RESEARCH ON METAL COATINGS

BRITISH IRON AND STEEL RESEARCH ASSOCIATION, SOUTH WALES LABORATORIES

IN 1932 a research group was set up in the Metallurgy Department of University College, Swansea, to study various aspects of sheet steel production. It operated under the auspices of the South Wales Siemens Steel Research Committee and the Welsh Sheet and Plate Manufacturers' Association and was led by Mr. D. Luther Phillips.

When the British Iron and Steel Research Association was formed in 1945, it absorbed this research group, the functions of which were carried on by the Association's Mechanical Working Division. The policy of the British Iron and Steel Research Association had been to site its laboratories in areas traditionally devoted to specialized aspects of ferrous technology, so that it was natural that its Coatings Laboratory should be set up in South Wales, which is the main centre of the flat-rolling, tinning and galvanizing industry. In 1946 laboratory accommodation was obtained at Sketty Hall, a large building in Singleton Park, close to University College and about four miles from Swansea. A Pilot Plant Laboratory has recently been added in a building adjacent to Sketty Hall to house small-scale continuous strip-coating and processing plants. Mr. Phillips retired in 1954 and research is now directed by Mr. S. S. Carlisle.

The research programme of the Coatings Laboratory works in two main fields. It endeavours first to develop new and improved coating for steel, so as to extend the applications of steel products, and secondly, it seeks to improve such existing processes as strip tinning, galvanizing, aluminizing and lacquering in order to reduce costs and increase productivity. The ultimate aim is to prepare the way for automation in the steel strip finishing industry.

The many projects currently being studied at Sketty Hall were displayed at open days, held on June 19 and 21, when representatives from the iron and steel and allied industries toured the laboratories. A short description of the main displays is given here.

Research has been undertaken into coating steel with plastics, so as to combine the strength of steel with the attractive and durable finish of the plastic. An improved process has been devised, whereby a film of polyvinyl chloride is bonded to steel, the laminate so formed ('Plasteel') being amenable to bending, deep drawing and roller forming without harm to the plastic film. A pilot line is producing continuous strip laminate up to 3 in. wide in a variety of plain, coloured or embossed polyvinyl chloride on steel. The process is being patented, and wide interest has been shown by potential users of the new material.

It is an advantage in can-making to be able to apply a thicker coating to the inside than to the outside of a can, since the interior may be subjected to corrosive action from acid fruits. A problem in subsequent working of the tinplate is to identify which side has the thicker or thinner coat. A method developed by the Association, and being patented, is to dull one side by applying a thin electrodeposit of iron (about 0·25 micro-inch thick) after tinning but before flow brightening.

Lacquered steel has been found suitable for use in can-making where protection against corrosive fluids, other than acid fruits, is required. Although this is a useful technique when tin is scarce, lacquered steel has the disadvantages that it presents no tinned surface for soldering, it cannot be used for all food-packs, and the lacquer is easily damaged. However, it is now possible to tin the edges for soldering, and to lacquer one side of a sheet and tin the other. The British Iron and Steel Research Association has built an experimental line for lacquering steel strip, on which coatings of epoxy-resins up to 0·5 mil thick can be applied and cured by heating. A coating as little as 0·15 mil thick has been found to protect steel.

Thick coatings are more economically applied by hot-dip coating than by electro-deposition. The hot-dip technique also opens up the possibility of its application to high-speed strip working. Many studies of hot-metal coating practice have been made at the Laboratories, and a new method of hot-strip tinning has been developed.

It was decided to investigate applying tin to steel strip by roller coating, when the weight of tin applied could be controlled by varying the speed of the roller relative to the strip. An experimental continuous strip-coating line using this principle has been built, and good-quality coatings have been applied at speeds of up to 50 ft./min. Lead–tin alloy has also been successfully applied.

The process opens the possibility of cutting costs in tinplate production by placing the hot-tinning mechanism at the end of a continuous strip-annealing line. It may also be possible to use the process for applying such other coatings as zinc and aluminium, and it has therefore been decided to develop it further. To this end a 12-in.-wide strip-processing plant has been built, similar in principle to the small experimental line. It is capable of speeds up to 1,250 ft./min. and is fully instrumented.

A method for continuously coating steel strip with aluminium has been developed in the Laboratories, so making available a material generally resistant to corrosion and useful at temperatures too high for tin or zinc coating. The 'Sendzimir' process, which has been successfully operated in America, is mainly suitable for large-scale production of coated sheet, for which there is as yet insufficient demand in Britain. In the Association's method, continuous strip up to 3 in. wide has been successfully coated, and the difficulties normally encountered in the past from oxidation of the ingoing strip have been overcome. The surface of the strip is carefully prepared and coated with glycerol; it enters the aluminium bath through a protective box, preventing the oxide film which forms on the surface of the bath from reaching the strip. By burning the glycerol the strip is kept free from oxidation before entering the bath, also, and the addition of small amounts of silicon to the bath inhibits the formation of brittle iron–aluminium alloy on the steel surface. One firm has applied the Association's process to the manufacture of aluminized steel wire; aluminized steel, with its negligible tendency to scaling and its good anticorrosion properties, will probably become a material of greater application in the future.

Copper wire is being coated by drawing it through a die, the entry side of which is flooded with molten tin. Provided that the wire surface is clean and freshly drawn, no flux is needed; uniform coatings about 40 micro-inches thick with reductions in wire diameter of 5–10 per cent have been obtained and drawing speeds are about 100 ft./min. The process is being patented.

A necessary preparation for steel sheet or strip before working or coating is 'pickling' by immersion in sulphuric acid, which has the effect of removing the surface oxide film. The severe shortage of sulphuric acid a few years ago, coupled with the problem of disposing of spent pickle liquor, led the Association to investigate the possibility of recovering acid from waste liquor. This is achieved by precipitating ferrous sulphate monohydrate from the liquor by evaporation, roasting the sulphate to sulphur dioxide, and oxidizing this to sulphuric acid in a contact plant using vanadium as catalyst.

Several difficulties were encountered. Research had first to be undertaken to determine the practical limits to the removal of ferrous sulphate from acid solutions. The solubility curves so produced were an original contribution to the literature since the published data had been found inconsistent. Then it became evident that the relatively small output of sulphate from pickling lines made it uneconomic to use the contact process for its conversion to sulphuric acid. The Laboratories therefore developed an alternative process of autoxidation.

In this method, sulphur dioxide is released from the ferrous sulphate by roasting with coal and is then dissolved in weak acid circulated through an absorber tower from an acid storage tank. The acid with sulphur dioxide in solution is then aerated by having small bubbles of air blown through it. This takes place in a diffusion tank and the oxidation of sulphur dioxide to sulphuric acid occurs at the bubble–liquor interfaces, catalysed by manganese sulphate which has been added. The liquor is thus enriched in acid, which is drawn off to storage tanks for supply to the pickle line. The iron oxide residue from roasting the sulphate can be used as blast-furnace feed.

Trials on a pilot plant indicated that the process becomes economic when 10 tons or more of acid are being handled. With acid strengths above about 23 per cent the catalyst needs to be kept active by injecting a little ozone with the diffuser air; acid strengths of up to 40 per cent can then be provided. A plant has been designed which could treat all the ferrous sulphate processed at the Trostre tinplate works of the Steel Company of Wales, Ltd., and this design is now being considered. A plant has been proposed, also, for a closed-cycle pickling system with acid recovery using this autoxidation process.

VACUUM DEPOSITION OF METALS

A MEETING organized by the Department of Physics of the University of Cambridge and the British Scientific Instrument Research Association was held in the Cavendish Laboratory on May 17 and 18 by invitation of Prof. N. F. Mott. Attendance was by invitation, and scientists were present from the Imperial College of Science and Technology, London, the University of Bristol, Tube Investments, Ltd., the University of Cambridge and the British Scientific Instrument Research Association.

In the first paper of the meeting, Dr. D. W. Pashley (Tube Investments) described recent experiments on the occurrence of epitaxy in deposits of copper, lead and thallium on crystalline substrates. Dr. Pashley's experiments showed that, while the nature of the initial deposit varied, according to the metal, from a uniform monolayer to a scattering of widely separated atom aggregates, the crystalline form was always that of the metal itself, undistorted by the supporting crystal, which, nevertheless, imposed a regularity of orientation. Later in the discussion, Dr. M. Blackman (Imperial College) examined the relation, for a wide range of materials, between epitaxy and the degree of misfit between the lattice structure of the film and its substrate, and showed that in contradiction to the theory of Frank and van der Merwe there appeared to be no critical degree of misfit above which orientation of the film could not occur. Following Dr. Pashley's paper, which also included a mention of a new technique for revealing dislocations by means of a moiré pattern, Dr. A. J. Forty (Tube Investments) gave evidence of the role of crystal boundaries as sites for nuclei from which the growth of films could start.

Dr. K. M. Greenland (British Scientific Instrument Research Association) described in general terms the work which is being done from the point of view of new and improved detecting elements, and explained that, in the first instance, the British Scientific Instrument Research Association is concerned with the binding forces between films and their substrates with special reference to gold and silver on glass and silica.

Dr. J. W. Mitchell (University of Bristol) described an investigation of the texture of copper films deposited by evaporation at very low pressures on glass held at the temperature of liquid nitrogen. The surface area of the film was measured by an adsorption method. In these experiments it was found that the porous structure of the film was independent of the thickness. As the temperature of the film was raised the structure became more compact, but the admittance of oxygen had a strong stabilizing effect.

Mr. D. G. Anderson and Mr. J. E. Knowles (both from the British Scientific Instrument Research Association) continued the account of work at the laboratories of the Association. Mr. Knowles gave a description of a method which was being investigated for the comparison of the energy released when gold films condense on substrates of differing kinds. This method might lead to a direct measurement of the energy of reaction between the gold film and the substrate. Mr. Anderson showed how chemical bonds might be formed between metal films and a silica or glass substrate and described some experiments designed to demonstrate the importance of oxygen in film bonding. Taking account of the ionic nature of the glass structure, the possibility of the existence of reactive ionic centres in a fractured glass surface was also considered. Prof. F. C. Frank (University of Bristol) summarized the Frank–van der Merwe theory of critical misfit in epitaxy and also discussed the stability of nuclei of cubic crystal forms.