Diffusion Treatment of Semiconductor Materials

A LONG-ZONE PLATINUM-WOUND FURNACE OF EXCEPTIONAL TEMPERATURE UNIFORMITY

The increasing versatility of the silicon-based diffused semiconductors has posed problems in the fulfilling of stringent specifications. The characteristics of such devices are critically dependent on the depth of diffusion of the doping element and, as diffusion rates vary with temperature, it is essential that the hot zones of the furnaces employed in the processing of these materials should have high temperature uniformity, be accurately adjustable to any specified temperature within a wide working range, and be capable of very precise zone temperature control. To meet these requirements a new design of rhodium-platinum wound furnace has been developed by Johnson Matthey.

The operating range of the furnace is from 900° to 1350°C and the temperature of the 16 inch by 2½ inch diameter hot zone where diffusion is carried out may be controlled to within ±1°C. This uniformity in the hot zone is produced by the use of three precision wound elements of 10 per cent rhodium-platinum embedded in a high grade alumina refractory. For temperature control, five platinum sheathed mineral insulated platinum: 13 per cent rhodium-platinum thermocouples are inserted through the elements into the bore of the furnace.

The main controller is a potentiometric three-term instrument with a suppressed zero to give a very open scale. This instrument controls the temperature of the furnace and is connected to a thermocouple placed at the mid point of the centre element. Two centre zero proportional galvanometer controllers, each regulating one of the end elements, automatically correct any temperature deviations and operate from the difference in e.m.f. between the respective thermocouple in each end element and its reference thermocouple in the centre zone. Variations in cold junction temperature are automatically corrected by means of a nickel compensating coil incorporated within the main controller.

The power to the end windings is first preset to produce uniform temperature in the 16 inch centre zone. The current to each winding is regulated by silicon controlled rectifiers that are protected against voltage surges. Once the controls have been adjusted to give a selected uniform temperature over the length of the diffusion zone, the temperature of the zone may be altered simply by a single adjustment of the main controller.

To ensure that the temperature of the furnace cannot rise above a specified safe level, in the event of a fault developing in the main controller an independent controller that monitors the temperature of each element in turn cuts off the power supply to the furnace as soon as the temperature of any of the elements reaches a pre-set level.

In the design and development of the furnace, every effort has been directed towards the production of a reliable apparatus that will give continuous production runs and that requires little maintenance. Even at 1350°C, the elements operate well within their capacity and have long working life. Power consumption is low and the standard unit operates on 200 to 250 volt single phase supply, although units for other voltages can be produced.

One of the new Johnson Matthey platinum-wound semiconductor diffusion furnaces in use in the research laboratories of The Plessey Company (U.K.) Limited at Caswell. A charge of silicon slices is being positioned in the hot zone of the furnace prior to the diffusion heat treatment operation.

The furnace is 37 inches long and has a bore of 2½ inches. The axis of the bore of the furnace is 63 inches above floor level.

This furnace is ideal for semiconductor diffusion processes that are carried out at fixed temperatures. Further development is being directed towards achieving a zone uniformity of better than ±0.5°C, to increasing the maximum operating temperature, and to providing a temperature programming and recording device.

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The Isomerisation of Olefins

CATALYSIS BY PALLADIUM COMPOUNDS

During an experimental study of the Wacker process carried out by Dr M. B. Sparke and his associates at B.P. Chemical Co. Ltd, Sunbury-on-Thames (M. B. Sparke, L. Turner and A. J. M. Wenham, paper AB4-30, presented at the I.U.P.A.C. conference, London, July 1963), it was observed that if the reaction was arrested before completion the original olefin used had isomerised. The isomerisation process was further investigated and was found to proceed rapidly under mild conditions: thus in the case of methyl pentenes and n-hexenes, the thermodynamic equilibrium mixture of isomers was formed when the olefin was refluxed with catalytic quantities of palladous chloride for ½ to 2 hours. No side reactions were observed.

The induction period which was observed when palladous chloride was used disappeared when ethylene palladous chloride or bis-benzonitrile palladous chloride was used instead. The mechanism of the reaction was not definitely established, but the migration of the double bond in the olefin molecule was shown to proceed stepwise along the carbon chain. Palladium bromide complexes and π-alkenyl-palladium complexes were virtually inactive, but ethylene platinous chloride had a definite but low isomerisation activity.