

elevated temperature in the course of preparation. The polycrystals were not as ductile as the single crystals but withstood considerable deformation at low rates of deformation. At high rates of deformation they showed a greater tendency to brittle behaviour but in any case withstood rolling 30 per cent in one pass at a speed of 10 cm/sec. The fracture tended to be intercrystalline.

The impurities present are very much lower than those usually necessary to produce grain boundary weakness but it may be that the significant impurities are non-metallic or otherwise not easily determined.

In a similar investigation on rhodium, Calverley and Rhys (9) were able to deform zone refined single crystals by 90 per cent without intermediate anneal and yet the annealed polycrystalline wires could only be deformed to a very limited extent before inter-

crystalline fracture occurred. These authors were also forced to the conclusion that grain boundary segregation of unknown impurities is the cause of embrittlement.

The authors are indebted to Johnson Matthey & Co Limited for providing the iridium specimens and for carrying out the analyses.

References

- 1 B. L. Mordike, *Z. Metallkunde*, 1961, **52**, 587
- 2 R. L. Fleischer, *J. Mech. Phys. Solids*, 1958, **6**, 301
- 3 E. Schmid and W. Boas, *Kristallplastizität Springer Verlag*, 1935, Berlin, page 66
- 4 P. Haasen, *Phil. Mag*, 1958, **3**, 384
- 5 T. H. Blewitt, *Phys. Rev.*, 1953, **91**, 1115
- 6 M. Ahlers, *Z. Metallkunde*, 1962, **53**, 302
- 7 W. Köster, *Z. Metallkunde*, 1948, **39**, 1
- 8 W. Köster, *App. Sci. Res. Section A*, **4**, 329
- 9 A. Calverley and D. W. Rhys, *Nature*, 1959, **183**, 599

Ethylenediamine Complexes of Ruthenium

By F. M. Lever, A.R.C.S., Ph.D., and C. W. Bradford, B.Sc.

Research Laboratories, Johnson Matthey & Co Limited

So far as is known, no ethylenediamine complexes of ruthenium have been isolated. The reaction between ethylenediamine and chloro complexes of ruthenium such as ammonium chlororuthenite or ruthenium trichloride results in the formation of dark brown viscous solutions from which it seems impossible to separate any crystalline derivatives. A technique earlier discovered and used to prepare ammino derivatives of ruthenium II has now been successfully used to produce an ethylenediamine complex of ruthenium II, $[\text{en}_3\text{Ru}]\text{ZnCl}_4$. This compound should provide a suitable starting-point for the preparation of other ethylenediamine complexes, particularly of ruthenium III since in its reactions it appears to be analogous to the ammino complex $[(\text{NH}_3)_6\text{Ru}]\text{ZnCl}_4$.

In a Ph.D. Thesis (London University, 1955) Lever, and at the International Conference on Co-ordination Chemistry, London, 1959 (Special Publication of the Chemical Society, No. 13, 1959, 135-136) Lever and Powell described the preparation of hexam-

minoruthenium dichloride, $[(\text{NH}_3)_6\text{Ru}]\text{Cl}_2$ and the chlorozincate $[(\text{NH}_3)_6\text{Ru}]\text{ZnCl}_4$ by the reduction of ruthenium chloro complexes with zinc dust in ammoniacal ammonium chloride solution. It has now been shown that by cautiously adding zinc dust to a solution of ruthenium trichloride in 25 per cent ethylenediamine, boiling under reflux, filtering, cooling, and carefully acidifying the solution to pH 1-2 crystals of $[\text{en}_3\text{Ru}]\text{ZnCl}_4$ can be separated. (Found Ru 20.55, Zn 13.37, N 17.22, Cl 29.10, C 14.93, H 4.93 per cent; $[\text{en}_3\text{Ru}]\text{ZnCl}_4$ requires Ru 20.69, Zn 13.39, N 17.21, Cl 29.02, C 14.75, H 4.95 per cent).

It has, so far, not been possible to investigate fully the reactions of this compound but it has been found to behave similarly to the analogous ammino complex. Thus, when it is boiled with hydrochloric acid, hydrogen is evolved and a deep blue complex is formed. Mercuric chloride and chloroauric acid are both reduced indicating the powerful reducing properties of the compound. It is hoped that, in the future, these and other reactions will be studied and the products isolated and identified.