

## APPLICATION OF A SELF-CLEANING ROTATING ELECTRODE (S.R.E.) FOR POLAROGRAPHIC ANALYSIS OF INDUSTRIAL SLAGS

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Preliminary results obtained for  $\text{MnO}_2$  determination point to good perspectives in practical analysis.

The self-cleaning rotating electrode (S.R.E.) constructed by Noninski<sup>1,2</sup> was successfully applied in the polarography of water solutions<sup>3,4</sup> and has proved its advantages. Very good results were obtained with the same electrode in the polarography of fused salts as well<sup>5-6</sup>.

In the present paper the SRE is applied for the analysis of industrial ferromanganese slag. The main components of this slag are the oxides  $\text{MnO}_2$ ,  $\text{CaO}$ ,  $\text{MgO}$  and  $\text{SiO}_2$  and traces of other oxides.

### EXPERIMENTAL

All the polarograms in the present paper are taken on a platinum S.R.E. and using apparatuses shown in Fig. 1. Registration of the polarograms was done on Philips X-Y recorder PM 8120 with the help of the polarograph LP-7. The chemicals were of the purity grade and were twice recrystallized in bidistilled water.

The supporting electrolyte polarogram was taken at 850°C, the potentials being measured *versus* a Pt electrode of 50/50/0.5 mm dimensions. All the polarograms in the present paper are taken at a sweep rate of 4 V/min and at a rotation rate of 500 rpm.

### RESULTS

The experiments made with various melts showed that  $\text{K}_2\text{CO}_3\text{--Na}_2\text{CO}_3$  melt was the most suitable as supporting electrolyte because it dissolves the slag to the necessary extent thus enabling to carry out the polarographic analysis.

In the supporting electrolyte polarogram a clearly expressed wave at 0.4 V can be seen. The appearance of this wave is probably due to the formation of carbon monoxide whose bubbles cause a specific concentration polarization when  $\text{CO}_3^{2-}$  neutralize. Limiting currents of this sort were observed by other authors as well<sup>7-10</sup>. When

TABLE I

Experimental results from the polarographic analysis with the help of S.R.E. of probes of unknown  $\text{MnO}_2$  content in industrial ferromanganese slag from "Kremikovtzi" plant in Sofia compared to the results from other kinds of analyses of the same probes

No	Probe No	Slag mg	$-\phi_{1/2}$ V	$h$ mm	$i_d$ mA	$\delta, \%$	$\text{MnO}_2, \%$		
							express analysis	quantometer	our data
1.	8 750	15	0.88	16	8	4.18	15.97	16.35	16.33
2.	8 530	15	0.86	11.5	5.8	4.11	18.85	12	12
3.	8 710	15	0.88	10.5	5.3	4.34	10.4	10.7	10.7
4.	8 658	15	0.87	15	7.5	3.78	18.57	16.6	16.71
5.	9 595	15	0.88	14	7	4.24	15.2	15.27	15.31
6.	8 676	15	0.86	11	5.5	4.41	9.68	12	11.88

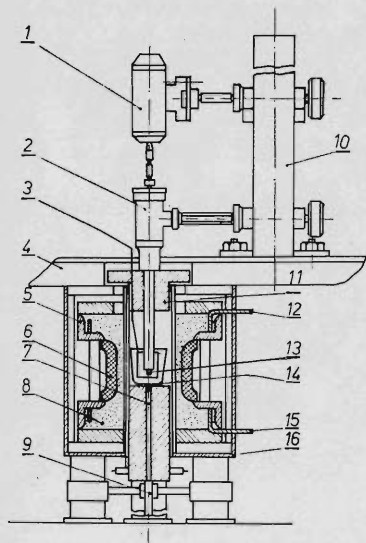


FIG. 1

Polarographic apparatuses based on S.R.E. 1 electric motor, 2 S.R.E. (Pt), 3 counter electrode, 4 concrete basement, 5 ring, 6 cryptol element, 7 thermocouple Pt-Rh/Pt, 8 cryptol, 9 rotating system, 10 leading column, 11 protecting corundum lid, 12, 15 electrodes of the furnace, 13 cleaning element (sharpener), 14 corundum crucible (polarographic cell), 16 coat of the cryptol furnace

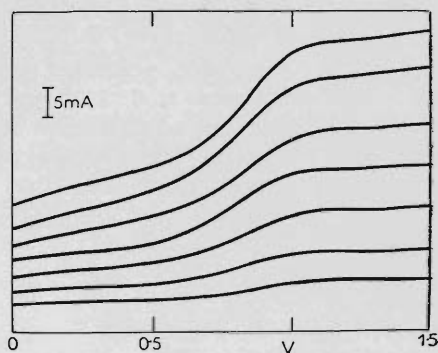


FIG. 2

Polarograms of industrial ferromanganese slag in  $\text{K}_2\text{CO}_3\text{-Na}_2\text{CO}_3$  melt on platinum S.R.E.; concentration of  $\text{MnO}_2$  varies from 1 up to  $14 \cdot 10^{-3}$  w. %

increasing the temperature the wave gradually becomes less distinct and at about 1 000°C it disappears almost entirely.

The main component which should be determined in the slag is  $\text{MnO}_2$ . Therefore, it was convenient to study first of all the behaviour of pure  $\text{MnO}_2$ . On the basis of the obtained waves a calibration curve was plotted for pure  $\text{MnO}_2$  in the supporting electrolyte  $\text{K}_2\text{CO}_3\text{--Na}_2\text{CO}_3$  at 1 000°C.

In Fig. 2 the polarographic waves of  $\text{MnO}_2$  in the industrial slag are shown. We made analysis of various probes of industrial slag of unknown  $\text{MnO}_2$  content. The obtained results were compared with the results for the same probes obtained with the help of a quantometer and an express (wet) analysis. The results are shown in Table I. It is seen that a very good coincidence of our data with the quantometric ones has been obtained. The results obtained by us (*e.g.* the relative error 3.5%) show good perspectives for the application of the S.R.E. in the analysis of industrial slags.

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