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ELECTRON BEAM ELECTROLYSIS

by

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I. INTRODUCTION

Electron beam electrolysis is a method of electrolytically decomposing either fused or solid salts or ionically conductive ceramics. The ionic material rests on a metal contact which serves as anode. The cathodic lead is the stream of electrons which comes from a heated tungsten filament.

The apparatus consists of an evacuated vessel, about 10^{-5} torr, with a tungsten filament heated to incandescence by the ordinary house alternating current. A potential of about 1000 volts is applied between the filament (negative) and the metal in contact with the ionic material. Electrolysis occurs on the surface of the material where the stream of electrons impinges. For these experiments the electron stream (hereafter referred to as a "beam") does not need to be focussed. A current of 50 to 100 ma passes in a typical experiment. Simple compounds, such as silver chloride and nickel chloride, have been electrolyzed to yield the metal. Faraday's law of electrolysis appears to hold.

At the present time, we have not determined the advantages of electron beam electrolysis over ordinary electrolysis involving two metal connections (for anode and cathode). In electron beam electrolysis the liberated metal is not in contact with another metal and, therefore, presumably might be prepared in a state of higher purity. Metals of low boiling point, such as sodium and

cadmium, volatilize when formed and presumably could be caused to form a film on objects exposed near the region of electrolysis.

The present work is concerned with attempts to electrolyze ceramic materials.

II. EXPERIMENTS DURING THE FIRST QUARTER

A. Electrolysis

Two electrolyses were conducted. In one experiment, solid nickel chloride was electrolyzed to yield nickel. In the other, sodium borate was electrolyzed. Sodium deposited on the walls of the vessel but could not be recovered, since the thin film reacted with the air as soon as the vessel was opened. A transparent lump of boric oxide was formed over the remainder of the sodium borate.

Attempts to electrolyze ceramic material (aluminum fluoride, magnesium phosphate, and aluminum phosphate) were not successful. The failure was owing to two causes: (1) inadequate heating of the material to provide electrical conduction between the anode contact and the electron beam, and (2) leakage of the current from the beam to exposed places on the anode lead. Consequently, the main emphasis during the past month has been on blank experiments to determine the factors governing electrical leakage, and on redesign of the equipment to permit heating of the electrolyte and more convenient collection of products which condense on the walls of the reaction vessel.

B. Observations on current flow

We observed the current flow between the tungsten filament and a plate of copper serving as anode, no ionic material being present. The flow of current was about 100 ma with 1000 volts between the filament and the copper plate. The distance between the filament and the copper plate was a minor variable as the current flow was only about 10 percent less when the filament was about 5 cm distant instead of two cm distant.

Covering the copper plate with a beaker reduced the current almost to zero, thus showing that the beam cannot flow easily into reentrant angles. However, covering the copper plate with a large watch glass did not reduce the current flow appreciably. These experiments indicated the need of carefully insulating the anode lead. The presence of a crack or a small exposed area through the insulation appeared to be sufficient to permit a flow of current. Further orienting experiments are in progress.

III. REDESIGN OF EQUIPMENT

No diagram is given of the presently used equipment, as it is not satisfactory and is now being modified. Three improvements are in process.

(1) The new equipment provides for the reaction vessel to be in three sections. The middle section, which is the one in which the electrolysis will occur, will be readily removable. This will permit examination of deposits on the wall without hindrance from the presence of electrodes and other paraphernalia.

(2) The receptacle for holding the electrolyte is being constructed of a high temperature refractory material which is not conductive under the conditions of the experiment. Materials of construction are aluminum oxide, mullite, or fused silica. The insulation for the anode wire is a ceramic tube, which is an integral part of the receptacle. This obviates any joints through which electrical leakage can occur.

(3) A susceptor which is heated by induction is being placed around the ionic material. This will permit independent control of the temperature. In previous experiments, the solid salts had to be heated by the heat radiated from the tungsten filament to a temperature of about 400 C before adequate conductivity through the mass occurred. The ceramics which we wish to electrolyze apparently require a considerably higher temperature.