

Growth Spirals in Electrodeposited Copper

It was first pointed out by Frank¹ that the termination of screw dislocations in a crystal face would produce steps, by the propagation of which crystal growth could take place. Such steps would obviate the need for two-dimensional nucleation and thus allow crystal growth to proceed at considerably lower supersaturations than those required to form two-dimensional nuclei. Frank predicted that the presence of an isolated dislocation on a close-packed crystal face would allow this to grow *ad infinitum* in the form of an ascending spiral. This prediction was afterwards substantiated experimentally by observations of crystals grown both from the vapour and from solution. The spirals of silicon carbide grown from the vapour by Verma², and those of cadmium iodide grown from solution by Forty³ may be quoted as examples.

No observations of spirals grown by electro-deposition seem, however, to have been reported. A recent examination of the surface of electrodeposited copper has led to the identification of spirals, and these must be assumed to have originated in a manner similar to that discussed in the previous paragraph. The copper was deposited at an average current density of 10 m.amp./cm.² on a polycrystalline sheet of copper which had been cold-rolled and annealed so that a high proportion of the grains were oriented with their (100) planes parallel to the surface of the sheet and their [001] directions parallel to the

rolling direction. The cathodes were electropolished prior to plating.

Two types of spiral have been observed: (a) those deposited from aqueous $N/2$ copper sulphate + $N/2$ sulphuric acid solution, in which the rate of growth appears to have varied with crystallographic direction (see Fig. 1); (b) those deposited from neutral aqueous $N/2$ copper sulphate, in which growth was insensitive to crystallographic direction (see Fig. 2).

The conditions under which these spirals grow are now the subject of investigation. Plans are in hand to produce a continuous photographic record of the development of these spirals and of other features of crystal growth encountered in electrodeposition. This will be done by placing a suitably designed electrolytic cell on the stage of a microscope fitted with a reflecting objective. Such an objective may easily be corrected for the refractive index of the plating solution.

Since writing this communication, my attention has been directed to a photograph of a spiral on the surface of electrodeposited silver-indium alloy, reproduced in a paper by Raub⁴. This spiral is visible to the naked eye, but no details are given of the way in which it has been produced.

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¹ Frank, F. C., *Disc. Farad. Soc.*, No. 5, 48 (1948).

² Verma, A. R., "Crystal Growth and Dislocations" (Butterworths Sci. Pub., 1953).

³ Forty, A. J., *Phil. Mag.*, **42**, 670 (1952).

⁴ Raub, E., *Metalloberfläche*, **A.**, **7**, 17 (1953).



Fig. 1. Growth spirals in copper electrodeposited from an acid copper sulphate bath. ($\times 570$)



Fig. 2. A growth spiral in copper electrodeposited from a neutral copper sulphate bath. ($\times 940$)

Partition Chromatography of Synthetic Detergents

IN the course of a study on the adsorption at liquid/solid and liquid/air interfaces, difficulties were encountered in identifying small quantities of adsorbed detergents and in separating mixtures of such substances containing alkyl groups differing in length.

A convenient method of separation has now been developed by means of paper partition chromatography. The compounds mainly dealt with were the normal sodium alkyl sulphates, the number of carbon atoms in the hydrocarbon chain ranging from twelve to eighteen. However, with minor modifications the method was successfully applied to alkyl aryl sulphonates, secondary alkyl sulphates, quaternary ammonium and pyridinium salts.

The methods of ascending and circular¹ chromatography were employed. Whatman No. 1 filter paper was impregnated with a long-chain fatty alcohol, usually cetyl alcohol, which acted as stationary phase; this was applied from a 1 per cent ethanolic solution. The mobile phase consisted of an aqueous ethyl alcohol solution saturated with cetyl alcohol. The concentration of this solution varied according to the alkyl sulphates to be separated. The chromatogram of the detergent mixture was developed for 10–24 hr., and the paper dried. It was then immersed in 0.5 per cent cupric acetate and sprayed with a 0.05 per cent solution of rhodamine 6GB. After drying, the detergent spots appeared crimson on a pink background in daylight, and dark purple on a yellow background under ultra-violet light. R_F values were determined for various concentrations of ethyl alcohol and are shown in Table 1.