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Crystal Growing by Electrodeposition from Dense Gaseous Solutions

Single crystals and dendritic formations of silver have been grown on platinum electrodes by electrodeposition from a dense gaseous solution of silver nitrate in ammonia. The process is essentially a modification of the standard high temperature, high pressure, hydrothermal process for growing crystals. However, unlike the standard hydrothermal process, which uses concentration gradients or chemical rates of solution as its driving force, the new method is driven by electric force in the form of electrodeposition. The method also differs from standard electrodeposition techniques in that it permits single crystals to be grown from a hydrogen-bonded solvent such as water or ammonia. In normal, liquid-state electrolysis using such a solvent, hydrogen gas is evolved and trapped at the cathode surface as bubbles. Such bubbles often interfere with the continuous growth of crystalline metallic deposits. In electrodeposition from a dense gaseous solution, however, because of the absence of surface tension, evolved hydrogen diffuses away from the cathode, and no bubbles are formed.

In the experiments described, ammonia was selected as the solvent because of its convenient critical point (132.9°C; 112 atm.) and its chemical inertness toward metals. Silver nitrate was chosen as the salt because of its solubility in ammonia and the insolubility of the electrodeposited silver in ammonia.

The electrodeposition was accomplished in a closed cell containing electrodes of platinum wire. The cell, made from 30 mm (OD) borosilicate glass tubing, was about 8 cm long with an internal volume of 35 to 40 cc.

Reagent grade silver nitrate was placed in the cell and the cell was evacuated. Approximately 20 cc of high-purity liquid ammonia was then condensed into the cell, which was subsequently sealed and placed in a pressure vessel. During heating and electrodeposition, nitrogen gas pressure within the vessel was maintained at about 30 atm above the predicted internal cell pressure.

Runs were made using current densities of 1 to 2 mA/cm^2 for time periods of from 600 to 800 sec, at temperatures of from −70°C to +140°C. The silver deposits became increasingly more crystalline as the critical temperature was approached. At temperatures above the critical point, needle-like single-crystal wires and dendrites up to 7 mm in length were produced. The observed triangular and square cross-sections of the central stems and branches of the dendrites suggested screw dislocations in the (111) and (100) planes.

The ease with which fairly large single crystals of silver were grown in the super-critical region suggests that the technique of electrodeposition from dense gaseous solution might be useful for growing whiskers or other desirable crystal forms of various metals.

Note:

Requests for further information may be directed to:

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