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LIGHT METALS

MARCH
1947

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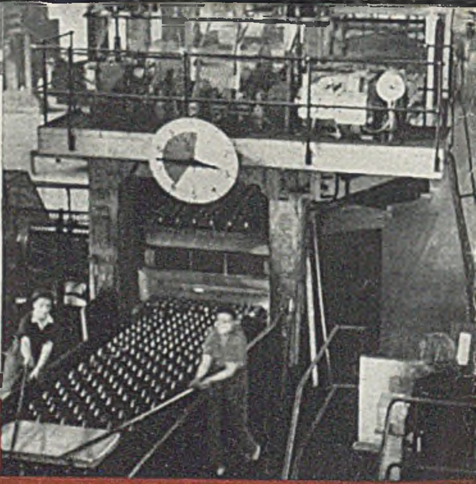

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P.109/47

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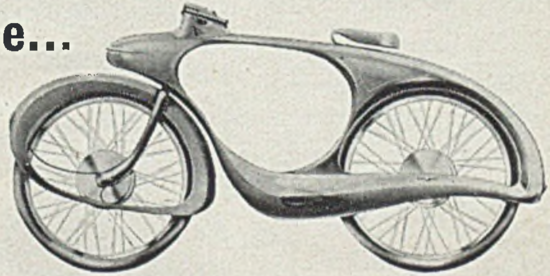

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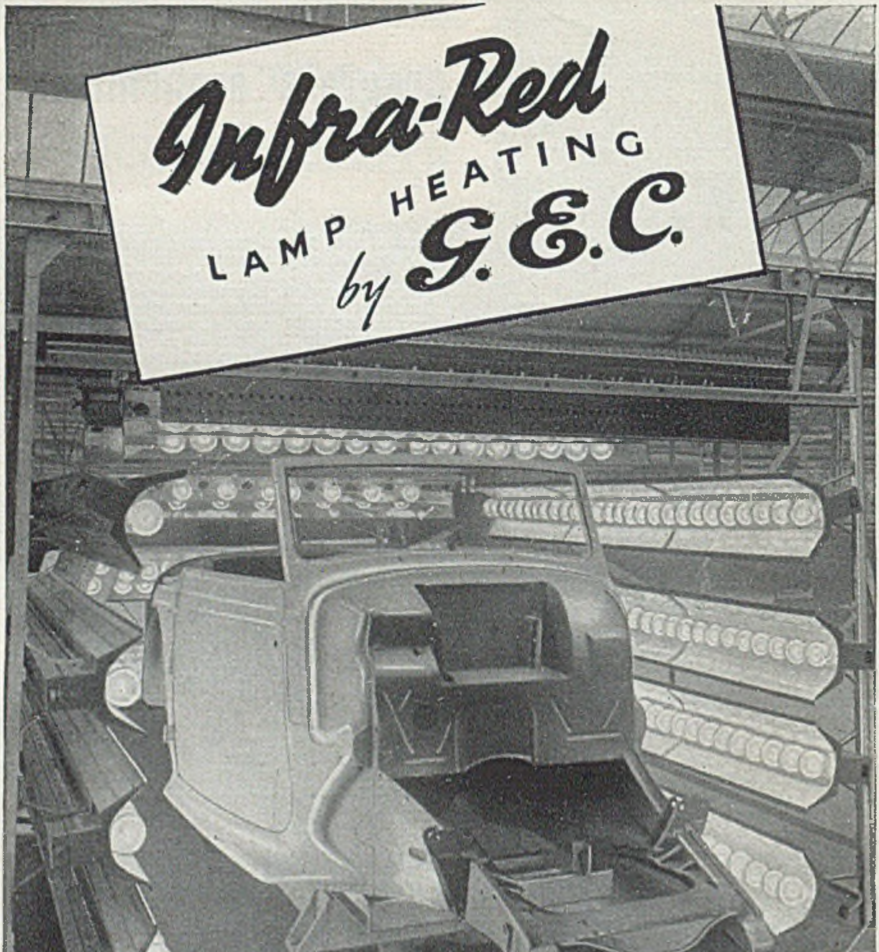
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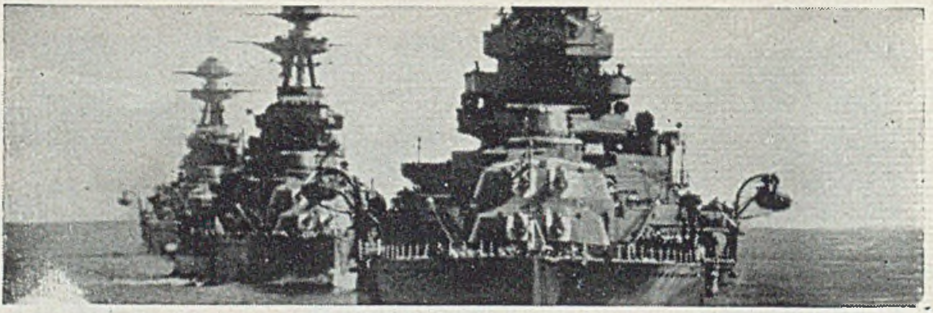


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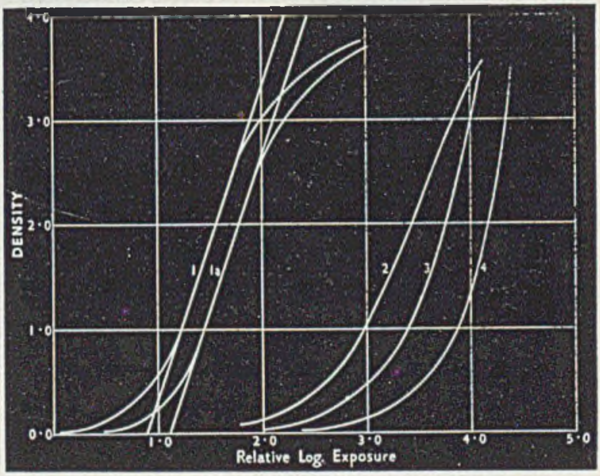

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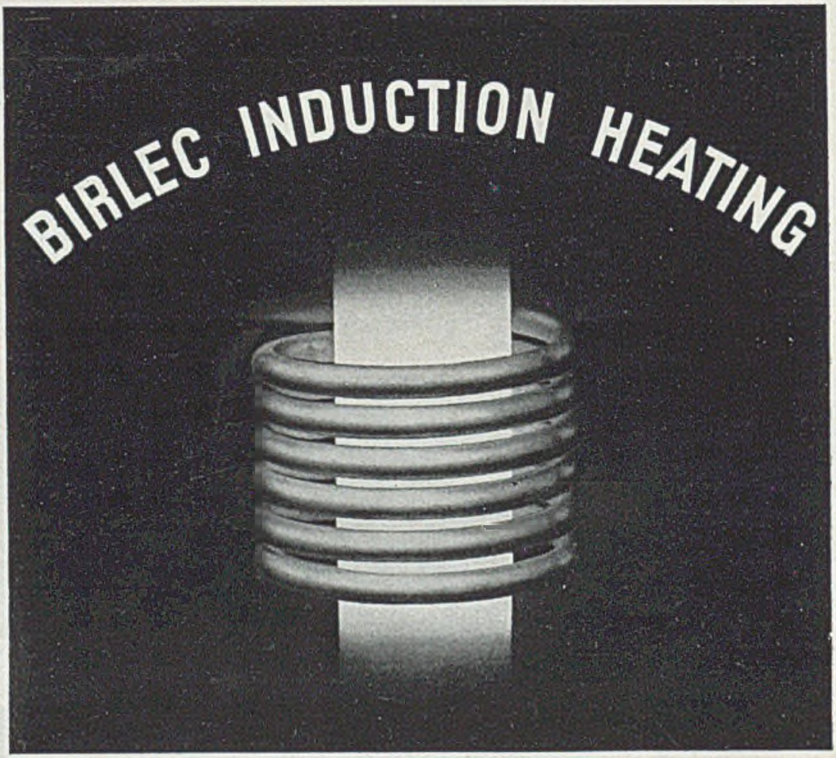

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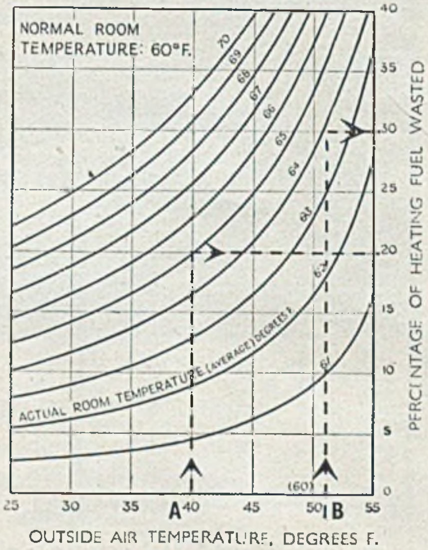

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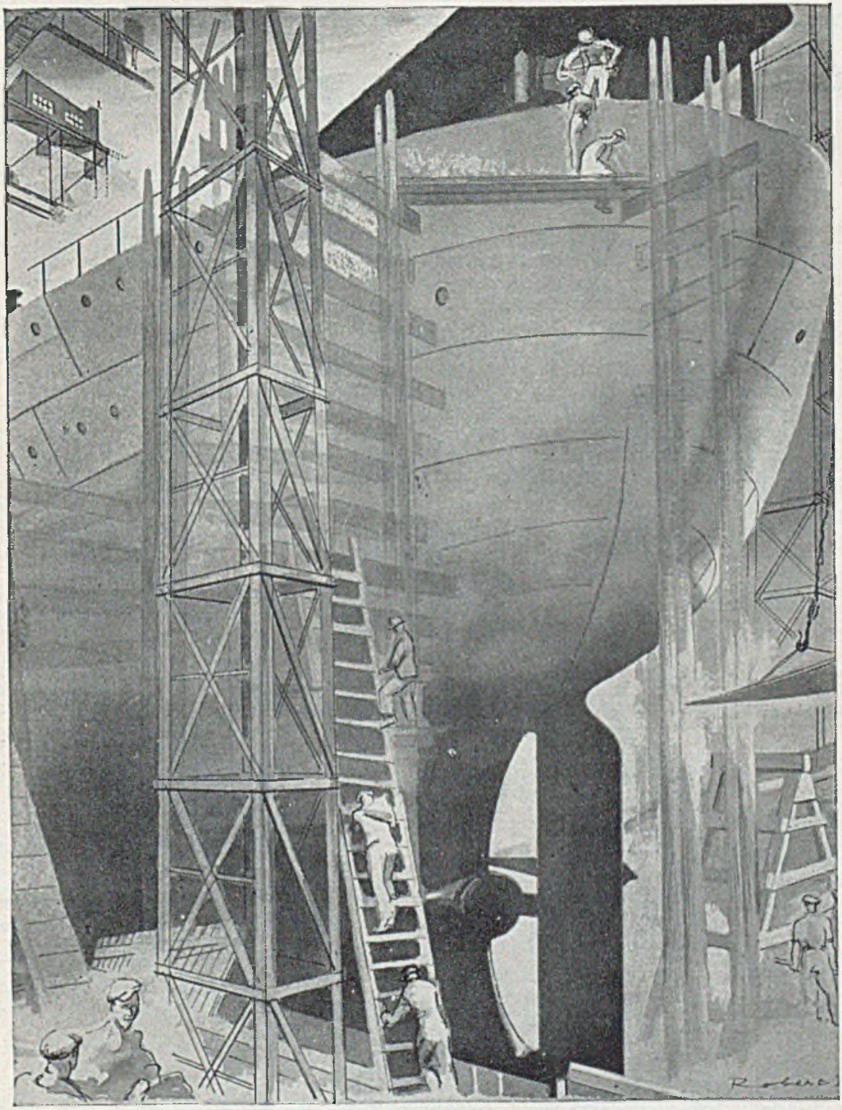

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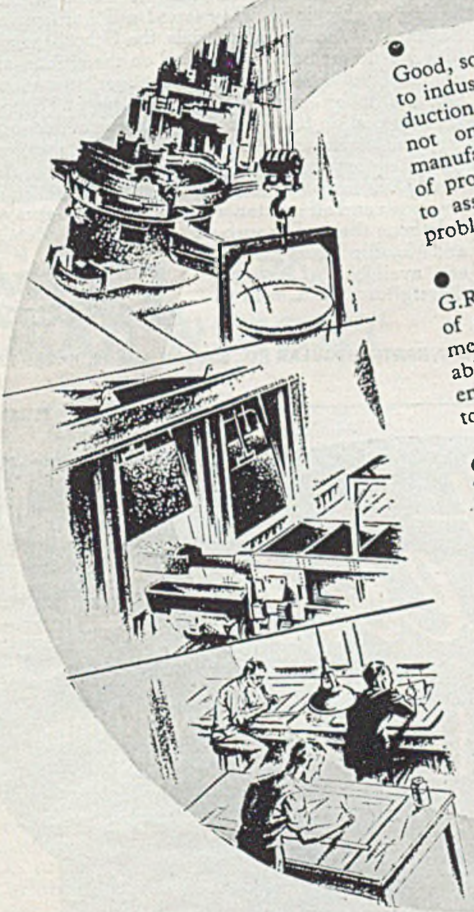

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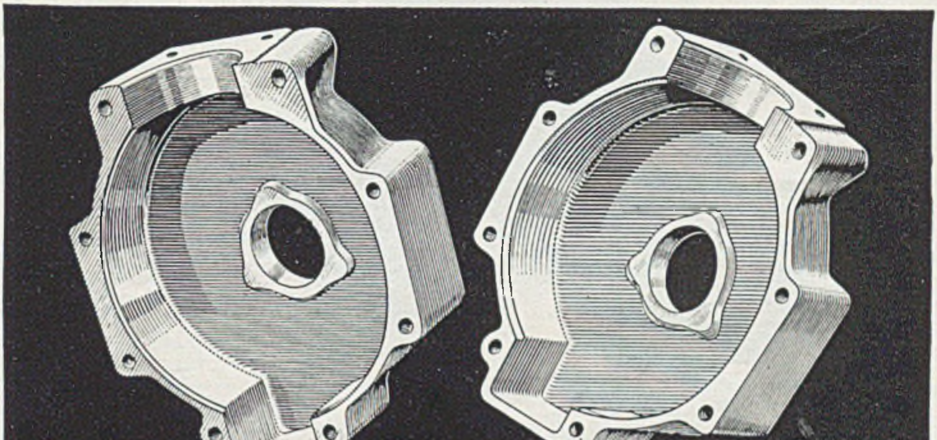

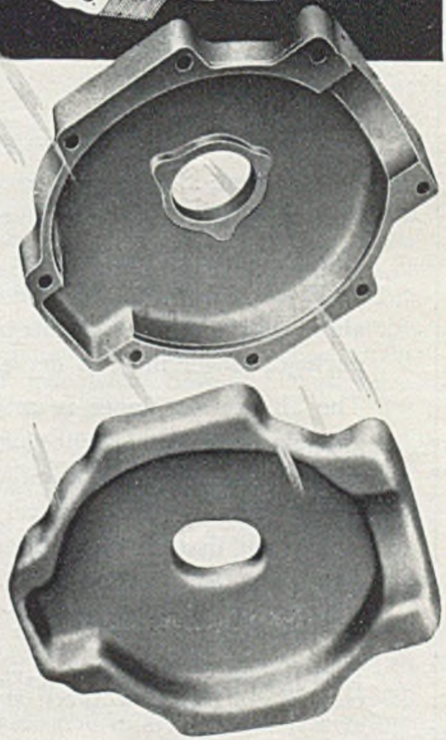

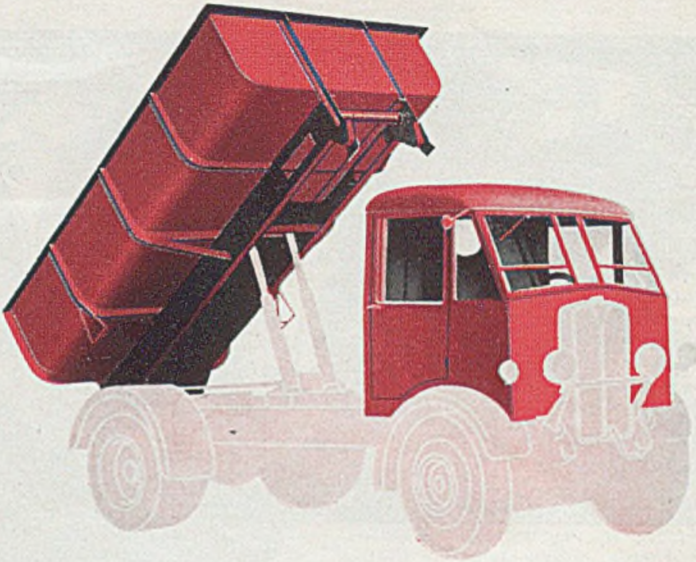

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LIGHT METALS

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Dealing Authoritatively with the Production, Uses and Potentialities of Light Metals and their Alloys

Editor :
E. J. GROOM, M.Inst.MET.

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◆

EDITORIAL OPINION

In a Persian Market

PRICE tickets and fixed charges are, it would appear, institutions peculiar by tradition to Western economy; Oriental commerce (at lower levels, at all events) is still run on the haggling system, with price cutting on the buyer's part as an end in itself, irrespective of the nature of the goods being purchased. All of us have experienced, and still encounter, attempts at similar practice here, although, it must be admitted, the mere achievement of a low buying price is rarely the sole incentive. East and West have, however, this in common—a bargain is always attractive.

In the realm of modern metal-finishing technology, bargain hunting can, on first principles, rarely be considered a worth-while pursuit, for in most cases nowadays, finish is intended to do far more than merely please the eye.

Metal finishing is a science, the aim of which may certainly be, in part, to enhance shelf appeal, but much more often than not, is to invest the metal treated with improved service qualities. For this reason, of the varied and numerous surface treatments which may be given to metal ware, choice is usually made of that which accords best with the work which the item to be treated must fulfil. Details of the selected processes are then arranged to give, where possible, requisite decorative appearance or colour and price range.

If a certain minimum standard of serviceability be required, and if a certain definite level of artistic merit must be attained, comparative cost must necessarily be a secondary consideration. A brush finish with an air-drying lacquer is usually cheaper than a first-class stoved finish, and both, in general, are less costly and of more general applicability than a vitreous-enamel finish, but, in the truly technological sense, neither can replace the other, either as regards appearance or in ability to meet demands in the field; each has its specific merits and sphere of usefulness.

Certain readers have quarrelled with a statement by Frank Taylor, on page 18 of "Light Metals" for January, 1947, in which the author remarks that anodizing is not, contrary to most people's opinion, a cheap process. With equal truth he might have gone on to say that neither is silver plating, or the production of hard chromium finishes. Cheap finishes may frequently be the source of a double danger, that of cheapness in appearance and unsatisfactoriness in service. The decision as to whether or not a certain finish shall be used, cannot, obviously, on quality work, be decided solely on a cost basis; this is particularly true of anodizing. The anodic finish is of a highly specialized nature, whilst the anodized and dyed finish is virtually unique, and represents the only commercial instance of a surface treatment giving a coloured protective layer integral with the base.

To pursue our examination of the offending portions of Frank Taylor's commentary in more detail, he later goes on to say that, in the case of certain simple pressings, the cost of anodizing and dyeing may actually be greater than that of the material and production charges for the work itself. This may quite conceivably be true, but equally true is it to say, that whereas the untreated pressing would probably not be satisfactory for the service required of it, the anodically oxidized article is capable exactly of fulfilling the task assigned to it. The demands to be made may be those of electrical insulation, wear resistance, oil-retaining properties, permanence of reflection characteristics, or even those of a purely decorative type. Now only by anodizing with or without dyeing, can these demands be satisfactorily met at the present time, and if they must be met, then cost becomes, as we have said before, a secondary consideration because the "finish" is actually a part of the job.

For certain specific purposes it is true, where the quantities required will justify the step, continuous automatic plant will obviously be capable of reducing the charges entailed on ordinary batch operations. This is true, however, of any production cycle. Within limits some regulation of cost, too, is possible, by quality control. The preliminary finishing and anodizing of an ashtray do not call for the critical supervision necessary in the case of large aluminium reflectors for optical instruments. Because of this it may even be demonstrated that, for certain cheaper repetition lines, it may be less costly to anodize and dye than to stove enamel.

Contents

"LIGHT METALS" is published in London, England, on the first Wednesday of each month.

METALLIC JOINING OF LIGHT ALLOYS

In the Third Part of this Account, Continued from "Light Metals," 1947/10/108, are Discussed Fluxes for the Soldering of Aluminium, Theory and Practice of Hard Solders and Soldering for Light Alloys, and the Mechanical and Corrosion Properties of Soldered Joints

IN the preceding sections of this article soldering has been considered from the aspect of suitable solders and their ability to alloy and provide a strong bond with aluminium. The primary requirement in all soldering processes is to present to the solder a chemically clean metallic surface, and with aluminium the problem is effectively to remove the tenacious and readily formed oxide layer. As has been mentioned, this can be achieved by friction soldering methods, using either the soldering iron itself or a solder which includes an abrasive material. For soldering metals other than light alloys the established practice is to employ fluxes for cleaning and to promote alloying, and consideration will now be given to these methods as applicable to aluminium.

With regard to fluxes, there is even less positive information available than there is for the solders. Organic materials such as stearine, oleic acid and petroleum jelly are used, but they serve as blankets to protect the soldering during the operation rather than as fluxes. The true fluxes are somewhat complex mixtures of halides. They are deliquescent and corrosive, and must be completely removed by suitable washing operations immediately after soldering. The following represent typical groups of such fluxes:—

(a)	Potassium chloride	1 part by wt.
	Lithium chloride . .	1 ,,
	Sodium fluoride . .	1 ,,
	Beryllium fluoride	1 ,,
(b)	Potassium chloride	6 ,,
	Calcium chloride	3 ,,
	Cryolite	1 ,,

(c) Zinc chloride base fluxes, including ammonium chloride, sodium chloride and potassium fluoride as addition agents in various proportions.

(d) Zinc chloride base fluxes, including sodium fluoride, potassium chloride and potassium fluoride as admixing ingredients.

(e) Zinc chloride base fluxes with ammonium chloride and stannous chloride as addition agents.

(f) Basic aluminium chloride as the base, with potassium fluoride as additive.

These fluxes can be used in the usual way as finely ground powders, or as aqueous solutions or as suspensions in industrial methylated spirit.

These fluxes are very close in composition and nature to those used in the flame welding of light alloys, and, as such, can be expected to be more efficient at the upper end of the temperature range used for soldering. Drawing an analogy with fluxing in the soft soldering of copper, its alloys, and the heavier metals generally, there is no preparation with the fluxing activity of the hydrochloric acid base solutions such as "killed spirit" and aqueous zinc chloride, with their numerous modifications. Again, there is no equivalent to the non-corrosive fluxes of resin base, such as colophony resin in industrial spirit solution, or in the mixed solvent butyl alcohol and carbon tetrachloride. Therefore, in soldering light alloys, either a flux is employed and a subsequent washing operation applied to remove all traces of the flux, or friction soldering has to be relied upon. In the latter case, a "blanket flux" can be used. Petroleum jelly may safely be employed for this purpose. Fatty materials such as oleic acid and stearine should not be used if light alloy is being joined to copper or its alloys, unless the fat is subsequently removed, for example, by means of a solvent. Actually, a resin base flux is sometimes quite satisfactorily used for this purpose.

The soldering operation is assisted by making a separate operation of the preliminary "tinning" of the surfaces to be joined. This is performed by friction soldering with or without flux, or by dip soldering, applying the flux and then inserting the work in a bath of the aluminium solder at the required temperature. It is useful to have the flux mixture molten for this purpose because the work is fluxed and preheated simultaneously and can be quickly transferred to the dip solder pot. In most cases this preliminary "tinning" allows the use of flux and its subsequent removal.

Table 4.—Durability Tests on Friction-soldered Junction without Flux.

	Al-Si alloy section to Al-Mn alloy sheet	Light alloy sheet material, lapped joins, all combinations of Al, Al-Si, Al-Mn, and duralumin	Duralumin strip to copper strip	Al-Mn strip to copper wire
Before testing ..	All junctions had the appearance of neatly executed, sound solderings.			
After testing	The solder became heavily corroded to white corrosion products.	The solder and area around solder, heavily corroded to white corrosion products.	White corrosion heavy at the junction of the solder and the copper; solder became very dull.	Heavy white corrosion developed around the junction; the solder became dull and slightly white with corrosion.
	The junction was very weak and parted under light tension.	Cross-breaking strength at the junction of all specimens negligible.	Junction had become very weak in tension and in cross-breaking direction. Junction had appearance of a dry join, and gave no evidence of proper alloying.	Corrosion was so heavy that the wire fell away from the light alloy upon handling.

Following preliminary tinning of the mating surfaces with aluminium solder, the junction is effected by fixing the parts together and soldering. This fixing together is not always essential, but is very helpful in contributing to a sound joint. In the case of a wire to a tag, mere twisting of the former to the latter suffices. For heavy work, clamping together wtih some pressure is advisable. Lighter sections can be eyeletted or folded together. The parts are then soldered by friction soldering, running a fillet of solder along the junction, or by heat and pressure alone or with additional solder as a fillet. It is preferred that the solder should be of the same composition as the original material used for the tinning, although this is not essential. Actually resin-cored solder of the lead/tin series can be safely and successfully used, but naturally it will not wet and merge with the aluminium solder with the same facility as it will when applying it to a pure tin or tin-lead alloy base. It is, however, very useful for delicate junctions and thin gauges where much heating is not possible.

Where the joining of light alloys to copper rich metals, or to steels, is involved, the surfaces of these metals should be pre-tinned in the usual way, not necessarily with pure tin, but, at least, with a tin-lead alloy. The junction can then be made to the "tinned" aluminium in the usual way, using aluminium or tin/lead solder.

There is a strong tendency to produce

merely a "dry joint" in soldering without flux. This tendency is promoted by the reluctance of the solder to alloy with the light alloy, as well as by the presence of the oxide film. In practice, every effort is made to build a fillet of solder around the join to enhance strength. In this connection, a number of junctions made, using a solder containing 33 per cent. of zinc, 0.6 per cent. lead, remainder tin, were examined. The aluminium alloy was pre-tinned with this solder alloy by friction soldering. Copper was pre-tinned with soft solder. The parts were clamped together for the actual soldering, and were pre-heated before soldering with an iron. The junctions were examined before and after subjecting to W.T. Spec. K.110 durability test, comprising one dry and two wet cycles. The dry cycle consisted of six hours at 71 degrees C., cooling off in the test cabinet, and two cycles each of six hours at 60 degrees C. and 100 per cent. humidity, cooling off in the test chamber for the remainder of the 24-hour day.

The solderings before and after the test conditions were assessed as set out in Table 4.

In contrast with the foregoing, markedly superior results were obtained by giving special attention to the friction soldering. For example, junctions of light alloy to light alloy and to copper were examined. The solder in this case analysed 43.0 per cent. zinc, 1.3 per cent. silver and the remainder tin. The copper components were preliminarily tinned. The aluminium components were cleaned with abrasive, coated with stearine and heated. They were then tinned with the aluminium solder, which was applied by means of a soldering iron, and rubbed until a mirror-like surface was procured. The parts so prepared were assembled in position and clamped. They were then heated through the outer surfaces with the iron. Specimens prepared in this way and submitted to the same cycles of the above durability test were evaluated as set out in Table 5.

Table 5.—Durability Tests on Friction-soldered Junctions with Flux.

Aluminium to copper strip	Aluminium to copper wire	Aluminium to aluminium
White corrosion developed at all junctions.	White corrosion at the junction.	White corrosion at the junction.
The joint was still quite firm, the fillets of solder at the junction still being sound.	The wire still held firmly.	The junction was still strong and required considerable effort to break.
Upon peeling the copper strip away, blackening was found to have occurred at the interface, indicating that the joint was not properly made over this area, although sound around the periphery.	The wire could be peeled away, mainly by virtue of its greater strength than that of the solder.	—

* All these samples were noticeably superior to those previously detailed.

In view of the contrast in quality of the joints made with these two solders, as well as the fact that the second series still left something to be desired, a further study was made using the 33 per cent. zinc alloy. Meticulous care was taken with the first stage of friction soldering in order to give maximum opportunity of ensuring a metal-to-metal junction between the solder and the base metal.

The first raw material used was aluminium, 0.080 in. thick and approximately 1 in. wide. This material, thoroughly degreased by means of trichlorethylene, was cleaned with abrasive, and then "tinned" with the aluminium solder over an area (and edges) to make a junction with approximately 1-in. overlap. The tinned areas were held together and joined by heating with an iron, and by building a fillet around the periphery.

Half of these samples were submitted to the one dry and two wet cycles of the durability conditions of W.T. Spec. K.110. White corrosion resulted, heaviest on the solder and where abrasive cleaning had extended. The junctions were still firm.

All the test pieces were then broken in a tensile machine. It will be noted that, due to the lap, the pull was not purely in tension, but there was some cross-breaking component. Detailed results are given in Table 6. They show no deterioration due to the durability test, and indicate that all junctions were sound. Only one specimen came adrift at the lap, and this showed generally clean mating surfaces, with little white corrosion on the interfaces. It was considered that a good soldering job had been performed, and also that the good fillet of solder around the junction had protected the lapped join from interfacial penetration of moisture during the exposure test.

A second specimen tested comprised two strips of magnesium-containing light alloy, ⅛ in. thick and 1¼ ins. wide, soldered to a lap join with about 2-in. overlap. After soldering, the test piece was twisted without the joint showing any evidence of failure. Subjection to the durability test caused fairly heavy white corrosion products to form over the solder and adjacent to the soldered junction. The junction could then be peeled apart, but not by any means easily, by a longitudinal pull. Some penetration of moisture had occurred during the test, about 25 per cent. of the interfacial areas having whitened. The remainder of these areas were metallically clean.

Two pieces of sheet of the same material, one bent to an angle, were soldered together, providing a good fillet of the solder. General slight whitening occurred in the corrosion test, with heavy whitening over the solder. No strength deterioration resulted, and the pieces could not be peeled apart by hand.

A piece of the same sheet material was friction soldered or "tinned" over one-third of its surface and subjected to the durability test. Corrosion occurred generally, but was heaviest on the solder. The latter could not, however, be peeled off the surface.

Again, a piece of this material was soldered to a piece of 60/40 brass strip, bent to an angle. In this case the surface of the light alloy was first friction soldered and the brass tinned with an ordinary tin/lead solder. The two were held together and then soldered, using resin-cored solder of composition tin 60 per cent., antimony 1 per cent., lead 39 per cent. Under the durability-test conditions, the tin/lead solder alloy remained quite bright, although any areas of uncovered aluminium solder became fairly heavily corroded to white corrosion compounds. The light-alloy sheet became fairly generally corroded to a white film or spots, but it was noted that quite marked corrosion products, mainly white or grey in colour, were produced on the brass. The two pieces of metal were pulled apart, but only by holding in a vice, and with great difficulty. The mating surfaces were quite clean. The use of the ordinary soft solder for the fillet was considered very beneficial, and it no doubt contributed appreciably to the maintenance of the strength of the junction by its moisture-sealing properties.

The composition of the aluminium alloy used in these tests was: Magnesium, 2 per cent.; manganese, 1.5 per cent.; silicon, 0.3 per cent.; iron, 0.2 per cent.; aluminium, remainder.

Table 6.—Tensile Strength Test Values on Soldered Aluminium Strip Specimens.

Sample No.	Condition	Tensile breaking load, actual, lb.	Strength, lb./sq. in. of cross-section	Strength, lb./sq. in. of lap area	Nature of break
1	As soldered	1,030	13,000	1,037	In the aluminium strip
2	As soldered	1,008	14,000	995	In the aluminium strip
3	As soldered	1,020	13,400	1,025	In the aluminium strip
4	After durability test	1,123	13,400	996	In the aluminium strip
5	After durability test	1,133	13,860	906	Soldered lap came apart*
6	After durability test	1,182	14,080	1,057	In the aluminium strip

* Note.—This sample had the longest lap (1¼ ins.) and therefore the greatest cross-breaking component.

In the same investigation, the use of the aluminium solder was considered on aluminium-silicon alloy castings. In one case a hollow casting of wall thickness about $\frac{3}{16}$ in. was cut in two and the mating surfaces trued. They were "tinned" with the aluminium solder, then clamped together and soldered with this same aluminium solder. The junction was quite strong, but after the durability test the two pieces came apart under applied shock. Regarding corrosion, the aluminium-silicon alloy became generally white, with fairly heavy corrosion along the line of the join.

The composition of the aluminium-silicon alloy was as follows: Silicon, 10.8 per cent.; iron, 1 per cent.; copper, trace; manganese, traces; aluminium, remainder.

The general conclusions to be drawn from these experiments are as follow:—

(1) Sound, aluminium-soldered junctions can be made in the light alloys.

(2) To achieve the best possible results, pre-tinning by friction soldering is necessary and must be efficiently performed.

(3) A good fillet of solder around the periphery of the junction is desirable.

(4) Ordinary tin/lead soft solder as the fillet-producing material is beneficial.

(5) The tin/zinc type of solder corrodes under moist conditions to a greater extent than the light alloy.

(6) If coppers or brasses are soldered to light alloys, these, too, may corrode under damp conditions by virtue of the bimetallic or multi-metallic junction.

The adverse bimetallic effects can be mitigated, and often fully negatived, by varnishing or finishing with other organic media, such as paint, enamel or lacquer. The organic medium must be appropriately chosen to exhibit good adhesiveness and a high impermeability to moisture.

When the junction is in tension (solder in shear), it has been shown that good strength, equal to that of the base material, can be anticipated. Proof against vibration and shock has not been established, and the same good results cannot be expected if the joint is exposed to cross-breaking stresses or to anything that can promote "unpeeling."

Where thin gauges are concerned, two opposing factors have to be given attention. The actual strength of the thin material is lower, inversely proportional to thickness, and the strength of the soldering is approximately the same as that when used to join thicker material. From this angle, properly executed solderings should be quite satisfactory, especially if they are designed to take the strain in the plane of the junction rather than at right angles to it, and to be more reliable than joints in thicker gauges. Against this is the greater difficulty associated with cleaning thin material by abrasive means, and in friction soldering, due to the intrinsically low strength of the material. Very thin aluminium foils, such as those employed in fixed paper condensers (thickness usually about 0.0002 in. and generally less than 0.0010 in.) are thus not sufficiently strong for these processes to be applied. They can, however, be soldered when supported. The thicker foils, such as used in electrolytic condensers, acoustic diaphragms and other applications, can be dealt with satisfactorily because they have sufficient strength to withstand the mechanical operations.

Until research indicates sound reasons for complex alloy compositions, a straight tin/zinc binary alloy with a small pasty range is recommended for friction soldering. It is considered that the alloy of 70 per cent. tin, 30 per cent. zinc, having a pasty range from 197 degrees C. to about 250 degrees C., is a satisfactory choice for thin-gauge materials. It should be operated at a temperature of 270 degrees C. to 300 degrees C. For the actual soldering of the "pre-tinned" parts, a eutectic composition is advocated, preferably erring on the tin-rich side of this composition. The alloy of 91 per cent. to 92 per cent. tin, 8 per cent. to 9 per cent. zinc is suggested where it is desired or advisable to use the same composition as for "tinning." Otherwise, the eutectic tin-lead alloy is suggested, and there is slight preference for it. . Again, any deviation from the eutectic should be on the tin-rich side, e.g., 63 to 65 per cent. tin, 35 to 37 per cent. lead. This composition can advantageously be used in the form of resin-cored solder wire.

The surfaces to be joined by soldering require, after pre-tinning, holding together rigidly for the soldering proper. Light-gauge parts can be held by eyeleting or riveting (using, of course, light-alloy eyelets or rivets), or in simple clamps. Again, there is no reason why they should not be dealt with in the usual type of soldering fixtures, in which the controlled heating is applied between electrically heated carbon electrodes. There is the possibility of developing several procedures with tools of this type. For example, a thin strip or washer of the tin-lead eutectic solder can be included between the pre-tinned surfaces of parts to be joined, these surfaces having been thinly coated with resin flux in suitable spirit solution. Again, a "tinning paste" may be used, this consisting of the triturated solder powder in emulsion with resin in a suitable carrier (e.g., butyl alcohol). Such a paste is applied by brush to the pre-tinned surfaces.

Any of these procedures are applicable to parts previously prepared by a thin or thick copper plating, by a combination of copper and tin plating, or by a dual copper/silver plating. The sodium zincate pretreatment procedure for the plating is satisfactory for this soldering, and all details were given in previous issues of this journal. It is quite practicable to electro-plate components, small tags, or even strip in thin gauges, but economics have to be considered, although with quantity production there is often available the means to render such platings both practicable and economic.

Using the resistance electrode procedure for the soldering heat, it is necessary to hold the parts under the electrodes until they have cooled below the eutectic temperature of the solder employed before the pressure is released. Water-cooled electrodes assist here.

This suggests another apparatus using, for example, a pneumatic press of appropriate size, electrical resistance heating, water cooling, and all operations worked out to a balanced cycle with automatic control. In this method, the surfaces to be joined are pre-tinned. In the case of the light alloys, electro tin plating is suitable. This involves, for example, a preliminary copper plating followed by tin plating from sodium stannate solution. A thin layer of nickel is usually advantageous between the copper and the tin because it retards the rate of diffusion of tin into copper during the joining process, and ensures maximum soundness of join. In the soldering process itself, the two surfaces have to be accurately located in the press so that when the two electrodes are brought together the surfaces are truly in contact. Floating or spring loading of the electrodes assists in providing this accuracy. The electrodes are so designed as to concentrate the heat where required for the joining to the two surfaces; and they may be of hard, high-resistance carbon, or chromium-plated mild steel, or a combination of these according to the nature of the joint to be made. They are held by the tubular water-cooled copper electrodes proper, which are fixed, one to the base of the press, and one to the ram. Control, by means of a single press button, gently brings the parts together between the electrodes, applies a predetermined pressure, a brief heating cycle of predetermined wattage, this followed immediately by a cycle of pressure alone during which cooling occurs, and finally brings the electrodes apart.

The thickness of tin coating required for this type of joining is from 0.0003 in. to 0.0020 in., according to the nature of the parts and the accuracy of flatness of the surfaces. A very thin film of resin flux is helpful in the more difficult cases. There is no reason why this procedure

Table 7.—Composition of Aluminium Flux-cored Hard Solders.

Sample No.	1	2	3
Alloy composition %—			
Aluminium	95.0	100.0	—
Copper	5.0	—	32.0
Silver	—	—	51.5
Zinc	—	—	16.5
	100.0	100.0	100.0
Flux content %	17.5	15.5	15.0
Composition of flux	Chlorides and fluorides of sodium and potassium	Chlorides and fluorides of sodium and potassium	Borax and boric acid

should not be applied for soft soldering of light alloy parts having pre-tinned surfaces.

Instances have been reported of the soft soldering of very thin aluminium foils. In these cases, the foil in its assembly has been secured to Bakelite paper-laminated sheet with synthetic adhesive, and has formed part of an electric circuit. Copper leads have been soldered to such aluminium foil which in this condition will withstand friction soldering. Direct soldering with resin-cored solder and a paste-resin flux has been reported to be successful, but no important industrial applications have as yet been noted.

In the case of fine wires, these can be cleaned with abrasive paper, twisted together and friction soldered with aluminium solder, the 70/30 tin-zinc alloy having been successfully used for this purpose. For aluminium to copper joints, the copper wire must be pre-tinned. However, it is to be noted that the solder will not flow between the turns of the twists, but can only be superficially applied by friction methods. Good electrical junctions can be made in this way, despite the fact that only a sheath of the solder alloy with soldering proper only on the peaks of the wire, is provided.*

Hard soldering is applicable to all but the thinnest of foils. It is virtually the same as flame welding, but has the advantage that the hard solder can be obtained with a core of flux. Flux has to be removed after this form of joining, as with all other methods, otherwise corrosion to destruction occurs. Hard soldering is effected, using a torch in the same way as in the brazing of steels, copper and its alloys. The torch chosen should give the smallest and coolest flame appropriate to the particular job undertaken.

Table 7 gives test results on a number of hard solders. Usually it is advisable to choose an alloy of similar composition to that of the materials being joined, but this is not always possible; for example, when joining dissimilar metals. It will be seen that one of the materials mentioned (No. 3 in Table 7) is an ordinary copper/zinc/silver solder alloy; there seems to be no purpose in using this unless one of the base metals concerned is other than light alloy, e.g., copper, brass, etc.

In general the use of hard solders is not advocated because of the difficulties in removing the flux, particularly with thin gauge material. When this problem can be satisfactorily solved, successful joining can be assured. For example, joints made with the hard solders No. 1 and 2 (Table 7) in thin-gauge aluminium were examined by dissolving in 5 per cent. caustic soda solution. No selective attack by this reagent occurred, the samples gradually dissolving from the outside inwards, and no voids or discontinuities in the junctions were revealed.

Consideration of the Bimetallic Junction

One objection to the soldering of aluminium alloys is the unavoidable formation of a bimetallic junction. Much has been made of this shortcoming, and, admittedly, it is very right that serious

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* See "Light Metals," 1946, 12, 671. Italian experience showed that with heavier gauges, individual tinning of each strand was essential.

consideration should be given to this feature. At the same time, it is very wrong to allow this consideration alone to prevent the use of soft soldering as a method of joining. Truly under humid conditions local action can occur due to the electrolytic cell that is formed, and, moreover, under some adverse circumstances, the resultant corrosion may be very damaging. On the other hand, there are circumstances when no such action occurs, others when its occurrence is of little consequence, and many others in which the bimetallic junction can be safeguarded by means of a protective finish.

Bimetallic junctions are, of course, produced in the soft soldering of the other metals, steels, copper and its alloys, etc. Rarely is consideration given to this fact in practice, and the limelight that is

Table 8.—Electrode Potentials against Normal Calomel Cell for Various Metals in Artificial Sea Water at 25° C.

A. Base Metals—Irons and Steels

Metal	Potential V.
Pure iron	0.46
Mild steel	0.59
Medium carbon steel	0.42
Silver steel, carbon 1.1%	0.50
Average	0.49

B. Base Metals—Coppers, Brasses and Bronzes

Metal	Potential V.
Copper	0.25
Brass	0.26
Phosphor bronze	0.22
High tensile brass	0.26
Aluminium bronze	0.26
Average	0.25

C. Base Metals—Nickel and Nickel Alloys

Metal	Potential V.
Nickel	0.24
Monel metal	0.24
Cupro-nickel (70/30)	0.22
Nickel silver	0.24
Average	0.24

D. Base Metals—Aluminium and Aluminium Alloys (Copper-free)

Metal	Potential V.
Aluminium	0.80
Aluminium/silicon alloy	0.80
Aluminium-magnesium alloy	0.82
Average	0.81

E. Base Metals—Aluminium Alloys containing Copper

Metal	Potential V.
Duralumin	0.62

F. Jointing Metal—Ordinary Soft Solders and Their Ingredients

Metal	Potential V.
Soft solder (50/50 tin-lead alloy)	0.50
Tin	0.40
Lead	0.60

G. Jointing Metal—Silver Solders (Brazing Alloys)

Metal	Potential V.
Silver	0.02
Copper	0.25
Zinc	1.05
Tin	0.40

H. Jointing Metal—Brazing Brass

Metal	Potential V.
Brazing brass	0.26
Copper	0.25
Zinc	1.05

I. Jointing Metal—Copper

Metal	Potential V.
Copper	0.25

J. Jointing Metal—Aluminium Soft Solders

Metal	Potential V.
Tin-zinc (80/20) alloy	1.00
Zinc-cadmium alloy	1.04
Tin-zinc-cadmium alloy	1.00
Tin	0.40
Zinc	1.05
Cadmium	0.76

K. Jointing Metal—Aluminium Hard Solders

Metal	Potential V.
Aluminium hard solders	0.60 to 0.82
Aluminium	0.80
Silicon	—
Copper	0.25
Zinc	1.05

Notes.—(1) The values given are measured values. (2) The above values are all negative. (3) Of two metals in contact, the one having the more negative value—i.e., the higher numerical potential—will tend to corrode. (4) It should be pointed out that, in practice, service behaviour may show marked discrepancies from that which might be assumed from laboratory data of thi type. In particular, precise conditions of exposure—i.e., to air, water, spray or soil attack—and initial surface condition (painted, degreased or otherwise) markedly influence results.

thrown upon the same type of joint in the aluminium alloys is certainly missing. Some thought will therefore be given to this phase of the subject.

In practice, any corrosive action that may develop will be dependent upon the nature of the metals forming the junction; the physical compactness, porosity and surface roughness; the conditions of temperature, humidity and atmosphere with respect to corrosive agents such as sulphurous compounds, salt, acidic bodies, dirt, dust, etc.; and the tendency for either or both metals to form passive coatings or to lose passivity under these conditions. A good guide, although not always a certain one, to the extent of trouble likely to arise from bimetallic disparity, can be obtained from a study of the electrode potential of the metals in sea water. The greater the difference in the values for metals in contact, the greater the reaction that is likely to occur between them under damp or otherwise adverse conditions. The metal with the more negative potential will be more susceptible to corrosion.

Electrode potentials for the solders and the materials for which they are employed are given in Table 8. Under conditions of hermetic sealing or with air conditioning, when conditions of dampness and/or corrosive chemicals can be eliminated, no trouble from bimetallic junctions is to be expected. When condensation is likely, and salt, as in marine atmospheres, possible, the potential difference between metals in contact must be as small as possible, and certainly not more than 0.25 volt. For conditions intermediate between these circumstances, 0.50 volt difference may be tolerated. When voltage differences greater than these maxima are unavoidable, the junctions should be protected by varnishing or other means.

Comparing the potentials of copper and zinc with that for brass, it will be seen that copper is the predominating factor, and that zinc does not raise the value for brass. The same obtains with nickel silver, but, on the other hand, copper lowers the value for aluminium in the alloy duralumin. In the case of soft tin/lead solder, it will be seen that the potential is the mean of those for tin and lead. These points stress the importance of determining the values for the metals concerned rather than estimating them from the values for the constituent elements. They also indicate that any values should be considered with caution for conditions other than those due to salt or humid atmospheres.

From a study of the figures in Table 8, it will be seen that the soft solders harmonize well with the steels, giving practically no potential difference. With copper, brasses, bronzes, nickels and nickel alloys, the potential difference is about the 0.25-volt limit, which is quite satisfactory.

With the copper-free aluminium alloys, the aluminium solders show a difference of about 0.20 volts, which is very satisfactory. With the copper containing alloys, they show a disparity of about 0.40 volts, which is rather high, but still not considered bad except under the worst conditions. The tin/lead soft solders are very close to the aluminium copper alloys, and about 0.30 volts from the copper-free alloys. It is interesting to see how the zinc constituent is the predominating factor in the aluminium solders with regard to this potential.

Regarding hard solders, the silver and brazing materials are likely to be within 0.25 volts of steel, and similar to the value for copper, brasses, etc. The silver solders are too far removed from the aluminium alloys (copper free) to be safe, but are closer to the copper containing aluminium alloys. The true hard aluminium solders give little or no difference from the values for the aluminium alloys.

Summarizing briefly, if the recognized solders are employed for joining aluminium alloys, potential differences created are no greater than those established with the heavier metals, where soldering has been employed as a recognized joining process for years. The bogy of potential difference, although

Table 9.—Electromotive Series of the Metals.

Metal	Potential V.
Potassium	+2.6
Sodium	+2.4
Barium	+2.6
Strontium	+2.5
Calcium	+2.4
Magnesium	+1.3
Aluminium	+1.0
Manganese	+0.82
Zinc	+0.51
Iron	+0.2
Cadmium	+0.2
Cobalt	+0.05
Nickel	−0.02
Lead	−0.12
Tin	−0.14
Hydrogen	−0.24
Arsenic	−0.53
Copper	−0.58
Bismuth	−0.63
Antimony	−0.71
Mercury	−0.99
Palladium	−1.03
Silver	−1.04
Platinum	−1.10
Gold	−1.7

Notes.—(1) The figures quoted are single-electrode potentials of the metals in solutions of the gram-equivalent of their ions. They represent the charge of the *solution*—i.e., a positive value indicates a tendency for the metal to go into solution to produce this value ; a negative value shows a tendency for ions to leave the solution and to deposit on the metal. (2) Of two metals in contact, the one having the more positive potential in the list will tend to corrode, and the potential between them which accentuates this corrosion is the difference between the values given.

demanding proper attention, must not be advanced as an argument against soldering aluminium. Such an unfounded criticism only serves to fog the issue.

The electromotive series of the metals has been included in Table 9, because some of the elements mentioned in the aluminium solder compositions have not been included in Table 8. Their omission is due to absence of data. The single electrode potentials suggest that manganese is a safe addition, but that bismuth, arsenic and antimony should be regarded with suspicion.

It should be indicated, however, that every caution is required in the practical interpretation of the data given in Table 9. In the field, bimetallic couples are rarely free to manifest their properties unhindered, and frequently one is dealing, not with true metallic couples, but rather with, say, a metal in conjunction with a metal oxide; aluminium and chromium are cases in point here.

(To be continued.)

MAGNESIUM MOULDING SANDS

A Brief Survey of Magnesium Casting Technique by F. A. Allen, A.I.M. A New Method of Preparing Synthetic Moulding Sands is Suggested and Offered for Further Research

THE molten-metal technology of magnesium-base alloys is based upon the necessity of combating the somewhat vigorous oxidation to which magnesium alloys are subject at elevated temperatures. Protection from the air must be afforded during melting, and also from the air together with the moisture of the mould, in the process of sand casting. If molten magnesium alloy is not so protected, melting losses will be high and, indeed, undesirable compositional changes will occur, since it appears that the magnesium oxidizes preferentially to the remaining alloying constituents.

In extreme cases, of course, actual burning takes place. The presence of moisture in the moulding sand introduces a further complicating factor, for magnesium at high temperatures reacts with water, giving hydrogen and magnesium oxide. Unless this reaction is restrained, unsoundness in the casting results, even if violent evolution of hydrogen does not cause more or less dangerous explosions. When inhibition of the reaction is incomplete—allowing the action to proceed in some measure—local increases of temperature at the mould face are sufficient to cause the formation of what appear to be combination products of metal and silica.

Molten-magnesium alloys are protected during melting, and whilst they are in the molten condition, by a fused flux cover, consisting of mixtures of salts, chiefly chlorides and fluorides, of which anhydrous magnesium chloride usually forms the largest part. The composition of the flux is adjusted to give a suitable melting point; preferably, the flux should be molten at a temperature below that of the final fusion point of the alloy, so that by the time the alloy is completely molten the flux that has been added during the initial heating period can form an isolating cover. Physical properties, such as specific gravity and viscosity, are also important.

Opinions differ as to the requirements under these heads, but all are agreed that definite separation of flux and molten metal is essential, for if separation is not achieved contamination of the metal with chlorides is inevitable, serious corrosion soon resulting. It was, indeed, just this contamination that was responsible for the ill-repute of magnesium-alloy articles at the commencement of their industrial application. Thanks to greater understanding of the problem and modern specialized fluxes, these days are long past; this unfortunate history is only mentioned again to serve as a cautionary word to those founders who contemplate magnesium-alloy founding as the material becomes economically attractive.

The normal practice of superheating magnesium alloys to refine the grain, underlines the need for efficient isolation during the whole melting process, as a temperature of 900 degrees C. is reached. It is well known that the speed of a chemical reaction, of which oxidation is a special case, of course, is greatly increased with increase of temperature. When the metal has cooled to pouring temperature, the flux cover is prevented from entering the mould with the metal stream either by the removal from the metal surface or because its physical properties are so distinct from those of the metal there is little tendency for the cover to move forward as the pot is tilted. In both these cases, however, some transient protection from oxidation must be applied as the molten metal passes through the air in its passage from pot to mould.

In the first method of flux removal, temporary protection is afforded by the application to the molten metal of sulphur, which successively melts, vaporizes and burns to form sulphur dioxide. This gas restrains oxidation but does not completely prevent it, for burning, once commenced, cannot be stopped by the application of sulphur. In both pouring techniques indicated above the actual metal stream is dusted with sulphur as it passes through the air.

This brief discussion of molten magnesium-alloy practice will serve to introduce the main subject of oxidation inhibitors for moulding sand, during the development of which thesis will be described a novel method for the incorporation of an inhibiting substance having, in addition, it is believed, considerable practical advantages.

In addition to free oxygen in the air contained within the mould cavity, from which the molten magnesium must be protected, the sand of the mould contains moisture, and against this, too, protection must be provided. Control of moisture content of the moulding sand would, therefore, seem to be a fundamental requirement, but in practice strict control of sand for magnesium-alloy casting is necessary for other considerations also. For example, owing to the low specific gravity of the molten metal, comparatively little hydrostatic pressure is exerted on the air or gases contained within the mould, and a mould of the highest gas permeability must be achieved in order that back-pressures will not build up, hindering the metal from flowing into sharp corners or restricted sections of the mould.

Permeability values of the order

required can be obtained by the use of "synthetic" sand, consisting of silica sand of regular grain size with an addition of a clay-like substance to give the degree of plasticity necessary for moulding. Bentonite or a native china clay is added in the correct proportion to dry silica sand and, after intimate mixing, a known amount of water is added. By this method, therefore, moisture is controlled at some low figure, whereas in a natural sand the moisture may vary within wide limits.

Natural sands may contain up to about 10 per cent. water, and this precludes their use, and would do even if they were sufficiently permeable—which they are not. In contrast, the use of the so-called synthetic sand allows for a sand mix containing the minimum amount of water consistent with producing a mouldable sand. The amount of water necessary to achieve these conditions is normally about 3.5 per cent.

Many substances have been proposed for incorporation with the sand as inhibitors of the water-magnesium reaction. The use of sulphur and boric acid, either together or singly, are well known in this connection. Fluorides, particularly that known as ammonium bifluoride, have also a wide use. Such addition agents act in either of two ways: by producing a gas less reactive to magnesium than air, or by forming a continuous surface film on the molten metal and so protecting it from oxidation. Incidentally, the necessary use of these substances unfortunately results in the fume nuisance of magnesium-alloy sand foundries, and, as far as is known up to the present, no inhibitor has been proposed which is odourless and yet effective. Misguided attempts to use nitrogen as a displacer of air from the mould have only provided confirmation of the fact that magnesium readily combines with this element.

Synthetic sands containing the inhibiting additions indicated above have been successfully used for many years. Quite apart from their effectiveness in restraint of oxidation, however, these sands suffer slightly from a disadvantage from the moulder's point of view. Moulders accustomed to working in natural sands, as commonly used in aluminium or iron foundries, complain that silica-sand mixes are dry to work. It must be accepted that there is point in this practical criticism, and some probationary period, in which he may attain the correct " feel " for the material, must be allowed the moulder new to the magnesium field.

In further consideration of this aspect of magnesium moulding sands, it is generally accepted that the sand also dries quickly. That is, because of the low moisture content, the high permeability and the nature of the sand, an open mould loses water rapidly by evaporation at the mould surface, with the pronounced tendency to surface damage by crumbling. Moulds for magnesium-alloy casting should not remain open for long periods on the floor, but should be closed and poured as soon as possible. It would appear that moisture is more loosely held in the synthetic mixes than in naturally bonded sands.

Silica-sand mixtures detailed in the American Press frequently show the addition of small amounts of hygroscopic substances, of which glycerine is an example. Such additions are, presumably, made to combat excessive drying by reason of their water-absorbing property.

Arising from the foregoing discussion, it will be appreciated that the use of artificially bonded moisture-controlled silica-sand compositions, though necessary to the art of magnesium-alloy sand casting, nevertheless, brings attendant disadvantages. It is believed that the problem may be solved simply, bringing sand technique for magnesium alloys in one step near to the position enjoyed by aluminium and other non-ferrous moulding.

It has been found that, when solid boric acid and solid ammonium bifluoride—both substances commonly used as inhibitors—are mixed together, an unexpected reaction takes place, for a wet mass is formed. The reaction may, perhaps, be symbolized as follows:—

$$\underset{\text{Boric acid.}}{H_3BO_3} + \underset{\text{ammonium bifluoride.}}{2\,NH_4HF_2} = \underset{\text{ammonium borofluoride.}}{NH_4BF_4} + \underset{\text{ammonia.}}{NH_3} + \underset{\text{water.}}{3H_2O}$$

The ammonium borofluoride produced is known to act as an inhibitor; when, therefore, the required proportions of boric acid and ammonium bifluoride are separately added to the dry sand containing the bonding agent, and the whole mixed, the above reaction proceeds within the sand mix, thus effectively adding water and ammonium borofluoride, the ammonia gas escaping to atmosphere.

A sand so prepared has markedly different properties in respect of " feel " and drying from those to which free water is added separately. For instance, this sand loses moisture at a much slower rate and, further, it remains mouldable at a free moisture content so low that normal sands would be unworkable. It may be conjectured that the water formed within the mass as the result of a chemical reaction combines more closely with the bonding material than does added water, which, in comparison, is but poorly incorporated by such mechanical means as light milling and mixing in a foundry mill.

It has been accepted that it is almost impossible to repair accidental damage to moulds composed of synthetic sand, and this was largely because of the excessive drying speed of such sand. If, however, sand of the type suggested, that is with moisture added as the result of a chemical reaction in situ, is used, minor modifications may be made successfully. One of the incidental problems of magnesium sand casting mentioned above, that of having to pour moulds quickly to safeguard crumbling due to drying, would also be solved by the use of the sand described. Consequent upon the slower drying rate, it may be that better organization of melting can be achieved.

Briefly, some experimental data may be given. A mix of sand of the following composition was made:—

Dry Bedford sand	400 gms.
Bentonite	14 gms.
Boric acid	12 gms.
Ammonium bifluoride	12 gms.
Sulphur	12 gms.
Water	12 c.c.

It will be apparent that part of the moisture was added as free water and part as a result of the reaction between the boric acid and the ammonium bifluoride. This is confirmed by the fact that a determination of the moisture content gave approximately 5 per cent. water present. If all the moisture had been added by the adjustment of the quantities of acid and fluoride, it is likely that the permeability would have been seriously decreased.

A second mix, consisting of:—

Dry Bedford sand	400 gms.
Bentonite	14 gms.
Boric acid	12 gms.
Sulphur	12 gms.

was made, to which was added sufficient water to give approximately 5 per cent. At the end of 36 hrs. the first sand was found to contain 2.2 per cent. water and was still mouldable, while the second sand, typical of normal technique, contained only 1.2 per cent. water; further, it had become so friable that moulding, or repair to a mould made from it, would have been impossible.

The valuable effect of adding water by means of the chemical reaction suggested persists, for following the normal practice of sand æration and tempering with water after mould knock-out, the original properties reappear: the sand " feels " right to the moulder, and again does not dry out as quickly as sands to which all the moisture is provided by free water addition.

Publication of the above composition for a magnesium-alloy moulding sand is made so that further practical tests on the foundry scale may be made. It is hoped that the results will be communicated in due course, as it is by free exchange of information that progress in the magnesium foundry industry will be achieved.

METALLIZATION WITH ALUMINIUM

By C. R. Draper

An Exhaustive Study of All Current Techniques and Equipment for the Coating of Metallic and Non-metallic Bases with Aluminium. Theory and Practice are Considered in Detail, with Particular Respect to the Scope and Economics of Various Fields of Application

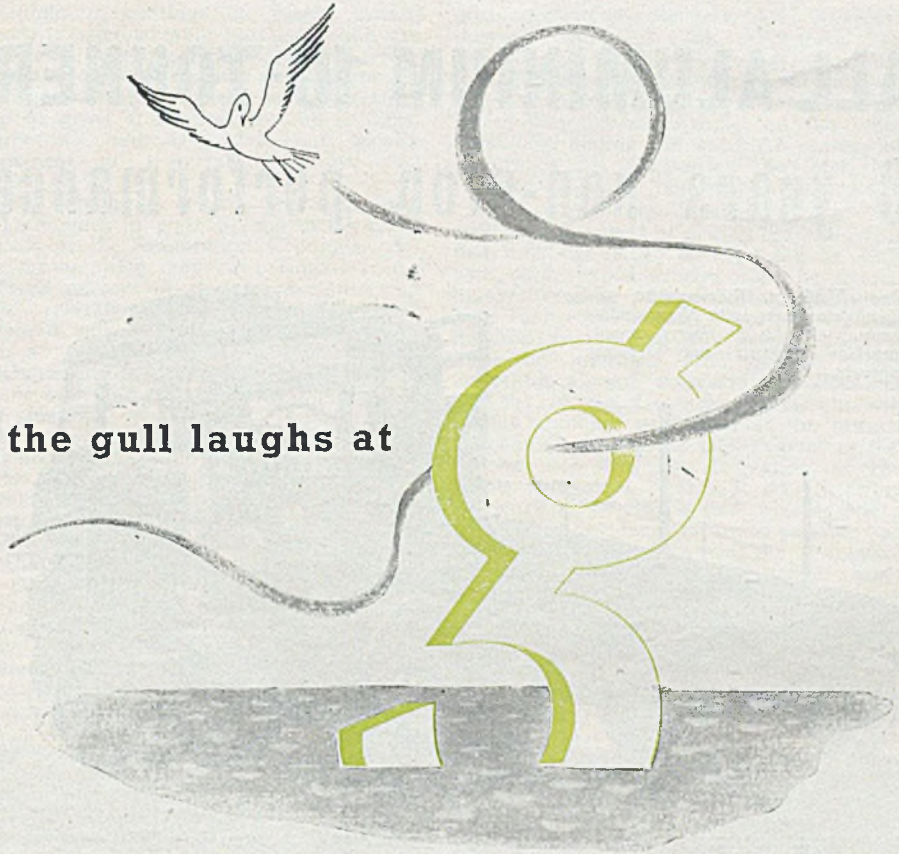
AN important branch of modern technology is the necessity for applying surface skins which are different in nature or character from the base over which they are applied. Very often they are applied for purposes of decoration; still more frequently to combine decoration with resistance to corrosion, whilst in other cases the achievement of an increased resistance to corrosion may be the predominating factor. These are generalities. More specific examples from the host which crowd to mind would include deposition for building-up worn or undersized components; coatings for obtaining increased resistance to high temperature oxidation; for reducing emmissivity; for producing a conducting surface on a non-conductor; for achieving selective chemical, physical or metallurgical actions as, e.g., in masking compositions for engraving, multicolour dyeing of anodized surfaces and for masking the non-working faces of soldering iron bits to reduce the rate of deterioration and to prevent the unwanted adhesion of solder to these parts.

Coatings may be metallic or non-metallic in character, depending on the desired properties of the skin and on the nature of the underlying base. Very often a number of alternatives is possible by means of which the same end may be achieved; then considerations of cost and convenience of application come into the picture. The science of surface treatment is, indeed, an involved subject about which few would claim to have more than a superficial knowledge of all branches.

In this article, we propose to deal with one very small section of this vast subject which, although of moderately recent development, already has certain important achievements to its credit. Coating with aluminium or with alloys of which aluminium forms an essential constituent is important for a number of reasons. Aluminium is recognized to be remarkably resistant to sulphurous fumes and by applying a surface skin of aluminium to steel or other metal, it is possible to achieve a similar measure of chemical resistance in cases where light-metal construction is not feasible for one reason or another. Much of the well-known resistance of aluminium to atmospheric corrosion may be transferred to other metals by applying an aluminium skin free from cracks, pores and gross amounts of oxide. Again, unalloyed aluminium and particularly super-purity metal are of greater corrosion resistance than many aluminium-base alloys and in cases where the mechanical properties of the unalloyed metal are inadequate or where bulk renders the use of massive super-purity metal prohibitive in cost, much the same desirable resistance can be obtained by applying a coating of the more resistant pure metal to the cheaper or stronger material.

Sometimes, where there is nothing against the use of light-metal construction for new equipment, as in the case of certain small items used in the bleaching and dyeing industries, there is a reluctance to scrap existing appliances which are quite satisfactory except in so far as their heavy metal or wooden construction renders them liable to excessive corrosion, or to the contamination or staining of solutions or materials with which they make contact. In such cases great improvements can be effected at low cost by applying an aluminium coat by the flame-spraying process.

Where items are subject to much abrasion, the relatively thin coating of soft unalloyed metal is soon worn away. In these cases, sprayed coats of harder aluminium-base alloy are beneficial. The potentialities of sprayed aluminium-base alloys have yet to be fully explored but, already, it is obvious that they possess important possibilities in numerous directions. To cite another example, sprayed hydronalium (Mg 3 per cent.-12 per cent.; Mn 0.2 per cent.-0.5 per cent.; Si 0.2 per cent.-1.0 per cent; Al remainder) not only possesses good wearing and corrosion-resisting qualities, but is also capable of taking a high polish, which has proved of value in applications where the production of a polished surface is also of importance.



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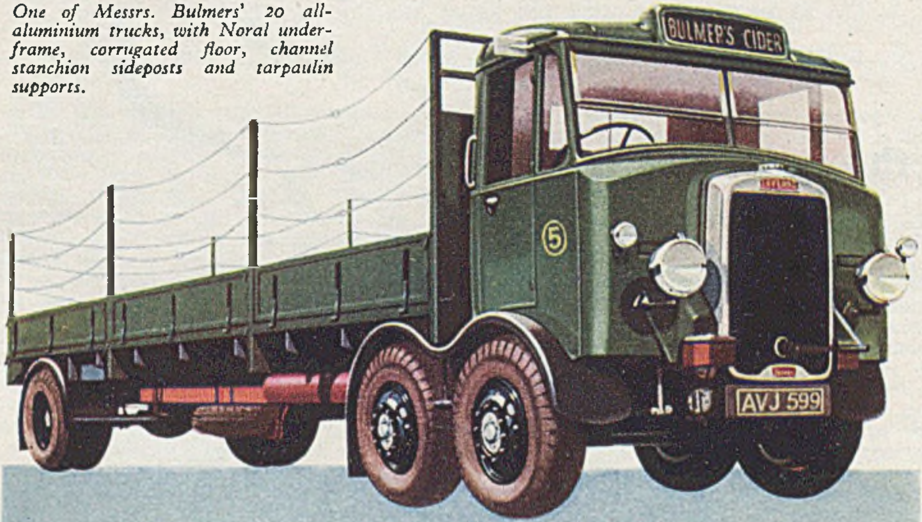

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Aluminium coatings on heavy metals, especially after suitable heat treatment, are capable of providing a high degree of resistance to oxidation at elevated temperatures. In the case of iron and steel, such protection may be given at temperatures up to 1,000 degrees C., although it is not usually permanent at temperatures above 800 degrees C.

Very different in effect is the application of aluminium to glass by the metal spray process or the evaporation technique. By this means there may be produced front-surfaced mirrors of great permanence and high reflectivity, pavement lights of increased efficiency, glass screens coated with a "silver" decoration which is also an electric heating element and blocks which can be built into high walls by a process of soft soldering.

All these and many other effects are obtained through the production of relatively thin coatings of aluminium or aluminium-containing materials on a base which may be metallic or non-metallic in character. There are seven main methods of producing these coatings, namely, spraying processes, methods in which the object to be treated is heated in contact with aluminium powder or an aluminium-iron alloy, dipping, electrochemical, evaporation, sputtering and mechanical processes.

Mechanical Methods of Aluminizing

Mechanical methods of plating have been known for very many years. Sheffield plate, for instance, which involves the laying of noble metal sheets upon a base metal and then uniting them by mechanical means is much older than electro-plating and the silver coating of copper articles was practised in this country long before the electrolytic method was discovered. In recent years, aluminium has been applied to other metals by this means. Thus aluminium layers can be produced on steel by rolling the two metals together under suitable conditions. Extensive use was made of this process in Germany in the years immediately preceding the war to obtain enormous quantities of aluminium-coated steel strip. In view of the great difference in hardness of the two metals, it was not possible to start rolling the combination until the steel strip had been reduced more or less to the required thickness and it was found preferable to complete the rolling of the steel strip first and then to apply the aluminium in the form of foil. Two processes have been used to secure the bonding of the two metals. In one case, the steel strip is pre-heated and then, with the aluminium foil in place, it is passed through the rolling mill whilst the temperature is maintained at between 300 degrees and 400 degrees C. In the second process, the strip steel, with the aluminium foil positioned on one or both sides, is passed through the rolls cold. Of these two methods, hot plating has the advantage that the adhesion of the aluminium to the steel can be obtained by using a comparatively low rolling pressure, while, on the other hand, cold plating eliminates the expense of a furnace for pre-heating the steel, but requires the use of high rolling pressures.

One of the big disadvantages of the mechanical method of plating lies in the fact that only flat (strip) material can be dealt with, and shaped articles must be treated before forming. Here, certain difficulties have been encountered. Thus if the plated material is required for deep-drawing it must be annealed after rolling. This annealing caused considerable difficulties in view of the fact that aluminium will readily combine with steel at the normal annealing temperatures (500 degrees to 600 degrees C.) with the formation of the brittle compound $FeAl_3$. It was therefore necessary to develop a steel with as low a recrystallization temperature as possible and one which would react with the aluminium at as high a temperature as possible. Some help in developing such material was obtained from experiments on the plating of basic Bessemer steel, which was found to react with aluminium at 600 degrees C. as compared with 490 degrees-500 degrees C. for open-hearth steel. It was then further observed that the setting-in of the reaction between steel and aluminium could be shifted to higher temperatures by certain additions to the steel; silicon was particularly effective in producing this effect.

In view of the fact that basic Bessemer steel does not possess sufficiently good deep-drawing properties it was found necessary to develop a similar non-reactive open-hearth steel. This problem was successfully solved, the temperature of the setting-in of the reaction in the case of particularly soft steel being raised from 500 degrees to 570 degrees C., this upper temperature being sufficiently high to completely soft-anneal a strongly cold-rolled strip steel for all deep-drawing purposes. The relevant patent specification shows that this property of the steel is obtained by increasing the oxygen and nitrogen contents. This, therefore, enabled the production of aluminium-plated steel strip without the formation of the brittle and undesirable intermediate layer of $FeAl_3$. The aluminium-coated steel strip thus obtained has many applications in diverse fields of which perhaps the most promising is the food-canning industry. Cans of this material must be designed with flanged and not soldered joints. Melchior(1) has described how Feran, a composite aluminium-strip-steel material in which the aluminium coat represents 6 per cent. of the total thickness, has been used in place of lead for the external covering of electric cables and insulation tubes.

Alclad and similar materials are made by the mechanical bonding of layers of relatively pure aluminium to sheets of copper-containing aluminium alloys. The latter, although mechanically strong, are less resistant to corrosion than unalloyed aluminium, and alclad combines the strength of the core with the resistance of the coating. It is important to note that the pure aluminium coating confers cathodic protection upon the alloy exposed at cut edges and also often confers some protection to rivets made of the stronger but less resistant alloys. Obviously the replacement of some portion of the total thickness by unalloyed aluminium must result in some reduction of overall mechanical strength as compared with a uniform slab of the strong aluminium alloy. Where this small reduction in strength cannot be tolerated, the surface layer may be of one of the high-strength aluminium alloys which are free from copper and other alloyed metals which make for reduced corrosion resistance. Duralplat is of this type.

Unfortunately, both these types of material suffer from the disadvantage that they are not suitable for lengthy exposure at high temperatures, diffusion of copper into the surface layers resulting in a steady decline in corrosion resistance. According to Keller and Brown(2) this danger is negligible at temperatures below 800 degrees F. (427 degrees C.); copper will reach the surface at 910-930 degrees F. (488-499 degrees C.) if the heating is prolonged.

Tests upon alclad, notably by Dix(3) and by Rackwitz and Schmidt(4) and upon duralplat by Meissner(5) and Stenzel(6) have provided evidence of the excellent protection afforded to the underlying light alloy by the more highly resistant coating. It should be noted, however, that not all light alloys can be protected in this way. As Edwards and Taylor(7) have pointed out, alloys containing $MgZn_2$ are anodic towards aluminium and would not be protected by the latter at cut edges. In point of fact, aluminium accelerates the corrosion of most, if not all, of the magnesium-base alloys.

Mechanically plated strip is of excellent quality, possessing a fine surface finish and bearing a substantial thickness of coating metal. Nevertheless, the fact that it is limited to sheet metals does impose a very serious limitation to its more general use.

Dipping Processes

A number of methods have been devised for producing coatings of aluminium on iron and steel by dipping the heavy metal into molten aluminium. Whilst this might at first sight appear to be a simple operation, it is, in fact, most difficult to control and often, in insufficiently experienced hands, gives very poor results. This is due, principally, to two factors. If a thick oxide skin separates the iron and aluminium, no coating is formed; on the other hand, if the skin is entirely absent, the alloying reaction between the two metals proceeds too rapidly. In all successful coatings produced in this way, some reaction between the two metals takes place so that no simple line of demarcation between the base and the coating, but a gradual transition involving the presence of compounds is developed. Proceeding outwards from the unaltered iron of the base material, there is first a region of mixed crystals becoming weaker in iron until only the component FeAl is present. Further towards the outside, some aluminium is also encountered so that the exterior surface, on which depends such characteristics as resistance to corrosion and high temperature oxidation, consists of a mixture of aluminium and $FeAl_3$.

From many points of view the presence of this intermetallic compound is unwelcome on account of its brittleness. Malleability decreases markedly as the thickness of the $FeAl_3$ layer is increased and the whole coating will be very brittle if the immersion time in the molten aluminium is too prolonged or the temperature too high. Because of this, the process of hot dipping is sometimes referred to as dip calorizing, the formation of this compound being also unavoidable in the calorizing process.

Modifications of the basic process have been devised to improve the adhesion of the coating in order to counteract, to a large extent, this brittleness, the most important being due to Fink, Delgren, Rodriguez, Nillson, Alerieff and that forming the basis of the Servarizing process. The Fink process(8) is one of a number in which this improvement is effected by the removal of the oxide film which tends to hinder proper adhesion. Fink achieves this by heating the iron or steel in hydrogen, which is taken up by the heavy metal not only removing the danger of the oxide reappearing but, also, probably favouring alloying with the aluminium. The process can readily be made continuous for aluminizing wire or strip. The material is first passed through dilute hydrochloric acid, then through a furnace, where it is heated to 600 degrees C., and immediately afterwards through boric acid and on to a second furnace in which it is heated to 900 degrees C.-1,000 degrees C. in an atmosphere of hydrogen. It then passes through the bath of molten aluminium, through a die to ensure correct diameter and to remove tears, and on to a revolving drum. The temperature of the aluminium is from 700 degrees C.-720 degrees C. and the wire (or strip) is passed through it at the rate of 6 ft.-15 ft. per minute.

Alerieff's method(9) depends on annealing the steel in an atmosphere containing hydrogen, nitrogen, and a quantity of water vapour carefully regulated so that the mixture is oxidizing at low and reducing at high temperatures. The gas mixture is passed through the plant counter-current to the steel to be coated. Thus, as the steel enters cold, it becomes covered with an oxide film which improves the absorption of radiation and thus accelerates its rise in temperature. When this has risen sufficiently, the oxide is reduced and the surface decarbonized. Before entering the dipping bath, the steel is cooled to the temperature of the molten aluminium and as the atmosphere at this temperature is neither oxidizing nor reducing, any troubles due to hydrogen supercharge are avoided and the presence of an oxide film is also eliminated. Because of the absence of oxide, only a short period of immersion is necessary to give a thick adherent coating consisting of unalloyed aluminium in the outer part and the component $FeAl_3$ below it. Nevertheless, despite the presence of this alloy layer, the coat is said to possess considerable flexibility, presumably because of the excellence of the bond between the two metals.

In the Nillson process, the surface of the iron is cleaned by pickling in a molten flux consisting of 10 per cent. cryolite, 20 per cent. $ZnCl_2$, 30 per cent. NaCl and 40 per cent. $CaCl_2$.

Similarly, the Delgren process for strip and wire makes use of a molten flux, but it is equally concerned with the method of passage of the work from the flux to the aluminium bath. The flux employed consists of a molten mixture of zinc chloride and ammonium chloride. The molten aluminium is kept in a cylindrical container open at the bottom and immersed in molten lead. The iron wire or strip enters the aluminium from below at the lead-aluminium junction, and the formation of aluminium oxide thus avoided. The whole apparatus is airtight and is filled with a reducing atmosphere.

Tinning or galvanizing the iron surface before immersion in the molten aluminium is a great help in obtaining adherent light-metal coatings free from excessive brittleness. The Servarizing process(10), following the same principle, makes use of an undercoat of cadmium to facilitate coating by dipping in molten aluminium. This process was developed for protecting furnace parts and heat-treatment boxes from oxidation, and has given excellent results in this application. Evans (11) has reported that some short-time laboratory tests have suggested that alloy coats made by this process also possess possibilities for resisting corrosion by moisture at ordinary temperatures.

It will be seen from the above that the essential steps in the hot dipping process comprise the removal of scale and oxide from the surface of the iron by methods involving the use of pickling acids, followed by heating in a reducing atmosphere or in a suitable liquid flux, and subsequent immersion in molten aluminium. It is essential to prepare the surface properly and to prevent it from oxidizing during transference to the aluminium bath. It is also important to adjust the conditions of treatment in such a way as to avoid as much as possible the formation of the brittle $FeAl_3$ layer, which proves troublesome unless the adhesion is very good.

The necessity for drastic pre-treatment in acids, molten flux mixtures or hydrogen at high temperatures, followed by immersion in molten aluminium, imposes severe restrictions on the nature of the article which can be so treated. Size is limited to that which can be accommodated by the melting pot and shape to that which avoids pockets and allows drainage of excess molten metal.

Calorizing and Related Processes

The Calorizing process consists essentially in heating the article in contact with aluminium powder, alumina and ammonium chloride when diffusion of the aluminium into the basis metal occurs. The process can be applied to ferrous and non-ferrous metals alike and it results in a reduction of the effect of high temperature oxidation, the wasting effect of some forms of corrosion and of the deleterious effect of certain gases. Examples of metals which have been successfully treated in this way include cast, wrought and malleable iron; various steels, including nickel and chromium steels; copper and brass. Equal protection is not afforded to all metals, however. Thus greater benefit is obtained by treating mild steel than cast iron, particularly if the mild steel articles have been subjected to some degree of working, e.g., by pressing, rolling, and spinning.

Sand blasting is an essential preliminary to treatment and as this also holds good for metal spraying, this subject will be dealt with separately.

The original Calorizing process was invented by T. van Aller in 1911 and developed by the G.E.C. of Schenectady, U.S.A., but, like many another valuable process, numerous variants have been patented and the methods in use to-day are modifications of, and improvements on, the original invention. The result, however, is fundamentally the same in all cases and comprises the formation of an alloy between aluminium and the basis metal with, usually, a thin film of unalloyed aluminium on the extreme surface. In the main process, this is achieved by sandblasting the article to remove scale, rust and grease, placing it in an air-tight retort which is partly filled with the calorizing mixture and subjecting the whole to a high temperature for several hours. The calorizing mixture consists of aluminium powder, alumina (to prevent the metallic grains from fusing together) and a small amount of ammonium (or, less frequently, sodium) chloride to help build up the coat; zinc powder or graphite are sometimes added. The proportions usually quoted are aluminium 49 per cent., alumina 49 per cent. and ammonium chloride 2 per cent. If chromium is present in the steel, diffusion of aluminium is greatly diminished and, to assist in building up a suitable alloy coat, the chloride content must be increased; Ipavic (12) recommends the mixture of 27 per cent. aluminium, 68 per cent. alumina and 5 per cent. ammonium chloride.

Throughout the process, the access of air, which would oxidize the aluminium powder, must be prevented. Sometimes this is achieved by the use of a hydrogen atmosphere; alternatively the retort may be fitted with valves which allow the passage of outgoing gases, but not of incoming air, and the vaporized ammonium chloride is relied upon to sweep the air from the chamber during the heating process.

The extent of permeation or alloying varies widely according to the composition of the calorizing mixture and the duration of the treatment. It may extend only a few thousandths of an inch or it may permeate the entire mass, in effect transforming the whole of the base metal into an aluminium alloy. The resistance of aluminium-containing iron alloys to high temperature oxidation is well known. With an aluminium content of about 9.5 per cent., scaling of such alloys becomes practically negligible in the region of 900 degrees C.; at this stage the alloy is covered with a reddish or white oxide film which inhibits further oxidation.

The calorizing process properly applied is capable of importing a similar degree of resistance to high-temperature oxidation. Curves published by Ruder(13) show a marked retardation of attack by high-temperature oxidation between 800-1,000 degrees C. Above 1,000 degrees C. renewed diffusion of aluminium into the iron impoverishes the surface layers, but some protection is still conferred. For ordinary purposes, therefore, the limiting temperature for efficient and reasonably permanent protection is considered to be between 900 and 1,000 degrees C. For service at higher temperatures, either the diffusibility of the aluminium must be reduced or its content in the body of the metal must be increased. As already noted, diffusion is diminished if the iron contains chromium(14), whilst with regard to the alternative, Rollason *et al.* note(15) that it is possible by a total impregnation of the metal, which gives an aluminium content of about 15 per cent., to render the article resistant practically up to its melting point.

As stated, many variants on the basic process have been described. E. D. Martin has suggested the use of a mixture of 50 per cent. aluminium powder, 45 per cent. alumina and 5 per cent. ammonium chloride, with a treatment temperature of 810 degrees C.-830 degrees C. This treatment was found to increase the life of iron and steel articles exposed at temperatures of between 800-1,000 degrees C. from five to sixfold as compared with the life of untreated articles exposed to the same conditions. A G.E.C. patent(15) specifies a mixture of 70 per cent. aluminium powder, 23 per cent. ammonium chloride and 7 per cent. zinc. The iron articles are heated with this mixture in airtight rotating drums at 450 degrees C., then removed and heated for another 15-20 minutes at 700-800 degrees C. to complete diffusion. As an alternative to the zinc addition, graphite may be used, the composition of the mixture then being aluminium powder 60 per cent., graphite 30 per cent., and 10 per cent. ammonium chloride, the reaction temperature being raised from 450 degrees C. to 700 degrees C.

The Aliting treatment which was used extensively by Krupps overcomes the necessity for maintaining an inert atmosphere during treatment by replacing aluminium powder with a powdered iron-aluminium alloy containing 40-50 per cent. Fe with 5 per cent. ammonium chloride. The treatment temperature is somewhat higher, cementation usually being carried out at 900-1,200 degrees C. for periods up to about 8 hours. The time and temperature of treatment depend upon the conditions under which the articles are intended to operate in service, but the surface skin generally contains 10-15 per cent. aluminium and is covered by an extremely thin layer of metallic aluminium. Further heating causes this aluminium to penetrate into the steel, the surface thereby losing its silvery white colour and taking on a dull grey appearance.

The mechanical properties of this coating are better than those of the coating obtained by the true calorizing treatment, but occasional disintegration of these coatings on carbon-bearing iron has been reported. Bauklo and Boke(16) attribute this to the formation of aluminium carbide, Al_4C_3, which is decomposed by damp air.

A disadvantage of the calorizing and Aliting processes is the fact that articles to be treated must be dismantled and packed with the calorizing or Aliting mixture into a suitable container. This naturally imposes limitations on the size of article which can be dealt with and it also means that components of equipment in use cannot be treated without dismantling. E. D. Martin has therefore suggested the aluminium chloride process in which a thin coating of aluminium is formed on the ironwork by treating the latter with aluminium chloride vapour at 900-1,000 degrees C. The aluminium chloride is produced separately by heating a mixture of 45 per cent. aluminium powder, 45 per cent. alumina and 10 per cent. ammonium chloride at 600 degrees C. So far as is known to the writer, this process has not been used commercially.

As one might anticipate, the main use of calorizing is for the protection of furnace parts against high-temperature oxidation, but some use has been made of it for the protection of iron against corrosion. Thus, the use of calorizing for buried pipes has been explored in America. Nealey(17) describes a process in which the pipes, together with the calorizing mixture, are sealed in steel retorts which are revolved in a gas-fired furnace heated to 1,500-1,800 degrees F. (815-980 degrees C.). The pipes are then removed, cleaned in boiling water and heated a second time in a fresh mixture. Logan and Taylor(18) report that calorizing buried pipes retards corrosion but does not completely prevent soil action. It should be remarked here that in Germany coatings of aluminium applied by flame spraying have been used in place of lead for covering underground electric cables. It is understood that satisfactory protection was afforded.

Flame Spraying

Of all the methods for metallizing with aluminium, flame spraying is the most versatile and it is, on this account, of considerable importance. Fundamentally, it involves melting the metal or alloy to be sprayed and projecting it, in the form of fine droplets, on to the article to be treated by means of a gaseous blast using a pistol which, although in some ways is reminiscent of the spray pistol used for applying paints and lacquers, is, naturally, of very different construction. In the powder process the metal is necessarily melted, but its temperature, on reaching the surface to be coated, must be above the sintering temperature of the metal powder.

Historically, it is interesting to observe that, although flame-spraying methods had been in use for a number of years for the production of metal powders for use in the manufacture of storage battery plates, etc., no one appears to have envisaged any practical application of the coatings which were unavoidably produced on screens, etc., during the operation of this process. Indeed, the idea of coating objects by a fine metallic spray, conceived by Dr. M. U. Schoop in 1911, resulted from an observation of a very different character, namely, that when firing with a small-bore rifle at targets placed in front of a stone wall, leaden bullets would adhere to the wall as flattened masses, and when touching or overlapping one another they appeared to be welded together. In fact, Dr. Schoop's early experiments on coating with a metal spray were conducted with the aid of a small cannon, without success as it so happened, since there is a limit to the velocity of the metal particles in the spray above which excessive rebound takes place. From this unpromising beginning, Dr. Schoop, with his assistants, Herkenrath, Bauerlin, Matzinger and Werner, Schoop, Jung and Schori have produced a number of robust and reliable types of metal spray apparatuses which have been based upon three main processes, namely, the use of molten metal, wire and powder.

The earlier designs of molten metal pistols were very cumbersome affairs, but the modern apparatus is very much improved. In a typical modern pistol the molten metal, previously melted in a separate furnace, is held in a malleable iron container heated by means of a Bunsen burner with gravity feed to the nozzle. The flame is also used to preheat the compressed air required for producing the spray. The air stream is carried by a stainless steel pipe coiled just in front of the burner to the nozzle, which is of silver steel (0.035-in. diameter). A limitation of the gravity-fed pistol is the fact that overhead work is not always possible since the pistol must always be slightly inclined downwards. In modified designs, the nozzle is made to project upwards and the whole design of the pistol is such as to overcome this disadvantage. However, it is also necessary to make these modified designs of molten metal pistol in miniature sizes only since the weight of the normal apparatus is such as to make overhead work extremely fatiguing. Another disadvantage of the molten metal piston is the rate at which the nozzle and container are attacked by the molten metal. Silver-steel nozzles have an average life of 1-1½ hours, and, although they are renewable within two or three minutes, in practice they are seldom replaced in anything like this time. Refractory liners of Morganite or fused corundum assist by prolonging their life to about 13 hours, but they are expensive and sometimes fail in a very short time, due to the different coefficients of expansion of the steel and the refractory lining.

Iron is dissolved by molten aluminium and in consequence progressive attack of the container takes place. Important, also, is the contamination of the aluminium, which is objectionable from many points of view. Refractory linings have been tried but they have not proved too successful, due, again, to expansion troubles.

It is possible to eliminate most of the contamination by working towards the further extreme of the pistol's range when the heavier particles fall out of the spray under the influence of gravity. This does, of course, involve a considerable wastage of metal and would not normally be attempted. The normal working distance is 5 ins.

As the nozzle wears, the spray becomes coarser. It also becomes somewhat irregular, since the wear on the nozzle is not regular, and to maintain a uniform thickness the speed of spraying is increased. The type of spray produced is influenced by the projection of the nozzle from the air feeder; the longer the projection the finer the spray. For heavy coatings, therefore, it is an advantage to shorten the nozzle, but if this is carried too far, the spraying becomes uneven.

Occasionally the nozzle becomes filled with dross which has to be removed by poking with wire. Other disadvantages of the molten metal pistol are that its weight and the temperature of the molten metal container which is in close proximity to the operator make the latter's task most uncongenial, whilst it is impossible to coat the inside of any but the largest containers and difficult to execute outside jobs where cold draughts playing on the reservoir and the need for frequent replenishment from a furnace which is probably inside the building lead to loss of time and difficulty in maintaining correct spraying temperature. In the case of aluminium and its alloys, the latter is 50-100 degrees C. above the melting point.

However, this process requires neither compressed oxygen nor compressed fuel gases and the fact that it allows the use of metals in their cheapest form, namely, as ingots, has proved attractive for some purposes.

The wire pistol is perhaps the most widely used form of metal-spraying appliance at the present time. The modern wire pistol is a robust and reliable piece of apparatus weighing some 3½ lb., and with it it is possible to coat any solid substance with a layer of aluminium, zinc, lead, tin, copper, iron, nickel, bronze, brass, etc., in thicknesses from 0.002 in. upwards. In this apparatus the metal to be sprayed is introduced into the apparatus in the form of a wire 1-2 mm. in diameter and is fed to the nozzle continuously and uniformly at the rate of 6 ft. to 30 ft. per minute, through mechanism operated by an air-driven turbine. At the nozzle, the wire is met by a reducing flame of oxy-acetylene or oxy-coal gas and is melted at a rate equal to the rate of feed. Under these conditions, the end of the wire is melted and is met by a blast of air at 40 lb. to 50 lb. pressure, converted into a spray and shot out in a diverging cone having an inner zone of reducing gas and an outer zone of air.

The turbine rotor is carried on ball bearings and runs at 12,000 to 40,000 r.p.m., the actual speed being regulated by means of the air control valve. For feeding purposes, the wire is gripped between two serrated rollers which are positively driven at equal speeds. Internal gears are frequently sealed in grease baths to prevent access of metal shavings and splinters, which would cause premature wear. As with the molten metal pistol, burning of the nozzle tip can be a problem, but this has been greatly reduced in certain designs of pistols by making use of heat-resistant materials such as nichrome. Stoppages may occur in use due to the blockage of gas passages by dust or to the adhesion of metal accumulations in the nozzle or in the wire or gas tubes; formation of these accumulations is hindered by rubbing the nozzle and tube surfaces with graphite. In operation, air and gas pressures must be carefully watched; on no account may the air pressure recommended for that instrument be altered, but a small latitude may be allowed in the adjustment of the gas pressure.

The wire pistol is versatile in character and can readily be adapted to meet particular requirements for which the simple hand pistol is inadequate. Thus, for outside work, or to meet cases where neither compressed air nor gas are available, complete portable or mobile plants have been designed and such equipment is in use both in this country and abroad. Such mobile equipment may take the form of a lorry fitted with an oil engine and a compressor, a sand blast apparatus, gas cylinders and the necessary loose equipment. Again, for spray coating the outside of large metal pipes, machines have been built on the principle of the lathe in which the tube is held between two centres and slowly rotated while the pistol is carried on a slide rest and slowly traverses the length of the pipe. If the size of the work to be coated warrants the provision of more complicated equipment, more than one pistol may be carried at intervals along the machine to hasten production, each pistol metallizing only a portion of the tube. This principle has been much extended in the case of the powder pistol, to be described next, to the coating of such complicated profiles as cylinder heads by mass-production methods.

For metallizing the bores of tubes with internal diameters of between 1 in. and 3 ins., rotary nozzles are used. These spin around the axis of the pistol and apply a uniform coat on a cylindrical surface of which the centre is the bore of the nozzle. Above 3-in. diameter, the metal is not thrown far enough to ensure a satisfactory coating, although this method of application may be used for applying a thin coating for building up worn or oversize housings which have to take a forced fit liner or bearing. Below 1-in. diameter, mechanical considerations preclude the use of rotating nozzles. The rotating nozzle is not too satisfactory with all metals. Tin and lead are particularly troublesome to spray by this means, but no difficulty is encountered with aluminium.

Extension nozzles consisting essentially of an extension tube fitted to a standard pistol can be used with advantage for coating surfaces not easily reached by the standard pistol.

For coating the bores of larger diameter tubes, deflector nozzles are useful in which the metal spray is projected at an angle to the pistol. These may be of two types, one in which an angle nipple is fitted to an extension tube to deflect the spray through an angle of about 45 degrees and the other in which an air jet is used to deflect the spray.

In the fan spray nozzle, two jets of air diametrically opposite to each other impinge on the atomized stream of molten metal issuing from the central orifice of the pistol and form a spray similar in shape to the flame from a bats-wing burner. This may be in the form of a thin wide or a narrow thick ribbon and it overcomes the defect exhibited by the standard spray in coating large surfaces, that the centre of the spray cone is always thicker than the outside regions, so that each section sprayed must overlap to secure a uniform coating.

For spraying heavy metal coatings for building-up purposes especially robust pistols have been designed capable of handling wire rods up to ⅛-in. in diameter. Naturally, such pistols weigh considerably more than the standard designs, but it is interesting to note that some reduction has been achieved by the use of light metals in this construction. Thus a pistol which in the standard model weighs 5½ lb. is also available with an aluminium head, in which case the weight is only 4 lb. This pistol is capable of depositing 4.1 lb. of aluminium per hour using 45 cubic ft. of oxygen and 45 cubic ft. of air per minute.

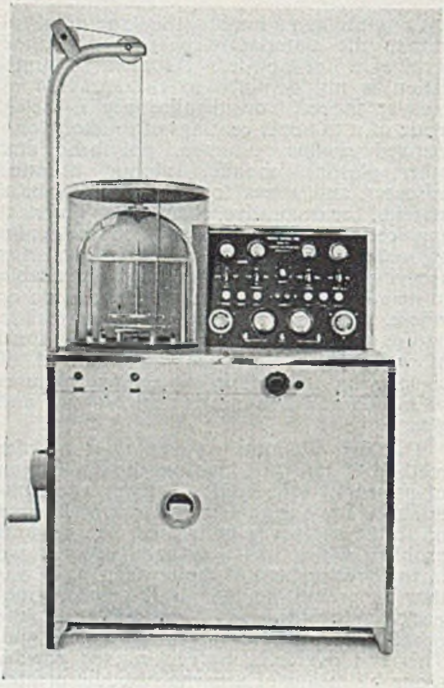
Wire pistols use compressed coal gas, hydrogen, dissolved acetylene, or propagas as fuel. Of these, hydrogen, acetylene and propagas are used from cylinders fitted with fine control regulators. Coal gas may be obtained from cylinders, but is more usually run in from the mains through a non-return valve and compressed to 25-30 lb. per sq. in. on the site by means of a small compressor. The air supply is obtained from a standard type of air compressor operating at 50 p.s.i. pressure/20 cubic ft. per min. minimum, the horizontal type being preferable, as it offers less chance for oil to get through to the pistol.

The Powder Pistol

We come now to the powder type of pistol which daily is assuming a position of greater importance in the metallizing industry. As the name implies, this type of pistol utilizes the material to be sprayed in the form of a fine powder. As such, its application is not confined to the spraying of metals; indeed, considerable use is being made of it to apply coatings of thiokol, polymerized shellac, bitumen, polythene and other plastics, ebonite, sulphur, asbestos mixtures, and waxes to many kinds of base material for decorative, chemical or electrical engineering purposes. Again, in the realm of metals, the powder pistol is ideal for applying metals where it is not practicable to maintain them in the molten condition or where, perhaps, because of their brittle nature, they cannot be obtained in the form of wire. It is also the only method of co-spraying immiscible metals or mixtures of metals and non-metals such as zinc and glass.

The first attempts to employ a powder method of spraying, involved the application of pressure to the inside of the powder container to force the cold powder at high speed on to the surface to be treated. An obvious problem which was very soon encountered in the development of the powder pistol was that of preventing the metal powder from packing together as it passed from the powder container to the nozzle of the gun. This was overcome by sucking the powder along by means of a partial vacuum created at or near the nozzle. It was then discovered that the mere projection of cold metal powder on to a surface was insufficient to produce an adherent coating and that it would be necessary to heat the powder. The experiment was then tried of heating the air blast, but this, too, was unsuccessful. It was, in fact, found necessary to heat the powder almost to melting point as it left the nozzle before satisfactory deposits could be obtained, and the simplest way of doing this was to blow it through a sufficiently hot flame.

The modern powder pistol has developed along these lines and is, essentially, a container from which a steady supply of powder is drawn into the pistol under a reduced pressure and then directed, by a blast of air, on to the surface to be sprayed through an annular flame of combusted gas. As with the wire pistol, the gases employed as fuel include acetylene and propane in conjunction with compressed oxygen. The pistol contains two injector arrangements. One in the handle for mixing the fuel gases and preventing back-firing in the pipes is similar to that used in any ordinary blowpipe, the other is used for aspirating the metal powder. The compressed air supply is split, the main stream going direct to the nozzle while the remander is passed through an auxiliary nozzle where a partial vacuum is created to draw along the powder. The chamber into which this nozzle opens is connected to the atmosphere by a small pipe



ABOVE. Universal apparatus for either evaporation or sputtering (Courtesy W. Edwards and Co. (London), Ltd.). At the right is the Schori metal spraying pistol (Courtesy Schori Metallizing Process, Limited).

opening into the back of the handle, where it is conveniently closed in service by the operator's thumb. The orifice must be closed before sufficient suction is generated to aspirate the powder, this arrangement thus providing a simple and reliable control which at the same time safeguards against the possibility of the spray coming into operation when the pistol is laid aside. The Schori powder pistol which operates in this manner is a handy and well-balanced tool, light to handle and easy to control.

Considerable ingenuity has been shown in the development of a satisfactory design of powder container with the object of ensuring a steady and controlled flow of powder. One successful design is rather like an egg-timer in design, the feed to the pistol being taken from the constriction between the upper and lower containers whilst a small eccentric air turbine creates a certain amount of vibration and tends to reduce packing.

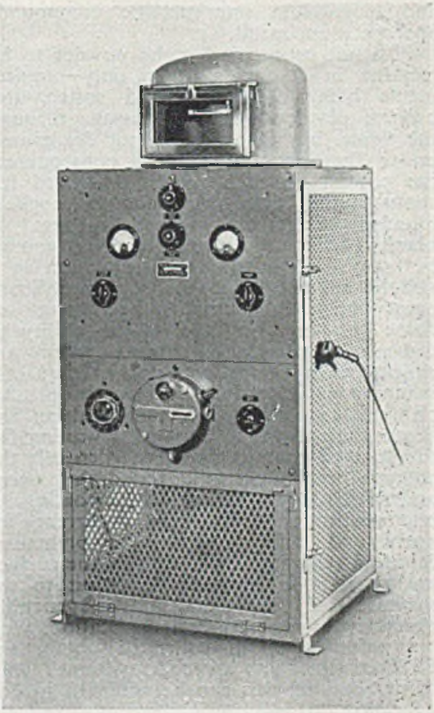
The success of the powder pistol is largely bound up with the quality of the powder employed. Careful attention must be paid to such factors as particle size, particle shape, chemical composition, oxide content, density and rate of flow. Particle shape is of importance because this largely controls the rate at which the powder will flow through fine orifices; purity and low oxide content are essential if the highest degree of protection is to be conferred upon the article being sprayed.

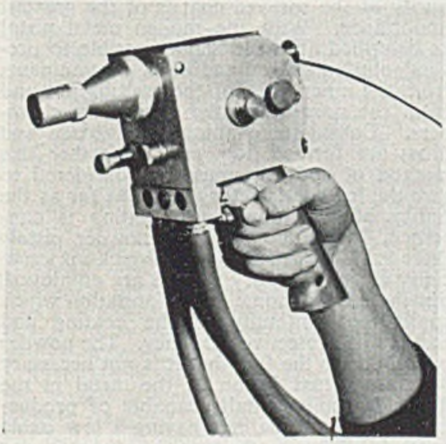
The Schori metal spraying pistol is illustrated below.

A Comparison of the Three Types of Pistol

Rollason, Turner and Budgen in their book on Metal Spraying [19] have given an excellent summary in tabular form of the principal characteristics of typical molten metal, wire and powder pistols. These early conclusions still hold good to-day; to them we would add a number of observations. Obviously the molten metal pistol is of limited applicability, but it does make use of metal in its cheapest form, namely, as ingot or bar. It can only deal with metals which will form homogeneous melts and even then it is not suitable for use with the higher melting point metals and alloys. Aluminium-silicon alloy with a melting point of around 600 degrees C. is generally considered to be the highest melting point material which can be sprayed satisfactorily by the molten metal pistol.

The wire pistol is the one which has had the greatest application, particularly in this country. Evans [11] in the section of his classic volume dealing with the subject of protection by metallic coatings recommends that " A robust pistol of the wire type should be used for applying aluminium coats " and makes no mention of either the molten metal or powder pistols. Rollason, Turner and Budgen state that wire pistols are essential for the building up of thick deposits in salvage work. Yielding coatings of metal which can be almost free from contamination except by oxide, and surfaces with good resistance to oxidation, can be produced. In the spraying of kitchen ranges, automobiles, motorcycles, household materials and so on with aluminium, the wire pistol employing wires 0.04-0.06 inches in diameter has given the most satisfactory results. The wire pistol is probably the most expensive of the three types to work and the fact that the metal to be sprayed must be in the form of a fine wire imposes a severe limitation to its potential range of utility. In practice, however, this does not prove such a disadvantage, as the largest proportion of spraying is carried out with zinc, aluminium and lead, all of which can be drawn into thin wires. The fact that the wire pistol, with the aid of suitable adaptors, is capable of spraying the internal surfaces of long tubes and deep bores is a great advantage.





ABOVE is pictured an apparatus for metallizing by the sputtering process (Courtesy W. Edwards and Co. (London), Ltd.) At the left is the Mark 16 wire-type pistol manufactured by Metallization, Ltd.

Perhaps the chief disadvantage of the wire pistol is the fact that it has a number of wearing parts and is not so robust as the other types. Nevertheless, it is the most widely used at the moment and has given excellent service since its introduction in 1922.

The powder pistol is noteworthy for its flexibility with respect to the variety of materials that may be sprayed. Thus, it is able to deal with metals and alloys whose melting points are too high for use in the molten metal apparatus or which do not form homogeneous melts or which cannot be drawn into thin wires. All metals and alloys can be obtained in the form of powder, although different methods of manufacture produce materials of very different quality from the point of view of the requirements of the powder pistol. It has recently been shown that the brittle nature of inter-metallic compounds is no disadvantage when they are applied as a thin film, and this has opened up wide possibilities for the utilization of metals with special characteristics which could not previously be employed. Such compounds which are easily powdered and admirably suitable to flame spraying include the zinc, aluminium and magnesium inter-metallic compounds and speculum metal.

It is not necessary for the powder to be metallic in character. The only requirements being that it should soften and become plastic during its passage through the flame, but that it does not decompose under these conditions. In consequence, the powder pistol has been employed for spray coating with such materials as glass, bitumen, plastics and synthetic rubbers. A number of these materials can, of course, be sprayed in solution or suspension in a volatile solvent by means of the ordinary paint spray. A particular advantage of flame spraying, however, is the ability to dispense with volatile solvents which are expensive, in very short supply, frequently inflammable and dangerous to the operator on the account of their toxic nature; the absence of any time lag whilst waiting for the sprayed coat to dry or harden off; the absence of the necessity for drying ovens and the ability to produce thick coatings in one application. Since the occurrence of pores and defects leading to premature failure of the film and attack of the surface are roughly in inverse proportion to the thickness of the coating it is generally necessary to make several applications with the paint spray in order to obtain the required thickness of coat, whereas with flame spraying this can be achieved in one operation.

Moreover, not all plastics are soluble in paint spraying solvents, in which cases flame spraying may provide the only practicable method of application. Polythene is one such example. This material, which was developed by the I.C.I. during the war, is a whitish translucent plastic, tough and flexible, odourless and tasteless, with outstandingly good dielectric properties and an amazing resistance to solvent materials. It has tremendous potentialities in the chemical, food, electrical and radio industries which were realized to a small extent during the last year or two of the war. The flame spraying process using the powder pistol appears to be the only method by which this important material can be applied in the form of thin or thick coatings to a metal basis; no difficulties are encountered by mixing the powdered polythene with other powdered materials and it thus becomes possible to spray the material either pigmented for decoration or filled for modification of mechanical, electrical and other properties. The powder pistol has also been used to good effect in the spraying of polymerized shellac-base compounds with pigments or fillets for coating transformer and junction boxes, switch gear, instrument boards and panels and as domestic finishes; the synthetic rubber thiokol for the protection of ships' hulls, tailshafts and brackets, condensers, chemical plant, vessels for oils and solvents, light alloys for use in the tropics and steel work exposed to chemical fumes etc.; bitumen compounds suitably filled for the waterproofing of concrete and brickwork, for insulation, sealing and impregnation in the chemical and electrical field and for the waterproofing and acid-proofing of floors, structures, vats, etc. Mixtures of metals and non-metals such as aluminium and glass or aluminium and bitumen have been sprayed by the powder pistol to fulfil particular requirements. Other advantages of the powder pistol include its low weight, the high rate of application, and the absence of moving parts or of components liable to become out of adjustment.

For many applications, including that of increasing the resistance to attack by atmospheric and other forms of corrosion, the purity of the sprayed coat is of the greatest importance. With the molten metal pistol as described above it is not possible to prevent solution of iron and other contaminants from the inner walls of the melting pot and from the nozzle and the connecting chambers. Considerable amounts of oxide and dross are formed, too, and altogether this method of spraying must be considered to give the least satisfactory result as far as the purity of the aluminium coating is concerned. With the wire and powder pistols there is little chance for metal pick-up, especially when refractory nozzles are employed, but there is still some oxide formation which spoils the continuity of the coating and reduces its protective efficiency. The powder pistol, due to the fact that it is not necessary to cause actual fusion of the metal in the flame, is fundamentally capable of producing aluminium coatings having a low oxide content. Prior to the war, however, it was not too satisfactory in this regard, simply because the powder itself was insufficiently pure and already contained an appreciable oxide content, together with a certain amount of other contamination. Sidery and Braithwaite(20) claim that better adhesion is exhibited by coatings of aluminium applied by the wire pistol than by those applied by the powder pistol. In intermittent salt spray tests carried out by Rollason(15), a sample sprayed with aluminium by the powder process developed rust spots within 26 days. Other samples were coated with aluminium-silicon by the wire process and by the molten metal process. That produced by the wire process was unaffected and bright after exposure for periods up to 170 days; using the molten metal pistol, the deposit soon became covered with a white corrosion product and was practically destroyed after 70 days exposure. During the war, however, a great deal of attention has been directed towards the development of greatly improved qualities of powder, more free from metallic contamination and with an oxide content of less than 0.5 per cent., and it is possible that, using this improved powder, coatings can be obtained which are more free from contamination than those with the wire pistol, although the writer is unaware of any experiments undertaken to prove this point. A further disadvantage of the powder pistol which now seem to have been largely overcome was that it sometimes produced irregular coatings containing "lumps." These, however, are recent developments not yet translated into general practice, and it is popularly considered that the most satisfactory coatings are obtained by using the wire pistol.

Actually, the amount of oxide formed during atomization is small. For one thing it is doubtful whether the metal is really molten once it has left the gun, and for another there is very little movement of the air stream relative to the metal particles, except for the very brief interval of time in which they are gathering speed. Consequently the oxygen content of the air layer immediately adjacent to the metal particles is rapidly diminished and is not replaced before cooling and deposition takes place.

On the question of cost, the molten metal method of spraying is generally cheapest because it uses ingot material. In awkward positions, and in endeavouring to cope with overhead work, this advantage is easily lost through slower working and greater fatigue. The wire pistol is a little more expensive to operate than the powder pistol due to the cost of drawing the metal into thin wires.

Recognizing the importance of minimizing the oxide content of the sprayed coat, Schoop designed a wire pistol in which the hot metal was maintained in a neutral atmosphere of carbon dioxide. Atomization was carried out by a blast of preheated carbon dioxide and the wire passed not through a flame but through a tube fitted with fins on its external surface which absorbed heat from two oxy-acetylene flames. The carbon dioxide was preheated by passing these fins. Although it is understood to have produced extremely homogeneous coatings, no extensive use of this pistol has been made in this country.

More recently a return has been made to the molten metal system for spraying super-purity aluminium. The details of the method adopted presents a great refinement over the method described above. As is well known, the corrosion and tarnish resistance of super-purity aluminium in many environments is greatly superior to that of metal of commercial purity, and it has for some time past been used as a cladding for stronger or less expensive metal for a number of purposes in the chemical industry and elsewhere; the super-purity layer being applied mostly by the mechanical method of rolling. The need for applying it by spray pistol arose from developments in the design of electrolytic condensers where, for purposes of increased efficiency, it was desired to coat the electrodes with aluminium. It was found that the efficiency and life of these electrodes was so dependent on the freedom of the aluminium coating from contamination that it was worth while to employ super-purity metal. This was applied as a spray, using a wire pistol employing super-purity aluminium wires of small diameter.

J. B. Brennan has pointed out(22) that this can be done more efficiently and more cheaply by spraying molten super-purity aluminium obtained by melting pigs, bars or billets of the metal in a fused alumina crucible. Essentially, the plant with which it was proposed to carry out this operation consisted of an open top crucible heated externally by electrical means and completely enclosed by a pressure chamber. A fused alumina pipe ran from inside the crucible to the outside of the pressure chamber, from whence a lead could be taken to the spray gun. In use, super-purity metal was introduced into the crucible, the pressure chamber closed and inert gas passed in to remove air from the chamber and the connecting pipe. The pressure was then reduced to atmospheric and the current was turned on until the aluminium was molten. To obtain a feed to the gun, the pressure of the inert gas was increased causing a stream of molten metal to issue from the pipe without imposing any strain on the fragile and expensive crucible of fused alumina. Another advantage of the gas chamber was that the gas in it lagged the crucible and thereby greatly assisted in the maintenance of correct temperature.

Characteristics and Applications of Sprayed Coats

The adhesion of sprayed metal coats varies widely according to conditions. Thus, with proper care, it is possible to produce a sprayed coating of aluminium on glass which can be separated only by the use of considerable force and which, when it is broken off, tears with it a great number of little glass splinters. Thus aluminium offers the key to the solution of finding a method of building glass assemblies whose tensile strength is that of the glass itself. Thus glass building blocks have been built into high walls and complete buildings by first spraying the surfaces to be joined with aluminium, and then with copper. Such surfaces can then be soldered together using a low-temperature furnace and low pressures. The issue of this journal for May, 1939, contains a photograph of a wall 15 metres high built in this way with hollow bricks of toughened glass with metallized edges. This method of soldering also provides a means of making sealed joints between glass and metals or by metallizing both components, then between glass and non-metals or, in fact, between any two materials capable of being metallized. An undercoat of aluminium can be applied to glass in cases where it is desired to produce an adherent coating of another material which is itself less strongly adherent to glass.

The high tension of aluminium to glass is believed to be due to the formation of a thin film of aluminium silicate at the interface, but is very dependent on a proper preliminary treatment being given to the glass, including heating to 700 degrees C. immediately prior to spraying. Some care and practice are necessary to secure the best results. Quartz, on the other hand, may be given an adherent and even coating of aluminium much more readily.

In spraying aluminium on to the other non-metals, however, it is not always possible to achieve as great a measure of adhesion as might be desired, and some roughening of the surface is indispensable. Materials such as wood, paper and millboard, may be sprayed satisfactorily with aluminium provided there is some mechanical keying. Coarse, unglazed papers and open grain unfilled woods give better adhesion as might be anticipated. Nevertheless, the adhesion produced is not always sufficient to withstand the stresses induced by the very different coefficients of expansion. In metallizing wooden surfaces with aluminium, for example, it is found that the coating, not infrequently detaches itself at sharp corners and at the edges and curls up even though the surface is well roughened, showing that the coating is under strain. It was, for example, found to be impracticable to aluminize wooden aeroplane propellers owing to the danger of peeling following damage to the coating at the tip and edges. Nevertheless, there are many less onerous applications, particularly in the decorative field, for which coatings of aluminium and other metals on wood and other non-metals are invaluable.

As regards metal-to-metal adhesion, this is due to the application of surface forces exerted between two metals in close contact, reinforced to some extent by mechanical effects due to "rooting" of the coating in pores and depressions in the metal surface. In these respects the mechanism by which aluminium and other metals adhere when sprayed on to cold or nearly cold surfaces is reminiscent of that occurring in electrodeposition processes. Moreover, as in electroplating, adhesion is influenced both by the preliminary treatment of the surface to be coated and by the conditions under which coating occurs, the former being the more important, provided that certain variables in the coating technique are kept within certain well-defined limits.

There are also these differences, however, (a) that in electro-plating, the deposited particles from which the coating is built up are of atomic dimensions, whereas in metal spraying they, although still small, are dimensionally of a very much higher order, and (b) that unlike electro-deposited metals, there is no tendency for the sprayed metal to follow the crystal structure of the base metal. Whereas in electro-plating the greater difficulty of securing good adhesion to a polished than to an etched surface is due in part to the disordered arrangement of the surface molecules resulting from the polishing operation, in metal spraying the same increased difficulty is due only to the decrease in the number of keying positions.

Good adhesion is promoted by securing intimate contact between the two metals. The first essential before applying a sprayed coating is, therefore, to clean the surface to be coated very thoroughly, paying particular attention to the removal of scale, oxide incrustations, and so on, and of films of grease and oil.

The size of the sprayed metal particles and the velocity with which they are projected on to the metal surface are reflected in a peculiar effect which results in a greatly reduced adhesion to a polished metal surface. The phenomenon has been investigated by Robert Hopfelt, Director of the Metallisator A.G., Altona, whose conclusions are summarized and illustrated in Rollason's book previously referred to.(15) If a metal particle, which is surrounded by a certain amount of air, strikes in a heated condition upon a smooth metal plate, it makes a gliding movement before coming to rest. A certain amount of rebound takes place and the particle during its gliding movement may leave the surface and return to it a number of times before motion ceases. The result is a wavy or slightly corrugated deposit which is in close contact with the substratum at only a few points so that inferior adhesion is obtained. If, however, the substratum is previously sand blasted, the surface is roughened and sharp elevations and depressions are produced. A sprayed metallic particle meeting such a surface is brought immediately to rest without having a chance to rebound. Moreover, the heat of the particle together with that produced by its sudden cessation of movement expands and removes much of the surrounding air so that it is brought closer to the substratum. Sand blasting is, in fact, the secret of success in metal spraying, and the coating operation will rarely give satisfactory results in the absence of this preliminary surface treatment.

Deliberate attempts have been made to remove the air layer between the coated and the coating metal in an endeavour to improve adhesion. The influence of this type of air cushion is a familiar factor. Thus, two smoothly polished and flat plates will adhere to each other much more tightly when wetted or warm than when dry or cold, the explanation, according to Hopfelt, lying in the fact that wetting removes the adhering layer of air from the surfaces and that warming acts similarly by causing expansion and therefore partial expulsion of the intermediate air layer. These principles have been applied in practice to the operations of soldering and autogenous welding with the addition of fluxes and high pressure respectively to break through the oxide film which is present, and attempts have been made to extend them to the operation of metal spraying. It was thought that it might be advantageous to maintain the article at an elevated temperature during spraying to effect expansion and partial removal of the adhering and occluded air-layer. Unfortunately, scorching and oxidation take place and the result is unsatisfactory. Similarly, if a very short nozzle distance is used, the particles may be projected on to the metal substratum with such force that welding takes place, but again the deposit is unsatisfactory, being so rich in oxide that it is useless for most practical purposes.

The fineness of the metal spray has an influence on the adhesion produced since small particles are able to penetrate more easily into the pores of the surface than are coarse ones, resulting in better mechanical keying. Since the fineness of the spray is largely connected with the air pressure employed and the position of the burner, these represent factors which are under the control of the operator.

The Sand-blasting Operation

Abrasive blasting may be carried out by one of three systems: (a) a pressure system in which compressed air is used to force the abrasive from a closed container along a pipe line and discharge it through a nozzle on to the work; (b) a suction system reminiscent of the mechanism of the powder pistol in which the abrasive is sucked up into a gun and is then projected on to the work by compressed air; and (c) a mechanical system in which the abrasive is fed on to impellers rotating at high speed and flung on to the work by centrifugal action. Of these three systems, the first is the most important and most widely used. The suction system is less efficient though of greater simplicity, whilst the mechanical system is best suited to the treatment of small objects handled in barrels or by a rotary table or conveyor belt which bring the object to the nozzle rather than the nozzle to the object.

An efficient exhaust system is required under Home Office Regulations to remove and filter the dust-laden air which is the inevitable result of abrasive-blasting operations, and in consequence this is not carried out in the open shop. Chambers or rooms are provided for the blasting of large objects and cabinets for treating smaller work. In the one case, the operator stands inside the room and directs the blast on to the work through a nozzle at the end of a flexible hose, whilst in the other he stands outside the cabinet and moves either a nozzle over the work or the work under a fixed nozzle. Small articles may also be dealt with on rotary tables or conveyor belts or in rotating barrels. In the last case, the articles are tumbled while the abrasive blast is projected through one or both ends of the barrel.

Naturally, protection must be provided for operators using hand-controlled nozzles, since the process is unpleasant, and in the absence of proper precautions may be dangerous to their health. Such protection may take the form of a combined head gear which completely covers the head and includes both goggles and respirator. Where the operator stands inside the cabinet, it is advisable for him to wear a complete protective suit supplied with fresh air.

Factors Affecting the Efficiency of Sand Blasting

We have already described how sand blasting can improve the adhesion of sprayed metal coats to non-metals, and particularly to metals, by thoroughly cleaning the surface, by providing numerous small indentations or pores in which the sprayed coat may anchor itself, and by arresting the flight of the sprayed particles, all of which result in a more intimate contact between the two surfaces on which ultimately adhesion depends.

Naturally, the extent to which this occurs in practice is dependent on the variables of the blasting operation. Thus, articles should be sprayed as soon as possible after blasting and handled only with gloves to avoid the deposition of moisture or the transference of grease. The size of the indentations or pores produced should be of the same order as the size of the sprayed metal particles, and considerable pressure and a hard abrasive must be employed to produce the open grain structure desired.

Sand and flint are both very good abrasives, but they powder rapidly in use and produce a dust which is dangerous to breathe; moreover, the use of sand or other substance containing free silica as an abrasive in blasting apparatus is now prohibited. Round steel shot has no cutting edges and is but a poor abrasive. On the other hand, crushed chilled cast iron is angular, hard and sharp, and possibly the best all-round abrasive to employ. It is more expensive than sand and flint, but it lasts 10 to 20 times as long before its efficiency is seriously reduced by pulverization. About 10 to 30 lb. per sq. in. is the range of pressures most used in sand-blasting operations preparatory to metal spraying. With steel abrasives, at least double the pressure is required to cope with the higher density of the metal abrasive. Fine angular shot used at the lower end of the pressure range is suitable for blasting non-ferrous metals and soft non-metallics, say No. 60 grit used at 20 lb. per sq. in. pressure, and the pressure is increased and a coarser grit is employed as the hardness of the material to be treated and the toughness of the skin to be removed increase. It is false economy to attempt to make do with air pressures which are too low. Below certain limiting pressures, the time for treatment increases enormously and the roughness produced may not be sufficient to promote good adhesion of the sprayed metal coating.

The nozzles of abrasive blasting guns are, naturally, subject to a good deal of wear. They may be inexpensively made from ordinary cast iron and replaced at frequent intervals, or they may be lined with some very hard material, such as boron or tungsten carbide. Lined nozzles are expensive, but they have the advantage of maintaining steady conditions over fair periods of time.

For best results the nozzle should be held at an oblique angle and not perpendicular to the work, at a distance of about 12 ins.

Porcelain, quartz and enamel are best given a preliminary matting of the surface by means of a very fine blast.

At this juncture, it is not inappropriate to remark that the metal-spraying operation itself is not without risk of injury to the health of the operator. Lead, cadmium, copper, brass, bronze, tin and zinc all give rise to various toxic effects and it would appear that aluminium is probably the least dangerous metal to spray from this point of view.

Appearance of the Sprayed Metal Coating

The appearance of a normal sprayed metal coating is always mat. As one would anticipate from its mode of formation, the deposition of metal particles of the size produced by the air blast of the spray gun which bond by a process of sintering rather than fusion can lead only to formation of a rough non-reflecting surface. Since metal spraying is usually carried out for purposes of protection only, this is not usually a disadvantage; in fact, it is a great advantage in cases where additional protection is afforded by subsequent coating with an organic coating composition, as will be explained later, since it provides a much better key for the coating. However, where appearance is of importance, sprayed metal coatings, particularly of aluminium, can be made to look much more attractive. Brushing with a fine wire brush imparts a satiny sheen of considerable decorative value. The brushes used on aluminium coatings should contain wires of steel or nickel silver and not brass, which might tend to discolour the aluminium surface.

A high polish may be conferred by ordinary methods, but, owing to the roughness of the sprayed surface, it is usually necessary to commence by emery bobbing followed by grease bobbing, mop with a tripoli compound and complete with a soft mop and finishing compo. In order to allow for the amount of metal removed during these operations, it is essential that the sprayed coat be initially not less than 0.015 in. in thickness. Hammered effects may be produced in the usual way, but care must be taken not to stretch the thin coat unduly, which would cause it to part from the under surface.

Chasing and patinizing may also be carried out. Patinizing is much employed on metallized articles of plaster of paris, terra cotta, etc., which are desired to resemble artistic metal ware. On such surfaces it is essential to obtain the patina by a dry process, using, e.g., hydrogen sulphide. If acids and liquids are employed they tend to soak into the under surface and then to seep out later, causing stains and other damage. This can be prevented by impregnating the article with paraffin wax before metallizing, but since this in turn reduces the adhesion obtained by closing the pores in the surface, it is not generally recommended.

Porosity of the Sprayed Coating

One would not anticipate that a coating formed by the deposition of metal particles, which agglomerate only through a process of sintering and whose surfaces include a fair proportion of oxide, would be very free from pores. Indeed, such is the case, and one of the chief objections to sprayed metals is porosity. Actually, with certain of the lower melting-point metals and by careful manipulation of the air pressure employed, it is possible to obtain conditions under which the deposited particles fuse together and produce a substantially non-porous coating. Rollason(15) mentions the spraying of a piece of metallic sodium with lead and tin to produce a coating so free from pores that no action occurred when the sprayed sodium was thrown into water.

T. Everts(23) has investigated in some detail the effect of various operating conditions on the porosity of the sprayed coat using a wire pistol and oxy-hydrogen flame. In the case of aluminium, increasing the thickness of the coating appeared to have very little effect, although thick coatings of steel showed a greatly reduced permeability to gases. The porosity of aluminium coatings increased rapidly when the distance of the nozzle from the surface was increased from 10 cms. to 15 cms. and decreased just as sharply when the hydrogen pressure was increased from 1.3 to 1.8 atmospheres. The presence of excess oxygen in the flame had very little effect on the porosity of the aluminium coats, neither had an increase in the air pressure employed, but in all cases an increase in the rate of feeding the wire over that normally employed tended to increase porosity due to a failure to liquefy the wire. The original paper contains data on a number of metals investigated and is most instructive, but, of course, it relates only to the particular pistol and system of spraying, fuel gases, etc., employed in the investigation. Under these conditions, the variables best suited to the production of sound aluminium sprayed coats are: spraying distance 2 ins., hydrogen pressure 2.4 atmospheres, oxygen pressure 2.3 atmospheres, air pressure 2.7 atmospheres, and rate of wire feed 18.3 ft. per min. A very considerable improvement was effected in this way, and it would appear that of all the metals investigated, aluminium coatings are the most susceptible to a reduction in porosity by attention to the actual spraying conditions.

Everts also attempted to estimate the number of pores in sprayed coatings by direct counting under the microscope. His results indicate that, in comparison with zinc and lead, the pores in sprayed aluminium coats are few in number but large in size. His actual figures are:—For a porous aluminium coating, 22 pores per sq. mm., each pore being 0.002-0.02 mm. in diameter; for a dense aluminium coating, 45 pores per sq. mm., each pore being 0.0003-0.0007 mm. in diameter; for a zinc coating, 1,000 pores per sq. mm. were found with diameters of between 0.0001 and 0.001 mm.

Schnorrenberg(24) has also investigated the effect of production variables on the porosity of sprayed coats, and has made recommendations for optimum values for use in spraying aluminium.

Treatments to Reduce Porosity

There are, however, a number of ways in which the porosity of a sprayed metal coat may be reduced, perhaps the best of which consists simply in the subsequent application of an organic coating composition which will also prevent deterioration of the metal coat. The combination of sprayed aluminium followed by an iron oxide paint gives really remarkable protection to iron and steel under the most severe conditions. Not the least important factor in this protective ensemble is the excellent key which the porous aluminium surface provides for the paint coatings. Not all organics are equally effective in this connection; iron oxide/linseed oil paint, e.g., has been found to give vastly better protection to sprayed aluminium on iron than unpigmented oil varnishes.

An alternative protection is to precipitate an insoluble compound in the pores of the sprayed metal coating by chemical means.

It is also possible to effect some improvement by mechanical means, such as polishing, hammering, wire brushing, burnishing and blasting with round shot, which amounts to much the same thing as burnishing. Articles sprayed in rumbling barrels working on the principle of the sand blasting barrel, are known to acquire a metal coating with high adhesion and very low porosity. No doubt this is due to the burnishing action of one article rubbing against another.

Everts(23) has recorded the results of some tests on the effects of heating, grinding and polishing on the soundness of some sprayed metal coatings. Measuring the soundness of a coating by the time taken for 1 l. of gas to penetrate 1 sq. mm. of the coating 0.5 mm. thick under an excess pressure of 1 atmosphere at 15 degrees C., he found that heating produced an eleven-fold increase in the soundness of aluminium coats, whilst grinding gave a 235-fold and polishing a 320-fold improvement.

In some cases readily fusible and flowable metals (tin and zinc) can be used to fill the pores of a sprayed metal coat. Thus porosity in sprayed brass or nickel may be eliminated by applying a coat of tin and then heating to about 240 degrees C. The tin melts and fills the pores. If the tin is applied as an intermediate coating between two layers, the appearance and other characteristics of the upper layer of the principal coating metal are not greatly altered, although the pores in it may be effectively sealed. Zinc is a particularly satisfactory underlayer for treatment with tin in this way, due to the production of a tin-zinc alloy, and this form of protection has given good results in the treatment of the interior surfaces of milkcans; the zinc alloy is said to be as harmless from the toxicity point of view as tin.

Thick coatings of aluminium are very satisfactory for filling cavities in iron, brass, copper and aluminium vessels. They do not exhibit any permeability to water, even under pressure, but metals other than aluminium used in this way are liable to give rise to some leakage.

Density of Sprayed Metal Coats

Schnorrenberg(24) has measured the density of a number of sprayed metals and compared these with the densities as cast.

He found that the density of the sprayed aluminium was some 9 per cent. less than that of the cast metal as compared with 6.4 per cent. for tin, 8.6 per cent. for zinc, 11.3 per cent. for bronze, 11.8 per cent. for brass, 14 per cent. for lead and 15.9 per cent. for copper.

This difference in density is, of course, a measure of the unsoundness of the coating. It is, however, not a very reliable measure, partly because castings may themselves be unsound unless very carefully produced and, therefore, not very suitable for use as standards of comparison and partly because it gives no information about the size and disposition of the voids, or about the oxide content, which naturally affects the density. Provided the voids are sufficiently fine, one unsound coating may be much less permeable than another exhibiting a small number of much larger pores, especially where liquids are concerned.

Abrasion Resistance of Sprayed Aluminium Coatings

Sprayed coats of unalloyed aluminium are rather soft and for use in conditions where they are liable to abrasion by dust particles, it is as well to protect them with a hard coat of paint. An alternative is to use a coating of aluminium alloy, employing methods such as those which have been developed for applying aluminium-base coatings specifically for resisting high temperature oxidation. In the Alumeticren process,(25), for example, aluminium is sprayed on to iron furnace bars. These are then treated at 1,000 degrees C. to produce an iron-aluminium alloy of considerable hardness. Similar alloys may also be formed at much lower temperatures. Alternatively, an aluminium-base alloy may be sprayed or aluminium and alloying ingredients sprayed simultaneously, using a powder pistol to produce an aluminium-base coating harder than unalloyed aluminium.

The Nature of the Sprayed Metal Coat

A great deal of speculation, false theorising and practical experiment have been undertaken in an attempt to solve the problem of why fairly sound adherent metal coatings can be produced using the flame pistol. It would be simple to understand if the metal particles reached the surface to be coated in the molten condition, but except when the nozzle is held very close to the object, this is not so, since combustible materials such as fine fabrics can be metallized without danger; the hand held a few inches from the nozzle receives a metallic coating and yet experiences little more than a warm blast of air, whilst, according to Rollason(15), a thermometer held in the spray 4 to 5 ins. from the nozzle of a gun spraying iron registers only about 70 degrees C.

Yet it is clear that, in the wire and molten metal pistols at least, the metal must be melted at some time or it could not be atomized. The theory has been put forward that the molten atomized particles are first chilled by the air blast, but that the energy of the cool particles on impact with the surface to be coated is converted into heat sufficient to cause the former to revert for a moment to the plastic state. Now, during this very brief period the particles become welded into a firmly adhering and coherent layer, whilst the high velocity air stream maintains the surface being sprayed at a safe temperature. This theory was first ventilated by Gunther and Schoop and although no one doubts that the air blast chills the atomized particles and keeps the depositing surface cool, the remainder of the theory was questioned almost from the first. Several authorities spoke against it in proceedings against German Patent 258,505 on April 27, 1909. Arnold(26) pointed out that photomicroscopic examination of sprayed metal surfaces proved only that the particles were in the soft condition when, or immediately after, they reached the surface being sprayed and gave no evidence to prove that any fusion or welding took place under normal conditions. He further showed that the kinetic energy of the particles was, in fact, theoretically insufficient for welding by fusion.

Other theories have been advanced, including one in which the cold metallic particles are assumed to be hammered into the pores of the surface by the great force behind them. The hypothesis favoured by Rollason,(15) which would appear to be the most plausible and which is founded upon a considerable volume of experimental evidence,* is that the particles are melted completely or partially in the flame, depending on the spraying apparatus, and cool fairly slowly while travelling in the air stream, since there is little relative motion between the two. When the pistol is held close to the article (1-3 ins.), the particles are still molten when they strike the surface. Splashes are formed which interlock. After deposition, the particles are rapidly cooled by the air stream which rushes by, and which also keeps the base material cool. At greater distances from the nozzle, the particles are cooled below their freezing point, so that the particles do not form splashes, but their kinetic energy is sufficient to deform them into a laminated packing

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* Rollason, Turner and Budgen's work on Metal Spraying contains a summary of the bulk of this evidence, together with a number of informative photomicrographs and X-ray photographs.

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SOME FACTS ABOUT LIGHT METALS . . .

Aluminium alloys are as strong as steel, machine as fast as brass, and are only one-third of the weight. In rapidly-moving machinery, light-weight aluminium alloys reduce wear on bearings, cut power costs. Aluminium is abundant and is cheaper than ever before

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enclosing fine pores. At still greater distances, the particles form a heaped sand-like mass with high porosity. It is possible that sintering processes, as understood in powder metallurgical operations, are involved. It is conceivable that the particles of metal reaching the surface to be sprayed are above the sintering temperature, but, unlike powder metallurgical processes, the pressures involved by the impact of the particles cannot be very high. Nevertheless, since much of the pressure employed in the sintering of powdered compacts is necessary to overcome the resistance of the grains to the movements required in compaction, it is not improbable that the pressure due to the impact velocity of the particles is quite sufficient to produce a sintered compact in thin layers at a time.

Finally, the possibility of under-cooling of the metal particles, due to the extremely rapid chilling, should not be overlooked. It is difficult to predict the possible extent of under-cooling under such conditions, and it may be that this is the key to the solution of this problem.

The Cost of Metal Spraying

To ask for the cost of metal spraying is rather like saying "how long is a piece of string?" It varies according to the size of the article, the material of which it is made and its condition, the ease with which it can be handled, the metal to be deposited, the method employed and the skill of the workmen. When edges are coated, a good deal of the metal sprayed is lost, whilst with heavy articles a good deal of time may be wasted manipulating the article.

The cost of metal spraying tends to be based on the area covered in contrast to galvanizing, which is charged on weight. Aluminium costs a little more than zinc and lead to spray, a little less than copper and considerably less than tin, steel and cadmium.

Some Applications of Sprayed Aluminium Coats

1. As a Protection Against the Corrosion of Iron and Steel

It is a good many years now since Sir Robert Hadfield made his much-quoted estimate of the cost to this country of the corrosion of iron and steel, and named a minimum figure of 28 million pounds per year. Evidence would seem to suggest that this is certainly not an exaggeration, especially when one also considers deterioration, which is due indirectly to corrosion. Within the past few years fundamental research by F. P. Bowden and his collaborators has shown that wear of attrition takes place only under conditions in which an oxidation or similar chemical action is possible, and the deduction has been drawn[27] that the great majority of discarded and worn-out machines are examples not of wear but of corrosion.

Many forms of protection against corrosion are, of course, commonly practised—painting, galvanizing, electroplating, e.g., but all methods have their limitations and few give protection for long periods on outside exposure. The one bright spot in the picture of rust and deterioration is the success which has been obtained in recent years with sprayed coatings of metallic aluminium. The versatility of the flame process for spraying aluminium is such that this method of coating is of almost universal applicability and the excellent resistance to corrosion which is conferred for long periods of time does offer the prospect of materially reducing the present losses through corrosion.

Fundamentally, there would appear to be two chief ways in which iron and steel may be protected against corrosion by the application of a coating. One is by the application of a coating which is unbroken and completely non-porous forming an effective barrier between the ferrous metal and the corroding environment. Such an ideal state of affairs is, of course, difficult to attain and is seldom reached in practice. When it is reached it is difficult to maintain since a deep scratch or a hard knock may penetrate or remove a small portion of the coating, in which case its continuity and maximum protective efficiency is destroyed. It is not limited to metallic coatings and, in fact, non-metallic materials are often safer to use. Paints and varnishes fall into this category. They function very largely by excluding the corroding medium, and when the latter succeeds in penetrating the pores of the coating, corrosion begins. The passivating effect of chromatic pigments, the sacrificial effect of metallic zinc pigments and other pigment functions may serve to delay attack, but ultimately corrosion is inevitable, and it proceeds under the paint film with ever-increasing rapidity as the latter is lifted to expose more of the base metal to attack.

Metallic coatings also may be employed to function as excluding agents, but they, too, can seldom be produced free from pores. Moreover, should they be higher in the electrochemical series than the base metal, there is a danger that they may accelerate corrosion at discontinuities in the coating. Electro-deposited nickel on steel is an example of a metallic coating protecting by exclusion. Thick coats of nickel substantially free from pores give good protection against atmospheric corrosion, but with thinner coats, which are always porous, rust spots soon appear at the pores and rusting is accelerated by the presence of the surrounding nickel. A pre-requisite of any material employed in this way for the reduction of corrosive attack is that it shall itself be stable in character and of adequate resistance to the corroding medium.

The second method of protecting a base metal is by the use of a metal which is more electronegative, but which is itself less readily corroded than the base metal. This is sometimes referred to as sacrificial protection, since, of the two metals concerned, it is the coating which is corroded first—sacrificed to preserve the base. Galvanized iron is an example of a combination of this type. This type of protection is obviously limited to metals on metals, since it involves the transference of electric charges, but it does not require a completely homogeneous coating. The zinc coating on galvanized iron continues to protect the iron from atmospheric attack even where the base metal is exposed at pores, scratches and cut edges.

Sprayed aluminium on iron, steel, copper-containing aluminium-base alloys, and on many other metals, appears to function in this way. Its protecting influence is not as powerful as that of zinc, but the beauty of the flame-spraying process is that it can be applied to fabricated articles and erected structures, so that any discontinuities in the aluminium coating are small in size and well within the range over which aluminium will exert a satisfactory protective influence. In comparison with zinc, tin and lead, which have been favoured for this purpose in the past, aluminium is more slowly oxidized by air and thus retains its appearance and protection over a longer period of time.

If one goes a step further and closes the pores in the sprayed aluminium coat, with an oil varnish or a paint, for example, then the coating begins to function in both ways, by exclusion and by a sacrificial effect at any discontinuities which may occur. This combination gives really outstanding protection. It is important to realize that paint failures on steel work occur principally through lifting by corrosion products formed initially at pores or other discontinuities in the coating. Organic coatings applied on aluminized iron are not subject to this defect and their life is very greatly prolonged.

There has been a considerable degree of controversy regarding the ability of aluminium to protect the underlying material at exposed parts, but the most reliable evidence is to the effect that it does do so in the case of steel specimens sprayed with aluminium, using the wire pistol, and exposed to atmospheric corrosion. For example Hoff(28), in long-time atmospheric exposure tests, observed no such effect: Britton and Evans(29), on the other hand, have reported very differently. In 1931 they exposed sand-blasted steel specimens carrying coats of sprayed aluminium of different purities and thicknesses at four stations, some of the specimens being doped with varnish or paint. In all cases, the aluminium showed itself capable of preventing rusting of steel exposed at pores, and this was presumed to be due to the conferring of cathodic protection; the aluminium itself was only slowly attacked.

Some particular specimens exposed at Cambridge were purposely scratched to expose the iron; along these scratches a little rusting was observed at first, but this soon ceased and after five years was inconspicuous, although uncoated steel specimens suffered actual perforation in about the same time. After seven years' exposure in London, Cambridge, or Grantchester Meadows (country atmosphere), and after four years' exposure at Selsey Bill (marine atmosphere), the specimens were still free from rust, although some change was observed in the aluminium layer, especially of the specimens exposed in London where softening had penetrated almost through the 0.003-in. coats and half-way through the 0.006-in. coats. This attack seemed to proceed sideways, undermining the aluminium flakes and causing the coat to rise in microscopic blisters or even to peel off when scratched, which lead the investigators to suggest that the deterioration might proceed more rapidly in situations where abrasion or bending stresses were present. It should be pointed out that this deterioration was less pronounced with relatively pure aluminium (iron 0.06 per cent., silicon 0.005 per cent.) than with commercial aluminium containing iron 0.7 per cent. and silicon 0.35 per cent.

It should also be noted that at all four stations the application of a single coat of ordinary iron oxide/linseed oil paint over the aluminium completely prevented the deterioration of the light metal, even in London, and that the paint retained its colour and good appearance long after similar paint applied directly to steel was destroyed by under-rusting. Unpigmented varnish, however, had not the same protective action as the iron oxide paint. So impressed was Evans with the results of these tests that he has suggested that the combined procedure of spraying with aluminium followed by painting should be applied to the protection of steel houses.

During the war, confirmation of the protective efficiency of aluminium spray plus paint was obtained from several applications of the process to steelwork operating under onerous conditions. Thus, interesting results have emerged in connection with the protection of cylinders containing hydrogen for balloon barrages. These cylinders had to be exposed to all sorts of conditions, including marine and tropical atmospheres, but the protective system of aluminium spray plus paint proved reliable and gave good protection under severe conditions.(11) These cylinders were made of high tensile steel with thin walls since it was essential to reduce weight to a minimum, and only a small amount of pitting would have had serious consequences.

Turner(20) exposed at the Forth Bridge steel test panels coated with aluminium to a thickness of 0.004 in. by the wire and powder processes and obtained rather different results by the two methods. The coatings applied by the powder method failed in less than a month, whilst those applied by the wire pistol withstood two years' exposure before failure finally occurred along the "strokes" of the application. As previously remarked, Sidery and Braithwaite(20) claim that better adhesion is obtained with the wire pistol than with the powder pistol, and that this is an advantage where the aluminium coatings are exposed to seawater spray, particularly at elevated temperatures.

Rollason(15) in intermittent salt spray and field tests obtained better results with the wire pistol than with the molten-metal pistol and with pure as compared with alloyed aluminium. Rollason also reports that some experiments of Gruber showed that coatings sprayed in the presence of inert gases were greatly superior in corrosion resistance to those sprayed in the ordinary way using compressed air. No doubt the influence of the method of spraying is connected with the content of oxide and, in the case of the molten-metal pistol, with iron pick-up from the container. It is a pity that no comparable results have yet been published for coatings on steel produced with a recent design of powder pistol and the low oxide-content aluminium powder which was developed during the war.

It is well known that the resistance of aluminium itself to corrosive attack increases with the purity of the metal, and that the alloys with copper and zinc possess a lower resistance than unalloyed metal of commercial purity. Thus, sprayed coatings of unalloyed aluminium have been applied to the duralumin parts of aircraft to afford additional protection. The value of super-purity aluminium for resistance to atmospheric corrosion and to many chemical reagents which attack aluminium of ordinary purity is now well recognized, but its high cost and low mechanical properties have stimulated interest in methods by which it might be applied as a protective sheathing at the same time without producing any serious contamination. This has proved easy enough in the case of sheet, the two metals being rolled together under the influence of high pressures and room temperature or moderate pressures and elevated temperatures, but rather difficult in the case of flame spraying due to the considerable reactivity of the molten metal and its high solvent action on other metals. We have already described a patented method of spraying super purity aluminium for the electrodes of electrolytic condensers using a molten metal pistol supplied with molten aluminium from a fused alumina crucible by compressed inert gas(22).

The atmospheric and chemical resistance of aluminium may be increased by anodic oxidation of the surface. This same treatment may be applied to sprayed aluminium coats, but is not to be recommended in view of the danger of electrolyte being trapped in the pores and seeping out at a later date, causing damage to the coating and perhaps to nearby surfaces on which the acid may fall. It is, in fact, an exaggeration of the problem encountered in anodizing porous aluminium castings.

Whether or not one metal will afford cathodic protection to another depends not only on their nature but also on the corrosive environment and the conditions under which corrosive attack takes place, including such factors as temperature, concentration of reagent, and aeration. Thus, Evans has shown(11) that steel specimens sprayed with aluminium have rusted in carbonate solution at cracks in the coating, but not in sodium chloride. Again, although under most conditions of atmospheric exposure aluminium is able to offer cathodic protection to iron and to copper-containing aluminium-base alloys, the reverse is true for magnesium and for alloys containing the compound $MgZn_2$. Considerable discretion must, therefore, be used in the application of electrode potentials to a prediction of the efficiency of an aluminium coating on another metal.

Resistance to Sulphurous Gases

Aluminium excels in its resistance to sulphurous gases which are so destructive to iron, steel and copper-base metals especially, and aluminium-sprayed articles retain this valuable characteristic to a large degree.

Rollason(15) has described some experiments of Gruber in which the latter sprayed aluminium, chrome iron and 18/8 stainless steel in carbon dioxide, nitrogen and compressed air and then exposed the specimens to hydrogen sulphide at elevated temperatures in a bubble tower of an oil refinery. It was found that aluminium headed the list from the point of view of corrosion resistance. Ballard(30) mentions an interesting case of a gas holder in an advanced state of corrosion which was effectively treated by sand blasting, spraying with zinc and then with aluminium. Evans(11) gives further details including the history of the gas holder and the composition of the gas.

On the other hand, sprayed aluminium does not seem to have been so successful on exposure to flue gases. Rollason(15) refers to experiments in which Pessel determined the relative resistance of 23 sprayed metals and metal combinations, in coatings 0.008 in. thick, to flue gas containing SO_2, CO_2 and air. Of the coatings tested only lead by itself and lead over Endura KA2 were found to protect the steel. Sprayed molten silver, copper and monel metal coatings were fairly intact, but all other coatings, including aluminium, were attacked severely with the production of sulphites and sulphates.

Erdmann(21) credits hydronalium (3–12 per cent. Mg., 0.2–0.5 per cent. Mn, 0.2–1.0 per cent. Si^1, remainder aluminium) with excellent corrosion-resisting qualities, and describes the application of the alloy to the protection of kitchen ranges, automobiles, motorcycles, household materials, etc. The wire pistol using light metal wires 0.04-0.06 in. diameter gave the best results.

Corrosion Fatigue

One very important factor to be considered in engineering calculations is the influence of corrosion on the fatigue of the metal in question. The effect of protective coatings on the corrosion-fatigue resistance of a 0.5 per cent. carbon steel such as is used for streamline wires in aircraft has been studied by Gough and Sopwith using rotating beam-type tests in the presence of a 3 per cent. salt spray. At the conclusion of the tests, the aluminium was covered with a white deposit and it was stated that aluminium spraying, plus enamel, gave about as good protection as galvanizing or skeradising, whereas spraying alone gave about 20 per cent. less protection. The corrosion-fatigue resistance of streamline wire steel in salt spray could be increased by a coating of aluminium plus enamel to such an extent that the endurance limit at 20 million cycles was little if any inferior to the fatigue limit of the bare steel in air. With aluminium spraying alone, the decrease in fatigue resistance was 20 per cent. against 80 per cent. for the unprotected steel.(15)

Sprayed Aluminium for Resistance to High Temperature Oxidation

Resistance to high temperature oxidation is almost as important as resistance to atmospheric and general chemical corrosion. Plain iron and steel parts such as furnace bars and carburizing boxes which are used at elevated temperatures oxidize and scale badly, the scale flakes off and the component soon becomes useless. Protection is afforded by the use of certain iron-base alloys which possess a greater resistance to high temperature oxidation than does plain iron. Structures may be composed entirely of such alloys as nickel-chromium iron, in which case their resistance to high temperature oxidation may be very high, but so also is their cost. For temperatures below about 750 degrees C. it is generally equally satisfactory and certainly more economical to employ a coating of iron-aluminium alloy for the purpose of conferring resistance to oxidation over a base of cast or wrought iron, the alloy coat being produced by spraying with aluminium and subsequently applying a heat treatment to promote alloy formation with the ferrous base. For many purposes, aluminized steel is satisfactory for exposure at temperatures up to 1,000 degrees or even 1,100 degrees C., but above about 750 degrees C. the strength of the iron base is likely to be reduced. The calorizing, alitieren and Servarizing processes for forming an iron-aluminium skin on iron and steel parts have already been described. Coatings produced by flame spraying and subsequent heat treatment are similar in character, of more general application due to the versatile nature of the spray process but inclined to be thinner than those produced by the methods previously described.

The object of heat treatment after spraying is to cause some aluminium to diffuse into the iron and an iron-aluminium layer to be formed. Diffusion is believed to occur in two stages, firstly, a dissolution of iron in liquid aluminium with the formation of a solution in equilibrium with the solid phase at a given temperature and, secondly, diffusion of the iron-aluminium solution into the iron. Aluminium is more soluble in ferratic than in austenitic iron which is believed to account for certain characteristics of the diffusion layer, notably the presence of columnar crystals and a concentration of pearlite in front of them.

Protection must be given to the sprayed aluminium layer to prevent oxidation of the light metal during the heat treatment process before alloy formation occurs. This is provided by coatings of such materials as bitumastic paint and the result is a compound coating which consists fundamentally of three layers; adjacent to and partly fused into the iron or steel base is a solid solution of iron and aluminium; next comes a layer of iron-aluminium alloy ($FeAl_3$), together with some free aluminium; and finally a thin coating of aluminium oxide. In subsequent use, the two outer layers soon flake away, but as long as the $FeAl_3$ is intact, oxidation and scaling are impossible. On exposures at temperatures above 1,000 degrees C., the light metal continues to penetrate into the steel, thus impoverishing the protective layer until eventually it is so reduced in aluminium content that oxidation occurs.

As with sprayed aluminium coatings for corrosion resistance, it is found that the resistance to high-temperature oxidation which is conferred by sprayed and heat-treated coatings of aluminium depends very largely on the conditions under which the coatings are produced and treated. It has been found that most metallic and non-metallic contaminants of the sprayed aluminium coatings and, in particular, the presence of oxide and silicon, have a harmful effect on the diffusion process. In consequence, it is not surprising to find that spraying for high-temperature oxidation resistance has almost invariably been carried out using the wire pistol, which is generally regarded as producing less oxide and other contamination than the powder or molten metal pistols.

As to the temperature of heat treatment, Commentz(25) recommends 1,100 degrees C., and claims that parts so treated will withstand products of combustion at temperatures up to 1,100 degrees C., provided the coating is not less than 0.6 mm. thick. This treatment is an essential feature of the German Alumitieren process which has been adopted for grate bars on various ships and on German railways, as well as for superheaters. In a later paper, Reininger(31) recommends that the heating be conducted at 1,000 degrees C., and that the sprayed aluminium coating be covered with a layer of flux to protect it from oxidation. In practice, however, temperatures much below these recommendations are commonly employed, particularly where the coating has less onerous obligations to fulfil.

Rollason(15) has described tests to determine the relative efficiencies of coatings of aluminium, "doped" in three different ways before heat treatment, with coatings of aluminium-cadmium alloy, 18/8 austenitic steel and the alloy nickel 65 per cent., chromium 15 per cent., iron 20 per cent., and with untreated mild steel, Silal and 18/8 nickel-chromium steel. All three types of pistol were used to produce the aluminium coatings, unalloyed aluminium being used for the wire and powder pistols and aluminium-silicon alloy in the molten metal process. The three doping and heat treatments were commercial processes patented in England, France and Germany. They comprised: (a) The English method. Coating with bitumastic paint, heating rapidly to 780 degrees C. and maintaining at that temperature for 10 mins. (b) The French method. Coating with a saturated solution of borax and annealing at 600 degrees C. for half an hour. (c) The German method. Coating with water glass, heating rapidly to 800 degrees C. and maintaining for 15 mins. The aluminium-cadmium alloy, also the subject of a British patent (No. 400,752), was heat treated at 900 degrees C. for 10 mins. The nickel-chromium-iron coating was sprayed from wire, and both this coating and that of 18/8 austenitic steel were annealed at 1,100 degrees C. for two hours without protection in order to form a diffused layer. The Silal specimen (6 per cent. silicon cast iron) was used in the form of a rod 0.8 in. in diameter.

The testing conditions comprised heating for 5-hour periods at 900-925 degrees C. in a muffle furnace, followed by quenching in water, this constituting an exceedingly drastic cycle as far as surface scale is concerned. The specimens were inspected and weighed periodically.

The results showed that the best protection with aluminium coatings was obtained using the wire pistol and the English (bitumastic paint) or German (waterglass) treatment; the French treatment consisting of coating with saturated borax solution and annealing at 600 degrees C. for half an hour, gave decidedly inferior results. The cadmium-aluminium alloy layer was very good although slightly inferior to coatings of unalloyed aluminium produced with the wire pistol and treated by the English or German processes. Less satisfactory was the silicon cast-iron specimen, whilst the behaviour of the mild-steel plate was, as was anticipated, very poor. The best results were obtained with the specimen sprayed with the nickel-chromium-iron alloy, using the wire pistol, which showed a negligible change in weight after 250 hours of the test cycle and with the solid plate of 18/8 nickel-chromium steel; on the other hand, poor protection was afforded by the sprayed coat of 18/8 austenitic steel. The actual durations of the heating and quenching cycle required to cause a 50 per cent. loss in weight by oxidation and flaking, as recorded by Rollason, were:—

Mild steel plate	7 hrs.
M.S. plate sprayed with 18/8 Ni-Cr steel	50 ..
Stainless 18/8 plate	300 ..
M.S. plate sprayed with Ni-Cr-Fe, using the wire pistol	250* ..
M.S. sprayed with unalloyed aluminium, wire pistol, English treatment	140 ..
Ditto, German treatment	150 ..
Ditto, French treatment	45 ..
M.S. sprayed with unalloyed aluminium, powder pistol, English treatment	90 ..
Ditto, German treatment	80 ..
Ditto, French treatment	60 ..
M.S. sprayed with aluminium-silicon alloy, molten-metal pistol, English treatment ...	85 ..
Ditto, German treatment	50 ..
Ditto, French treatment	15 ..
M.S. coated with cadmium-aluminium alloy	130 ..
Silicon cast-iron specimen ...	100 ..

* Negligible change in weight after 250 hrs.

In view of the considerable degree of protection against high-temperature oxidation, which is conferred by spraying with aluminium, it is not surprising that this process is finding increasing favour for the treatment of furnace doors and door posts, carburizing and annealing boxes, and similar applications. A large number of low-temperature furnaces has been constructed for use at temperatures up to 550 degrees C. with an inner lining of steel sprayed with aluminium. Rollason refers to a cylindrical furnace approximately 31 ft. long used for tempering work at temperatures up to 550 degrees C., which was constructed in this way. This particular furnace had an inner baffle providing a path for the circulation of the furnace atmosphere by means of a fan mounted in the back. He also refers to some bright annealing furnaces comprising a cylindrical bell, which is dropped over the base carrying the charge, the base itself being of metal-sprayed steel and the charge cover of steel, metal sprayed and heat treated. After heat treatment, the furnace bell is removed and the container is sprayed with cold water. Metallizing has an added advantage in such cases in that it prevents the very bad rusting which would otherwise occur on an untreated iron surface. Large tanks used for salt baths are aluminium sprayed and heat treated; Rollason also reports that aluminizing is specified for aircraft manifolds.

Where resistance to sulphurous gases, in addition to resistance to high-temperature oxidation, is involved, the use of sprayed aluminium becomes doubly attractive. Thus, aluminium-sprayed steel work has been employed in the construction of distillation columns used in the fractionation of tar and petroleum oils. Sprayed-aluminium coatings are also of value in eliminating the corrosion and scaling which is inevitably associated with the use of iron flue pipes and for the iron components of conveyor belts which have been adopted in gas-fired bakery ovens.

In the case of furnace installations, which are protected against high-temperature oxidation by the application by spray of metallic aluminium, it should also be remarked that the low emissivity of the light-metal coating is a material help in reducing heat losses by radiation.

Difficulties are sometimes encountered in providing against high-temperature oxidation of cast iron over long periods of time, due to the tendency of most grades of cast iron to grow under the influence of prolonged heat. As the aluminium-containing skin does not grow at the same rate, a crazed effect is sometimes obtained and oxidation then proceeds along the lines where the skin has fractured. An obvious answer is to use an iron which is less subject to growth. Unfortunately, they are expensive, but since many of them have also a high resistance to high temperature oxidation, they are sometimes preferred in spite of their cost. An additional cause of breakdown may be the formation of aluminium carbide Al_4C_3 which is decomposed by damp air.(16)

The use of aluminium spray to protect against high temperature oxidation is not limited to ferrous-base articles. It has been used with great success, for instance, on copper parts; a highly specific application of sprayed aluminium in this connection is the finishing of soldering iron bits to increase the life of the non-soldering faces. This it does partly by reducing oxidation and scaling and partly by preventing the adhesion (and alloying) of unwanted solder. The surface of copper articles so treated turns a characteristic yellow due to the formation of an aluminium bronze.

Numerous experiments have been made with the use of alloys of aluminium, but so far without finding anything superior to the pure metal. Aluminium-cadmium alloy affords almost as much protection as pure aluminium and gives a harder coating; silicon and oxide reduce the protective efficiency of aluminium coatings. According to Rollason, two German workers, Bardenheuer and Muller, have sprayed mixtures of aluminium and chromium (50 : 50), aluminium and nickel (30 : 70) and various mixtures of aluminium, chromium, nickel and silicon, but without success.

Sprayed Aluminium for Building-up

Thick coatings of aluminium are very satisfactory for filling cavities in iron, brass, copper and aluminium castings and fabricated vessels. They do not exhibit any permeability to water even under pressure, but metals other than aluminium used in this way are liable to give rise to some leakage. Sprayed aluminium is a convenient medium for the repair of defective aluminium-alloy castings and for building-up worn components, whilst it may also be employed for "welding" aluminium components together.

In view of the application of the flame-spraying process to such operations as these, it becomes of interest to know something of the mechanical properties of sprayed coats of aluminium of appreciable thicknesses. This aspect of the metallization process has been investigated by Turner and Ballard(32) who built up comparatively large solid bodies by spraying aluminium, copper, zinc, tin and iron into steel moulds. Bars produced in this way could be sawn, filed, planed, turned and machined in much the same way as ordinary solid bars, but the mechanical and other properties varied according to the direction of spraying. Thus, sprayed metal could be chipped in one direction without flaking, but at right angles to this flaking was liable to occur. Again, on turning a bar in a lathe a smooth surface could be obtained on two sides, but on the other two sides at right angles to the first a comparatively rough surface was often, though not invariably, obtained. Similarly in some Izod tests, there was noted a sharp difference in strength between the tests taken parallel to and those at right angles to the laminations. Aluminium sprayed bars exhibited tensile strengths of around 4,900 p.s.i., this being higher than the values obtained for zinc, copper and tin (4,610, 4,400 and 3,000 p.s.i. respectively).

Sprayed metal bars and, in particular, bell-shaped forms produced by spraying over an iron mandrel to a thickness of about ¼ in. exhibited a definite metallic resonance; in the case of zinc, curiously, this characteristic was not developed until a few days after it was sprayed.

Some Typical Miscellaneous Applications of Sprayed Aluminium

In the manufacture of automobile manifolds, where a very clean core is demanded, aluminium-sprayed cores have been used with great success.(15) Wiring has in many cases been eliminated owing to the support afforded by the sprayed metal coating. Hydronalium has been employed in Germany with great success in the manufacture of automobiles, motor cycles, kitchen ranges, and household goods. In some instances, the applied coatings have been polished.(21) Tank wagons used in the transport of chemicals and oils are sprayed internally and externally to protect them from corrosion by the material being carried and from atmospheric corrosion. Similarly, pressure vessels used in oil refining are sprayed internally with aluminium.

Jones records(27) that during the war aluminium spraying using the power pistol and high-quality low-oxide grades of powder, particularly when mixed with small percentages of zinc, was undertaken to an extensive degree for the protection of many articles sent to the Far East and used for the treatment of drinking water and the like. In all branches of the food industry aluminium is of importance because of its hygienic nature and its resistance to attack by many articles of food. Where violent stirring is an essential operation, solid aluminium is naturally to be preferred but for many purposes it is found that adequate protection is afforded to iron vessels by spraying thickly with aluminium and thus avoiding the cost of all light-metal construction.

Conversely, coatings of other metals may be applied to aluminium in cases where the light metal is exposed to attack. Thus duralumin rollers used, for lightness, in the manufacture of photographic films are coated with nickel to prevent reaction between the film and the aluminium. Again, during the war, unalloyed aluminium was sprayed on to light-alloy cylinder heads at the works of the Wright Aeronautical Corporation, Paterson, New Jersey. Extensive experience had shown that for sea-going aeroplanes some form of protection was desirable on the light-alloy cylinder heads of sea-going planes fitted with air-cooled engines to guard against the inevitable corrosion caused by salt spray. Enamelling was tried but proved relatively unsatisfactory under service conditions. Eventually, it was determined that the most favourable conditions were obtained by spraying with unalloyed aluminium, the metallized coating so obtained proving to be highly resistant to salt-water attack.

This application is of particular interest in view of the high degree of mechanization of the process which had eventually to be undertaken to cope with the enormous output under the U.S.A. defence programme. Details of the steps by which this was achieved have been given in earlier pages of this Journal,(33) and constitute probably the most important of the few published accounts of the application of mass production methods to the flame-spraying technique.

At first sight, the Wright cylinder heads appeared to be a poor subject for automatic spraying, owing to the complicated form of both the head and the barrel, and hand spraying was performed for some little time. However, when an increase in output beyond the limits of manual handling became imperative, application was made of simple rotating tables and rising and falling spray guns, undercuts and narrow gaps being finished off by hand. From this first step, the degree of mechanization was slowly increased until, finally, a six-stage process was evolved which required little more than an occasional hand spotting to make good minor defects. A special "Wheelabrator" sand-blasting machine was built by means of which the time required for pre-treatment was reduced from 15 minutes to 65 seconds. Mechanical slinging of the abrasive was found to give better results than compressed air, and in this machine seven separate throwing wheels revolving at 2,250 r.p.m. were used to throw 10 lb. of abrasive against the outer surface of each cylinder. In practice, the complete cylinder assembly was inserted into the blasting machine, small components being protected by wooden blocks, ports and machined faces by masks, and cylinder skirt and flange bottom by a heavy rubber cuff on the supporting arbor.

The machine, built to make aluminium spraying of cylinder heads and barrels automatic, comprised six stations, each controlled by reduction gears and fully adjustable to various motions and speeds. The parts being metallized were progressively covered with an increasingly heavy coating of aluminium by metal-spraying guns fed with town's gas and compressed air.

In the first station a cylinder head was loaded and automatically adjusted. It moved to the second station for the first or strike coat. Here the cylinder head rotated on a turntable, but, instead of being moved alternately up and down, the turntable remained in a fixed horizontal plane. A spray gun, mounted on an hydraulically operated post, rose and fell, subjecting the revolving cylinder to a spray of aluminium from two different directions, as it changed angle at the top and bottom of its movement.

The next station of the machine brought the cylinder in front of two guns mounted at fixed opposing angles, which likewise moved up and down, spraying the upper and lower fin surfaces. At the fourth station, two more guns, also mounted at opposing angles, but moving above the level of the guns at the third station, sprayed the side surfaces of the head only. At the fifth station manual labour finally took over for the operations of spraying the upper head surfaces. At the final station, No. 6, the cylinder was inspected, touched up with a hand gun if necessary, and unloaded.

Each gun had its own gauges and controls mounted on a panel on the upper part of the machine, within easy reach of the operator. Compressed air was supplied by a 60 h.p. six-cylinder water-cooled Schrann air compressor with a 260 cubic-ft. delivery. The air was dried by passing it through a water-cooled air drier of 300 cubic-ft. capacity. An exhaust fan connected by a duct to the top of the machine removed the fumes of the burned gases, but, as an added precaution, operators were provided with respirators to keep out airborne metal dust.

In the bleaching and dyeing industries, sprayed aluminium is of importance for the coating of much of the plant and accessories which the processes undertaken require. Aluminium has a specially important place in the bleaching industry because it is the only practicable material to use, and, at the same time, is neither affected by nor has any gross deleterious action on hydrogen peroxide. There is no doubt that such equipment is best constructed either of solid aluminium or of some material such as wood lined with sheet aluminium. However, this may not always be possible on the grounds of expense. In such cases sprayed aluminium offers a useful alternative at moderate cost. Again, there is some reluctance to scrap existing wooden equipment or accessories in favour of aluminium, but these wooden components can be given a moderately thick coating of aluminium by the flame-spraying process, thus providing the advantages of aluminium construction to a limited extent without the necessity for new equipment. Perhaps the greatest advantage of the flame-spraying process in this direction is the fact that it enables the application of aluminium to be made to fully fabricated plant in situ. Gaps and crevices and faults in aluminium linings may be closed or bridged; components in other materials, such as bolts and rivets, joints and repairs in silver solder or other material may all be covered with a uniform layer of aluminium possessing a chemical resistance commensurate with its freedom from contamination.

Evans has reported(11) a very different application of sprayed aluminium, namely, for preventing cavitation on the cast-iron propellers of trawlers.

Sprayed aluminium is used for various purposes to cover non-metallic materials. Wood is one example and the value of aluminizing on wood is mainly a decorative one. Sprayed coats of aluminium on wood do not reduce the susceptibility of the latter to changes in moisture content and dimensions with variations in humidity, neither do they reduce the absorption of water on soaking. This applies to all the higher melting metals applied by the flame-spraying process (bronze, brass and copper in particular), but metals of lower melting point (tin, lead, and zinc) have been found to reduce the sensitivity of wood to changes in the humidity of the atmosphere by from about one-half to one-third(15). Frequent swelling and shrinkage of the wood due to changes in moisture content does, however, weaken the bond, never very great, between the metal film and the wood surface, resulting in cracking and later peeling. Thus, it appears that the major application of sprayed aluminium on wood is likely to remain a decorative one for the moment at least.

The same is not altogether true of the aluminization of other non-metallics. Thus, although the striking results produced by the aluminization of dress fabrics and lace, sometimes using stencils to obtain patterns, is for effect only, it is stated that airship and balloon fabrics have been treated in this way. Not only does this add to the life of the fabric but, in addition, the surface is made electrically conducting which greatly reduces the danger of fire following the discharge of static electricity.

Aluminium spraying has been used for the metallizing of paper and board, although this type of material is now more usually produced by the adhesion of aluminium foil or by printing with an aluminium-pigmented ink. According to Rollason(15) the metal spray process has been used in several factories for coating embossed or moulded paper, cardboard or papier-maché goods, whilst a manufacturing works at Frankfort utilized the process for producing carbons with capacities up to 20 gallons and hoop-bound barrels of 70 gallons capacity from specially prepared and hardened paper cylinders. These cartons and barrels were supplied to dyeworks, and to manufacturers of chemicals, margarine, soap, foodstuffs and other articles. The metal spray was applied after fabrication of the carton and no doubt it helped to seal the joints. Cinematograph projection screens have been made by spraying aluminium onto a base of linen, cardboard, papier-maché or glass; for this application the mat surface of brilliant aluminium particles which is produced is ideal. Nameplates have been produced by spraying aluminium onto a plate glass or other surface over a suitable stencil, whilst other applications of aluminium spray include the decoration of shop fronts and exhibition stands of wood, plastic, etc., to give them the appearance of solid metal, and the coating of plaster casts and effigies.

One application of the metallizing process which has received considerably more attention in other countries than in this depends on the reflectivity of the aluminized surface. We have already referred to the production of cinema projection screens by spraying aluminium onto a suitable background "canvas." Similarly, circular cast glass pavement lights have been aluminized on their lateral faces, the reflection from which has greatly enhanced their light transmission.

But, in this paragraph we are thinking more of the reflection characteristics of polished sprayed aluminium coats and especially of polished sprayed aluminium-magnesium alloys. For many purposes a dry method of decorative plating would be extremely welcome and this development may well possess important future potentialities.

In view of the fact that the individual particles of deposited metal are insulated from one another by oxide films, except perhaps at points of small surface area where sintering has taken place, it is not unexpected that films of sprayed aluminium possess a much higher resistance than that of the cast metal. It is, therefore, quite possible to employ such films as heating elements, and when these take the form of patterns sprayed over stencil onto a glass surface some surprising possibilities are revealed. Thus, a radiator built up from two panels of glass each 50 by 40 cm. was sprayed with aluminium in the form of a Greek fret consisting of 30 strips each 45 by 10 mm. Each panel consumed 1,000 watts per hour at a pressure of 110 volts and reached a temperature of 130 degrees C. Such a radiator occupies little space, is elegant and modern in appearance and can be manufactured very cheaply.

An alternative possibility is to build up floors of glass blocks (or other heat-resistant insulating material) with an aluminized undersurface made to operate at a temperature of 30 degrees C.

Aluminium Paint

Perhaps the most universal method of applying a coating of aluminium in situ consists in the use of aluminium paint. If the organic medium is driven off by heat, there is little other than aluminium left behind and this is capable of alloying with the undersurface in the same way as sprayed aluminium; on the other hand, if aluminium pigment is applied in a leafing medium and allowed to stay in that condition after drying, the surface layer consists of interleaved aluminium flakes covered with such a thin layer of organic medium that they do constitute, to all practical purposes, a layer of metallic aluminium though with a very different structure and totally different method of adhesion from aluminium coatings applied by the flame-spraying process. However, aluminium paints and lacquers are a subject in themselves, and since the structure of the aluminium layer produced and the mechanism by means of which it adheres to the undersurface are so different from those of aluminium coatings produced by any of the other processes of aluminization dealt with in this article, it is not proposed to deal with them in any detail. This section is, in fact, no more than a cursory glance at the subject to see how aluminium-pigmented organic coatings line up with other methods of aluminization.

Taking the first of the two cases mentioned above, painting with aluminium paint and subsequently driving off the organic medium by the application of heat (or by allowing it to be driven off by the heat developed in service), does constitute a method of applying aluminium for the purpose of increasing resistance to high-temperature oxidation. Since, however, the thickness of aluminium applied in one coat is very small, resistance to oxidation is only effective at relatively low temperatures (up to 400 degrees C. or so). Moreover, although the organic medium does tend to act as a flux during the carbonizing process, facilitating good adhesion of the aluminium to the iron base and protecting it to some extent from oxidation, nevertheless, considerably more oxide is formed than during the heat treatment of sprayed coatings using the English or German methods previously described.

In this connection, choice of vehicles is all-important where the temperature of exposure is likely to exceed about 200 degrees C. For temperatures between 250 and 400 degrees C., gloss oil and spirit varnish types of vehicle, and, best of all, synthetics with high volatile content generally give the best results. With a suitable vehicle and provided the iron surface is clean and free from grease, rust and paint before application of the aluminium coating, a remarkable degree of adhesion is obtained giving one the impression that an alloy has been formed; this, however, does not occur. Below 200 degrees C., the choice of vehicle is less important, but it is generally preferable to use one of little body, that is to say, of low viscosity, and to dilute it to give a freely flowing paint. In other important respects, emissivity, corrosion and chemical resistance, etc., these coatings are not greatly different from those produced by spraying, except in so far as their greatly inferior thickness limits the degree of protection afforded and renders them more vulnerable to mechanical damage and attack.

Coatings of the second type are very different from sprayed metal coatings in another important aspect, namely, their brilliance and low porosity. These characteristics are due to the peculiar property of aluminium flakes, in a leafing medium, of tending to concentrate at the surface of the medium, and to arrange themselves in overlapping layers of tiny plates each more or less parallel to the surface. In consequence, we find that aluminium paint coatings are often applied where increased visibility or reflection is required, e.g., on bridge facings and inside factories. B.P. 578,887 claims the application of a phosphorescent material to such building materials as wood, plaster, brick, stone and concrete over an undercoat of aluminium paint, the function of the aluminium paint being to provide a good reflecting background for the phosphorescent material. The organic medium in which the aluminium powder is suspended tends to close any remaining pores and crevices and thus a metallic paint coating of remarkably low porosity can be obtained.

The aluminium coating applied in the form of paint retains the chemical and atmospheric corrosion-resisting characteristics of the massive metal or sprayed aluminium, although again its efficiency is somewhat limited by the thickness of metal which can be applied in a reasonable number of coats. It is, therefore, widely employed for protecting structures of all types and of various materials from the deteriorating effects of many agencies. Unlike sprayed coats of aluminium, which adhere to the base metal by the forces which operate between metals in extremely close contact, aluminium paint relies upon the adhesion of an organic medium which itself determines the life of the coating and the length of time for which the aluminium coating remains efficient.

Nevertheless, suitably formulated and carefully applied aluminium paint coatings are capable of performing very careful service. Edwards(34) quotes the results of a number of field and laboratory tests to illustrate the efficiency of aluminium paint both as a primer and as a top coat in the protection of iron and steel against rust. In some early tests it was found that one coat of aluminium paint over one coat of red or blue lead, or two coats of aluminium paint were still in very good condition after 52 months' exposure at Washington. In tests carried out to determine the suitability of paints for protecting steel water tanks, aluminium paints proved to be as efficient as hot bituminous coatings and superior to any of the other 12 classes of organic protective examined.

As with all other forms of aluminizing, the care with which the process is carried out has an important effect on the results obtained. In comparison with other processes, however, there are more variables attached to the painting treatment when one takes into consideration the number of possible variations in the composition of the paint, and Edwards gives some information on the effects of varying pigment concentration, particle size of pigments and vehicle characteristics.

Owing to the presence of a thin film of oil over the aluminium layer and also to a slight darkening or dulling of the aluminium flakes which inevitably occurs during the comminution process, the reflectivity of the aluminium paint film never equals that of the polished massive metal, polished sprayed coatings of aluminium or deposits on polished surfaces produced by the processes of evaporation or cathode sputtering which will be described later. In general, the average performance of good paints varies between 60 per cent. and 75 per cent., but this is still high in comparison with most other organic and inorganic coatings and, in consequence, aluminium paint is often employed in factory and warehouse interiors to increase the general level of illumination obtainable with existing lighting installations. Objects on the road have been coated with aluminium paint to render them more easily visible at night: an aluminium-painted bridge has been shown to be more readily visible to an oncoming motorist than one freshly coated with white paint whilst the deterioration of reflectivity with age is greater with white paint than with aluminium. Aluminium paint on the iron framework and apparatus in the telephone exchange does much to increase the attractive appearance and lighting efficiency of the exchange rooms.

On the exterior surface of oil tanks, aluminium paint is of value not only for protection against corrosion, but also for reducing evaporation losses by steadying the temperature inside the container. During the day the high reflectivity of the aluminium paint coating minimizes absorption of heat from the sun's rays, whilst during the night its low emissivity (about 30 per cent. of that of a black body) reduces the dissipation of heat to the cooler atmosphere. Sheet-iron magazines for the storage of explosives and refrigerator cars are two further examples in which aluminium paint is employed because of its high reflectivity and low emissivity to reduce temperature variations.

The reflectivity of aluminium paint is influenced by the care with which the powder is made and the degree of leafing which occurs, but the colour and opacity of the medium employed has little effect provided leafing occurs. This is because in such cases the film of oil over and above the leafed layer is too thin to have appreciable influence.

In electrical characteristics, aluminium paint films are very different from other types of aluminized coatings. Where leafing takes place, the tendency is for each flake of metal to be surrounded first by an exceedingly thin film of polishing agent and then by a thicker (but still quite thin) film of paint medium. Since both polishing agent and paint medium are non-conductors, leafed aluminium paint films have generally a very high electrical resistance to moderate voltages; above a certain potential, generally around 100 to 300 volts, the resistance of these films may be broken down and current will then pass. Where leafing does not occur, the film of polishing agent is absent and the reduced suspension of the pigment in the vehicle results in the former squeezing out the vehicle from between the flakes which can then make contact. Thus, non-leafing aluminium paint coatings will often conduct electricity under much lower potentials. Aluminium paint may, therefore, be employed as an insulating or a conducting medium according to circumstances.

The low permeability of aluminium paint films to moisture is a property of great practical value and in opposition to the characteristics of aluminium coatings produced by the methods of aluminization previously described. Moisture permeability is affected by the thickness of the film, the grade of powder employed and its concentration in the paint, and on the nature of the medium employed. Apart from the value of the characteristic in protective applications of aluminium paint on wood and metal, it also makes aluminium paint of value as an intermediate coat over bitumen or creosote to prevent these materials "bleeding" through the top coat.

The resistance of aluminium to sulphur and sulphur fumes is retained in aluminium paint films and makes the latter of value for use in many chemical environments, in applications ranging from the painting of outside steel work at gas-producing plants and oil refineries to the painting of laboratory fume cupboards. The use of white lead in such applications is ruled out owing to the unwelcome formation of black lead sulphide and the use of such pigments as zinc oxide and lithopone, the colours of which are not affected by sulphurous fumes, leads to a much lower durability, a disadvantage which is not attendant on the use of aluminium paint.

Electrodeposition of Aluminium

The methods of producing coatings of aluminium so far described are practical methods with wide applications which are in regular use to-day for the treatment of considerable quantities of iron, steel and other materials. We come now to three methods of more limited application, of which the first, the electrodeposition of aluminium is, at the moment, no more than a laboratory curiosity. It has long been appreciated that aluminium cannot be electrodeposited from aqueous solutions. Deposition is, however, possible from a fused electrolyte containing aluminium and sodium chlorides at 160-200 degrees C. and this method has been given some study(35). Another method developed by Blue and Mathers(36) makes use of the compounds formed by dissolving aluminium bromide in benzene, with xylene as a brightening agent. Both those methods suffer from grave disadvantages; the first is due to the use of a fused electrolyte which produces incrustations of salts which have to be removed and the temperature of which is liable to give rise to troubles by the distortion of parts of small cross section. In the case of Blue and Mathers' method, disadvantages are inherent in the organic materials employed as solvent and brightener, in particular their toxicity and inflammability.

It would thus appear that electrochemical processes for the deposition of aluminium in their present stage of development are not suitable for practical application. Convenient alternative methods of deposition are available (flame spraying, cathode sputtering, evaporation). Some of these automatically produce a smooth, highly reflecting film of aluminium on a polished surface, but after flame spraying, buffing is necessary to obtain a bright finish. It is interesting to note that polished flame-sprayed coats of aluminium-magnesium alloy were officially recommended in Germany as a substitute for polished electro-nickel plate. Cathode sputtering and evaporation processes which enable a highly reflecting film of aluminium to be deposited on a polished surface might therefore be compared with the modern bright nickel processes of electrodeposition which also eliminate the need for polishing.

Evaporation Processes

The techniques of both the evaporation and the cathode sputtering processes have been dealt with in some detail in earlier pages of this journal.(37) Hence, in as far as practical details are concerned, the account here presented is no more than a brief summary, and the earlier contribution should be consulted for fuller information.

As its name implies, the evaporation technique for the deposition of aluminium (and certain other metals) involves, fundamentally, evaporating metallic aluminium and allowing some of the vapour to condense on the article to be coated. The apparatus employed consists essentially of a chamber capable of maintaining a high vacuum and the pumping equipment for providing this vacuum. Within the chamber are a support for the object to be coated and the mechanism for holding and evaporating the aluminium.

The form of the chamber and the material of which it is constructed vary widely according to the nature of the object to be coated. In dealing with small parts, glass bell jars of heavy construction, as used in the laboratory, standing on an accurately ground glass slab will suffice. In other cases steel jars are employed, whilst in coating larger objects such as telescope mirrors with a diameter of, say, 3 ft. or more, a steel chamber is built up round the object which itself rests on a steel slab. Naturally this is expensive and time-consuming, but in certain specialized applications the results achieved by the evaporated aluminium film are so striking as to make the operation well worth while.

The joint between the chamber or jar and the base plate must be vacuum tight, since a high vacuum is necessary partly to protect the metal from oxidation and partly to lower its boiling point. This is achieved first by accurately grinding the bottom rim of the jar on to the base plate and, secondly, by the use of beeswax, picein wax or, preferably, one of the Apiezon compounds. An electric heating element is often included round the periphery of the base plate to soften the wax and assist in making the vacuum seal. Only the minimum amount of wax should be employed and it should be confined to the outer edges of the rim of the jar to minimize contamination of the atmosphere inside the jar which renders evacuation more difficult.

Vacuum connections are taken through the base plate to a suitable pump; diffusion and oil pumps have both been used satisfactorily, the latter evacuating down to a pressure of 1 to 10 mm. of mercury. The pressure required for the evaporation process is lower than this, around 10^{-4} mm. of mercury, and this further exhaustion is achieved by the use of an activated charcoal-liquid air system attached to the vacuum connection. During the initial stages of exhaustion the activated charcoal is baked out with an electric heater. Later, the level of the liquid air is raised to cool the lower portion of the charcoal which then absorbs the bulk of the gas, leaving the upper layer still active. Raising the liquid air still further brings these active layers into operation for the final stage of absorption. An indication that the required vacuum has been obtained is the absence of any discharge when the base plate and the object or the filament are connected to the opposite sides of a spark coil.

In some forms of apparatus a device is sealed in the system between the bell jar and the carbon trap to give some indication of the vacuum obtained inside the bell jar. In one instance, this comprised a discharge tube having two aluminium discs as electrodes 24 mm. diameter and 12.8 cm. apart. Potential was applied to one electrode from a hand-operated Tesla coil and the other was earthed, when the form of the discharge gave an approximate idea of the pressure in the jar; when the pressure was sufficiently reduced, discharge ceased.

Considerable investigation has been directed towards ascertaining the best method of handling and evaporating the metal to be deposited, as a result of which there are three principal methods in use to-day, namely, direct evaporation of the metal off an electrically heated coil, evaporation from the surface of a ceramic or silica container heated electrically by an internal coil, and the use of a condenser discharge to " explode " the material to be deposited. In the early experiments both tungsten and platinum wires were successfully employed for the heating coils in the direct-evaporation method. The material to be evaporated was merely slipped in the coil, the equipment evacuated, and then the coil was rapidly raised electrically to the required temperature for evaporation by contact. During the brief liquid stage, surface tension would hold the material in the coil.

The success of such an arrangement must depend on several factors. Thus, the metal must adhere to the filament over the full range of operating temperatures; that is to say, the molten metal must wet and adhere to the filament and must not fall off in drops. This presupposes that chemical reaction between the two metals to be evaporated and the filament must be non-existent, or at least insignificant. It is also to be preferred that no alloying takes place between the two metals. If an alloy is formed with a melting point below the temperature at which complete evaporation occurs, the result is disastrous. A further requirement is that the vapour pressure of the coating metal at a temperature below the melting point of the filament material must be sufficient for its rapid and complete evaporation. For the evaporation of aluminium, it would appear that the most satisfactory filament materials, in order of merit, are tungsten, tantalum, molybdenum and columbium, and, in point of fact, the filaments most generally employed in practice are molybdenum and tungsten. Whilst satisfactory results have been achieved with platinum filaments for the evaporation of aluminium, failure is more common due to the burning-out of the filament. This is probably the result of the formation of a low-melting-point alloy. Care is necessary in the use of tungsten filaments to obviate a similar, but less grievous, effect. The solubility of tungsten in liquid aluminium is limited, and provided the filament wire be of sufficient thickness, saturation of the aluminium is reached before the wire diameter is dangerously reduced. One experimenter employed a coil of tungsten wire 1/32 in. diameter spaced eight turns to the inch. With closer spacing, the aluminium was found to short the turns and cause inefficient heating, whereas with wider spacing the coil burnt out, due to excessive ratio of aluminium to tungsten. The dissolved tungsten is, naturally, deposited back on the coil as the aluminium evaporates and, between each use, the coil may be heat-treated to sinter it. The coil used was about 1 in. long and $\frac{1}{8}$ in. internal diameter, and the pure aluminium to be evaporated was cut from foil and rolled into a cylinder to fit easily into the helix. Such a coil would be used for about a dozen evaporations.

Hiram W. Edwards(38) noted that if he used a short piece of aluminium wire laid inside a tungsten coil, failure usually occurred due to arcing when one end of the wire broke contact with the coil. This led to his using a number of pieces of wire hung from the openly spaced loops of the heater coil, and later to a similar arrangement using a straight filament wire. As each piece of aluminium melts, it draws up to globular form on the wire, then evaporates, with reduced danger of the filament burning out. Next, he successfully tried using a heater wire in the form of a vertical sine wave, supporting the aluminium, cut into H-shaped pieces, on the waves. Again, two straight heater wires, spaced about a millimetre apart in a horizontal plane, were used with the metal supported on them. This gave better contact and evaporation at a lower heater temperature.

In modern commercial evaporation processes for aluminium, Edwards's ideas are often adopted. Thus, a common procedure is to use a coiled heater arranged horizontally with the aluminium cut into the form of "flags" which are inserted between the turns of the coil. A number of such heater coils are suitably arranged in the same chamber to produce the quantity of evaporated metal and distribution required.

As with other methods of producing coatings of metallic aluminium, the more free from contamination the better the characteristics of the condensed film. Increased freedom from oxide inclusions can be obtained by melting the aluminium in a helix in the absence of the object to be coated, this freeing the aluminium from oxide and gas. The object to be coated is then introduced and the evaporation proceeded with normally.

When the material to be evaporated reacts with the filament, an indirect method of heating may be used consisting of a small crucible of magnesia, alumina or graphite, which may be fitted into the filament. This method of evaporation is said to be preferred for magnesium, although it is also stated that satisfactory results have been achieved with filaments of tungsten, tantalum, molybdenum and columbium, as with aluminium.

Metal deposition processes in general require very thorough cleaning of the surface of the object to be coated in order to obtain adequate adhesion, and the evaporation process is no exception. Metal articles are thoroughly degreased, descaled where necessary and preferably given a mild etch. Non-metallic articles are degreased and preferably slightly etched or roughened by methods or reagents appropriate to their chemical and physical properties. It is important that all traces of polishing compounds and detergents be removed, and where possible it is an advantage to "glow" the article in the vacuum chamber before commencing evaporation. Glass may be given a preliminary alkali wash, using a mild alkali (sodium carbonate, silicate, phosphate mixture), then a water wash, followed by a sulphuric-chromic acid soak and water wash. The cautious use of dilute solutions of alkali fluorides in dilute sulphuric acid has also been suggested. With glass it is important to remember that this material does not readily give up its absorbed water, and deposition on to this material heated to 200 degrees C. yields a more tenacious coating.

Evaporation takes place in all directions, and, therefore, to minimize contamination of the apparatus and the extent of cleaning required, some form of screening is often employed. Copper discs and mica diaphragms may be used for the purpose. Alternatively, a number of objects may be arranged round the filament or filaments to make the maximum use of the evaporated metal.

Production of a condensed film is not necessarily limited to the deposition of pure aluminium or any other pure metal. Certain alloys can be produced as evaporated films by means of this apparatus by the simultaneous deposition of the ingredients from separate heater filaments. Again, it has been possible to "explode" alloy wires by means of condenser discharge and so deposit them. When alloys are placed in the heating spiral the results obtained depend upon the vapour pressures of the constituent metals under the conditions of evaporation. In experiments, speculum metal was a success, probably because tin and copper have approximately the same vapour pressure at the evaporation temperature. Brass fractionated, zinc depositing before the copper; similarly with jeweller's silver, silver deposited before copper. It is possible that the high magnesium alloys, deposited from two heaters, may prove to have valuable practical applications. Neither is the technique limited to the evaporation and condensation of metals. Non-metallic materials which have been evaporated include quartz, fluorite, alkali halides and silver chloride. Glass itself fractionates, leaving an opal residue.

Any material can be coated by this process provided it will withstand the high vacuum conditions; there is no appreciable elevation of temperature, and such materials as paper, cloth, rubber and nitro-cellulose may be safely coated. The thickness of deposit is controlled by weighing the amount of material to be evaporated. In practice this is very reliable and permits of the production of films of accurately controlled thickness, for example, for the preparation of partially reflecting films for interferometric work. Attention must be paid to the distance from the filament to the object since obviously the density of deposited metal decreases rapidly as the object is moved away from the filament. On the other hand, uniformity of coating is improved by using a large filament-object separation.

One piece of apparatus incorporated a device to watch the rate of evaporation. A potential of 200 volts was maintained between filament and metal base. This activated the hydrogen used for cleaning the metal to be evaporated and made the evaporating metal become luminous. With correct rate of evaporation, the discharge was bright enough to be seen in a well-lighted room. Incidentally, the discharge was thought to improve the adhesion of the evaporated metal to glass.

Aluminized Mirrors

The applications of condensed films of aluminium include the coating of quartz crystals for oscillators, aluminium being employed for contact and capacity reasons, the coating of evaporated silver films, the thin film aluminium oxide subsequently formed giving an important increase in tarnish resistance, and the coating of glass and quartz to produce front-surfaced mirrors with high reflecting efficiency and low rate of deterioration for precision optical instruments and as internal mirrors for infra-red lamps for radiant heating equipment.

Of these probably the most important and certainly the most spectacular is the application of the evaporation technique to the production of front-aluminized mirrors for use in telescopes and other optical instruments. Front-surfaced mirrors are essential wherever accurate observations are required to eliminate the multiple reflections and lower reflecting efficiency of an ordinary silver- or mercury-backed plate-glass mirror, but of course it exposes the reflecting surface to tarnishing and mechanical damage. The reflectivity of pure aluminium evaporated films for visible light is nearly as good as that of silver, which is the finest reflector material known. An aluminized mirror possesses roughly the same reflectivity as good quality silver-backing and is greatly superior to mercury-backing. In the ultraviolet region, aluminium has a reflectivity greatly superior to silver and, indeed, to other reflector metals. This is a factor of the greatest importance when it is remembered that most modern astronomical work depends on photography by low intensity illumination.

An equally important point, however, is the low rate of deterioration of aluminized mirrors, which is believed to be due to the formation of a very thin natural oxide film, the same type of film, incidentally, which has given such good results in the protection of silver films from tarnishing. Such mirrors are, in fact, more resistant to tarnishing than are silver mirrors protected by an evaporated quartz film. Again, the evaporated aluminium adheres to glass surfaces tenaciously, whereas silver is not so firmly bonded and is more easily scratched. Aluminium can be washed with soap and water at regular intervals without harm, whereas silver becomes scratched under similar treatment. Another big advantage is that whereas silver films have a marked colour selectivity which varies with age and density, aluminium films are fairly grey and stable at all densities.

In view of the advantages of evaporated aluminium for front-surfaced mirrors it is not surprising to find that some considerable application of the process has been made in the production of mirrors for astronomical telescopes, which it is a great advantage to be able to treat as permanent fixtures. Front-surfaced mirrors of silver glass have been used for many years in optical apparatus, but the inevitable tarnishing and softness of the film prevent them from giving lasting satisfaction. Aluminium, however, is much less susceptible than silver to tarnishing and after only three weeks' indoor exposure, even in the absence of sulphurous fumes, silver has usually tarnished sufficiently to bring its reflectivity below that of ordinary polished aluminium exposed alongside it. The big mirror at the Mount Wilson observatory, for instance, used to be resurfaced every six months; aluminium front-surfaced mirrors, on the other hand, have been in use under similar conditions for upwards of three years with hardly any loss in reflectivity. Among the first mirrors to be treated in this way were certain auxiliary reflectors of the 60-in. and 100-in. telescopes at the Mount Wilson observatory. This was in November, 1934, and the results were so satisfactory that in February, 1935, the 60-in. mirror itself and in March, 1935, the 100-in. mirror, too, were aluminized. In November of the same year, the British Astronomer Royal, Dr. H. Spencer Jones, announced the change-over of the mirrors at Greenwich Observatory from silvered to aluminized glass on account of the smaller degree of tarnishing obtained in the latter case. Other big operations of this type included the aluminizing of the mirror for the 200-in. reflecting telescope at Mount Palomar, near San Diego, just before the war, and the 72-in. mirror at the Dominion Astraphysical Observatory.

The usual method of procedure in the case of the larger mirrors is to first build up a vacuum chamber around the glass on which the aluminium is to be deposited. The cleaning of the glass surface to receive the metal film is of the greatest importance, and it has been found advisable to carry this out in two stages, the first chemical cleaning in which detergents such as certain sulphonated fatty alcohols, followed successively by potassium hydroxide, nitric acid, water and alcohol, are employed, and the second is a physical method, using a powerful electric discharge from a special electrode, while the bell jar is being evacuated. For the heating element, tungsten wire is used in the form of helical coils $\frac{5}{16}$ in. in diameter and pitched four turns to the inch, containing 10 turns of wire. The aluminium, in the form of U-shaped wire 1 mm. in diameter and about 1 cm. long, is clamped to each turn of the spiral, and fusion of the aluminium occurs in about 4 secs. in vacuum when a potential of 20 volts is applied to the tungsten wire.

The number of coils used for any one mirror depends upon the size of the mirror, and for those of large diameter the aluminium is distilled from a sufficient number of sources to permit of the equal distribution of the aluminium film over the whole surface of the mirror.

The thickness of the actual aluminium film aimed at is a uniform 10 μ over the whole of the reflecting surface of the mirror, and this results in the production of a surface which possesses high reflectivity, is largely immune from tarnishing, of good adhesion provided the undersurface has been adequately cleansed, and which offers considerable resistance to scratching and abrasion.

So far developments have been practically confined to the deposition of pure aluminium, but there is evidence to suggest that, for this type of application, certain alloys of aluminium may exhibit advantages over the pure metal. Thus, an alloy of magnesium and aluminium has been found suitable for deposition in vacuo and has a reflecting power some 4 per cent. higher than that of aluminium itself.

Mirrors have been produced commercially in this way under the trade mark "Pancro," and are claimed to be suitable for use in telescopes, ophthalmic instruments, stereoscopy, galvanometer mirrors, moving pictures, and applications in which semi-transparent mirrors are required. Another variation is the use of Chroluminium, a name suggested by R. C. Williams(18) for the combination of an under-deposit of chromium upon which a film of aluminium is later deposited.

Such deposits of aluminium are comparatively soft when first formed, but harden almost instantly when washed with either water or alcohol, and are then so hard as to be scarcely affected when rubbed with a blunt steel instrument. The reflectivity is as good if not better than that of pure aluminium, and the deposit has the advantage that the aluminium layer can be easily removed if and when desired by a solution of caustic potash, without removing or affecting the chromium. Another method has also been developed for removing the double film without injury to the glass.

German investigators have called attention to the reflecting properties of sprayed aluminium-magnesium alloys, and such coatings were officially recommended in that country as substitutes for polished nickel-plating. Edwards(39) has contributed a note on aluminium/magnesium mirrors produced by the simultaneous evaporation of these two metals in vacuum. He does not give accurate analyses to show the composition of the film owing to the practical difficulties associated with analysing films of which the weight is of the order of only 1 mg. Results that were obtained gave 10 per cent.-50 per cent. magnesium, remainder aluminium.

Edwards obtained the best result by evaporation of the two metals in separate pieces, using weights in the proportion 10 parts aluminium, 1 part magnesium. It is evident that it cannot be assumed that the film composition is of similar order. First, the evaporation process causes general deposition, so that uniform coating of the object by the two metals does not necessarily follow. Secondly, although the melting points of the two metals are very close (aluminium 658 degrees C., magnesium 651 degrees C.), their boiling points at atmospheric pressure are not (aluminium 1,800 degrees C., magnesium 1,120 degrees C.). Thus it can be anticipated that the vapour pressures under vacuum evaporation conditions are also widely different, with that of magnesium higher, and with the possibility of larger deposition of this metal.

It was claimed that mirrors produced by the evaporation of the best proportions of aluminium and magnesium should prove invaluable in optical intrument manufacture where face surfaces of high reflecting power are required. The combination deposit is exceedingly hard and non-tarnishable, apparently due to the corrosion and mechanical protection afforded by a very thin superficial layer of aluminium oxide. These mirrors have advantages over silver in both these respects.

Edwards tested mirrors from evaporated aluminium and from the aluminium-magnesium combination for reflectivity, with light incident at an angle of 10 degrees. The source was a tungsten filament, from which the white light was filtered and three ranges tested by means of the standard set of Wratten three-colour-taking filters as used for tricolour reproduction. The red filter (A5) transmits approximately the band 7,000 to 5,800, the green (B58) from 6,200 to 4,800, and the blue (C5-47) from 5,200 to 3,600 Å. Intensities were measured by a Weston Photronic meter.

The pure aluminium films reflected 89.5 per cent. in each of the three regions, and aluminium-magnesium 94 per cent. Chemically deposited silver, deposited on four surfaces, showed 95 per cent. reflectivity at the red and only 85 per cent. at the bue end of the spectrum. Therefore, the aluminium-magnesium combination not only shows high reflectivity, but uniformity over the visible region, with every possibility of good values in the ultra-violet.

One further field in which thin deposited coatings of aluminium are valuable as a reflection medium is that of gratings for use in interferometry. Aluminization improves the reflectivity of speculum metal gratings by about 50 per cent. for visible light, and to a much greater extent in the ultra-violet band. When the films are thin and the grooves are shallow, the spectrum formed by an aluminized grating, when viewed without a lens, appears to be crossed by light and dark bands. These do not appear when using ordinary gratings, and only show in spectra of the lower order when the films are thinner than 4,000 Å.

A further advantage is that the aluminium coating on the speculum grating can be removed by a suitable solvent (potassium-hydroxide) without attacking the speculum, and thus the usefulness of the bare speculum grating is not sacrificed. Partially transmitting films of aluminium have also been deposited on Corex glass and on fused quartz, and examined with a view to their being used for interferometry. Their reflections and transmissions were measured over the range 2380-4080 Å and all showed the same general behaviour—a slow decrease in reflectivity and a nearly corresponding increase in transmissivity with decreasing wave-length. The thicker films had the higher efficiency. In the earlier experiments with ruled aluminium gratings, the first attempts were made with pure aluminium films evaporated direct on to glass, but it was found that the diamond point, with which the rulings were made, stripped the aluminium off the glass in patches along the ruling. Pure chromium was next tried, but it proved so hard that when the diamond was loaded sufficiently to rule it the glass was disfigured.

Finally, aluminium was deposited on to chromium and success was attained, because the aluminium adhered more firmly to the chromium than to the glass. No stripping took place when the ruling was made, there was no glass to be affected by the ruling, and if, by chance, the first ruling proved a failure, the aluminium could be removed, a fresh film evaporated on to the chromium and a new ruling made. Moreover, the depth of the ruling could be accurately controlled by regulating the thickness of the aluminium film.

These rulings have proved very successful and are capable of producing spectra of greater intensity than those obtained with prism instruments. As a result, it has been possible to obtain photographs of iron arc spectra with thousands of lines perfectly defined, using an exposure of only five seconds. As the iron arc spectrum is the most generally used secondary standard for interpolating wave-lengths, the value of the ruled aluminium grating can be appreciated.

Sputtering

Of considerable interest but of less practical value at the moment is the sputtering process for depositing films of aluminium. This method possesses considerable potentialities because it supplies the means of depositing extremely thin layers of aluminium of controlled thickness which are of interest scientifically and which do possess certain practical possibilities.

Cathode sputtering is effected in a rarefied atmosphere between two electrodes. One of these, the cathode, is the material to be deposited as a sputtered film. The other, the anode, is of suitable material. The cathode is maintained at a high negative potential with respect to the anode, and an intense electrostatic field is created between them. In this field the relatively heavy positive ions of residual gas, directed towards the cathode, impact upon it and dislodge or tear out particles of material from it. These particles are probably of molecular dimensions, and carry a negative charge, and therefore travel towards the anode along the electrostatic field. If one or more objects be placed between the

cathode and anode in the electrostatic field, that is in the path of the negatively charged cathode particles, they will mechanically intercept these particles and become coated with the cathode material. By arranging for uniformity of field, and placement of the objects in that portion of the field that is uniform (that is, constant number of lines of electrostatic force per sq. in. of cross-section), a uniformly cathode-sputtered layer can be built up. The thickness of this layer is determined by the total time period of the operation.

The primary fields of application of sputtered films are optics and electricity, and the industries concerned are usually of the more highly specialized kinds manufacturing instruments and apparatus. Sputtering is also employed for the deposition of films of chemical compounds (e.g., quartz), or of alloys or metal mixtures (e.g., aluminium/magnesium).

Established commercial applications for the sputtering process include the following:—

Gold or platinum metals sputtered as thin films transparent to light on to selenium coatings to provide a low resistance electrical contact for photo-electric cells; gold sputtered as thin films on to copper-oxide-type rectifier discs for electrical contact; gold or silver sputtered as relatively thick films on to ceramic base, especially low-loss type, for fixed and variable condensers; gold sputtered as thick films on to precision-ground quartz crystals for oscillators; and palladium as a sputtered coating on the wax masters of gramophone records prior to the electrodeposition of copper. In connection with this last application, gold or silver has been employed for this purpose, but palladium is less expensive, quicker in sputtering, harder and less tarnishable and, above all, it does not possess the shortcoming of tending to diffuse into the electroplated copper matrix. Industrial applications of sputtered aluminium films have yet to be investigated.

The technique of cathode sputtering has been understood for about two decades. As long ago as 1933 the Naval Research Laboratory (U.S.A.) described a technique(40) utilizing a simple, yet highly adaptable, type of apparatus. The sputtering chamber consisted of a glass bell jar approximately 40 cm. high by 18 cm. diameter, having its flanged bottom periphery ground to fit on a heavy glass baseplate, vacuum wax being used to make a tight joint. Connection to the vacuum pump was made via a glass tube passing through the centre of the glass baseplate and sealed into it by means of vacuum wax. This tube also carried the lead to the anode, the latter consisting of an aluminium plate supported on a glass disc, which was itself loosely supported on a glass cylinder. The cathode assembly entered through the top of the bell jar and consisted of an aluminium disc attached by an aluminium rod to a metal plug fixed in the bell jar and sealed with vacuum wax. The work to be sputtered was supported on a table, which was suitably constructed either of glass or aluminium wire. Shielding glass sleeves and discs were fitted around the anode and cathode assemblies to confine the discharge as nearly as practicable to the space between the anode and cathode. They prevented the discharge plating upon metal connections to the electrodes and upon the seals. This had to be avoided as otherwise the liberation of gas (which upsets fixed conditions and makes control impracticable) would occur and, moreover, seals might be melted. Rectified and unrectified currents were employed at potentials of 1,000 to 3,000 volts and currents of 25 to 50 M.A. It was found that powers above 100 watts caused excessive heating, which resulted in poor films and difficulty in maintaining the seals. When exceptionally pure sputtered films were desired, the cathode and anode were made of the same material and the work was so placed that it could receive its deposit from either or both of these electrodes. An approximately correct working rule states that best films are obtained if the pressure be maintained such that the edge of the Crooke's cathode dark space is, roughly, tangent to the object to be sputtered, but apparently this is not critical. In practice, the work always receives some deposit on the faces remote from the cathode, although only slight. This phenomenon is common, especially with slow sputtering. The deposit on the remote face is usually found to be more uniform, although, of course, thinner, than that on the near side.

Every care has to be taken to have everything chemically clean, particularly the cathode. This can be cleaned chemically or by means of emery or glasspaper, but must be free from grease, including fingermarks. When a cathode is used for the first time the deposit may be slow in forming, but after an hour or more this rate increases. The surface of the cathode appears to become in a more readily sputterable condition, which is retained even if the cathode be removed, exposed to air and refitted, provided its surface is not handled or contaminated in any way.

Another form of cathode sputtering chamber was developed in researches by G. L. d'Ombrain and C. L. Fortescue at City and Guilds' College.(41) The anode consisted of an aluminium plate of appropriate size and of sufficient area to dissipate the heat developed in it. The cathode plate parallel to the anode was of the material to be sputtered, and was of about the same surface area as the object to be coated. This object was supported from a vertical pillar in a plane approximately parallel to that of the cathode, on the side of the cathode remote from the anode. Argon, recommended for sputtering any easily oxidizable metals, was used for the atmosphere and at a pressure that gave a cathode dark space of from 1 to 2 cms. at a supply voltage adjusted to give a current density from 2 to 3 milliamperes/sq. cm. of cathode surface. The position recommended for the surface to be covered was about 0.5 cm. beyond the end of the dark space. About 1,000 volts was required between the electrodes, with a series resistance having a similar voltage drop between its ends in order to control the discharge.

Uniformity of the cathode sputtered layer was checked by depositing on glass and examining in transmitted light. It was found that the apparatus gave very uniform films when operated as recommended, but with the object positioned between the electrodes the film was very uneven.

The form of chamber and vacuum system can be of any type that gives the results required. These workers used a glass bell jar with ground flange on a firm brass base plate, the junctions being held vacuum-tight by means of Apiezon compounds.

Films were deposited upon metals and non-metals (plastics, glass and ceramics). They were strongly adherent, markedly more so than sprayed or electro-plated metal coatings, or metal coatings applied from colloidal solutions and baked. The rate of formation of sputtered films was of the order of 1½ cm. by 10^{-6} in 10 mins. for copper and one-tenth of this for gold, calculated from resistance determinations.

The exhausting arrangements recommended for this equipment utilize a small Hyvac pump driven by a 1/6 h.p. motor. The capacity of such a pump is 7 litres per min., and it can maintain a vacuum of 0.0004 mm. of mercury. The pump is connected to the equipment through an oil trap and a phosphorus pentoxide drying chamber for removing moisture, and to the argon supply line.

The latest sputtering outfits are essentially modifications of this type of apparatus to suit the particular needs of the articles to be coated, the metal to be sputtered and the purpose for which the film is to be used. Sometimes the orthodox bell-jar type of sputtering chamber is replaced by a cylinder with a removable top plate; in other cases the work may be loaded through a hinged door. Heavy cast iron glazed internally with a vitreous enamel coating and provided with a 1-in. thick plate-glass observation window are popular materials of construction for commercial equipment. Anodes are generally of aluminium, whilst the cathode may be either of the metal to be sputtered or of brass polished and electro-plated with the metal to be sputtered. It is essential that this coating be uniform, continuous and free from defects. The work platform is frequently of glass, positioned on the side of the cathode remote from the anode. Occasionally, it is positioned on the side of the anode remote from the cathode.

Aluminium is one of the metals which sputter extremely slowly, but the rate can be increased enormously by the introduction of an inert gas, and a residual atmosphere of argon is employed in modern commercial apparatus. Air leakage has to be avoided or films of oxidizable metals will be tarnished.

The relative sputtering rates of various metals and the influence of inert gas are well demonstrated by the results of some experiments carried out by Hulburt(40). Employing a residual gas atmosphere of air corresponding to a dark space of 5 cm., total current 50 milliamperes, and cathode diameter 5 cm., he found that opaque coatings of antimony, bismuth, cadmium, gold, lead, platinum, silver, tin and zinc were formed in about 1 hr.; opaque coatings of cobalt, copper, iridium, iron, nickel, selenium and tellurium were formed in about 2 hrs.; molybdenum, tantalum and tungsten required several hours to give an opaque coating; whilst aluminium, beryllium, carbon, chromium, magnesium and silicon sputtered extremely slowly. In the cases of aluminium, chromium and silicon, the rate of sputtering was increased enormously by the introduction of mercury vapour, helium or argon into the sputtering chamber. In this manner, optically perfect opaque films could be obtained in less than 15 hrs. The influence of these monatomic gases in this direction was not explained. With beryllium, and residual gas of hydrogen and mercury vapour, 60 hrs. failed to produce entirely opaque films, although they were optically good. With the residual gas consisting of air, graphite (both electrodes) gave nearly opaque films in 30 hrs., and mercury vapour did not improve the rate. The films were uniform, optically smooth, blackish in reflected light, and the thinner coatings yellowish in transmitted light. They were moderately soft.

Hulburt speculates that the discharge from the cathode is of atomic dimensions and that much of it proceeds in straight lines from the cathode. This is shown by the fairly well-defined shadows that can be cast upon an object by interposing on obstacle such as a diaphragm. The "shadowed area" receives no deposit, or very little. In practice areas not required to be sputtered can be masked by the formation of shadows or by means of closely fitting screens. On the other hand, the walls of the vessel and all the remote surfaces of fixtures, etc., within the chamber receive some deposit, much being due, presumably, to diffusion.

Complex interference effects also occur. Thus, when the cathode emission passing through a hole 2 mm. in diameter in a glass plate 2 mm. thick was allowed to fall on a glass plate 3 mm. distant from the tube, a film was formed roughly corresponding to the geometrical shadow of the hole, but having an uncoated central spot. With glass tubes interposed between cathode and object, the cathode particles entered the tube only to a depth about equal to the diameter. With a concave cathode, particles leaving it normally could be focused, but the spot produced had a central hole in it. Similarly, a cathode curved in one axis only (that is, a portion of the curved surface of a cylinder) and which should yield a straight strip of film, exhibited the same peculiarity, that is, a central line of no deposit.

Use is made of various cathode arrangements according to the configuration of the surface to be coated. With a flat cathode plate fixed vertically, the objects to be coated can be positioned on each side of it and the two loads of work receive their deposit simultaneously. Using a vertically positioned cathode in the shape of a wide U, an object within it can be coated on opposite sides in the one operation. Again, using a wire-form cathode, the internal surfaces of a tube can be coated. It is evident, however, that individual difficulties have to be solved. For example, in coating tubes, as the length becomes greater than the diameter, the uniformity of the coating becomes less, due to the discharge from the anode not reaching all points on the cathode wire. During the sputtering operation, the work to be coated becomes hot owing to the absorption of thermal radiation from the cathode, and if this is likely to cause harm, as, for example when wax gramophone masters or plastics are being sputtered, water cooling must be employed or the process must be conducted intermittently, allowing suitable intervals for cooling.

In view of the small amount of total current employed, it is not surprising that deposition is slow. In fact, the sputtering process is essentially a means of producing very thin films of aluminium and other materials which are at the same time fairly uniform and of controllable thickness. If such films of aluminium have no important applications at the moment, the sputtering process is nevertheless of interest because it is the final link in the chain of processes by means of which aluminium and alloy coatings may be produced in any desired thickness—flame spraying for coatings for which there is theoretically no upper limit to the thickness which may be produced; calorizing, Aliting and similar processes for producing alloy coats; organic painting compositions forming thinner coats; evaporated aluminium films; and finally, sputtered aluminium films, the thinnest yet produced.

Although every endeavour has been made in the foregoing review to present as complete a survey as possible of the various techniques dealt with, it has, by the nature of things, proved virtually impossible within this short compass to cover in detail every possible variant of each, or to detail every possible application. In this last respect, those spheres of utilization developed during the war years have only recently come to light and complete data are, in general, not accessible.

Equally, little or no reference has been made to those applications of an earlier date, which, having been demonstrated at the time, were put on one side, possibly because no use could be found for them in practice, or because economic considerations favoured the adoption of alternative methods. One particular instance may be quoted here; namely, the production of a flame-sprayed coating of aluminium on cold rolled bright annealed mild steel sheet, the finished product being afterwards cold rolled to give a dense bright aluminium finish. The appearance of such sheet was very attractive, and its corrosion-resisting properties were surprisingly high, but it was not found practicable in any way to use it in place of tinplate, this idea having originally prompted the experiments. During the war years, gas bottles for the filling of barrage balloons were flame-sprayed with aluminium by means of the wire pistol, and it has been stated that, in terms of area of metal covered, this represents by far the largest job of the type undertaken with aluminium as the applied metal.

As in the case of every other technological advance, practical application of the processes described here, and in particular that of the flame-spraying systems, is governed, not only by purely technical considerations, but by others of an economic, and sometimes even of a political nature. In this last instance we will recall the attempts made in Germany to replace nickel-plating by sprayed films of aluminium-magnesium alloy. Economic factors, however, may prove to be of the highest importance in furthering the sphere of usefulness of the spraying technique.

Where, in pre-war days, service requirements demanded special corrosion-resisting properties for example, or qualities for resisting scaling at high temperature, it was possible, if the expense would warrant it, readily to obtain and use special nonferrous alloys with high resistance to chemical attack, or alloy steels designed specifically for high temperature use.

These materials, even if expensive, were readily available and no difficulties were entailed in delivery either for original manufacture or for replacement. The position now, however, is very different indeed; delivery dates for copper-base alloys are uncertain, and in fact they can be obtained only on the basis of high priority. Alloy steels are in short supply and, in addition, cannot be obtained at all readily in the multitude of forms which were on the market prior to 1940.

Mild steel itself is far from plentiful, but it is probably right to say that it is a little easier to get at than those other materials we have mentioned. Construction, therefore, in mild steel treated initially by spraying with molten aluminium, with or without any subsequent processing, may often prove, even for the most stringent requirements, an avenue of escape from otherwise almost insuperable difficulties.

Much controversy still surrounds certain of the theoretical aspects of sprayed metal coatings, and finality has by no means been reached. In a supplement to this review, to be presented at a later date, will be summarized more recent research, particularly that set out in a paper by Ballard and presented to the Physical Society. Modern testing methods for sprayed coatings will be considered at the same time.

Comment so far has been devoted principally to flame-spraying techniques: the position with respect to cathodic sputtering and evaporation is somewhat different. These arts were practised in specialized fields for many years before the outbreak of war; they were regarded, however, as methods more proper for application to precision instruments, than in the field of general manufacture, and, excepting for a very small number of plants, could be applied only to relatively small surfaces. The outbreak of war prompted the immediate use of the sputtering and evaporation techniques to a large mass of scientific and technical equipment for warfare purposes, and for the first time probably, these processes may be said to have found a place in mass-production organization.

With widening field of use, so, also, the flexibility of the equipments was increased, and more important, apparatus was constructed capable of dealing with much larger surfaces than heretofore. Improvements were effected both as regards speed of evacuation and efficacy, more particularly in connection with the production of the very high vacuum required in massive chambers. In some cases, it is believed, developments resulted in the creation of equipment capable of what might be described as continuous production, not only, incidentally, for the evaporation of aluminium, but also of other metals, and of non-metals. This increased use and the development of advanced techniques enabled economies to be effected in the processes at large, and this again reflected in a further widening of their field of usefulness. Some details of the latest types of equipment will be given in the forthcoming supplement, together with further examples of current applications of the processes as operated for commercial production purposes.

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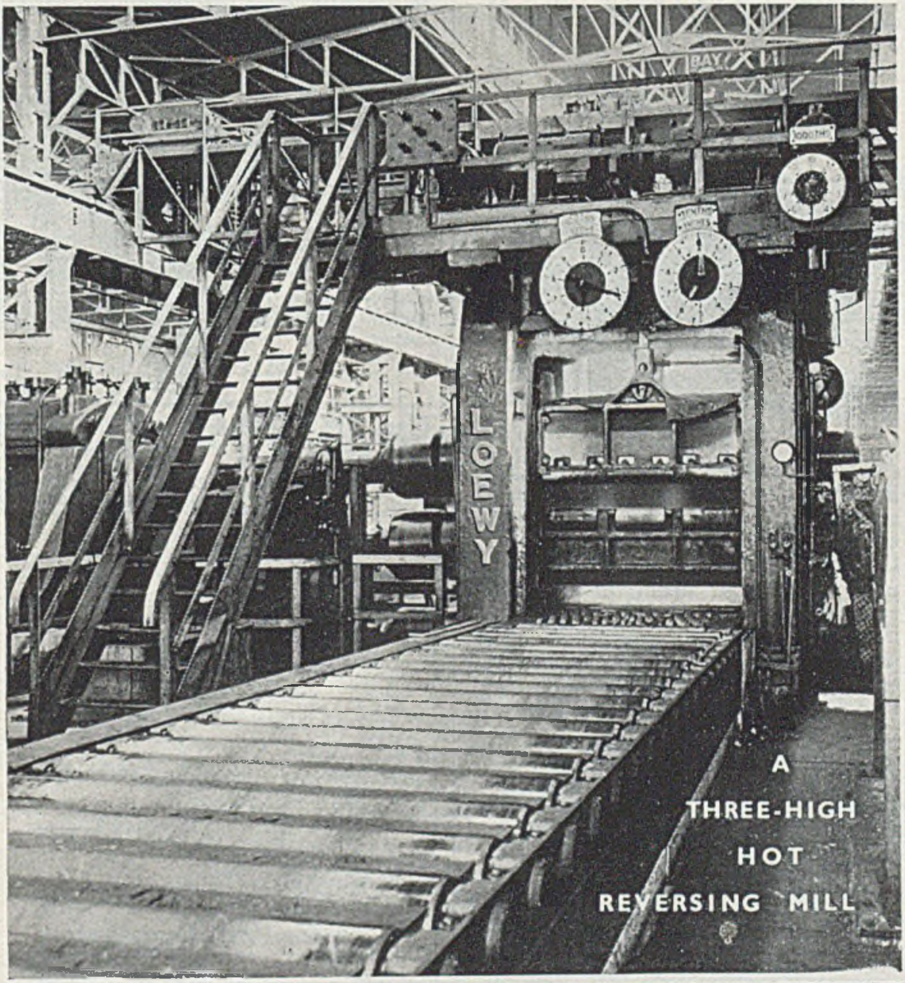

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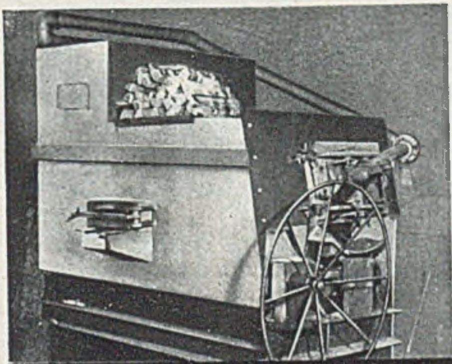

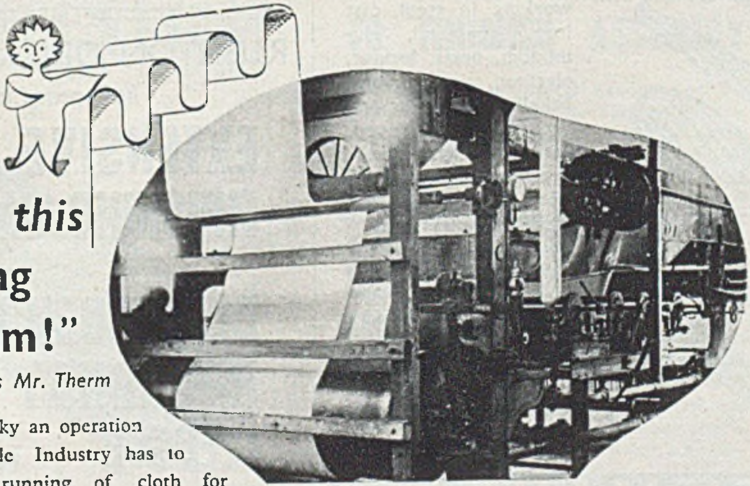

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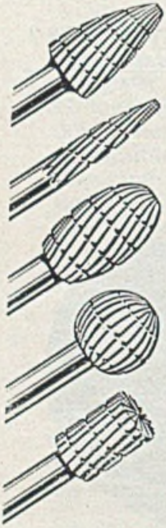

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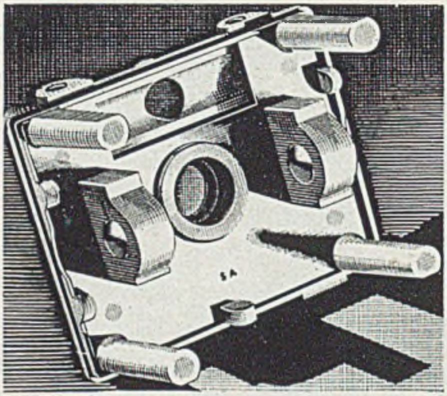

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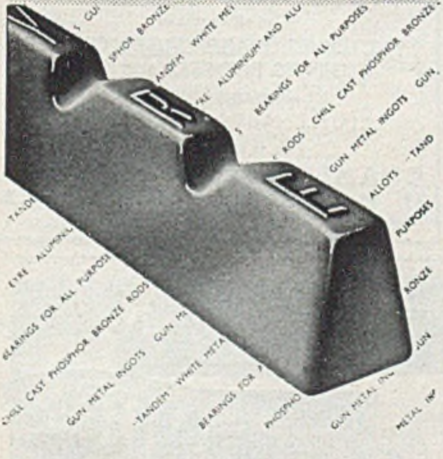

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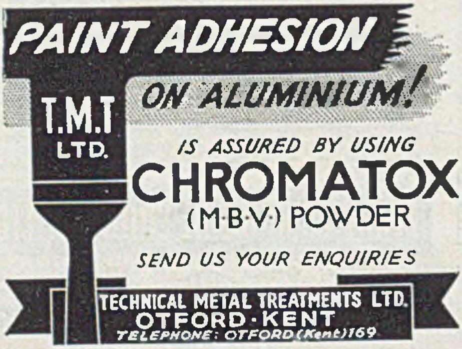

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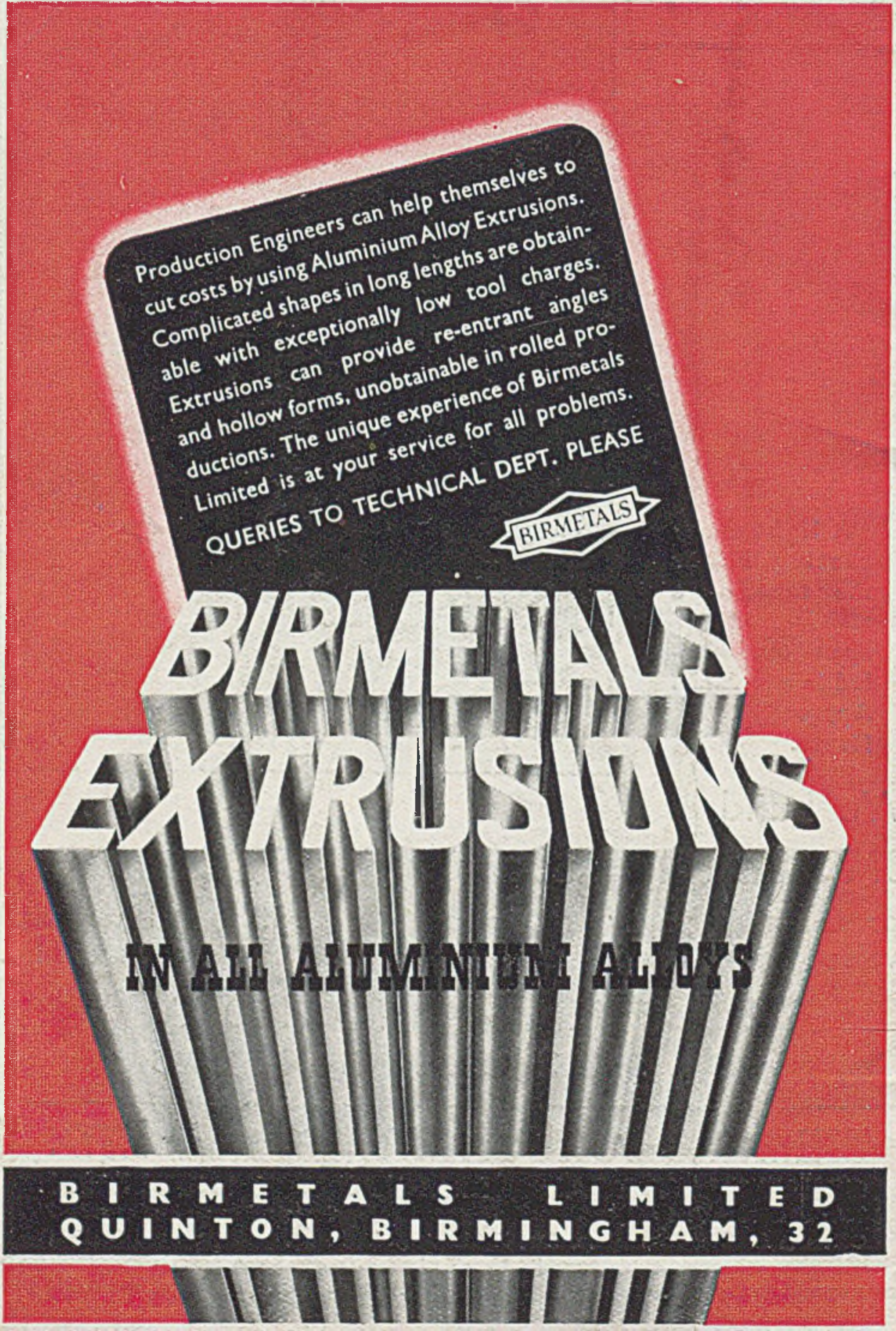

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