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Zinc Electrowinning under Periodical Reverse Current (PRC) Behavior of the Cathode and Effects of Lead Impurities

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ABSTRACT

Laboratory-scale experiments of zinc electrowinning under periodical reverse current (PRC) were carried out in acidic sulfate solutions. To study the effects of lead impurities on the zinc cathode, 0.3–15 mg/l of lead were added to the electrolyte. A square wave of current was used to produce the PRC. Current densities of 500 and 1500 A/m² were investigated under both direct current and PRC. In the case of PRC, the direct and reverse pulses had respective durations of 100s and 0.1–1.0s. The current density of the reverse pulse was set at half the value of the direct pulse. Comparisons were made with conventional dc electrolysis. The presence of 5 mg/l of lead lowered the current efficiency in dc from 94.1 to 93.2%, while in PRC it raised it from 93.2 to 97.6%, using a reverse pulse duration of 0.1s. Correlation between the current efficiencies and the presence of the crystallographic plane (101) was also observed. It suggests that lead in the zinc deposit and the use of PRC influence the crystallographic growth of the zinc deposit. The presence of plane (101) increases the overpotential of hydrogen evolution on zinc, and this results in a higher current efficiency. A way of obtaining the beneficial plane is to use PRC and lead in the electrolyte together.

The increasing cost of energy in the zinc industry has driven research towards less energy intensive processes (1–7). The use of periodical reverse current (PRC) during zinc electrowinning has shown impressive preliminary results (8–11). During electrowinning of zinc in acidic media the authors found that desorption of a shielding chemical such as OH_(ad) occurs at the lead-silver anode during current reversal (11). This results in a lower anodic overpotential, improving the specific energy consumption.

Even if no additives or impurities were purposely added in the latter study, some lead coming from the anode during the experiments could have contaminated the zinc and influenced current efficiencies.

The present investigation aims at a better comprehension of the effect of PRC techniques on the mechanisms of the cathodic process involved during zinc electrowinning in the presence of lead impurities in the electrolyte. This made it possible to explain the results of previous work in this laboratory (11) about the relation between the use of PRC and current efficiencies. The same experimental setup was used in this study except for the use of insoluble anodes in order to control the effects of lead concentration in the electrolyte. Chemical analysis, crystallographic orientation, and morphology of the zinc deposits as well as current efficiencies were recorded in order to characterize the phenomena.

Experimental

Cell.—The cell was composed of one 6 cm² aluminum cathode (supplied by Noranda) placed between two dimensionally stable anodes (DSA) 3 cm² each (supplied by Eltech Corporation). The DSA were made from titanium covered with iridium oxide. The electrodes were held by plastic grips and set in a Teflon support to maintain an interelectrode spacing of 3.8 cm, in order to simulate industrial conditions. The aluminum electrode was polished with 600 grit sandpaper before each experiment to assure reproducible surfaces, but the DSA did not need any kind of treatment. Each surface was delimited with electroplating tape (3M no. 484) and lacquer (Microstop from Michigan Chrome and Chemical Company). The whole cell was contained within a 2 liter beaker. After electrolysis, the zinc deposit was rinsed, dried, and peeled. A Mettler analytical scale (Type H-16) was used to weigh the deposit and determine the current efficiency.

Electrolyte.—The solution was made of 60 g/l of zinc from ZnSO₄ · 7H₂O and 200 g/l of H₂SO₄. Although no lead was added to it, this solution contained 0.3 mg/l of lead. It came from the zinc sulfate as an impurity and is considered to be within the reagent specifications. Lead was added to the electrolyte from a 2 g/l PbSO₄ solution in order to obtain concentrations of 5, 10, or 15 ppm. Because solubility of PbSO₄ in sulfuric acid is equal to 6.13 mg/l or 3.31 ppm Pb²⁺ (12), some particles sometimes remained in suspension in the electrolyte, and for this reason it was vigorously agitated before each experiment. All chemicals used were reagent grade, and water was bidistilled. The temperature of the electrolyte was maintained at 40° ± 0.5°C by placing the cell in a thermostated water bath. This temperature was found to be optimum for energy consumption, current efficiency, and Pb-Ag anode corrosion (13–15). No stirring was done during the 2h experiment, and the volume of the electrolyte was large enough (1 liter) to limit the depletion of zinc ions to less than 5%.

Electrical circuit.—The electrowinning current was generated by a PAR potentiostat/galvanostat (Model 173) equipped with an IEEE-488 programmable interface. Current densities of 500 and 1500 A/m² were investigated under direct current and PRC. In the case of PRC, square wave was preprogrammed on an IBM-PC microcomputer and stored into the erasable memory of the interface. The direct pulse had a duration of 100s while the reverse pulse was set at 0.1, 0.5, and 1.0s. The typical risetime of such a current wave is on the order of 500 μs.

Chemical analysis.—The flame spectroabsorption technique was used to measure the lead content of each zinc deposit. The limit of detection of this method is on the order of 0.1 ppm with an accuracy of 1–2%, and the results obtained gave a global value of the lead content.

Roughness analysis.—Each zinc deposit was glued to an antivibration marble table. A probe was placed perpendicularly to the surface and was activated by a "Clevite Brush Surfdrive 100" recorder. The cutoff of the probe was set at 0.76 mm, and the variations recorded were transmitted to a "Clevite Brush Surfdrive MS-1000-01" integrator which gives the mean, maximum, and minimum values of the height of surface peaks. The roughness is defined here as the average of 5 times the mean.

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Crystallographic orientation.—X-ray diffraction was used to determine the preferred crystallographic orientation during the deposition of zinc (16). To achieve this, each deposit was cut in such a way that a 1.5 cm² area was available. It was then coated with Bakelite and polished up to 15 μm. This last step caused some plastic deformations, and they were removed by etching the surface with a 10% HNO₃ solution for 30s. This procedure permits good sample reproducibility.

Samples were then put in a Phillips PW 1130 diffractometer to obtain diffraction patterns. Since a material has a preferential crystallographic orientation when its grains do not grow randomly (17, 18), the diffraction pattern of each deposit corresponding to a specific experimental condition was compared with the pattern obtained from a zinc powder (Reagent Grade type) which had its grains randomly oriented. Fifteen crystallographic planes were obtained per pattern: (002), (001), (101), (102), (103), (110), (004), (112), (200), (201), (104), (202), (203), (105), and (114). Each plane corresponds to a peak on the diffraction pattern. The position of each peak is defined by Bragg's rule (18). Its height on the diffraction pattern is referred to here as its intensity. These intensities were first used to calculate the intensity ratio, which is the intensity of a plane (hkl) divided by the summation of the intensities of all the fifteen planes

$$R_{(hkl)} = \left(\frac{I_{(hkl)}}{\sum_{a=1}^{15} I_{(hkl)_a}} \right) \cdot 100\% \quad [1]$$

From this, a texture coefficient was calculated for each plane by dividing the intensity ratio obtained from a deposit for a plane (hkl) by the intensity ratio of the zinc powder for the same plane (hkl). This is expressed by Eq. [2]

$$TC_{(hkl)} = R_{(hkl)} / R_{\text{zinc powder}(hkl)} \quad [2]$$

Finally, the absolute texture coefficient ($ATC_{(hkl)}$) was calculated. $ATC_{(hkl)}$ is given in the form of a percentage of the

surface which is covered by the (hkl) plane and is calculated by Eq. [3]

$$ATC_{(hkl)} = TC_{(hkl)} / \sum_{j=1}^{15} TC_{(hkl)_j} \cdot 100\% \quad [3]$$

Like the texture coefficient ($TC_{(hkl)}$), it expresses the importance of a given orientation (hkl) compared with the one obtained from the zinc powder.

Microscopy.—To study the morphology of zinc deposits, a scanning electron microscope (SEM) Jeol T-840 was used. It made it possible to assess the effects of lead and PRC on the surface growth.

Results

Tables I and II summarize the results for 500 and 1500 A/m², respectively. For each experimental condition, the tables give the mean value (in μm), the minimum value, and the maximum value of the roughness of the deposit. They also give the current efficiency and the amount of lead in the deposit.

A typical relationship between the lead in the electrolyte and the amount of lead in the deposit is given in Fig. 1. It shows that for a given concentration of lead in the electrolyte, the use of PRC, compared to dc, lowered the amount of lead in the deposit.

Figure 2 shows that an increase in the current density decreased the amount of lead in the deposit for the experimental condition of PRC with 0.1s of reverse time (t_r). The effect of current density was similar for values of t_r equal to 0.5 or 1.0s. The results agree with other works (20-24) made in dc.

Figure 3 shows the effect of lead in the zinc deposit on the current efficiency at 500 A/m². The amount of 5 mg/l of lead in the solution (corresponding to 60 ppm in the deposit) combined with the use of PRC at t_r equal to 0.1s gives the best performance. The current efficiency in-

Table I. Experimental results for dc and PRC electrowinning at 500 A/m²

t_r (s)	Lead in solution (mg/l)	CE (%)	Roughness lo-hi-me (μm)	Lead in deposit (ppm)	Current of lead (mA/m ²)
0.0	0.3	94.1	6.9-10.4-8.6	12.6	1.9
0.0	5.0	93.2	2.8-4.3-3.6	122.0	17.9
0.0	10.0	92.8	3.3-4.0-3.7	176.0	25.8
0.0	15.0	92.3	3.4-4.8-4.0	214.0	31.2
0.1	0.3	93.2	3.6-7.1-5.3	18.8	2.8
0.1	5.0	97.6	2.0-4.0-3.0	61.0	9.4
0.1	10.0	96.5	3.0-4.6-3.9	117.0	17.8
0.1	15.0	96.0	3.3-4.6-3.9	168.0	25.4
0.5	0.3	92.6	6.9-9.7-8.3	15.7	2.3
0.5	5.0	95.1	2.3-4.3-3.3	94.0	14.1
0.5	10.0	94.5	3.6-4.8-4.2	170.0	25.3
0.5	15.0	92.9	3.0-4.8-3.9	198.0	29.0
1.0	0.3	93.1	6.6-8.6-7.6	9.5	1.4
1.0	5.0	94.1	3.0-4.3-3.7	98.0	14.5
1.0	10.0	94.5	2.5-4.8-3.8	164.0	24.4
1.0	15.0	93.8	3.3-4.3-3.8	224.0	33.1

Table II. Experimental results for dc and PRC electrowinning at 1500 A/m²

t_r (s)	Lead in solution (mg/l)	CE (%)	Roughness lo-hi-me (μm)	Lead in deposit (ppm)	Current of lead (mA/m ²)
0.0	0.3	91.0	13.7-16.3-15.0	0.0	0.0
0.0	5.0	90.9	11.4-13.7-12.6	86.0	37.0
0.0	10.0	90.9	11.2-13.0-12.1	120.0	51.6
0.0	15.0	91.2	10.2-12.7-11.4	148.0	63.9
0.1	0.3	89.1	14.0-15.8-14.7	9.9	4.2
0.1	5.0	93.4	8.1-10.2-9.1	58.0	25.6
0.1	10.0	93.0	9.1-11.7-10.4	100.0	44.0
0.1	15.0	92.3	4.8-11.4-10.8	130.0	56.8
0.5	0.3	90.4	13.7-15.8-14.7	6.6	2.8
0.5	5.0	91.1	9.1-11.2-10.2	63.0	27.2
0.5	10.0	91.9	10.2-12.5-11.3	109.0	47.4
0.5	15.0	90.5	9.7-12.7-11.2	127.0	54.4
1.0	0.3	90.1	12.7-15.8-14.2	13.4	5.7
1.0	5.0	91.0	9.7-11.7-10.7	75.0	32.3
1.0	10.0	90.2	11.9-14.5-13.2	107.0	45.7
1.0	15.0	90.4	12.7-14.7-13.7	123.0	52.6

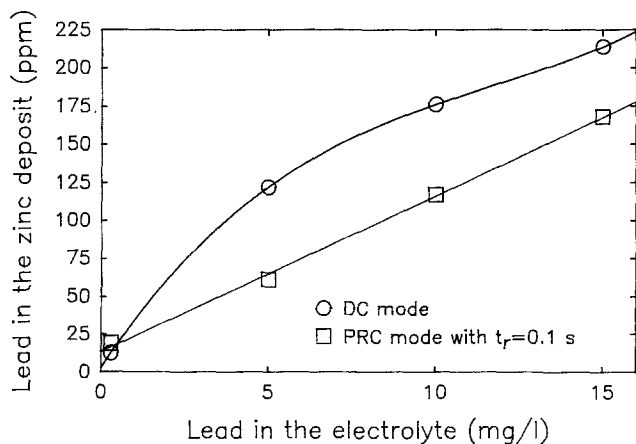


Fig. 1. Lead in the zinc deposit for different value of lead in the electrolyte. Current density at 500 A/m^2 .

increases from 93.2% in dc to 97.6% using PRC. The same effect was observed at 1500 A/m^2 , as shown in Fig. 4. The current efficiency increases from 90.9% in dc to 93.4% using PRC.

Microscopic aspects of the zinc deposit obtained in direct current are shown on Fig. 5 with 5 mg/l of lead and without lead. It appears that the lead affected the morphology of the zinc deposits. This observation agrees with other works (20, 24) made in dc, where it is reported that lead gives a finer grain. From Fig. 6, which represents PRC conditions, the same deduction about lead can be made, because it also decreased the grain size.

Figure 7 gives the relation between the roughness of the zinc deposit and the amount of lead contained in it, for 500 and 1500 A/m^2 . The only factors influencing the roughness were the amount of lead in the deposit and the current density. Applying PRC (a square wave) that generates pulses of inverse current did not affect the roughness, as is shown in Fig. 7.

Based on the data from Tables I and II, there is no correlation between the roughness and the current efficiency. The different current efficiencies observed in the various experimental conditions were not attributable to the roughness of the zinc deposit.

In order to evaluate the effects of surface morphology on the current efficiency at 500 A/m^2 , x-ray diffractions were performed on zinc deposits. A total of 10 experimental conditions were studied, and diffraction patterns were obtained for each deposit. On each pattern, 15 crystallographic orientations were registered, and for each orientation an absolute texture coefficient was calculated (see Experimental section for details). Table III gives the absolute texture coefficients and the experimental conditions for each of the 10 deposits.

Statistical analysis (25) was undertaken on the crystallographic orientations of zinc deposits to evaluate how they

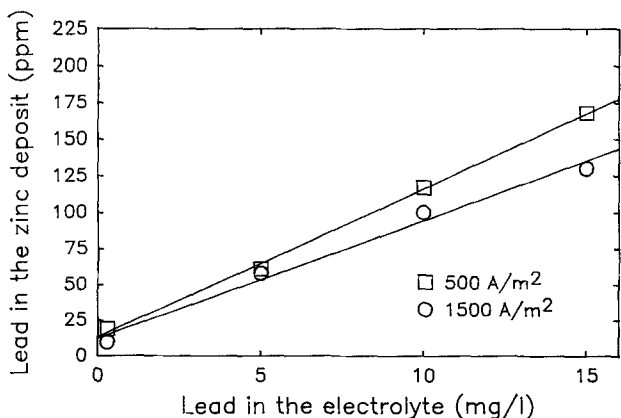


Fig. 2. Lead in the zinc deposit for different value of lead in the electrolyte.

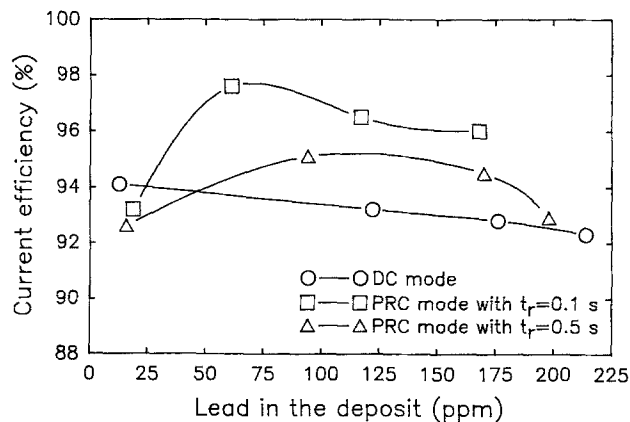


Fig. 3. Effect of the lead content of zinc deposits on the current efficiency at 500 A/cm^2 under dc and PRC.

were related to the current efficiency. Because of the limited amount of data available, the first step was to point out the predominant crystallographic orientations. Thus, planes having very low $ATC(hkl)$ were discarded. Planes having $ATC(hkl)$ below 4% were not considered for future treatment. Six planes are in this category ((002), (100), (110), (004), (200), and (201)), leaving 9 planes for more mathematical treatment.

In order to find the predominant crystallographic orientations affecting the current efficiency, mean square error (MSE) values were calculated (Table IV). To calculate a MSE for a given plane, the 8 remaining planes are used to compute multiple regression and MSE. Discarding a plane that correlates well with the current efficiency results in a greater error in the multiple regression of the 8 other planes. The higher the value of MSE, the greater is the predominance of the corresponding crystallographic orientation. This procedure was repeated 9 times so that for each plane there is a corresponding value of MSE. This made it possible to establish a table showing the importance of the correlation between a plane and the current efficiency. Table IV gives the MSE values for the nine planes studied. From this table, plane (101) is the most predominant, followed by (105).

Figure 8 shows that the relation between current efficiency and $ATC(101)$ is linear. A higher value of surface covering of (101) plane increases the current efficiency. Figure 9 shows a more scattered relationship, but it can still be seen that a higher value of $ATC(105)$ tends to decrease the current efficiency. The presence of plane (105) will be detrimental for the zinc electro-winning process.

Discussion

PRC affects the crystallographic orientation.—It is known that the hydrogen overpotential on zinc deposits varies, depending on the crystallographic orientation

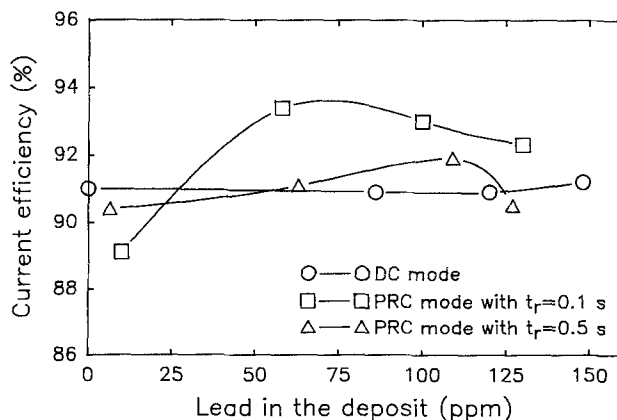


Fig. 4. Effect of the lead content of zinc deposits on the current efficiency at 1500 A/m^2 under dc and PRC.

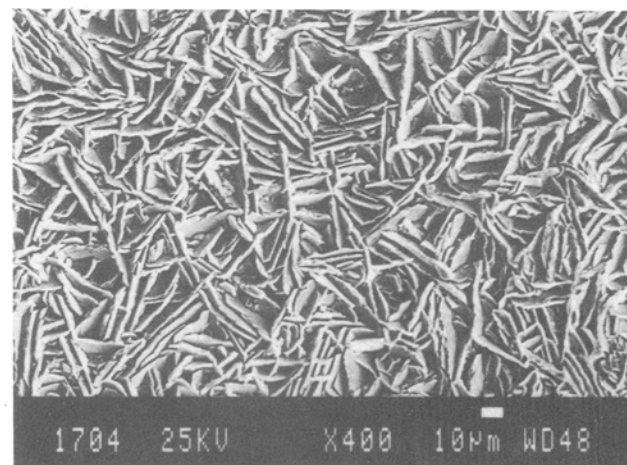
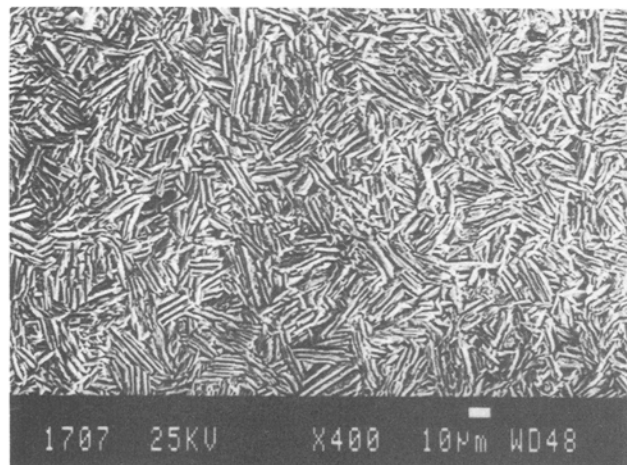
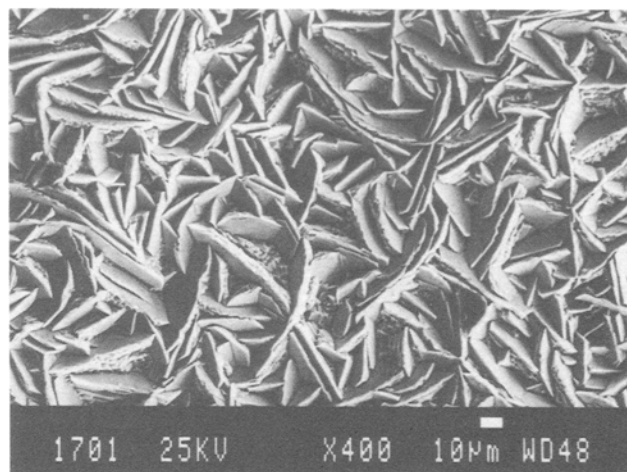
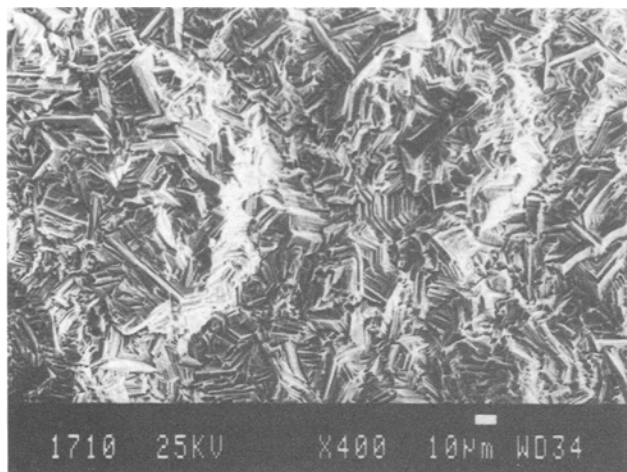


Fig. 5. Morphology of the zinc deposit obtained in dc mode at 500 A/m² without lead (top) and with 5 mg/l of lead (bottom).

Fig. 6. Morphology of the zinc deposit obtained in PRC mode at 500 A/m² without lead (top) and with 5 mg/l of lead (bottom).

(19, 24). If the presence of a given plane (hkl) caused the hydrogen evolution on the zinc surface to be low, a large surface covering of that plane will increase the current efficiency of the zinc deposit. Thus, a preferred orientation will have a direct effect on the current efficiency of zinc deposition.

As shown in Fig. 8, the presence of crystallographic orientation (101) had a beneficial effect on the current efficiency. Moreover, values of ATC(101) were higher when PRC and lead were used simultaneously. This suggests that there is an effect between PRC and lead in solution that influences the crystallographic growth of the plane (101).

Figure 9 shows that using PRC lowered the ATC(105) compared to dc, independently of the amount of lead in the electrolyte. The highest value of ATC(105) in PRC was 4.03% (with $t_r = 1$ s and 0.3 mg/l of lead in the electrolyte) compared to the lowest value in dc equal to 5.9% (with 0.3 mg/l of lead).

In dc, lead influences the morphology of zinc deposits (20, 24). It acts as a germination site for the zinc, and the result is a smoother surface. The same observation was made in this study for dc (Fig. 5) and PRC (Fig. 6). The differences of morphology observed in these figures between PRC and dc were not attributable to the roughness. Figure 7 revealed that PRC did not influence the roughness compared to the dc mode. Thus, the different morphologies were caused by the change in crystallographic growth between PRC and dc. The pattern of Fig. 6 with 5 mg/l of lead in solution gives the best current efficiency obtained in this study, with 97.6%.

Concentration of lead influenced by PRC.—Lead is more noble than zinc and its deposition will be ruled by mass transport, because of its low concentration in the electrolyte (from 0.3 to 15 mg/l of lead). Thus, stirring of the elec-

trolyte or an increase in the amount of lead in the solution will increase the content of lead in the zinc deposit.

As mentioned in the Experimental section, there was no stirring of the solution. However, hydrogen evolution at the cathode caused local agitation of the electrolyte near the electrode. Thus, a higher hydrogen current, caused by a lower current efficiency, will increase the amount of lead at the electrode, and this will result in a higher level of lead.

Deposition currents of lead were calculated for PRC and dc. The relation between the effect of hydrogen current, lead in solution and lead current is shown in Fig. 10. For 5, 10, or 15 mg/l of lead in the electrolyte, an increase in the

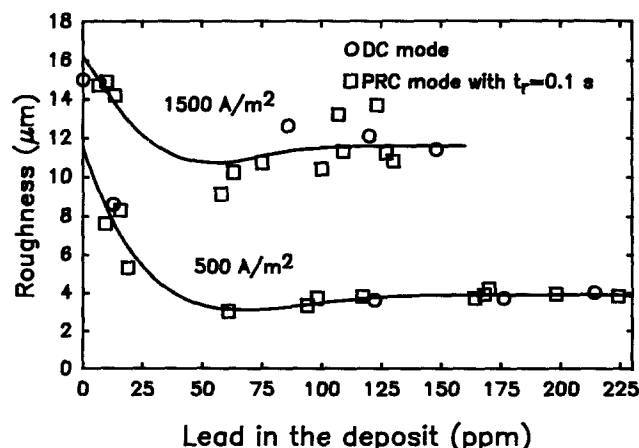


Fig. 7. Effect of lead in the zinc deposit on its roughness at 500 A/m² and at 1500 A/m².

Table III. Texture coefficients of zinc deposits obtained by x-ray diffraction

Crystallographic planes	PRC mode with reverse time, t_r (s)									
	DC mode		Lead in the electrolyte (mg/l)							
			0.1				0.5		1.0	
	0.3	10.0	0.3	5.0	10.0	15.0	0.3	10.0	0.3	10.0
(002)	4.18	1.77	0.40	0.40	1.44	0.90	1.79	0.69	2.19	1.15
(100)	3.48	1.11	0.82	1.01	1.97	1.24	0.64	0.64	1.69	1.53
(101)	8.37	3.90	6.7	18.2	12.88	9.39	4.59	5.87	5.01	3.85
(102)	12.63	11.68	21.43	17.12	15.28	21.89	24.71	21.25	15.4	17.20
(103)	10.31	21.70	6.30	5.37	5.18	15.74	18.51	17.33	10.94	12.45
(110)	3.59	1.51	0.58	0.75	1.72	1.50	0.00	0.05	1.82	2.18
(004)	3.27	1.45	0.00	0.00	1.21	0.00	1.08	0.33	2.34	0.92
(112)	10.95	3.81	13.05	14.36	15.09	8.51	7.89	8.75	11.99	13.08
(200)	3.14	0.36	0.00	0.00	1.55	1.46	0.3	2.27	1.57	1.46
(201)	3.50	1.17	1.23	2.06	3.88	2.09	1.05	3.08	2.35	3.15
(104)	6.33	18.82	0.00	1.40	3.19	3.87	5.15	4.25	8.78	5.59
(202)	5.99	2.72	16.75	11.80	11.48	7.54	4.82	6.50	4.11	7.84
(203)	8.83	5.37	22.52	14.36	14.24	12.37	8.37	14.21	7.70	13.97
(105)	5.90	11.86	0.00	1.33	2.41	1.93	2.21	2.80	4.03	3.26
(114)	9.53	12.76	10.22	11.84	8.48	11.57	18.87	11.97	20.05	12.37

hydrogen current resulted in an increase in the lead current. However, with 0.3 mg/l of lead, a higher hydrogen current (more stirring of the solution) did not significantly increase the lead current.

It is reported in the literature (24) that in dc mode, an increase in the applied current density will lower the amount of lead in the deposit for a given value of lead in the electrolyte. In the case of PRC, the same observation was made (Fig. 2). Figure 10 shows that a higher hydrogen current density caused more stirring and resulted in a higher current of lead. However, more zinc is deposited at higher applied current densities and the zinc current increases faster than the lead current. The result is a lower lead content in the deposit.

The improved current efficiency observed in PRC mode for a given applied current density (Fig. 3) can explain the observed decrease of lead content in the zinc deposit compared to the dc mode, as shown in Fig. 1. An increase in the current efficiency decreases the evolution of hydrogen at the zinc cathode, resulting in less stirring of the electrolyte near the electrode. Less lead then reaches the surface to contaminate the zinc.

With 0.3 mg/l of lead in the solution, corresponding to a lead content lower than 20 ppm in the zinc deposit, there was no major difference in Fig. 3 between the current efficiencies obtained by PRC and dc. However the current efficiency in PRC was lower, and resulted in a higher amount of lead in the deposit, as shown in Fig. 1.

Balance changed by PRC.—Using PRC and lead in the electrolyte will affect the crystallographic orientation, which in turn tends to vary the current efficiency. As discussed earlier, current efficiency affects the amount of lead in the zinc deposit and therefore the crystallographic orientation. It is believed that a balance tends to build up between the amount of lead in deposit, the current efficiency, and the crystallographic orientation. Using PRC will tend to change this state and result in certain cases in better current efficiency.

The primary use of PRC is to lower the anodic overpotential of the lead-silver electrode used in the industrial process. The present study shows that using PRC alone could not increase the current efficiency. The presence of lead is necessary to create a balance that could affect the

crystallographic orientation. In the preceding study (11), the lead-silver anode was used. Thus, lead coming from the anode reached the zinc deposit and resulted in better current efficiency when using PRC.

Although the results show that PRC affects the crystallographic pattern of the surface of zinc deposits, more investigation is needed to determine the influence of the other crystallographic orientations on the current efficiency as well as the mechanism that maintains the balance previously mentioned.

Conclusion

It was shown that simultaneously using PRC and lead in the electrolyte changes the crystallographic orientation, increasing the surface coverage with plane (101) and de-

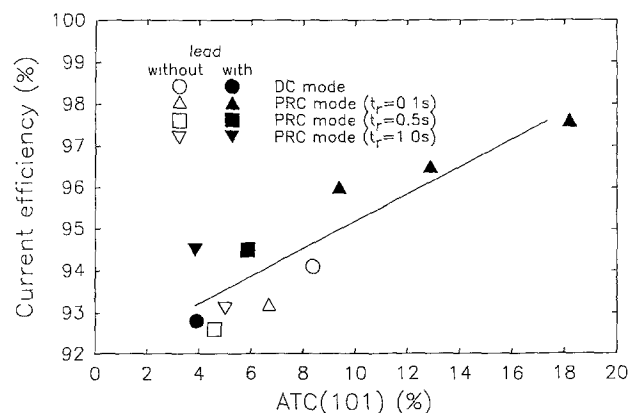


Fig. 8. Effect of the crystallographic orientation (101) on the current efficiency.

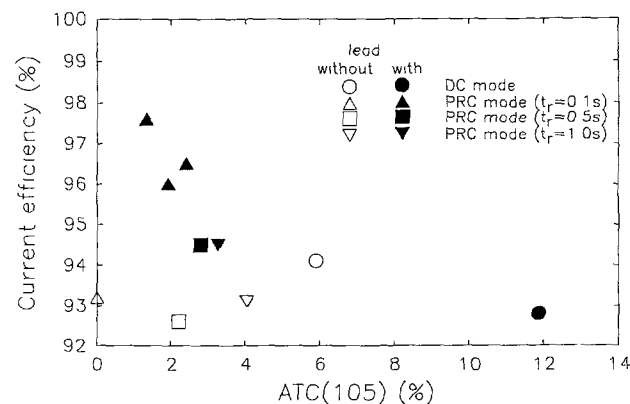


Fig. 9. Effect of the crystallographic orientation (105) on the current efficiency.

Table IV. Values of mean square error of crystallographic planes

Planes	MSE
(101)	1.577
(105)	0.840
(103)	0.605
(102)	0.505
(112)	0.412
(114)	0.364
(104)	0.347
(203)	0.269
(202)	0.268

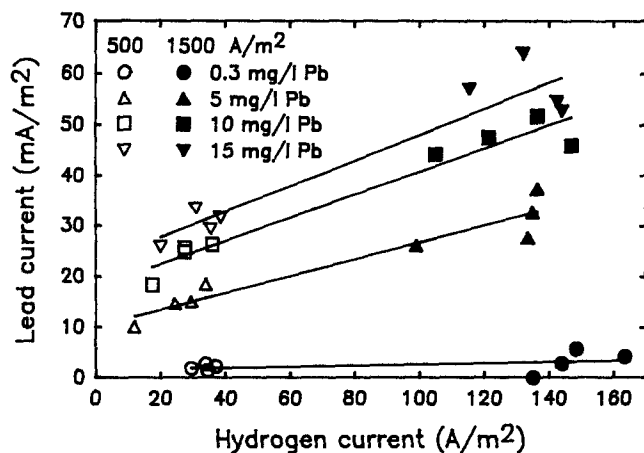


Fig. 10. Effect of convection resulting from the hydrogen current density on the lead limiting current density.

creasing it with plane (105). This resulted in a higher current efficiency.

Using PRC without lead or lead without PRC did not result in an increase of the current efficiency. With the presence of lead in the solution, PRC decreased the lead content in the zinc deposit. A balance between the current efficiency, the amount of lead in the zinc deposit and the crystallographic orientation is established during zinc deposition. Using PRC changed this balance, with the result that the current efficiency is increased in certain conditions.

In PRC mode an increase in the applied current density decreased the amount of lead in the zinc deposit. This was already observed in dc mode but not in PRC.

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LIST OF SYMBOLS

$I_{(hkl)}$	intensity of the (hkl) plane
$\sum_{q=1}^{15} I_{(h_1k_1l_1)}$	summation of the intensities of all planes
$R_{(hkl)}$	intensity ratio of the (hkl) plane
$R_{\text{zinc powder}(hkl)}$	intensity ratio of the (hkl) plane for the zinc powder
$TC_{(hkl)}$	texture coefficient of the (hkl) plane

$\sum_{i=1}^{15} TC_{(h_1k_1l_1)}$ summation of all texture coefficients

$ATC_{(hkl)}$ absolute texture coefficient of the (hkl) plane

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