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**THE EFFECT OF ALUMINA PROPERTIES AND
SMELTER OPERATING CONDITIONS
ON THE DISSOLUTION BEHAVIOUR OF ALUMINA**

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Abstract

Alumina performs several functions in a modern smelter - it is used to scrub the pot gases to remove fluoride, acts as a thermal insulator on top of the cell and, when added to cryolite-based electrolytes, it is the raw material used to produce aluminium. Alumina is also expected to have good flow and handling properties, and dissolve well in the bath. Unfortunately, it does not always dissolve rapidly and this leads to the formation of "sludge", which creates operating disturbances in the cell; it is difficult to remedy this problem without a basic understanding of the process of alumina dissolution. Consequently, the objective of the work presented in this thesis was to develop an apparatus and technique that would allow the dissolution behaviour of powder alumina to be measured as realistically and objectively as possible, and then determine the important factors affecting dissolution.

The method developed involved the merging of three different techniques:

- electroanalytical measurement of dissolved oxide concentrations
- recording of the associated thermal phenomena
- visual observation of the interaction of alumina with the bath

which were then used to investigate the effect of a range of alumina properties and smelter operating conditions on dissolution behaviour. A series of dissolution parameters was selected to evaluate the relative dissolution behaviour of the different variables.

It was found that slow dissolution behaviour resulted primarily from poor feeding and/or dispersion, coupled with poor heat transfer in the first few seconds of the dissolution process.

If the flow properties of the alumina were good, alumina flowed easily out of the dropper on to the surface of the bath forming relatively thin but well-distributed rafts. If not, alumina could flow out into a localized area producing very dense rafts which clumped together and eventually sank to the bottom forming sludge. Properties such as loss on ignition enhanced the dispersion of the alumina through the release of volatiles, which caused the alumina to "effervesce" on the surface. Conversely, bulk density aggravated the clumping problem as the density of the formed rafts increased with increasing bulk density.

Good feeder design can help to counteract deficiencies in the flow properties of an alumina, if the importance of wide coverage and the imparting of a horizontal velocity component to the alumina are kept in mind. It was also found that if the bath agitation was increased, either by increasing the amount of existing stirring or by reducing the bath volume in the feeding area for a given bath velocity, the initial heat transfer to the alumina could be dramatically improved. The presence of bath superheat was important for maintaining optimum heat transfer conditions for fast dissolutions, as alumina was found to dissolve better when a higher proportion of the heat required was supplied from the bulk of the liquid bath as opposed to localized freezing.

Increasing the initial alumina concentration in the electrolyte retarded the dissolution process, with the retardation becoming increasingly more significant at higher concentration values. Similarly, reducing the cryolite ratio, which also decreased the alumina solubility, resulted in the dissolution being inhibited in the later stages, as more alumina dissolved. In situations where the mass transfer in the bath was improved, the impact of these concentration effects was minimized.

The video recordings were useful for indicating potential operating difficulties with the samples, such as excessive emissions and flow problems. Six different raft formations, characterizing the degree of bath surface coverage and the raft density or cohesion, were identified from the videos and the aluminas were classified accordingly.

Preliminary heat balance calculations were performed using the data obtained in this study and were used to estimate the heat of dissolution for $\alpha\text{-Al}_2\text{O}_3$, for a variety of alumina concentrations in cryolite-based electrolytes. The results indicated heat values of 112 ± 15 and $55 \pm 5 \text{ kJ mol}^{-1}$ for alumina concentrations in the bath of 0.43 and 2.83 wt% respectively. Further work is necessary to refine these calculations, however.

Reacted ore was found to dissolve more slowly than parent virgin alumina but it was difficult to ascertain which property caused the difference - whether bulk density, flowability, influence of HF, or presence of other impurities. As a result, it is recommended that a series of dissolution runs be performed on reacted samples of different compositions to establish which property associated with reacted ore causes the problem.

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List of Symbols and Abbreviations

Symbol	Description	Unit
A	a constant	
A	interfacial area between the crucible and the bath	m ²
ALPHA	α -Al ₂ O ₃ content	wt%
b	Tafel slope	V
B	a constant	
BET SA	B.E.T. surface area	m ² g ⁻¹
C	actual concentration at time t	kg m ⁻³
C	alumina concentration	wt%
C	a constant	
C(1)	alumina concentration at the addition point	wt%
C _{exp}	negative exponential fit at time t	
C _p	heat capacity	J g ⁻¹ K ⁻¹
C _{pa}	heat capacity of the solid alumina	J g ⁻¹ K ⁻¹
C _{pb}	heat capacity of the bath	J g ⁻¹ K ⁻¹
C _{pb,1260}	heat capacity of the bath at 1260 K, 1.7970 J g ⁻¹ K ⁻¹	J g ⁻¹ K ⁻¹
C _{pc}	heat capacity of the graphite crucible	J g ⁻¹ K ⁻¹
C _{pc,1260}	heat capacity of the graphite crucible at 1260 K, 1.9173 J g ⁻¹ K ⁻¹	J g ⁻¹ K ⁻¹
C _s	saturation concentration	kg m ⁻³
C ₀	saturation concentration	mol cm ⁻³ or kg m ⁻³
%C	percent alumina concentration change, 0-100 %	%
ΔC	theoretical concentration change for the addition	wt%
$\Delta \%C_{F-s}$	scaled Fourier-smoothed percent concentration change	%
$\Delta \%C_{raw}$	scaled raw percent concentration change	%
Conc	alumina concentration	wt%
CR	cryolite ratio (wt NaF to wt AlF ₃)	
D	diffusion coefficient	m ² s ⁻¹
D	a constant	
ESP	electrostatic precipitator	
f ₁ (x)	mathematical function of x, where x is a variable	
f ₂ (x)	mathematical function of x, where x is a variable	
F	Faraday's constant, 9.652•10 ⁴ C mol ⁻¹	C mol ⁻¹
F	frequency of readings or seconds between readings	s
F _g	mass fraction of the alumina that is non-alpha alumina, 0-1	
FFT	flow funnel time per 100 g alumina sample	min

h_x	heat transfer coefficient between the crucible and the bath	$\text{W m}^{-1} \text{K}^{-1}$
H	Hann multiplication factor, 0-1	
H_{required}	total heat required for the dissolution of alumina	J
H_{supplied}	total heat supplied for the dissolution of alumina	J
H_1	total heat supplied using the purely conductive heat transfer approach	kJ mol^{-1}
H_2	total heat supplied using the combined convective/conductive heat transfer approach	kJ mol^{-1}
ΔH_{phase}	heat for the phase transformation to $\alpha\text{-Al}_2\text{O}_3$ at 1260 K	J
ΔH_{diss}	heat of dissolution	kJ mol^{-1} or J
i	current density	A cm^{-2}
i	number of a point or reading	
i_p	peak current density	A cm^{-2}
k	a constant	
k_T	temperature dependence constant at T °C	
k'	a constant	
K	dissolution rate constant	m s^{-1}
K	total number of cycles	
K_{add}	number of the addition cycle	
LBD	loose bulk density	g l^{-1}
LOI	loss on ignition (300-1000 °C)	wt%
LSV	linear sweep voltammetry	
m	amount of alumina dissolved per unit time	$\text{kg m}^{-3} \text{s}^{-1}$
m_a	mass of alumina dissolving	g
m_b	mass of the bath	g
m_c	mass of the crucible over which ΔT_c acts	g
MLSV	modified linear sweep voltammetry	
MOI	moisture on ignition (0-300 °C)	wt%
n	number of electrons transferred	
PBD	packed bulk density	g l^{-1}
PH	90th percentile of the particle size distribution	μm
PM	50th percentile of the particle size distribution	μm
PS	10th percentile of the particle size distribution	μm
q	dynamic heat flux	W or J s^{-1}
q_s	steady state heat flux or convective heat transfer rate	W or J s^{-1}
q_{xs}	increased heat flux	W or J s^{-1}
Q	area under the voltammogramme or quantity of electricity value	C
r	radius of the sample disc	m
r_{av}	average regression coefficient	
R	gas constant, $8.317 \text{ J mol}^{-1} \text{K}^{-1}$	$\text{J mol}^{-1} \text{K}^{-1}$
spf	single point feeder	
S	dissolution surface area	m^2

t	time	s
t_0	time at the end of the initiation period	s
Δt	change in time	s
t_{init}	initiation time	s
$t'50$	time taken to reach 50 % dissolved minus t_{init}	s
$t'66$	time taken to reach 66 % dissolved minus t_{init}	s
$t'80$	time taken to reach 80 % dissolved minus t_{init}	s
$t'f$	time taken to reach 100 % dissolved minus t_{init} (based on initial rate of dissolution)	s
T	temperature	K
T_{av}	average temperature of the crucible wall	°C
T_b	bath temperature	°C
T_{bath}	bath temperature	°C
T_c	crucible wall temperature	°C
T_{op}	bath operating temperature	°C
$T_{op,add'n1}$	bath operating temperature for the 1st addition	°C
$T_{op,add'n4}$	bath operating temperature for the 4th addition	°C
T_w	wall temperature of the outside of the crucible	°C
δT_b	transient change in the bath temperature	°C
δT_c	transient change in the crucible wall temperature	°C
ΔT_b	bath temperature change or drop	°C
ΔT_{bath}	bath temperature change	°C
ΔT_c	crucible wall temperature change or drop	°C
TcQ	temperature-corrected quantity of electricity value (coulombs)	C
T_{max}	maximum temperature drop recorded by the bath thermocouple	°C
v	sweep rate	$V s^{-1}$
V	solution volume	m^3
w	width of the Hann window	
x	addition number, 1-4	
α	angle of repose	°
$\alpha-Al_2O_3$	alpha alumina	
$\beta-Al_2O_3$	beta alumina	
$\gamma-Al_2O_3$	gamma alumina	
$\delta-Al_2O_3$	delta alumina	
$\theta-Al_2O_3$	theta alumina	
ν	kinematic viscosity	$m^2 s^{-1}$
π	a constant, ≈ 3.141592654	
τ	transition time	s
ϕ	angle of internal friction	°
$\chi-Al_2O_3$	chi alumina	
ω	angular velocity	s^{-1}

To the memory of Fritz,
The best dog an engineer could have.