Electrodeposition of Antimony Utilizing Battery Wastes in A Divided Cell

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THE choice of a suitable bath for electrowinning of antimony from secondary sources like discarded lead-acid batteries, cable sheaths etc., and the successful operation on a laboratory scale, of a fluoride-sulphate bath have been discussed in an earlier paper[1]. Employing a current density of 12 asf at an average cell voltage of 1.7 volts, a current efficiency of 85 per cent was realised. It was indicated that the process could be improved by employing a diaphragm cell. The use of a diaphragm was suggested to prevent the formation of pentavalent antimony which does not deposit and which will make the cyclic process impractical without being reduced to trivalent state. The results of laboratory scale experiments in a 5 ampere diaphragm cell are presented now.

A battery container of size 9″ x 6″ x 8″ made of hard rubber was used as the cell. Microporous rubber sheet used in lead acid storage batteries as separator (brand name MITEX) was used as diaphragm material. A rectangular anode box (7″ x 2½″ x 7″ ht.) was made with the diaphragm material suitably fitted to a teakwood frame treated with chlorinated rubber paint[2]. Sulphuric acid of 20-25 per cent strength was used as anolyte with lead or lead-antimony alloy anodes of size 6½″ x 5″ x 1/8″ thick. Monel sheet of size 8″ x 5″ x 1/32″ thick, polished with fine emery was used as the cathode. Two anodes and one cathode were used in these experiments.

The antimony concentration in the catholyte has been found to have a marked effect on the nature of the deposit. Grey, powdery deposit is obtained when antimony content is below 40 g/l. Higher concentrations gave a smooth, silvery deposit. In our experiments the catholyte composition was as follows: antimony 70-100, free HF 30-40, H_2SO_4 300 g/l. Clove oil and beta-naphthol were used as addition agents. It is found that clove oil is beneficial only in very small quantities and therefore not more than 0.1 c.c. of clove oil per litre of catholyte was added to the bath. It was also necessary to stir the catholyte well after adding the clove oil and allow it to stand for 4-6 hours before starting the electrolysis.

On conducting the electrolysis at a current density of 12 asf, a cell voltage of 2 volts was maintained over a period of six to seven hours in a number of experiments. A smooth, glistening white deposit adhering to the cathode was obtained. The deposit was washed and dried and removed as flakes by flexing the cathode. A current efficiency of over 95 per cent (with a maximum of 98 per cent) has been obtained under normal experimental conditions.

A comparative table showing the results with diaphragm and non-diaphragm cells is presented below:

From the above it may be seen that the use of diaphragm has served to increase the current efficiency, mainly through the prevention of formation of pentavalent antimony. The process is therefore made cyclic without any additional step. Although normally it is expected that the use of a diaphragm would mean increase in electric energy requirements, yet in this case it remains practically the same owing to higher current efficiencies made possible.

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REFERENCES

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TABLE I

	Diaphragm Cell	*Non-diaphragm Cell*
Catholyte	HF-H_2SO_4 bath with 70-100 g/l Sb	HF-H_2SO_4 bath with 120 g/l Sb.
Anolyte	20-25% H_2SO_4 in Mitex diaphram boxes	No separate anolyte
Cathode	K-Monel	K-Monel
Anode	Lead or Pb-Sb Alloy	Acheson Graphite
Inter-electrode distance	2"	1.5"
Current density	12 asf	12 asf
Cell voltage	1.95-2 V	1.7 V
Duration of electrolysis	7 hours	5 hours
Current efficiency	95-98%	85-86%
Nature of deposit	Smooth, silvery, adherent	Smooth, silvery, adherent
Purity of the deposit	High Purity	High Purity
Energy consumption	0.60 Kw.hr/lb average	0.60 Kw.hr/lb average
Addition Agents (per litre of catholyte)	Clove oil 0.1 cc, beta-naphthol 0.01 gm.	Clove oil 0.1 cc, beta-naphthol 0.01 gm.