of the Executive Summary is re-read at this time since it summarises this work and places a context upon the achievements of the project.

Technical Achievements of the Work

**Information in Support of the Gas Safe Project: 13**.

Submission 13 completes the technical side of the Gas Safe story by describing the work of other researchers in the field. This provides support for the Gas Safe concept and allows the performance of the Gas
Safe to be compared with different approaches. It also provides information on the way this work is likely to develop in future.


Submission 11 describes a new fire testing technique to collect data for use in computer models. This greatly reduces the amount of fire testing needed and so compresses the development time of new fire protection products. Since this submission was written the method has been put into use to improve and reduce the cost of one of the company's products.

Section 5 of the Executive Summary summarises the technical progress and innovations made by the R.E. during the Engineering Doctorate.

Commercial Exploitation of the Project

Although the R.E.'s work was principally technical he was also heavily involved in presenting the Gas Safe product to the outside world.


Initial Comments on Manufacturing the Nullifire Gas Safe Abroad: 7.

Submissions 6 and 7 indicate the way in which the company attempted to exploit the Gas Safe in the first few months after testing was completed. This should be regarded as background information only.


Submission 9 describes the design of one of the Gas Safe variants created by the R.E. This is an example of the work carried out by the R.E. to adapt the Gas Safe concept to meet real market requirements.

Analysis of the marketing of the Gas Safe: 10.

This Submission critically examines the way in which the company researched and developed the market for the Gas Safe. Although this work was carried out in June 1999 it is still pivotal to the outcome of the project.


Submission 12 compiles the material presented in public by the R.E over the course of the Gas Safe project. It demonstrates a detailed knowledge of the subject and an improvement in the presentation technique.

Section 6 of the Executive Summary describes how the innovations generated by this project have been exploited commercially. It also provides a measure of the impact that the Engineering Doctorate will have on the company in the longer term. The final section of this document seeks to analyse the less successful aspects of the project in an attempt to discover the root causes and to learn from them for the future.

The Personal Profile describes how the competencies of the R.E. have developed and so provides a measure of the impact of the Engineering Doctorate on the individual.
3. BACKGROUND TO THE ENGINEERING DOCTORATE

3.1 COMPANY HISTORY

Nullifire Ltd. is one of the primary suppliers of passive fire protection products in the U.K. Passive fire protection is designed into the fabric of the building to slow the spread of fire and to protect the structure so that fire damage does not cause the building to collapse. Such products are fundamental to ensuring the safety of the occupants, fire fighters and in limiting the cost of fire damage. The company was formed in the early nineteen seventies to supply intumescent fire protective coatings to the construction industry. An intumescent is a material that swells or foams when exposed to the heat of a fire and so provides a thick layer of insulating ‘char’. These coatings dramatically reduce the flow of heat into a substrate in the event of a fire and delay fire spread and structural collapse.

As the first entrant in a new market Nullifire Limited was soon the market leader in the U.K. producing a range of intumescent products to protect both wooden surfaces and steel structures from the effects of fire. Increasing competition in the nineteen eighties led to diversification into other areas of fire protection. Part of this strategy was the acquisition of the rights to an epoxy fire protective coating from I.C.I. Advanced Materials in 1988. Nullifire now markets two epoxy based intumescent materials for spraying, casting or trowel application by approved contractors. Typical uses for these materials include protection of industrial structural steel work, bulkheads and decks on off-shore oil platforms.

At the same time the 'Coating Services' department was formed to apply epoxy coatings to specialist components in an in-house facility. This department has remained quite small, making up around 10% of Nullifire's turnover of about £7 million. However, over the past 13 years a few high value, long term military and aerospace contracts have provided a stable core of high margin business.

In 1998 Nullifire was bought by a much larger American company, RPM, and integrated with their Carboline industrial coatings division. The Nullifire branded fire protection products remain the core of the product range, although various other heavy-duty coatings from around the RPM group are also sold. Then, in 2000, Nullifire was transferred to another RPM division, Tremco, a manufacturer of specialist industrial sealants and coatings. Both companies now benefit from pooled buying power and the production of Nullifire coatings in Tremco manufacturing facilities.

3.2 PRODUCT RANGE

Over most of the company history company activities have consisted of three distinct parts; intumescent coatings, "fire stopping" products and the services provided by the Coating Services division.
3.2.1 Intumescent Coatings

At the beginning of the 1990's Nullifire depended on four main intumescent products:

- S602/3, an interior grade thin film\(^1\) intumescent for structural steel,
- S605, a weather resistant thin film intumescent for structural steel,
- System W, fire protection for wooden surfaces,
- "Firec" epoxy intumescent\(^2\) for fire protecting steel for over 60 minutes and also used in aerospace and defence applications.

At this time Nullifire was unquestionably the market leader in this field, both in technical and commercial terms. In 1993 two new intumescent products were launched, the waterborne S607 thin film intumescent and the heavy duty System E epoxy intumescent, intended for the fire protection of off-shore oil platforms and heavy industrial applications.

- S607 has been an extremely successful product and has formed the backbone of company sales ever since. It is only eight years later that competitors' products are beginning to match it and the launch of a replacement is needed.
- System E was technically the best epoxy intumescent in the world for a number of years, and the American companies dominant in this market were forced to develop improved products. Unfortunately, Nullifire had underestimated the difficulty of penetrating the competitive off-shore oil market and was never able to recoup the development cost.

3.2.2 Fire Stopping

Fire stopping refers to the prevention, or delay, of fire spread through a building. Modern buildings above a certain size are normally divided into a number of separate compartments by fire resistant partitions. Invariably, these "fire barriers" are penetrated by doors, linear gaps between walls and floors, and services like cables and pipes. Fire stopping products consist of special sealing materials and assemblies for preventing the spread of fire through these penetrations and so between fire compartments in buildings.

Nullifire began marketing of fire stopping products quite early in its history and by 1993 they accounted for a substantial proportion of sales. Even so, they were never core business for Nullifire in the same way as intumescents. While intumescents are specialist, technically complex products, most fire stopping materials are comparatively basic. As a result they have become quite standardised and most passive fire protection companies carry a similar range. This means that competition is primarily based on price and, to a lesser extent, on brand.

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\(^1\) "Thin film" intumescents are normally a single pack paint, generally understood to be between 250 microns and 2 mm in thickness.  
\(^2\) In contrast epoxy intumescents are a two pack system applied at thicknesses of up to 30 mm.
3.2.3 Coating Services

This department has always been somewhat different from the rest of the company. This is mainly because it has always been the only area where any production takes place, the intumescent and fire stopping products are all contract manufactured.

The Coating Services department has relied almost exclusively on two steady aerospace contracts for 13 years. The larger of the two is the fire protection of missile canisters that are used as transportation packaging and as launching silos on board ship. The outer surface of the canister and various canister components are coated with an epoxy intumescent to reduce the amount of heat that would reach the missile in the case of a fire. The smaller contract involves the fire protection of aircraft engine actuators that are used to control turbine blade orientation in flight. These must operate for a short time in case of an engine fire and so are coated in a thin layer of epoxy intumescent.

3.3 TESTING AND DEVELOPMENT METHODS

3.3.1 Fire Test Methods and Procedures

Since passive fire protection products are safety critical a good level of confidence in their performance is essential. To this end procedures for the independent certification of products have existed for many years. In theory, this framework allows suppliers, customers and those providing insurance cover to operate under equal technical and commercial conditions. Although Nullifire operates primarily in construction, it also supplies the off-shore, defence, aerospace, and mass transport industries. Each of these operates quite different certification regimes designed to suit its own conditions.

In the U.K. the construction industry depends upon standard test methods that are set by the British Standards Institution. These test methods are intended to cover the various ways in which materials and constructions react to and resist fire. They specify a range of test rigs, burners and furnaces that aim to recreate fire conditions in a reproducible way. Historically the various parts of BS 476 have covered most of the tests relevant to Nullifire. The European Committee for Standardization is now issuing European Standards to replace these, but it is unclear exactly when they will all take force.

In the construction industry the process of certification depends upon the independent test centres to carry out and report on the test results to ensure impartiality. This process can be very time consuming, a waiting list of six months for some tests is not unusual. The expense of testing is always a major consideration since a single large scale furnace test may cost as much as £10,000. It is quite normal for the cost of certifying a new product range to be as high as £50,000 and certifying System E cost much more because of the wide range of different tests required. In other industries, particularly defence, the customer may select the test method, carry out or witness the test itself and take ultimate responsibility for the reliability of the results.
Fire test methods may be divided into a number of categories designed to establish the behaviour of materials and elements of construction when exposed to fire. In some industries tests on how easily materials are ignited, how quickly flame will spread across their surface and how dense or toxic the smoke emitted from their combustion are important. Some fire tests use a "standard" burner or an intensely hot radiant panel to ignite samples. Large scale and dangerous tests often surround the test specimen with a pool of burning hydrocarbon fuel. The "jet-fire" test, which is generally regarded to be the most severe, plays a supersonic jet of burning fuel upon the specimen to subject it to thermal shock and erosion as well as intense heat.

The tests that are usually most important to Nullifire are those that measure the temperature rise of a protected item or the time it takes for flame to penetrate a fire barrier. Most of these tests use a gas fired furnace to recreate the conditions of a fire in a reproducible, although not particularly accurate way. BS 476, part 20 [1] defines test furnace temperature rise to follow one of the standard heating curves shown in figure 2.

![Figure 2: BS476: Part 20 Cellulosic and Hydrocarbon heating curves.](image)

Other national standards define different curves but the two curves shown are quite typical. The curve chosen for a test should represent the severity of the fire the product is likely to be exposed to. A "cellulosic" fire is generally regarded to represent an ordinary building fire, fuelled mainly by wood and paper. A "hydrocarbon" fire is much more severe because it is intended to represent the rapid ignition and intense heat from fires burning fossil fuels.
The Nullifire test furnace has internal dimensions of 1m x 1m x 1m and is built of refractory bricks held in a welded steel frame as shown in figure 3. The temperature inside the furnace is measured using four type K thermocouples which are read by a PC programmed to control the four gas burners so the furnace temperature follows the selected heating curve.

According to the standard the test specimen should, as far as is possible, be identical to, or at least representative of, the element of building construction that is to be evaluated. All the critical aspects of the element, including joints, penetrating services, finishes etc. must be incorporated in the specimen. This is to ensure that the test is as representative as possible of an actual fire.

Standard test methods specify the location of type K thermocouples placed at strategic points on a test specimen. During the fire tests the furnace control computer records the rise in temperature at each site every 10 seconds. This is related to theoretical temperature limits to determine whether the specimen has 'passed' or 'failed' the test. These methods work well for their intended purpose; to define whether a material or element of construction will remain intact or provide protection for a given time period.

Figure 3 shows a typical Nullifire development test of a sample of structural steel coated in intumescent. The sample thermocouples can be seen attached to the specimen which is covered in reacted intumescent char. The important factor in this test would have been the temperature of the steel section which is insulated by the intumescent char. The failure criteria will have been the steel reaching an average temperature of 550° C, which represents the earliest likely point of structural collapse.
3.3.2 Engineering Issues in New Product Development

The high cost of certification means that most companies carry out a preliminary, "indicative" fire test programme to minimise the risk of failing the witnessed test. Indicative tests may also be used to increase the chances of commercial success by optimising the formulation or design of the product. Many passive fire protection companies have their own test facilities specifically so that they can use fire testing as a development method. Nullifire carries out this kind of testing on a routine basis, usually operating the furnace once a day.

However, even fire testing development specimens in a company's own furnace is an expensive process. At Nullifire a figure of £500 per fire test is used to keep track of project costs internally. In addition, the speed at which iterative fire testing can develop products is limited by the number of times a furnace can be used per day. It is difficult to use a large refractory based furnace, like Nullifire's, more than twice per day. As a result it can take many months to develop a product to the point at which external testing can take place. This also limits the number of projects that can be carried out and so the furnace is considered to be the principle development bottleneck.

The company has evolved small scale test rigs to simulate furnace tests but correlation with full scale test results is unreliable. As a result these methods are normally confined to initial screening of ideas and have had a limited impact. Some fire protection companies are making an increasing use of computer modelling to address this problem. Unfortunately, these methods are only really useful when dealing with systems that can be described in a mathematical way, such as the flow of smoke or the expansion of structures under heating. Nullifire's intumescent products make use of the chemical and physical reaction of complex materials in fires.

A conventional starting point in engineering design is the calculation of various parameters that may be used to select suitable materials. Some data that are apparently relevant to fire engineering is available for many materials, but it is not usually actually that useful. Material properties change dramatically as the temperature rises from ambient to typical fire conditions of 900 to 1100°C. Thermal conductivity, for example, is a crucial property in fire engineering but the range of standard values quoted by manufacturers rarely exceed 400°C. This is compounded by the fact that virtually all materials degrade, gradually or rapidly, as they are exposed to flame and high temperatures. This alters properties in an unpredictable way as they change chemically, or crack or simply disintegrate over the course of a fire test.

Manufacturers of standard fire protection materials, like plasterboard and calcium silicate board, provide basic data on the results of standard fire tests. However, it is very difficult to predict likely fire test results when different thicknesses of insulation or different fire test heating curves are used, or when layers of other materials are added. This problem is compounded when the product under development is physically complex. As the furnace rises from ambient to several hundred degrees in a couple of minutes it imposes severe
thermal shock on test specimens. It can be very difficult to predict how
different materials, geometries and joints will expand, or contract, or distort
under these conditions.

Under these circumstances, it is not yet conceivable that accurate modelling
of the kind of products that Nullifire produce can take place. The behaviour of
new formulations can only be determined with any accuracy by fire testing
them by the standard methods. At the beginning of this Engineering
Doctorate this also applied to products constructed from Nullifire materials,
even when the basic fire properties of the materials were known.

3.4 MAKING USE OF EPOXY INTUMESCENTS

Since the Gas Safe project was originally initiated to find a new use for the
epoxy intumescent System E all the early work carried out by the R.E. was
based on that material. This section describes this material in more detail and
outlines the state of the art in the use of System E at the beginning of this
Engineering Doctorate.

3.4.1 Nullifire System E

Intumesce literally means 'to swell', and in the fire protection industry it is
used to describe this reaction under conditions of heat and flame. Some
natural materials exhibit this property, for example some types of mica and
graphite expand when heated because of mechanical stresses locked into
their structure. Most recent intumescents rely on the release of steam, or the
evolution of gas, at high temperatures to create an expanded "char". This
property can be used to produce fire protective coatings that appear similar to
ordinary paint by adding intumescent ingredients to paint resins.

System E is a more complex product because it is formulated to resist the
most extreme fire conditions and also be resistant to weather and mechanical
damage. It is based on epoxy resin because this provides the required
durability, the slight degree of inherent intumescence that epoxies have is
also useful. The epoxy used is a two pack system in which the bulk of the
resin is contained in the "A" pack and the curing agent makes up the "B" pack.
Unlike most paints, which dry to a solid state as the solvents or water in which
their resins are dissolved evaporate, System E contains no solvent and cures
by chemical reaction once the two packs are mixed together.

To become an intumescent, both the "A" and "B" packs of System E are filled
with various powders. A good intumescent requires several different things to
happen in the right order when flame is applied. Firstly, the resin matrix must
soften by just the right amount. Secondly, steam or gas must be evolved in
the right quantities, and at the right rate, to "blow" a foam that is considerably
thicker than the original matrix. Many intumescent ingredients are available in
powdered form, suitable for this type of formulation, but the exact types and
grades of those used by particular companies are secret. Those that
simultaneously absorb large quantities of heat, using it to drive the
intumescent reaction itself, are particularly effective.
Once blown, the soft, thick foam must then be stabilised so that it remains in place and is able to resist the intense heat and erosive effect of flame. This is normally achieved by adding materials that degrade under heat to generate large quantities of carbon. Other materials that form sinter or form ceramics under heat are also sometimes used.

System E also contains a proportion of chopped carbon fibre that improves its mechanical characteristics and the strength of the intumescent char. Unfortunately, it also restricts the char expansion rate so that it only reaches 3 to 5 times its original thickness. Some thin film intumescents expand by over 20 times their original thickness. The insulation provided by a char does depend to a large extent on char thickness, but this was the necessary penalty to be paid for one of the toughest chars of all intumescents. This is particularly important because, as a material formulated for protecting oil platforms, it must resist the highly erosive effect of "jet-fires" of burning gas.

3.4.2 Existing Certification for System E

As has already been explained independent certification is necessary for the sale of fire protection products. The exact type and scope of the certification depends upon the market that the product is intended for. Products for the oil industry need to pass many tests because of the demanding environment they are exposed to. The System E certificates are summarised below:

- Hydrocarbon tests data for structural steel, bulkheads and deckheads for up to 4 hours protection. Certified by Lloyds and Det Norsk Veritas.
- The equivalent certification for the American market, this also includes stringent ageing and durability testing. Certified by Underwriters Laboratories.
- Jet fire test data on various steelwork configurations and for test durations from 15 minutes to 2 hours.
- Blast testing of typical steel elements of construction. Certified by Lloyds.
- Cellulosic fire test certificates to allow the use of System E in ordinary construction at lower, and more competitive thicknesses. Up to 3 hours protection, assessed by Warrington Fire Research Centre.
- Surface spread of flame data with a variety of different overcoats of decorative top-seals. Certified by the Loss Prevention Certification Board.

This certification took several years to complete and cost Nullifire a great deal of money. The most reliable estimate that the R.E. has been able to obtain is in the region of £500,000. It can be seen that for a company with a turnover of around £6 million at the time this was a considerable investment. This goes some way to explaining the pressure to find other applications for the product when sales were disappointing.

However, certification is of limited use outside its intended area where fire conditions and the geometry of the protected item varies from the test specimen. Although the test results may be used for making preliminary estimates, which is often useful, most customers require that solid test data are provided before a sale takes place. It is sometimes possible to obtain
independent "assessments" of performance from independent fire engineering consultants but these professionals are suitably cautious in their approach. In many cases this means that a specific test is required and the cost and time required for this usually frustrates the sale.

3.4.3 Practical Application of System E

To use System E, the two packs of material are normally pre-mixed with a high powered, spiral headed mixer for approximately 10 minutes. It is important to ensure that no unmixed areas remain since they will never cure, or perform properly in a fire. Although System E is considered to be a 100% solids product a small amount of solvent is often added to aid the mixing process. The mixed material is then spray applied using a powerful industrial spray pump at pump pressure of about 50 p.s.i. (tip pressure of around 2800 p.s.i.) and using a large tip orifice of up to 35 thousandths of an inch.

System E was optimised for fire performance and high build characteristics intended to give a functional, industrial finish. To be competitive on large structural steel contracts it is important that application costs are minimised. Therefore, it was formulated to be viscous so that up to 10 mm thickness of material could be applied in a single pass. However, this and the fact that the fibres and fillers sometimes clump, blocking the spray nozzle, means that it is a very difficult material to spray even at high pressures and large tip sizes. The material does not atomise, even at such high tip pressures, and so it is very difficult to achieve an even "fan" when spraying.

As a result of these factors, the final texture does not resemble what is normally understood as a sprayed finish, as can be seen in figure 4. The material actually tends to impact the spray surface as quite large globules, or as "strings" of material. This leads to a porous and very rough finish; a surface texture of 2-3 mm is quite typical. In extreme form this is known as "bird-nesting" because the surface actually resembles randomly scattered, tiny twigs. This arises from the impact of many "strings" of material on the substrate surface that are too viscous to consolidate to form a film.

The rough surface finish is usually improved by rolling it with a solvent dampened roller which gives a smooth, undulating surface and also helps to consolidate the material, reducing porosity – at the surface at least. After this a light "mist" coat of material may be applied to give a relatively even textured surface. The spray characteristics may be somewhat improved by higher levels of solvent addition. Nullifire does not recommend this because excessive solvent retards cure and may affect fire performance.

Specialist spray equipment is available that warms the material, mixes it automatically and then provides a powerful spray capable of atomising most materials. This is known as "plural kit" and has been shown to work well with System E. These machines provide a superior spray finish and give faster curing because the material is pre-heated. Unfortunately, they typically cost £30 - 40,000 which is too expensive for most coating contractors. To date it
Figure 4: A sample of structural steel sprayed with System E.

has also been too expensive to justify for the Coating Services workshop since the throughput of System E work has always been quite limited.

Like all epoxies the gel time and curing rate of System E are heavily dependent on ambient temperature. Pot life, or working time, of the mixed epoxy is usually slightly under one hour. However, the curing reaction is exothermic and the action of the pump adds heat so the reaction can be accelerated significantly. Since a mixed kit can normally be sprayed in less than 15 minutes this is not usually a problem, although the material does occasionally cure in the pump or feed lines.

Despite this, the material takes a long time to cure at 10-20°C, normally 24 hours is allowed before the surface can be worked or a second coat applied. On a large structural steel contract this is not a problem since it is convenient to work in phases, returning to the same area much later. However, cold temperatures slow the cure rate dramatically. At 5°C it may take a week before reasonable cure is achieved, below that temperature, cure will not take place at all. In addition, even small quantities of water will retard or prevent cure so rain, high humidity and surface condensation must be strictly avoided.

These difficult characteristics were judged to be acceptable for a heavy-duty industrial coating eight years ago, when it was formulated. In fact, System E was generally regarded to be more user friendly and easier to spray than most of the competing epoxy intumescent.
4. THE STORY OF THE GAS SAFE PROJECT

4.1 THE COURSE OF EVENTS

Historically Nullifire has mainly sold into construction and the company has experienced the effects of each recession on this industry. The company also holds a number of valuable defence contracts, but winning more has proved difficult. In the mid 1990's part of the company strategy was to diversify into other markets to reduce risk and expand the business more rapidly. Several months before the beginning of the Engineering Doctorate a brainstorming session was held to identify unexploited opportunities, using the company's existing materials and skills.

The objective of the session was to find an alternative use for the System E epoxy intumescent which was experiencing disappointing sales. The ubiquitous domestic LPG cylinder was identified as being highly vulnerable to fire, based on anecdotal evidence from fire fighters known to the Managing Director. It was also known that no way of fire protecting was offered on the market and so the entire cylinder stock was completely unprotected from fire. A project to develop an enclosure to protect portable Liquid Petroleum Gas (LPG) cylinders from the effects of fire, using System E, was launched in May 1996. This was designated the "Gas Safe" project.

4.1.1 Internal Development and Fire Testing

The original specification for the Gas Safe assumed that the fire protection would be achieved by spray applied System E epoxy intumescent. The required period of protection was given as 15 minutes for domestic "gas bottles". The possibility of coating the cylinders directly was quickly eliminated because they are owned by the LPG distribution companies, and they were hostile to the idea. Instead a simple, removable steel enclosure for cylinders, coated with intumescent, was conceived. The R.E. began a programme of experimental fire testing by building prototypes and burning them in a furnace, this is described in detail in submission 8.

After an intensive programme the 15 minute target was reached in October 1996. It was achieved by combining the epoxy intumescent with a thick layer of cheap fire resistant foam insulation. By this point the project had drifted some way from the original assumption that a coated steel enclosure would be sufficient. However, it was decided to take advantage of the properties of the System E and foam composite and increase the target period of protection to 30 minutes. Following further intensive testing this was also achieved in January 1997. The natural rigidity and strength of System E also allowed the elimination of the heavy, fabricated steel shell, which reduced the cost considerably.

At this point an informal review of the prototype concluded that its appearance needed to be improved greatly. The rough, sprayed finish and the basic cylindrical shape were identified as being unappealing to the intended consumer market. To improve this the shape of the prototype was changed
into a cylinder with a profiled lid that fitted snugly over the top of the gas cylinder. It was more difficult to improve the finish of the prototype because the epoxy material was difficult to spray, and hence very rough. Attempts were made to mould the material but it was found that the high viscosity meant that pressures were too high for the simple and cheap tooling that was available.

This problem was solved by spraying the material onto the inside of open moulds made from thermoplastic sheet or vacuum forms. The moulds were designed so that this created a smooth finish on the side of the moulding that became the outer surface of the Gas Safe. It was also possible to apply simple in-mould painting techniques so that the de-moulded component was immediately ready for assembly. These new methods allowed the construction of attractive prototypes with a 30 minute furnace performance by December 1997. Alternative insulation materials and foams were also tested at this time to improve the strength and reduce the cost of manufacture. A glass reinforced phenolic foam was selected in February 1998 and used in all future Gas Safes.

4.1.2 External Testing

At this point the Gas Safe was very close to its final form and had been shown to perform extremely well in furnace testing. However, in the fire protection industry some kind of independent testing, preferably to an acknowledged standard, is necessary before a product can be sold. This was difficult in the case of the Gas Safe because there was no formal test standard for this kind of product. Investigation by the R.E. found that researchers in the field normally used pools of hydrocarbon fuel, propane burners or cribs of wood burning in the open air to test potentially explosive items.

The R.E. discussed possible test methods with three different testing centres, including the H.S.E. laboratories at Buxton who have carried out numerous tests on LPG cylinders and vessels before. It was found that all three centres could perform the required “pool fire” testing but the fire testing facility at DERA Porton Down was finally selected on the basis of cost.

The initial pool fire test was carried out on a partially full propane cylinder inside a standard Gas Safe. The fire was fuelled by aviation fuel and engulfed the cylinder completely for over 40 minutes. The Gas Safe protected the cylinder for 40 minutes, at which time venting from the pressure release valve occurred in a safe manner. The results clearly showed that the cylinder was in absolutely no danger of exploding at any time during the test. This test is described in detail in the third submission “Gas Safe Pool Fire Test”, and the fifth also gives some additional information.

Now that the Gas Safe had met the technical specification the company's attention turned towards the marketing of the product. The first part of this process was to gain official certification for the product, and this was the responsibility of the R.E. To do this it was necessary to establish a recognised test standard in some way. The Loss Prevention Certification
Board (LPCB) were very supportive of the Gas Safe concept and agreed to write a new standard to cover the testing of this type of product. The standard was based very closely on the testing carried out by R.E. at Porton Down. This process is described in more detail in submission 13, “Information in Support of the Gas Safe Project” and the standard is reproduced in the appendices to that submission.

Figure 5: Pool fire test of the Gas Safe, after 20 minutes.

Despite this success no LPCB test was ever carried out to this standard. This was because an official test would have cost two or three times the cost of an indicative pool fire test. The R.E. realised that a number of design changes would probably be necessary to meet the needs of particular customers, as yet unidentified. This would probably have meant that a test specific to that design would have to take place. Therefore, it was decided that official testing would only take place when a firm customer for a large number of Gas Safe units had been identified.

Nevertheless, another four indicative pool fire tests took place at Porton Down, all of which confirmed that over 35 minutes protection could be expected. Three of these tests were carried out for the purposes of publicity with various television broadcasters. The fourth test was on a much larger Gas Safe containing a 47 kg propane cylinder. The success of this test showed that the concept could be applied across the range of portable butane and propane cylinders.

In addition the Swedish Fire Brigade carried out their own test and several tests on Gas Safe variants were carried out by independent test centres in France. These tests are described in more detail in submission 13, “Information in Support of the Gas Safe Project”.
4.1.3 Sales Activities and Pilot Production

The sales and marketing activities carried out are described in some detail in the tenth submission, "Analysis of the Marketing of the Nullifire Gas Safe". This describes a great deal of promotional activity, including high profile press and television coverage. This generated a great deal of interest and there were many high quality enquiries for a period of over a year, reaching several per day at the peak of activity. These leads were from a diverse range of LPG users and, as expected, their requirements were usually somewhat different to the existing Gas Safe.

Pilot product of 13 kg Gas Safe units began soon after the first successful pool fire test. The company decided to postpone a redesign for manufacture exercise to avoid delaying the availability of pre-production samples. A total of 50 pre-production Gas Safes were manufactured by the Coating Services department, under the guidance of the R.E. All the units were hand built in the same way as the prototypes and fire test specimens so that expenditure on tooling could be avoided until a firm, final design was reached.

The existing Nullifire sales network was used to convert the leads generated by the publicity into sales, at a price of £150 to £200. Most of the 50 units were sold, usually to companies interested in acting as distributors rather than individuals. Most of the rest were distributed free of charge to Nullifire agents world wide as sales samples.

4.2 PROJECT ACHIEVEMENTS

4.2.1 Fire protecting LPG cylinders

Detailed information about LPG cylinders and their behaviour in fires is given in submission 13, "Information in Support of the Gas Safe Project". It will not be repeated here, except in so far as it is necessary to explain the difficulties involved in protecting them from fire satisfactorily.

The design and manufacture of LPG cylinders is tightly controlled by national standards. In the U.K. they are all fabricated from welded mild steel, a typical domestic cylinder has a minimum skin thickness of about 2.3 mm. In construction this would be regarded as highly vulnerable to fire, even if the failure temperature were to be as high as the 550º C typical for structural steel fire protection.

A method for determining the amount of insulation needed to fire protect structural steel has existed for many years and is widely used. The cross-sectional area, 'A', of the steel is a useful measure of the thermal mass of a section. The proportion of the steel exposed to the fire is considered as the 'heated perimeter', or 'Hp', of the cross-section. The ratio Hp/A, also known as the "section factor", indicates how quickly the section will heat up and how vulnerable it is to fire.
If this principle is applied to a typical domestic LPG cylinder the $H_p/A$ may be calculated from a wall thickness of 2.3 mm and a diameter of 320 mm to give a $H_p/A$ of 438 m$^{-1}$. Nullifire has not carried out any tests at section factors this high and would normally politely decline any such enquiries from customers. The highest $H_p/A$ certified for protection by System E is 320 m$^{-1}$, which equates to a steel skin thickness of over 3 mm.

However, this is actually only a measure of the rate of heating of a sample and so the failure temperature and the period of protection required must also be considered. LPG cylinders have two basic failure modes that must be considered and these give two widely different failure temperatures.

**Venting**

As described in previous submissions, the pressure inside LPG cylinders is highly temperature dependent. Storage pressure for propane is about 8 Bar at normal ambient temperature, but exceeds 25 Bar at about 70°C and rises steeply beyond that. Although cylinders are designed to a safety factor of 4, they cannot cope with the pressures generated by temperatures in fires.

To prevent explosion, cylinders are fitted with pressure release valves (PRVs) that open at about 25 bar. According to Calor Ltd. this equates to a bulk propane temperature of 630°C. Although this is a safety device the jet of gas usually ignites in a fire and can spread the fire rapidly or be a danger to fire fighters, as shown in figure 6. Therefore, an internal cylinder temperature of 630°C must be taken to be the first failure criterion.

![Figure 6: The reaction force of a 5 m long vent of flame spins a propane cylinder into the air after only 5 minutes exposure to a small fire.](image-url)
The fact that the cylinder is filled with LPG does have a positive effect because the contents have a significant thermal mass. A full cylinder is made from about 13 kg of steel, with a specific heat capacity of 473 J kg\(^{-1}\)°C\(^{-1}\), and will contain 13 kg of propane, with a specific heat capacity of 2430 J kg\(^{-1}\)°C\(^{-1}\). It can be seen that the thermal mass of the propane is actually about 5 times as significant as that of the steel skin. However, cylinders may be nearly empty and still contain enough LPG to generate dangerous pressures. Therefore, the worst case principle demands that assumptions, calculations and the level of fire protection are based around empty cylinders.

A simple calculation may be carried out to estimate how long a cylinder may take to reach the failure condition of 63°C.

\[
E = S \times M \times \Delta T
\]

Where:

- \(E\) is the energy required to heat the cylinder to 63°C,
- \(S\) is the specific heat capacity of steel (473 J kg\(^{-1}\)°C\(^{-1}\)),
- \(M\) is the mass of the steel cylinder (13 kg),
- \(\Delta T\) is the temperature rise (48°C, assuming an ambient is 15°C).

The energy required to evenly heat a full propane cylinder to failure point is about 1811 kJ, and that required for an empty cylinder only 295 kJ. Fully engulfed by a fully developed cellulosic fire a cylinder would be exposed to a heat flux of around 50 kW m\(^{-2}\). Under these conditions even a full cylinder would reach 63°C in only around 72 seconds. In reality, fires take at least several minutes to develop and would not fully engulf a cylinder in early stages so the heating conditions would actually be much less severe.

These rough calculations compare quite well with actual test data. An early furnace test showed that an empty cylinder would reach this point after only 2 minutes exposure. The R.E. later carried out several live pool fire trials on full cylinders and witnessed others. These showed that small fires, not much larger than a domestic barbecue, could cause venting conditions to be reached very quickly. Times to first vent, even on full cylinders, could be reached in only just over two minutes and were rarely as long as 5 minutes.

When compared with Nullifire's usual structural steel, a LPG cylinder is extremely vulnerable to heat and also has a very low failure temperature. This made the task of keeping it safe for any length of time, let alone 30 minutes, a difficult fire protection task.

Boiling Liquid Expanding Vapour Explosions (B.L.E.V.E)

Under severe conditions, as described in submission 13, "Information in Support of the Gas Safe Project", a PRV is not able to prevent explosion. When a LPG vessel ruptures the high pressure ensures that it will be a violent event, as shown in figure 7. The biggest hazard is usually the projection of sharp fragments of the steel vessel that can travel over a hundred metres with
great force. Vessels are also sometimes propelled great distances as high speed 'rockets' by escaping gas, depending on the conditions at the time of failure.

B.L.E.V.E. may occur if a region of the cylinder skin becomes so hot that it begins to weaken and cannot contain the 25 Bar pressure that the PRV maintains. It may also happen if the rate of heat input into the cylinder is so high that the rate of flow from the PRV cannot prevent the pressure from rising. Obviously, these two factors interact and, unless a cylinder is well instrumented for pressure and temperature, it is difficult to determine which causes a particular failure. Other conditions that may occur in fires can also cause or contribute to B.L.E.V.E. Impact damage, following explosions, or flaming jets from other cylinders can overheat or weaken spots on cylinders fatally.

Figure 7: BLEVE of a 13 kg propane cylinder during a Nullifire test, the fire ball is approximately 10 m high and over 20 m wide.

Although these factors are complex, it can be assumed that, provided the PRV is operating correctly, B.L.E.V.E can be prevented if the steel skin of the cylinder is kept reasonably cool. It can also be assumed that a thermally protected cylinder will never experience the intense heating that might overload the PRV. Precisely what the limiting temperature for the steel skin should be is debatable but the R.E. suggests that a limit of 400° C is used. Steel in tension retains around 70% of its relative strength at this temperature and the safety factor of the cylinder ensures that it should easily be capable of withstanding 25 Bar.

In comparison with delaying venting, ensuring a cylinder does not experience B.L.E.V.E. is relatively easy. Approximately eight times as much energy is required to raise a cylinder to 400° C compared to the point of venting.
Therefore, by specifying that the Gas Safe delay venting by 30 minutes it became certain that it would also be safe from B.L.E.V.E. for much longer.

Complicating Factors

A number of practical considerations, outside the scope of the specification, complicated the Gas Safe project.

1. LPG cylinders are owned by the distribution companies, whether a deposit is paid or not. Therefore, any fire protection solution must be entirely separate from the cylinder, not require any modification to the cylinder or alter its normal operation in any way.

2. Different LPG companies use slightly different cylinder sizes and designs. This became an important factor in the design of an enclosure intended to suit all cylinders of a given size in the U.K.

3. As gas is drawn off a cylinder more liquid evaporates to replace it. This requires energy, reducing the temperature of the cylinder which gradually absorbs heat from its surroundings to replace it. Thermal insulation intended to protect a cylinder from fire also tends to inhibit this important process.

4. Gas cylinders are heavy, robust objects and they tend to be treated quite roughly. Insulating materials and intumescent coatings tend to be quite fragile and careful design was necessary to maximise the toughness and durability of the Gas Safe.

4.2.2 Advances in Insulation

The first few furnace tests of the Gas Safe concept revealed that the concept of using a thin coating of System E on a sheet steel enclosure did not provide nearly enough insulation. Calor Gas Ltd had provided information that LPG cylinders would begin to vent when the internal temperature reached 63°C. Since testing cylinders containing LPG would have been far too dangerous, the temperature of the air inside an empty cylinder was taken as a worst case equivalent. However, the concept design reached this failure criterion after only about six minutes.

Over the seventeen minute period of this test the average temperature of the steel cylinder rose from 33°C to 219°C, therefore ΔT is equal to 186°C. The mass of the steel cylinder was 13 kg and the steel shell of the gas safe was a further 6 kg. The specific heat capacity of steel, S, equals 473 J kg⁻¹ °C⁻¹.

The energy input, E, over this period can be calculated using equation (1):

\[ E = 0.473 \times 19 \times 186 \quad = 1671 \text{ kJ over 17 minutes}. \]

This is equivalent to an input of 1639 watts of heat through the surface of the Gas Safe, about 0.9 m², throughout the test. Clearly, the intumescent char
did play a significant role because a later test carried out on a bare cylinder reached an equivalent point at around 4 minutes.

However, it was clear that a significant advance in the amount of insulation provided by the Gas Safe would be needed, even to meet the initial specification of 15 minutes to failure. The obvious solution of increasing the thickness of System E was made much less attractive by the additional cost; the cost price of the material to Nullifire was around £3/kg at the time. Over the surface area of the Gas Safe, at a thickness of 4 mm, this equated to about £12.00. Therefore there was some reluctance to increase the thickness. Later in the test programme, a test on a 6 mm thick coating was carried out and found to give a performance boost of only three minutes for an additional cost of £6.00.

The use of foam insulation was suggested by the R.E. because of its low cost and weight and its reputation as an efficient insulation material. A fire resistant grade of polyurethane foam was chosen after promising indicative tests using a propane burner on small plate samples. The addition of foam insulation proved to be effective and after many tests it was found that 30 mm of foam and 4 mm of System E provided comfortably over 30 minutes protection. The performance of the Gas Safe had been improved from 6 minutes to 34 minutes after five months work and 22 tests.

![Figure 8: Time to failure, concept Gas Safe compared with best furnace test.](image)

The steel cylinder inside the 22nd Gas Safe reached 98° C after 41 minutes, a temperature rise of 81° C. If the calculation carried out above for the original Gas Safe is repeated, the improvement in insulation is evident. The fact that
the fabricated steel shell of the Gas Safe had been eliminated must be taken into account, so the mass of steel is reduced to 13 kg. Using equation (1):

\[ E = 0.473 \times 13 \times 81 = 498 \text{ kJ over 41 minutes.} \]

This is equivalent to an input of 202 watts of heat through the surface of the Gas Safe throughout the test, an eight-fold reduction on the original result. This was achieved by the addition of only £8 of foam. The elimination of the steel shell actually saved much more than that, approximately £25, and also reduced the weight from 15 kg to just under 10 kg.

Achieving the same result by using System E intumescent alone would have been much less satisfactory. Although no tests were carried out, it is possible to make a rough estimate from existing fire test certification:

The 127 mm x 76 mm x 13 kg universal beam has the same mass of steel as the LPG cylinder in these tests, and a lower surface area which makes it less vulnerable. The minimum failure temperature in the System E cellulosic certification is 400°C. It can be calculated that a steady heat input of 202 watts would take just over 190 minutes to reach this point. The certification shows that this equates to a beam protected with 30.5 mm of System E.

This calculation is approximate because it assumes steady conditions, which is not the case, and uses a different failure temperature. However, it was the most accurate calculation that could be made from the certification at the time. It is also quite adequate at demonstrating that System E alone could never be as efficient as the new material combination. A Gas Safe constructed of System E at a thickness of 30.5 mm would require 30 kg of material, which would cost £90.

When foam insulation was originally proposed it was accepted that although it might be a cheaper and lighter solution, it would probably be quite bulky because of the thickness of foam needed. It was accepted that this would be a reasonable compromise because of the three factors, a small increase in overall size was the least important. However, it can be seen that a pure System E solution could actually have been nearly as large as well as much heavier and much more expensive.

Once the effectiveness of polyurethane foam insulation had been demonstrated, several alternative types of insulation were tested. These included cellular foamed glass slabs, plaster mixtures containing high levels of chemically bound water and a blown phenolic foam. This was because, apart from the value of testing the effectiveness of other materials, the polyurethane foam was not ideal. The fact that it was flexible and compressible meant that the thin shell of System E on the outside of the Gas Safe was effectively unsupported and vulnerable to damage.

Many other materials, selected on the basis of low cost and availability, were discussed, but were dismissed for various reasons. For example, ceramic
fibre based insulation provides excellent fire performance but raises serious health and safety issues. Mineral fibre also performs well in fires and is cheap and plentiful, but becomes water logged easily. Many other materials were deemed impractical because of the difficulty in obtaining them in tubular form or processing them to reach that shape.

Ultimately, a glass reinforced blown phenolic foam was selected because it was rigid and strong and achieved nearly the same fire performance as the polyurethane. It had the additional advantage of being mouldable so that it could be fashioned into the tubular Gas Safe sleeve and profiled lid relatively easily. The phenolic foam and System E Gas Safe was pool fire tested several times and achieved over 30 minutes to first vent on every occasion. This confirmed that the performance of the insulation met the enhanced specification under the most severe fire conditions that a domestic cylinder was likely to encounter.

Figure 9: Section through the lid of a fire tested Gas Safe showing condition of char and insulation.
4.2.3 Comparison with Other Solutions

Various groups around the world have been fire testing LPG vessels to improve safety for many years, as summarised in submission 13, "Information in Support of the Gas Safe Project". Until recently this work has mainly concentrated on the larger LPG vessels such as rail and road transport vessels and bulk storage tanks. This is where the danger is greatest, as shown by numerous incidents and various solutions including water deluge systems and various insulating coatings, including intumescent. However, the primary method of protection is the same as for mobile cylinders, the pressure release valve.

Much less testing was carried out on the fire protection of smaller cylinders although it is known that various intumescent solutions have been tested before. A twin skin cylinder that is cooled by gas, released from its own PRV, flowing between the skins has also been tested. However, no practical method of fire protecting mobile cylinders was commercially available. Over the period of the Engineering Doctorate the amount of testing carried out has increased and a variety of different solutions have been proposed as shown in figure 10:

- Cementitious coatings. These are widely used in structural fire protection and are cheap and effective. However, they do require a large thickness to be effective, have a very rough finish and are vulnerable to damage.

![Figure 10: Different methods of fire protecting automotive LPG tanks, including Nullifire System E.](image-url)
- Ceramic fibre contained in a woven silica fabric sock. This is an effective insulation material but is not ideally suited to low temperature insulation and is surrounded by health concerns. It is also bulky and vulnerable to damage.
- Mineral fibre insulation in a fabricated stainless steel jacket. Another effective solution, but it is bulky and the low cost of the insulation is balanced by the cost of the jacket.
- Fire resistant silicone on an insulating fabric liner. This solution diverged from a Nullifire collaboration with another company and originally used System E. However, although the solution was compact and effective, the epoxy was judged to be too brittle. It is not known whether the new material is as effective.
- Direct coating of cylinder with intumescent. This method has the advantage of providing good insulation from a coating only a few millimetres thick, and if epoxy intumescents are used the coating is tough and durable. However, the amount of insulation provided by intumescent is limited as has been discussed in earlier sections. Nevertheless it is sufficient in some applications and Nullifire has proposed this solution at times and has current projects exploiting this option, as described in submission 13.

Only one other group that the R.E is aware of has tested foam and intumescent, although not in combination. This group, from the All Russian Scientific Research Institute for Fire Protection [2], achieved reasonable results, delaying venting until 20 minutes using 5.5 mm of intumescent. They also tried layers of foam up to 100 mm thick, and delaying venting for over 40 minutes. However, this group did not experiment in combining intumescent and foam and so did not achieve results that compare with the Gas Safe.

It is possible that there are also other approaches that the R.E. is unaware of, but none have achieved commercial success to date. Although suppliers and users demonstrate an increasing awareness of the problem, the favoured approach remains the alteration of the cylinder itself. The more modern standards relating to LPG vessels show that the flow rates from PRVs are increased and the use of thermal fuses is also sometimes recommended.

4.3 EXPERT OPINION ON THE GAS SAFE

4.3.1 Independent Opinion

The Gas Safe concept was first tested by independent opinion when the Loss Prevention Certification Board were approached by the R.E. This was done because the Gas Safe needed certification of some kind from an independent authority. The LPCB were very supportive of the concept since, from the point of view of the insurance industry, LPG cylinders frequently increase fire losses. Their most favoured application was in protecting cylinders on construction sites, particularly those used to fuel burners melting asphalt on flat roofing.
The LPCB agreed to write a new Loss Prevention Standard to allow products like the Gas Safe to be tested and approved. The engineers who wrote this standard were sufficiently impressed by the severity of the Nullifire pool fire, and the performance of the Gas Safe, to base the standard on it. The design of the pool fire rig, fuel fire specification and method of instrumentation all remained unaltered. They added durability and wear tests intended to prove that the Gas Safe would survive in service.

The Gas Safe was also tested by expert opinion in two public arenas. The R.E. was invited to demonstrate the Gas Safe at the 1999 Swedish International Association of Arson Investigators Conference and supervised a pool fire demonstration there in front of 50 delegates. The Gas Safe sample performed well, protecting a cylinder for a total burning time of 30 minutes. The experienced firemen, police arson investigators and insurance representatives were quite surprised and impressed by this. Some did not actually believe that the cylinder had not vented inside the Gas Safe until they were able to inspect it the following morning. The Gas Safe also won an award for "excellence and innovation", judged by a panel of industrialists and experts on fire, at the 1999 International Fire Exposition.

Since the Gas Safe concept originated from information given by fire fighters it was logical to ask their opinion once it was complete. As soon as the company was confident that it would perform in real fire conditions, it was presented to them for assessment. It was well received at the London Fire Brigades "Beyond the Cause" conference in May 1999 and plenty of useful feedback was received. Warwickshire Fire Brigade were invited to Nullifire to inspect the Gas Safe and review the test evidence demonstrating its performance. They were impressed at the safe time period that it would provide fire fighters and authorised Nullifire to use their logo on promotional literature to show their support.

4.3.2 Collaborations and Customer Involvement

Probably the best measure of the soundness of the Gas Safe concept and its performance has been the interest from major LPG suppliers. Although LPG distributors were originally hostile to the concept, and some have remained so, other companies have been far more positive. One major supplier has attended two of the Gas Safe pool fire tests and spoken in support of the Gas Safe in a television interview. Their senior fire advisor also planned a joint fire test programme with the R.E. but later withdrew because of concerns from his company's marketing department.

Another supplier has gone much further as described in section 4.6 of submission 13. Normally, fire regulations require that cylinders on sale are stored in cages outside, away from combustible materials. However, this company has been able to negotiate new regulations for fire protected cylinders that will allow them to be sold "off-the-shelf". This company believes that it will gain a significant market advantage over its competitors by supplying LPG cylinders through ordinary shops like any other commodity. This company approached Nullifire after hearing about the achievements of
the Gas Safe programme. This joint development project has now been running for over a year and the R.E. has tailored a solution to match the new requirements, which will be described further in section 6.2.

Collaborations have also taken place with other companies who are interested in exploiting the technology developed in the Gas Safe project. Many of these have taken place within the burgeoning automotive dual fuel industry that installs propane cylinders into vehicles. This has included a joint development with a French company and several successful fire tests at the French CNPP fire test centre. However, the most crucial project to date has been with a car manufacturer and this remains active, although it progresses slowly.
5. THE BENEFITS OF THE GAS SAFE PROJECT

The Gas Safe project generated quite significant publicity and probably improved recognition of the Nullifire brand to some extent. More tangibly, there has been widespread interest in the technology employed in the project and this has created a large number of specialist fire protection enquiries over the last two years. Some of these ultimately led to development projects and successful products, which will be discussed further in section 6.

However, most significant in the long term will be the practices and technologies that have been introduced to the company over the course of the project. Over the last two years it has become apparent that the improved capabilities of the Coating Services division allow a wider range of more demanding and profitable projects to be undertaken. This section summarises these improvements and explains their origins and potential.

5.1 NEW METHODS OF TESTING AND ANALYSIS

5.1.1 Traditional Development

The R.E. has frequently heard the opinion that successful design of fire resistant products is "an art" and that experience is more valuable than tables of data or engineering calculations. Certainly, in the past new product development at Nullifire has generally relied upon the construction and destructive testing of a series of prototypes. Although this appears to be a quite effective way of tackling simple problems, it does tend to result in over engineered products. Such solutions tend to be based on well-known materials and technologies and so are rarely well optimised. The early work undertaken on the Gas Safe, summarised in section 4, followed this approach. Despite the technical success of this work it was found to be a slow, expensive process, as described in the 11th submission.

Clearly it would be preferable to be able to make use of engineering principles to carry out calculations to inform the early design process and reduce the number of physical prototypes and tests needed. In the case of the Gas Safe project it is possible to model the problem as a simple thermal mass (the steel cylinder), with a given surface area exposed to a fire with a known heat flux. Once this simplification has been carried out it initially appears quite straightforward to calculate how much insulation is needed to keep the thermal mass below a given temperature for a chosen period of time.

Unfortunately, the problem is complicated somewhat by the fact that most insulation materials will degrade substantially when exposed to the heat of a fire. It can be difficult to calculate the effectiveness of even suitable high temperature insulation materials because material properties and thermal conductivity vary greatly over the range of temperatures under consideration. Although manufacturers often quote values for thermal conductivity this is normally only at a single temperature, or rarely across a limited range, which is not useful when considering the extreme temperatures found in fires.
5.1.2 Modelling heat flow through porous materials and chars

However, much work has been done to model the thermal conductivity of composite solids such as porous chars. This has been carried out for a wide range of reasons including the modelling combustion of carbon particles, estimating the thermal conductivity of UO₂ pellets, improving the insulation properties of building materials and understanding the behaviour of char forming plastics and their fire retardant properties.

Dawson and Briggs [3] carried out a review of methods for predicting the thermal conductivity of insulation materials. They divided the approaches into three basic groups:

a) Flux law models,
b) Ohm’s law models,
c) Empirical relationships.

Flux law models make use of Fourier’s fundamental heat conduction law. This states that the heat flux between two points is proportional to the temperature gradient between them. The multi-phase characteristic of insulation materials is accounted for by considering the volume fractions of the different phases and their particular conductivities. The drawback of this approach is that the model is very dependent on the geometry of the phases. A typical model is of spheres distributed in a matrix but this does not lend itself to the complex geometry of real multi-component materials.

Ohm’s law models use an electrical analogue of a system of resistances. These assume one dimensional heat transfer through unit cells of material that may be built up to reflect the structure of material being considered. For example, a three element parallel resistor may be constructed by adding blocks of poor and good conductivity in series, a block of poor conductivity and a block of good conductivity. The resulting equation may be weighted to model the proportion and structure of the different phases. Alternatively, if a random arrangement of the phases is assumed it is possible to use a geometric mean equation.

There are also many different equations that make use of an empirical relationship to describe the effective conductivity of a composite solid. These are normally derived from the known volume fractions and conductivities of the constituents and often modify an equation derived from a model to give better fit with empirical data.

Dawson and Briggs note that all these methods neglect radiative and convective heat transfer and therefore limits their application to situations where these components are negligible. However, they quote empirical evidence that convection may be neglected in insulation materials and that the contribution of radiation is negligible for small pore sizes. They found that the geometric mean equation was most suited to use in a computer program and used it to predict the conductivities of a variety of materials. They found
good agreement with measured conductivities for stable materials under stable temperature conditions.

Bakker [4] employs the Finite Element Method (FEM) to calculate the conductivity of complex porosity structures. It makes use of a two dimensional (2D) photograph of a section through the structure which is potentially far more accurate than a geometrical model based on simple shapes. The geometry of the photograph is transferred into a FEM program that computes the 2D conductivity from the 2D thermal flux profile.

Bakker also takes account of the fact that the extra degree of freedom of the flux in 3D reality will cause the conductivity to be higher than in the 2D representation. He uses the 2D conductivity as a lower bound and the conductivity of a matrix containing spheres as an upper limit to develop a relationship between the 2D and 3D conductivity. This allows the 3D conductivity to be computed from the 2D conductivity generated by the FEM.

Apart from the use of photographic sections this work is theoretical and no comparison with experimental data is made. Nevertheless, this type of method could certainly be used to compute the conductivity of an intumescent char structure. It would take account of the complex shape of a typical char structure by using photographs of representative sections of char in a FEM program.

Staggs [5] notes that for a porous material consisting of a solid matrix and holes it is straightforward to calculate the specific heat capacity and density of the whole. The thermal conductivity is far more difficult, although it is possible to calculate upper and lower bounds, providing the thermal conductivity of the individual phases and the degree of porosity is known. He states that in the Fire Science literature the upper bound is normally used but that this will significantly overestimate the effective thermal conductivity if the ratio between the thermal conductivities of the phases is large and if the porosity is significant. Intumescent chars certainly fall into this category, with expansion ratios generally falling between 4 and 100.

Staggs uses a network of resistor elements to model heat transfer through a porous material. A unit cube is approximated using the classical 3D network of six resistors and one central node. The thermal conductivity of each resistor is either equal to that of the solid matrix or the hole and this is determined randomly based on a probability depending on the porosity of the whole. The network is built up from a large number of these elements, 45,000 in the case of the results presented. The effective thermal conductivity, $k_{eff}$, is calculated by iteration and between 100 and 3000 iterations are typically required for a solution, depending on the porosity. A comparison with experimental results for alumina with spherical inclusions is made and compares reasonably well for porosities below about 0.3.

Unlike Bakker, this approach cannot take account of complex pore structures but is three dimensional. Staggs shows that Bakker's formulae for calculating 3D from 2D is only really valid for porosity up to about 20%. The UO$_2$ pellets
that Bakker was working with fall into this range but intumescent chars are generally much more porous than this. This is unfortunate because Bakker's method can apparently model the subtleties of complex char structures, whereas the resistor network method cannot.

The porosity of System E char is generally assumed, since it has never been precisely measured, to be about 0.8 from the expansion ratio of 5 for properly developed char. This falls into the region of porosity that needs the maximum number of iterations to reach a solution using Staggs method. It is also the region where $k_{\text{eff}}$ changes by two orders of magnitude from porosity ranging from 0.6-0.8. This implies that we should expect a huge difference in the performance of System E char as an insulator depending on its degree of expansion. To some extent this certainly true, in that a well expanded char with an assumed porosity of about 0.8 performs much better than one that has expanded poorly, perhaps with a porosity of only 0.3. However, there is no evidence to suggest that small differences in expansion have a large effect. This may because $k_{\text{eff}}$ is only one factor in determining the performance of an intumescent system, thickness of the char is equally important and the endothermic reactions are also significant.

Clearly, the conductivity of the individual phases must be known for these approaches to be useful and this would require accurate measurements to be made. However, the main drawback of this type of method would be that it can only be applied to a fully developed and stable char. It must be noted that even once a char has completed its expansion it will continue to increase in porosity, as organic materials slowly burn out, and that the thermal conductivity of the polymer based solid phase will alter as it undergoes pyrolysis.

Kantorovich and Bar-Ziv [6] present a detailed review of heat transfer mechanisms for highly porous chars. They consider chars to be porous materials that are formed by the removal of elements from the original structure by combustion. This is different from the formation of an intumescent char by foaming but the end result appears to be similar in many respects. They comment that most previous models consider the internal structure as overall fractions of different phases and that while this is appropriate for non-consolidated porous materials, chars actually consolidated with finely contacted solid grains. This means that the detail of the structure will have a strong effect on thermal conductivity.

They also deal with the relative importance of convection, radiation and conduction as mechanisms of heat transfer in porous media. Since heat transfer by convection can only play a significant part if the circulation of gas within the pores is intensive they discount it for pore sizes below 1 cm. This would include good intumescent char structures. They state that radiation can be a significant mechanism if the solid is a weak conductor and the pore sizes are large but that it can be neglected for the majority of carbonaceous materials for temperatures below 1000 K. Using these criteria it is not clear whether radiation would be significant in an intumescent char or not, not least
because temperatures in the outer layers would exceed this limit in most fire tests.

The rest of the work presented by Kantorovich and Bar-Ziv deals mainly with conduction mechanisms within crystals and inter-crystal joints and is no immediately relevant. However, they do describe two methods by which the thermal conductivity of 100 \(\mu\text{m}\) particles can be measured. These methods could be a way determining the thermal conductivity of particles of intumescent char. This could provide a figure for the "solid" phase, actually microporous in reality, of a macroporous intumescent char.

The problem of determining thermal conductivity is more extreme in the case of intumescents because the situation is dynamic. Such materials react dramatically when exposed to fire, increasing in thickness and changing structure to become much better insulators. The work of Anderson and Wauters [7] is more directly relevant because it concerns the dynamic behaviour of intumescents and seeks to model the insulation they provide. They follow the original work of Clark et al. [8] and propose a one dimensional thermodynamic heat transfer model developed by application of equations of conservation of mass and energy. They also define that the crucial expansion factor is a function of the quantity of outgassed material.

Data on the loss of mass was obtained by thermogravimetric analysis and then represented using a Fourier series. A differential scanning calorimeter to was used to provide information on the rate at which the coating absorbed heat through endothermic reactions such as phase changes. This information was used in the numerical analysis as a direct function of temperature.

The calculation was carried out by dividing the coating into zones, each of which is represented as a grid point. The equations are applied to each point and the model is moved forward in time using the known values from the previous time step. Since the grid expands with time extra zones are added when appropriate and the new grid values calculated.

The model was used to predict the behaviour of a sample of intumescent on a small steel plate exposed to the heat of a furnace. The model is in general agreement with the rate of temperature rise and can predict the basic shape of the experimental substrate heating curve. However, it is far from precise and is only applied for a very short time period of three minutes. This is a result of the application, protection of munitions against brief fuel fires, but suggests that it is mainly modelling the absorption of heat by chemical processes during the formation of the char rather that heat flow through a developed char.

Buckmaster et al. [9] propose that instead of the coating foaming throughout its thickness, it only actually foams in a very narrow region of the coating, a "front" that moves from the coating surface to the substrate in time. They correctly state that Anderson and Wauters' assumption that the degree of intumescence is directly related to mass loss is simplistic because the coating must be in the right viscoelastic state when evolution of gas takes place to
allow foaming. Experimental evidence does indicate that foaming takes place in a limited band of coating that moves in time, but defining a very narrow band is perhaps a little extreme.

However, it is convenient because it allows the coating to be divided into two regions by the reacting band. These are given different properties to correspond to their "pre-heat" (solid) and "post-tumescent" (foamed) condition. Another significant difference in their approach is that they do not account for the endothermic effects that are thought to be significant by other workers.

Broadly speaking, both Anderson and Wauters and Buckmaster et al. present experimental and theoretical substrate heating curves that show a similar form. This is an initial rapid rise in temperature followed by a quite brief period in which the gradient of the temperature rise is much reduced and then a final period of rapid temperature rise. It must also be noted that this does not match the R.E.'s experience at all well. Curves of this form imply that the insulation provided by the foamed char is not actually much better than the original coating. Buckmaster et al. actually suggest that the beneficial performance of intumescents "in this context" is purely a result of the fact that the expansion of the coating physically delays the arrival of heat at the substrate by its motion.

This is certainly not the case with the modern intumescents and much longer time periods that the R.E. has worked with, although this effect would clearly be much more significant over the three or six minute test period discussed by Buckmaster et al.

Mamleev et al. [10] use equations of Mass conservation to describe an expansion coefficient and find a thickness of foam that will be generated from a starting volume of coating. Equations of energy conservation are used to describe heat transfer through this volume of foam. It is assumed that the dominant factor is the thermal conductivity of the gasses in the pores, especially when the expansion coefficient is high. However, it is recognised that this approach does not account for the effect of pore size distribution on heat conductivity.

Having assembled a series of equations to study the dynamics of intumescents numerically Mamleev et al. also carries an experimental investigation into the temperature field within a developing intumescent char. This was carried out by heating a small disc of intumescent with a propane burner and reading the temperature at set points away from the substrate using fixed thermocouples. As the coating expanded it progressively engulfed the thermocouples and revealed the temperatures at different layers of the developing coating. The thermal conductivity is one of the main parameters in this model and is estimated using the experimental results of this experiment. Thermogravimetric analysis of the simple intumescent formulation used in this experiment was also carried out and analysed to provide constants for use in the numerical analysis.
A second model to describe the char formation is also described in terms of foam formation and draining. This provides an alternative set of results describing the thickness of foam that is formed for comparison with the expansion coefficient already described. The calculated temperature field in the expanding coating is similar for both methods. However, these results do not match those measured experimentally well.

Despite this, the model predicts the same sorts of effects that are seen in reality. For example, the distribution of the expansion coefficient with depth and the rate of char development are both important and complex factors. The authors clearly have a good understanding of the development of intumescent chars and have succeeded in modelling their complex behaviour, at least in general terms.

The authors ascribe the imperfect match of the models predictions to reality to the uneven expansion of the coating over the small sample tested. This is a common problem in the study of intumescent chars and it seems probable that, as well as the small specimen size, the use of a single gas burner would have provided an uneven heat source. It is likely that a better match with the model could be achieved by testing larger specimens and controlling the heating of the specimen more precisely.

5.1.3 The behaviour of intumescent materials in fires

Intumescent char is generated from a thin coating by heat in a complex manner. Most intumescent coatings consist of resin matrix heavily loaded with fillers. Under fire conditions the resin softens to become a viscous liquid and simultaneously certain fillers react to generate steam or gas that blows the matrix into a foam. The more sophisticated formulations contain other fillers that improve the char formation in some way. For example, surfactants that aid the foaming process and help ensure that bubbles of the optimum size range are formed. As this foam heats further it begins to carbonise and gradually becomes a rigid char with a thickness that is much greater and a density that is much lower than the original coating.

This change means that the char has a far lower thermal conductivity than the original coating. Four main factors decide the performance of an intumescent char:

1. **Char structure.** The density of the char and the size distribution of the pores in the char has a large influence on its thermal conductivity. The degree of interconnection between the pores is also important.

2. **Thickness.** Clearly, if all else is equal, the insulation provided by a char will improve as the thickness increases.

3. **Rate of reaction.** The more quickly an intumescent can form a thick, stable char the less heat will pass through it to reach the substrate.
4. **Endothermic reactions.** The insulation benefit is also enhanced by the fact that the changes of state are driven by reactions that also absorb energy from the fire during the crucial char forming stages.

Most of those working in the field understand this process well and appreciate its complexities. The process of intumescence has been well studied over a period of over 30 years. Much of the early work in the field concerned the protection of munitions from fire and explosion following jet fuel fires on aircraft carriers.

Anderson and Wauters [7] describe the effect of heat flux on the solid matrix; raising the temperature and causing activation of the various fillers. In that case melting of hydrated solid powder fillers, distillation of water, the evolution of water vapour and other volatiles causing an expansion of the softened matrix. This description makes it clear that these reactions absorb significant quantities of energy and that this benefit was understood early in the development of intumescents. They also point out that the resulting char is graphitic and therefore resistant to burning. In consequence, as it gets hot enough it will glow and radiate back a significant proportion of the energy, mitigating the overall heat flux.

Later workers in the field, such as Mamleev et al. [10], describe intumescence in more detail. Mamleev emphasises that the softening, foaming and hardening process occurs from the outer surface inwards and that these processes must be synchronised correctly to form a good char. This explains the common observation that different layers of a char demonstrate different a significantly structure and density through the fact that each will have experienced a different heat-history. They also demonstrate a good understanding of the realities of char structure, including foam formation, structure and deterioration. This description goes some way to explaining the formation of different char types through an understanding of bubble formation, matrix viscosity and pressure.

It is well understood that these factors vary substantially between different generic types and specific formulations of intumescent. Different manufacturers formulate their products carefully to tailor this behaviour and achieve optimum performance for the chosen application. The rate of expansion, degree of char expansion, and the type of char produced are different depending on whether the material is intended for rapid reaction, but short term protection, or for long term resistance to intense fire.

Workers in the field also understand that the actual behaviour of a specific intumescent will vary according to a number of factors. The most crucial is the temperature regime that the intumescent is exposed to during the fire or simulated fire conditions, specifically the rate of temperature rise and heat flux. This is most critical in the early stages of the fire, once the char has formed into a stable layer it will not be greatly effected by normal variations in fire conditions, although it will gradually lose carbon content, weaken and erode.
The R.E.'s work has primarily been on a single material, Nullifire's System E. This is an intumescent based on epoxy resin that falls into the heavy duty, "thick film" category. This means that it is normally applied in thicknesses between 4 and 20 mm and expands by up to 5 times to form a hard and durable char. System E was formulated for use on structural steel substrates to protect them against severe hydrocarbon fuelled fires. The R.E.'s work looked at using the material on other substrates and under different fire conditions.

Figures 11 and 12 demonstrate the effect of different heating rates on nominally identical specimens. Under cellulosic heating conditions the 4 mm thick layer expanded to over 20 mm of char. This char is made up of a mixture of irregular pores about 5 mm across and many more roughly spherical pores less than 1 mm across. There are also several distinct layers apparent where it is clear that the balance of viscosity, foaming and hardening conditions were different. This is a typical System E char.

![Figure 11](image1.png)

Figure 11: 4 mm of System E on 20 mm ceramic board. Exposed to BS476, part 20 "cellulosic" furnace for 40 minutes.

The specimen shown in figure 12 was exposed to a furnace that was heated to 600°C in less than 5 minutes and then held at a steady temperature. Comparison with figure 2 shows that this heating regime is not radically different to the cellulosic regime experienced by the previous sample. Yet, the chars produced are very different. Figure 12 shows a much thinner, denser char made up of many small pores ranging from less than 1 mm to about 3 mm. It also shows that the layer of material closest to the ceramic substrate has not undergone the foaming process. The reflects the fact that it had not yet reached the same temperature as the equivalent layer on the other specimen.
This comparison demonstrates that intumescents can be very sensitive to heating conditions. The degree and type of foaming and the extent of the increase in thickness can be greatly affected. Clearly this level of difference will also have a definite affect on the thermal properties of the char.

When, as in much of the work carried out by the R.E., an intumescent is combined with another material the problem becomes even more difficult. Mathematical models tend to assume an inert, impermeable substrate, either highly conductive or highly insulative. A highly conductive material with a high thermal mass will draw a lot of heat from the char, on the other hand, a char in contact with a perfect insulator will become much hotter. This may lead to a more rapid reaction, and a better char, but may also "bake" the material so that it becomes solid before the foaming process has been completed.
The sample shown in figure 13 was originally very similar to that shown in figure 11. It was made at the same time, with the same thickness of the same intumescent and was exposed to the same temperature conditions. However, the substrate material was a syntactic phenolic foam board rather than a pressed ceramic fibre board.

This single change has had a great affect on the char formation. The char reached a thickness of over 60 mm in some places compared to only about 20 mm on the ceramic board. The char is also a more open, reticulated structure showing evidence of tearing and the start of a process of delamination of different layers.

The thermal performance of the two boards at room temperature is similar, according to manufacturer's data. Interestingly, the performance of the two systems on the furnace was also similar with cold face temperatures on both being much the same throughout the test. This may suggest that the more open structure of the char on the phenolic foam sample, indicating inferior insulation, was compensated for by the greater thickness.

Figures 14 to 16 show three more samples, all made up of 4 mm of System E on different substrates of 20 mm thick insulating materials, all tested under the same conditions. They show a typical System E char developed on Calcium Silicate board and very different results on two different types of vermiculite based panel. The char on a low density, loosely bonded panel is almost completely un-foamed and the thermal performance of the system was poor. The char on the high density panel foamed to a typical thickness, but it is highly laminar, quite flaky and still performed quite poorly thermally.

Figure 14: 4 mm of System E on 20 mm low density Calcium Silicate board.
Exposed to BS476, part 20 "cellulosic" furnace for 40 minutes.
Previous experience has shown that the difference between steel and the various types of insulating board is significant. Char on steel tends to be more compact, with smaller pores, while that on insulating board is thicker with larger and more irregular pores. These results reveal that char formation of System E is also highly dependent on the type of insulating substrate. Although this must be partly a result of the degree of insulation, such significant differences on materials that are broadly similar thermally suggest other factors too.

For example, whether the substrate will permit passage of gasses may also be significant. If the gasses generated during intumescence are able to move in either direction through the softening matrix this is likely to affect the char
structure. The materials described above ranged from quite dense, impermeable materials to those with open structures. Finally, it is possible that substances released from certain substrates may effect development. Some materials tested by the R.E. released steam and/or smoke during heating. In these cases some interaction may have taken place.

As described in the previous section, it is possible to calculate the heat flow through stable porous "chars" quite accurately using mathematical models. However, modelling a developed intumescent char is only a practical proposition if one can be certain that the char thickness and structure will be of a certain type. The figures shown above demonstrate that intumescent chars are relatively unpredictable and their formation is easily influenced by a number of factors. The work carried out by the R.E. involved employing different substrates as secondary insulation and it quickly became apparent that this factor dominated the formation of the char.

In a paper on the modelling of the formation of non-intumescent chars Staggs [11] comments that many models exist for well-developed char layers but that modelling the formation of chars is more complicated. Although the degradation of the polymer, PVC in this case, is described in detail he concludes that a realistic mathematical model of the dynamic char-forming process is unlikely. This is because the behaviour of the material as it is transformed from molten polymer to solid char is complex and this is crucial in determining the transport of volatile products which is fundamental to reaction to fire properties. This problem is even more severe when considering intumescent chars because it is the behaviour of gasses that determines the structure, and therefore the thermal conductivity, of the char.

When System E epoxy intumescent is exposed to a cellulosic heating curve the char is still developing after a period of 30 minutes. Most of the applications that the R.E. worked on concerned protection periods of 30 minutes to one hour. Since mathematical models cannot describe the complex development of the char and the dramatic changes in its properties they are least useful during this phase.

In the case of intumescent materials in particular it is easy to understand why many of those in the field shy away from seeking to model behaviour.

5.1.4 Heat flow measurement and modelling

Although Nullifire is quite experienced in the design and testing of fire barriers, the Gas Safe is an enclosure that is completely immersed in fire. This means that any hot gas that penetrates the enclosure through joints or holes is there for the period of the fire test, unlike fire barriers where it escapes and heat is radiated away. During the Gas Safe development it was found by experiment that the very low failure temperature of propane cylinders meant that the amount of heat that could be allowed into the enclosure was actually very small. Much later, it was calculated that the energy required to evenly heat a full propane cylinder to 63° C was only about 1811 kJ. A fully developed fire is often quoted as producing a heat flux of 50 kW m$^{-2}$. Therefore, under these
severe conditions a full cylinder could reach venting point in only around 72 seconds.

The Gas Safe prototypes were intended to represent the intended finished product as accurately as possible. Therefore, they included complex geometry and details such as joints, ventilation holes, handles and these had an unknown effect on fire performance. After a number of tests the R.E. realised that these details were making interpretation of results difficult and began to exclude them. This helped to some extent but was only a partial solution.

The R.E. developed a combination of intumescent and passive insulation materials capable of providing the high level of insulation required to protect LPG cylinders. Although this composite approach was effective it was very difficult to predict the performance of different material combinations. Very little data exists on how much insulation is provided by most materials under fire conditions. The behaviour of these materials when over-coated with intumescent was completely unknown, and unique to each type and thickness of intumescent and the particular fire test conditions experienced.

After this experience, the R.E. would now approach a similar development programme in a different way. As before, the starting point would be to establish the properties required from the product and then review the alternative materials available. Where possible a shortlist would be chosen using manufacturer’s data, but unfortunately good fire test data is rare. Using the methods developed during Gas Safe testing, a series of screening tests would be devised to select the best materials, while excluding the effects of the design itself.

A good example of this would be fire testing of small sample panels which gives good information about the reaction of the material to fire and the level of insulation it provides. The use of small samples reduces the cost of early testing, eliminates the labour of prototype construction and may increase the number of alternatives that can be tested. It also increases the speed of development because a larger number of samples can be tested at once.

Unfortunately, the performance of a flat panel can not be directly related to an enclosure like the Gas Safe. Simple temperature data from the unexposed face of a panel is not equivalent to the surface temperature of the inside of a Gas Safe, because there is no heat loss from an enclosure. In addition, this data does not indicate how hot the contents of an enclosure will get because it contains no information about the amount of energy entering it.

However, it is possible to measure the amount of energy flowing through a surface using heat flow sensors. Once this was realised the R.E. began to work on this in parallel with the work on the Gas Safe. A method for measuring heat flow through panels was developed and is described in the submission "Collecting and Making Use of Data on Heat Flow Under Fire Test Conditions in the Development of a Fire Resistant Enclosure".
It was found that this data was more useful than the surface temperature data that are normally collected from such tests. The data from different panel samples may be compared directly to assess which is the most effective insulator. More importantly the heat flow data may be used to construct simple computer models that predict the temperature rise of an object in an enclosure over time under given fire conditions. An example of this is given in figure 17.

![Graph showing the performance of a Gas Safe predicted from a measured heat flow curve compared with actual fire test data.](image)

**Figure 17:** The performance of a Gas Safe predicted from a measured heat flow curve compared with actual fire test data.

### 5.1.5 Discussion of the Heat Flow Model

**Limitations**

The method described above obviously requires that the heat flow profile through a particular material or combination of materials is known. This means that representative flat samples must be made and tested on the furnace and that heat flow measurements are taken. Although this can be carried out much more quickly than testing of complete prototypes it does still require significant work.

It also means that each measured heat flow profile is unique to:

- a) the time/temperature or furnace regime,
- b) the combination of materials and substrates,
- c) and the thicknesses of materials used.
This limits the use of the measured heat flow curve to modelling something made from an identical material cross-section exposed to the same furnace conditions. As shown above the significant effect of the substrate on char formation also means that it is not possible to deal with the intumescent as a separate layer. Therefore, the heat flow through each proposed cross-section of materials must be measured individually.

Accuracy

The usefulness of this simple modelling technique as a design tool is dependent on its accuracy. The measurements of heat flow through the panels were subject to normal experimental errors. The accuracy of the sensors and data logger was thought to be subject to an error of ±20 W m⁻² in a final heat flow of 2000-4000 W m⁻². Air flow currents over the sensors were found to cause a much more significant error in the reading of about ±250 Wm⁻². However, this was a regular fluctuation about the mean reading and would not have had a large affect on the overall heat flow over the period of the test. Therefore the largest uncertainty about these measurements is the inherent variability of furnace testing. Over fifteen years of furnace testing at Nullifire indicates that results are subject to a variation of about ±5%.

It is certain that far more significant errors were introduced by the assumptions that were made in constructing the model:

1. The geometry of the modelled objects was taken to be a simple surface area evenly exposed to the heat flux. In practice the surface area was not precisely known in every case and had to be estimated. In addition, the heat flux would not be even and would certainly vary according to the location and elevation of the samples in the furnace.
2. The heat flow through the flat panel samples was used to represent heat flow through the curved surfaces of the enclosures.
3. The heat flow through the panel that was measured was taking place between the furnace, at a temperature rising to about 800° C in 30 minutes, and the laboratory at about 20° C. In an enclosure the "cold face" temperature is actually rising, in the case of the Gas Safe this would be to about 100° C. This effect was assumed to be relatively insignificant in the model but would actually have reduced the heat flow slightly in the later stages of the tests.
4. The wall thickness was taken to be uniformly the same as the tested panels although it varied in parts of some designs.
5. It was assumed that the development of the intumescent char would be the same on the curved surfaces of the samples as on the flat panels. In practice, intumescents are prone to cracking on curved surfaces and char thicknesses will not develop evenly because of differences in exposure to heat.
6. Although the thermal mass of the items in question was known quite accurately, since the mass and materials were known, it was assumed that heating of this mass was even. It practice this would certainly not have been the case since the model does not take
account of the effects of air gaps, vertical temperature differences, motion of air and convection within the enclosure

These assumptions were made to keep the model as simple as possible while maintaining a useful level of accuracy. They are largely within the control of the modeller and can be adjusted to ensure that a worst case prediction is given. This is best evaluated by comparing the predictions made by the model with actual test results.

For a basic steel and intumescent Gas Safe the model predicts that the steel cylinder will reach 100° C after 6⅝ minutes. A fire test on that design showed different parts of the cylinder reaching that temperature between 4 and 10 minutes. This variation reflects the uneven heating issue raised in point 6 above.

In the example of a more sophisticated foam and intumescent given above, in figure 17, the model predicted 16⅛ minutes to reach 63° C. This was only 75% of the 22 minutes the specimen actually took to reach that temperature. This is accounted for by the fact that the thickness of insulation in the upper potion of the Gas Safe sample was actually greater than the tested panel.

Later use of the model in predicting the performance of an electronics enclosure (described in section 6.1.1) proved to achieve similar levels of accuracy. Actual performance of an enclosure in a 300° C test was 16% better than predicted but in a 400° C test it was 13% worse.

The Model as a Design Tool

This model was developed for use in the initial stages of design to reduce the amount of prototype testing required. It allows proposed materials to be evaluated by testing simple panels more rapidly and cheaply. Although the predictions of performance that the model makes are not especially precise, the examples given above show that they typically fall within about 25% of actual performance. This is accurate enough to allow various concepts to be evaluated rapidly and for promising ones to be selected for testing in prototype form.

Although this method is quite simple, the effects of this more analytical approach can be very significant. The information provided by a small number of panel tests can completely eliminate the first stage of prototype testing. As discussed in submission 11, using this method in the first stage of Gas Safe development would have reduced the 23 month period and £172,000 cost by half. The long-term importance of this piece of work is that the value of an early analysis and engineering approach has been demonstrated. In future the development methodology in Coating Services will reflect this.

This approach was based around the equipment available at Nullifire, principally the fire testing furnace. Using the furnace ensures that the heat flow data fully represents the way in which the materials in question will react
to later fire testing. Although the more sophisticated and fundamental methods of modelling heat flow through char discussed above are more widely applicable they cannot reflect the behaviour of developing char as well. They would also require thermogravimetric analysis or results from a differential scanning calorimeter, which is equipment that Nullifire does not have ready access to.

Although this method was too late to be of real use in the Gas Safe development it was used on another project, as described in section 6.1.1. This product is also an insulated enclosure, constructed from similar materials as the Gas Safe. The R.E. is also testing a series of different insulation materials combined with different intumescents. Data from these tests will be used in similar computer models to improve the performance and reduce the cost of that product. It will avoid the necessity of building large numbers of complex prototypes and so save time and money.

5.1.6 Making Use of Existing Data

As has already been stated, the performance of Nullifire intumescent products is independently certified. In section 4.2.2 an example was given of a crude calculation using data from System E certification to estimate the thickness of intumescent needed. Calculations of that type are inaccurate, and require careful judgement, but are better than guesswork. They are often used at Nullifire as a first estimate of the practicality of a project in terms of performance or cost. However, the heat flow approach may be applied to the same reservoir of data to give much better results.

The intumescent thickness tables in the certification are generated from an extensive independent testing process. This means that data already exists on the temperature rise of steel sections coated with a range of different thicknesses of intumescent under standard fire test conditions. These data are of a high quality, with temperature readings from several locations on the section every 10 seconds. The section type and mass is also known and so the amount of heat that has flowed into the section in every 10 second period is known accurately.

This means that, for each fire test, a graph can be drawn of heat flow through the developing intumescent char as the test progresses. The fire tests are designed to give a range of intumescent thicknesses so it is possible to generate a series of heat flow curves for each intumescent formulation. This family of curves provides excellent information for engineering design calculations. Once the performance requirements and approximate geometry of a product are known it will be possible to make a comparison with the heat flow curves. This will allow a much more accurate initial selection of intumescent type and thickness than is currently possible.

Nullifire also carries out many in-house tests on its intumescents and these data could also be used. The main advantage of this would be to allow cross-checking of the heat flow curves at similar thicknesses from several different
tests. This would ensure against the inherent variability in fire testing and also provide an indication of the likely variation in performance.

Finally, fire tests are carried out on a range of different sample geometries, including flat panels, "I-sections", and square and circular hollow sections. It is known that this has a quite significant effect on the performance of intumescents. This is because the forming intumescent char is liable to crack at corners, flange tips on "I-sections" for instance, and on curved surfaces, circular hollow sections for example. Intumescents may also be liable to "stickability" problems on large flat surfaces, i.e. detachment of the char and exposure of bare steel. It will be possible to produce different heat flow curves for each of these geometries and so the curves most appropriate to the geometry of the new product may be chosen.
5.2 MATERIAL COMBINATION

When the Gas Safe project began it was specified that the product must be made of the System E epoxy intumescent. This was an attempt to recover the cost of developing System E that was suffering very poor sales. However, System E was formulated and optimised for the fire protection of structural steel which has a failure temperature of around 550° C. The R.E. realised very early in the project that this meant it was not ideal for the protection of gas cylinders with a low failure temperature around 70° C.

5.2.1 System E and Passive Insulation Materials

The R.E. found that the addition of a layer of foam to the internal face of the Gas Safe improved the fire performance greatly. Experimentation showed that 4 mm of System E alone provided about 6.5 minutes protection while foam alone provided only 4.5 minutes. Yet both materials could be combined to give a fire performance of over 30 minutes. Once the characteristics of each material is considered it becomes clear why this synergy exists.

The resin matrix containing intumescent ingredients must soften at around the same time as the blowing agents activate so that a foamed char can be generated. A relatively thick layer of System E, 4-5 mm in the case of the Gas Safe, takes some time to soften and react to the heat of a fire. The unreacted material is dense and actually quite a poor heat insulator and so quite a lot of heat may pass through it in the five or so minutes before the char forms. This is not especially significant where the failure temperature is high. However, where it is low, around 100° C for instance, enough heat can flow through the unreacted material to cause failure before the char has even formed.

Foams are generally very good insulators at low to normal ambient temperatures, but even the fire rated foams used in these experiments crack and erode rapidly when exposed to the temperatures found in fires. When placed behind a layer of intumescent they can provide crucial insulation in the early stages of the fire before the char has had time to react. As the fire proceeds the reacted intumescent protects the foam from the worst of the fire and so the foam will continue to provide additional insulation, even though it will degrade gradually.

The Gas Safe project showed that several different types of foam could boost the performance of System E greatly. The benefits of this approach are that foams are lightweight and comparatively cheap. However, they do increase the thickness of the protection required significantly, from 5 to 35 mm in the case of the Gas Safe, and tend to be vulnerable to damage. Therefore, it is necessary to consider the relative importance of these factors early in the design stages. The cost of these materials and potential processing difficulties are also very relevant. The R.E. is currently undertaking a project to evaluate a wide range of passive insulation materials, using the heat flow methodology described above, so that a database of all these properties can be assembled.
5.2.2 System E and Fabric Insulation

A collaboration with a French company, described in more detail in the "Information in support of the Gas Safe" submission, showed that similar benefits could be obtained at much lower thicknesses. The company produces and markets a range of knitted fabrics made up of carbon fibre precursor and kevlar fibres. These fabrics were originally intended as flame resistant barriers, also capable of catching blast fragments.

It was found that a 1 mm thick layer of this fabric was nearly as effective as 30 mm of foam in boosting the performance of System E. This only seems to be effective where the fabric is placed directly between the intumescent and the substrate material. It is thought that it has very low thermal conductivity and so prevents the heat in the char being conducted to the substrate. It is also thought that the knitted structure traps many tiny cells of air and so prevents transfer of heat by convection. Finally, it is possible that the fabric may absorb heat by phase change of one of its constituent fibres.

Figure 18: The performance of System E/fabric composites compared with that of the same thickness of System E alone.

Figure 18 shows that one layer of fabric doubles the time taken to reach 100°C from 13 minutes to 26 minutes, while two layers increase it to 46 minutes. Although this combination of materials appears to be extremely effective, potentially reducing the Gas Safe wall thickness from 35 mm to 6 mm, no products are currently under development. However, further research and fire tests on various fabrics are planned to establish how this really works and precisely how effective it is.
5.2.3 Improving Performance using Thin Film Intumescents

The realisation that the slow reaction of System E to fire allows significant quantities of heat into the substrate early in a fire has led to another material combination. Thin film intumescents, also produced by Nullifire, produce a voluminous char much more rapidly in a fire. This is partly because the heat quickly reaches the entire thickness but also because the char expansion of System E is restrained, by carbon fibre, so that it retains a greater degree of strength. The cost of thin film intumescents is low relative to epoxy intumescents and so it is possible to overcoat a System E component without greatly increasing its price.

This has been shown to be a cost-effective way of improving performance, especially late in the development process when more drastic design changes are impractical. It was used in this way by the R.E. on an insulated actuator cover to allow it to pass a more severe test when the specification was increased by the customer. The disadvantage of this method is that the additional drying time of thin film inturnescent slows production and adds several days to the process. It is also vulnerable to chipping and so makes the product less durable.

Thin film intumescent has also been used by the R.E. to allow System E to meet tunnel specifications. All materials used in tunnels must meet the "Surface Spread of Flame" test, or its local equivalent, to "Class 1". This limits the speed at which flames travel along the surface of a panel of material under severe radiant heat conditions. Normally, System E only performs to "Class 2" and this precludes its use in tunnels. However, a thin coat of Nullifire's System S reduces the spread of flame sufficiently to pass this test. As a result System E is now specified for two Channel Tunnel contracts that are expected to begin in the next few months.

5.2.4 Summary

The original attitude in the development of new products for Coating Services was that they must be made from System E, whether it was the most appropriate material or not. The approach to problems that has developed over the course of the Engineering Doctorate programme is very different. When designing a product, a material selection process now takes place and the required properties of an ideal material for the job are considered. When the basic properties of the ideal material are known it is possible to select from the Nullifire range, consider material combinations or search for materials from other companies.
5.3 NEW PROCESSING METHODS

During the Gas Safe project the surface finish of the product became a very serious issue. It was apparent that the rough texture would not be acceptable to the intended consumer market that is accustomed to smooth injection moulded surfaces and high quality paint finishes.

Figure 19: A sprayed System E Gas Safe.

It was known that the material could be re-formulated to improve application characteristics and surface finish, although this may have reduced fire performance or increased cost slightly. However, reformulation was judged to be too expensive and too slow to be a practical option. This was exacerbated by the fact that the Gas Safe was a development project, with unknown long term potential. As a result the only option available to the R.E. was to change the application method to try to improve the surface finish.
5.3.1 Injection of System E into simple moulds

The injection of System E into moulds was an existing alternative to spraying. To do this the usual mixing method and spray kit was used, but the spray gun was removed and the material injected into a mould using a small nozzle. The moulds were usually very simple, often simply flat trays for creating slabs of epoxy. However, the use of large spiral wound cardboard tubes as moulds was also well known at Nullifire.

Normally they were used in concentric pairs, a large outer and a smaller inner, with a gap of 10-20 mm between them, which was filled with System E by injection. Once the epoxy had cured the entire assembly was cut in half along the length and the cardboard disposed of. This method has been used for many years to make large "half-shells" of epoxy intumescent which are glued over circular structural columns in buildings to provide fire protection.

Although this worked well, the mouldings were always deeply marked by the spiral windings from the tubes and contaminated with the essential silicone release agent. This always meant that a great deal of finishing work was required on the tubes before they could be painted, raising the installed cost of the system greatly.

5.3.2 "Spray Moulding" of System E

When it became clear that spraying could not produce suitably aesthetic Gas Safes the R.E. began to search for ideas. The first part of this was a review of plastics and moulding journals in search for new ways in which System E could be processed. Visits were also paid to several companies specialising in the moulding of "difficult" materials like epoxies and composites manufacturers. This process generated several new ideas and a great deal of advice.

One of the clearest and simplest concepts was the use of a release film to allow de-moulding from re-useable tooling. Further investigation revealed that although System E bonded quite well to a number of thermoplastics it would release very well from PVC, Acetate and also from polypropylene. The R.E. realised that since thin thermoplastic films are quite flexible it would be possible to line shapes with one degree of curvature very easily.

Since the main body of the Gas Safe was cylindrical it was obvious that the inside of a cardboard tube could be lined with film. It was found that the elasticity of the film was dependent on its thickness and that this quality could be used to hold it in place inside the tube. If the film was precisely the right size to match the circumference of the inside of the tube it could simply be curled and then dropped into the tube. The natural spring of the material would then hold it tightly against the sides of the tube with an almost imperceptible seam.

Experiments were carried out with injecting the trunks of the Gas Safe directly onto inner tubes of the insulating foam in an attempt to produce a finished
component. However, it was found that the pressure necessary to fill a 4 mm wall section with the viscous System E was so high that it tended to destroy the fragile foam insulation.

Fortunately it was found that, since the diameter of the Gas Safe trunk was quite large and the length quite short, it was possible to spray the epoxy into a lined tube from either end. Once the material had cured it was easy to slide the liner out and peel it away from the thin tubular shell of epoxy. This created a very smooth outer surface and a rough inner surface, which was ideal for bonding to foam. It was also found that, provided care was taken, it was possible to re-use the film and the cardboard tube up to ten times which made the method economically viable.

This method offered a practical way of producing very smooth and round Gas Safe trunks, but did not solve the problem of the Gas Safe lid. To improve the appearance of the enclosure the lid had been profiled to fit tightly over the top of gas cylinders. It was now a truncated cone with a boss to give space for the regulator and so was quite a complicated shape, impossible to mould using release film.

However, the research carried out by the R.E. had shown that polypropylene sheet was routinely formed into quite complex shapes using the vacuum forming process. It was found that the only barrier to this method was the cost of tooling for creating the vacuum forms, between £500 and £1000 for a tool the size and shape of the Gas Safe lid. It was clear that this method would only be appropriate for products where a high enough number of mouldings would be manufactured to absorb the cost of the tool. The Gas Safe was expected to be produced in considerable numbers so a vacuum form master tool was produced.

A number of vacuum forms were produced from 3 mm polypropylene and spray trials carried out. It was found to be quite easy to lay down System E to approximately the right thickness by spraying and then rolling it to even the film thickness and consolidate it. De-moulding of the cured "shell" of epoxy was reasonably easy, even without any release agent, and the outer surface of the moulding was very smooth and an accurate reproduction of inside of the mould.
Figure 20: Spray moulding of System E into an open mould

The "spray moulding" method was judged to be very successful and used for the production of all subsequent Gas Safes. Even so, a number of limitations were noted:

- The inner surface of the moulding was rough and it was not easy to control wall thickness of the moulding.
- It would only be possible to spray quite large vacuum forms with an open form, since small, narrow moulds would result in excessive over-spray and wastage.
- The vacuum form method was limited to shapes that could be de-moulded and required generous draft angles, experience indicates a minimum of 5°.
- Trimming the moulding was time consuming and difficult to carry out accurately.

The first three limitations were not problems for production of the Gas Safe, and the roughness of the inner surface was actually beneficial in bonding the inner layer of foam. However, the problems with trimming did contribute to the high labour content in trial production.
5.3.3 Improved cardboard tube method

Production of Gas Safes by "spray moulding" showed that two of the weaknesses of cardboard tubes could easily be overcome by lining them with release film. This led to the introduction of improvements in the way in which System E is moulded for structural steel circular hollow sections.

This was first used on the Buchanan Centre contract, which the R.E. was heavily involved in. The method relied upon using cardboard tubes, cut in half down their length, to mould System E directly onto the circular steel columns on site. Although there were serious problems with this method they were eventually solved, the contribution of the R.E. to this is described in more detail in the Personal Profile. The use of release film inside the tubes created a very smooth surface over 90% of the finished casting and greatly reduced the amount of finishing work required.

More recently the production of "half-shells" in the Coating Services workshop, as described above, has been reintroduced as an alternative method. This is because all of the moulding takes place in the workshop and
so the process can be more easily controlled, reducing the risks inherent in producing such large mouldings. The use of release film is now an integral part of this process and it has been estimated that this saves about 2 hours of labour per m² of moulding for a material cost of only £2/m².

Figure 22: System E protected columns in the Buchanan Centre.

5.3.4 Injection moulding into vacuum forms

The limitations of the "spray moulding" technique became serious when attempts were made to transfer Gas Safe technology to other projects. Most epoxy parts were smaller and deeper in section than those of the Gas Safe and this made spray moulding difficult and wasteful. The high cost of System E makes a high rate of wastage unacceptable. In addition, a smooth inner surface was also desirable in most mouldings to allow accurate fitting to other components or to the item to be protected.

The R.E. soon realised that the spraying method would have to be improved upon to make moulded System E components generally practical. The success of the closed moulds used to make "half-shells" indicated that a form of injection moulding would be possible. A closed tool could potentially give a good control of accuracy and good surface finish all over a component and also reduce wastage to almost zero. By now it was known that vacuum forms were reasonably cheap, a known number of components could be produced from them and de-moulding System E from polypropylene was relatively easy.
However, experiments on closed tools constructed from vacuum forms soon confirmed that there were major obstacles to be overcome. The high pressures required to force the epoxy around the mould caused distortion, bulges and uneven wall thickness. It was apparent that the rigidity of the tooling would need to be improved and that the lower the wall thickness of the moulding the greater the problems would be.

This demonstrates an inherent contradiction in the moulding of System E clearly. An ideal tool will be very rigid, accurate and have a smooth surface that will release from epoxy easily. However, the long curing time of the material means that the in-mould time will be at least 24 hours. This dictates that large quantities of cheap tools are needed for economic production of a run of components.

The R.E. was familiar with the concept of composite tools and tool liners which can allow more economic and rapid production of tooling. It seemed possible that the "soft" polypropylene vacuum forms could be reinforced with a "hard" backing material to give a cheap, rigid tool that also released well. Experiments were carried out with cast epoxy and plaster backing materials and this showed that the concept could work, although the prototype moulds were heavy, unwieldy and too expensive to be practical.

Over the course of three projects the concept was improved and principles that allow design of such tools have evolved. Two of these projects relate to the production of parts for commercially successful contracts while the third remains a technically successful development project for the time being. These projects are described more fully in section 6, this section is confined to a description of the tooling design improvements that made production possible.

**The Tay House column caps**

The wall section of this moulding was a minimum of 10 mm and so moulding pressures were quite low. The tool was designed as an upper and lower vacuum form that fitted together along a flange. This flange was clamped shut using two bolted rings that also held the vacuum forms concentric. An additional benefit of these rings was that they removed any inherent distortion from the vacuum form by holding the flange completely flat.
The upper surface of the moulding needed to be convex and it was fortunate that this shape is naturally rigid, as is the circular form of the inner and outer rim. The inner base of the moulding was reinforced with a plate since it needed to be completely flat. The centre of the domed upper surface was selected as the injection point and the injection was carried out upside down to minimise the risk of air entrapment. The mould vented air from a ring of holes around the rim and flow of material from these indicated when the mould was full.

This tool design was very successful and 100 components were produced from 20 tools. In reality this was quite conservative since each mould could have produced at least 20 parts. However, the required rate of production meant that there was only time for five injection cycles, and so this number was chosen to minimise tooling cost.

**Thin Moulded Coating on a Steel Vessel**

This project has already been introduced in submission 13, "Information in Support of the Gas Safe Project" and will be further discussed in section 6. It also demonstrates a important advance in the design of closed vacuum form tooling at Nullifire because it has a wall thickness of only 3-4 mm and incorporates a complex insert.
The insert is an approximate sphere about 300 mm in diameter with a prominent welded seam and four welded studs at each end. The required coating thickness was thought to be the minimum achievable by moulding in System E. A very smooth finish and a good seam at the joint line were also specified.

The shape allowed two identical hemispherical vacuum forms to be used and this shape provides excellent resistance to internal pressure so no distortion takes place. The vacuum forms are held in place by the eight studs and these are also used to ensure an even gap between the vacuum form and the insert. Intumescent rubber washers of the correct thickness are placed over each stud and each mould-half fitted, the studs passing through small holes in the vacuum form. Bolts are then tightened down on each stud that holds the vacuum form tightly against the washers and ensures accurate spacing.

![Diagram of moulding process](image)

**Figure 24: Moulding a thin coating onto a steel vessel.**

The vacuum forms are clamped together using flanges and clamping rings in a similar fashion to the Tay House moulding. The epoxy is injected through a hole in the centre of one end, equidistant from four of the studs, and vented through a similar hole at the opposite end.

This method has been shown to be successful with over 40 components being produced for customer trials. However, for successful volume production a considerable amount of development remains. For example mould set up time must be greatly reduced from the current 30 minutes per unit and problems with the mould filling
resolved. The flow in the mould is often uneven so that one side fills more rapidly, trapping an air pocket when the vent hole is obstructed. It is likely that applying a vacuum to the mould at the vent point will solve this, but this remains to be tested.

**Actuator Cover Mouldings**

This product actually pre-dates the two previous projects but the problems with moulding have only been solved recently. The wall thickness of the moulding is only 4 mm and both the lid and base incorporate fragile foam inserts. In addition the shape of the moulding consists of flat surfaces, that are susceptible to bulging, because the vacuum form was designed with spray moulding in mind. However, spray moulding proved to be too difficult and wasteful for such small parts.

Extensive experimentation on tooling for this product took place over a period of nine months. During this time production continued using open moulds and primitive moulds consisting of vacuum forms reinforced with plaster. The method that was finally chosen uses the bolted flange rings familiar from the previous two projects. It also reinforces flat surfaces using an outer, wooden "hard tool" and braces the inside of the foam insert using polypropylene ribs to prevent collapse during moulding.

![Diagram of mould assembly](image)

Figure 25: A section through the mould assembly for the Actuator cover lid.
The components of this tool are cut using a CNC controlled machine to ensure that all the various plates fit together perfectly and means that all parts are completely interchangeable. This method also keeps the cost of the mould within reasonable limits so that they can be considered disposable after 25 mouldings. It is anticipated that this will allow gradual improvement of the tooling as more is learnt about the process. This design has been shown to work well but it is hoped that set up time, currently 12 minutes per mould, can be reduced and the life of the tools extended.

5.3.5 Moulding Using Machined Tooling

The use of vacuum forms as moulds has also led, by a convoluted route, to the use of highly accurate machined aluminium tooling for moulding coatings. This occurred because the vacuum form method was oversold as being suitable for the moulding of precise thicknesses of a silicone based ablative coating onto rocket motor parts. This was done without the knowledge of the R.E. and during prototyping it became obvious to colleagues that the required precision of ± 0.2 mm was completely unrealistic. However, by that time Nullifire was committed to a years production, consisting of 120 examples each of two different parts. The prices for this had been estimated from a costing based on the estimated cost of vacuum form tooling. At this point the R.E. was asked to design and implement a new production method to meet the customer's specification.

The ablative material itself was extremely expensive, approximately £700 for a 6 kg kit, or nearly £120 per kg, with a consumption of 0.6 kg per component. This indicated that, relative to the vacuum form method, the budget for tooling could be quite generous providing wastage of material was minimised and labour per unit controlled. The cost constraints on the required method were very clear because a known volume of products were to be produced at a firmly quoted cost. Therefore it was relatively easy to balance a series of different budgets for tooling against estimates of labour cost given more or less sophisticated production methods.

An examination of the ablative showed it to be two part; part A being a highly viscous paste heavily filled with carbon fibre and part B being a clear liquid. The material was difficult to mix by hand and also needed to be vacuumed to remove air and volatiles before application. It quickly became clear that the only way of keeping labour within reasonable limits and meeting the mixing specification would be to buy a machine capable of mixing, vacuuming and injecting the ablative. Fortunately, such machines are used quite frequently with two pack silicones and it was easy to find a suitable one.

The machine cost £5000, indicating an additional cost of approximately £20 per component. However, given the extreme cost of the material and the importance of eliminating waste, calculations based on the estimated level of wastage showed that the machine would actually reduce expenditure on material per component by more than this. The convenience of having a
properly mixed and vacuumed material on tap would also reduce the labour content significantly.

The viscosity of the mixed paste demonstrated that injection would require very high pressures and a very rigid tool. The accuracy of ± 0.2 mm required in some places and ± 0.7 mm in others indicated that a machined tool would be needed. This was complicated somewhat by the fact that there was no draught angle on the moulded parts and this meant that a multi-part tool was necessary. After discussion with several toolmakers it was decided to use machined aluminium lined with self adhesive release film. The tools were built up of flat panels, machined with the necessary detail and radii, which bolted together and to the components to form the necessary cavities. Each cavity, a single cavity on the larger component and eight on the other, is injected directly from the mixing machine. Curing of the material takes place in an oven at 70° C for one hour and using two tooling sets per component, ten of each part can be produced per day.

Figure 26: Rocket motor parts coated with ablative and tooling.
5.3.6 New finishing methods for System E

Although components produced by the new moulding methods are smooth and accurate there is often a requirement for a surface finish of some kind. This may be to protect the component from the weather, UV, abrasion, or simply to provide a gloss finish in a particular colour. Several methods have been introduced to the company by the R.E. and tested on various projects:

**In mould painting**

To date this is the only new method in common use and it has been shown to work extremely well. This method was inspired by the work of another Engineering Doctorate R.E. and solves one of the main finishing problems inherent to System E. A cured sample of System E is slightly porous because of air entrained during manufacture, in the mixing process or during spraying. The worst example of this is the surface of a spray-moulded component because the spraying tends to trap tiny pockets of air at the surface. Although these surfaces appear to be very smooth they are extremely difficult to paint well.

This is because a sprayed film of wet paint is drawn into the surface of the epoxy where there are defects, leaving "pin holes" in the cured film. When painting a System E component up to five pinholes per square centimetre is not unusual. Although a good finish can eventually be achieved by applying many coats this results in excessive labour and use of material.

However, when sprayed or closed moulding methods are being used it is normally possible to paint the surface of the mould before application of the epoxy. Typically, a heavy coat of epoxy or polyurethane top seal is applied to the tool surface and allowed to begin the curing process. The mould is then assembled and sprayed or injected as usual. Once full cure of both materials has taken place the component may be de-moulded. The top seal will have bonded well to the System E but release easily from the polypropylene and so the component will emerge with a smooth coat of paint.

This paint will reproduce the surface of the mould and when using vacuum forms this will usually mean a good matt finish. If a gloss finish is required then a second, thinned coat of paint may be applied. Although two coats of paint may be necessary, this is a considerable improvement on the five or so that would be needed to conceal pin holing. All fifty of the pre-production Gas Safe units were produced in this way, with a labour saving of over one hour per unit. Since that time this method has become standard practice where a paint finish is required on System E mouldings.

**Gel Coating**

An occasional problem with painted System E surfaces is that they are relatively easily scratched and chipped compared to painted steel. Limited trials have taken place using polyester gel coats in place of the in-mould
paint film. The polyester may be brush or spray applied to the inside of the mould at thicknesses of up to 500 microns and is allowed to cure fully. The System E is then applied, cured and de-moulded as usual.

As with the in-mould painting technique this produces a smooth, if slightly matt, finish. However, the gel coat may buffed up to a reasonable gloss and is extremely resistant to damage. Trials indicate that this method has good potential but further development is required before it can be introduced to any production work.

**Vacuum formed paint films**

The R.E. has recently discovered that it is possible to obtain very high quality paint films on thin sheets of thermoplastic, such as ABS. These materials are used in the automotive industry to create pre-painted plastic components like wing mirrors and bumpers. The paint film may be vacuum formed, like ordinary ABS, to considerable depth and geometric complexity without affecting the quality of the paint film. Normally, this shell would then be placed in an injection-moulding tool and filled with the same type of thermoplastic melt.

The trials carried out by the R.E. on various types of plastic film showed that System E would bond well to ABS. It would therefore be possible to inject the epoxy into a vacuum formed paint film to create a pre-painted component. In addition, it may be possible to eliminate the usual polypropylene vacuum form entirely by using a high quality hard supporting tool instead. This method could have the substantial advantage of allowing immediate de-moulding from the supporting tool, reducing cycle times and costs.

This proposed method has not yet been tested, although materials to do so have recently been obtained. The high cost of the material, approximately £12 per m², unprocessed, would limit the method to smaller components where a very good finish was required.

**5.3.7 Summary**

The R.E. has introduced a number of new manufacturing methods to Nullifire by adapting techniques from the outside world. These allow accurate, high quality mouldings to be made in difficult materials like epoxy intumescent and silicone ablative.

These new methods of processing allow the creation or coating of parts with complex geometry, dimensional accuracy, smooth surfaces and durable coloured finishes. These new capabilities open up a number of new markets and exploitation of this has begun, as described in the next section.
6. NEW TECHNOLOGY LED PROJECTS

In the last year of the Engineering Doctorate programme it became increasingly apparent that the Gas Safe project was misconceived. It was agreed that future development work must be focussed on areas where there was known to be a customer need. It was originally thought that these customers would be in certain sectors of the LPG market where fire risk was high and sensitivity to cost relatively low. Although these areas do exist it has been found that most of the useful enquiries have come from other sectors.

6.1 COMMERCIALLY SUCCESSFUL PROJECTS TO DATE

6.1.1 Smoke Extract Duct Actuator Cover

In large buildings the air conditioning systems can be quite complex and can present a serious hazard by distributing smoke from a fire. This may hinder evacuation, cause death by smoke inhalation and conceal the seat of the fire. It is normal to use temperature activated dampers to close off these ducts in case of a fire and to use fans in the same ducts to extract smoke so that the build up of smoke is delayed. The dampers and fans in such a system are controlled by an electronic central control system. The dampers are moved by electrical actuators that consist of an electric motor, gearbox, electronic control and a spring loaded return mechanism.

Theoretically, these actuators may be used to control the system during a fire so that various areas may be selectively cleared of smoke to allow fire fighters to operate more safely. Unfortunately the actuators are very vulnerable to heat because their electronics will fail at about 120° C. Since the roof spaces where these actuators are located are liable to fill with hot smoke, thermal protection is often needed. As a result the world leader in smoke extract duct actuators supply a "Thermally Protected" version of their actuators. Until recently this consisted of a thick layer of epoxy intumescent moulded directly onto the actuators, by a competitor of Nullifire.

In reality intumescent is not particularly well suited to this application since the hot smoke test temperatures only range from 300° C to 600° C whereas intumescents are formulated to perform in flame at closer to 1000° C. To overcome this the existing solution relied mainly on the thermal mass of a thick layer of epoxy to delay heating of the actuator. As a result that solution is very heavy and is also permanently fitted around the actuator. This means that if an actuator fails in service the entire unit must be discarded. This is particularly painful because the moulded fire protection actually costs more that the actuator itself. In addition this means that a stock must be held of a thermally protected version of each of the many different types of actuator.

Nullifire realised that the low failure temperature may mean that the insulating technology developed for the Gas Safe may be more suitable for this application. The R.E. held a preliminary meeting with the customer to discuss the fire protection requirements in detail, which are summarised below:
Low temperature test: 300°C for 60 minutes
High temperature test: 400°C for 30 minutes

Failure criteria:
   a) Actuator circuit board temperature of to remain below 100°C.
   b) Actuator to remain operational throughout fire test.

Actuator size: 290 mm x 140 mm x 60 mm
Actuator mass: 3 kg

Once this information was known, the R.E. carried out a preliminary calculation to determine whether it would be possible to meet this specification.

**Calculation of Sensitivity**

It was apparent that the insulation used for the Gas Safe was very effective. Since the surface area of an enclosure could be estimated from the actuator size and the thermal mass from the actuator mass, it was reasoned that the vulnerability of the actuator could be compared with that of a LPG cylinder.

It has been previously calculated that the surface area of typical Gas Safe design is 0.87 m² and that the energy required to heat an empty cylinder to the failure point of 63°C is 295 kJ. Using equation (1) once again:

\[ E_f = S \times M \times AT \]

- \( S = 0.473 \text{ kJ kg}^{-1} \text{ °C}^{-1} \) (assuming actuator is completely steel)
- \( M = 3 \text{ kg} \) (assuming actuator is completely steel)
- \( \Delta T = 85 \text{ °C} \) (assuming a failure temperature of 100°C and an ambient of 15°C)

Therefore, the energy to heat the actuator to failure, \( E_f \), is equal to 121 kJ.

The surface area of the enclosure, using a preliminary assumption of a 20 mm thick layer of insulation, is 0.167 m².

The rate of heating is linearly dependent on the surface area exposed to heat and the thermal mass of the object. Therefore, the figures above indicate that the actuator is nominally only 47% as vulnerable as a LPG cylinder. It therefore seemed very likely that it would be possible to fire protect the actuator in the same way, particularly since the heat input from the cooler furnace would be much lower.

After reporting this preliminary finding the R.E. was tasked with designing an enclosure that would be more attractive to the customer than their existing
product. An initial prototype was designed, built and presented to the customer as a proposal for a joint development programme. The design relied upon the same combination of System E and phenolic foam as the Gas Safe and was designed to make use of the same manufacturing methods.

It was also designed to offer features that the existing product did not:

- It was an enclosure, rather than a moulded coating, so that it was completely separate from the actuator. This allowed servicing and replacement of the actuator and also meant that a single unit was suitable for any of the different actuator models.
- It was intended to be as easy to fit and install the actuator as possible. The actuator was fitted to the duct on a flat base that allowed easy access to the actuator and quick wiring of the cable glands.
- It was less than half the weight of the existing moulded solution, since it was known that it sometimes causes problems during installation on ductwork in ceiling voids.

The design solution was accepted by the customer and a joint development programme began. Seeking to avoid the lengthy iterative process used to develop the Gas Safe, the R.E. turned to the methods of analysing Heat Flow and temperature rise that had been developed towards the end of the Gas Safe project. Following the work described in submission 11 it was now possible to carry out more sophisticated calculations using the Heat Flow model.

Heat Flow Model Results

This model is described in detail in submission 11 so that information will not be repeated here. The model uses experimental heat flow data, measured over the period of a fire test, to calculate the heat rise of a known thermal mass. This data accurately reflects the reaction of intumescent char, and its improving insulation characteristics throughout the test, and combines this with the insulation provided by an inner layer of phenolic foam.

Unfortunately, the only heat flow data available was that from cellulosic fire tests that are considerably hotter that the fire conditions specified for this product, as shown in figure 2. However, this was initially accepted to be an acceptable "worst case" for a first run of the model. The model predicted failure taking place during the 23rd minute, as shown in figure 27. To improve the accuracy of this prediction an attempt was made to factor the heat flow according to the actual temperature of the furnace.

The equation for steady state heat flow, which is used in the model, states that heat flow through a panel is linearly dependent on the temperature difference between its surfaces. On this basis, the heat flow input in the model can be factored in a straightforward way using the new furnace temperatures. In practice this will not be accurate, not least because the reaction rate of the intumescent will alter and this will affect the heat flow.
However, it was thought to be an acceptable approximation for the second trial of this model.

![Graph showing actuator temperature rise predicted by the Heat Flow Model.](image)

**Figure 27:** Actuator temperature rise predicted by the Heat Flow Model.

The model predicted just over 30 minutes performance in the 400°C test, meeting the requirement, but only 36 minutes in the 300°C test, considerably lower than the 60 minutes required. In addition, the enclosure would be penetrated by four M6 studs, used to fix the actuator to its mounting bracket, and the 20 mm diameter drive shaft. These would all be exposed to heat for approximately 50 mm of their length and would therefore conduct additional heat into the enclosure.

More positively, it was known that the surface area used in the calculation was excessive. In reality the enclosure would be mounted 50 mm away from a cold block work wall or duct surface. The temperature in this gap would undoubtedly be lower than the rest of the furnace and this would substantially reduce the heat input over one of the largest faces. In addition, the R.E. had recently found a company that supplied a phenolic foam likely to be much superior to that used in the heat flow trials. It had a much superior measured insulation, at ambient temperature, and had also been specially formulated to resist very severe "jet-fire" conditions.

On this information, it was thought to be worth constructing two prototypes and carrying initial fire tests. This was done and the R.E. constructed a test rig allowing the actuator to be fire tested while electrically live, so that it could be motor driven throughout the test. This demonstrated that the actuator was operational by turning a loaded pulley using a shaft passing through the
furnace wall. The results of the first set of fire tests, at both temperatures, were as follows:

<table>
<thead>
<tr>
<th>Time to failure</th>
<th>300° C test</th>
<th>400° C test</th>
</tr>
</thead>
<tbody>
<tr>
<td>Predicted:</td>
<td>36 minutes</td>
<td>31 minutes</td>
</tr>
<tr>
<td>Actual:</td>
<td>42 minutes</td>
<td>27 minutes</td>
</tr>
</tbody>
</table>

NB: The time to failure was taken from the average actuator temperature so that it would equate to the even heat rise assumed by the model.

The performance in the 300° C test was 16% better than that predicted by the model, while in the 400° C test performance was 13% worse than predicted. Nevertheless, the results of the fire test were much closer to the model than expected, given the effects of the steel shaft and stud penetrations and the non-linear behaviour of intumescent with temperature. Therefore, this was regarded as a significant success, even though it was clear some development work remained to meet the specification.

Figure 28: An actuator cover before and after fire testing.

Ultimately, it took five 300° C tests to meet the specification but only three attempts were needed at the 400° C test. To do this, improvements were made to the joints and seals, to reduce any ingress of hot gasses, and a further 10 mm of insulation was added to the top surface of the enclosure. The customer then added an additional requirement that the enclosure pass a
30 minute, $600^\circ C$ test so that it would give an additional market advantage to the combined actuator/enclosure product. This was achieved by adding an external coat of Nullifire thin film intumescent that reacts quickly and provides a more effective char early in the test, as described in section 5.2.3. In total the development took 12 fire tests, to meet three different fire test curves, whereas the Gas Safe took 28 fire tests to meet a single temperature regime.

Once development was complete this test method was endorsed by the independent certifying body TÜV and the product certified as protecting the actuator for 30 minutes at $600^\circ C$ and 60 minutes at $300^\circ C$. Since the launch of the product in November 2000 over 600 units have been sold and the current rate of supply is 100 units per month. A long-term relationship has now been established with the customer, who predicts that potential sales across Europe will be a steady 6,000 units per year. If Nullifire is able to develop similar relationships with the customer's overseas divisions, worldwide sales could be several times greater.

### 6.1.2 Emergency Generator Diesel Tanks

As often happens, Nullifire was approached with an unusual fire protection problem. A new computer centre in London needed three large oil storage tanks in its basement to provide fuel to the emergency generator in case of power failure. Normal procedure is to enclose this type of fire hazard in a separate fire rated room but space constraints made it impossible in this case. The client hoped that an intumescent coating would be able to keep the steel tank cool enough to avoid adding fuel to the flames, despite exposure to a severe hydrocarbon fire for 120 minutes.

Once the R.E. began to analyse the problem a familiar and frustrating problem arose. The existing fire test certificates for System E are quite general and are aimed at typical elements of construction like columns, beams and bulkheads. Any unusual construction is not covered by these tests and the cost, and time, required, for fire testing usually rules out specific certification. As a result it is normal for architects and engineers to continue using familiar and traditional methods.

However, at this time the R.E. had recently completed the work eventually submitted as "Collecting and Making Use of Data on Heat Flow Under Fire Test Conditions in the Development of a Fire Resistant Enclosure". Methods learned from this work enabled the R.E. to perform calculations, based on standard certification to show that the failure conditions would not be reached in the 120 minute time period.

This was done by showing that the amount of heat flow through a given thickness of intumescent during a standard fire test would not be able to heat this twin skinned tank to the failure point. Even under the worst case assumptions that the tank was almost empty, and so had no additional thermal mass, and that the air gap between the skins would not provide any additional insulation were made, the failure temperature was not reached.
The R.E. submitted these calculations in the form of a structured argument, to the Building Control Officer responsible, and this solution was accepted.

Like most building works, this was a once off project and so the rate of return for the amount of work that the R.E. put in was modest. However, the experience will make it considerably easier to pursue and carry out similar work in future. In addition, the fact that the method has been accepted and installed before will be a powerful sales tool in future.

6.1.3 British Museum Fuse Box Enclosures

At about the same time another unusual fire protection problem was brought to Nullifire by the fire protection contractor responsible for the extension of the British Museum by renovation of the old Library Reading Room. In this case an error had been made in the installation of the emergency lighting system. The fuse boxes and switching mechanisms had been placed in unprotected areas. This meant that if a fire occurred it was quite likely that the emergency lighting system would not come on, or may go off at some point, which was clearly unacceptable.

The failure temperature of the units was around 100°C, like most electronics, and a total of 27 enclosures needed protecting all over the site. It seemed likely that the type of insulation developed for the Gas Safe and the Actuator Cover would be suitable. However, it was clear that a fire test and some form of independent certification would be necessary in this case since the size and design of the enclosures would be quite different from any previous test.

The main difficulty was that the museum extension was due to open in the new year and this only allowed 14 weeks to design, develop, test and install all 27 enclosures. To make matters worse, the emergency lighting system, and all other electrical systems were already completely installed. In practice this meant that the fuse boxes were all bolted to walls, closely surrounded by tightly packed cabling, other enclosures, ducts etc. This meant that each enclosure would have to be different and this would complicate testing, manufacture and installation enormously.

The R.E. began this process by negotiating the design of a single test, to represent all installations with the independent test witnesses TÜV and Building Control. A prototype installation of a fuse box and protective enclosure was then built into a furnace test frame and fire tested while electrically live. This showed that the design and level of insulation was slightly too low and an improved test sample was built. This second test was witnessed by TÜV and passed the criteria of one hour's protection against cellulosic fire. The experience of combining intumescent and foam insulation to provide protection to items with failure temperatures around 100°C, gained from the Gas Safe and Actuator Cover projects, was absolutely crucial to this project.

Each installation was surveyed by a draughtsman and drawn individually so that the idiosyncrasies of each site were recorded. The R.E. designed a flat
pack of parts for each installation that could be fabricated on site with hand tools. Once again the Coating Services division was too busy to provide labour for this contract so sub-contractors were used for manufacture and installation.

Once again, like most construction industry projects, this was a once off contract. The long term potential from this work is unclear and will depend on how it is exploited by the sales department. However, it is known that electronics enclosures of various types quite frequently require fire protecting. Usually this protection would be included at the design stage, rather than being an emergency addition. Clearly, this type of work would be much more attractive if a standard product, or range, similar to the actuator enclosure described above, could be offered since this would minimise the development required.

6.1.4 Ablative Protection of Rocket Motor Parts

The technical background to this project has already been described in some detail in section 5.1.3. It concerns the precision application of an ablative coating to a pair of rocket motor components. The rocket motor is being developed for use in a new missile being developed for the US Navy. At the moment this is in advanced development and a limited number of parts are required for prototype work and live testing. The order is for 112 pairs of components over the first year and this contract is approximately one third complete, although much delayed by design alterations.

Although the R.E. designed the tooling and method to be reasonably profitable on this limited production batch, it is hoped that there is long term potential. It is thought that once the prototyping phase of these rocket motors is over, more substantial production orders will follow. It is known the requirements of the US navy may be substantial and that several other countries have placed orders for the system.

The significance of this project may eventually be quite great. Coating Services holds two existing contracts of this type and has found them to be valuable in the long term. Although they require a great deal of development work and investment to begin with, and many such contracts never reach full production, they can form a stable core of work in the long term. Therefore, work of this type is thought to be very desirable and acquiring further contracts will form part of the Coating Services strategy that is under development. In addition, the final customer for this particular contract is completely new to Nullifire and the use of ablative is also new. Both of these factors may indicate the potential for further new business.

6.1.5 Circular Hollow Column Tops

This contract was a simple, once off moulding job. Weather resistant fire rated caps were needed for the tops of 100 columns on a large building. The experience of new moulding techniques meant that it was possible to design the fire protection solution and tooling for production in less than two hours.
The new methods also meant that it was easy to produce moulded System E caps at reasonable cost. Before the advent of closed vacuum form moulding this work would have been uneconomic because of the extremely wasteful and labour intensive nature of the old methods.

6.1.6 Feature Columns

The cardboard tube method, described in section 5.3.1, was in use for many years before the Engineering Doctorate but was improved by the addition of release film and other small design changes. This has reduced the installed cost of the system to the point where it is competitive and two contracts worth a total of £175,000 are currently in production. The sales department has tendered another seven bids and it is likely that some of these will be won.

The long term potential of this type of work is good as long as the construction industry is healthy. It is hoped that this will form a significant part of Coating Services production in the future.

6.2 ON GOING AND FUTURE OPPORTUNITIES

6.2.1 Automotive LPG vessel protection

One of the most technically interesting developments of the Gas Safe project has been the fire protection of the LPG vessels in dual fuel vehicles. The low cost of LPG fuel has increased the number of these vehicles greatly in recent years. However, a number of explosions have occurred after these cars have caught fire and there have been a few injuries and deaths. As described in submission 13, "Information in Support of the Gas Safe Project", the R.E. has undertaken joint projects with several other companies to develop a product to prevent this.

The objective of these projects has been to produce a compact, cheap and durable version of the Gas Safe to be fitted to the LPG cylinders in the boot or under the floor of vehicles. A certain amount of technical success has been achieved with the development of a much thinner wall section that gives equal protection. However, the best estimates are that protecting the LPG vessel in a car would add £100 to the cost and this is too high for car manufacturers at present.

The United Nations Economic and Social Council Working Party on the Construction of Vehicles is considering the possibility of making "thermal protection" for automotive LPG vessels mandatory. This means that it is possible that a market for this type of product may be created quite suddenly. However, it is more likely that the vested interests of the automotive industry and LPG supply companies will be powerful enough to prevent this happening. Nevertheless, if a market does arise, Nullifire is well placed to exploit it because most of the necessary development has already taken place. For the time being the company remains in contact with several potential customers and will engage in any joint projects that arise but will not carry out further work on its own.
6.2.2 European Domestic LPG cylinders

This project has already been described in submission 13, "Information in Support of the Gas Safe Project". It concerns the fire protection of small butane cylinders for a domestic European LPG market. It was initiated by a LPG distributor in order to obtain a market advantage over its competitors. Supermarkets and DIY shops will be permitted to sell cylinders that are fire protected for 20 minutes directly from their shelves. Theoretically this will give that brand a huge advantage over competitors' products that must be collected from depots.

Ironically, the enthusiastic involvement of the LPG cylinder owners has allowed direct coating of the cylinder to be possible. Very early in the Gas Safe project this method was discounted because of the hostility of British LPG suppliers to the Gas Safe concept. Direct coating of the cylinder reduces the fire protection to the minimum practical cost of a simple coating. In addition, because these cylinders do not include a pressure release valve, and do not rupture until an internal pressure of 90 bar is reached, only 3 mm of intumescent provides 20 minutes of protection. The predicted additional cost of the applied fire protection to each cylinder is approximately £10. The advances in moulding developed by the R.E., described in section 5.3.4., means that the 3 mm thick coating of System E is smooth and seamless.

So far 40 prototypes have been produced for evaluation by the customer and the decision about whether to proceed further will be made soon. Production would take place in Europe at the point of cylinder manufacture. Nullifire would profit from sale of the System E and from a modest royalty on each cylinder, or a technology transfer fee. A total of 700,000 cylinders would be protected if the project goes ahead. It can be seen that the potential revenue for Nullifire could be very considerable.

However, it is very difficult to judge the likelihood of this taking place and, although the solution is technically satisfactory, the ultimate decision will depend on the results of trial marketing. Therefore the success of this project will depend entirely on whether the theoretical market advantage really exists.

6.2.3 Heavy Industrial Valve Actuator Protection

Long before the R.E. joined Nullifire, part of the Coating Services portfolio of products was the fire protection of heavy industrial valve actuators. These items were fire protected by spray application of epoxy intumescent and were rather rough and ready. This work was lost to a competitor about 8 years ago because they were able to mould a similar epoxy intumescent to the valve components to give a smooth, neat finish. The reason for the loss of this work was not fully appreciated at the time and did not become clear until the customer returned to Nullifire last year.

After many years of use and thousands of components it had become clear to the customer that the competitor's material was not fully weather resistant.
Specifically it had been shown to be vulnerable to high temperatures and humidity, which were quite common conditions in some important markets. Therefore, the customer was interested in re-evaluating Nullifire as a supplier, providing that it could be shown that System E has superior all-weather durability and that it could be moulded to the components in an attractive way.

It is known that System E has excellent weather resistance because it has been extensively tested for use in all climates as part of its certification for use on oil platforms. Therefore it is expected that the material will pass the planned environmental test regime easily. It is also fortunate that the closed vacuum form moulding method developed by the R.E. is capable of producing the mouldings required. It is only in the past year that this capability has been developed, and comparison with mouldings produced by the competitor indicates that it will be possible to improve on them.

Nullifire is now six months into a two year joint development project with the customer. This project is being managed by the R.E. and will include environmental testing, fire testing of prototypes and the design of tooling to apply fire protection to the five actuator sizes that make up the customer's range. Past history suggests that this will be a long-term relationship if the development is successful.

6.2.4 Improved Moulding of Aircraft Engine Actuators

Nullifire has protected a particular aircraft engine actuator against fire using a spray applied epoxy intumescent for nearly a decade. The specification for this requires that a 3 mm thick layer be applied accurately to certain areas of the component with a smooth finish. The limitations of the spraying process, described earlier, mean that this is a very labour intensive process involving a great deal of masking and highly skilled spraying and trimming operations. There is also quite a high risk of rework being necessary, particularly when skilled staff are absent or training is taking place.

A project to replace this method by a moulding process similar to the one used to apply ablative to rocket motor parts, described in section 5.3.5., has recently been launched. The R.E. has proposed that accurately machined tooling will allow direct injection of the epoxy onto the surface of the actuator. This can potentially de-skill the process, greatly reduce material wastage through over-spray and improve the surface finish and accuracy of the finished parts. The customer has agreed to support this project by helping to fund prototype work and tooling costs.
6.3 FINANCIAL CONSIDERATIONS

6.3.1 The Cost of the Engineering Doctorate and Gas Safe Project

The cost of the project was estimated in the 11th submission, "Collecting and Making Use of Data on Heat Flow Under Fire Test Conditions in the Development of a Fire Resistant Enclosure". This information was presented as part of an argument to show that the use on heat flow data and modelling could greatly reduce development costs. However, the estimates of cost were only carried out up to the point of the first external fire test and so are completed in figure 29.

<table>
<thead>
<tr>
<th>Resources</th>
<th>Estimated cost</th>
</tr>
</thead>
<tbody>
<tr>
<td>Furnace costs (inc. O/H):</td>
<td></td>
</tr>
<tr>
<td>30 tests at £500 per test</td>
<td>£15,000</td>
</tr>
<tr>
<td>External Testing costs:</td>
<td></td>
</tr>
<tr>
<td>5 pool fire tests at DERA, £5000 each</td>
<td>£25,000</td>
</tr>
<tr>
<td>Material and equipment costs:</td>
<td></td>
</tr>
<tr>
<td>Estimated from purchasing records</td>
<td>£28,000</td>
</tr>
<tr>
<td>Engineering costs (inc. O/H):</td>
<td></td>
</tr>
<tr>
<td>4 years at 100% utilisation, roughly 4300 hrs,</td>
<td>£40,000</td>
</tr>
<tr>
<td>at EPSRC sponsored cost</td>
<td></td>
</tr>
<tr>
<td>Labour cost of fire test samples (inc. O/H):</td>
<td></td>
</tr>
<tr>
<td>35 at 16 hours each at £25 p.h.</td>
<td>£11,200</td>
</tr>
<tr>
<td>Labour cost of pre-production units (inc. O/H):</td>
<td></td>
</tr>
<tr>
<td>50 at 8 hours each at £25 p.h.</td>
<td>£10,000</td>
</tr>
<tr>
<td>Promotional costs</td>
<td></td>
</tr>
<tr>
<td>(Literature, advertising &amp; various trips)</td>
<td>£15,000</td>
</tr>
<tr>
<td>Management time (inc. O/H):</td>
<td></td>
</tr>
<tr>
<td>(15 hours/month on average at £75 p.h.):</td>
<td>£54,000</td>
</tr>
</tbody>
</table>

**Estimated total cost:**

£198,200

Figure 29: The Cost of the Engineering Doctorate and Gas Safe Project to Nullifire Ltd.

The figure of just under £200,000 compares with an estimate of £172,575 for first stage of Gas Safe development. The main reason that the figure is not considerably higher is that the cost of the R.E. has been reduced to reflect the real cost of employment to Nullifire.
6.3.2 Commercially Successful Projects to Date

The sponsoring company has gained a number of new capabilities as a result of the Engineering Doctorate and the Gas Safe project. The outcome of this has been a number of new projects that have all achieved some degree of commercial success. The financial effects of these projects are summarised in figure 30. To maintain commercial confidentiality the names of the customers are not given and the value of the contracts should be considered to be approximate. The real measure of commercial success, the gross margin of each project, is given as a proportion of the target margin for the Coating Services division.

<table>
<thead>
<tr>
<th>Project Name</th>
<th>Value</th>
<th>Proportion of Standard Margin</th>
<th>Potential</th>
</tr>
</thead>
<tbody>
<tr>
<td>Actuator Cover</td>
<td>£100 each</td>
<td>0.25 currently</td>
<td>1,200 p.a. currently</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.0 achievable</td>
<td>6,000 p.a. likely</td>
</tr>
<tr>
<td>Diesel Tanks</td>
<td>£25,000</td>
<td>0.5</td>
<td>Once off</td>
</tr>
<tr>
<td>British Museum Fuse box covers</td>
<td>£80,000</td>
<td>0.75</td>
<td>Once off</td>
</tr>
<tr>
<td>Ablative protection, Rocket Motor Main Body</td>
<td>£150 each</td>
<td>1.5</td>
<td>112 units ordered</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Repeat potential</td>
</tr>
<tr>
<td>Ablative protection, Rocket Motor Cover</td>
<td>£450 each</td>
<td>1.5</td>
<td>112 units ordered</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Repeat potential</td>
</tr>
<tr>
<td>Column Caps</td>
<td>£10,000</td>
<td>1.5</td>
<td>Once off</td>
</tr>
<tr>
<td>Feature Columns</td>
<td>£175,000</td>
<td>Ongoing</td>
<td>Repeat potential</td>
</tr>
</tbody>
</table>

Figure 30: Financial summary of recent Coating Services projects.

The actuator cover project has suffered initially from a lack of investment in production engineering time and in labour saving tooling. As a result production was initially very labour intensive and early units were made at a loss. This situation has now been resolved, by team action between Quality, Production and the R.E, and a modest profit is now being made on this product.
The confidence in the long-term potential of this product is such that the R.E. is also undertaking a cost reduction exercise. This exercise depends on an improved design that reduces part count and the labour needed for moulding and assembly. A fire test programme is also taking place to supply the Heat Model with data so that the insulation materials used can be optimised with respect to cost. These improvements will result in much improved margin when the new design goes on sale in January 2002.

Three of the projects listed above are described as being "once off", that is to say that they were concerned with a single order with no definite repeat business. However, each of them should result in occasional repeat work based on the improved capability developed for that specific project. For example, the Column Cap project would have been unthinkable three years ago. Even six months ago, before the advent of the closed vacuum form method, it would have been uneconomic. Following development work on other projects, the R.E. was able to design the tooling for this project in about two hours and it was the most profitable project undertaken.

The ablative protection of rocket motor parts is also theoretically very profitable. However, the end product is still currently under development and this has resulted in erratic "call off" orders and frequent design changes. Nevertheless, it is likely that this product will transfer successfully into volume production and the annual production would be equal or greater than the initial order. At that point, genuine profits would be achieved and the effort invested in developing a reliable, precision application method would be rewarded.

The market for Feature Columns is known to be considerable, many construction projects include atria that require slender, smooth and hard wearing columns to achieve maximum impact. Although this is a longstanding Nullifire product, the improvements to the production process made by the R.E. have reduced the cost and improved the quality considerably. Two large contracts have now been won, suggesting that the cost of the installed system has now fallen to the point where it is affordable. There are currently another seven bids for further work under consideration by architects and main contractors. There is clearly plenty of this type of business available, the key question is whether the predicted margin can be met since it is highly dependent on wastage and scrap rates in production and installation labour.

It should also be noted that the introduction of moulding as the main method of production has reduced material wastage. The reduction in over-spray alone is estimated to be 5% of the total material usage. The recent acquisition of a meter fed plural injection machine should be even more significant. In recent times up to 20% of each kit of material was wasted during the mixing, pumping and spraying process. It is fully expected to be below 5% now and the cost of epoxy intumescent will make this a considerable saving. It is estimated that at the current rate of usage of System E this saving will amount to around £5,000 per year.
6.3.3 Commercial Potential of Ongoing Projects

Predicting the effect of ongoing projects is more difficult because some may not be successful; contract value and margin is also much less certain. Current development projects are summarised in figure 31, ranked from the one least certain of completion to that most likely to succeed.

<table>
<thead>
<tr>
<th>Project Name</th>
<th>Value</th>
<th>Proportion of Standard Margin</th>
<th>Potential</th>
</tr>
</thead>
<tbody>
<tr>
<td>Automotive LPG</td>
<td>Unknown</td>
<td>Unknown</td>
<td>Unknown</td>
</tr>
<tr>
<td>Domestic LPG</td>
<td>£3.2 M</td>
<td>0.33</td>
<td>Over 3 years</td>
</tr>
<tr>
<td>Valve Actuator Protection</td>
<td>£400,000 p.a.</td>
<td>1.0</td>
<td>10 Year estimated</td>
</tr>
<tr>
<td>Aircraft engine actuator moulding</td>
<td>£100,000 p.a.</td>
<td>+ 0.2</td>
<td>5 year estimated</td>
</tr>
</tbody>
</table>

Figure 31: Possible financial effects of future Coating Services projects.

The automotive LPG vessel protection, based on improved Gas Safe technology is the hardest to predict. Even though Nullifire and others have developed the required compact insulation technology the market is completely dependent on new safety legislation. Although it is known that legislators in Europe are moving in this direction it is not at all certain when thermal protection will be required, if ever. The domestic LPG project is also very hard to predict because, although the customer's technical specification has been met, the authorisation for the project depends on the results of a market trial of the concept. There is also some doubt about the viability of this project because the low margin does not allow much room for error and it has inherent risks relating to production.

However, the heavy industrial valve actuator protection is far less risky. Nullifire System E is capable of providing the fire resistance and environmental durability required without further development. Although the mouldings required are slightly more complex than those carried out to date there do not appear to be any serious problems likely. Although this project requires another 18 months of steady development, there are few technical risks associated with it. The main concern must be that the customer requirement, or perspective, may change over the development period.

The improvement of the intumescent application technique to the aircraft engine actuator contract is even less risky. It is based entirely on known technology, injecting epoxy intumescent into machined tooling. The only complicating factor is the geometric complexity of the three parts in question. However, it appears very likely that rapid prototyping technology will allow the
creation of tooling at reasonable cost. The benefits in reduced material waste through over-spray, reduced labour content and skill requirement and the virtual elimination of rework will be considerable.

6.3.4 Long Term Potential of New Technology

Although 50 pre-production Gas Safe units were made, only some were actually sold at a rate of £150 - £200. Many were given away as samples and many went abroad, to Hong Kong, Brazil, India, Sweden, Italy and Israel to name a few countries. In these cases the carriage was normally charged but the samples were free. As a result, although the potential income from the 50 units was £10,000, the actual income was much lower. The Gas Safe project ran for four years and it is estimated that it consumed approximately £200,000 of company money. In comparison, any income from the project is negligible.

In early documents stating the objectives of this Engineering Doctorate, two parallel themes were identified. The first theme was the development of new products, initially the Gas Safe, to meet the companies commercial needs. The second theme was the improvement of the fire testing and production technology available to the company. This second theme has been seen as less important by the company for most of the period of the doctorate. However, it has ultimately been more successful than the first and the positive commercial impact of this will outweigh that of the Gas Safe project.

This section attempts to predict the impact of the Engineering Doctorate on the Coating Services division over the next four years, making use of the commercial information presented above. To do this three scenarios will be constructed, ranging from the minimum likely impact to the most optimistic reasonable outcome.

**Scenario 1: Actuator Cover Only**

The first scenario assumes that no further projects are successful and that no further orders arise from the "once off" projects. This would mean that the only ongoing business would be the steady orders for actuator covers, and the current, low level of orders are assumed to continue.

<table>
<thead>
<tr>
<th>Projects</th>
<th>2001</th>
<th>2002</th>
<th>2003</th>
<th>2004</th>
</tr>
</thead>
<tbody>
<tr>
<td>Actuator Cover</td>
<td>£120,000</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Diesel Tanks</td>
<td>£25,000</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>British Museum</td>
<td>£80,000</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ablative Rocket Motor</td>
<td>£67,000</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Column Caps</td>
<td>£10,000</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Feature Columns</td>
<td>£175,000</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Valve Actuator Protection</td>
<td>0</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Domestic LPG</td>
<td>0</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Automotive LPG</td>
<td>0</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td>£477,000</td>
<td>£120,000</td>
<td>£120,000</td>
<td>£120,000</td>
</tr>
</tbody>
</table>
Scenario 2: Further success based on technical probability

This scenario is more optimistic and is based on the assumption that those projects that have been commercially and technically successful will continue to be so. Therefore, more orders are won based on the technology developed for the "once off" projects, with, for example an order similar to the British Museum contract won every other year. In this scenario the Actuator Cover achieves the level of production predicted by the customer and the ablative protection of rocket motor parts becomes regular business at an equal level of production. In addition, the Feature Column market continues at the current level. This scenario also depends upon some further technical progress so that the Valve Actuator Protection project achieves modest commercial success in 2003, according to the programme.

<table>
<thead>
<tr>
<th>Projects</th>
<th>2001</th>
<th>2002</th>
<th>2003</th>
<th>2004</th>
</tr>
</thead>
<tbody>
<tr>
<td>Actuator Cover</td>
<td>£120,000</td>
<td>£280,000</td>
<td>£440,000</td>
<td>£600,000</td>
</tr>
<tr>
<td>Diesel Tanks</td>
<td>£25,000</td>
<td>£25,000</td>
<td>0</td>
<td>£25,000</td>
</tr>
<tr>
<td>British Museum</td>
<td>£80,000</td>
<td>0</td>
<td>£80,000</td>
<td>0</td>
</tr>
<tr>
<td>Ablative Rocket Motor</td>
<td>£67,000</td>
<td>£67,000</td>
<td>£67,000</td>
<td>£67,000</td>
</tr>
<tr>
<td>Column Caps</td>
<td>£10,000</td>
<td>£10,000</td>
<td>£10,000</td>
<td>£10,000</td>
</tr>
<tr>
<td>Feature Columns</td>
<td>£175,000</td>
<td>£175,000</td>
<td>£175,000</td>
<td>£175,000</td>
</tr>
<tr>
<td>Valve Actuator Protection</td>
<td>0</td>
<td>0</td>
<td>£125,000</td>
<td>£250,000</td>
</tr>
<tr>
<td>Domestic LPG</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Automotive LPG</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td>£477,000</td>
<td>£557,000</td>
<td>£897,000</td>
<td>£1,127,000</td>
</tr>
</tbody>
</table>

It is also important to state that although this level of success does not depend upon further technical progress, it does require that company sales activities continue at the current level.

Scenario 3: Most projects prove successful

This most optimistic scenario makes three extra assumptions in addition to those described above. Firstly, that the production volume of the rocket motor parts is double the current level, which is simply of prototype, test and pre-production units. Secondly, that the volume of fire protected Valve Actuators meets the levels predicted by the customer rather than the more cautious figures used above. Thirdly, that the domestic LPG cylinder has a successful market trial and the product is taken into production by the customer.
### Summary of Commercial Potential

The three scenarios described above cover the full range of likely outcomes and these are presented in graphical form in figure 32. The R.E. favours scenario two, which predicts that the income derived from products and

![Figure 32: Sales from new Coating Services products projected to 2004.](image-url)
projects arising from the Engineering Doctorate will rise from nearly £500,000 to just over £1M over the next three years. This analysis does not take account of competitive action or the possibility of recession, which could have a serious effect on all the projects described above. However, it is firmly based upon the new technical capabilities of the Coating Services division and so it is perfectly achievable.

This possible future must be compared with the £200,000 that the company invested in the Engineering Doctorate and the Gas Safe project. It is not possible to draw a curve to show the cumulative expenditure and income for these projects without revealing the gross profit margin for the division. However, if it assumed that the sales predicted by scenario 2 are reached, and that these achieve the standard level of gross margin then the money invested will be recovered before the end of 2002.
7. SUMMARY AND CONCLUSIONS

7.1 THE CONTRIBUTION OF THE ENGINEERING DOCTORATE

7.1.1 Improvements In Development Theory And Practice

When the R.E. began the Engineering Doctorate the methods of development and testing used at Nullifire were not well suited to engineering new products. The existing methods had arisen from testing the predominant product group within the company; intumescent coatings. These materials are a complex blend of resins, polymers, intumescent materials and rheology agents. Predicting the behaviour of such materials under fire conditions is very difficult and, at best, is guesswork based on experience.

Under these conditions the most appropriate method of developing a new coating is to create a range of formulations and to test them under fire conditions. The most promising is then selected and gradually refined through an iterative process of fire testing. It has long been understood at Nullifire that this is a time consuming and expensive process. As a result various screening tests have been developed that compress the early stages of testing by reducing sample size and cost, and by allowing more than one sample to be tested at once.

Although these methods are acceptable for developing coatings they are very inappropriate for developing the engineered fire protection solutions that the R.E. has been working on. This is because the complex geometry, design details and material combinations means that interpreting results is difficult and so the number of tests required is very much higher. This was demonstrated by the development of the Gas Safe which took 28 furnace tests over a period of 23 months.

The R.E. has now demonstrated the value of carrying out engineering design calculations, based on data on heat flow, at the beginning of the development process. For new materials, or material combinations, this data may be collected quickly using the type of screening test methods already used by the company. For existing materials it may often be calculated from historical test data. This more analytical approach means that the first prototype to be constructed can be based on sound predictions of performance. This greatly increases the chance of early success because it is more likely that the correct thicknesses of the most suitable materials have been chosen first time.

The potential of this method has been demonstrated by the development of the thermally protective actuator cover. Although considerably smaller than the Gas Safe the situation was much more complex. The fire resistant enclosure was penetrated by bolts, cables and a drive shaft, all of which affect performance in an unpredictable manner. Despite this, the use of a simple heat flow computer model allowed a good choice of materials and thickness to be made first time. As a result, only 12 tests were needed for three different temperature regimes whereas the Gas Safe required 28 for only one.
7.1.2 Manufacturing capabilities

At the beginning of the Engineering Doctorate the Coating Services division used two epoxy intumescent products in two different ways. They were normally spray applied, and it was accepted that this was a wasteful process which was difficult to control. Applied thicknesses of material were quite variable and the surface finish was poor. This method was also extremely labour intensive because a great deal of masking, de-masking and trimming were required. The materials were also sometimes cast into simple moulds. Geometry was limited, normally to flat sided shapes using moulds built out of polypropylene sheet and chipboard, or to tubular shapes cast from disposable cardboard tubes.

The introduction of new and improved production methods by the R.E. has dramatically increased the complexity and quality of parts that can be made from epoxy intumescent. The use of vacuum form moulds allows complex parts to be created with a good surface finish, with relatively little material waste and a much lower labour content. This method also allows the incorporation of other insulating materials or fixings as inserts and in-mould paint finishes so that the mouldings are essentially complete at the point of de-moulding. The design of the tooling may be tailored to suit the required life and accuracy, ranging from cheap, single use, disposable tools to machined aluminium tooling to provide precision parts for the duration of a contract.

In the past the initial approach by Coating Services to any fire protection enquiry was to ask how the problem could be solved using System E. This placed severe constraints upon design solutions and often made proposals impractical, uneconomic or unsatisfactory to the customer in other ways. The R.E. has gradually changed this approach by introducing different materials, such as the phenolic foam used in the Gas Safe. It is now accepted that access to a wide range of different insulation materials is necessary to meet customers needs in the best way. Although a natural bias remains towards Nullifire products, especially where intumescents are required, the initial approach is now open minded.

7.1.3 Product range

When the R.E. joined Nullifire the Coating Services division only really had two products. These both involved spray application of epoxy intumescent to defence and aerospace components. Both contracts were quite long term and reasonably profitable but left the division very vulnerable to changes in customer demand and it was difficult to obtain more work. Some moulding work also took place but this was very erratic and small scale.

As a direct result of the R.E.'s work the division now has four long term customers and both new customers are in new market areas. There have also been several substantial one off contracts that demonstrate a greatly improved capability in design and manufacture. Therefore, the division is less vulnerable to changing customer demand and has a larger pool of potential customers so the potential for expanding the business is much greater.
7.2 SUMMARY OF INNOVATIONS

7.2.1 New test methods and analytical techniques

- The measurement of heat flow is quite commonplace in some industries and is used to collect information on heat loss and the performance of thermal insulation. It is not generally used in fire testing and the benefit of this additional information on the performance of materials in fires is not well understood. The R.E has successfully used data on heat flow through materials under fire conditions to predict the heat rise of components inside insulated enclosures under similar conditions. This engineering approach allows calculations to be carried out during the design process to help material selection and reduce the amount of fire testing on prototypes needed. Use of this method in practice has shown the reduction in development time and cost to be real and significant.

- The R.E. has also shown that the mass of historical fire test data within the sponsoring company is of use. It may be used to calculate the heat flow through given thicknesses of the entire range of intumescent products under fire test conditions. This allows the generation of a series of curves showing heat flow against time for each thickness of each intumescent. Information of this quality is extremely useful in the design process because it is not specific to geometry and allows the performance of a range of options to be calculated before testing begins.

7.2.2 Innovative Combination of Materials

- The R.E. has been innovative in the combination of existing materials to achieve superior levels fire performance. This has evolved from a fresh understanding of the behaviour of different materials under fire conditions and a realisation of their strengths and weaknesses.
  - System E epoxy intumescent and various types of fire resistant foam have been combined to give levels of fire performance eight times higher than either alone. This solution allows high levels of fire protection to be achieved at low cost and weight.
  - The R.E. has also experimented with combining System E and layers of knitted fire resistant fabric in partnership with a French company. This has shown similar levels of fire performance to the epoxy/foam composite, at a greatly reduced thickness but increased cost.
  - Finally, the R.E. has combined System E with Nullifire thin film intumescents to minimise the time it takes to produce insulating char. This improves the reaction speed of the system to fire and greatly reduces heat flow through it in the early stages of a fire.

7.2.3 Innovative Products

- The Nullifire Gas Safe is an innovative product that has created a great deal of interest and won praise for its design and fire performance. It has shown that fire protecting LPG cylinders in a practical way is possible for the first time. Although its high cost prevented it from being a commercial
success it has generated several more carefully targeted projects to fire protect LPG vessels in specific niches. So far, one of these projects has achieved complete technical success within the budgeted cost and has a good chance of becoming the first commercially available fire protected LPG cylinder.

- The Actuator Cover was directly derived from the technology developed for the Gas Safe and used the new analytical methods described in 7.2.1 in its design. It is commercially successful, with over 600 units sold to date and a healthy, long term market open to it. It is innovative in its design which separates the fire protected enclosure from the actuator, improving flexibility in the customer's stockholding and reducing installed cost.

- The British Museum Fuse box enclosures were also derived from the Gas Safe and also achieved commercial success. No other fire protection company was able to offer a solution to this problem. The speed of development, certification and installation that the methods developed by the R.E. allowed were crucial to this successful project.

7.2.4 New processing methods

- In order to permit economic production of high quality parts made from epoxy intumescent the R.E. introduced a series of moulding techniques to the sponsoring company. These methods have been adapted from production methods used elsewhere, particularly the composites industry. Nevertheless, achieving the required accuracy, surface finish and, most especially, unit cost has required considerable innovation:
  - The use of commonly available thermoplastic films as a re-useable moulding surface has allowed production of parts with one degree of curvature with a very smooth surface finish. It has also been possible to combine this with in-mould painting techniques to permit the moulding of finished components with a lower labour content and production time.
  - Spraying epoxy into vacuum formed moulds has allowed the production of geometrically complex parts with a very good surface finish at low cost. Using a vacuum form in conjunction with a thermoplastic plate, or another vacuum form, has allowed a form of injection moulding to take place. It is now possible to produce accurate moulded components, incorporating fragile or complex inserts, out of epoxy intumescent. The key innovation is that the use of carefully designed moulds made from cheap materials means that the 24-48 cure time of System E is acceptable.

7.3 FINAL STATEMENT

The cost of these innovations to the sponsoring company is estimated to be approximately £200,000, over a four year period. In the 2001 calendar year the exploitation of these new products and technologies has added over £400,000 to company sales. A realistic scenario for future growth indicates that this figure should rise to over £1 M in the next three years providing that the momentum of sales activities is maintained.
References


