A STUDY OF NEW PRODUCT MARKET SEARCH AND USER ADOPTION.

Volume II

A Thesis, submitted for consideration for the award of Doctor of Philosophy.

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<u>VOLUME II</u>

Contents

APPENDIX A

Carbon/Carbon Case History

A(ii) Information sources for carbon/carbon

All of the information for this case study was gained while the author was working at Dunlop Ltd. Much of it came from either the personal experience of the author or other employees associated with the carbon/carbon development programme. Other information came from manufacturers of carbon/carbon in the USA and most of the reactions by potential users were in response to direct approaches by the author.

A1 Introduction

Technology transfer between the UK and USA (and other countries) is common place nowadays - particularly if the transfer is from the UK. It makes a pleasant change then to find a company in this country which has successfully exploited an American bred technology. For that is what has happened with one of the latest twentieth century materials - carbon/carbon. The name may sound ambiguous, for it is in fact all carbon - but made as a composite material; with carbon fibres embedded in a carbon matrix.

Dunlop, the only British manufacturer has exploited carbon/carbon's exceptional thermal properties for use in aircraft brakes, particularly on Concorde. Whilst Dunlop certainly won the first round in getting the first carbon/carbon brakes off the ground, the American manufacturers have done much to redress the balance. Whether Dunlop can find sufficient business to rely solely on aircraft brakes for carbon/carbons or have to diversify the usage of the material into other fields remains to be seen. This history describes their efforts to develop a high technology material for aircraft brakes and later their attempts to identify applications for more general use.

A2 Discovery of carbon/carbon composites

To say which establishment first discovered carbon/carbon is rather difficult. Here in the United Kingdom there is no doubt that we generally had a lead over the rest of the world in carbon fibre technology. Not only had carbon fibre been produced here first from polyacrylonitrile (PAN) at the Royal Aircraft Establishment, Farnborough by Dr. W. Watt, FRS and his colleagues, but developments with the final composite (generally carbon fibre reinforced plastics - CFRP) were more advanced than elsewhere, though we later suffered a setback with the Rolls Royce RB 211 engine.

Engineers and scientists are constantly seeking better materials and the field of fibre composites is no exception. Even with a material with a high specific strength, engineers wanted a higher temperature composite than that obtained with CFRP. Here the limiting factor is the matrix which melts or degrades at relatively low temperatures. Hence the search for a better matrix material giving rise to fibre reinforced metals and fibre reinforced carbons.

Scientists at the Atomic Weapons Research Establishment (AWRE) Aldermaston began looking at the concept of carbon/carbon composites about 1966 (1). They investigated a method of manufacturing the carbon matrix by charring the resin matrix in a CFRP composite.

At the same time in America, Speciality Carbons (now taken over by the Carborundum Co.) noticed that the charring action of resins gave ablative protection for such uses as rocket motor nozzles and, later, heat shields for space re-entry vehicles. Such action led to the study of carbon fibre reinforced plastics being deliberately charred to give a carbon matrix and hence a carbon/carbon composite. Even before this, another organisation - Vought Corporation at Dallas - claimed (2) to be "actively engaged in the development and application of carbon/carbon since 1958", but information as to how and why they started is not available. Another firm, Super Temp Company in California (part of Ducommun Incorporated) manufactured graphite and high temperature alloys. During 1969, one of their engineers noticed an interesting phenomenon which occurred during the processing of pyrolytic graphite (3). Carbon felt, which was used as an insulating material in the processing furnaces, became encrusted with carbon during the CVD (chemical vapour deposition) process. Ever since, Super Temp have been developing such composite materials - looking for stronger and less expensive carbon/carbon materials.

From these beginnings, an industry for the manufacture of carbon/carbon started. Though it may be difficult to say who first discovered the principle of carbon/carbon composites, it was certainly the Americans who pioneered the commercial production of this material following both the resin route* and the CVD route. It is this technology, using the

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* A description of the resin route and CVD route for manufacture of carbon/carbon is given on page 387.

CVD method of manufacture in particular, that Dunlop has been able to exploit in the manufacture of brake materials, and gave rise to Dunlop leading the Americans with their brake technology.

Since those early days other organisations have become involved with developments in carbon/carbon, such that now, the following American and Japanese companies are working in this sphere:

Abex	Hitco
Atlantic Research Corp	Mitsubishi Electric
Avco	Monsanto
Bendix	Pfizer Incorporated
Carborundum	Raytheon
Ferro Corp	Super Temp
Fiber Material Incorporated	Stackpole Carbon Company
Fiberite Corp	Toho Beslon
General Electric Company	Torray and Nippon Carbon
Goodyear	Union Carbide
Hercules Incorporated	Vought Corp

In addition work is going on in various US Government Departments:

US Atomic Energy Commission

US Air Force Materials Laboratory

Oak Ridge National Laboratory

NASA

In Europe the following organisations have been involved with carbon/carbon.

AWRE)
Fordath) in the UK
Dunlop)

SEP)
Le Carbone) in France

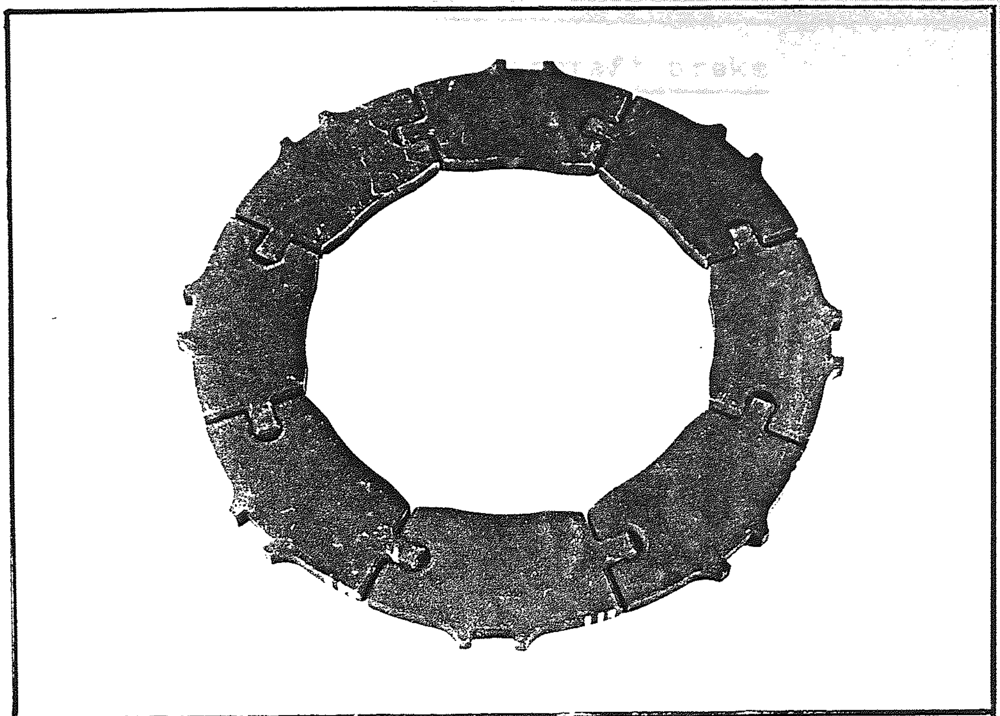
Sigri in Germany

A3 Dunlop look for a lightweight brake

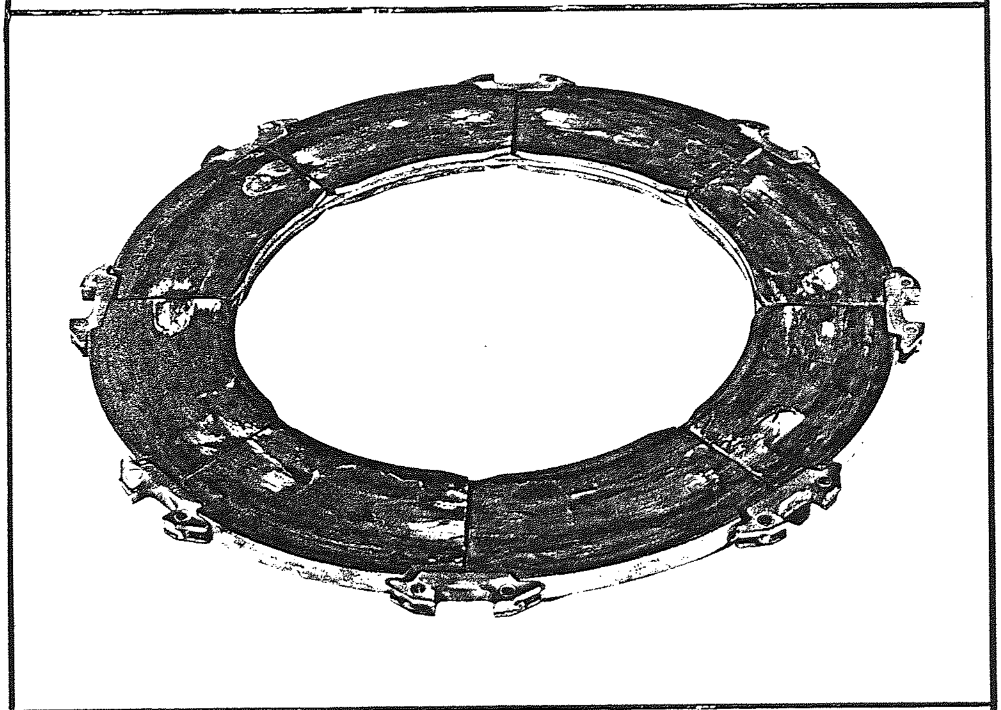
During the late 1960's Dunlop realised that a lightweight brake would be necessary for the aircraft of the 1970's and 1980's. The advent of heavier aircraft, with greater payloads, possibly landing at higher speeds meant that a lighter, more efficient brake would be needed by the airframe manufacturers. Until this time, nearly all aircraft brakes used steel as the brake friction material. Even today most aircraft use steel heat sink discs of which Dunlop produce three types as shown in figure A.1. The brake itself can be represented by a basic 'H' section as in figure A.2, the pressure and thrust plates being connected by the central torque tube.

The concern here, is the material selection for the heat pack - the area contained within the 'H' section. The heat pack is constructed from discs of the desired material, so that a rotor - rolling with the wheel - rubs face to face with a stator - located on the torque tube.

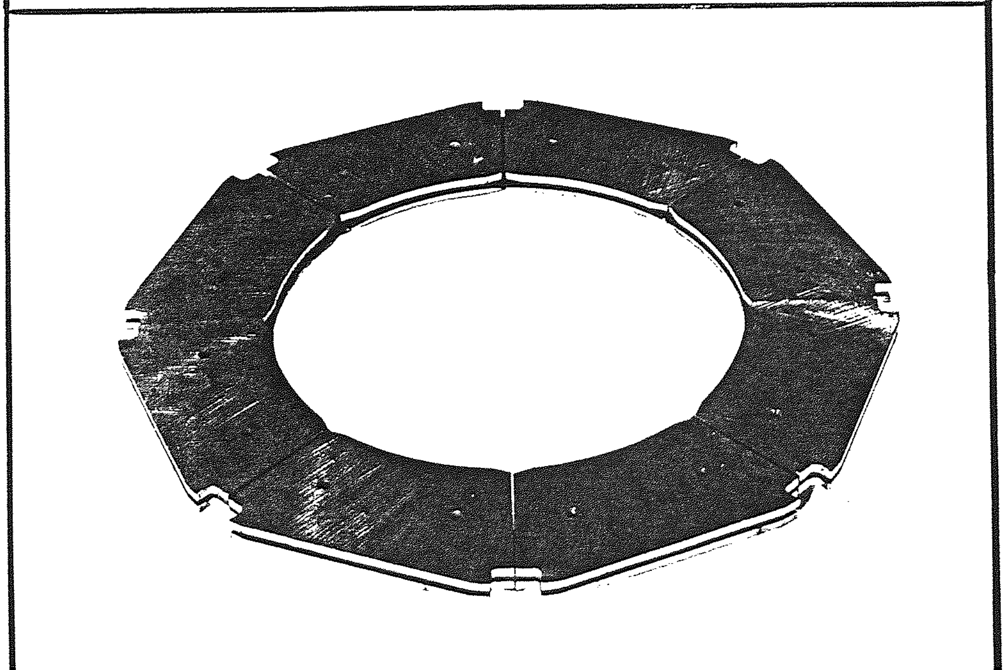
Figure A.1 Three Types of Steel Brake Disc Manufactured by Dunlop



(a) Jigsaw Rotor

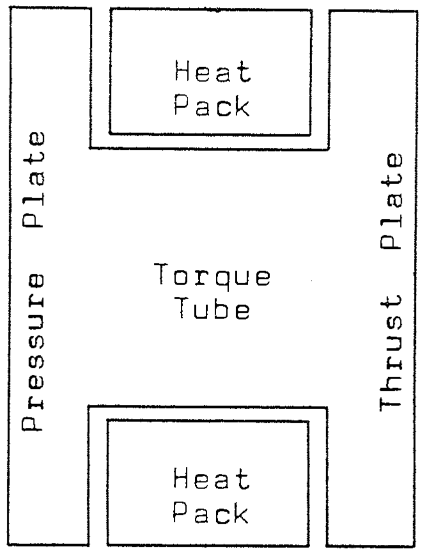


(b) Spider Rotor



(c) Window Type Rotor

Figure A.2 The basic 'H' section representation of the aircraft brake



(Source: Weaver 1972)

About 1968, Mr. Bayly (then Director of Aviation Division) encouraged a much greater effort in the development of lighter weight brakes. The task of searching for a suitable material fell to Mr. Ian Stimson (Engineering Manger Wheels and Brakes). The main function of an aircraft brake material is to convert the kinetic energy of the rolling aircraft to thermal energy and dissipate this heat as rapidly as possible - similar to a car disc brake. Thus, the material chosen as the heat sink must have a high specific heat. According to Dulong and Petit's Law, the product of the atomic weight and the specific heat, i.e. the atomic heat, is a constant, approximately equal to 6·4 calories per gram-atom. Table A.1 below indicates those elements with low atomic numbers and their corresponding specific heat value.

Table A.1 : The atomic number and specific heat of some elements

Element	Atomic No	Specific Heat (kJ/kg °C at 1000°C)	Density (g/cc)	Melting Point	Comment
Hydrogen	1	15 approx	-	-	) Gas
Helium	2	5·2 approx	-	-	)
Lithium	3	4·3 approx	·534	186°C	
Beryllium	4	3·3 approx	1·85	1280°C	
Boron	5	2·1 (at 900°C)	2·3	2300°C	
Carbon	6	1·9*	1·6	3500°C	(sublimes)
Nitrogen	7	1·2	-	-	Gas
-					
-					
-					
Iron	26	0·7	7·86	) 1530°C	
(Steel)		0·49		)	

* figure quoted is for carbon/carbon

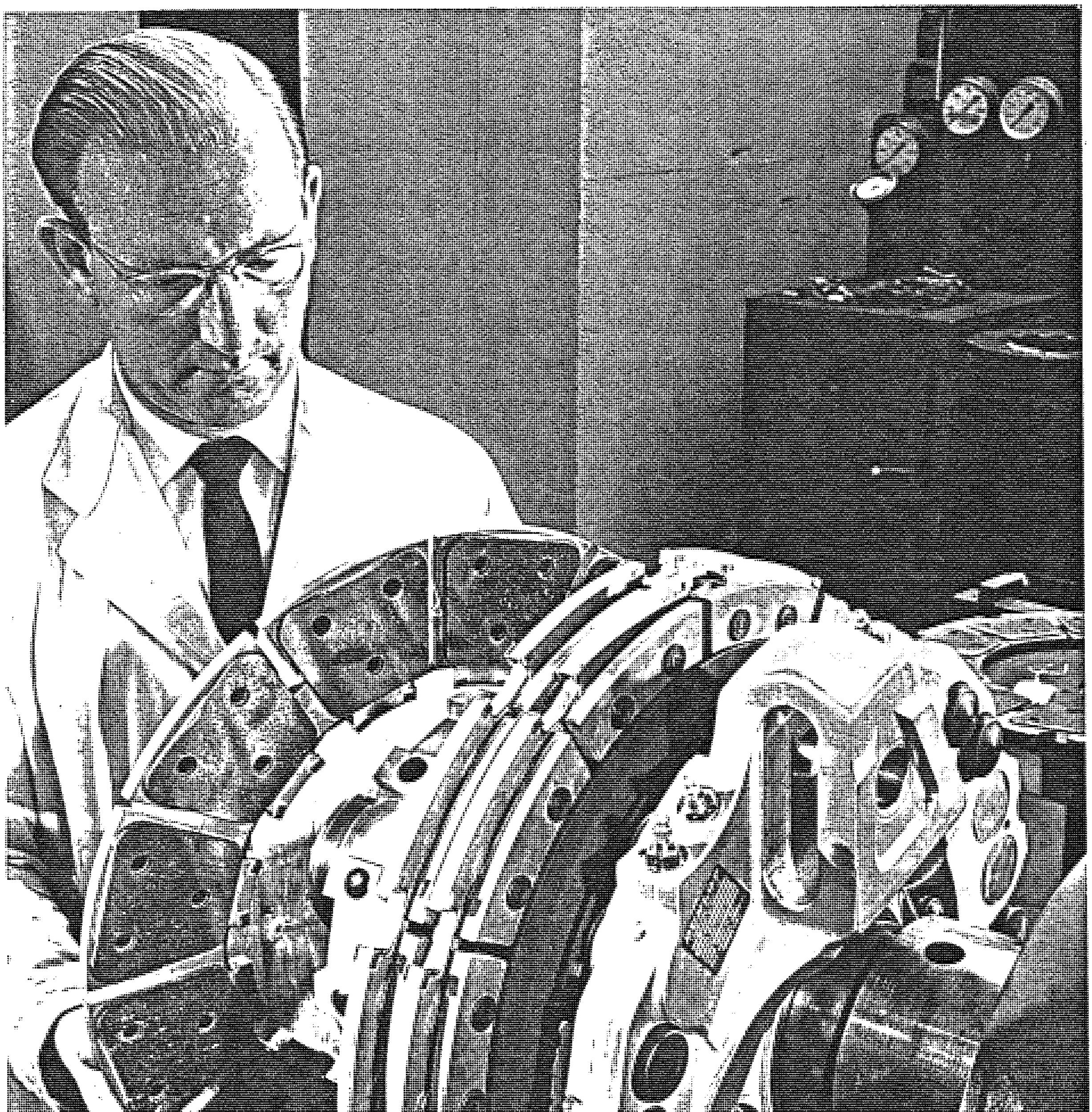
Obviously some of the elements ruled themselves out - the gases for instance and lithium because of its low melting point. This meant beryllium was the first choice material. Boron was not considered because as far as was known no one had ever manufactured a structural component with the material. By 1964 development work with beryllium had started. There was difficulty initially in forming beryllium to the desired shape, and the first made was a 'cased' beryllium brake. This was beryllium powder encased in a steel disc housing - the steel acting as the friction material and the beryllium acting as the heat sink. Even had it been possible to manufacture beryllium as a structural disc at that stage, it would not have been feasible to allow beryllium discs to run one against the other because of the toxicity of beryllium oxide. However, by 1966 Dunlop had perfected a method of manufacturing cased beryllium brake discs - and had the brake undergoing trials in a BEA Trident aircraft.

During 1967, Dunlop were approved by the manufacturers of Concorde to be the brake suppliers; either conventional sintered steel brakes or beryllium brakes.

By 1968, it looked as though beryllium would form the next generation of aircraft brakes. It had been fully type-tested to meet the Air Registration Board's (ARB) requirements and a structural beryllium brake was under development (see figure A.3). However, there still remained the fear of toxicity from the beryllium oxide which necessitated all operators wearing protective gloves and masks.

So when Bayly heard of developments with a new structural carbon in the USA, Dunlop decided to investigate this material. Stimson visited all the carbon manufacturers in America during 1969, with the aim of buying suitable carbons for brake manufacture. He came back with sample material from two companies - Union Carbide and Super Temp. It appears there were four companies making structural carbons - also known as carbon/carbon - at that stage, the other two being Carborundum and Monsanto. Of these four, Super Temp only were developing the material by the CVD route - the others were using the resin route. Carborundum was

Figure A.3 Structural Beryllium Brake Assembly



supplying their material to Goodyear, one of Dunlop's main rivals in the aircraft brake field. Monsanto's development had not reached a commercially acceptable standard and apparently they no longer wished to be involved with carbon/carbon for they asked several companies if they would purchase their carbon activity. Dunlop said 'no', and as it turned out Bendix, another of Dunlop's competitors in the brake field, bought the technology. They and Goodyear have gone on to supply most of the carbon/carbon brakes to America's military aircraft in recent years, although they have both changed their technology to include manufacture by the CVD route.

By the end of 1969, Dunlop had investigated the potential of the two types of carbon/carbon - from the resin and CVD routes. Mr. Norman Smith (Chief Metallurgist) recommended that material made by the CVD route would offer the greater development potential. In essence, the CVD route was chosen for Concorde because the size of the heat pack was likely to be less than with the resin route. In fact it was doubtful at that stage if there was sufficient space even for a CVD heat pack. In addition the material had better mechanical strength properties, better friction properties in wet and dry conditions and superior oxidation resistance. Another factor must have been, that Dunlop's competitors had chosen the resin route, and Dunlop wanted to lead the technology rather than follow it.

When Stimson first visited Super Temp, they (Super Temp) had no thought of using their material for aircraft brakes. They had no knowledge of brake design and it was the Dunlop staff, led by Fred Dowell, who pioneered the design configeration. Super Temp were purely being used for their expertise in manufacturing such materials. They were admittedly very enthusiastic over the possibility of using their material for aircraft brakes, and must have realised early on that they were in with a chance of manufacturing material for Dunlop, and licensing their technology.

During 1970, test and development work on carbon/carbon from Super Temp started in real earnest both at Coventry and Dunlop's Research Centre in Birmingham. Even so, the beryllium brake was still being developed at this stage. About this time Norman Smith and Dr. Ron Fisher (Manager Advanced Materials Laboratory) realised they would need someone with expertise in the understanding of fibre reinforced composites, if they were to progress with carbon/carbon. That someone was Dr. John Weaver who had just finished a PhD at Nottingham University on fibre reinforced metals. He was taken on in January 1971. Before doing any work on carbon/carbon for brake discs, he went to work with Dr. G. M. Jenkins (who acted as a consultant to Dunlop for a few years) at the University of Swansea, who was also investigating the material, its properties and method of manufacture. This enabled Weaver to look at carbon/carbon in its own right rather than as a brake material. With this behind him, he returned to Dunlop and proceeded to work on friction problems associated with the material and later on to solving many of the production problems that occurred.

By the end of 1971, Dunlop felt they could convince the appropriate authorities that they had the ideal brake, wheel and tyre package for Concorde. Those responsible for the commercial development of the brake project arranged a suitable seminar but omitted, initially, to invite those responsible for the technical developments. And on the 10th December personnel from Aerospatiale, BAC, the British and French air authorities and ministries were treated to a presentation of Dunlop's technology. The main selling point being the interchangeability of the three brakes that Dunlop had developed for Concorde - conventional sintered steel, structural beryllium, and carbon/carbon. At the close of the presentation Dunlop management were more hopeful of selling their brake technology.

No doubt Dunlop felt they had fought off the competition for the Concorde contract - but no sooner had they thought that, when Goodyear stepped into the picture. With not a little panache, they flew the French officialdom from Toulouse over to their Akron site in America to try and rescue the Concorde brake from Dunlop. The outcome certainly looked as though they had succeeded for they ended up with an order for four sets of brakes - and Dunlop were virtually told they were off the aircraft.

This setback no doubt made Dunlop fight harder. After all, the Concorde contract still promised much. Whoever won it would be supplying brakes - both original and replacement for the airlines - for hopefully well over one hundred aircraft, as the manufacturers were then claiming. Remember between March of 1967 and the end of June 1972, the manufacturers had seventy four options on Concorde - then Air Canada cancelled their options, followed by other major airlines - Pan Am, TWA, American Airlines, etc. (4). In January of 1972 Dunlop successfully stimulated the full "reject take-off" requirements for Concorde (the heat sink had to absorb 70 million joules of kinetic energy) with the structural carbon brake. At the same time they were insisting that both their brake and Goodyear's should be subjected to technical tests by an independent authority. So in April of 1972 the French aviation authority, S.T.Ae at Toulouse tested carbon/carbon brakes from Goodyear and Dunlop. Dunlop's confidence was justified, for the Goodyear brake broke up whilst the Dunlop brake cracked, but continued to function.

With this obvious success behind them, development work on the carbon/carbon brake continued rapidly. A trial set of brakes for the Super VC.10 aircraft was soon completed and was in operational use by October. Of the eight brakes on the VC.10, one set was replaced by carbon/carbon. The aircraft was instrumented to record all the operating variables for the carbon/carbon brake and the associated steel brakes on the same landing gear.

More importantly, the success of the Toulouse trials gave Dunlop the confidence to order 2000 brake discs from Super Temp and take the decision to build their own manufacturing facility at Coventry. In winning the contract to supply carbon/carbon brakes to Concorde, Dunlop had to agree to there being two suppliers of the material. This meant they could not rely on Super Temp as the sole suppliers. The reason the product support people of BAC and Aerospatiale insisted on a dual supply source probably lies in the fact that Super Temp (at Santa Fe Springs, California) is sited near the San Andreas fault and is liable to earth tremors. On March 8th, 1973 a letter of agreement was signed with Super Temp to provide the necessary technical expertise. It took over a year though before the necessary licensing agreement was drawn up and signed on the 5th September, 1974. Not that this delayed developments with the brake - the first set of brake discs were put on Concorde during 1974 and a Super VC.10 had become the first aircraft to fly regular airline service equipped with carbon/carbon brakes in 1973.

Following the signing of the letter of agreement in the spring of 1973, Dunlop arranged for some of their people to visit Super Temp to acquaint themselves with production and quality control methods. Duncan Cunningham (carbon technologist) spent one month and Stan Worvell (supervisor of the manufacturing facility) two months with Super Temp towards the end of 1973.

Whilst the CVD facility was being built at Coventry, Mr. Lockwood-Goose who was Product Support Manager and made frequent visits to the USA, acted as project co-ordinator. He flew many times between Coventry and Santa Fe Springs in California, smoothing out any problems that arose; he saw the CVD facility completed by the early summer of 1975.

Dunlop had thus completed their search for a lightweight brake material, and had their own manufacturing site some five years after the search was instigated. Not only that, they had won the contract to supply the brakes on Concorde; and had the satisfaction of knowing that it was they, who pioneered the first carbon/carbon brakes on a commercial aircraft. With such a success, they no doubt felt confident in being able to supply future generations of aircraft, both civil and military using the most advanced brake materials available. The search for further applications has continued since with some limited success to date but the widespread acceptance of carbon/carbon brakes has yet to happen in the airline business.

A4 The Concorde Brake

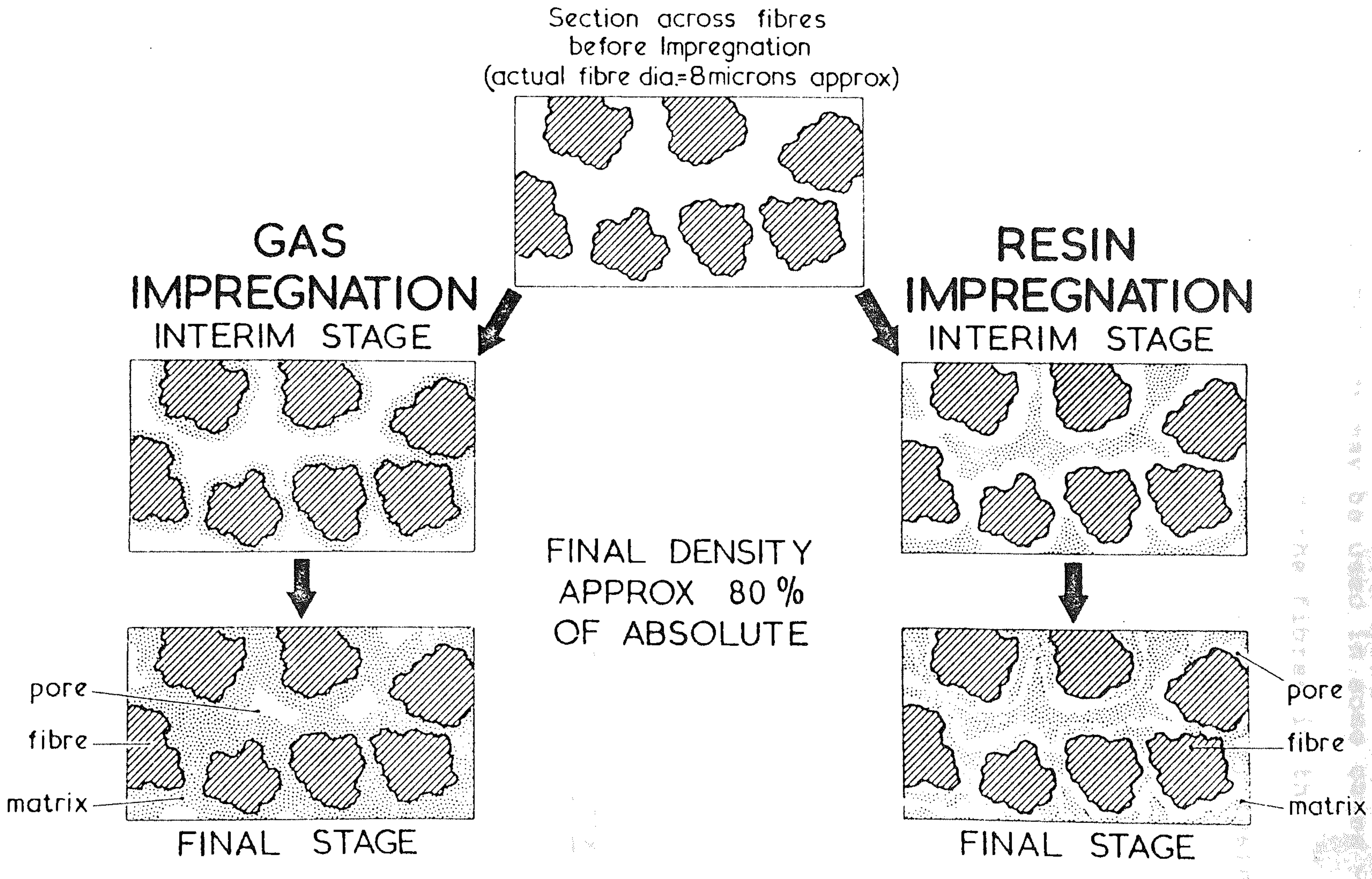
A4.1 Manufacturing Method

The process for manufacturing structural carbon, involves permeating the appropriately shaped assembly of carbon fibre with a hydrocarbon gas. At temperatures up to 2000°C the gas is cracked - depositing carbon on the individual carbon fibres and thus joining them together within a matrix of carbon. This process is generally termed chemical vapour deposition (CVD).

Another method of manufacturing the carbon matrix is to impregnate the bundle of carbon fibres with a resin and then to char the composite. This cycle of impregnating with resin and charring is repeated until the desired density for the material is achieved. Figure A.4 illustrates the two

Figure A.4

MECHANISMS OF IMPREGNATION



DIAGRAMMATIC REPRESENTATION

different methods for impregnating the carbon fibres. A combination of the two methods may be used in some cases - for instance, when one needs to hold the fibres in the desired shape before infiltrating by the CVD method, resin may be used to bind the fibre bundles together initially. Though the resin char method is more rapid than the CVD, the properties of the finished composite are generally inferior. Certainly a more dense material can be obtained with the CVD process; and this leads to a material with a better resistance to oxidation and contaminants, improves the strength and also the wet friction characteristics. Table A.2 illustrates the difference for density and flexural strength for composites made by the CVD and resin routes.

Table A.2 Mechanical strength of various fibre - matrix system composites

	CVD Matrix		Resin Matrix	
	Rayon Precursor	PAN Precursor	Rayon Precursor	PAN Precursor
Density (g/cc)	1·55-1·60	1·75	1·45	1·60
Flexural (MN/m^2)	103	186	69-83	103
Strength (p.s.i.)	15×10^3	27×10^3	$10-12 \times 10^3$	15×10^3

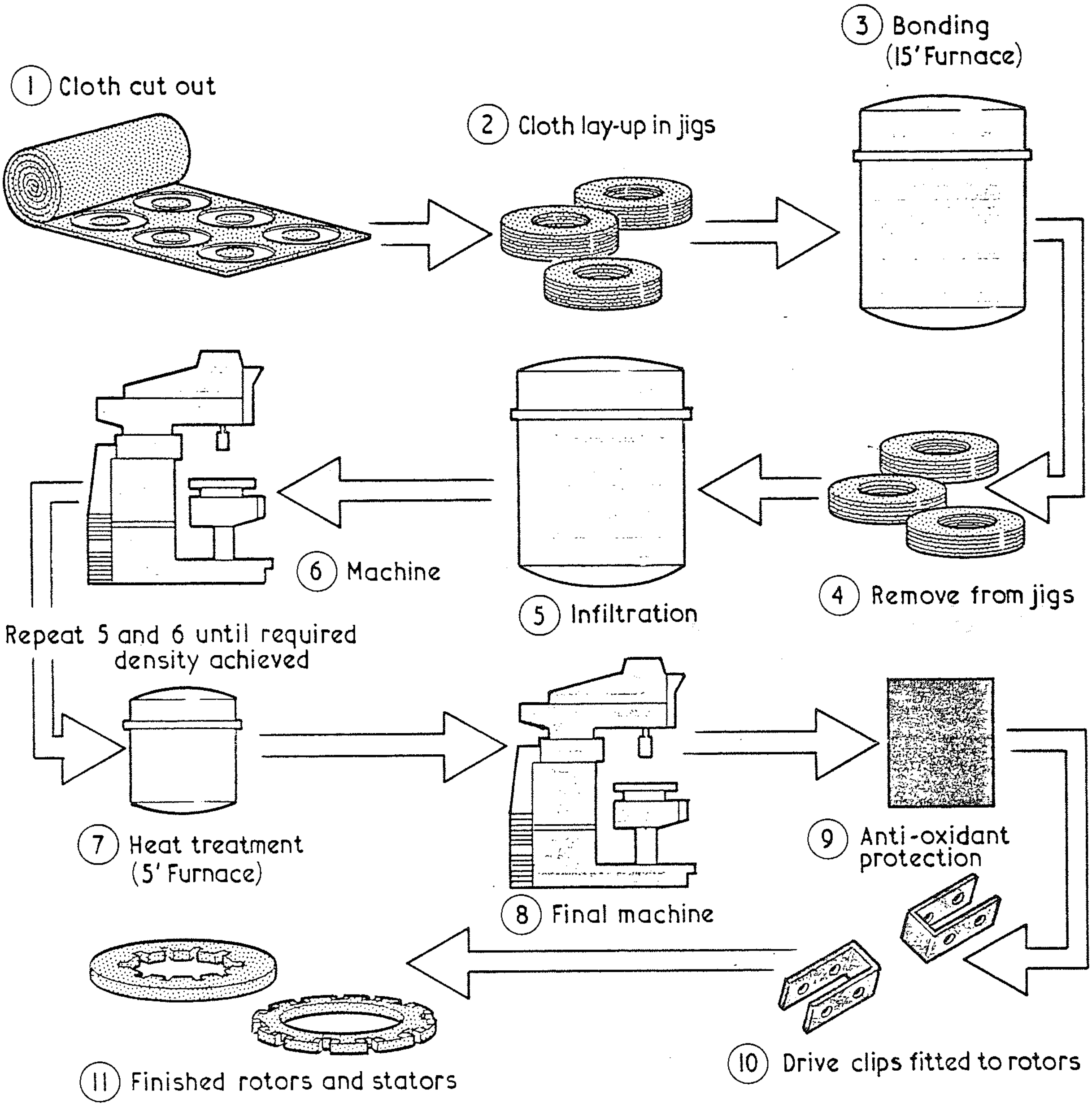
As has already been stated the decision to opt for the CVD route was taken by Dunlop because it offered a more versatile system and gave a better mechanical properties to the finished composite. Having said that, there is still room for improvement with the CVD process. And Dunlop continue to look at ways of improving the process technology to give a better composite material, in terms of its properties and price. A recent report (5) indicates the potential developments that could occur with further studies of the chemical and physical aspects of the process; the effects of pre and post treatments to the composite. The use of alternative, possibly cheaper substrates and a better fundamental understanding of the philosophy of design with the material may also lead to more effective, less costly processing.

In the case of Concorde disc brakes the process starts with a woven fibre cloth, whose precursor was rayon. Other forms of carbon fibre can be used depending on the article; for example chopped strand mat, felt, tow, etc. The precursor and the form of carbon fibre affects not only properties but may also influence the price of the final article.

Several layers of cloth are cut to shape, cleaned and laid up between graphite jigging plattens to form a pile some twenty five per cent thicker than the final thickness required. Figure A.5 illustrates the production process. The work piece is then processed in a vacuum furnace (see figure A.6) for several weeks. During this time, the discs will be removed from the furnace at several stages. Firstly after an initial bonding run, the jigging plattens will be removed once the work piece has been consolidated. The discs will again be removed from the furnace at later stages for machining operations. This is necessary because the pore size in the composite reduces as the carbon is deposited. A point is reached when no more gas can permeate the composite because a surface crust of carbon has closed off all the pores. This crust must be removed periodically by machining,

Figure 3.1

CARBON DISC MANUFACTURE - C.V.D. PROCESS



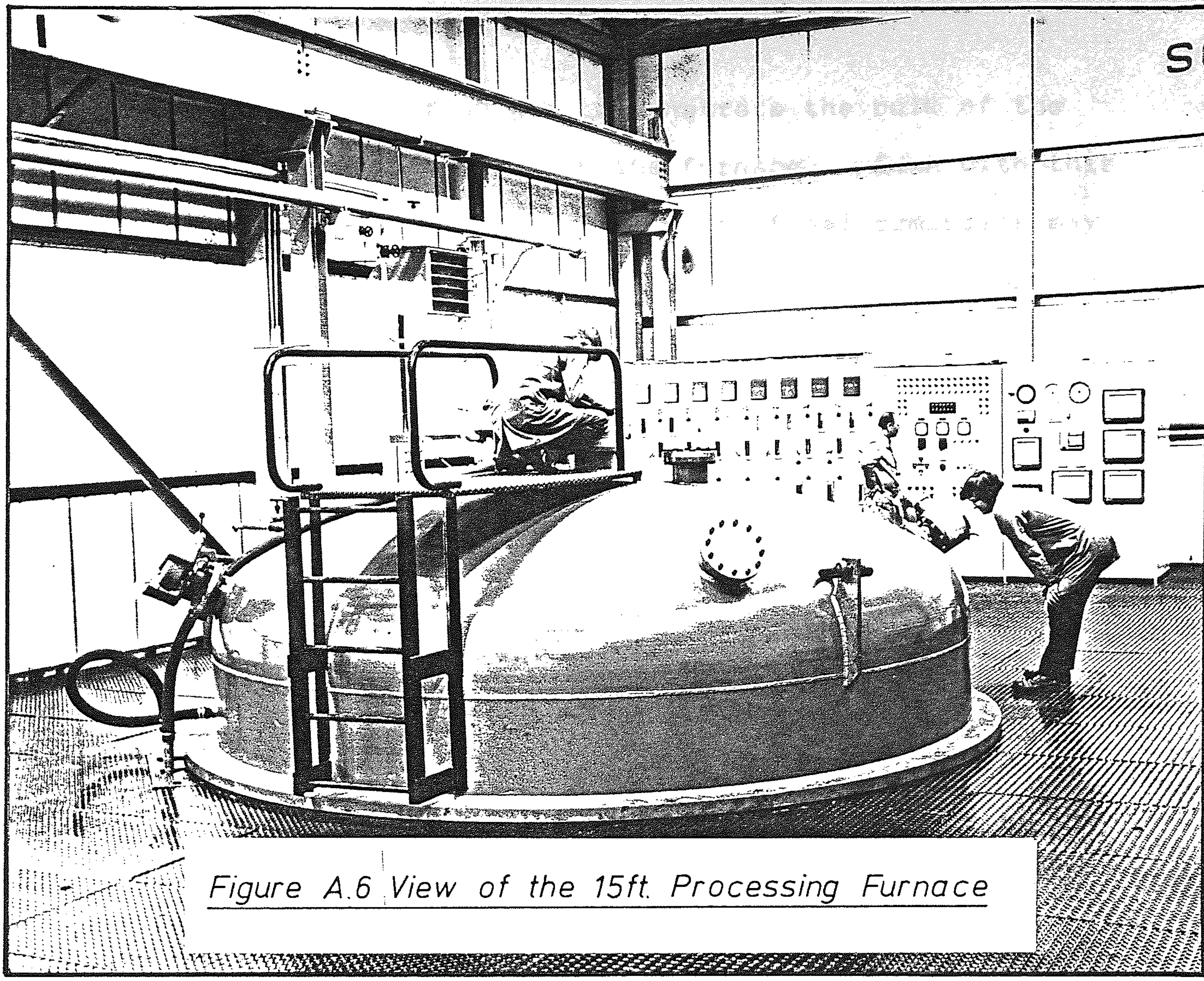


Figure A.6 View of the 15ft. Processing Furnace

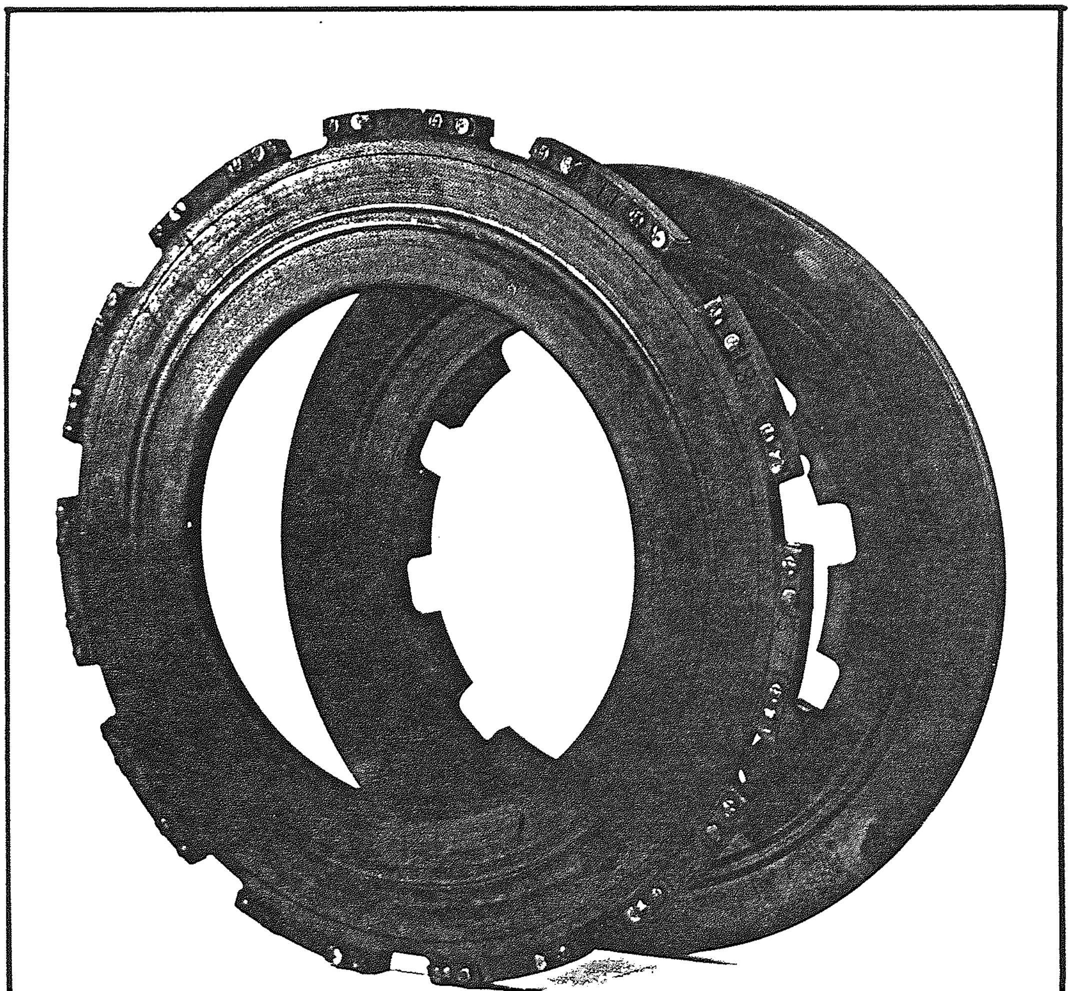


Figure A.7 Concorde Rotor and Stator

which will allow fresh gas to penetrate the bulk of the material when placed back in the furnace. Even with this repeated machining the density of the final composite may vary from say 1·7 g/cc at the outer surface to 1·5 g/cc at the centre.

Following the infiltration process, the composite may undergo a heat treatment operation to modify the properties of the material. This operation again takes place in a furnace at temperatures in excess of 2000^{o}C, and has the effect of increasing the thermal conductivity, whilst decreasing the overall strength of the composite. In some instances the heat treatment operation may precede the final one or two infiltrations.

After the final machining operations, the edges of the disc are given an anti-oxidant treatment, and drive clips are fitted to the rotors - see figure A.7. The end result is a composite having a thirty per cent fibre volume faction - seventy per cent is deposited carbon with a pore size of 10-15 microns. The density of the finished material varies according to the thickness of the composite - about 1·6 g/cc for 13 mm thick material, and 1·45 for 25 mm thick material. Thinner composites obviously take less time to process and will be correspondingly cheaper. The same applies with composites of lower density - if only one or two infiltration runs are necessary the processing costs will be that much less.

A4.2 Properties of the composite

A composite material such as that described above, has anisotropic properties, being made from distinct layers of cloth. This anisotropy causes the properties of the material to vary depending on the orientation of the fibres.

Some of the general physical and mechanical properties of the material are shown in Table A.3 below and compares them with other materials.

Table A.3 Some of the general physical and mechanical properties of carbon/carbon in comparison with other materials.

	Carbon/ carbon	Aluminium	Steel	Metal Impregnated carbon	CFRP
Density (g/cc)	1·6	2·78	7·86	2·8	1·34
Temperature capability (°C)	2500**	260	750	750	300
Modulus of elasticity (GN/m^2)	12	70*	207*	2	109*
($X10^6$ p.s.i.)	1·7	10	30	0·3	16
Flexural strength (MN/m^2)	100	427*	1990*	9	1410*
($X10^3$ p.s.i.)	15	62	290	1·33	204
Coefficient of Thermal expansion ($10^{-6}/^oC$)					
Longitudinal	+0·5	+24	+11	+5	-0·7
Transverse	+1·0	-	-	-	-1·0
Specific heat (cal/gm°C)	·168-·46	·22	·12	·17-·45	
at temps. quoted (°C)	(20-1000)	(15-185)	(20-100)	(20-1000)	

* Strength and modulus quoted are the maximum possible at the upper limit of their temperature capability.
** In inert atmosphere. (900 in oxidising atmosphere).

A more detailed breakdown of the properties that can be expected on the Concorde brake discs are given in tables A.4, A.5 and A.6 and figure A.8. In general though, carbon/carbon composites exhibit a unique combination of features, and these are listed as follows:

- Chemical inertness to most corrosive agents
- High thermal and electrical conductivity (with heat treatment)
- Low thermal expansion coefficient
- High resistance to thermal shock
- Low density
- High specific heat
- Absence of melting behaviour
- Good machinability
- Non-toxic
- High operating temperatures - 2500^{o}C in inert atmosphere, otherwise 900^{o}C with anti-oxidation treatment.
- Biocompatible

The items which have been made so far, are generally of simple shape - plates, discs, cones, tubes, rods, etc., although some success has been achieved with moulded items (see figure A.9).

Generally tolerance maintenance is not as good as say, steel, and low temperature creep and movement after machining is noticeable, but not so much as with plastics.

Table A.4 General data sheet of structural carbon by Dunlop

Property		
Fibre Volume%		34
Density (g/cc)		1·6
Flexural Strength	(MN/m^2)	103
	(p.s.i. x 10^3)	15
Compressive Strenth	(MN/m^2)	138
	(p.s.i. x 10^3)	20
Interlaminar Shear	(MN/m^2)	17
Strength	(p.s.i. x 10^3)	2·5
Elastic Modulus	(GN/m^2)	11·72
	(p.s.i. x10^6)	1·7
Thermal Expansion:	Longitudinal	+0·5
($\times 10^{-6}$ cm/cm/^oC)	Transverse	+1·0
Thermal Conductivity:	Longitudinal	0·17
cal/$cm^2 s ^oC$	Transverse	0·060
Friction Coefficient		0·1 - 0·35
Electrical Conductivity	$(mho/cm)^+$	625
Thermal Shock resistance*	(W/m)	
	Longitudinal	350,000
	Transverse	37,700
Strain to failure		0·9% approx.
Impact Strength	(Nm/mm of notch)	310
	(ft lbs/in of notch)	0·950

* Thermal Shock Resistance determined using Gangler Parameter

i.e. Gangler Parameter $A = \frac{\sigma . K}{\alpha . E}$

σ = Stress
K = Thermal Conductivity
α = Thermal Expansion
E = Elastic Modulus

Ref: J. J. Gangler - Journal American Cermaic Society 33 (1950)

See also Table A.5 for comparison with other materials

\+ See also Table A.6 for comparison with other materials.

Table A.5 Thermal shock resistance of various materials

Gangler Parameter $A = \frac{\sigma \cdot K}{\alpha \cdot E}$

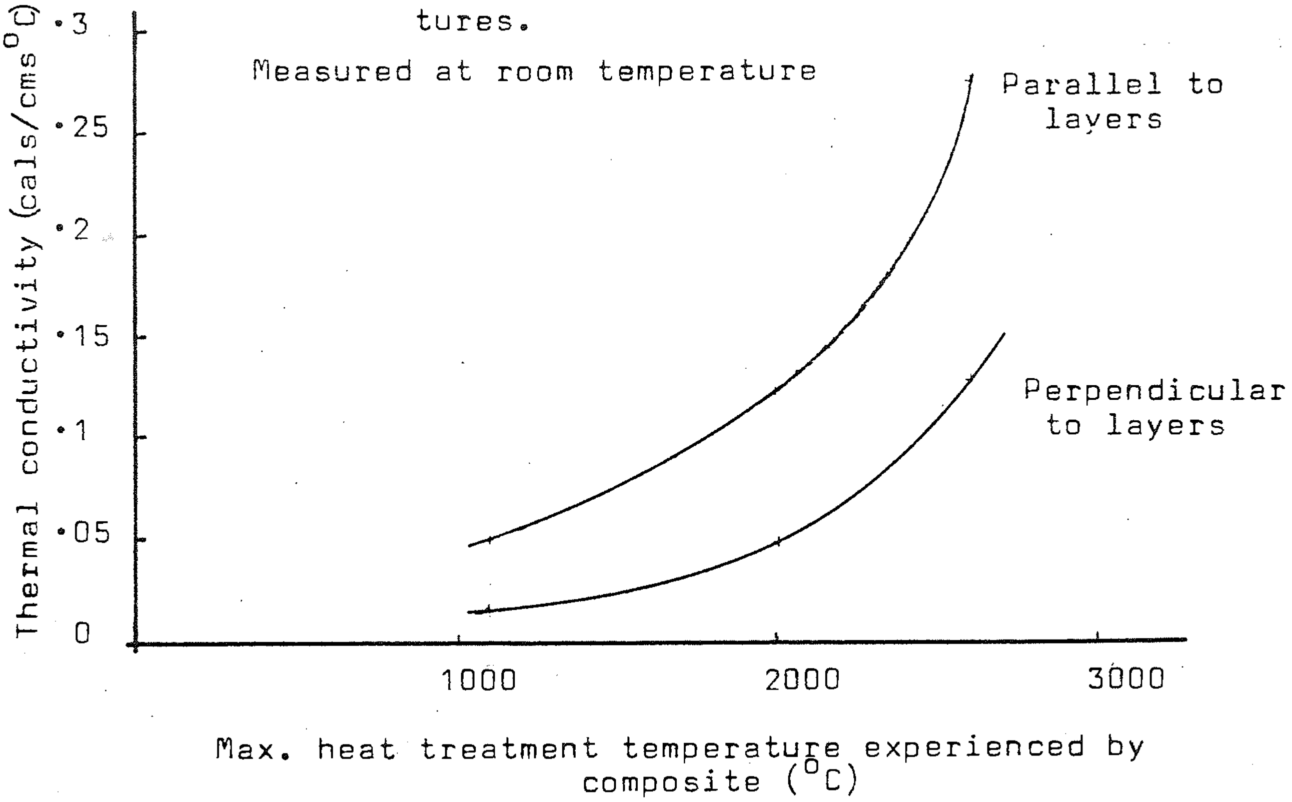
Material	A Value Cals. $cm^{-1}s^{-1}$	W/m
Metal		
Copper	100·0	42,000
Oxides		
MgO	3·0	1,250
AL_2O_3	27·0	11,300
Mechanical Carbons		
CCA	21·0	8,800
CCP	47·0	19,700
AUT	79·0	33,100
Commercial Grade Carbons		
CS	277·0	116,000
ATJ	270·0	113,000
AGW	330·0	138,000
Carbon Composite		
Cloth laminate heat treated to 2700°C.		
(a) Parallel	(a) 831·0	350,000
(b) Perpendicular to layers of cloth	(b) 90·0	37,700

Table A.6 Comparisons of electrical conductivity.

Substance	Electrical Conductivity mho/cm
Carbon/carbon	$6{\cdot}25 \times 10^2$
Graphite	$3 \times 10^3 - 1{\cdot}6 \times 10^4$
Carbon	$1{\cdot}4 \times 10^2$
Iron	$1{\cdot}1 \times 10^2$
Aluminium	$4{\cdot}0 \times 10^5$
Alumina	$10^{-11} - 10^{-14}$

Figure A.8 Variation of thermal conductivity and specific heat in carbon/carbon composites.

Variation of thermal conductivity with heat treatment temperatures.



Variation of specific heat with temperature

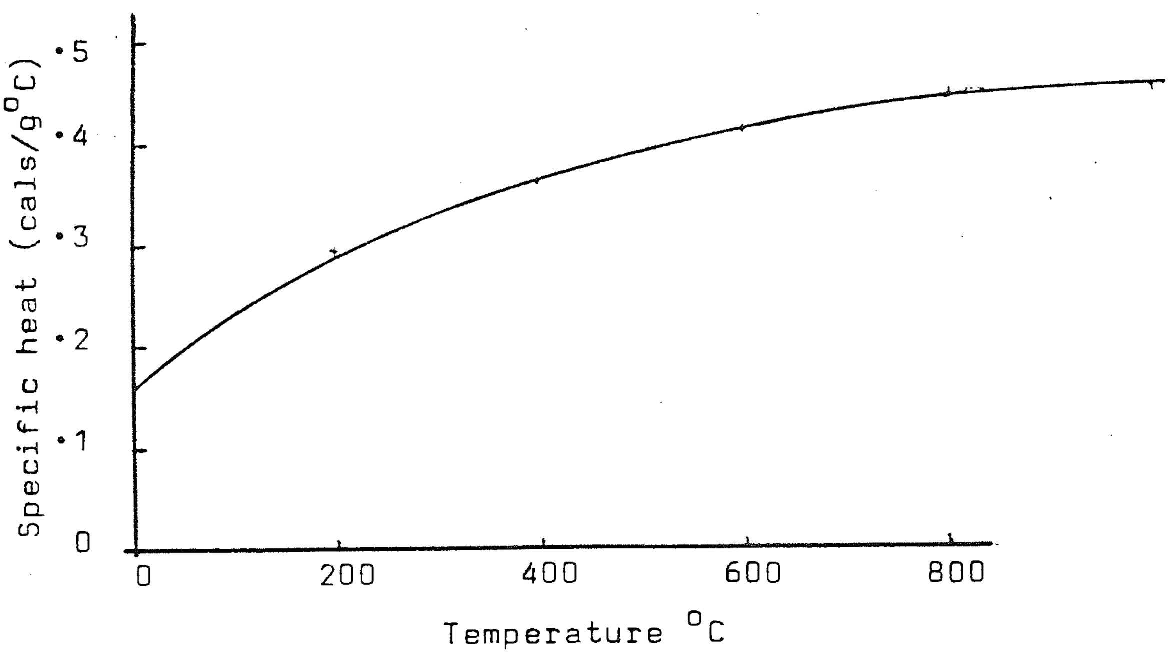
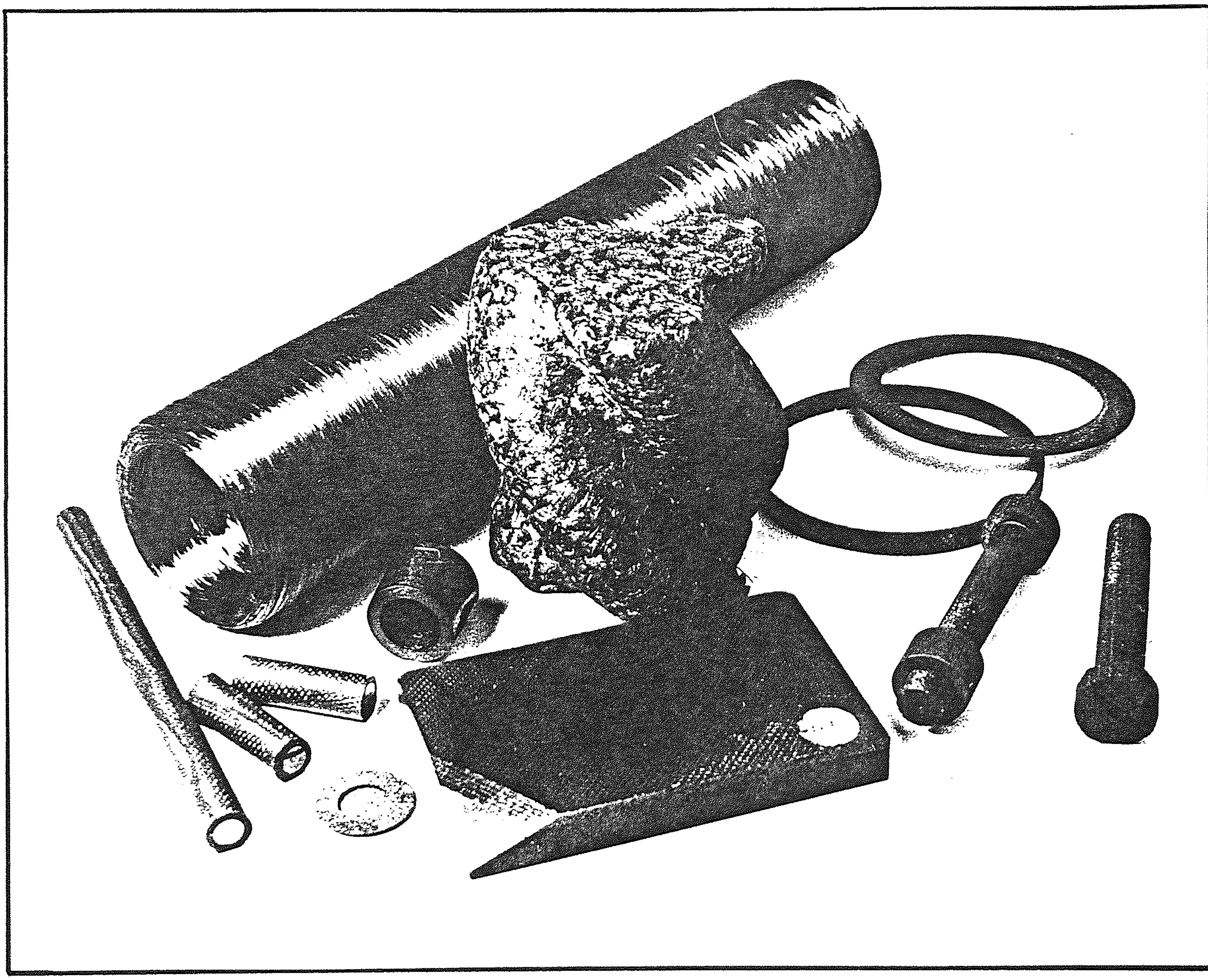


Figure A.9 Some Components Made From Carbon/Carbon, Including Rod, Tubes, Rings, Bolts, Plate, and a Moulded Air Motor Rotor



A4.3 Cost of carbon/carbon composite

The cost of a carbon/carbon product is difficult to compare with that, of say, an aluminium extrusion, which may be so many pence per metre; or with steel, which may be so many £'s per kilogramme. The cost of any carbon/carbon article is made up of several components - the raw material e.g. carbon fibre costing £30 to £40 per pound which varies depending on whether a cloth, tow or felt is chosen, and whether it is from a rayon or PAN precursor; laying-up the material to the desired shape; processing, which will include infiltration and machining between infiltrations. Other factors which will affect the cost include: the finished size of the component - a thick component will require more processing time than a thin one; the properties required from the composite - again, a dense material of 1·7 g/cc will require more processing time than one of say 1·4 g/cc.

On high-performance military aircraft the value of weight saving may justify the additional cost of carbon brakes to the order of $100- $150 per pound. However on subsonic commercial aircraft the target is lower still and the current aim is to produce carbon discs at figures less than $100 per pound. This sort of figure serves to indicate just how expensive carbon/carbon is - and the reason why only a few specialised industries can afford it in cost effective applications. See also comments in the supplement p. 425.

A5 The spin-off into other aircraft

Having invested over one million pounds in a facility to make aircraft brakes in a new, some may say exotic material, it was obviously Dunlop's aim to get the material used on other aircraft. Unfortunately, carbon/carbon is not as cost effective on present day sub-sonic civil aircraft as for super sonic aricraft - and there is only one design of a super sonic civil airliner in the western world. Such brakes as carbon/carbon do show advantage on military aircraft though, enabling a greater weapon payload and, or longer range. However, most of the latest European designed fighter aircraft have already had more-conventional brakes assigned to them. As yet none of the other military aircraft planned in Europe have settled on their final brake technology. The story in America is rather different. There, the whole of the aviation industry closed ranks on outside suppliers after Dunlop had won the contract on Concorde. And, although there have been several aircraft fitted with carbon/carbon brakes - for example McDonnell Douglas' F-15 and F-18, General Dynamic's F-16 and Northrop's YF-17 - the contracts for these have always gone to American manufacturers, either Goodyear or Bendix.

The success of the Concorde brake has not therefore, brought aircraft manufacturers rushing to fill the Dunlop order book with more carbon/carbon brakes - even though Dunlop have sold the concept hard and long. Development work continues on brakes for various civil and military aircraft projects but only development orders have so far been placed. This may not have mattered had the early predictions of the number of Concordes in service - BAC were estimating to break even on 120 aircraft - been fulfilled. As it is Dunlop have nearly completed the order for Concorde's brakes for BAC and Aerospatiale, Air France and British Airways - a total of nine aircraft. However other carbon brake manufacturers are no better placed - Goodyear having only one contract left in the States, i.e. F-16 while Bendix have just two, the F-18 and F-15 follow on aircraft.

Only one other aerospace application for carbon/carbon - other than brakes - has arisen. And that was a small, flat ring (see figure A.9) used as a pressure pad for screw jacks operating the flying surfaces on the European Airbus. Such rings can be made from the material used for the Concorde discs. But ten rings per aircraft, on the hundred aircraft that were planned was hardly going to keep the production facility going.

As can be seen, Dunlop had the potential for supplying a material ideally suited to energy absorption - either brake or clutch applications - and also for bearing applications. They will no doubt find applications on other aircraft; but experience has made them realise that reliance on one customer (or one industrial sector) does not always pay.

A6 The search for other users of carbon/carbon

Before describing how the search for carbon/carbon users has proceeded, it is as well to understand the marketing activities undertaken by Aviation Division of Dunlop.

A6.1 Aviation Division's marketing activities

Aviation Division do not have a marketing department as such. They do have a sales department which is responsible for the acquisition of orders and sale of equipment to the aircraft and military industries in general.

It is not Dunlop's aim to sell specific items to the user - their aim is to promote the idea of being in the aircraft equipment business. It is assumed that the manufacturers of airframes and aeroengines know that Dunlop are in the business of supplying equipment. An airframe/aeroengine manufacturer will therefore enquire if Dunlop is able to supply a particular part whenever necessary. Enquiries may come from the UK, Europe or the USA. As a summary of Dunlop's business in these three areas they can expect to receive enquiries:

- definitely from the UK manufacturers
- generally from the Eurcpean and American manufacturers.

The procedure for acquiring business is generally as follows:

- Dunlop will receive a design specification (or ACS - aircraft control specification) from the customer, which will give very specific details of the item they require.

- Dunlop will submit a proposal to the customer within thirty days. This may be a detailed document covering the statement of compliance (interpretation of the ACS paragraph by paragraph), description and operation of product, the design philosophy, a weight statement, reliability statement, relevant drawings and alternatives or variations.

- If Dunlop's technical and commercial proposals prove to be competitive and subsequent negotiations on contract terms and conditions are considered by both parties to be satisfactory, then an order will result.

This method of tendering for business is general to the aerospace and military industries, who specify their needs and let the equipment manufacturer's compete for the contract. Such a system works quite well in this particular environment but it is only to be expected that a company orientated to supplying the needs of the aviation industry would have problems introducing a new material. The senior management of the company probably recognised this for it would explain why the sales department has never been asked to sell carbon/carbon.

This illustrates the problem that a company oriented to a specific market might have, in trying a new venture in an unknown market. They have fixed ideas of working within their known sector but no experience for tackling new areas. In Aviation Division's case this is not quite true because a new foamed metal 'Retimet' had been launched by the company some years ago. The development, production and marketing of this material was put in the hands of one man in the laboratory. It was a case of developing a material with no end product in mind, and indeed it was introduced on the BBC's Tomorrow's World as a material looking for a problem to solve. Even the response to such publicity was under-estimated and a lack of follow-up material added to the problem. Insufficient analysis of Retimet's properties together with a lack of information about the material at the right time illustrate just two of the problems that Dunlop had whilst trying to introduce this new material. As a result, this product has never been regarded as a success even by the company. It is probably fair to say that they did not want to go through the same process with carbon/carbon and have therefore tackled the problem in an entirely different way.

A6.2 Publicity given to carbon/carbon by Dunlop

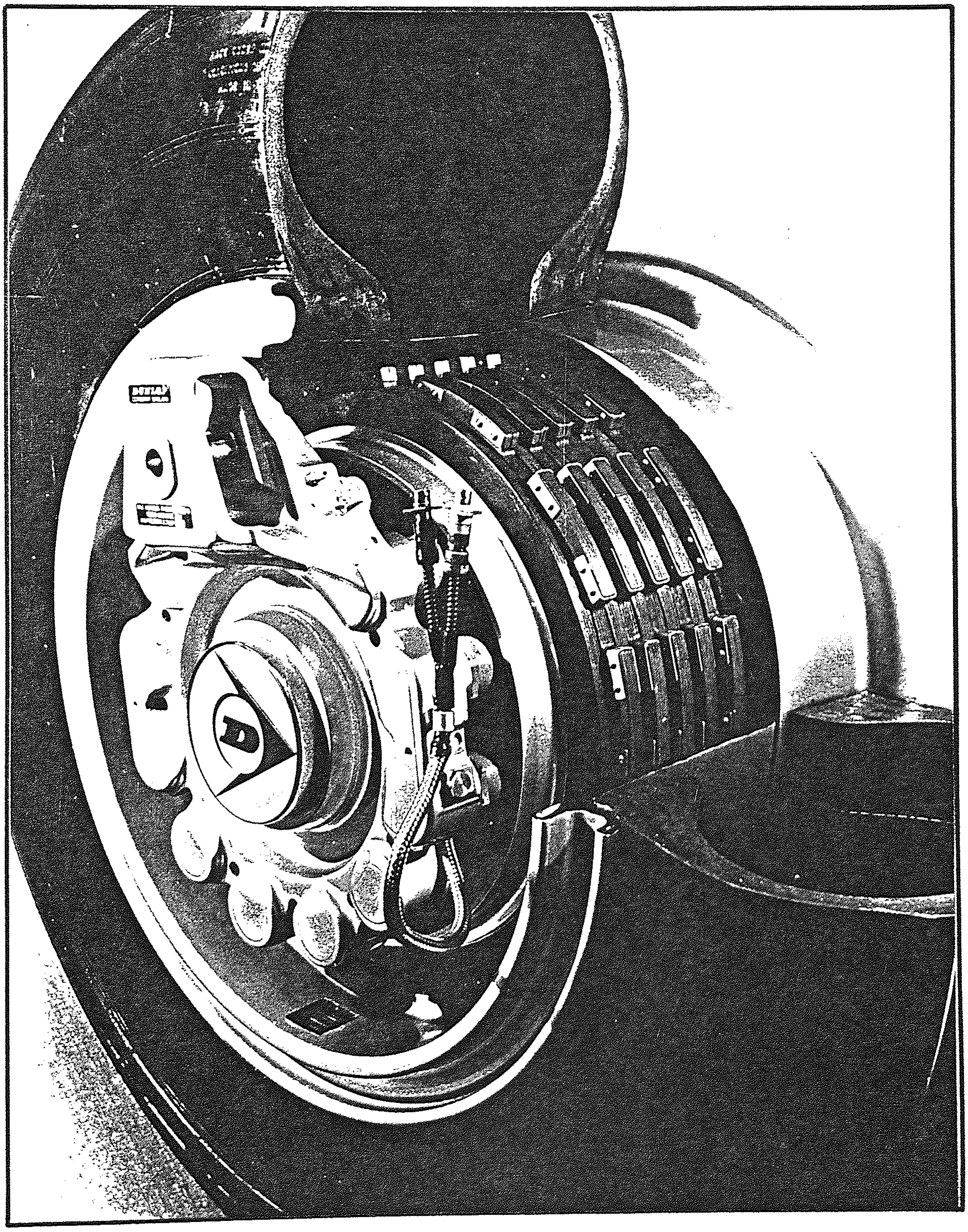
The publicity given to carbon/carbon by Dunlop has been minimal. Following the symposium given to the various aviation authorities (see page 384) on 10th December, 1971 a press conference was given. And some while later Mr. John Dent (Director Engineering Group) suggested that Aviation Division might compete in The Royal Aeronautical Society's annual competion for the N. E. Rowe Medal. Of the three papers given to the Coventry Branch of the Society, John Weaver's "Advanced Materials for Aircraft Brakes" went on to win the 1972 N. E. Rowe Medal for the 21-25 years age group (6). Another paper, "Design development of lightweight wheel braking equipment" (7) was given by Stimson and Dowell in 1973 at the Conference for the Society of Allied Weight Engineers. In 1974 a paper (8) was given by Fisher and Weaver at the carbon conference in London. These have been the only technical papers published by Dunlop on carbon/carbon.

Other announcements have occasionally occurred, for instance The Financial Times (9) carried a short column when the CVD furnace was installed during the summer of 1974. Full page advertisements (selling Dunlop's brake technology in carbon/carbon) have appeared in Aviation Week and Space Technology, but there are no records of the response. Formal lectures have been given to various societies - for instance the Rubber and Plastics Institute, Birmingham Branch meeting in the autumn of 1977 - giving a brief history of Dunlop's involvement with carbon/carbon and their associated brake technology. Similar lectures have been presented at foreign trade shows; for example a tour of India in early 1978 was aimed at promoting Aviation Divisions technology. Until recently (see page 411 and references 12, 13, 14, 15 and 16) no journalistic publicity has been given to carbon/carbon by Dunlop, apart from a public exhibition run at the Design Centre, London during the summer of 1977 (17). This was essentially to promote an overall image of Dunlop rather than to push the carbon/carbon technology in particular. Other exhibitions pushing the carbon/carbon brakes have been the biennial SBAC exhibition, the Paris Air Show, the Hanovar Show and more recently a similar demonstration in Japan. Mock-ups of the Concorde brake have been shown (figure A.10) at some of these exhibitions.

A6.3 A market survey by ERA

Following Dunlop's decision to build their own CVD facility and make carbon/carbon on site, they began to think of other possible applications that the material could be used for.

Figure A.10 Section Through the Concorde Main Tyre and Wheel Showing Multi Disc Structural Carbon Plate Made From Carbon/Carbon Composite



A market research study was obviously called for, but who should do it? Research Centre felt they could handle such a task, but the people at Coventry favoured an outside organisation, equipped with the necessary skills in market research. Dunlop had previously seen one of ERA's (Electrical Research Association) long range planning reports "The commercial exploitation of carbon fibres to the 1980's". Such background information led Stimson to ring Dr. B. C. Lindley at ERA early in 1973.

Apart from ERA's long range planning report on carbon fibre they had also investigated insulating materials for the electrical industry. Otherwise their experience lay with electrical and electronic projects. They had not long been in the business of performing marketing studies - only since 1968 had they had a Technological Planning Unit (now Planning and Market Development).

As Dunlop had no experience in the electrical components field, but felt that a structural carbon might be of importance in that area, they followed up the ERA contact. An exploratory meeting was held at ERA, and an interim report was submitted by ERA to Dunlop in July 1973. This laid out their understanding of the problem, details of carbon/carbon's manufacture, its properties, and the method they proposed to follow to search for new market areas for the material. Most of the contacts they made were from reference to their own library of contacts built up over past years. Where they had no knowledge of a contact in a particular industry; telephone calls to various establishments were made to identify the 'right' person. In conducting such a search they were aware of the physical properties of the material, as indicated on pages 395, 396 and 397, and were seeking applications where cost might be regarded as secondary to achieving functional objectives. Using product/market matrices and relevance tree techniques they identified twenty seven possible areas for carbon/carbon usage. Of these only five were recommended to Dunlop as being worthy of any follow-up in the final report of January 1974 (10). These were, in order of potential:

Medical implants

Engine seals

Diecasting dies

Bearings

Batteries

The report was regarded by Dunlop quite favourably, although Roger Bull of ERA felt Dunlop "had used the study as insurance against the brake programme failing" (11). Some of these applications were followed up and development work on a few started. But none have yet come to fruition as we shall see later.

A6.4 The Total Technology approach

With seemingly no new applications being developed for carbon/carbon, Dunlop's head office - or rather their personnel department - played a part in the carbon/carbon history. Mr. Rupert Brooks, Career Development Officer, was looking for new graduates to join the company. He and the author met through Cambridge University, where the author was enrolled for a Production Management course. Brooks, knowing the authors background in aviation, with experience on CFRP structures, proposed a project working with structural carbon whilst undertaking a Total Technology PhD at Aston University. This was set up during the summer of 1975 with Bayly and the Chief Engineer of Aviation Division - Mr. Stan Beasley. The author was placed under the Chief Metallurgist - who unfortunately had not been involved in the preceeding negotiations. The project - "to find new markets for carbon/carbon outside the aerospace and military sectors" - did not get off to a good start because of this. However, once the author had been accepted and his position and responsibilities clarified (which took about six months) the work proceeded satisfactorily - as testified by the thesis "Study of new product market search and user adoption". The project does not claim to have found new uses for carbon/carbon in the three years that it has been running - but rather to indicate the ways in which a material producer should tackle the problem of finding uses for a new material. The project discussed:-

- the factors affecting the innovation process, related to material developments, both internal and external to the producing organsiation.

- the search procedures available to a material manufacturer.

- who the "material decision maker" is, in various industrial sectors, what his function is, and how important he rates material innovation.

- which information sources/channels should be used to reach the "material decision maker".

- the time period that can be expected for material adoption in different industrial sectors.

The results of this project led to publication of several articles and advertisements, in various journals (12, 13, 14, 15, 16) which may well lead to future applications for carbon/carbon.

In carrying out such a project the author was able to study various applications that arose, and to follow their history.

A7 Some applications considered for carbon/carbon

During the first two months of 1976, the author got in touch with most of Dunlop's known contacts who had an interest in carbon/carbon development. These contacts followed either from the ERA report (10) or directly from Fisher and Weaver. The applications discussed here are generally those that have yet to come to fruition.

A7.1 Medical (implant) applications

There have mainly been two areas of work concerned with carbon/carbon developments. The first, a heart valve, stemmed from contacts that Dr. Fisher had forged with the nuclear industry - in particular the ill-fated Dragon Project in Dorset. And the second, dental implants, arose from contact with Dr. G. M. Jenkins at Swansea - who had by then finished his consultancy contract with Dunlop.

A7.1.1 Heart valve

The Drgaon Project had become involved with the Societa Ricerche Biomediche of Italy and asked to coat a heart valve with pyro carbon. This valve was later passed to Dunlop who were asked to seal the surface by processing in their CVD furnace. By the end of 1975 though, it became known to Dragon that the valve which the Italians were experimenting with was a virtual copy of the Bjork-Shiley valve - designed and patented in the United States some years ago. Dr. N. Macleod of Edinburgh University pointed this out to Dragon in December 1975. He was himself working on a new heart valve design to try and overcome some of the inherent problems with the Bjork-Shiley and Starr-Edwards* type valves. Problems such as being noisy (Starr-Edwards), with high pressure drops and generally thrombogenic (promotion of blood clots) were common. Macleod, a chemical engineer, applied his knowledge of fluid dynamics to the problem and propsed an aerofoil shaped, pivoted valve, housed in a conical ring - see figures A.11 and A.12. Mr. F. Ridealgh of Dragon suggested in January 1976 the author should contact Macleod.

Macleod responded very rapidly to approaches from Dunlop and on the 8th of March he met the author at The Institution of Mechanical Engineers where a discussion on "flow dynamics of heart valve prostheses" was being held. Over dinner that evening he and Professor David Taylor (an eminent surgeon of The Royal College of Surgeons) convinced the author that the

* the Bjork-Shiley is a restrained butterfly valve
  the Starr-Edwards is a caged poppet valve.

Figure A.11 Macleod Heart Valve

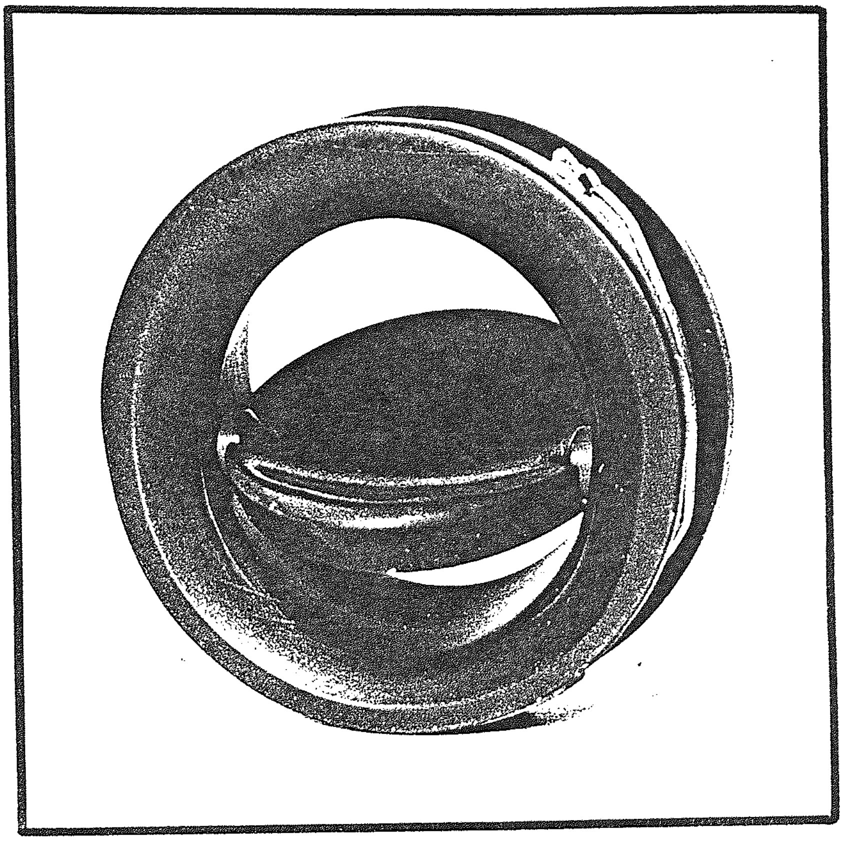
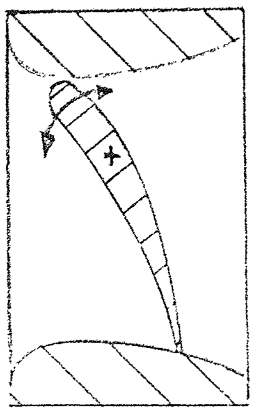


Figure A.12 Schematic of Macleod Heart Valve



Macleod valve was superior to any other type of mechanical valve. He had previously made the valve from vitreous carbon and various polymers, and was keen to try and make it from a reinforced carbon. Carbon was regarded as the ideal material because of its previously proven biocompatibility with the human body. Unfortunately, the Americans appeared to have virtually covered the bio-engineering field with patents on components made from pyrolytic carbon. Because of this, vitreous carbon was the first choice material and Macleod had at first been able to get material from Plessey's. When they stopped their process he turned to Fordath and then the Atomic Energy Authority at Springfield for materials. His patents (1,327,192; 1,327,371; 1,407,621; 1,447,871) on "improved fluid control valves" extend back to 1969.

Following this first meeting, sample material - both cloth and felt reinforced material - was supplied to Macleod. He had difficulty machining the cloth based composite, and thought both had far too coarse a grain structure for his purposes. At this stage very little effort was made by Dunlop into developing better materials for such an application. This was mainly due to (a) pressures to overcome Concorde disc production problems and (b) the uncertainty members of the Advanced Materials Laboratory had as to management policy for developing medical devices. For example, there were fears being raised over the possibility of Dunlop being involved in large insurance claims, should someone die when fitted with one of their valves. Nobody had any real answers to such fears, and no one was asked to find out ways around such problems. Nothing more happened until 6th January, 1977 when Beasley (Chief Engineer) intimated that a policy document being prepared for Engineering Group, Dunlop, included diversification plans for carbon/carbon. The medical field was in no way restricted. Indeed Beasley was keen to have the potential of heart valves more fully investigated. So on the 24th January, 1977 Norman Macleod visited Coventry and presented his valve to a group comprising N. Smith (Chief Metallurgist), J. Weaver and R. F. Fisher (Advanced Materials Lab) Mr. H. Wagstaff (Sales Engineer) and the author. Macleod estimated the market for such valves to be five thousand valves per year in the UK alone at a value of £1·5 million (18). Most of the valves are currently imported from the United States and cost between £200 and £800 each. A feasibility study with small carbon/carbon discs (1" dia x 1/8" thick) was planned as a result of the meeting. This was essentially to establish that carbon/carbon had the required mechanical properties.

Prior to this feasibility study (between March of 1976 and January 1977) Macleod continued his efforts to interest other manufacturers with his valve. He very nearly succeeded with Morganite but came up against bureaucratic red tape when Morganite asked for guarantees from the Department of Health and Social Security, who were slow to pursue the matter. Had they succeeded Dunlop would have got some work anyway because Morganite were approaching Dunlop to seal the components with pyrolytic carbon. Macleod made contact again with Dunlop during this period in the hope that they would sponsor such work. When the author could give no firm indication along these lines, he put Macleod in touch with Dr. G. M. Jenkins at Swansea in September 1976. Jenkins had earlier in the year forwarded a pre-published MRC (Medical Research Council) report (19) to the author. This clearly showed the biocompatibility of carbon/carbon with living tissue, especially when implanted percutaneously. It described bone implants and also heart valve fabrications. Even though Jenkins was able to make the occluder and ring for the Macleod valve from vitreous carbon, neither were really satisfactory according to Macleod. In any event, Jenkins had no production facilities and the enterprise fell through.

The work proposed for the feasibility study at Dunlop never really got off the ground, and association between Dunlop and Macleod gradually petered out with no real conclusions either way as to whether or not carbon/carbon would have been a useable material. Furthermore, Aviation Division had no procedure for actually looking at the benefit of such ideas by investigating the market. With such experiences behind him - nearly ten years trying to interest a UK sponsor to manufacture and market the valve - Macleod then published his work in the USA. Two firms immediately came forward. One of these, a fairly big organisation with experience in this field is considering taking on the project.

A7.1.2 Dental implants

Dr. G. M. Jenkins (Swansea University) put the author in touch with Professor A. O. Mack at Eastman Dental Hospital, who was doing work on tooth replacement. Mack was contacted in early February 1976 and a meeting in March arranged. Dr. John Hobkirk (senior lecturer in Prosthetics Dept) had for some years been investigating the potential of various ceramics and carbon products as implantable materials. As a result of the meeting between Hobkirk, Mack and the author, some small sample rods of carbon/carbon were sent to Hobkirk for investigation as tooth roots (May 1976). Some of the samples together with vitreous carbon acting as a control were inserted into the jaws of rhesus monkeys. Such implantation trials obviously take a long time, and even by April 1978 no firm results had been obtained, although histological tests were being undertaken.

The replacement of tooth roots with a biocompatible material such as carbon/carbon is still very much in the experimental stage. The hope is that the porosity of carbon/carbon will allow tissue ingrowth into the artificial root, after which the tooth root may be capped. However, Eastman Dental Hospital is a private institute. Their patients can afford titanium and vitreous carbon tooth roots which currently cost £15 - £40 each. Whilst there is the possibility of producing tooth roots by the thousand each year, it is unlikely that national health patients would be able to afford such treatments, as Dr. Wilson of the Birmingham Dental Hospital pointed out (20). Even should such developments succeed, the financial return to Dunlop would be minimal.

A7.1.3 Concluding remarks on developments with the medical world

Although the medical field is undoubtedly an area where new advanced technology materials are making an impact (titanium for instance), it is not an area where large quantities of material are consumed. The medical profession is however, prepared to pay the high prices that these materials cost, providing of course they do the job correctly. An area of concern here for all material manufacturers though is that many surgeons have their own whim as to which joint replacement, heart valve, etc. should be used. A recent course (21) at the Insitution of Mechanical Engineers showed quite clearly that many surgeons had their own design joints for the shoulder, elbow, wrist, etc. and would use no other. Consequently there are no great numbers of any one design, and for a material producer the answer is obviously to have his material used by each and every surgeon regardless of design - very difficult for carbon/carbon, where the material has to be laid up in a particular configuration, for a given product.

Although the experience of Aviation Division with the medical world is both short lived and apparently haphazard, the overall Dunlop experience is not so bleak. In 1977 the directors at Dunlop head office decided to set up a new division - Bio-activities. A major aim of this division is to provide suitable procedures for investigating the marketability and desirability of Dunlop pursuing 'biological' activities. Such a unit will hopefully identify and analyse those biological activities that are suitable for commercial exploitation. This will remove the responsibility of carrying out such investigations from the various Dunlop divisions who may lack the appropriate biological and marketing expertise in this area but have the required technology.

7.2 Seals and bearings

The ERA report (10) of January 1974 gave an indication of the potential both for seals and for bearings. Aviation Division appear to have moved quite rapidly on this, for some of their hydraulic and pneumatic control equipment could well have used carbon/carbon bearings and seals. Many of the seals in such devices are made from unreinforced carbons and are prone to operator abuse. It is not unknown for three seals to be broaken before a sound one is fitted - due to the inherent brittleness of the material.

Under the direction of Mr. Brian Bull (Senior Design Engineer), modified drawings for piston rings and bearings - to be made from carbon/carbon - appeared from the design office. A test rig was designed and made, so that the new components, could undergo operational trials. In the Advanced Materials Laboratory though, no one had experience of the necessary filament winding techniques to make carbon fibre tubular components. Nevertheless Weaver rapidly converted one of the lathes on the shop floor and began to investigate the manufacturing methods. Having tried the known methods of making CFRP tubular components - and found them unsuitable for carbon/carbon - a more suitable technique was gradually evolved. In doing so, care was taken to ensure that no patents would be infringed by this developed method. By November of 1974, the first tubes had been infiltrated in the furnace and were ready for machining. This raised the next problem, for a single point tool on a lathe automatically unravells the carefully wound carbon fibre tows. Even though Weaver had foreseen such problems and suggested a wet grinding technique should be used, it took the machine shop several ruined tubes to relaise the same. Wet grinding, using cylindrical or centreless grinders gave satisfactory results.

The initial success gained with such filament wound rings looked promising - hoop strengths of 390 MN/m^2 (57 x 10^3 psi) with a Youngs Modulus of 139 GN/m^2 (20 x 10^6 psi) were common place. An inherent advantage of such rings is revealed when failure of the ring occurs. The fibres running around the outer circumference break when the ultimate load is reached - but the inner fibres remain intact and the ring could presumably continue to function, though less efficiently until replacement. This might well be advantageous for piston ring applications where a carbon or steel ring would fail completely giving total pressure loss and in the case of a steel ring, the fragments could ruin the bore of the cylinder.

Such good results became more difficult to achieve as more components were made - and this variability in the properties of the product has slowed down many a development project (see next section). Apart from the initial test work carried out to investigate manufacturing methods and the physical properties of such filament wound tubes, no further development work ever took place. The test rigs for carbon/carbon seals and bearings have never been used for that purpose. Hardly any resources - less than two hundred man hours, and only a few hundred pounds for raw materials, lay-up, processing and machining - have been devoted to the project. Even though Dunlop have never felt able to proceed any further with seals for their own products; other potential customers do not share that view. In particular, work for the Ministry of Defence is continuing in this area, though on a different product. The advantage of such a reinforced carbon over conventional grades of carbon or such devices as seals and bearings is obvious - greater strength and therefore less prone to operator abuse. Presumably other companies recognise this more than Dunlop, for several wish to investigate the material's potential. The publicity during early 1978 (12, 13, 14, 15, 16) has brought forward such enquirers. Another, local enquirer for reinforced carbon seals was made in April of 1978 when one of the shop floor machinists was chatting to some friends in his local pub. This Rugby based concern, is now investigating carbon/carbon for a sealing application. Presumably, if a successful product for an outside customer is completed, Dunlop will again review the situation for their own products!

A7.3 Batteries

Again, this was a product area suggested by ERA. The batteries in question are the sodium/sulphur battery and zinc/air battery. A great deal of development work on batteries is going on at present (22), particularly with the sodium/sulphur type, where the aim is to produce a battery with four times the energy density of a conventional lead-acid battery hopefully for use as and when the 'energy' shortage becomes more apparent. Research with the sodium/sulphur battery has been going on for ten years now and Dunlop's association with it started in 1974. It came about partly as a result of the ERA report and partly because of Dunlop's contacts with British Rail for braking developments (see page 424). British Rail have a staked interest in sodium/sulphur battery developments and they put Stimson in touch with the firm that was researching the battery.

The application in question did not acutally use carbon/carbon but rather made use of Dunlop's facility for sealing carbon using the CVD process. Many materials have been investigated for use as the electrolyte tubes*, and Dunlop's carbon is just one of them. As with the bearing/seal developments the early results with the carbon tubes looked very promising. The tubes housed the molten sodium and allowed the sodium ions to pass through the wall to react with the sulphur anions. All seemed well with the first few made, and with forecasts of one million tubes being required in ten years time Dunlop looked set to play an important role in this new battery's development. Unfortunately, the quality of the product varied enormously as the numbers being made were stepped up from the odd one or two to hundred's.

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* For an understanding of the operation of the sodium/sulphur battery see reference 22.

This variability in quality - which has occurred with other products - must be explained by the processing technique. It has been the hope of the Dunlop engineers that small items - such as these tubes, or bearings, seals, etc. - could be fitted in or around the brake disc stacks.

Whilst the processing conditions are ideal for the brakes (though even this is still being investigated - reference 5) the conditions are almost certainly not ideal for many smaller items stacked over a height of several metres. It has not been possible to develop more suitable manufacturing facilities for these smaller items due again to a lack of resources - manpower and money.

Stimson had been hoping that the original cost estimates of 50p per tube could have been reduced to 15p by: loading the furnace more efficiently, reducing the processing time and, ultimately by designing a furnace to suit high quantity production of this product. Unfortunately none of these have been put to the test due to the battery manufacturer departing from the carbon tube because of unpredictable variation in the end product and lack of time to develop the materials and processes.

A7.4 Supplement

A supplement has been included to try and tie up a few loose ends and give some further information that could not be included under the previous headings.

The development, or otherwise of the applications mentioned above have tended to be in the unsuccessful areas. This is due mainly to the simple fact that there has been no other product success to rival the Concorde brake programme. To complete the list of applications that ERA suggested, a little will be mentioned about diecasting dies. The refractory alloy steels used for dies have low heat conductivity and are subject to 'heat checking' after a few thousand shots, with the result that there is a network of tiny fins on the surface of the casting. With carbon/carbon's superior thermal conductivity and resistance to thermal shock, it was hoped that the material might be investigated for use as dies. GKN - one of the main diecasters in the UK - were prepared to investigate new or improved materials for such uses. However, when Stimson and Smith followed it up with them, they found another firm was well established in developing an unreinforced carbon die. And it appeared that Dunlop's costs would be at least ten times as much as this other firms. As a result the idea was taken no further.

So far, this history has not mentioned Dunlop's efforts to capitalise on their expertise with energy absorption devices - brakes. That is, apart from their efforts to interest other aircraft manufacturers with carbon/carbon brakes. There are other areas where vehicles with high kinetic energies have to be stopped - railway vehicles for instance. Dunlop was not slow in recognising this, and British Rail could see advantages for vehicles such as their High Speed Train. Although much design and development - including prototype manufacture and testing - has been done over the past four or five years, a viable product has yet to be achieved.

An indication of Dunlop's pricing technique for developing these new products should also be given. It appears to be of a very simplistic nature, in as much that all too often a product will be priced on a direct comparison with a Concorde brake disc. For example, if a tube is to be made, a quick calculation will show how much furnace area that will take up in place of a brake disc. The cost of the tube will be pro-rata to the brake disc, regardless of whether the processing conditions should be the same or not. The overheads associated with making aircraft brakes to exceptionally high standards are far in excess to those that say a product for a general engineering firm might involve. This may indeed be an over simplified view but no one has seen fit to make the author - or many in the Advanced Materials Lab - any the wiser. If this is the case, it is not surprising that many a promising product has fallen by the wayside.

An area of development which has taken place that was not suggested with any great enthusiasm by ERA is in the nuclear field. The contacts made here initially by Weaver followed a Carbon Conference in 1972. Mr. F. Ridealgh later rang John Weaver about the possibility of investigating carbon/carbon at the High Temperature Reactor (HTR) - Dragon - in Dorset. The initial suggestion was for carbon/carbon to be used as a high temperature insulating material*. Gas

* There is considerable variation in the thermal conductivity characteristics of a composite made from cloth. There can be greater than a 20 fold difference between parallel to ply directions - the factor can be reduced to a factor of 4 to 1 or lower if necessary; see also page 398.

ducts - carrying helium - of one metre diameter were talked of, stretching over lengths of 3 km. The only work ever done on this was to prove the feasibility of wrapping a carbon cloth into a tube about 24 cm dia and infiltrating this. There would obviously have been problems when bends and junctions were encountered, but the idea never reached that stage - Stan Beasley (Chief Engineer) was not prepared to back it.

Contact continued however with Dragon - remember it was they who instigated the heart valve - and some sample pieces of carbon/carbon were planned for irradiation studies. Unfortunately, before these could be placed in the reactor core, the Dragon Project was run down by March of 1976, when the British Government pulled out of the programme. The team that had been responsible for the high temperature materials programme was salvaged to some extent and regrouped under Dr. Graham at Flight Refuelling's establishment at Wimbourne, Dorset. They were being financed as an international organisation by American and German concerns. But no further work has been done by them with Dunlop's carbon/carbon - and they intimated (23) in May of 1976 that other establishments, such as at Harwell and Risley had also ceased the carbon fibre programmes. Such a closure was unfortunate for Dunlop, for there were long term hopes (by the Dragon people) for developing seals, bearings, and even turbine blades and discs with carbon/carbon. No real contact has been had with any nuclear establishments - for developing carbon/carbon products - between early 1976 and April 1978. However, following recent publications (12), the authors' father - a member of the Nuclear Inspectorate - passed a copy of the article in The Engineer around his colleagues. As a result, it was suggested that further copies should be sent to the Secretary of State for Energy (Mr. A. Wedgewood Benn), Head of the CEGB (Mr, E. Pugh), Head of the South of Scotland Electricity Board (Mr. D. R. Berridge) and to the Chairman of the British Nuclear Engineering Society (Mr. P. M. Wolff). The letter to Benn has led to talks being opened with the Materials Development Division at AERE Harwell. So developments in the nuclear field may once again get underway for carbon/carbon.

This history can not be concluded until mention is made of the great efforts that Dunlop have made to ensure the success of the Concorde brake programme. Many production problems have arisen over the years and these have all been put right as rapidly as possible. It is to Dunlop's credit that they have a fine aircraft brake for Concorde. But it may be a result of this tremendous effort that has been expended on Concorde, that other, smaller projects have not succeeded. However, to compete with other brake manufacturers like Goodyear, Dunlop have had to work hard to ensure the manufacturing process is giving products of the best quality and reliability possible. The tremendous effort exerted to develop one successful brake has to a large extent meant that other projects have suffered in their development. Whilst it is commonly recognised that getting one product to commercial fruition is good product management rather than developing several projects at the same time - it is not so commonly recognised that concentration on one project can stifle the creativity of the project team. Something like this happened in the Advanced Materials Laboratory when hardly any time could be spared to develop material for products other than Concorde. For some of the team this created a longing to move on to something new. Weaver, for example has moved to a different job within the company, whilst Smith has left the Company altogether, though not necessarily for the same reasons. The loss of such experienced men may only cause a hiccup in the development of carbon/carbon. On the other hand, if no one of similar calibre can be found to solve the material processing problems, it could mean Dunlop lose their market leadership with carbon/carbon.

Finally, it should be pointed out that the structural carbon brake programme which started out with such a flourish in 1969/1970 has lost much of its initial momentum. By May of 1978 Dunlop will have completed the supply contract for Concorde brakes - not only as original equipment for British Aerospace (formerly BAC) and Aerospatiale, but also the spares (replacement brake discs) for British Airways and Air France for the foreseeable future. A project which started out with over a hundred aircraft being planned, has, unhappily produced only sixteen Concordes - seven of which are unsold. The fate of Dunlop's carbon/carbon still runs in hand with the Aviation industry.

A8 Discussion and Conclusion

This history of Dunlop's experience with carbon/carbon might on the surface be regarded as just one innovation. In fact there are three separate innovations rolling together into one. If one was to judge the overall innovation to-date, it might be accepted by all, that success or otherwise has yet to be proved. But then carbon/carbon is still a relative baby in terms of its life, and is really only just at the start of the innovation path. Materials like copper and tin might be regarded as nearing the end of the innovation path - though they are not on their deathbed by any means. To judge the success or otherwise of Dunlop's efforts with carbon/carbon it is better to look at the three innovations that have occurred in more detail. First there is the overiding product innovation with the carbon/carbon brake. Second there is the process innovation that has taken place in developing the facility for making carbon/carbon. And third there is a cluster of product innovations associated with the development of other uses for carbon/carbon. These will be discussed in turn.

Aviation Division's business has, since its conception, been aircraft and their ancillary equipment. Wheels and brakes have played a very important part in this business, and small product improvements take place regularly. However, the dawning of not only SST aircraft but jumbo jets as well, brought about the realisation that lighter, more efficient materials would be needed for these future 'planes. This meant the introduction of materials with better strength to weight ratios into the airframes and engines. It was hardly likely that steel brakes would be ignored in the quest for lighter, high performance aircraft. Fortunately, Bayly (Director of Aviation Division) recognised this fairly early on, and, he it was who started and directed the search for this lighter, more efficient brake material. And a thorough search it was too. As can be seen, a considerable effort went into the beryllium brake development - to the extent that it was fully type tested for Concorde. To reach that stage and then start another investigation into another, unheard of material is quite remarkable. The doubts over beryllium's safety because of its toxicity must have far outweighed the efficiency of the brake. The investigation into carbon/carbon itself was very thorough, looking as it did at both methods of manufacture - the resin route and CVD route - getting samples from American manufacturers and testing them for their suitability in brakes. One person was put in charge of this search - Mr. Ian Stimson. Through his efforts, the most efficient brake material available was found for Dunlop. And he did that, not by sitting behind his desk, but by going out into the world to find out whether new technologies were being developed, how and by who. He might have remained at home and got the Dunlop Research Centre to investigate the possibilities but instead he looked outside to see what the rest of the world had to offer. As it was Stimson who sifted all the information about new materials coming into and out of Dunlop - so it was that Bayly backed his efforts. Probably not because of his faith in carbon/carbon but rather in his conviction that Concorde was going to be a success and Dunlop was going to be a part of it. This alone meant Dunlop must have the best brake available to win the contract for Concorde.

In developing new brakes, such as beryllium or carbon/carbon there is nothing new in the braking concept. It is still rotors and stators (both acting as friction surfaces) rubbing against one another, converting kinetic energy into thermal energy - just like a more conventional sintered steel brake. Even with the sintered steel brakes, developments take place - the friction and heat dissipation characteristics are improved by adding new additives to the mix before sintering. Though important, such incremental innovations rarely bring home the big rewards. The philosophy behind the change to a superior material is hardly radical - the same braking principle is being used - but there are some who would surely say that the change to an unknown, untried, advanced technology material is indeed radical. It was for Dunlop - the stakes were high, and the rewards hopefully would be just as great.

In developing a new advanced brake - beryllium or carbon/carbon - Dunlop were careful to ensure the product was what the customer wanted. This is easy to say because all equipment manufacturers are handed an ACS but it does go further than just meeting the specification. It meant meeting the customer (BAC/Aerospatiale), showing them the technology, demonstrating the product - both on simulators and aircraft - convincing them you have not only the know-how but also the organisation with the capability of meeting all your claims. In short, you must be able to demonstrate the whole wheel, tyre and brake technology if you are to succeed. Dunlop did; and they eventually won the Concorde contract. On that score they have had a successful product innovation - or have they? It could be argued that Dunlop misread the market as badly as the Concorde manufacturers. If there had been a hundred SST's Dunlop would have been very wealthy. But supplies for sixteen aircraft falls far short of the hoped for success. However, if they win contracts for some large military or civil projects in the near future, all could change again. Maybe it is too early to judge the overall success of the carbon/carbon brake. As a technical achievement it is a success - but as a business venture? - the answer to that is not so clear cut.

Moving on to the next aspect of this history, let us consider the process innovation that took place. Super Temp must take the greatest praise for developing the basic CVD technology for commercial carbon/carbon projects. But Dunlop too have played their part in developing and improving the process to give a better, more consistent material for brake discs. Improved lay-up and jigging fixtures, refining the processing variables, improving machining techniques and developing anti-oxidation treatments are all areas that Dunlop has helped in for getting better structural carbons. Smith, Fisher and Weaver, as well as the Research Centre staff, have all played important parts in not only getting the process working, but getting satisfactory material out of the works door at the end of the day. The implementation of the CVD process was a successful process innovation - led by three technologically minded men, all metallurgists. Weaver stands out most, as the one who pioneered new ways to improve the process, and end material, with his detailed knowledge of fibre reinforced composites.

This new, advanced technology was accepted within the company quite readily, and does show just how quickly new technologies can be adopted. Dunlop did not even know of carbon/carbon before 1969 - but by 1973 they had committed themselves to a new technology - and by 1975, were successfully making brake discs themselves. It took them just five years to learn of carbon/carbon, educate themselves in its peculiarities of manufacture and finally complete successful composite items. In doing so the development team contrasts sharply with other manufacturers - Dunlop have never had more than a dozen working on the material, a comparatively small team. That Dunlop should have won the contract for Concorde brakes from Goodyear surely speaks volumes for their material.

If we move onto the final area of the innovation - new products for carbon/carbon outside the aerospace industry, we see Dunlop have not been so successful. This must, in all honesty, stem from their lack of experience in marketing. The whole of Aviation Division is geared to selling products to the airframe/aeroengine industries. To fulfil this function, there is a sales department with representatives that travel the world. Such a set up, keeps them in touch with all their customers' developments, but does not help in finding new markets. That is not to say there are not 'sales drives' for there are, and quite regularly, but the company has never felt the need to look at the whole marketing concept for the aircraft industry. Had there been a department responsible for marketing, not selling, there may have been a chance they could have helped with developing outlets for carbon/carbon, other than brakes. The system, as it is, works satisfactorily for the aerospace industry, though one might be tempted to ask "for how mcuh longer?" Perhaps as a result of the lack of marketing expertise, Aviation Division turned to an outside organisation more able to investigate the potential for carbon/carbon. Outside help is never to be sneered at if that helper is more expert than yourself, be it for technical, financial or marketing matters. ERA provided the necessary marketing research and presented Dunlop with some challenging areas to enter. If as has been suggested, it was done purely as an insurance exercise against the carbon brake programme failing, one can understand why none of the suggested product areas have succeeded. There are other reasons of course, but a major factor must surely be that the management of Aviation Division believes - quite rightly - that it is in the business of supplying aircraft equipment. This did tend to leave one with the feeling that management believed all carbon/carbon products should be disc shaped with a hole in the middle: "if it's not a brake, we don't want to know", could sum up the attitude. Such an attitude did unfortunately prevail, even though it was stated policy that carbon/carbon would be diversified into any viable product. It was publicly stated that any application would be considered, but in reality non aviation type products found it very difficult to get off the ground. The ideas from the medical field were a good example of this.

Another problem area, that has not been made clear in the actual history was one of communication. The problem was in two areas - one of communication between departments at the same level - and the other was communication down the line of command. The first type can be illustrated by the communication barrier that existed between the design people and the people from the laboratory. The laboratory tested carbon/carbon to investigate its properties, and the design people rejected the figures quoted for say strength - and would not design products according to the laboratory's strength figures. Such a problem was understandable when one realised that the design specification had to be altered (lowered) because too many products were going through only on concession. Instead of resolving such matters a personality clash between the various people evolved prohibiting further discussion - and both departments worked on their own data. The other communication problem arose because of Aviation Division's horizontal type of management structure. One representative would inform the lower rank and another representative would inform the next rank and so on down to the bottom. By-passing this lot from the bottom to the top could be very tortuous. However, it did help to explain why such products as the heart valve only got the go ahead for a feasibility study nearly twelve months after the initial contact was made.

The search for new products for a new material is not an easy task. Ideally the material manufacturer wants to develop products where there is a need. He first has to push out information about the material, before anyone can come back saying "my product needs your sort of material, can we try it?" This was one of the main reasons for giving publicity to carbon/carbon during early 1978. It is doubtful whether any new products will accrue to Dunlop immediately. It is more likely the information will be stored away (hopefully not forgotten) until a problem needs solving.

This has been the hope, that customers will come to Dunlop with the aim of carbon/carbon providing a possible solution to their material selection problem. Far better, it should be that way round, rather than Dunlop pushing their ideas out into the world with no real need to fulfil. To some extent this has already happened, with some customers investigating carbon/carbon for a particular product idea. And this raises the next dilemma for Dunlop - should they be raw material producers or finished product manufacturers. Management's view is that they would rather make a complete product than just supply the material. But here-in lies the dichotomy. Aviation Division has the expertise for handling products in the aviation and military sectors of industry but not for other areas such as the medical or nuclear fields. As a result the company is prepared to look at any product idea and try to make it unaware of the engineering and distribution problems. All this points to a lack of project management: all product ideas are considered - not by a project management team - but by the Advanced Materials Laboratory. And these people are metallurgists, not engineers. They have the knowledge for understanding the material problems and associated environmental problems but not the detailed understanding of engineering problems. It is suggested that a better approach to a new product selection would be achieved if there were an interdisciplinary team, possibly in the form of a new venture group to handle such things. Coupled with the lack of a project management team is the lack of resources (both manpower and money) to do justice to even feasibility studies. Too often it is a question of supplying material to a potential customer 'under the counter'. This is alright when the product ideas has only just arisen (that is, a customer telephones in with a possible application for the material). By all means send a block of material for an initial appraisal by the customer, but as the product idea progresses to a feasibility study, it must be put on a more formal footing. This has not really happened at Dunlop, not with every product idea anyway, e.g. seals, heart valve, etc. Essentially this means the company is bad at exploiting product ideas, once they have been generated. And this in turn could be construed that the company suffers from the NIH (not invented here) factor, or more aptly, not interested. As far as aviation products are concerned this is not true - the carbon/carbon brake and CVD facility would never have come about otherwise. But as far as other products go, there is a tendency to be not interested.

The final nail in the coffin for some of the product ideas is the variability in the product performance. Even the brake discs suffered some variability, but so important were they that sufficient effort was put in to minimise the problem, if not remove it altogether. This has not happened for products such as the battery tubes and seals, and consequently they have never reached fruition. It could be argued that they would suffer an inherent variability no matter how much time and effort was expended to put it right. But that is just the point, not enough effort has been put in to find out one way or the other.

To conclude then, the introduction of the CVD facility has been an unqualified success as a process innovation; the technical achievement with carbon/carbon brakes is a success in its own right and may even become a successful product innovation given time for winning further orders but the various product innovations attempted outside the aviation industry have to date been a failure. The search for new product ideas may reverse this failure if the communication problems, lack of marketing and project management experience, NIH problems and product variability problems can be overcome.

A9 Acknowledgements

The author would like to thank the many people at Dunlop who have given their time freely to help build up a picture of carbon/carbon's development.

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APPENDIX B

Polyethylene Case History

B(ii) Information sources for polyethylene

The problems involved in finding the history of polyethylene stem from the fact that its history started in the 1930's. Much of the information gained was therefore taken from refe-rences as many of the individuals concerned with the development had either retired or died. However, case histories on polyethylene - dealing with the technical aspects of the innovation - have been previously written: by Allen and Jewkes for example. Various research associations and journals concerned with polymers were contacted in the hope of pinpointing the person(s) who were closely involved with the materials development.

To discover the organisations involved with the early developments, various research associations and journals concerned with polymers were contacted, see tables B(i) and B(ii). Most of the contacts suggested ICI and the British Plastics Federation. ICI proved the most helpful, whilst at British Plastics Federation no one appeared able to help when contact was eventually established. Whilst at ICI, previously unpublished company records were made available, which have been a great help. In the course of building up background information for the case, several 'red herring' references were suggested, for example:

Raine, H.C. (1961) 'Technical Developments in Olefine Polymers'. Plastics Progress.

Bausback, G.H. (1961) 'High Density Polythene in Europe', Plastics Progress.

Woodcock, W.A. (1961) 'Worldwide Commercial Aspects of low Density Polythene', Plastics Progress.

As the story behind polyethylene evolved, the part the early users played became more obvious. It has proved nearly impossible to find out detailed information about Metropolitan Vickers or TC & M Ltd. even though their parent

Table B(i) MATERIAL SOURCE INDICATOR

Material: Polyethylene

PEOPLE RECOMMENDED \ PEOPLE CONTACTED	RAPRA (Mr.Painter)	PERA (Mr.Goldsmith)	Fulmer Res Inst.	H.J. Stern Lab	Brit. Polymer Journal	European Plastics News	European Polymer Journal	Rubber & Plastics Inst.	BXL (Grangemouth)	Brit. Plastics Fed.	Rubber & Plastics Industries Training	Birmingham University	ICI Publicity
RAPRA													
PERA													
Fulmer Res. Inst.													
H.J. Stern Lab													
Brit. Polymer Journal													
European Plastics News													
European Polymer Journal													
Rubber & Plastics Inst.		x											
BXL (Grangemouth)		x											
Brit. Plastics Fed.		x		x				x					
Rubber & Plastics Industries Training			x					x					
Birmingham University Prof. Haward					x								
ICI Publicity			x			x						x	
ICI Secretary													x
Shell			x										
Phillips Petroleum			x										
BP			x										

TABLE B(ii) : INFORMATION SOURCES FOR POLYETHYLENE

Contact	Comments
R.A.P.R.A. (Mr. Painter)	Offered the services of their library but did not recommend any industrial organisations to talk to.
F.E.R.A. (Mr. Goldsmith)	Recommended several references: 'Plastics in the service of Man' by Cousens and Yardley. : 'Rigid X, High Density Polythene' BP Chemical Technical Manual 22. : Transactions of Institute of Rubber Industry, 1960 by M. Jones. Also suggested contacting: Rubber and Plastics Institute : British Plastics Federation
Rubber and Plastics Institute (Mr. Walford-Coleman)	Recommended contacting: Rubber and Plastics Industries Training Board : British Plastics Federation
Fulmer Research Institute (Mr. Turner)	Suggested contacting: Phillips Petroleum or BP for high density polythene : Shell for the Ziegler process : ICI Plastics Div. - publicity people Recommended reference: 'First 100 years of Plastics' by M. Kaufman.
British Polymer Journal (Mr. Leonard)	Suggested contacting Professor Haward at Birmingham University who was involved with the Ziegler process in the early days.
European Plastics News (Mr. Telbott)	Suggested ICI.
Professor Hayward Birmingham University	Recommended reference: 'Discovery of Polythene' by R.O. Gibson Royal Institute of Chemistry Proceedings Suggested it might be possible to trace Gibson through ICI.
ICI (Mr. White, Marketing Services)	Recommended two references: 'Polythene' by Renfrew and Morgan, published by Iliffe. : 'ICI - A History' by W. J. Reader. Also suggested that the company secretary for Plastics Division might be able to help with early history.
ICI (Mr. Dickinston, Div. Secretary.)	Mr. Dickinson suggested that the licensing manager might help. Mr. Lowe (Licensing Manager) found some historical reports in the archives and made them freely available. He also tried (unsuccessfully) to trace some of the persons involved with the early developments.
BICC Research and Engineering Ltd. (Mr. Slaughter)	As the history progressed it became apparent that further information was needed about the users of polyethylene. Eventually tracked down to Mr. Slaughter, having contacted BICC Head Office and Telecon Plastics Ltd. He provided further background information on TC & M Ltd.
GEC Switchgear (Mr. Dawson)	After contacting Marconi Radar Systems and AEI Cables, eventually established contact with Mr. Dawson for information on Metropolitan Vickers.

organisations were identified - GEC Switchgear and BICC Research and Engineering respectively. Identifying the best person to talk to in these organisations proved to be very difficult.

B.1 Introduction

Case histories of the invention of polyethylene as well as the innovation are fairly well documented (1, 2, 3) but none deal with the search for new uses in any great depth. Much has also been written by ICI about polyethylene (4-12) and maybe a brief introduction to the case would be to describe the events preceeding the discovery.

During the summer of 1930, senior staff members of the Alkali Division* of ICI at Winnington considered the prospects of a long term research programme for their own laboratory and other research interests of ICI.

One of the projects selected was the effects of the high pressures on chemical reactions put forward by two young students, J. C. Swallow and M. W. Perrin, and this was put to the local Board in January 1932.

The proposals were accepted and work started on the reaction of organic chemicals at high pressures. Some of the first reactions were with carbon monoxide on ethylene which gave a polymer of acrolein and not acrolein itself. Many other liquid phase and gas phase reactions were done and amongst

* This unit is now part of the Mond Division.

them was the reaction of ethylene and benzaldehyde suggested by Professor (Sir) Robert Robinson, a consultant of the Dyestuffs Division later President of the Royal Society. It was hoped to obtain proprial phenone but this did not occur - a leak developed in the equipment, and the steel gas inlet tube was coated with a thin waxy solid. This reaction took place between 24th and 27th March 1933. The reaction was repeated several times and in July 1933, the white waxy solid was eventually identified as a polymer of ethylene - but after several explosions when repeating the experiment with ethylene alone, it was decided to halt the programme until better equipment was available.

When the results of the high-pressure work were re-viewed in 1935 it was decided to continue the study of the polymerization of ethylene. During December 1935, the experiment was carried out first unofficially by Perrin and W. R. D. Manning (Research Engineer) and then officially by E.G. Williams, J.G. Paton and F. Bebington when about 8 gms. of the polymer were obtained. There had again been a 'fortunate' leak in the apparatus, allowing just the right amount of oxygen in for the polymerization. The polymer was now recognised as something new and exciting and this prompted some more detailed work to be done. By January 1936 some properties of the polymer had been evaluated and by April the first British patent specification was filed under the brand name Alketh (later changed to Alkathene). British patent 471,590 was granted to ICI on 6th September 1937 and it was not long before other patents followed in this country and the USA.

B.2 Development and Innovation of Polyethylene

The innovation of polyethylene has a long and interesting history but before describing it, it is probably worth noting what Perrin had to say about the discovery some years later:

> "Since a chance discovery may, and probably will lie outside the field of immediate interest to those who make it, rapid and effective recognition is more likely if the research man concerned has as wide a knowledge as possible of science and its application in industry. The contribution of the specialist is certainly needed and this, again, also serves to emphasise the importance of collaboration in, and appreciation of, the work of others in widely different fields of academic and applied research." (4)

This collaboration was certainly tried by ICI, whether it was successful and as rapid as was hoped is open to debate - the idea is sound, its just not certain how sound the application was, particularly before the Second World War.

During 1936 and 1937, three immediate tasks were set:
- Produce sufficient material, so that a more widely based evaluation of its properties could be made. This was done, firstly by increasing the reactor size from about 80 ml to 750 ml to produce material on a pound scale and then, from 750 ml to 9 litres early in 1937. A plant to make 50 tons of ethylene per year was also built at this time.

- Improve equipment to compress ethylene to the required pressures. This unit became available in early 1937 - it was designed by Professor A. Michels, a Dutch scientist who had long been acquainted with ICI.

- As the scale increased, it became imperative to learn how to control the polymerization and dissipate the heat generated by the reaction. This led in March 1938 to a small experimental plant being built on the site, capable of continuous operation - and by the end of that year 1 ton of polyethylene had been produced.

Simultaneously to these developments, attempts were made to find uses for the material:

1. All the divisions within ICI who might be interested in the polymer were visited, and samples left with as many as possible. In fact, during August 1936, a small sample (3/4 oz) was sent to the Dyestuffs Group by W. H. H. Demuth (Development Director). There, B. J. Habgood of the Rubber Laboratory saw it - he had recently joined Dyestuffs Group from Telegraph, Construction and Maintenance Ltd.(TC&M). It was he who pointed out the similarities between it and gutta percha* which at that time was the best known low-loss insulator for submarine cables - he requested a further 1/2 cwt for further evaluation. The importance of this will be seen a little later.

* Gutta percha had been used as a cable insulant since 1856. With the advent of higher transmitting frequencies there was a need in the cable industry for a material with a lower electrical power loss. As if a further incentive for change was needed, gutta percha was a jungle product and therefore, subject to erratic supply.

2. A detailed investigation of the electrical properties was carried out by the Research Department of Metropolitan - Vickers Ltd. on the understanding that the results would be freely available to anyone interested.

3. It was decided in June 1937 at a marketing meeting held with W. F. Lutyens, chairman of Alkali Group that development of uses should be split between various divisions - moulding uses by Mouldrite Ltd.*, textile uses by Dyestuffs Group and electrical and other unspecified uses by the Alkali Group. A circular note to this effect was sent to all ICI Sales Offices on the 23rd December, 1937.

4. Sales representatives were mentioning the polymer to their customers and:

   "Already by September 1937, a Mouldrite representative (at a meeting concerned mainly with Perspex) was discussing with the Air Ministry the possible use of polythene for de-icing, due to its chemical and electrical properties at low temperatures, and by December were working with Explosives Group on extrusion of polythene." (11 part 2, page 94).

5. In November 1937, the Cable Makers' Association (CMA) was supplied with all the information then available about polyethylene.

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* Mouldrite Ltd. later became Plastics Division.

ICI had good relations with the CMA and they were therefore one of the first contacts. A small technical committee was formed, "which reported favourably on the electrical possibilities of polythene", and at CMA's request "ICI agreed to refrain from approaching other cable makers for a limited period and to give CMA a slight price advantage". (11 part 2, page 94).

At about the same time, (November 1937), the Sales Committee fixed the price of polyethylene at 15 shillings (75p) per pound for powder and crepe form polymer and £2 per pound for thin silk or thread. The high price was fixed to prevent too keen an interest in the material at this time - and in fact from the very start polyethylene has been "priced," even sample material to potential customers, and between Groups was charged. Mr. Preston and Mr. J. L. S. Steel (commercial director of Alkali Group) were appointed the responsibility for development sales of the product at this same meeting.

It was not until late 1937, early 1938 that polyethylene was taken seriously as a substitute for gutta percha - previously it had been thought of only in its waxy phase, as indicated by Swallow: "This is a new point of view, since it has been our habit to regard polythene as a very superior paraffin wax." (11 part 2, page 98).

Because of the technical agreement between ICI and Du Pont*, Alkali Group were in constant touch about polyethylene's evaluation and it was due to the wishes

* ICI/Du Pont technical agreement dated back to 1929.

of Du Pont that polyethylene publicity was delayed. In March 1938, Swallow wrote to A. E. Hodgkin (chairman of Mouldrite Ltd.)

"In deference to the wishes of Du Pont Company who, in their own words 'wish to nurse this product as carefully as if it had been a Du Pont development' we agreed not to make any formal publications in the literature on polythene, a course which is contrary to that originally intended by the Alkali Group, whose aim was publication of this sort in order to prevent the taking out of a large nubmer of user patents by third parties". (11 part 2, page 98).

Du Pont acted very cautiously over the development of polyethylene and it would appear that they wished to keep the developments within the organisations at that time - though they did say that outside contacts would be necessary for electrical uses. They had investigated film but "thought (it would be) too soft for wrapping foil", and coated paper of which they said "attractive hightly water repellant creaseproof papers have been prepared". (11, part 2, page 98).

Another development took place in March 1938 when Caress and Renfrew visited Metals Group at Witton to discuss polyethylene's use in cartridge cases - it was decided to continue the research work on these.

Then in April 1938 the Research Department reported on the comparative properties of Diakon, Polyethylene, Formwar and Mipolam.

It was at this time that Alkali Group decided against allowing polyethylene patents being used as a basis for some of the new resins prepared by I. G. Farbenindustrie in Germany - this proved most fortuitous in later years during the Second World War when Germany was without polyethylene and hence lacked a very important material.

Several developments took place in May 1938 and throughout the summer of that year. Firstly, Batten reported favourably on the injection moulding of polyethylene and advised Alkali Group to file patent applications.

Secondly, when the Commercial Committee met on the 18th May, 1938 they appointed Swallow to be in charge of the development of polyethylene with Steel instead of Preston.

Thirdly, whilst CMA were investigating the uses of polyethylene in electric cables (except submarine cables) Telegraph Construction and Maintenance Co. Ltd. (TC & M) became interested, quite by chance. This occurred when J. N. Dean "learnt of polythene (by reading the report of Lord McGowan's speech at the Annual General Meeting of the company) and after examining a small sample recognised its potential value for insulating high frequency submarine cables". (11, part 6, page 200, quoted from ICI Magazine, September 1954). Thus it was, that the chance recognition of polyethylene's cable possibilities by Habgood back in August 1936 (remember he was a former TC & M employee) and Dean's realisation of the potential in the early summer of 1938 started the submarine cable application.

TC & M had many years of experience with the problems and techniques of submarine cable manufacture, and it did not take long to test the insulating properties of polyethylene when they received a sample in May 1938. Tests were being done in conjunction with the Post Office and by September 1938 a polyethylene insulated cable had been laid between the Isle of Wight and England and so successful was it that an order for 100 tons was placed with ICI for delivery by the middle of 1939. This was done despite the fact that there were some technical problems associated with the extrusion of polyethylene. The use envisaged was for two submarine cables between Scotland and Norway - and the order was later raised to 150 tons for delivery in batches from February 1939 after price quotes from ICI.

The first mention of an order of this magnitude (100 tons) came on the 6th September, 1938 and ICI realised there was not the slightest hope of meeting such an order on the present plant. An extension to the plant (up to 300 tons/year) was considered but a decisision was delayed until it was known whether TC & M would accept or reject the quote of 5/- (25p) per pound for 150 tons.*

The quote was accepted and with the first order for 100 tons of polyethylene for the submarine cable industry the decision was taken to build the first commercial plant. In ICI at this time "hopes of a good market ran high, as there was a strong possibility that a Transatlantic telephone cable would

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* Sales Committee agreed to quote 5/- per pound on 21st November, 1938.

be started within five years and with one laid, a second competative one would be almost certain. If for this application polyethylene was decided on, the usage would be 800 tons per cable". (11 part 2, page 100). This plant was completed and came into operation in September 1939, prophetically on the day on which Germany invaded Poland. The plant, built at Wallerscote used 50 litre vessels - far larger than hitherto, and severe technical problems were encountered, besides the problem of supplying ethylene of the required purity. Technology at that time, had developed in the ICI group for ammonia synthesis and other high pressure work, but only up to a few hundred atmospheres - and not up to the two thousand atmospheres required. Whilst time was given to the engineers to develop the necessary equipment, a piece of laboratory apparatus designed by Michels was scaled up and used, even though it was condemned by every engineer who ever saw it.

It might be as well to remember the background on which the decision to build the first commercial plant at Wallerscote was taken. J. A. Allen reveals some of the problems in his case study (1).

> "While polythene in other physical forms, for example film and solvent spun fibre, had been made in experimental amounts in 1938, the only firm market appears to have been the submarine cable industry which could scarcely be described as one with a large growth potential. Hunter recalls that at the time the estimated demand was 2,000 tons per year. The technology was new in an extreme form as evidenced, the research and

development charges extending over a number of years cannot have been inconsiderable, the costing, at best, was uncertain and the proposed price of five shillings a pound probably as experimental as the plant and product itself. There have been several points in this narrative at which the work in research and development might have been justifiably terminated, and yet this was not done. Seldom can there have been so little commercial encouragement arising from an estimated return on the investment or from any of the more or less sophisticated indices useful in arriving at an investment decision, but these considerations, if indeed they were seriously contemplated, were to be summarily removed by the demands of war."

But returning to the summer of 1938, other developments were still taking place:

- Several uses for polyethylene were being explored - bonding of textiles with emulsions, water proofing of paper, electrical uses other than submarine cables, e.g. condensers.

- A wrangle was developing between ICI and the Cable Makers Association over the exclusive supply of polyethylene wanted by CMA. Remember it was November 1937 that ICI had given CMA the chance to develop electrical uses exclusively (excepting submarine cables) and not much had been forthcoming. CMA still wanted a further six months trial, with no guarantee of a satisfactory conclusion in January 1939. ICI could see no way of meeting them - in the end they withdrew their offer of exclusive terms to CMA and started "to negotiate with go ahead firms outside the ring, notably Telegraph Construction and Maintenance Company". (11 part 2, page 100).

- Political arguments within ICI were developing over who should have responsibility for the development of polyethylene and sales. This continued through to December of 1938 when it was suggested that a "Polythene Development Committee" should be formed "under the direction of Alkali Group but with members from other interested groups and from the ICI Development Department". Even this idea was hotly argued over, by Lutyens in particular who said it would be "just another committee which would not do anything". (11 part 2, page 102). Regular bi-lateral meetings with other interested groups were suggested but the argument appears to have continued with nothing firm decided on.

- Fears about third party users taking out patents gave rise in July 1938 to the decision that "to prevent obstructive patenting, the Plastics Group should publish their research work on various products at quarterly intervals". (11 part 2, page 128).

Throughout 1939 interest in polyethylene was increasing rapidly, so much so that in March 1939 ICI tried to restrict third party users patenting by preparing two booklets - one general the other 'secret', to include all known uses of polyethylene.

The 'secret' booklet was to be sent to the Department of Scientific and Industrial Research as a form of protection against such outside patenting.

It seems rather strange therefore that whilst they were trying to restrict outside interest the Sales Committee should in the same month (March) reduce the experimental price from 15/- (75p) to 5/- (25p) for 2 cwt lots, with higher prices for smaller orders.

The booklets were never completed satisfactorily due to firstly, holdups pending a market survey which was authorised by the Delegate Board in May 1939 - and the survey was delayed in June 1939 because of unsatisfactory discussions with Alkali Group on policy: and secondly when it was completed, it was found to be completely out of date. So a compromise was reached and a list of all the possible uses was prepared and sent to the DSIR on the 16th August 1939.

Early in 1939, Plastics Group were anxious to develop polyethylene, and put one man almost entirely on polyethylene - they took provisional patents out on bottle closures and in March 1939 sent some special closures to Kork-N-Seal. However, Kork-N-Seal were not interested due to the "smell of polythene, and work was put in hand to eliminate this if possible". (11 part 2, page 95).

During July of 1939 Alkali Group received an enquiry from Telegraph Constructions & Maintenance Ltd. for between half a million and one million polyethylene cable spacers. The enquiry was passed to Plastics Group at Billingham and in early August, 1939, agreed to make them even though they thought "it was undesirable to undertake moulding as a normal function". (11 part 2, page 95). It appears that policy had still not been agreed on as to whether they were raw material manufacturers or finished product manufacturers.

Du Pont were kept very much in the picture throughout the year and were sent samples of 100 lb in January and 112 lb in February. On the 28th February 1939 a team of Du Pont experts visited ICI Winnington and the ICI engineers werepraised for their work by Mr. Dittmar of Du Pont. The next month (March), Lord Melchett wrote to Jasper E. Crane of Du Pont offering polyethylene as a "major invention" - this was acknowledged on the 4th April, 1939. In June of 1939 Du Pont started to approach other companies in the USA about polyethylene - the General Electric Company who were not particularly interested, the US Rubber Company who Du Pont said were not very promising and the Bell Telephone Company who were regarded as a promising contact. They were following with interest the developments in the UK with submarine cables.

Other interest abroad was being developed:

- By the Chemical Export Sales Department who sent polyethylene information during May 1939 to their agents and received enquiries from Belgium, Norway, Sweden and even Latvia.

- Information and samples were sent to Cables de Lyons and M. Bommelaer of the Societe Alsacienne de Constructions Mecaniques in France after suggested co-operation for super tension cables, in July 1939.

- Two Dutch representatives from the Ministry of War visited Winnington in July 1939 after polyethylene had been introduced to the Dutch Government by Michels.

By the time the Second World War broke out eighty one companies had been contacted by Alkali Group and there were at least seventeen applications under consideration. This had been achieved even though arguments about policy between the Groups still raged. The possible applications under discussion when the war broke out included:

Balata belting
Balloon Barrage, treatment of ropes
Battery Boxes
Battery spacers
Bottle closures
Cables - submarine, power and high frequency
Candle ingredient
Cartridge cases
Chemical plant protection
Condensers
Golf balls
Metal coating
Petrol tanks for aircraft
Printing ink ingredient

Surgical applications - bandages,
sleeving

Textiles and paper - impregnation and
coating of

Windolite substitute

B.3 The Effect of the Second World War

The advent of the Second World War effectively changed the development of polyethylene. No longer was the polyethylene from Wallerscote destined for submarine cables alone, for other important uses had been found - mining cable, and shortly after flexible high frequency cable for radar installations. The development of polyethylene in radar had started a couple of years prior to the war, when Sir Robert Watson Watt (the inventor of radar) contacted Metropolitan Vickers. It was on the 22nd of January, 1937 that he gave Metropolitan Vickers the transmission details of RDF* (13, page 181), and it cannot have taken MV long to realise that polyethylene (which they were investigating for ICI at the same time) would fill an ideal need in radar. Indeed the significance of the development of polyethylene was not lost on Sir Robert Watson Watt:

> "The availability of polythene transformed the design, production, installation and maintenance of airborne radar from the almost insoluble to the comfortably manageable. Polythene combined four most valuable properties in a manner then unique. It had a high dielectric strength, it had a very low loss factor even at centimetric wavelengths, it could fairly be

* S... 470

described as moisture repellant, and it could be moulded in such a way that it supported aerial rods directly on watertight, vibration proof joints backed up by a surface on which moisture films did not remain conductive. And it permitted the construction of flexible very high frequency cables very convenient in use. A whole range of aerial and feeder designs otherwise unattainable was made possible, a whole crop of intolerable air maintenance problems was removed. And so polythene played an indispensable part in the long series of victories in the air, and the sea and on land, which were made possible by radar". (Quoted by J.C. Swallow in Polythene ed. A. Renfrew and P. Morgan Lliffe and Sons, London, 1957, page 7.)

The output of polyethylene from the Wallerscote plant soon became inadequate and another plant was built - the process was not altered they simply increased the scale of things. The 100 ton plant built at Winnington, largely due to P. Allan's instance came about because the Ministry of Aircraft Production had a need for chlorinated polyethylene as a 'dope' for aircraft canvas. No such use ever developed but the extra capacity was no doubt welcomed. (12). By this time though control of the process was easier and dur- ing 1940 the polymer was characterised by the melt flow index. This is a measure of the viscosity and hence the molecular weight which it is directly related to.

Whilst the applications were being developed, work was still continuing on characterising polethylene and in 1939 C. W. Bunn showed that the polymer was not as fully crystalline as supposed. This led to the realisation that materials with different crystallinity could be made and hence materials of different densities and other physical properties. A short time after, in 1940, this picture was modified when Fox and Martin in England showed by infrared analysis that methyl groups were present in the polymer. This proved conclusively that the chains were branched but in what manner was not known for several years. In fact the producers of polyethylene were so fully occupied in supplying the conventional type of polymer that it was not until the mid 1950's that these results were made use of.

Turning back to the effects of the war - the submarine cable having proved successful between the Isle of Wight and England set the precedent for two cables to be laid across the English Channel after the invasion of Europe for military communications.

Then in 1941 information about polyethylene was given to the United States via two channels - the war reciprocation agreements between the governments of the two countries, and also by the technical agreements, which was in existence between ICI and Du Pont. Probably the most important of these two channels was the ICI/Du Pont link up, and in November of 1941 a team from Du Pont visited the Winnington plant. Sir Harry McGowan of ICI made the offer to Mr. Walter Carpenter of Du Pont after recognizing the signal importance of polyethylene to the war effort. The Du Pont team then went home at the end of their visit with all the engineering drawings, specifications and operating instructions of the Wallerscote and Winnington plants.

After seeing the ICI plants Du Pont decided later that same year to develop a different kind of plant using a wet tube process instead of an autoclave - this was largely due to their worries about dissipating large amounts of heat from the polymerization reaction.

Union Carbide and Carbon Corporation now came into the picture quite independently of ICI and Du Pont. They developed a dry tube process and by 1943 had built two plants in West Virginia with assistance from the US government - these went into production that same year.

Back at ICI the war period was allowing many technical problems to be solved such as "What caused voidages in extruded cable coverings? How was the extruded cable best cooled and how could large mouldings be made which would be undistorted and unstrained on cooling?" (7, page 178).

Many of these problems were solved by introducing extrusion and moulding equipment into the laboratory and working in close co-operation with the users. A plasticizer, polyisobutylene was added to polyethylene to help extrusion and also improve low temperature flexibility - an anti-oxidant was also needed to help whilst processing because extrusion was causing cross-linking and increasing the dielectric loss due to an oxygen pick up.

Then towards the end of the war, machinery capable of processing more viscous materials became avialable. This allowed the introduction of polymers with greater molecular weights which had better low temperature flexibility and solvent embrittlement resistance. The technology in the US plastics industry was more advanced than in the UK and they experienced no difficulties in extruding even the higher molecular weight polymers. One might wonder why the technical agreement between ICI and Du Pont did not allow this information to filter back across the Atlantic - there certainly does not appear to be any record of this happening.

B4 Post War Developments

By the end of the war, (1945) the US production was 1,500 tons per annum greater than the UK production. There was an immediate expansion in production capacity, particularly by Union Carbide, and polyethylene now became available for other uses. ICI realised at this time, that if polyethylene was to become more than just a speciality product a cheaper method of production for ethylene must be found. This would appear to be the first instance of ICI looking for some economies of scale for polyethylene. It was not until 1951 that a plant for cracking naptha was completed - this gave a cheap and abundant supply of ethylene to the Wilton works of ICI in particular.

The companies in the USA stole an immediate lead over ICI at the end of the war for several reasons:

* they were more consumer oriented, with a large home market.

* there was a more favourable, competative economic climate than in Europe.

* they already had established expertise in plastics manufacture which led to developments in the film and injection moulding fields.

However, these factors must be tempered by two features outside ICI's control:

* The cost of ethylene fell sharply - particularly in the USA.

* The US government filed a case against ICI-Du Pont under the Sherman Anti Trust Act, which resulted in ICI being obliged to offer licenses to other US companies - four companies purchased the complete know-how on polyethylene, whilst two others purchased patent rights, as from 1952.

ICI was also licensing other companies throughout the world and Europe in particular and as a result production in the UK and USA doubled and the price fell from 49 cents/lb to 41 cents/lb between 1953 and 1955. This had the effect of encouraging growth and led to an increase in the research and development of olefine polymerization techniques.

Then in 1956, another submarine cable was laid, this time across the Atlantic - the first polyethylene telephone cable to link the UK and USA. This concentration on cable development certainly played a very prominent role in the polyethylene story and it was some years later that Swallow said:

"This concentration on one use in the years before the war was, I believe, correct and it certainly saved a great deal of money. During the war the entire British and American output was employed in high frequency uses so that no problem existed, and after the war it was applied immediately for uses in the growing field of television and commercial radar cables."

The search for new uses for polyethylene continued throughout the 1950's, and by 1952 it was becoming apparent that polyethylene had extensive uses in not only cables but also film/sheet, moulding, pipe, coatings and bottles.

B5 High density polyethylene

It has already been stated that during the war years, the polyethylene manufacturers were fully occupied in producing the conventional polymer with a density of 0·92 g/cc. However, during the early 1950's three new processes appeared for making polyethylene - all coming about by chance when researching into something quite different. The three were developed by Standard Oil of Indiana in 1951, Phillips Petroleum in 1953 and the most important by Karl Ziegler (of Max Planck Institute for Coal Research at Mulheim) in November 1953. These processes* produced a polymer using low pressure and yielded a higher density material - 0·94 to 0·96 g/cc.

* Details of the processes can be found in Studies in Innovation in the Steel and Chemical Industries by J. A. Allen.

It was not until 1954 that ICI discovered how to make a high density polyethylene - up to 0·94 g/cc, by modifying their process. This was done to protect their position as inventors of the material. The higher density material is stiffer than the lower density type and hence the wall thickness of many components could be reduced, enabling it to compete very effectively on a cost basis.

B6 Commentary and discussion

The story of polyethylene, its development and search for uses has been given for the period between 1930 and the mid 1950's. However, as you will have realised the history concentrates on the period before the war; for two reasons (i) the study is concerned with the early search that the company undertook and (ii) the effect of the war must surely have had an effect on polyethylene development and the study really wishes to deal with materials subject to commercial pressures.

B6.1 Search methods used by ICI

After the 'official' discovery of the polymer of ethylene, ICI quickly realised thatthey had a potentially important material and they sought potential uses, whilst improving production techniques, and the material's characteristics. The strategy involved in the search was basically one of an inter disciplinary approach and they made five specific acts which made it so:

- they involved other ICI divisions by visits, leaving sample material and the known technical details in the hope that uses would be suggested.

- they allowed experts in the electrical field (i.e. Metropolitan Vickers) to investigate the electrical properties and to inform anyone interested of the results.

- the development of potential uses was split between the various divisions.

- sales representatives mentioned the polyethylene development to the customers they came in contact with.

- the Cable Makers' Association were given the opportunity of developing uses in their field exclusively.

A short time after setting this search process into operation, ICI informed Du Pont of polyethylene's developments. As has already been intimated this was due to the technical agreements then in operation between the two companies.

But if we examine the two early commercial products which came into being, namely submarine cables and radar cables the picture of how needs and technology were brought together will become clearer.

B6.1.1 Submarine cables

It is interesting to see that as early as August 1936 a potential use for polyethylene had been recognised by Habgood (of Rubber Lab., Dyestuffs Group). Remember he had only recently joined ICI, having come from a cable manufacturer TC & M Co. Ltd., and, therefore had prior knowledge of the cable industry's needs. The similarity between polyethylene and gutta percha was immediately recognised by Habgood but the development of polyethylene as a cable insulant was delayed until the potential was recognised in the cable industry itself. Presumably Habgood's findings were not pushed back to TC & M because the Cable Makers' Association had been given the chance of developing uses exclusively in their field. But polyethylene moved very slowly in the CMA at that time and ICI began to despair of them during the summer of 1938. A further reason for the delay in such developments must surely have been because ICI did not regard polyethylene as a serious substitute for gutta percha, but were considering uses only in its waxy phase. This would tend to indicate that even though an inter disciplinary approach for new uses had been adopted, the decision makers in the company still held to their own disciplines and were slow to accept new ideas.

Fortunately though for ICI, J. N. Dean, owner of TC & M read of polyethylene in what must be a fairly unusual manner, in the annual report of ICI. It should be pointed out that submarine cable manufacturers were seeking better dielectric materials at this time as higher transmission frequencies came into being. Developments with K-gutta and Paragutta were going on but a fundamentally new dielectric was really needed.

Dean quickly realised it to be a potential substitute material for gutta percha as Habgood had done in August 1936. Once the material had been evaluated at laboratory scale, things moved very rapidly indeed and by September 1938 a test cable had been laid from the Isle of Wight to England and very quickly proved itself. This led to an order of 150 tons of polyethylene for delivery during 1939.

B6.1.2 Radar cables

With the establishment of polyethylene as a cable insulant, really it was inevitable that other cable applications should come along. But to begin with it was only in those areas where established dielectrics were failing or a suitable dielectric was unavailable. The development of radar cables falls into the second category and evolved rapidly once the need area had been recognised.

The concept of radar had been known by Sir Robert Watson Watt (the inventor of radar) and the British Government for some time (1935) - the development of the hardware was not instigated though until January 1937. It was then that Watson Watt gave details of RDF* to two companies - Metropolitan Vickers Ltd. and A. C. Cassor Ltd. Metropolitan Vickers were asked to do some research work on the transmission side. And according to Watson Watt he chose those two companies because of his "personal knowledge of the qualities of their research people and a knowledge of their resources in general".

Now Metropolitan Vickers had previously been asked to investigate polyethylene's electrical properties by ICI and it did not take them long to realise that polyethylene could fill the need for a dielectric in radar cables. However the development of such a use seems to have been slower than submarine cables which had been field tested by September 1938 - it was not long after this though that radar cables became the major user of polyethylene.

* RDF stands for radio direction finding and was a spurious title given to radar to confuse unwanted observers. The title 'Radar' was adopted during World War II and stands for, Radio Angle, Direction and Range.

B6.1.3 Lessons to be learnt

It is apparent from both of these early polyethylene uses that they were fulfilling a need, either because the present material (gutta percha) was failing and hence polyethylene could be substituted or, as happened with submarine cables; because there was no other material that would do the job.

The polyethylene development follows the discovery/push type model, and it is apparent, that ICI had difficulty in actually identifying the first user. However, it was fortuitous for them that the first two applications for polyethylene were areas where there was a positive need.

In communicating polyethylene developments to the outside world it is interesting to note that at the outset ICI selected their audience, i.e. the Cable Makers' Association, Metropolitan Vickers and known customers that sales representatives visited. It was not until they removed this selection process, by communicating through the AGM report that the first 'need' application arose. And it is doubtful that they realised that the AGM report was a potentially effective information channel. One should remember though, that ICI had wanted to publicise polyethylene to the world through technical publications but Du Pont stalled them on this.

Both applications in submarine and radar cables illustrate just how quickly new materials can be adopted into useful products. If we take submarine cables first, then we see that TC & M first learnt of polyethylene during the early part of 1938 - and by November 1938 had evaluated the material, performed field trials and ordered over 100 tons - a matter of six months. The radar cable adoption was not quite so rapid but still very fast for a new material. Metropolitan Vickers knew of polyethylene round about the end of 1936 beginning of 1937 and were producing polyethylene coated radar cables by mid 1939 - approximately eighteen months between user awareness and adoption.

B6.2. Other search methods used

As has been stated above Du Pont stalled ICI's attempts to publicise details of polyethylene but by July 1938, ICI had decided to go ahead. Whether the decision to publicise was taken with the aim of using it as possible information channel to stimulate new uses, or because of the fear of third party user patents, is not made clear. However it does not alter the fact that various pieces of research work were publicised at three monthly intervals. Unfortunately the relative success of this as an information channel is unknown.

Apart from information about polyethylene being sent to the USA via Du Pont, European and Scandanavian countries were told of the developments. This information was sent to ICI's agents by the Chemical Export Sales Department and in the case of the Dutch enquiries by Professor Michels (of Amsterdam University who had done a great deal of the early research work with Alkali Division). As far as is known no new uses were forthcoming from these channels.

It appears that most of the seventeen possible applications being considered before the war were due almost entirely to the sales representative team. These uses ranged from the high technology e.g. chemical plant protection, condensers etc. to the low technology e.g. golf balls, bottle closures etc. Whether any of these uses would have been developed as major products is hard to say because the war affected the polyethylene output so much. It must be said though, that the low technology uses of polyethylene which we all know of today were to a large extent known then - for example plastic bottles, polyethylene/textile aprons, polyethylene film. Interestingly enough the film uses thought of were nearly all high technology, for condensers as a dielectric at high frequencies although one enterprising customer bought some sample material to try to make beer aprons. Discussing such possibilities brings us on nicely to the policy which ICI adopted towards polyethylene.

B6.3. Polyethylene and ICI Policy

The policy regarding the technical aspects of the material has already been stated in the study - produce sufficient material to evaluate its properties, improve manufacturing equipment and learn to control polymerisation. And the policy for searching for new uses has been discussed above in part.

The points not so far covered include pricing strategy, raw material producer or finisher product manufacturer and development.

B6.3.1. Pricing

A price of fifteen shillings (75p) per pound was initially set by the Sales Committee in November 1937 and even though this was a high price it appears to have been arrived at in an odd fashion. It was not set at a "production cost plus" or even what ICI though they could get for it on the market but at a price which it was hoped would discourage too keen an interest.

The Sales Committee decided that this price should be paid for all sample material to potential users. No sample material was given away not even to Du Pont whom they had a technical agreement with. This may certainly have encouraged customers to think hard about potential uses when it was bought but it probably also had the desired effect of discouraging interest - would that be done nowadays?

The price did come down to five shillings (25p) per pound on the 6th of September 1938 when ICI quoted TC&M for 150 tons. But whether this was fixed as a realistic price is dubious; an extension plant of 300 tons per year was being planned and could not go ahead until orders for the material were forthcoming. It was on the basis of this 150 ton order that new plant was planned for commercial production; with the hopes of new transatlantic cables using 800 tons per cable in the offing.

As the output increased, the price slowly fell and by 1945, ICI was selling over 1200 tons per annum and the price was down to three shilling nine pence (19p) a pound.

B6.3.2. Raw Material Producer or Finished Product Manufacturer?

The question of whether a company with a new material is going to be a raw material producer of finished product manufacturer is often asked. In the case of ICI it appears they were prepared to look at anything and everything.

As it turned out for the submarine and radar cables they supplied the raw material to fabricators but had to stimulate the end product in the process. Indeed throughout the history ICI have been closely involved with the development of end products on their own sites - even if only to pass on their technical expertise to fabricators and hence ensure products did not die premature deaths. And in some instances they manufactured the finished product, for example cable spacers were made for TC&M even though moulding was not regarded as a normal function at Plastics Group at that time.

Throughout the early history ICI endeavoured to patent all possible applications or publish the details so that no one else could take out patents. This was clearly illustrated during 1939 when Plastics Group took out provisional patents on bottle closures before approaching Kork-N-Seal with the idea. The development of such a use was delayed through for two reasons - Kork-N-Seals' objections to the smell during manufacture and the oncoming war.

B6.3.3. Research & Development

As has been stated the development of uses was split between the various divisions, and researching the material's characteristics was carried out by Alkali Group. If anything, R&D commitment was increased as time went by, across the spectrum of basic research to product development.

For instance research was being carried out on cartridge cases, injection moulding, comparing polyethylene with other plastics, besides investigating the basic structure of the polymer.

Swallow was one of the men who instigated the initial ethylene research and it was largely he who followed the developments through - being put in charge of polyethylene's commercial development with Steel in May 1938. However, the development of polyethylene did not follow a smooth passage and many arguments arose over who should be responsible and how it should be organised. It is interesting to see that an innovation committee or as ICI called it a 'Polythene Development Committee' was suggested but not taken up. Neither were regular bi-lateral meetings between interested groups - in fact to the outsider it appears to have moved along in a rather erratic fashion. Not that that appears to have harmed polyethylene's development - maybe such political arguments show enthusiasm across a firm for such projects, but if carried too far one can imagine the whole thing collapsing.

The development programme continued through the war, probably even more intensely - but concentrated on specific products rather than spread across a range. It gave ICI a mainstream outlet for their material in cables which gradually filtered downstream after the war into television and commercial radar cables. Such an outlet could be regarded as a "bread and butter" product, that is, a safe market you know you have whilst developing other product areas.

B6.4. War Effects

One of the most important effects the war had, certainly in its early years, was to create a very large home market for polyethylene. This helped ICI who were only just beginning to feel their way commercially during the late 30's when decisions to build larger plants had to be taken. Particularly relevant was the decision to build a 1000 ton plant at Winnington in 1940 - to meet a demand for chlorinated polyethylene as aircraft 'dope'. This was never developed but its effect gave ICI a large capacity plant. Plants of that size had never been envisaged two years earlier say in 1938, when the growth rate was thought of in hundreds of tons rather than thousands.

As has been stated the development programme throughout the war concentrated on improvements to cable insulation and manufacture, and it is doubtful that a similar effort would have been exerted in peace time.

Developments after the war are not really relevant to this study, because by that time polyethylene had become accepted and could no longer really be classed as a new material. After initial set backs like a deflated Europe and Anti trust acts being filed against ICI and Du Pont polyethylene uses grew and grew, and today it is probably regarded as one of the most successful material innovations.

B7 Acknowledgements

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APPENDIX C

Silicon Nitride Case History

C(ii) Information Sources for Silicon Nitride

Much of the early history was gained by scanning through abstracts - Ceramic Abstracts and British Ceramic Abstracts. Such references indicated the individuals and companies involved, and the way in which the innovation developed technically. These gave a basic understanding to the material's innovation.

A cross check with the reference book "Industrial Research in Britain" showed the research organisations involved with silicon nitride:

- United Kingdom Atomic Energy Authority, Harwell
- British Ceramic Research Association
- Fulmer Research Institute
- International Research and Development Co. Ltd.

Companies involved with the commercial exploitation of silicon nitride were found through the abstracts and confirmed by colleagues at Aston University and Dunlop.

- Admiralty Materials Laboratory
- Associated Engineering Ltd.
- Birmingham Small Arms Ltd.
- British Leyland *
- Carborundum Co. Ltd.
- Clarke Chapman - John Thompson Ltd. *
- Doulton Ltd. *
- Joseph Lucas Ltd.
- National Research Development Corporation
- Plessey Co. Ltd.
- Ransome Hoffman & Pollard Ltd.*
- Advanced Materials Engineering Ltd.*

The four research organisations mentioned above were contacted, and recommendations were made by them as to the best person/organisation to contact for information regarding the innovation history, see table C(i). The recommendations from N. Smith (Chief Metallurgist, Dunlop) came because Dunlop was a major user of silicon nitride powder. And information from H. Child came whilst interviewing him for information on titanium. It was not previously realised

*AME formed from consortium companies marked *.

Table C(i) MATERIAL SOURCE INDICATOR

Material: Silicon Nitride

CONTACTS / RECOMMENDED CONTACTS	H. Child Aston University	UKAEA	Brit. Cer. Res. Assoc.	Fulmer Res Inst.	Int R & D Co. Ltd.	N. Smith, Dunlop	
H. Child Aston University							
UKAEA							
Brit. Cer. Res. Assoc.							
Fulmer Res. Inst.							
Int. R & D Co.Ltd.							
A.E.D. (Rugby)	x			x	x	x	
A M E (Newcastle)	x	x	x	x	x	x	
Norman Parr - ex AML now with MoD	x						
AML (Poole)			x	x			
LUCAS				x	x		
Dr. Riley, University of Leeds					x		
DYSON (Sheffield)					x		
DOULTON (Stoke)					x		
NRDC					x		
Prof. Ken Jack, Newcastle University					x		

that he was an ex-BSA man with responsibility for silicon nitride. Further information about silicon nitride's innovation was gained from the Admiralty Materials Lab., Advanced Materials Engineering and NRDC.

C1 Introduction

Although silicon nitride has been known since 1857, this history deals with its commercial exploitation since the mid-1950's. The first patent (US 2,628,896) was taken out by Hendrick de W. Erasmus and William D. Forgeng in February 1953, for the Union Carbide and Carbon Corporation. This was for bonded silicon nitride abrasive products. Apart from this, the first published account was probably that by Collins and Gerby (1) at the AIME Annual Meeting in February 1955. The Americans can only just have beaten the British for starting work on silicon nitride, because although one of the first published accounts (2) by the British did not appear until 1959, it is apparent that a lot of work had been put in before this. It was the Admiralty who sponsored this work - initially as a search for a suitable stator blade material for gas turbines operating at 1200°C. Silicon nitride was actually material number 245 in the search carried out by G.F. Martin and E.W. May. Norman Parr - who is regarded by many as the father of silicon nitride - realised the importance of their work, and homed-in on silicon nitride. He has since led the developments with the material from his base at the Admiralty Materials Laboratory (AML) in Poole.*

* AML is now known as the Admiralty Marine Technology Establishment.

The fortunes of silicon nitride have fluctuated madly over the years. Many companies have entered the silicon nitride race - some remain, others have fallen by the wayside, but even when they do, another party seems to appear over the horizon to pick up the pieces. This history cannot do justice to the political arguments that must have bedevilled the material. What it tries to do is follow some of the milestones and show how various British organisations have tackled the problem of getting users to adopt this ceramic as an engineering material. The story, then, begins with AML; diversifies, as a cluster of companies pick up the challenge after gaining licences, and converges onto one or two companies who remain in the race, with the aim of being the first to exploit silicon nitride successfully. To date, the prize has eluded all.

C2 Silicon Nitride - How You Make It

To understand the developments that have taken place with silicon nitride it is as well to know how it is made and what it can do - as indicated by the physical properties of the material. Ceramics have never been readily accepted by engineers as engineering material. They are only interested in them when other conventional materials, such as metals, reach their physical limits. This was recognised early on by the developers of silicon nitride, and they tried to give engineers a knowledge of the manufacturing methods and the properties of the material (2,3,4,5).

Silicon nitride is normally made by heating elemental silicon in an atmosphere of nitrogen:

$3Si + 2N_2 \longrightarrow Si_3N_4$ at temperatures between 1000 and 1400°C

This method was chosen for the commercial production of silicon nitride, although another route is available:

$3SiCl_4 + 4NH_3 \longrightarrow Si_3N_4 + 12HCl$ at 1400°C

The reaction between silicon tetrachloride and ammonia has never been developed for mass production of silicon nitride, and in any case, the elements required for the first reaction - silicon and nitrogen - are readily available.

If a lump of coarse granular silicon is used, the reaction is confined to the surface. But if a powder (of 200 mesh or finer) is used, and the powder is compacted so that the angular particles are in multiple-point contact, it is possible to induce silicon nitride crystals to connect and build up a coherent structure.

The reaction between silicon and nitrogen takes place at about 1400°C, and is a two stage operation. A preliminary reaction takes place between 1200 and 1400°C and gives a cellular structure of silicon nitride confining the elemental silicon giving a high temperature rigidity. This partially-fired material - known to be in the "biscuit" state - can be clamped for machining to final shape by turning, milling, drilling, etc. An alternative, and frequently preferred method, is to form the biscuit by argon-sintering of silicon without nitrogen present. The second stage is more rapid and takes place at 1400-1500°C. It gives a material which is much harder and denser than that in the biscuit state. Machining this final material can best be done with grinding equipment. The reaction time depends on the size and thickness of the ware being fired, but it generally takes several days. It is this lengthy firing process that escalates the cost of the material, making the end product quite expensive. An early nitriding furnace developed at AML is shown in Figure C1 and a simplified flow sheet for the fabrication of silicon nitride is illustrated in Figure C2.

The methods for shaping silicon nitride components have developed tremendously over the years. Most of the early methods were taken straight from the ceramics industry - such as slip-casting for hollow shapes, pressings using first, closed steel dies, and later, isostatic (also known as hydrostatic)methods, and tamping; an exception being the flame-spraying technique developed by AML in 1958. As the years have gone by, more sophisticated methods have evolved (although many of the origianl processes are still used) using techniques developed in the metal, plastic and paper-making industries. For example, extrusion, injection moulding, foam moulding, band casting and bandage wrapping. All these methods have a common starting point - silicon powder in the "green" state - though they may vary in the additives to the mix, plasticisers for instance, may be added to act as a binder.

If plasticisers are used, they must be "burnt out " prior to the nitriding in a separate furnace. Products made in silicon nitride in this manner are known as reaction bonded (RBSN).

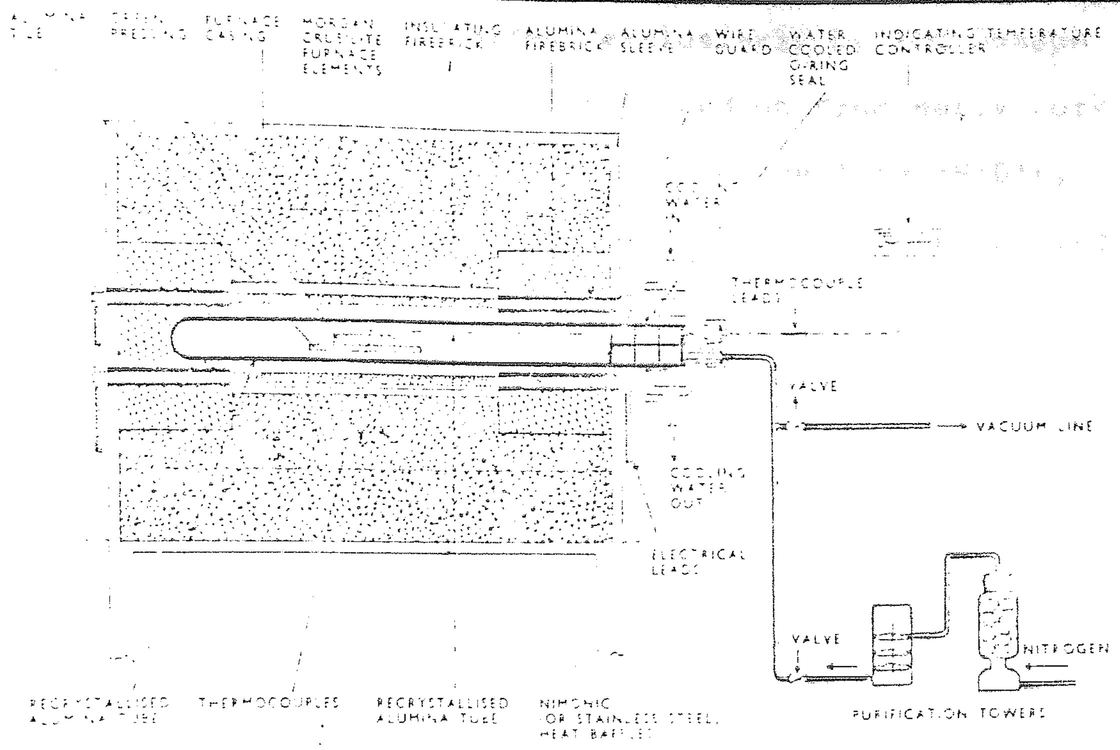

Figure C.1 Schamatic View of Nitriding Furnace as used by AML

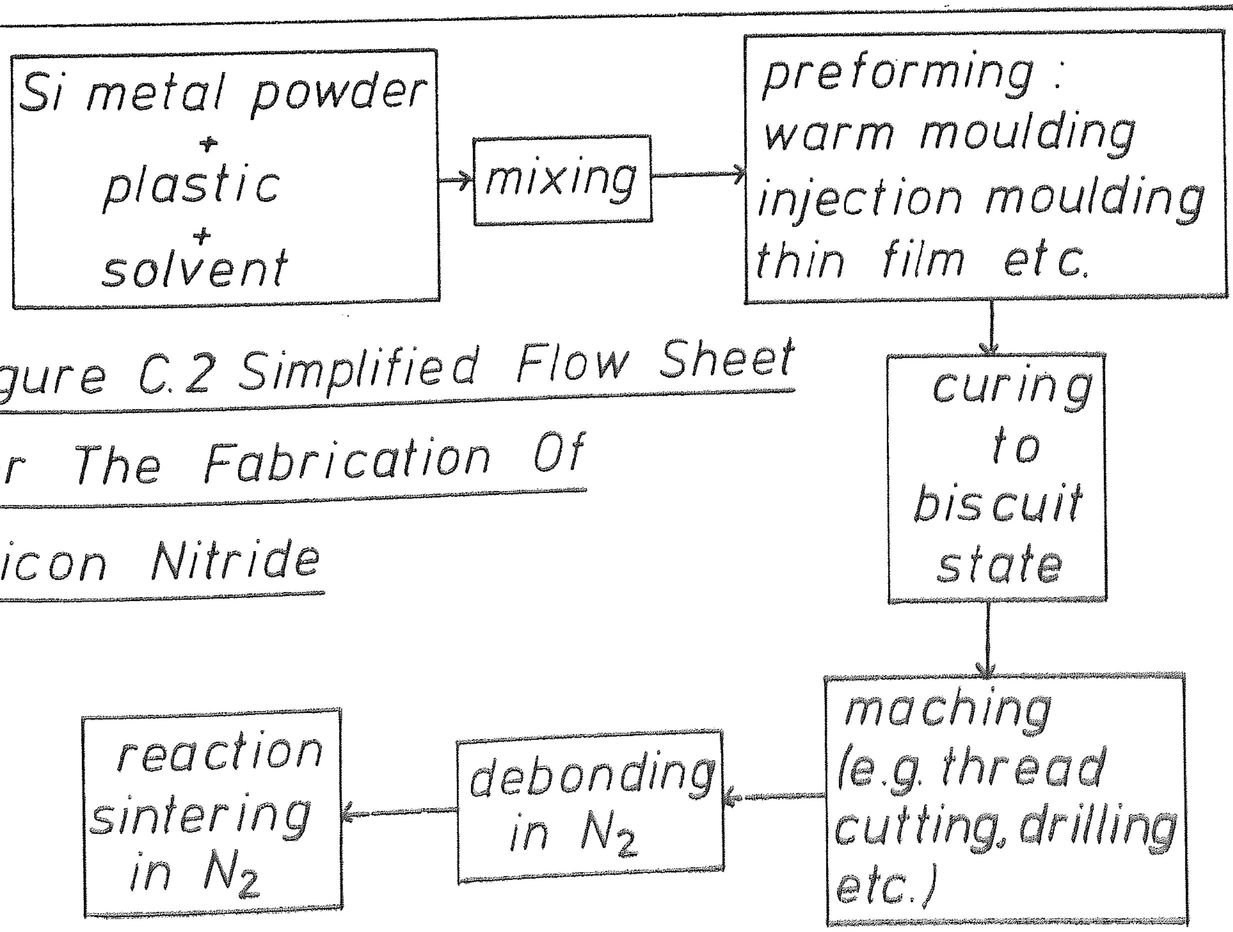

Figure C.2 Simplified Flow Sheet For The Fabrication Of Silicon Nitride

Another method of manufacture is the hot pressing technique, pioneered in this country almost exclusively by the Joseph Lucas Group Research Centre and followed on from early work done by Deeley, Herbert and Moore (6) in the late 1950's, early 60's. Using this technique, silicon powder is reacted with nitrogen to form silicon nitride powder. The silicon nitride powder is then mixed with additives and hot-pressed (the material is known as HPSN) in graphite dies to the required shape. This method gives a denser (3·2 g/cm^3 as opposed to 1·8 to 2·7 g/cm^3), stronger material than that produced by the reaction bonded process. Lucas have, over the years, developed new silicon nitride additive mixes by partially substituting aluminium for silicon and oxygen for nitrogen within the silicon nitride unit cell, to give a new breed of materials called "sialons". The strength of such materials is comparable with that of hot pressed silicon nitride but the creep resistance and chemical stability are superior.

C2.1 Properties of Silicon Nitride

As early as 1958 many of the properties of silicon nitride were known (3). Most of the early publications (2,4,5,7,8,9) carried details of the properties of the material as well as the methods of manufacture, application, design considerations and so on. They included not only the basic physical data, but clear information regarding its resistance to various molten metals and chemicals.

The most up-to-date information on the properties of reaction bonded silicon nitride has been taken from the literature supplied by AME (Advanced Materials Engineering Ltd.) and is shown in figure C3. To generalise these properties, the table below indicates its attributes and deficiencies:

Attributes

- High thermal shock resistance
- Dimensionally stable over wide temperature range (up to 1750°C)
- HPSN mechanically stronger than most, if not all other ceramics
- Hard and more resistant to abrasion than most, if not all other ceramics
- Low dry friction
- Non-heat absorbent
- Non-magnetic
- Resists molten metals including aluminium
- Good corrosive resistance (better than other ceramics)
- Easily formed and worked
- Low shrinkage on processing (less than 0·1%), better than most, if not all other ceramics

Deficiencies

- Low fracture toughness
- Inferior emissivity to silicon carbide
- Moderately low thermal conductivity and low specific heat (i.e. poor heat absorption)
- May be porous under high pressure conditions (RBSN is porous under all conditions)
- May be more granular on polished surface than finest tungsten carbide
- Difficult to get homogenous nitride bond thickness greater than 10 mm, therefore thickness limited to 20 mm approximately
- Alkali resistance no better than other ceramics

Figure C.3 Properties of Silicon Nitride

PHYSICAL PROPERTIES

Chemical Formula Si_3N_4
Crystal Structure: Hexagonal/Two Phases (Alpha & Beta)

C3 Commercial Development of Silicon Nitride in the United Kingdom

It has already been indicated that the Illinois Carbide Corporation were among the first to develop silicon nitride in the USA. Whilst they were developing small components - crucibles and boats for crystal growing and zone refining in the electrical industry, rocket motors, thermo-couple sheaths and so on - the Admiralty Materials Laboratory were hoping that silicon nitride would fulfill their expectations for use in gas turbines, for turbine discs and blades. Their first work along these lines was with silicon nitride, stiffened with a fine dispersion of silicon carbide. With this as the basic material, they went on to investigate production methods (they patented the flame sprayed reaction bonded fabrication process in 1958), properties of the material and carry out field trials. Where they didn't have the expertise for investigating various properties they employed others more expert than themselves. For instance, the National Gas Turbine Establishment investigated the thermal shock characteristics of the material for them. Field trials were also carried out for them where they did not have the facilities. PAMETRADA did some test work with silicon nitride wafers to simulate bending of stator blades in gas turbines. The Admiralty Engineering Laboratory ran an experimental turbine fitted with two silicon nitride guide vanes. Even though the rotor blade failed after 250 hours of intermittent service (when a metal blade failed), the AML people had faith that the problems could be overcome. The hope was that the next generation of gas turbines should, to a large extent, incorporate silicon nitride. Whilst that may have been considered as the ultimate use for silicon nitride, they were not slow in looking at other applications. Electrical insulators, high temperature catalysts, thermocouple sheaths, supports for heat treatments, furnace chamber linings, shackles and components for high temperature testing, supports in high temperature/high pressure water systems and brazing jigs were all applications being talked of (2) and developed. This was in addition to other MoD work being carried out at AML for combustion test rigs and rockets. All such work, sponsored by the government, was undertaken by the Metallurgy division of AML during the late 1950's and led by Norman Parr. By 1960, it was felt that the material had been proved in gas turbine applications (7) and it was time for industry to be brought in to exploit the material commercially. Accordingly, all patent and licensing rights were handed over to the National Research Development Corporation. About the same time, a contract was placed with the BSA Group research centre by AML to help in the developments with silicon nitride. BSA had extensive R & D facilities for investigating the powder metallurgy aspects with silicon nitride.

C3.1 NRDC license AML's Silicon Nitride Technology

Towards the end of the 1950's it was becoming apparent that several companies were becoming interested in silicon nitride. Besides Union Carbide and the Carborundum Company, both of whom had a history in this class of ceramics, other organisations such as Plessey (who were interested in the material because of its good creep resisting properties necessary for gas turbine blades) and the British Ceramic Research Association began investigating silicon nitride. None though, would commit themselves to commercial production until the potential market had been analysed (7).

It was not long, however, before the NRDC started granting licences to various organisations. In 1960 there were four organisations holding licences - BSA, Clarke-Chapman, Doulton and Hoffmans. BSA, as has been stated, had interests in the powder metallurgy aspects, Clarke-Chapman were interested because of the possibilities a high temperature material had in the nuclear industry, Doultons had a long history of developing ceramics and Hoffman were interested for the potential in plain rubbing bearings.

Although Union Carbide (8) - who were developing silicon nitride without an NRDC licence - were apparently leading the field in 1961, no great developments took place throughout the 1960's. The hoped-for breakthrough with gas turbine stator blades never came about: the engineering problems raised by its inductility were considered insuperable. Despite much publicity - mainly by the Americans - silicon nitride applications appeared to remain in the doldrums. There was plenty happening on the process front, with many organisations patenting new and novel ways of producing silicon nitride (see refs. 10 to 16 for example) but still no real money-spinner in the form of a product appeared. Doulton were pushing their "Roydazide" silicon nitride for thermocouple sheaths, special jigs and fixtures for brazing and welding in the jewellery industry, mandrels for heat treatment equipment and control equipment for the flow of molten aluminium. Hoffmans, with their "Hofsil" silicon nitride were having no great luck in the bearing field. And probably, like the others, BSA were only really sounding out the market for silicon nitride in areas such as the light metal foundries for handlong molten aluminium, heat treatment applications, bearings and seals. They all had technical literature (brochures detailing properties and applications) prepared, and all were trying for roughly the same markets, but getting nowhere fast.

C3.2 AME is formed

Not long after Doultons had held a symposium "The Future of Silicon Nitride" in London in 1969 - with still no great success - the NRDC decided to try and bolster up the exploitation of silicon nitride, and floated the idea of forming a consortium between the main producers of the material. By July 1970 the consortium had been formed and a new company to run the silicon nitride interests was created - Advanced Materials Engineering Ltd. (AME). The consortium comprised five organisations - Doulton, Clarke Chapman - John Thompson Ltd., Ransome-Hoffman-Pollard, British Leyland, and the NRDC itself - with a registered capital of £400,000 "to start commercial production" of silicon nitride. British Leyland joined the consortium because of their keen interest in gas turbine developments - research with gas turbine cars at the Rover plant had been going on since the early 1950's. For them, silicon nitride offered the chance of being the new material for not only stator blades but also in a unique form of heat exchanger (see Figure C4). However, at the formation stage, Leyland had only an 11% share in the company, and no-one was nominated from them for a senior management post at AME. The senior management team of AME at that time comprised of: Chairman, Dr. Bard (NRDC); Managing Director, Dr. Stoddard (Clarke-Chapman); Production, Mr. Lindop (Doulton); Marketing, Mr. Egenolf (Hoffman); and Development, Mr. Graham (Clarke-Champan).

The new company had the backing from each parent company; the techniques and know-how which each had built up over the years, including process patents; as well as further technical backing from Government laboratories, notably the Admiralty Materials Laboratory and the ceramics research division at the Atomic Energy Research Establishment, Harwell. It is worth noting that since the consortium's conception, most of the basic research associated with silicon nitride has been carried out by AML, AERE, Leeds University and the British Ceramics Research Association, and not by Advanced Materials Engineering. AME has been used primarily as the production and sales base for silicon nitride, and to begin with, this was based at Doulton's site at Stone.

C3.3 BSA Run the Lone Wolf

BSA, believing that they had established a technical lead over the other NRDC licencees, decided they could afford to stay out of any consortium as far as silicon nitride was concerned. Indeed, at about the time AME was being formed, BSA were studying (3rd July 1970) the results of a market

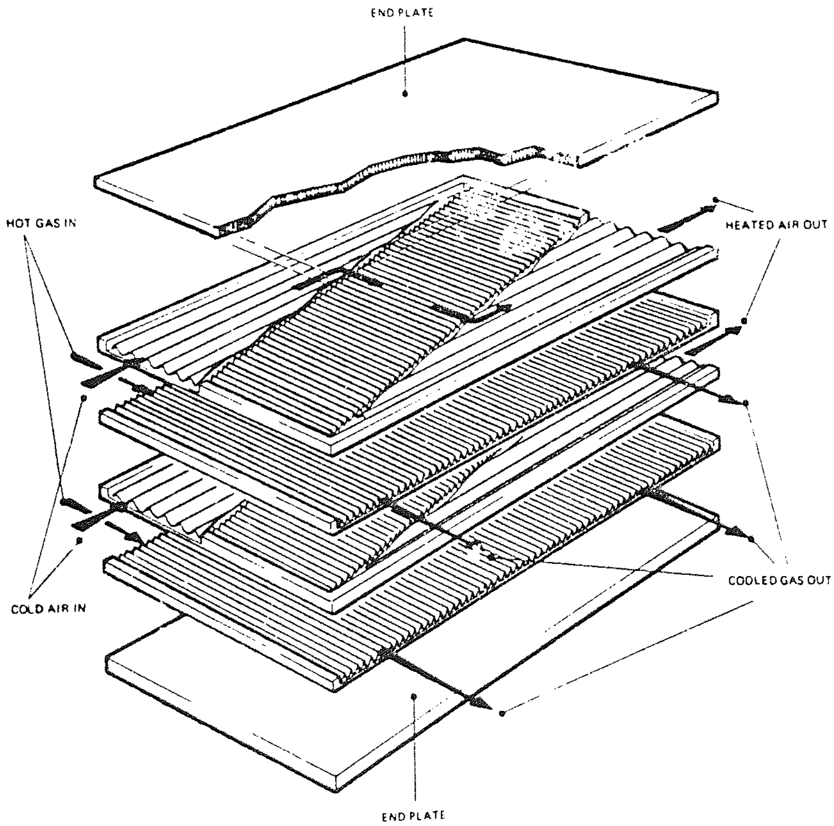

Figure C.4. Silicon nitride heat exchanger survey that they had commissioned. Marketing and Economic Research Ltd.(MER) had been given the task of assessing the potential market for silicon nitride. For BSA were now considering the consequences of making investment decisions should they go for commercial production of silicon nitride. The survey (17) was commissioned to:

"(i) discover the likely size of the market over the next 5-10 years or

(ii) discover if BSA could sell £150,000 worth of silicon nitride components within two years of plant commissioned and £500,000 within four or five years, and

(iii) discover industries and applications likely for silicon nitride

(iv) discover likely customers to whom BSA would be selling their requirements."

Of thirty five possible applications suggested, sixteen were looked at in detail, and of these seven were ruled out either for technical or commercial reasons. The most promising application appeared to be wire-drawing dies. The other eight applications considered to have "probable" or "possible" potential were telemetal pots (for ladling molten metal), Buhler type molten metal pump, low pressure stalk tubes, induction furnace tubes, radiant tubes, induction jigs, Wankel apex seals, and mechanical seals.

In assessing the market for silicon nitride, MER looked at the image that the material and its suppliers had gained for itself over the years. The conclusions they came to were not very promising. Too often, laboratory tests showed promise, but there were frequent disappointments when it came to commercial applications. The users of silicon nitride felt that the suppliers were not convinced of the commercial potential. A possible symptom of this is shown by the silicon nitride manufacturers being slow to follow up any trials that were arranged. Further, the users felt too little promotion and advertising had been given to the material. And finally, as has already been indicated, the NRDC were disappointed at the rate of commercial exploitation of the material.

Nevertheless BSA regarded the report favourably (18) and would no doubt have started to push silicon nitride harder. Unfortunately, the financial position within the company as a whole was becoming quite tight by then (1971-2). Even though new kilns were put in, the silicon nitride side of the business never got a chance to grow, for in March 1973, the company collapsed (19). Part of the company (those parts with motorbike interests) were rescued by the government in June (20) but the silicon nitride interests along with the rest of the company was sold off. Associated Engineering Developments at Rugby bought the new kilns and other silicon nitride assets from BSA. They had for some time been doing research for the Ministry of Defence, and obviously felt that reaction bonded silicon nitride could play a part in their developments for such things as pistons for diesel engines and cooling systems.

Meanwhile, Lucas and Plessey continued to develop their hot-pressed silicon nitride interests. After all, they had no ties with the NRDC, not needing any of their licences for the hot-pressed material. Indeed, Lucas and Plessey were leading the field in this area and were patenting new processes regularly (for example, refs. 15, 21, 22, 23). Plessey were developing both hot-pressed and reaction bonded silicon nitride for their Solent gas turbine which was sponsored by the government. Lucas also continued their developments and in 1971 they sold an exclusive licence to the Norton Company in the USA and by 1973 Norton were claiming (24) to have perfected a material which could compete with high-performance bearing steels. At the same time, though, they were counselling caution over (i) the brittleness of the material - "the material should not be used in applications involving high impact loads, and care is required in handling and installation" - and (ii) problems that thermal expansion might cause - "because its thermal expansivity is much lower than that of metals, innovative design approaches are required for mounting complete silicon nitride bearings on metal shafts". Providing such problems could be overcome, a very useful bearing would accrue. To illustrate the development expenditure which the Americans were prepared to put into new material developments, it should be mentioned that Nortons were reportedly (25) taking part in $10·5 million contract with the US Department of Defense Advanced Research Project Agency. This was for a five year programme aimed at developing ceramic components for both large and small gas turbines.

C3.4 The Ups and Downs of AME

At its conception in 1970, AME was based at the Doulton site in Stone. But in January 1973, they moved to a new green field site at Gateshead. A 25,000 sq. ft. factory costing in the region of £250,000 opened with twenty-five people being employed initially. At this stage, prospects for AME were still looking up. Their marketing strategy looked sound enough. They did not regard themselves as finished product manufacturers, but were prepared to sell silicon nitride powder as a raw material. Two of their largest buyers were probably Rolly Royce (1971) and Dunlop, who bought the material as an additive for other materials. As far as products went, they were looking to substitute silicon nitride for materials that were causing problems. This in itself brought problems though, for the customer was not always fully open about the application he had in mind. On several occasions this led to a silicon nitride component being produced and it failing very rapidly because the coustomer did not say "it will be subject to a slight shock loading", or "it will be running mounted to a hot steel assembly and liable to encounter thermal stress problems. Such problems did not give silicon nitride a good image when the user encountered snags with the product. Following on with the marketing strategy, though, it was AME's policy (26) to publish information about the material and successful applications either by article or advertisement in journals they thought appropriate. For instance, The Engineer and Materials Engineering for general engineering products, Welding Journal for heat treatment and so on.

Two full-time salesmen were employed to follow up all enquiries that might arise from such publicity. Attending exhibitions also became one of their publicity means, not just in the UK either. They sold silicon nitride abroad quite strongly, for instance, when they attended the Washington Gas Turbine Exhibition and Conference in 1973. The response to this was "fantastic" according to John Egenolf, their marketing director (27), AME being the only manufacturer showing silicon nitride. Throughout 1973 considerable publicity was given to silicon nitride, no doubt with the aim of renewing the engineering profession's interest in silicon nitride and acquainting them with the fact that AME were now the main producers in the UK. Free samples of silicon nitride were made available to any interested enquirer, until it was realised that they were spending more time and money on making samples and not getting a lot of business in return. Some samples are now sold to prospective customers. Much of this publicity appears to have taken place through The Engineer (27, 28,29, 30), probably due to the fact that Norman Parr was editorial consultant. For example, the feature on the heat exchanger (28) (see Fig. C4) being developed for British Leyland mainly through the development efforts at Harwell brought an immediate reaction from a competitor - Cornings. The reaction came in the form of another feature a few weeks later (29) and gave the impression that Corning were at last wondering whether they should have been pursuing a silicon nitride-type heat exchanger rather than their own glass ceramic. Whether such speculative features were good for either material is open to debate.

The silicon nitride industry as a whole, though, showed no real sign of picking itself up and going places. Not that any of the other complex ceramics like the sialons, fibre-reinforced ceramics or silicon carbides appeared to be having any more success. There were still too many companies - AME, Lucas, Plessey, Associated Engineering Development - doing their own thing with silicon nitride with no real programme to work at. It had been suggested (27) that the British government, like the Americans, might play a more decisive role and get a development programme for a ceramic gas turbine under way. This has never happened.

AME then suffered the loss of one of its best potential outlets for silicon nitride when the gas turbine programme was cut at British Leyland in mid 1974. British Leyland pulled out of the AME consortium and sold their gas turbine interests to Lucas. Between then and 1976 other major developments occurred. AME linked first with Rosenthal of Germany - to try and exploit silicon nitride in some of their energy saving programmes - and secondly with KBI (Kawecki, Berylco Industries) - to try and secure a footing in the American market. Whilst these organisations were joining the consortium Ransome Hoffman and Pollard were pulling out. All these comings and goings brought management changes to the AME set-up and no doubt altered policies on how silicon nitride should be exploited.

Throughout all this, one of the few products that seems to have been a success is the welding nozzle. This is a component which has successfully ousted alumina even though the silicon nitride component costs three times as much as the alumina. Its great advantage in thermal shock resistance gave it a life some thirty times greater than alumina. Such a small component though, however successful, technically, did not bring AME the financial rewards they were after. They were still seeking access into the gas turbine field.

Then shortly after Lucas had sold a licence to AME for their hot pressed silicon nitride technology in early 1976 a successful breakthrough was claimed in January of 1977 (31). Ricardo, the Sussex based engine research and development firm had run a silicon nitride rotor disc at temperatures of 1275-1325^{o}C. And the rotor disc was made by AME. To say that this heralded the era for the ceramic gas turbine would be premature, particularly when just six months later (June 1977) AME was brought near to bankruptcy and saved only at the eleventh hour by Allied Insulators (32).

Allied Insulators had previously bought Plessey's ceramic interests (except the silicon nitride interests) for £1 million. This was no doubt to improve their position with high tension insulants and the acquisition of AME must have strengthened this position further. The demise of AME must have been particularly galling for the management - the more so for the fact that in the same month they were claiming (33) wider industrial usage of silicon nitride for such things as machine cutting tool tips, wire drawing dies, thermocouple sheaths, riser stalk tubes for the low pressure die-casting of aluminium, and welding nozzle. With this latest upheaval, Doulton and Clarke-Chapman took the opportunity of pulling out of the venture and the shareholding was left 75% Allied Insulators, 15% KBI and 10% NRDC. Such ups and downs leave one feeling rather wry about an earlier NRDC statement (34) on the development of silicon nitride with AME when they said in 1971, "The initial market open to the company is limited and some time may elapse before successful applications are proven". They have at least been proved right in that.

C4 Discussion and Conclusions

Developments with silicon nitride started well over twenty years ago and yet there are few who would say that it has had a successful innovation. The obvious thing to say about it is that it has been slow - slow in finding a "bread and butter" type use. Most successful innovations are adopted quite rapidly when there is a need for that particular innovation by the user. Certainly silicon nitride cannot be faulted for not having that need. If the premise that thermodynamic processes work more efficiently as temperature increases is accepted, then it must also be accepted that there is a need for materials capable of operating at these high temperatures. The Admiralty Materials Laboratory stated quite categorically that they were looking for a new high temperature material which could help in applications such as the gas turbine, rocket propulsion and industrial fields. That was in the 1950's, and after evaluating many materials, silicon nitride was chosen as the material with the best potential of succeeding in such applications. So there was a need, in particular for gas turbine blades and discs operating at temperatures above 1200°C. Most innovations succeed when there is need, so why not silicon nitride?

In trying to answer such a question, perhaps one should look at the factors that affect the acceptance of new materials. Sambell and Davidge (35) have suggested that for a material to be accepted it must:

- have adequate properties
- have suitable fabrication techniques
- be economically viable
- pass various political and social criteria (for example, state of raw material resources, environmental effects, energy requirements).

In 1974, Sambell and Davidge believed that silicon nitride met most of these factors, when compared with metallic superalloys in similar applications - bar one. And that was the acceptance by engineers of the use of a brittle material. They believed engineers had a built in prejudice against such brittle materials and that metals that had built in safety factors would win. All the other factors - suitable fabrication techniques; economically viable; acceptance that silicon and nitrogen were plentiful compared to, say, competing nickel alloys; lower energy requirements for making similar products from silicon nitride rather than nickel alloys; more acceptable environmentally than nickel alloys in terms of conservation of land - were met by silicon nitride. The problem they claimed, rested with the education of engineers - getting them to accept new materials such as silicon nitride by presenting the data in a new way: statistical variations for strength/creep strength, strength-probability-time (SPT) diagrams and so on. What they say may well be right - certainly the design information presented to the engineers in the late 1950's, early 1960's was limited: avoid stress concentrations by sharp corners and abrupt changes in section: take account of the low coefficient in expansion and room temperature brittleness: design for compression loading rather than tension if possible: allow for difference of coefficient of thermal expansion between mating components of different materials (2, 3, 4, 5). All new materials go through this phase, wherein the properties of the material are being discovered by the materials technologist over a number of years. How such a phase can be shortened is debatable. What is certain is that a material may behave beautifully under laboratory conditions, but when production is scaled up or the product put into actual service conditions, other unforeseen factors come into play. And if the product fails, as silicon nitride has on many occasions, then the material rapidly gains a bad image which is difficult to restore, even when the problem has been overcome.

There is no doubt that the brittleness of silicon nitride has contributed to its poor acceptance as an engineering material. And the claims of the manufacturers have, in some instances, preceeded the success of a given product. Too often it has been stated that silicon nitride has been successfully used in gas turbines only to be publicly refuted a little later. This has again produced a bad image for the material and its manufacturers. This in itself has contributed to the slow development of the material and the general belief that other uses will not accrue until the successful development of gas turbine parts has arisen. For example, Duckworth and Flint (25) have stated "It would.....appear that the main potential commercial applications for silicon nitride are in the manufacture of gas turbine parts, and until these are seen to be operating satisfactorily in full commercial operation it is unlikely that other potentially commercial developments will be actively pursued". In stating this, it is noticeable that two companies at least believed the potential lay outside gas t.rbines, namely AME and BSA. The market report prepared for BSA made no mention of the possible returns in this area. BSA perhaps believed they could have made a commercial success of silicon nitride without the need for gas turbines. They unfortunately were never able to prove that whereas AME has yet to show the full commercial promise outside this area.

Other scientists - Dr. Godfrey at AML for instance - believe that too much emphasis has been placed on the development of silicon nitride parts for gas turbines. He contests (36, 37) that the feasibility of silicon nitride components in internal combustion engines is well proven; indeed he has successfully demonstrated their use in a small petrol engine and two diesel engines rated at 690 and 1580 MPa brake mean effective pressure. The development of ceramics for gas turbines, particularly aircraft turbines, is manifestly more difficult than for internal combustion engines where the ability to resist ingested debris is pre-requisite. The development of silicon nitride parts for internal combustion engines is perhaps nearer than for gas turbines for this very reason and also because of the growing concern to use hydrocarbon fuels more efficiently.

In trying to see why silicon nitride has been slow to develop, it might be prudent to ask if the right information channels were used to promote such a material. Are journals such as Research Applied to Industry, the Refractories Journal, Powder Metallurgy and the NRDC Bulletin the best media for promoting a new, advanced technology material to engineering professions as a whole? Such were the journals used in the 1950's and 1960's. Later, once AME had been formed, they favoured more widely read engineering journals such as The Engineer and Design Engineering. But has silicon nitride received too much or too little publicity? The answer is not easy. During the late 1950's and early 1960's over-optimistic claims were being made about the success of the material. During most of the 1960's publicity was kept at a very low key. One eminent scientist, Dr. Godfrey, who has been close to the development of silicon nitride, was told by a member of the silicon nitride industry that "if we had advertised (at that stage) we would have been overwhelmed" (38). And yet the BSA report seems to be critical of the promotion efforts "The methods used by the present licensees (BSA, Doulton, Clarke-Chapman, Hoffman) to promote silicon nitride, seem mainly confined to press releases and articles by technical staff in trade journals. There appear to have been few paid space advertisements". Such criticism could not, however, be levelled at AME during the 1970's; their promotion efforts have, if anything, resorted to the original over-optimism which so frequently disappointed the user.

The NRDC being the guardian of much of Britain's silicon interests have quite rightly felt disappointed with the slow growth of the material commercially. It was they who instigated the formation of the AME consortium from three or four of their licence holders. In principle, this was probably the right thing to do, bringing together the breadth of experience of these three companies.

However, the management of AME appears to have changed with every change of the AME shareholding. For instance, the post of Managing Director changed at least three times between 1970 and 1977. No doubt the changing management structure also brought changing policies as to the best exploitation of silicon nitride. One is left wondering whether there was anyone left to champion new product ideas through to fruition.

A final question to ask about the poor development of silicon nitride is: could a more successful result have come from AME if more had been invested in the programme. By 1973, £750,000 had been ploughed into developments at AME, whereas the Americans were reportedly devoting \$10·5 million on a government contract for developing gas turbine components in silicon nitride. This history does not intend to answer such a question, although there is an impressive body of experience being accumulated in the USA as indicated at the 1978 Gas Turbine Conference (39). It is apparent from this and previously published work that the Americans have had a mapped-out defence project in which silicon nitride has been closely involved. The British on the other hand have at least three different companies working on their own, going in their own direction without any real goals to aim at.

In conclusion then, silicon nitride is an advanced technology material which meets most of the factors for acceptance, except one fundamental physical property that engineers abhor - brittleness. With further education, this problem might be overcome. The material had an initial "need", for use in gas turbines; but too much publicity too soon gave silicon nitride a bad image before it had proved itself. The AME consortium has not been able to overcome the problem; and the engineering profession is left waiting for the day when either AME, Lucas or Associated Engineering Developments makes the successful breakthrough into gas turbines or perhaps internal combustion engines before the material will really be accepted as a general engineering material.

C5 Acknowledgements

The author would like to thank H. Child of Aston University, and personnel from AML for their help in compiling this history.

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APPENDIX D

Titanium Case History

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D(ii) Information Sources for Titanium

As with the other materials studied, the method for finding relevant information sources was basically the same - contact relevant institutions, journals, and known contacts within the University or Dunlop. Table D(i) - 'material source indicator' - shows the result of this search exercise. In the course of this search some interesting discoveries about the initial contacts were made.

The organisations initially contacted were:

*British Non Ferrous Metals Research Association.

*Institute of Metals.

*Institute of Metallurgists.

*Journal of Historical Metallurgy.

Apparently the British Non Ferrous Metals Research Association deals with Aluminium, copper and their alloys - not titanium, which seems strange for an organisation claiming to deal with non ferrous metals. The contact there was even unsure of the manufacturer in the UK until he had a word with his colleagues who recommended contacting New Metals Division of IMI.

Things were not much better at the Institute of Metals, they did not know IMI manufactured titanium, or whether there was much work being done with the metal in the UK. They were, however able to recommend a reference - "Titanium Science and Technology", 1972 edited by Jaffee which is the proceedings from an international conference at which IMI were much in evidence!

One seemed to go from bad to worse in contacting these organisations - the Institute of Metallurgists knew nothing about

Table D(i) MATERIAL SOURCE INDICATOR

Material: Titanium

RECOMMENDED CONTACTS \ CONTACTS	Prof. W. Alexander, Aston University	I. H. Lowe, ICI	British Non Ferrous Metals Res. Assoc.	Inst. of Metals	Inst. of Metallurgists	Journal of Historical Metallurgy	A. Carter, IMI	J. Langrish (author Wealth from Knowledge)	H. Child, ex Jessop Saville	J. Mountford, IMI
Prof.W.Alexander Aston University										
I. H. Lowe, ICI										
Brit. Non Ferrous Metals Res. Assoc.										
Inst. of Metals					x					
Inst. of Metallurgists										
Journal of Historical Metallurgy										
IMI	x		x					x	x	
Dr. M.McQuillan, IMI	x							x		
A. Carter, IMI	x									
J.S. Millar, IMI		x								
H. Child - ex Jessop Saville	x									
J. Mountford, IMI							x			
P. Morten, IMI									x	
M. Clapham, F.B.I.									x	
N.D.J.Compton, ICI, Gen. Chem. Div.										x

titanium's development and recommended contacting the Institute of Metals!

The Journal of Historical Metallurgy is interested in the technical history only of various metals and processes, and not with associated developments of uses for materials. In any event, titanium it appears is too recent a material to fit in a 'historical' journal especially if it is the business history of such a material that one is interested in.

As can be seen, the larger organisations were not exactly helpful or in some cases even in a position to be helpful. This left the only known contact within the university of Professor W. Alexander who more than made up for the lack of contacts and information provided by the other concerns. It was he who opened most of the gates for the author to investigate titanium's history, including persons in IMI and Dr. H. Child, an ex Jessop Saville man directly concerned with their titanium development.

D1 Introduction

Titanium is considered to be one of the 'new' metals and indeed it is - but its ancestory extends back to the 18th century. The oxide of titanium was first isolated by the Reverend William Gregor (1) in 1790 in Cornwall. He named it mennaccanite after the bay in which he found the black sand - Mennaccan (six miles south of Falmouth). Five years later a German chemist, Martin Heinrich Klaproth noticed the similarity of his oxide extracted from rutile in Hungary to that of mennaccanite. He acknowledged Gregor's priority and gave his discovery the temporary name 'titanium' taken from Greek mythology. The name titanium however appears to have been more acceptable than mennaccanite, and has lasted the test of time.

Even though titanium is one of the earth's most abundant elements, and the fourth most abundant metal after aluminium, iron and magnesium - it was not isolated as a metal until nearly a century after Gregor's initial discovery. Indeed all the early pioneer work was commercially unsuccessful, though much credit must go to those early pioneers - Nilson & Petterson (1880's), M. A. Hunter (1900-1910), Lely & Hamburger (1914) and Van Arkel and deBoer (1925). It was not until June of 1937 that W. J. Kroll in Luxembourg showed that pure titanium, which was ductile when cold, could be produced.

The Kroll process was later to become world famous for the production of titanium, although it took several years to convince people of its potential. With the coming of the German invasion Kroll fled from Luxembourg to the USA in 1940. The US Bureau of Mines started to look at titanium in 1938 (after Kroll's first visit) and began investigations of the Kroll process in late 1940. And by 1944 the first titanium plant was built in Boulder City, Nevada producing 100 pounds a week - the incentive coming largely from the 2nd World War, with the search for high strength to weight ratio materials.

A new concept in aircraft propulsion arrived when in 1941 Frank Whittle's little 855 lb thrust turbo jet took off. To a very large extent it was the jet engine which nurtured the titanium industry in the USA and started the British R & D effort at the Royal Aircraft Establishment in the early 1940's.

By the end of the forties, people were really getting excited about the potential of titanium. It was hailed as a great metal and "on the threshold of a brilliant career" filling the gap between iron and aluminium to form a "complimentary trio" - "Iron and steels are strong and cheap, but not light. Aluminium alloys are light and cheap but not particularly strong. Titanium alloys will be light and strong but not cheap". So proclaimed the journal Scientific American (2).

It was against this background that British industry first became involved with titanium, and why not, when the uses being suggested looked diverse and lucrative -

Aircraft
Instrument precision parts
Spindles in cotton mills
Food processing plant
Prosthetic devices
Battle ships
Automobile pistons
Handles for aluminium pans and cooking utensils
Sports equipment - tennis rackets
Springs
Tool mountings
Pen points, styluses
Very stable high electrical resistance glass

D2 IMI's Involvement

The story really begins with Imperial Chemical Industries Limited as early as 1945 when interest in titanium was shown by General Chemicals Division at Widnes (now Mond Division). They had a history of producing not only established chemicals but also unusual metals including sodium, cerium and uranium. Their natural interest in titanium continued to grow after visits to the US Bureau of Mines and other interested parties in 1946 and 1947 - and as a result of publicity in America around 1947, they received numerous enquiries for trial amounts of titanium from aircraft makers, and the major engine manufacturers in particular. An initial costing for the production of 100 lbs of titanium sponge* by the Kroll process was instigated in March 1948, in which ICI Metals Division (now Imperial Metal Industries Limited) were to study the conversion of sponge to a wrought metal and its properties and end uses.

With things looking optimistic at this stage (through large American effort and publicity, for example refs 2 and 3) development work escalated rapidly, with General Chemicals Division looking at three sponge production processes - using magnesium, iodide and later sodium. Metals Division pushed ahead with studies into melting and fabricating techniques and also supplied small development quantities to potential customers. They were by early 1951 being supplied with sponge from the first Kroll plant commissioned by General Chemicals Division (12 tons/year) in their Research

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* name given to product obtained from the reduction of titanium tetrachloride and before its conversion into ingot form. For technical details of sponge production see page 549.

Department. Because of the increasing demands from Metals Division a second plant of 100 tons/year was built by General Chemicals Division at their Gaskell-Marsh Works (Widnes), coming into operation in 1954.

D3 The Government plays a part

ICI were just gaining experience of titanium, when in 1951 the Government made its views felt - it wanted titanium for military aircraft, and was anxious to get to full scale production (4). For ICI this must have posed a problem. General Chemicals Division were not convinced that the magnesium reduction route was the most economic and had just started investigations using sodium. No doubt ICI felt pressurised into going for the magnesium route especially when W. Kroll himself made his unkind views of the sodium process known.

However General Chemicals Division pushed ahead with their experiments using sodium, starting on a 7 lb scale at the Widnes laboratories (5). After all, they had years of experience with the manufacture of sodium and knew all about its nasty problems of catching fire readily and its explosive reaction with water. It took a year to sort out the problem of getting pure titanium without any oxygen embrittlement which first plagued them, but eventually the contamination sources were tracked down. The 7 lb plant soon dispelled the technical objections to sodium, at least on the small scale - and a relatively cheap process for the separation of sodium chloride and titanium looked feasible.

With this first success behind them General Chemical Division went on to build a 100 lb reactor in mid 1952 and by the end of 1952 a 900 lb reactor.

Whether by good luck or judgement - or both, no great snags were met during this year, and when in 1953 the Divisional Board had to decide on a commercial plant, they accepted the Research Department's recommendation and went for the sodium process. The decision to go ahead with a commercial plant was taken whilst there was still an air of optimism about the demand for titanium. Because of the Government's positive interest in a titanium industry discussion with them on the national requirements for titanium were held. This resulted in a contract being signed on the 6th August, 1953 with an undertaking by the Government to purchase up to 75% of ICI's crude titanium for up to four years - provided the full scale plant was operational within two years. The decision to invest in a large commercial plant cannot have been that difficult when the purchase of the product was guaranteed for at least five years at a presumably attractive price of 21 shillings per pound. And by 1955, production had started on a 1500 ton per annum sodium reduction plant, at the Bain Works (Wilton) of General Chemicals Division.

D4 Developments within Metals Division (1948-1957)

The process innovations which took place in Metals Division in the melting and fabricating were no less important than the sodium process developments at General Chemicals Division. Granular titanium*, packed in drums, arrived at the Witton

* Production methods for titanium sponge are described on Page 549.

plant of Metals Division to be made into wrought and semi-fabricated products for sale.

Research into titanium fabrication began during 1948 and in 1950 the first titanium ingot of 5 lbs was produced (6). To state the process as - melt titanium powder - produce ingot - shape it - is to state the process in its simplest terms without regard for the technical intricacy involved. For instance, molten titanium will pick up virtually any impurity, and has therefore to be melted either in a vacuum or under an inert gas such as argon or helium, and in a container which will not contaminate the metal. Metals Division first went through a phase of melting titanium using an electric arc with graphite electrodes in water cooled copper cruicibles - and then to a method using a compacted titanium electrode, which is progressively lowered as the electrode melts and the ingot builds up.

Having an ingot, one is still only part way to the finished product - you have yet to roll the metal to its finished form - sheet, plate, bar, rod, etc.. This again posed problems, for the metal readily oxidises at the hot rolling temperatures and great care is needed (6). Initially the metal was rolled on mills for copper and brass but with titanium tying up such mills for up to 20 times as long as copper or brass, it became apparent that titanium would require its own mill. With the advent of the government's contract the ICI Board sanctioned a new titanium sheet and rod rolling mill at Waunarlwydd in Swansea - becoming operational in 1957.

To introduce a new metal such as titanium required a lot of development work - studying the melting, rolling, extruding and drawing techniques, as well as looking at the properties of the metal and its alloys*. From the very first, Metals Division involved the customer (airframe and engine manufacturers, in particular Rolls Royce) to see how he would use titanium and try to meet the special requirements of the user. This involved learning how the wrought metal could best be machined, welded and otherwise fabricated into its final shape.

So it was, that by 1954 a 150 ton per annum pilot melting plant had been completed and a year later a 1500 ton per annum production plant.

D5 <u>ICI Policy</u>

If the search for uses for titanium was not on before, it certainly got underway with the production facilities in full swing. Literature detailing the metal's impressive properties was published with the advent of commercial wrought titanium - especially its high strength to weight ratio and corrosion resistance. Even with its impressive properties, titanium suffered limitations - strength decreasing rapidly above 500^{o}C, but probably above all else price was its main limitation, for example sheet metal was costing over £7 per pound, and tube £16 per pound. The poor creep strength could have been a serious blow to titanium's development, after all, the early forecasts had all made mention of the potential for use in the high pressure end of turbine

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* Structure and properties of titanium described on Page 552.

engines, requiring good creep properties. Even so it was ICI's intended policy - according to Dr. W. H. G. Lake, Director of Titanium Production, that with "a limited output of wrought products, it was invevitable that the development and sales effort should in the first instance be directed to the aircraft industry, which was able to justify the relatively high cost of titanium in the interests of weight saving" (7). Apart from Rolls Royce, two other companies investigated titanium very early on - deHavilland and Bristol Aircraft. The aim initially was a straight replacement for heavier metals, realising though that full benefit would not come until components were specifically designed for titanium.

How did ICI let the "world" know of titanium and its potential? The answer seems to be that very early on they decided on as wide a technical coverage as possible. Apart from well versed technical sales people visiting potential customers, a very large effort was put into the production of technical literature, the attendance of exhibitions and presenting papers whenever the opportunity arose. As early as September 1955 they had arranged a stand at the Engineering Marine & Welding Exhibition and published 'Wrought Titanium' - a practical handbook for all those who might wish to make use of the metal. Advertisements in technical journals, pushing a particular property of titanium also appeared quite regularly - typical of such advertisements are those shown in fig. D.1 taken from Aircraft Production during 1957. An abstracting service was also created by ICI in 1955 when they introduced 'Titanium Abstracts' -

Table D.2 Applications of IMI titanium in chemical and general engineering

Industry	Environment	Alloy used	Types of plant	Reason for using titanium
Chemical	Organic acids; chlorides/bromides; chlorine compounds; chlorinated	CP Ti IMI 260 IMI 261	Loose lined, solid and clad plate vessels; tubular and	Excellent corrosion resistance in a wide variety of oxidizing

Engineering	Various	CP Ti Ti alloys	Various.	Good specific strength

(Table courtesy of IMI)

apparently as a promotion medium. Clearly for anyone interested in titanium's development it was a useful reference source detailing developments under the following headings:

- Properties and Characteristics
- Alloys
- Metal Fabrication
- Metal Treatment and Workshop Practice
- Uses and Applications
- Titanium Compounds
- General
- Bibliography

D6 The Developing Titanium Industry Outside ICI

D6.1 Europe

Undoubtedly the production of titanium sponge in the early 1950's rested solely with ICI throughout the whole of Western Europe. With the advent of the government's contract for titanium sponge it virtually ruled out any other supplier in the UK. However one company did try to get in on the titanium sponge scene, and that was McKechnie Bros who had a pilot Kroll plant operating at Widnes in Lancashire by 1955. Recognising that all defence requirements would be met by ICI and that the commercial market in the UK at that time was small, they concentrated their activities to small overseas markets. None too successfully it would appear, because by December 1957 they had been forced to close their plant.

The growth of titanium fabricators was far more impressive, with five companies, other than ICI engaged in the industry as early as June 1955 (8). Of these William Jessop and Sons

Ltd provided most competition for Metals Division, ICI - not only producing mill products but also fabricating their own products. And to begin with they were extremely well positioned to offer this competition, particularly for the aircraft industry. They had a history of supplying structural steels to the aero engine manufacturers in particular, and to defend this position they had to join the titanium industry. So it was, that in 1951 Jessops had become interested in titanium; the driving force behind their interest being Mr. G. T. Harris, the Research Director. Their experience of alloy development stood them in good stead and as early as 1953 it is claimed, they had produced an alloy* far stronger than any developed in the USA or ICI. During 1954, both Jessop and ICI bought the technical know-how of furnace design and melting technology from Titanium Metals Corporation of America - in particular compacted titanium electrode technology. This had the effect of putting them on equal terms with Metals Division of ICI who had experienced difficulties with impurities in the titanium from their electrode process - they had, at about the same time switched to a compacted titanium electrode method. Jessop's three furnaces, capable of producing 300 tons per annum of titanium came into operation during the middle of 1956 at Sheffield.

Competition between Jessop-Saville and Metals Division, ICI remained intense until 1967, when Jessop's titanium interests were bought by the then IMI. The fortunes of both companies in the market development will be discussed later.

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* Hylite 50 was patented in 1954

The titanium industry in Europe, other than the UK has rested mainly with four companies - two in Germany (Krupp and Contimet - then 50% Thyssen and 50% Titanium Metals Corporation of America, and now 100% Thyssen Edelstahlwerke), one in France (Péchiney and Ugine - now merged - Péchiney Ugine Kuhlmann Group), and one in Sweden (Avesta) (9).

D6.2 United States

The USA has from the very start been the undisputed leader of the titanium industry. Growth of the industry during the late 1940's and early 50's proceeded at an incredible rate, and was almost entirely due to colossal Federal support (for sponge production only). By 1955 the US Government had committments with five major companies with contracts worth $186m for the construction of plant and purchase of output.

Until the middle of 1955, all the titanium ouput was committed to military uses, but by August 1955 it was reported that 10% of production would be available for civilian use. Having established a titanium industry, the US Government then dealt it a severe blow when in 1957 military strategy changed from manned aircraft to missiles. Military projects collapsed and with it the titanium market - and as we shall see with the UK market, manufacturers were forced to look for other users. The impressive growth of titanium in the USA is illustrated in figure D.2 and although it fluctuates, is apparently ever upwards.

During 1955 the US Government took another measure, obviously aimed at expanding titanium's usage when they established the Titanium Metallurgical Laboratory at the Battelle Memorial

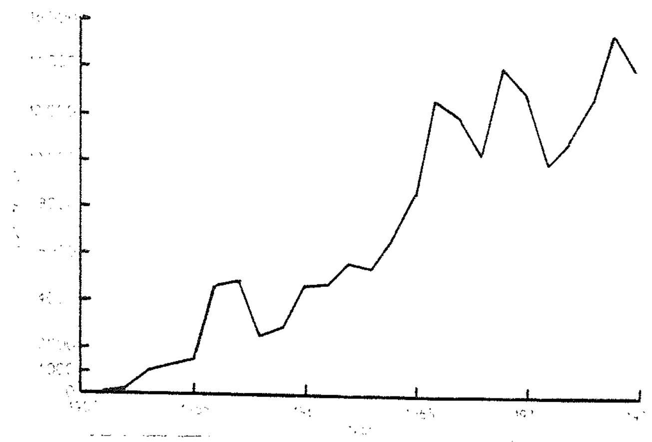

Fig. D.2 United States titanium sales (mill products).
(Courtesy of IMI)

Institute, Ohio. The aim of the organisation was to:-

- supply advisory services to constructors under the government's titanium programme.
- advise government agencies
- collect and disseminate information on titanium technology.

The slump of 1957 forced several companies out of the titanium industry until by the mid 1960's there were only two sponge producers. Thus the grandeur of the titanium industry was tarnished and as T. W. Farthing suggests 'the main casualty ..... was the credibility of the industry' (10). Whether this lack of credibility in the industry rubbed off in the UK is debatable, but to a certain extent the fortunes of the UK industry have followed in the footsteps of the USA.

D6.3 Japan and USSR

The growth of a titanium industry (starting in 1954) in Japan seems quite incredible when one realises they have no defence committments - what the industry was built round in the USA and UK! Why then should they produce titanium? The answer probably lies with the aid programme between the USA and Japan after the Second World War with America suggesting possible growth industries. In any event the Japanese industry was expanding rapidly with plans in early 1956 to increase production from its three plants to 2600 tons per annum - with over 90% of the output to the USA.

The Japanese industry continued to grow, with exports of titanium going not only to America but also Germany and the UK - providing ICI's decision to close down their extraction plant between 1960 and 1964 - when there was only a low demand and the Government stockpile was being run down. Capacity within Japan now stands at about 11000 tonnes for sponge (1974 figures).

Development of the titanium industry in the USSR can have been no less impressive than the rest of the world, though details of such development are hard to come by. Their industry started in 1950 following the Kroll route and their capacity for sponge production is currently estimated at 40,000 tonnes per annum from three main plants.

The Russian market is effectively closed to imports from the West but their records of exports to the West has been quite remarkable - for example over 4000 tonnes to the USA in 1974. According to T. W. Farthing, they are currently supplying the free world with about 20% of its consumption - no good thing when existing facilities are under utilized especially as some people consider the metal to be of strategic importance.

D7 Development of Markets in the UK

Unlike the sponge production at General Chemicals Division, Metals Division had no Government aid - all the development work was privately funded. Indeed by 1958 ICI were reported (11) to have spent £2m on research with about £500,000 being spent annually on further development. Whilst in plant and working capital about £9m had been invested by the time the new Welsh rolling mill was completed in early 1958.

The air of optimism shown by most people in the titanium industry was not shared by at least one of ICI's personnel. W. Alexander had been sent by ICI to the USA between 1955 and 1958 to try and sell the sodium process to the Americans, in preference to following the Kroll route. Whilst there, he worked closely with the Battelle Memorial Institute's new titanium metallurgical laboratory and other companies associted with titanium. From what he saw there he gathered that ICI were behind the Americans in titanium development and that they (the Americans) were at last realising that it was not the wonder material that everybody thought - particularly as the creep properties were lacking, many of the early alloys suffered from hydrogen embrittlement and prices were far too high for normal usage such as in transport. A report to this effect was sent back to ICI but far from deterring them it seems to have strengthened their resolve to continue with developments.

With this background in mind developments in the aerospace and non-aerospace markets will be described.

D7.1 Aerospace

It has already been stated that ICI's initial titanium development programme was aimed at the aircraft industry - and it is interesting to note that Rolls Royce were involved almost from the start, with this development. In fact ICI did not have to search for their first user - there was one knocking at the front door.

As has been indicated the growth of the titanium sponge industry was rapid by any standards. The growth of titanium products has not been so rapid, probably because the users of the metal demanded alloys that could work at the limit of their properties from the very start. Titanium is one of the new generation metals that have been put to the high technology use first, and the low technology second.

D7.1.1 Engines and Airframes

To make the most of titanium, you have to make use of its unique properties and for the aircraft industry this meant its high strength to weight ratio. After all the optimism about titanium in the late 1940's early 1950's for use in turbines it must have come as something of a shock to find such a promising material had a low creep strength. This effectively meant that it could not be used in the high pressure end of a turbine (the hot end). Nevertheless engine manufacturers were still eager to make use of titanium in other areas, in particular, turbine blades and discs in the low pressure end.

Interestingly enough it appears to have been Rolls Royce who exploited titanium first rather than Bristol Siddeley - yet most of the defence contracts were placed with Bristol Siddeley. Bristol Siddeley were soon to catch up with any lead that Rolls Royce may have established when they started a titanium programme with William Jessop & Sons Ltd. - who had been associated with the aircraft industry for a good many years with high quality structural steels, and high temperature alloy steels for gas turbines. Early alloy development for ICI's Metals Division led directly to the introduction of the first commercial alloy, 2Al-2Mn (designated IMI 315) with Rolls Royce as compressor blades in their Avon engine. This went into service in the Comet, Canberra, Lightening, etc. (Table D.1 from T. W. Farthing's paper illustrates the alloy development and associated uses). Other alloys developed by ICI followed over the next few years, the main ones being IMI 679 (11Sn-1Mo-5Zr-2$\frac{1}{2}$Al-$\frac{1}{4}$Si) in 1958; and IMI 318 (6Al-4V) developed in the USA and adopted in the UK in 1958 has probably been the work horse of titanium alloys over the years. Jessop's were also at the forefront of alloy development and probably stole advantage over ICI with their 'Hylite' 50 (4Al-4Mo-2Sn-$\frac{1}{2}$Si) - later to be known as IMI 550: the equivalent alloy by ICI being IMI 679, which had much more tin and was consequently denser. Alloy research not only centered on creep resistant alloys for compressors - but also high strength alloys for airframe forgings - and cold formable sheet alloys for by-pass ducts and airframe skinning. Indeed IMI were later to be commended for their achievements in these areas with the Queen's Award in 1967.

The alloy developments which took place throughout the 1950's were really quite remarkable not only at ICI and Jessop's but

Table D.1 Titanium aerospace developments

		Major titanium appli-	Associated

ciature.

(Courtesy of IMI)

particularly in the USA. The search for the ultimate properties seemed to be the be all and end all - using quite expensive alloy elements.

During the 1950's things were looking particularly rosy for the titanium industry with two major aero defence projects on the drawing boards - the TSR2 (with Olympus engines) and the Harrier jump jet (with Pegasus engines); Concorde was also being developed. The Fairey delta research aircraft was breaking new grounds in March 1956 when it reached 1,132 miles per hour - pointing the way to new metals for aircraft skins. But at the same time as the Fairey delta was showing the way for possible titanium uses, ICI Metals Division had to reduce their titanium output. Demand was falling, simply due to aircraft manufacturers changing their requirements. And people other than ICI were beginning to ask whether there was another market for this metal (2, 4).

The next blow to hit the titanium industry came in April of 1957 when Duncan Sandys, Minister of Defence announced the Government's outline of future policy - in which many defence projects were cut, and missiles were favoured rather than manned aircraft. By now though, ICI had realised they must be looking for uses outside the aircraft industry - but we shall see this later. This decision by the Government rocked not only the aircraft makers but most of their suppliers as well, including the titanium suppliers ICI and William Jessop. As Dr. Lake pointed out a year later "the Defence White Paper ... administered a sharp but, by and large, salutary warning against complacency" (7). It must indeed have been a salutary warning, particularly for ICI who were committed to their expansionary programme, the Waunarlwydd rolling mill in particular.

Throughout the development of uses in the aircraft sector a policy of substitution had been adopted. For instance with the aircraft frame manufacturers, titanium was substituted for light alloy and stainless steel skins in unstressed areas on the Bristol Britannia - round the engine nacelles and in firewalls. This policy seems to have been adopted throughout the industry, for even the US Bureau of Mines were saying in 1954 that:

> "Titanium can be used in aircraft construction as a substitute for stainless steel in such non-structural parts as engine firewalls, engine mount struts, ducting and miscellaneous brackets and fittings around engines or exhaust systems." (13).

The strategy for finding new uses in the aircraft sector seems to have been one of informing the sector of developments whenever and wherever possible; with the aim of getting a feedback on developments. For ICI this meant attending exhibitions, presenting technical papers at meetings and articles in journals and giving in-house symposia. A few examples of these should illustrate their thinking:-

D7.1.2 <u>Exhibitions</u>

ICI attended the exhibition organised by the Society of British Aircraft Constructors at Farnborough annually from 1956. It should be noted that other titanium fabricators also attended regularly, including William Jessop & Sons Ltd., Chesterfield Tube Co., Accles & Pollock, G. K. N. and Marston Excelsior - one of ICI's subsidiaries. (It should not be overlooked that Marston Excelsior played an important role in the development of titanium fabrication including welding and later the fabrication of linings for chemical plant components and heat exchangers). ICI later expanded their role of attending aviation shows when they went international with the Hanover Air Show in 1962.

D7.1.3 Conferences/Symposium

ICI not only attended conferences, they also organised them - for example on 15th February, 1957. At this they presented the following papers:

- The general picture, by M. Cook
- The general commercial position, by M. J. S. Clapham
- Outline of ICI's production policy, by St. J. Elstub
- Production of wrought titanium by J. R. Crane
- Research and development activities, by N. P. Inglis
- Current problems of usage, by R. L. Preece
- The economic applications for titanium in civil air transport by H. W. Shaw

They not only reached the audience present, but far more, for wide coverage was given by several journals - Metal Bulletin, Engineer and Aircraft Engineering.

Similarly on May 4th and 5th of 1960 ICI organised a symposium at their new Waunarlwydd plant on the theme 'Titanium production methods in the aircraft industry' - again reported in many journals.

D7.1.4 Articles in Journals

The benefits and successful applications of titanium could be quite easily put over to a large audience using the journal medium. Frequently the case was argued for titanium usage rather than conventional materials - for example, an article in Aeroplane (14) of 1958 entitled "The economic value of weight saving"*. Similarly, articles that mentioned titanium in passing appear to have been encouraged - for example an article in Aircraft Production (15) of 1959 entitled "Handley Page Victor" outlines the structure of the bomber and describes some of the production process - and mentions where titanium is used.

D7.2 Non-Aerospace - Chemical, General Engineering, Medical

The development of uses for titanium outside the aerospace industry differed from that used for the aerospace industry itself. For instance there was no one knocking at ICI's door just waiting to make use of titanium's unique properties as Rolls Royce had. For ICI, it meant they had to look at titanium's properties - other than high strength to weight ratio - and find uses that would make the most of these properties. The obvious property of titanium to develop was therefore its remarkable corrosion resistance - which had been recognised and reported as far back as 1949 (2, 3) but never exploited, either in the UK or USA.

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* Author employed by G.K.N. (Midlands) Ltd.

The 1957 slump in the titanium industry caused by changes in the aircraft defence committment may have been regarded as a bad omen by ICI - but it at least reinforced their decision to look for non-aerospace uses taken in 1955. Having taken the decision to look for other uses, New Metals Division then appointed a manager responsible for non-aerospace developments - Dr. K. W. J. Bowen. Under Bowen a team with considerable experience was built up, particularly in the chemical industry. And as time went by other key personnel were appointed in the search for non-aerospace uses - nuclear (Mr. A. Carter) medical (Mr. Kay). Carter spent a considerable time actually working on site with personnel at one of the Atomic Energy Authority's stations, helping with material problems.

Both before and during the search for alternative uses of titanium a massive research effort was put into the understanding of its corrosion resistance (which is generally better than that of stainless steel). In the event most of the applications found have used commercially pure (CP) titanium with very little alloy development being necessary. There is of course one exception and that is IMI 260 - an alloy containing 0·15% palladium, developed in 1959. This was developed for use in non-oxidising acids or in reducing conditions where CP titanium was not always satisfactory. It had the remarkable effect of reducing the corrosion rate of the pure metal by as much as 1500 times.

Another property of importance, is the metals fatigue resistance. Indeed, it was shown quite early on that the fatigue limit was unaffected by corrosion - a property which not even the best stainless steels could match.

The search, therefore, for titanium applications was concentrated to begin with on the chemical industry and subsequently in more diverse areas - paper pulp and bleaching industries, metal finishing, power generation, medical and the auto industry to name but a few.

All the applications in these areas had to satisfy rigorous value - cost comparisons, and this was complicated by the cost of titanium. New Metals Division of ICI knew full well that the only way that the cost could be reduced was if the throughput could be increased. Their efforts to reduce the price of titanium were considerable - economies which scale could give were treated very seriously. Between 1956 and 1961 the price of sponge and wrought titanium products was reduced no less than six times but still sheet could cost anything between £3 and £7 per pound.

As in the aircraft industry, the very early applications for titanium were straight substitutions where other materials were failing in small and simple components - valves, springs, nozzles, etc. In many instances the new products were tested in ICI's own plant - for example at the Billingham Works of ICI (1957) stainless steel valve springs used in compressors for nitrogen and oxygen were failing regularly from stress corrosion (16). Titanium was used successfully to replace not only the springs but also the valve plates. Many such successful developments like this were patented by ICI themselves - for example, B.P. 866,482 relates to improvements made in anodising jigs and B.P. 889,147 relates to improvements in electrode structures for use in electrolytic oxidation processes.

So how did New Metals Division search for non aerospace uses of titanium? Again, the strategy seems to have been one of informing the world at every opportunity, using technical sales staff, exhibitions (static and mobile), technical papers, journals, and conferences. To illustrate this a few examples are cited.

D7.2.1 Technical Papers

The presentation of technical papers appear to have been encouraged at every possible time - such as:

"The production and properties of titanium and its alloys" by N. P. Inglis presented to the Society of Chemical Industry and the Institute of Metals, 7th November, 1955.

"Titanium Manufacture" by Dr. J. Taylor presented to the British Association for the Advancement of Science, 1956.

"Titanium" by Dr. M. Cook presented at the Tenth Annual Conference of the Australian Institute of Metals, 22nd May, 1957.

"Titanium as a material of construction in the chemical industry" by Dr. K. W. J. Bowen presented to the Birmingham Branch of the Incorporated Plant Engineers, 20th September, 1957.

"Chemical plant in titanium", by R. J. Watkins presented in the Industrial Chemist of June 1958.

D7.2.2 Exhibitions

Probably one of the earliest exhibitions attended by ICI was the Engineering, Marine and Welding Exhibition during September of 1955. ICI and their subsidiary Marston Excelsior seem to have been attending exhibitions ever since:

* Cycle and Motor Cycle Show, November 1956 - showing a titanium bicycle by Phillips Cycles Ltd., in collaboration with ICI.

* Society of Chemical Industry, Corrosion Group held an exhibition on 23rd January, 1958 at which ICI demonstrated platinum - coated titanium anodes in cathodic protection applications.

* Chemical Engineering Exhibition, June 1958 - ICI showed a titanium probe for a liquid guage, titanium mesh, a lined pressure vessel, an emulsion bottle and implements for surgical use amongst other things.

One of the most interesting exhibitions that ICI organised was a mobile exhibition designed to tour Great Britain. It was first shown at the British Industries Fair in May 1957 and subsequently toured the country, exhibiting various titanium components from $\frac{1}{4}$ inch high pressure needle valves to a four feet diameter titanium lined mild steel vessel.

As with the aircraft developments, ICI went European when they attended the Chemical Exhibitions and Congress at Frankfurt for the first time in June 1961. Marston Excelsior attended with them showing such things as heat exchangers and titanium lined steel vessels.

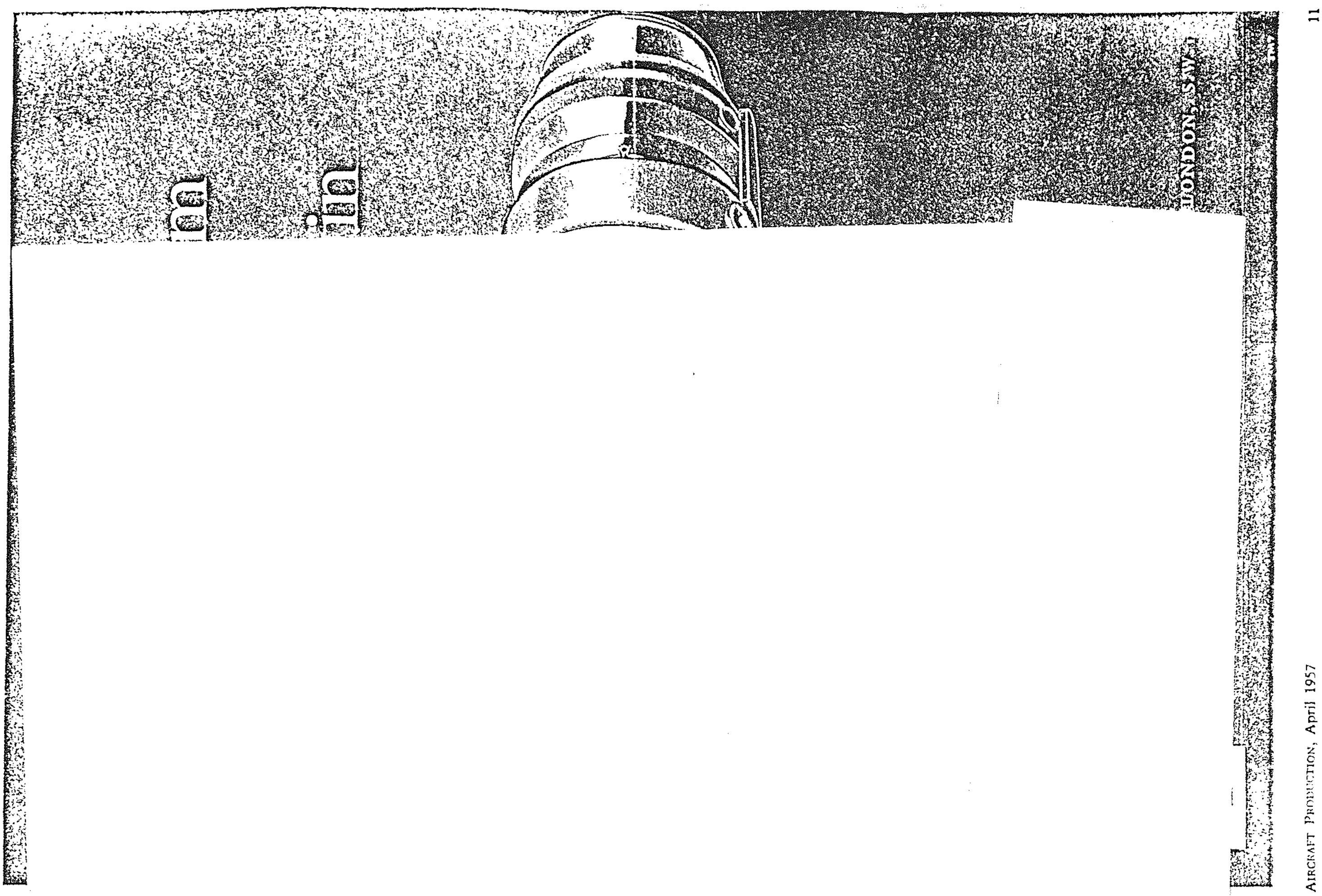
In many instances the showing of components at exhibitions was their finalé in a sense; for ICI had often had to provide new designs, data, fabricating techniques and training of the user personnel before titanium would be accepted as a worthy material - and not treated as a space age metal.

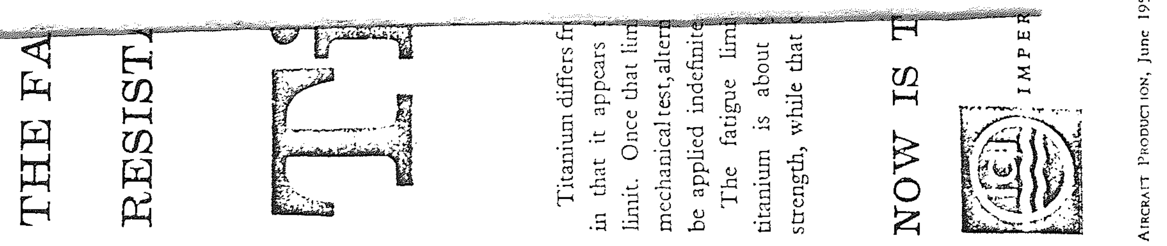
Some of the applications found for titanium, have been mentioned above, but a better indication of its usage outside the aerospace industry is given by referring to table D.2. Just two illustrations will be made of the development of titanium in non-aerospace use - condensor tubes and anodising jigs, under the heading of electrochemical processes.

D7.2.3 Steam Condensors

One of the uses for titanium that ICI did not foresee in their product forecast of the mid 1950's was that for water cooling in steam condensers using brackish or estuarine waters. The CEGB were experiencing difficulty with copper based alloy tubing during the late 1950's - problems such as erosion, impingement attack, general corrosion, stress corrosion and fatigue corrosion. When such a failure occurred at the Uskmouth power station in 1958 they decided to replace twenty of the steam condenser tubes with titanium instead of 70/30 cupronickel. Titanium showed its worth over the next few years and during the early 1960's trial quantities of titanium were put in a further six power stations. However, it was not until 1970 that the CEGB finally committed themselves to their first major installation using titanium - at the West Thurrock station, using about 100,000 feet of titanium tube. Subsequent stations have used titanium tube.

Figure D 2 Typical Titanium Advertisements Produced by ICI





D7.2.4 Electrochemical Processes

During 1958 ICI discovered that anodic-current continued to flow, when other metals were in contact with titanium, even though titanium forms a highly resistive oxide film under anodic conditions. This led to many applications in electro-finishing, cathodic protection, and other electrochemical processes.

Hoover (Washing Machines) Ltd. conventionally used plastic coated aluminium racks to support sheet aluminium components during anodising. These generally required a major over-haul every six weeks. During 1958/59 Hoover experimented with 400 aluminium racks fitted with titanium contacts and 60 racks entirely made from titanium (17). The change to titanium was fully justified, recovering the cost of the tipped racks in less than two months and the all titanium racks in six months.

A great deal of development work went on in many companies once it was realised that coated titanium anodes could offer advantages over other materials in industrial electrolytic cells. Titanium anodes coated with platinum for instance showed great promise for the production of chlorine by brine electrolysis and also for use in marine cathodic protection systems. ICI in fact were prepared to pool their knowledge with other companies for a more rapid development of such uses - and in August 1961 it was reported (18) that they would collaborate in the development and promotion of coated titanium anodes with another company - Magneto-Chemie N.V., Schiedaui, Holland.

D8 Commentary and Discussion

This case history has indicated the ways in which ICI and later IMI developed uses for their new material. The search for uses has not stopped, but is an ongoing process. Probably the latest developments have occurred since 1974 with the advent of oil rig applications - an area where the environment must suit the use of titanium admirably.

As has been indicated the titanium industry in this country owes much to the government of the day's belief that titanium was a necessary strategic material. But for their backing of ICI during the early years, the titanium story might have been very different. As it was there were arguments for and against the government's decision to give ICI the sole manufacturing contract of titanium sponge, but as The Economist (4) pointed out "ICI would argue that nobody else in Britain could have done this particular job".

However, to rely on a government for your sales of product can prove to be a dicey business, as ICI have surely found out. Changes in defence commitments in 1957 (missiles favoured rather than manned aircraft) and in 1964 (cancellation of TSR2, P-1154 and HS 681 aircraft projects) have had their toll on the aircraft industry and hence the titanium industry. Even on the civil aircraft side, there are risks which must be growing larger as the years go by - the Rolls Royce collapse of 1971 dealt yet another blow to the titanium industry. Such facts must surely make one wary of a reliance on one particular industrial sector. ICI and later IMI must have learnt this over the years and they have indeed tried to move their reliance from the aerospace to more diverse non-aerospace users, though even today sales of titanium are split something like 50/50 between aerospace and non-aerospace uses.

The development of uses for titanium has in many instances followed the lead set by the Americans and this has made it very difficult for IMI to expand sales in the USA. On the other hand sales in Europe have been quite significant, but as P. L. Bubb (19) has pointed out, as tariff and trade barriers are reduced competition in Europe becomes stiffer, particularly as there is considerable over capacity in titanium outside the UK. Indeed there is considerable argument in the industry regarding unfair trading, particularly as the main importers to Europe (USA, Japan and USSR) all operate from more highly protected domestic markets - which the European industry finds hard to penetrate. So even though IMI have been virtually in a monopolistic position for titanium in the UK, competition on the world market is far fiercer.

The US lead which Alexander mentioned in his report sent back to Metals Division, must have noted the tremendous alloy developments the Americans were making. It is understandable that some alloy development should take place when one realises that the uses of titanium were demanding the ultimate properties from the metal. Both Metals Division of ICI and William Jessop & Sons Ltd. were in the race for alloy developments - and the massive research effort must surely have added to the cost of an already expensive material. None of the titanium producers, it would appear, looked for uses for titanium which did not require this alloy development - in the early years at least. Most producers would probably argue that the defence applications formed the backbone of the industry, allowing other uses to be found at a more leisurely pace later. Whether the industry should have developed this wasy is debateable.

To say that ICI put a lot of R&D effort into titanium is putting it mildly - between 1951 and 1956 over 20% of General Chemicals Divisions' research staff were tied up on titanium and Metals Division were devoting some 40% of their research activity to titanium. As has been indicated, the development for uses of titanium was done on a very sound footing - involving the potential customer from the start, developing designs, providing data, learning and teaching the fabricating techniques to the user. Some parts were even developed and tested on their own equipment before introducing them to the world. This helped to ensure their success in the market place and in some cases allowed ICI to take out patents of their own on the developments. These actions by ICI may sound surprising to some people, particularly when you realise that Metals Division regarded themselves as producers of wrought products, and not of finished components. It serves to illustrate the lengths to which they were prepared to go to get people to buy their wrought product which could then be turned into the finished component by the customer.

To communicate the potential of titanium to the market, ICI used nearly every conceivable method possible - technical salesmen; articles in journals and papers; attending conferences to give papers; holding in-house symposia; attending exhibitions and even producing their own mobile exhibitions; producing handbooks and guides for the users of titanium. Whether they would be able to say which methods gave the best results, in terms of new business received in the company is doubtful but it does indicate their determination to make a success of titanium.

As an indication of the publication that ICI used, the list below shows nearly thirty used between 1955 and 1961 (excluding their own publications):-

Aeronautics
Aeroplane
Aircraft Engineering
Aircraft Production
Automobile Engineer
Birmingham Gazette
Birmingham Post
British Chemical Engineering
Chemical Age
Chemical and Process Engineering
Chemical Processing
Chemistry and Industry
Discovery
The Economist
The Engineer
Engineering
The Financial Times
Flight
Industrial Chemist
Light Metals
Metal Bulletin
Metal Finishing Journal
Metal Industry
Metall (Germany)
Metallurgical Reviews
Shell Aviation News
Sheet Metal Industries
The Times Review of Industry
The Times Survey of British Aviation

Their own publications included:-

ICI Titanium
Imperial Chemical Industries
Titanium Abstracts
Titanium for Chemical Plant
Wrought Titanium

ICI were not afraid of approaching other companies for collaborative developments of titanium, witness collaboration with another company for anode developments already mentioned. Another development that should not be overlooked was their approach with Columbia - Southern Chemical Corporation of Pittsburgh to the US government during 1955 for a five year contract to supply titanium via the sodium method. Both actions indicate ICI's strategy in developing titanium usage in other countries.

Having illustrated the methods used by ICI in searching for titanium applications, it is well to remember the time period that can be experienced between the users awareness of a material and his adoption of it into one of his products. For some applications where the metal relieved immediate problems, the time for adoption could be as short as one year (for instance, Hoover and the anode baskets). For others, where long term trials had to be carried out, the time could run into many years (15 or so for the Central Electricity Generating Board and condenser tubes).

The Economist (4) summarised the broader aspects of this materials innovation over 20 years ago:

> "...the whole history of titanium development...offers some provocative object-lessons for the study of industrial innovation in the second half of the twentieth century - and of the role of big business in the modern economy. Firstly, it exemplifies the great and growing demands upon the performance of industrial materials now being made at the cutting edges of advanced technology -

primarily in aircraft and in nuclear engineering. Secondly it emphasises that these most advanced technologies, and the demands on materials that they produce in their turn, stem primarily from defence, and would never have developed at such a rate had not that national need transcended economic considerations. Thirdly, whether or not a large company is essential to such ventures, titanium shows how big a proportion of the research and development staff...one single project of this calibre may take".

The same summary could be stated just as easily today, although defence projects are fewer now.

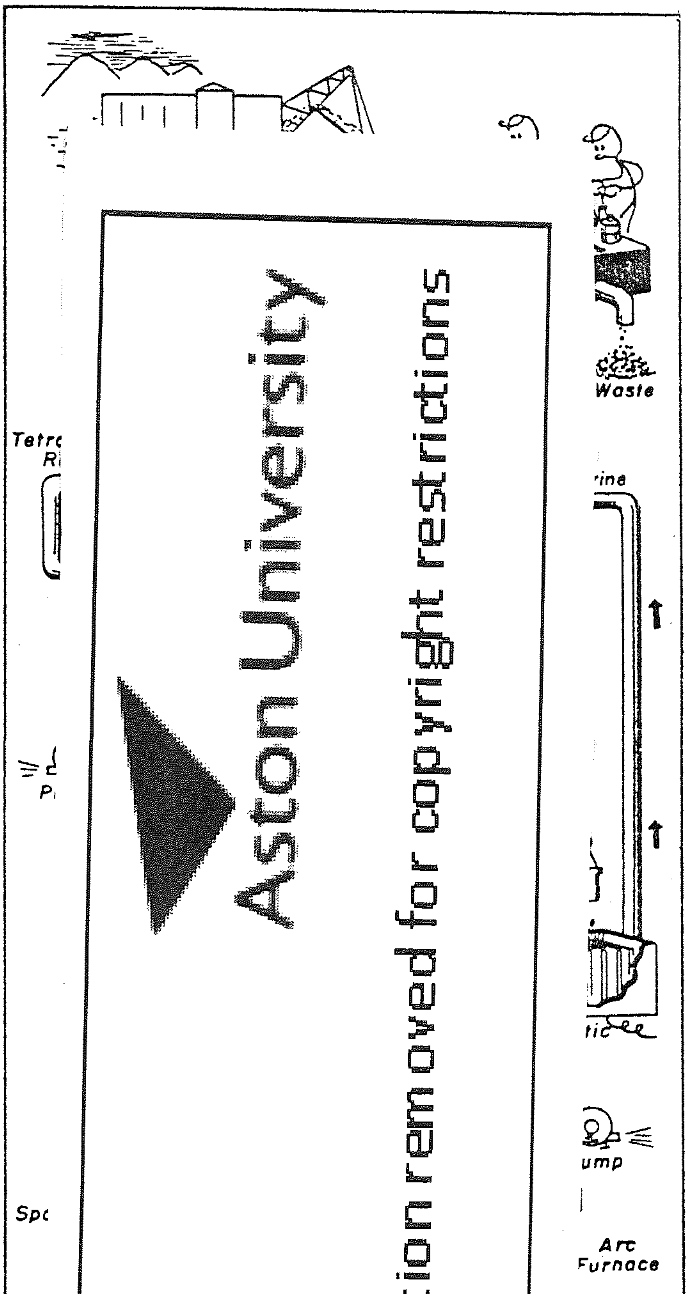
D9 Supplement

D9.1 Production Methods for Titanium Sponge

By far the most widely used method for the production of titanium sponge is the Kroll process - patented on June 25th, 1940: US Patent 2,205,854. Figure D.3 ably depicts the process through to the production of a titanium ingot. This illustrates the production from an ore called rutile (95% titanium dioxide); another rich ore of titanium is a black oxide mineral called ilmenite (50-60% titanium dioxide), after the Ilmen Mountains in Russia. There is no economic way of getting titanium straight from the oxide and one therefore has to chlorinate the ore and then reduce titanium tetrachloride.

The basic reaction in this process uses magnesium to reduce titanium tetrachloride:

$$TiCl_4 + 2Mg \longrightarrow Ti + 2MgCl_2$$



F … EARLY
T … SED ON
R … SANDS
A. … PPROX.
9 … UTILE
9 … TRACHLO
… FED
IN … AS THE
R … YIELD
… ANIUM.
M … FED TO
T … SOLID
AN ARC FURNACE FOR CONVERSION … SOLID
DUCTILE TITANIUM.
(DRAWING COURTESY OF WESTINGHOUSE ENGINEER.)

A different approach was pioneered by General Chemicals Division of ICI when they opted for sodium as the reducing agent:

$$TiCl_4 + 4Na \longrightarrow Ti + 4NaCl$$

Whilst this route is potentially more dangerous than the magnesium route, ICI believed it to be more advantageous to them. The decision to start work on the sodium was taken because (12):

(a) Sodium was cheaper than magnesium in the UK, even allowing for higher equivalent weight.

(b) The reaction is thermodynamically favourable.

(c) Some of the Kroll equipment was suitable for the initial experiments.

After the successfull development work with sodium, the decision was takento build a large plant based on sodium because:

(a) With increasing scale the sodium process promised a capital advantage and operating cost advantage.

(b) The reaction product of metal and salt was easier to break up than the Kroll product.

(c) The reaction product could be purified by leaching which was simpler than the high temperature purification required by the Kroll process.

(d) A continuous work up of the product could be envisaged.

(e) Sodium was available within the company: indeed the Wilton site sits on top of natural salt deposits and General Chemicals Division operated its own electrolytic plant.

D9.2 The Structure and properties of Titanium

Titanium can exist in two different phases - α and β. Below 882°C titanium has a close packed hexagonal structure - the α phase. Above 882°C titanium has a body centred cubic structure - the β phase. These different structures impart different characteristics to the Metal:

- α alloys are strong and maintain their strength at high temperatures but are difficult to fabricate.
- β alloys are less strong, easier to work but rather unsuitable at elevated temperatures - and little used commercially.
- α-β alloys have intermediate properties.

The unique properties of titanium are illustrated and compared with other metals in table D3 below (10). Of particular importance is its high strength to weight ratio and high melting point - and it was this, that led in its very early days of development to the belief that titanium would have very good creep strength - only to be shattered a few years later.

D10 Acknowledgements

The preparation of this history was made the easier for the help given by W. Alexander and H. Child of Aston University and also by N. D. J. Compton of ICI and various personnel of IMI. Grateful acknowledgement is given to T. W. Farthing for allowing the use of tables D1, 2 and 3 and figure D2 which he presented in his paper (10).

Table D3 Physical and mechanical properties of titanium compared with aluminium, copper, iron and magnesium

Physical Properties	Titanium	Aluminium	Copper	Iron	Magnesium
Melting point °C	1665	660	1083	1535	650
Density ($10^3 kg/m^3$)	4·505	2·70	8·94	7·86	1·74
Thermal conductivity at 20°C (cal/cm/cm^2/°C/sec)	0·0407	0·57	0·92	0·17	0·35
Electrical resistivity at 20°C (microhm cm)	55·4	2·68	1·72	10	4·4
Specific heat (0-100°C) (cal/g/°C)	0·126	0·211	0·093	0·109	0·245
Magnetic susceptibility (10^{-6}cgs units /g)	+1·25	+0·65	-0·086	Ferro-magnetic	+0·55
Mean electrode potential (volts) in dilute chloride solution. (Related to saturated calomel scale)	-0·10	-0·88	-0·29	-0·58	-1·73
Typical mechanical properties	IMI 125 annealed	99·5 Aluminium annealed	Deoxidized non-arsenical copper	Armco iron normalized	99·9 Magnesium annealed
0·2% proof stress MPa	340	35	54	185	69
Tensile strength MPa	440	80	230	340	180
Elongation %	29	47	56	39	5
Young's Modulus GPa	120	70	120	210	45
Fatigue limit as % of tensile strength	50	40	33	45	37

(Courtesy of IMI)

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16. 'Shut-downs at Billingham due to broken valve springs and plates avoided by use of titanium parts', Chemical Processing, 4, No. 2, p. 9, Feb. 1958.

17. Jones, J.C. 'Titanium jigs are justified', Product Finishing, 12, No. 12, Dec. 1959.

18. Financial Times, No. 22482, p. 15; 31st August, 1961.

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APPENDIX E

Survey on Materials - Results

Contents

Key to table ratings:

5 - essential	2 - rarely useful
4 - worthwhile	1 - no use
3 - occasionally useful	0 - not used.

Key to symbols:

* - abstracts specified by respondents

⊕ - journals and other channels specified by respondents.

Industrial Sector: Aerospace

Material Decision Makers rating of various information channels for keeping him in touch with (a) business developments and (b) material developments.

BUSINESS DEVELOPMENTS							MATERIAL DEVELOPMENTS					
5	4	3	2	1	0		5	4	3	2	1	0
	1				20	* Abstracts (give name) TOTAL	1	7	5			13
					1	BNF		1	1			
					1	DEFENCE RESEARCH ABSTRACTS		2				
					1	ENGINEERING INDEX	1					
					1	INTERNATIONAL AEROSPACE ABSTRACTS			1			
					1	INTERNATIONAL METAL ABSTRACTS		1				
					1	J. OF SPACE CRAFT & ROCKETS			1			
					1	PERA			1			
					3	TECH. LINKS		2	1			
	1	3	4	2	8	BBC's Tomorrows World		2	5	7	3	8
	2	7			9	Chartered Mechanical Engineer	2	1	7	4		10
	1		2	1	14	Composites		6	2	1	2	14
1	4	2	4	1	6	The Daily Telegraph		1		5	6	13
	4	2	4	1	7	Design Engineering		8	8	5		4
2	2	8	1		4	The Engineer	1	5	9	4		3
	3	3	3		8	Engineering		1	9	5		7
	2	2	7		7	Engineering Materials & Design		10	9	3		3
1	1	1	2		13	The Financial Times			4	4		17
	1	2	1	1	13	The Guardian				3	2	20
	1	2	2		13	Materials Engineering		4	6	2		13
	1		1		16	Materials Science & Engineering		2	1	1		21
	1	3	3		11	Metallurgist & Materials Technologist	1	5	4	2		13
	2	1	1		14	Metals & Materials	1	2	5	1		16
			1		17	Nature		1		1		23
		2	4	3	9	New Scientist	1		1	9		14
		2	4	1	11	Plastics Engineering		2	6	6		11
	4	4	1	1	8	The Sunday Times		1	2	3	3	16
		4	1		13	The Times				4	1	20
		2	3	4	9	Whats New in industrial products & Equipment			3	5	2	15
		1	4	3	8	Aeronautical Journal		2	3	6	2	8
	1	3	1	3	8	Aircraft Engineering		3	5	3	1	9
3	2	2	1	3	5	Aviation Week & Space Technology		3	8	3	1	6
						⊕ Other - Please specify.						
		1				ACCOUSTICS BULLETIN					1	
					1	ASME			1			
					1	ASTM PUBLICATIONS		1				
			1			BSI NEWS		1				
						CHEMISTRY IN BRITAIN				1		
		1				ELECTRONICS			1			
		1				ENGINEERING TODAY					1	
		1		1		INSULATION & CIRCUITS		2				
	1		1			INTERAVIA			2			
					1	J. OF COMPOSITE MATERIALS		1				
					1	J. OF MATERIALS SCIENCE		1				
					1	J. OF PLASTICS & RUBBER INSTITUTE			1			
					2	J. OF ROYAL AERONAUTICAL SOCIETY			2			
				1		LUBRICATION ENGINEERING		1				
		1				MACHINE DESIGN		1				
					1	POLYMER ENGINEERING & SCIENCE			1			
		1				RESIN NEWS		1				
1						TRADE & INDUSTRY			1			

Industrial Sector: General Engineering - survey 1

Material Decision Makers rating of various information channels for keeping him in touch with (a) business developments and (b) material developments.

BUSINESS DEVELOPMENTS							MATERIAL DEVELOPMENTS					
5	4	3	2	1	C		5	4	3	2	1	6
					35	Abstracts (give name) ⊕ SEE BOTTOM 5 ON LIST.						43
	3	6	13	10	8	BBC's Tomorrows World		8	18	17	2	4
	1	5	5	2	27	Chartered Mechanical Engineer		3	11	6	2	27
		2	1	2	35	Composites			3	2	2	42
	5	9	5		21	The Daily Telegraph			4	11	3	31
1	5	10	15	1	8	Design Engineering	3	18	15	6		7
	6	9	10	1	14	Engineering Materials & Design	2	13	13	8		13
9	5	10	2		14	The Financial Times	1	5	13	5	3	22
	2	4	1		33	The Guardian			3	2	2	42
	3	6	9		22	Material's Engineering	1	9	14	4	1	20
	2	1	4		33	Material's Science & Engineering	1	5	4	2	1	36
	2	2	3		33	Metallurgist & Materials Technologist	1	6	4	1	1	36
	3	2	6	1	28	Metals & Materials	2	7	6	2		32
					40	Nature			1	1		47
		2	1	1	36	New Scientist		2	2	4	1	40
		5	3	1	31	Plastics Engineering		3	3	4		34
	6	10	9	1	14	The Sunday Times		2	6	9	7	25
	4	5	5		26	The Times				6	4	39
		6	7	4	23	Whats New in industrial products & Equipment		5	11	6	1	26
		1				⊕ CBI BULLETIN					1	
		1				⊕ CHARTERED PRODUCTION ENGINEER		1	1			
					1	⊕ THE CONSULTING ENGINEER		1				
	2	2				⊕ THE ENGINEER		3	1			
	1					⊕ ENGINEERING		1				
		1				⊕ ENGINEERING TODAY				1		
		1				⊕ INDUSTRIAL EQUIPMENT NEWS			1			
		1				⊕ INDUSTRIAL PURCHASING NEWS			1			
		1				⊕ MAINTENANCE ENGINEER			1			
			1			⊕ METAL PROGRESS	1	1				
1						⊕ METAL WORKING PRODUCTION	1					
	1					⊕ ORIGINAL EQUIPMENT MANUFACTURE DESIGN		1				
					1	⊕ PLASTICS & RUBBER WEEKLY		1				
		1				⊕ PRODUCT FINISHING		1				
				1		⊕ OWN COMPANY ABSTRACT		1				
	1			1		* RERA		3				
					1	* [illegible] P.			1			
					1	* DER[illegible]NT BR[illegible]SH [illegible]T ABSTRACTS			1			
	1					* [illegible] BULLETIN			1			

Industrial Sector: General Engineering
- survey 2

Material Decision Makers rating of various information channels for keeping him in touch with (a) business developments and (b) material developments.

BUSINESS DEVELOPMENTS							MATERIAL DEVELOPMENTS					
5	4	3	2	1	0		5	4	3	2	1	0
3				1	19	* Abstracts (give name) - TOTAL	3	1	1			19
1						COMPANY ABSTRACT	2					
						RESEARCH ASSOC ABSTRACT - UNSPECIFIED		1				
2						RAPRA	1					
1	2	5	2	3	8	BBC's Tomorrows World		2	6	5	2	7
	2	2	1	2	14	Chartered Mechancial Engineer		2		1	4	15
	1		1	1	18	Composites		1		1		20
1	1	3	2	1	13	The Daily Telegraph			1	4	1	16
	6	4	2	1	8	Design Engineering		8	5	3		6
1	2	4	2		7	The Engineer		1	6	4		5
	2	3	2		9	Engineering		1	5	2		8
1	5	1	5	2	7	Engineering Materials & Design	1	8	4	3	1	5
2	4	2	2		11	The Financial Times			5	2	2	13
		2			19	The Guardian			1		1	20
		2	3	1	15	Materials Engineering		1	2	2		17
			2	1	18	Materials Science & Engineering			1	2		19
		3	1	1	16	Metallurgist & Materials Technologist	2	2		1		17
		4		1	16	Metals & Materials	2	2			1	17
				1	20	Nature					1	21
		1	1	1	18	New Scientist			2	1	1	18
		2	2	1	16	Plastics Engineering	1		3	2		16
	1		5		15	The Sunday Times			1	5	1	15
	1		1		19	The Times				1	1	20
	3	1	1	2	14	Whats New in industrial products & Equipment		5	1		1	15
						⊕ OTHER - PLEASE SPECIFY						
1		1				ENGINEERING TODAY			1			
					1	EUROPEAN PLASTICS NEWS			1			
			1			FACTORY EQUIPMENT NEWS		1				
1	1					EUROPEAN RUBBER JOURNAL	1	1				
	1					HYDRAULICS & PNEUMATICS		1				
	1		1			INDUSTRIAL EQUIPMENT NEWS		1		1		
1						INTERNATIONAL PRODUCT DIJEST			1			
			1			MATERIALS PROTECTION	1					
					1	MODERN PLASTICS INTERNATIONAL			1			
1	2					PLASTICS & RUBBER WEEKLY	1	3				
		1				PROCESS ENGINEERING		1				
1						RUBBER AGE	1					
					1	RUBBER JOURNAL		1				
1						RUBBER WORLD	1					

Industrial Sector: Material Producer

Material Decision Makers rating of various information channels for keeping him in touch with (a) business developments and (b) material developments.

BUSINESS DEVELOPMENTS							MATERIAL DEVELOPMENTS					
5	4	3	2	1	0		5	4	3	2	1	0
3		4			13	* Abstracts (give name) - TOTAL	3	5	2	1		15
					1	BCIRA PUBLICATIONS		1				
1						BNF TECHNOLOGICAL CENTRE		1				
		1				COMPANY ABSTRACT	1	1				
					1	N.P.L.			1			
1						RAPRA	1					
1		1				TRANSACTIONS OF THE BRIT. CERAMIC SOC.	1	1				
		1				TRANSACTIONS OF THE AMERICAN CERAMIC SOC.		1				
	1	1	5	3	8	BBC's Tomorrows World		5	6	6	4	4
	2		1	3	12	Chartered Mechanical Engineer		4	3	2	1	15
			1	2	15	Composites			1	1	1	22
1	3	3	5		6	The Daily Telegraph		2	1	6	5	11
	1	4	3		10	Design Engineering		3	9	1	2	10
	1	1	3		3	The Engineer		1	4	1		5
	1	1	3		3	Engineering		1	4	2		4
		4	4		10	Engineering Materials & Design		3	11	4	2	5
4	4	3	2		5	The Financial Times		3	6	5		11
	2		2		14	The Guardian				3	1	21
		2	1	2	13	Material's Engineering		1	7	3		14
		3		1	14	Material's Science & Engineering		1	5	1		18
	1		1	1	15	Metallurgist & Materials Technologist	1	3	2	1		18
	1	4		1	12	Metals & Materials	1	3	3	4		14
		1	1	1	15	Nature				2	1	22
		1	2		15	New Scientist			5	1		19
	1	1	2	1	13	Plastics Engineering	1	1		2		21
1	2	3	2	2	8	The Sunday Times			2	4	4	15
1	2	2	1	2	10	The Times				4	3	18
1	2	1	3	1	10	Whats New in industrial products & Equipment	2	1	7	3	1	11
						⊕ Other - Please specify						
		1				EUROPEAN PLASTICS NEWS	1					
					1	FOUNDRY PUBLICATIONS - GERMAN & U.S.A.	1					
		1				MODERN PLASTICS	1					
1						PATENTS OFFICE LIBRARY	1					
	1					SHEET METAL INDUSTRIES		1				
					1	SPORT AVIATION (EXPERIMENTAL AIRCRAFT ASSOC)			1			
	1					STEEL TIMES		1				
	1					WOLFSON JOURNAL - 'HEAT TREATMENT OF METALS'			1			

Industrial Sector: Nuclear and Power Industries

Material Decision Makers rating of various information channels for keeping him in touch with (a) business developments and (b) material developments.

Business Developments 5	4	3	2	1	0		Material Developments 5	4	3	2	1	0
	1	2	3		12	* Abstracts (give name) – TOTAL	3	2	5	1		10
			1			BRITISH CERAMIC ABSTRACTS			1			
					1	CHEMICAL ABSTRACTS		1				
		1				IEE ABSTRACTS				1		
					1	INTERNAL LIBRARY SERVICE	1		2			
			1		1	METAL ABSTRACTS	1		1			
	1					NUCLEONICS WEEKLY			1			
			1			PERA		1				
		1				RAPRA	1					
	1	2	4	1	6	BBC's Tomorrows World		4	4	2	3	4
	4	2	1		7	Chartered Mechanical Engineer		7	2			8
	1				13	Composites		1		2		14
	3	3	2		6	The Daily Telegraph			2	1	5	9
	2	2	4		6	Design Engineering		5	3	2		7
	3	3	4		3	The Engineer		3	7	2		4
	2	3	5		3	Engineering		4	6	2		4
	2	1	6	1	4	Engineering Materials & Design		9	3	1		4
1	7	1	1		4	The Financial Times		1	4	5	1	6
	3	2	2	1	6	The Guardian			1	2	4	10
	1	2	4		7	Materials Engineering	1	4	5	1		6
	1	1	2		10	Materials-Science & Engineering	1	3	3	2		8
	1	3	1		9	Metallurgist & Materials Technologist		8		1		8
		3	3	1	7	Metals & Materials		5	4	1		7
				2	12	Nature		1	2	2		12
		4	5	1	4	New Scientist	1	2	6	4	1	3
			1	1	12	Plastics Engineering				2		15
	6	2			6	The Sunday Times				3	5	9
	3	4	2		5	The Times				5	3	9
			2		12	Whats New in industrial products & Equipment			2	1		14
			2		11	AWRE News		1	1	1		13
			3		10	Carbon			2	1		13
						⌖ Other - Please specify						
		1	1			ATOM						2
		1				B.C.S.A		1				
					1	B.I.S.R.A.		1				
			1			BRITISH WELDING JOURNAL			1			
		1				CHEMISTRY IN BRITAIN		1				
		1				THE CONSULTING ENGINEER		1				
		1				ENGINEERING TODAY				1		
		1				[illegible]	1					
					1	FINISHING INDUSTRIES			1			
					1	INCO PUBLICATIONS		1				
	1					INDUSTRIAL EQUIPMENT NEWS			1			
					2	INSTITUTE OF METALS [illegible]		2				
					2	JOURNAL OF [illegible] SCIENCE	1	1				
					2	JOURNAL OF NUCLEAR MATERIALS		2				
			1			MATERIALS PERFORMANCE			1			
				1	1	METAL CONSTRUCTION			2			
						METAL SCIENCE			1			
				1		METAL TECHNOLOGY			1			
						METALL TRANSACTIONS	1					

Industrial Sector: Nuclear and Power Industries

Material Decision Makers rating of various information channels for keeping him in touch with (a) business developments and (b) material developments.

BUSINESS DEVELOPMENTS							MATERIAL DEVELOPMENTS					
5	4	3	2	1	0		5	4	3	2	1	0
					1	METALLURGIA & METAL FORMING			1			
		1				MODERN PLASTICS INTERNATIONAL		1				
		1				NEW CIVIL ENGINEER		1				
1	1					PLASTICS & RUBBER WEEKLY	1	1				
		1			2	WELDING & METAL FABRICATION		3				
			1			WELDING IN THE WORLD			1			
					2	WELDING INSTITUTE - RESEARCH BULLETIN		1	1			
				1	2	WELDING JOURNAL		2	1			

Industrial Sector: Road Transport

Material Decision Makers rating of various information channels for keeping him in touch with (a) business developments and (b) material developments.

BUSINESS DEVELOPMENTS 5	4	3	2	1	0		MATERIAL DEVELOPMENTS 5	4	3	2	1	0
		6	2		29	* Abstracts (give name) - TOTAL	8	1		2		30
		1				BCIRA	1					
		1				BIM						1
		1			1	BNF	1					
					1	MIRA	2					
		1	1			PERA	1	1				
		2	1			RAPRA	3					
		11	9	4	10	BBC's Tomorrows World		2	19	10	3	5
	3	5	4	1	21	Chartered Mechanical Engineer	1	6	10	1		21
		2	1	1	30	Composites		2	2	2		33
1	8	7	4		14	The Daily Telegraph			2	12	5	20
	7	7	8	3	9	Design Engineering	3	14	13	1		8
2	7	2	3		7	The Engineer	1	6	6	2	1	9
1	3	3	3		11	Engineering	1	4	7	1	1	11
1	3	8	8	4	10	Engineering Materials & Design	4	11	14	2		8
11	6	2	1		14	The Financial Times	·	4	8	6	2	19
	1	2	1	2	28	The Guardian				4	2	33
1		4	4	3	22	Materials Engineering	3	5	8	1		22
		2		2	30	Materials Science & Engineering		4	1	1	1	32
	2	2	1	3	26	Metallurgist & Materials Technologist	3	5	2	1		28
	1	2	3	4	24	Metals & Materials	1	6	4	2		26
			1	1	32	Nature			2		1	36
		1	2	2	29	New Scientist		1	2	3	2	31
1	3	2	3	4	21	Plastics Engineering	2	6	8	3	1	19
	7	3	5		19	The Sunday Times		3	2	7	5	22
·	7	1	3	1	22	The Times		1	3	5	3	27
		2	4	4	24	Whats New in industrial products & Equipment	1	1	8	5	2	22
1	3	4	5	1	7	Automotive Engineering	3	6	5	3	1	7
1	1	3	1		15	Automotive News	1	2	3		1	18
	2	4	1	1	14	Commercial Motor		2	4	1	2	17
					21	Motor Trade Executive with Motor Industry			1			24
	1	2		1	18	Motor Transport		1	2	1	1	21
1	2		5	2	11	SAE Journal of Automotive Engineering	1	5	4	2		13
						✦ Other - Please specify.						
1						AUTOMOTIVE INDUSTRIES	1					
			1			BRITISH PLASTICS & RUBBER		1				
					1	BSI NEWS		1				
			1			CHEMISTRY IN BRITAIN		1				
	1	1	2			EUROPEAN PLASTICS NEWS	2	2				
	1					FINISHING INDUSTRIES	1					
		1				INDUSTRIAL PURCHASING NEWS			1			
	1					INST. OF MEASUREMENT & CONTROL				1		
			1			INST. OF PHYSICS JOURNAL			1			
					1	IRON AGE METALWORKING INTERNATIONAL				1		
		1				MATERIALS HANDLING NEWS			1			
	1					METAL PROGRESS		1				
					1	METAL WORKING PRODUCTION				1		
			1			PERSONAL CONTACTS	1					
			1			PLANT ENGINEER & MAINTENANCE		1				
					1	PLASTICS & RUBBER INTERNATIONAL	1					

Industrial Sector: Road Transport

Material Decision Makers rating of various information channels for keeping him in touch with (a) business developments and (b) material developments.

BUSINESS DEVELOPMENTS							MATERIAL DEVELOPMENTS					
5	4	3	2	1	0		5	4	3	2	1	0
	1		1			PLASTICS & RUBBER WEEKLY		1	1			
		1				PROCESSING			1			
		1				PRODUCTION ENGINEERING	1					
		1				REINFORCED PLASTICS		1				
			2			RUBBER JOURNALS - VARIOUS: EUROPEAN, USA, USSR etc.		2				
	1					SHEET METAL INDUSTRIES		1				
					1	THE SPRING JOURNAL		1				
					1	TRANSACTIONS OF THE ASME			1			
	1					WELDING JOURNAL (AMERICAN W.S.)		1				

Industrial Sector: Research Establishments

Material Decision Makers rating of various information channels for keeping him in touch with (a) business developments and (b) material developments.

BUSINESS DEVELOPMENTS							MATERIAL DEVELOPMENTS					
5	4	3	2	1	0		5	4	3	2	1	0
-		3	3	2	12	* Abstracts (give name) – TOTAL	3	4	5			10
					1	BICERI ABSTRACTS		1				
			1			BRITISH CERAMIC ABSTRACTS			1			
				1	1	CHEMICAL ABSTRACTS		2				
		1				COPPER ABSTRACTS			1			
					1	ENGINEERING INDEX	1					
			2		1	METAL ABSTRACTS	1		1			
					1	NTIS WEEKLY GOVERNMENT ABS.		1				
		2		1		RAPRA	1		2			
	3	3	3	2	5	BBC's Tomorrows World		7	6	3		4
	2	2	1		11	Chartered Mechanical Engineer		2	4	1		13
		2	1	1	12	Composites		3	1	1		15
	2	3	3		8	The Daily Telegraph			2	4	3	11
	1	2	6		7	Design Engineering		7	3	3		7
	2	7	1		5	The Engineer		5	7	1		6
		6	2	1	6	Engineering		4	6		1	8
	2	5	2	1	6	Engineering Materials & Design	1	11	2	1		5
2	8	2			4	The Financial Times		4	8	1	1	6
	2	1	1	1	11	The Guardian		1	1	2	2	14
	1	4	2		9	Materials Engineering		6	2			12
		1	2		13	Materials Science & Engineering		3	3			14
	1	2	3	1	9	Metallurgist & Materials Technologist	1	4	3			12
		2	3	1	10	Metals & Materials	1	4	3			12
	1		1	5	9	Nature		3	4	2		11
		7	2	1	6	New Scientist	1	4	11			4
		1	1	1	13	Plastics Engineering		3			1	16
	5	4		1	6	The Sunday Times		1	4	7	3	5
	1	5		1	9	The Times		1		7	1	11
		2	1	1	12	Whats New in industrial products & Equipment			4		1	15
						⊕ OTHER – PLEASE SPECIFY						
				1		ADHESIVE AGE			1			
				1		BRITISH POLYMER JOURNAL			1			
		1	1			CHEMISTRY IN BRITAIN		1				
			1			CHEMISTRY IN INDUSTRY				1		
		1				CORROSION			1			
				1		DIE MAKROMAL CHEMIE		1				
			1			EUROPEAN CHEMICAL NEWS				1		
		1				EUROPEAN PLASTICS	1					
				1		EUROPEAN POLYMER JOURNAL			1			
					1	ENGINEERS DIGEST			1			
	1					JAPAN PLASTICS	1					
						JAPANESE IRON & STEEL INST. JOURNAL		1				
					1	JOURNAL MATERIAL SCIENCE	1					
				1		JOURNAL OF APPLIED POLYMER SCIENCE		1				
				1		MACROMOLECULES			1			
		1				METAL CONSTRUCTION				1		
					1	METALS TECHNOLOGY		1				
	1	1				MODERN PLASTICS INTERNATIONAL	1	1				
1	2	1				PLASTICS & RUBBER WEEKLY	1		1			
	1					PLASTICS TECHNOLOGY	1					

Industrial Sector: Research Establishments

Material Decision Makers rating of various information channels for keeping him in touch with (a) business developments and (b) material developments.

BUSINESS DEVELOPMENTS 5	4	3	2	1	0		MATERIAL DEVELOPMENTS 5	4	3	2	1	0
				1		POLYMER			1			
			1			P.R.I. JOURNAL				1		
				1		RUBBER CHEM. & TECH.		1				
				1		RUBBER WORLD			1			

Industrial Sector: Small Marine

Material Decision Makers rating of various information channels for keeping him in touch with (a) business developments and (b) material developments.

BUSINESS DEVELOPMENTS							MATERIAL DEVELOPMENTS					
5	4	3	2	1	0		5	4	3	2	1	0
					10	* Abstracts (give name) – TOTAL						13
	1	2	3	1	3	BBC's Tomorrows World		2	7	3		1
		1			9	Chartered Mechanical Engineer		1		1		11
			1		9	Composites				2		11
	2	2	1		5	The Daily Telegraph				4	2	7
	1	2	1		6	Design Engineering	1	1	2	1		8
		2			8	The Engineer			3			10
		1			9	Engineering			1	1		11
		2	1		7	Engineering Materials & Design		1	2	1		9
	1	2	2		5	The Financial Times				3		10
			1		9	The Guardian						13
					10	Materials Engineering			1	1		11
					10	Materials-Science & Engineering						13
					10	Metallurgist & Materials Technologist						13
					10	Metals & Materials			1			12
					10	Nature						13
			1		9	New Scientist			2	3		8
		1			9	Plastics Engineering		1	2	2		8
	2	1	2		5	The Sunday Times				2	2	9
		1	1		8	The Times					1	12
		2	1		7	Whats New in industrial products & Equipment			1	2		10
		1	1	2	6	Dinghy Sailing			3		2	7
		1	1		8	International Dinghy			3		2	7
	1	2	2	2	3	Yachting Monthly		2	2	2	2	4
1	1	3	2	1	2	Yachts & Yachting	3		4	3		2
						⊕ Other - Please specify						
		1				B.P.&R		1				
	1					CHANDLER & BOAT BUILDER		1				1
1						MOTOR BOAT & YACHTING						
					1	PRACTICAL BOAT OWNER		1				
		1				REINFORCED PLASTICS		2				
					1	SEAHORSE		1				
		1				YACHTING WORLD						1

Industrial Sector: Other

Material Decision Makers rating of various information channels for keeping him in touch with (a) business developments and (b) material developments.

BUSINESS DEVELOPMENTS							MATERIAL DEVELOPMENTS					
5	4	3	2	1	0		5	4	3	2	1	0
					11	* Abstracts (give name) - TOTAL			1			13
	1	2	4	1	3	BBC's Tomorrows World		2	3	5	2	2
	1	4			6	Chartered Mechanical Engineer		1	3	2		8
		2			9	Composites		1	1	2		10
	1	2	2		6	The Daily Telegraph			2	3	3	6
	2	2	2	2	3	Design Engineering	3	1	9	1		
	3	1	2		3	The Engineer		1	6	2	1	1
	1	1	2		4	Engineering	1	1	2	3	1	2
	2	1	4		4	Engineering Materials & Design		4	5	2		3
1	1	1	1		7	The Financial Times		1	2	2		9
		3	2		6	The Guardian			1	1	2	10
		1	1		9	Materials Engineering		1	1	2		10
		1	1		9	Materials-Science & Engineering		1		1		12
		1	1		9	Metallurgist & Materials Technologist	1		1	1		11
			2		9	Metals & Materials			1	3		10
		1			10	Nature			1			13
	1		1		9	New Scientist		1	3	3		7
			2		9	Plastics Engineering	2		2	1		9
		3	3		5	The Sunday Times			1	3	1	9
	1				10	The Times		1				13
		3	4		4	Whats New in industrial products & Equipment			4	4	1	5
						⊕ Other - Please specify						

Industrial Sector: Dunlop

Material Decision Makers rating of various information channels for keeping him in touch with (a) business developments and (b) material developments.

BUSINESS DEVELOPMENTS							MATERIAL DEVELOPMENTS					
5	4	3	2	1	0		5	4	3	2	1	0
3		1		2	8	Abstracts (give name) - TOTAL	3	1	2			10
				1		CHEMICAL ABSTRACTS		1				
1						INTERNAL COMPANY ABSTRACTS	1					
2		1		1		RAPRA	2		2			
	2	2	2	3	3	BBC's Tomorrows World		1	6	4	1	2
	1	1	1		9	Chartered Mechancial Engineer			2		2	10
			2	2	8	Composites			1	4		9
1	2	1	2	1	5	The Daily Telegraph				4	1	9
	3	2	3		4	Design Engineering		5	1	3		5
1	1	4	1		5	The Engineer		3	5			6
	1	4	1		6	Engineering		2	4	1		7
	4	3	1		4	Engineering Materials & Design	1	6		3		4
1	3	1			7	The Financial Times		2	4	1		7
	1	3			8	The Guardian			1	2	1	10
	3	1			8	Materials Engineering		2	1	1		10
	2	1			9	Materials Science & Engineering		2	1	1		10
					12	Metallurgist & Materials Technologist		1				13
			1		11	Metals & Materials		1		1		12
			1	3	8	Nature			3	1		10
		2	3		7	New Scientist		1	3	2		8
	1	1			10	Plastics Engineering		1	1	1		11
	1	3	1		7	The Sunday Times			2	3	1	8
		2			10	The Times		1		2		11
	1	1	1	1	8	Whats New in industrial products & Equipment		1	3	1		9
						Other - Please specify.						
				1		ADHESIVES AGE			1			
				1		BRITISH POLYMER JOURNAL			1			
		1				CHEMISTRY IN BRITAIN				1		
			1			CHEMISTRY IN INDUSTRY				1		
				1		DIE MAKROMAL CHEMIE ?		1				
		1				ENGINEERING TODAY				1		
			1			EUROPEAN CHEMICAL NEWS				1		
				1		EUROPEAN POLYMER JOURNAL			1			
1	1					EUROPEAN RUBBER JOURNAL	2	1				
	1					JAPAN PLASTICS	1					
				2		JOURNAL OF APPLIED POLYMER SCIENCE		2				
	1					MODERN PLASTICS INTERNATIONAL	1					
			1			PRI JOURNAL				1		
1	3					PLASTICS & RUBBER WEEKLY	1	3		1		
	1					PLASTICS TECHNOLOGY	1					
				1		POLYMER			1			
				1		RUBBER CHEM. & TECH.	1	1				
					1	RUBBER JOURNAL	1	1				
1				1		RUBBER WORLD	2		1			

Industrial Sector: Total

Material Decision Makers rating of various information channels for keeping him in touch with (a) business developments and (b) material developments.

BUSINESS DEVELOPMENTS 5	4	3	2	1	0		MATERIAL DEVELOPMENTS 5	4	3	2	1	0
6	3	15	8	5	161	Abstracts (give name)	21	18	21	4	0	166
		1			1	BCIRA	1	1				
		1				BIM						1
1		1			2	BNF	1	2	1			
					1	BICERI		1				
		2			1	BRITISH CERAMIC ABSTRACTS			2			
				1	2	CHEMICAL ABSTRACTS		3				
1		1		1	1	COMPANY ABSTRACTS	4	2	2			
		1				COPPER ABSTRACTS			1			
					1	DEFENCE RESEARCH ABSTRACTS		2				
					1	DERWENT BRITISH PATENT ABSTRACTS			1			
					2	ENGINEERING INDEX	2					
		1				IEE ABSTRACTS				1		
					1	INTERNATIONAL AEROSPACE ABSTRACTS			1			
			3		3	INTERNATIONAL METAL ABSTRACTS	2	1	2			
					1	J. OF SPACE CRAFT & ROCKETS	-		1			
					1	MIRA	2					
	1					NADFS			1			
					1	NPL			1			
					1	NTIS WEEKLY GOV'T. ABSTRACTS		1				
	1					NUCLEONICS WEEKLY			1			
	1	1	2	1	1	PERA	1	5	1			
3		5	1	1	1	RAPRA	7		2			
					1	RESEARCH ASSOC ABS. (UNSPECIFIED)		1				
					3	TECH LINKS		2	1			
1		1				TRANSACTIONS OF BRIT. CERAMIC SOCIETY	1	1				
		1				TRANSACTIONS OF AMERICAN CERAMIC SOC.		1				
					1	ZINC CORP. ABSTRACTS			1			
1	13	35	47	27	59	BBC's Tomorrows World		34	74	58	19	39
	17	28	13	8	116	Chartered Mechancial Engineer	3	28	40	18	7	128
	3	8	8	8	155	Composites		14	10	14	5	181
4	29	34	28	2	85	The Daily Telegraph		3	14	50	33	124
1	29	35	45	8	64	Design Engineering	10	65	67	23	2	57
5	22	30	16		40	The Engineer	2	25	49	16	2	43
1	13	21	20	1	53	Engineering	2	17	40	16	3	55
2	22	33	47	9	69	Engineering Materials & Design	8	75	63	20	3	55
31	37	24	13		77	The Financial Times	1	18	50	33	9	113
	11	16	11	5	139	The Guardian		1	7	17	16	183
1	6	21	26	6	120	Materials Engineering	5	31	46	16	1	125
	4	9	12	4	153	Materials Science & Engineering	2	19	18	10	2	173
	8	16	14	6	138	Metallurgist & Materials Technologist	10	33	16	8	1	156
	7	18	18	9	130	Metals & Materials	8	29	27	13	1	146
	1	2	4	10	165	Nature		5	10	8	3	198
	1	18	19	9	135	New Scientist	3	10	34	28	5	144
1	4	14	18	10	135	Plastics Engineering	6	16	29	22	2	149
1	33	30	27	5	86	The Sunday Times		7	18	43	31	125
1	19	22	14	4	122	The Times		3	3	32	17	169
1	5	19	26	16	115	Whats New in industrial products & Equipment	3	12	41	26	9	133

APPENDIX F

Carbon/Carbon Publicity

F.1 Carbon/carbon Publicity

Organised publicity was given to carbon/carbon during the early months of 1978, as indicated in table 9.1. Copies of the published articles and advertisement are reproduced herein, as is the brochure which was used in response to the enquiries.

A copy of the: article "How should Industry keep up-to-date with material developments?" which appeared in the Chartered Mechanical Engineer (March 1978, pages 105-106) is shown on pages 573 and 574.

: article "This hot composite is just waiting for the brakes to be taken off" which appeared in The Engineer (February 9th, 1978) is shown on page 575.

: article "Materials looking for markets" which appeared in The Metallurgist and Materials Technologist (March 1978) is shown on page 576.

: article "10 ways not to market a new material" which appeared in Engineering (March 1978) is shown on pages 577 and 578.

: advertisement selling carbon/carbon which appeared in the March 1978 issues of CME, Metallurgist and Materials Technologist, Engineering Materials and Design and Design Engineering is shown on page 579.

: brochure used to respond to all enquirers of carbon/carbon is shown on page 580.

How should Industry keep up-to-date with material developments?

by M W Commander

Do companies in the UK make any effort to keep in touch with material developments? Is there any need to investigate new materials when established materials will do the job satisfactorily?

Aston University

Content has been removed due to copyright restrictions

The objectives of a material monitoring system are outlined

Avenue, Bury St. Edmunds, Suffolk.

This hot composite is just waiting for the brakes to be taken off

Applications are being sought for carbon/carbon

the diverse disciplines needed by a practising engineer.

Commander started by looking at what had happened to other high technology materials and what efforts had been made to find new markets for them. From this work he was able to draw some general conclusions.

Next he studied the ways in which people found out about the availability of new materials. He sent out a postal questionnaire to a large number of people who had to make

by the chemical vapour deposition process by which the matrix is formed. E

Reply card No. 121

Materials looking for markets

What is the most effective way of bringing the producers of a new material into contact with potential users? This is not so simple as it sounds, since very often the full potential uses are not known to the maker.

When C. McCarthy studied the development were users for titanium carbide identified?— of getting samples into key laboratories

... in materials adoption, what is the best way ... opposite.

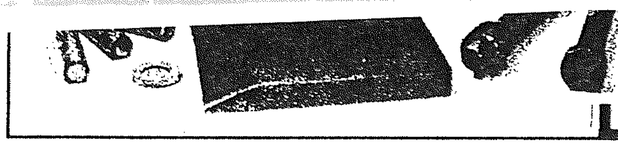
From the Metallurgist and Materials Technologist, March 1978, page 115.

10 ways not to market a new material

The problems facing producers of

DUNLOP

carbon/carbon



Heat-treatment [ ]

Medical [ ]   Nuclear [ ]

Send Cheque or postal order, made payable to Dunlop Limited to:
Dunlop Limited—Advanced Materials Laboratory. Aviation Division, Holbrook Lane, Foleshill, Coventry CV6 4AA

Manufacture of carbon/carbon composite materials using

Manufacture of carbon/carbon composite materials using a chemical vapour deposition process.

DUNLOP

DUNLOP

Manufacture of carbon/carbon composite materials using a chemical vapour deposition process.

PRESENT AND POSSIBLE FUTURE APPLICATIONS OF

A selection of components showing the shapes that are possible from Dunlop

Telex: 31677.

Printed in England

Publication No. L600