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MANAGEMENT OF ACUTE PARTICULATE FOULING IN A TITANIUM DIOXIDE REACTOR SYSTEM

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ABSTRACT

The gas phase manufacture of titanium dioxide is subject to acute fouling in the cooler unit located directly downstream of the reactor which quenches the reaction. A model of the cooler system was constructed, incorporating aspects of compressible flow, multi-mode heat transfer, fouling and changes in geometry. This indicated that deposition could be very rapid. The effect of deposit layer build-up required measurement of the thermal conductivity of the porous layer: this was achieved using a novel testing device similar to that reported by Tan et al. (2006), for measuring the thermal conductivity of surface coatings. Active mitigation techniques are employed to reduce the effect of rapid fouling. The effectiveness of adding an erodent, in this case sand, to the flow was appraised by studying the break-up of deposit layers by impinging particles. The experimental conditions (high temperature chlorine gas, high flow velocities) were simulated in cold experiments by matching the inertia and size of test particles to those of the sand. These studies showed that sand at the feed size would detach deposits, but could result in breakage of the sand particles. Mitigation efficiency is then determined by sand distribution and re-distribution.

INTRODUCTION

Powdered titanium dioxide is a widely used commodity, used in products as diverse as paints, suncreams, catalysts and foodstuffs. One of the major commercial routes for manufacturing powdered titanium dioxide from TiCl₄ involves the continuous generation of TiO₂ particles of several microns in size by reaction with an oxygen plasma. The hot particulate product is conveyed away in gaseous chlorine into a serpentine cooler, essentially a long stainless steel pipe immersed in a flowing water stream. As the suspension cools, the reaction is quenched, setting the particle size and morphology. Careful control of the cooling stage is required in order to obtain the desired product properties. Heat transfer through the cooler walls is hampered by the rapid build-up of fouling layers of particulate TiO₂ with high porosity which can reach thicknesses of several mm.

Active mitigation techniques are currently employed to reduce the effect of fouling. Sand, which serves as an erodent, is injected into the dusty gas leaving the reactor to break up the fouling layer by impaction and saltation mechanisms. The sand particle size is approximately 1 mm so that it can be readily separated from the product and recycled.

The TiO₂ chloride process represents an unusual case of particulate fouling, in that the fouling precursor is the valued product and the range of mitigation options is limited by the reactor operating conditions and need to maintain product quality. This paper describes a series of investigations into TiO₂ particulate fouling aimed at identifying mitigation options open to the plant operator. The studies built on the foundational work on gas-side particulate fouling (*e.g.* the work by Young and Leeming (1997) and studies of flue gas cooler systems by Rindt and co-workers; Abd-Elhady *et al.*, 2007, van Beek *et al.*, 2001) as well as other techniques developed by the heat exchanger fouling community.

The paper is organized into three sections. The first describes the construction of a model of the process which allows the rate of fouling expected under different process conditions to be estimated. This is an important step as the scope for experimental studies is limited: the process stream is predominantly gaseous chlorine, initially at approximately 1000°C. Particulate fouling is controlled by transport and sticking steps, and the maximum rate of fouling can be determined from a knowledge of the characteristic particle relaxation time (see Young and Leeming, 1997). Estimation of this parameter requires knowledge of the local gas phase temperature, pressure and mean velocity, so these were calculated from a process model. The flows are turbulent so a one-dimensional model is sufficient to estimate these parameters with the level of accuracy required for the modelling work, given the uncertainty associated with many of the input parameters. Detailed, three-dimensional models of particle flow in similar geometries have been reported by other workers for fouling and eroding systems (e.g. Ai and Kuhlman, 2011; Yao and Fairweather, 2012; Njobuenwu and Fairweather, 2012), but not where powder deposition and particulate (i.e. larger species) erosion occur

simultaneously. As a first study in this area, the 1-D approach was considered appropriate.

The modelling work highlighted the need to determine the thermal conductivity of the fouling layers, $\lambda_{\rm f}$, so a device for measuring $\lambda_{\rm f}$ was constructed, based on the technique reported by Tan *et al.* (2006). The effectiveness of sand, and other candidates, as an erodent was assessed by measuring their friability at different gas velocities. This information was combined with measurements of the energy required to fracture deposits to assess the efficiency of erodent-based mitigation strategies Much of this third strand of work was informed by the literature on erosion (*e.g.* Hutchings, 1983; Humphrey, 1990).

Some data, particularly the pressures and flow rates, cannot be reported for reasons of commercial confidentiality.

PROCESS MODELLING

The product stream leaving the gas reactor is a hot (around 1100 K) gas stream (predominantly chlorine) containing approximately 0.03 volume % of TiO₂ particulates log-normally distributed with a mean size of approximately 0.285 μ m and geometric standard deviation of 1.38. This passes into the cooler which is a long (~ 250 m) tube arranged in a serpentine pattern, as shown in Figure 1. The device features five bends, which have larger internal diameters than the straight sections to decrease the fluid velocity and reduce erosion at the bends by entrained sand.



Fig. 1 Schematic of cooler configuration. Bends are labelled A-E: Roman numerals indicate changes in pipe i.d.

The cooler is immersed in a long trough along which cooling water passes in approximately plug flow. The need to contain the reactants, in particular chlorine, means that there are no instruments measuring temperatures and pressures along the cooler. The inlet and outlet conditions are monitored continuously.

The flow is turbulent everywhere with Reynolds numbers of the order of 10⁶. A one-dimensional model was constructed to find the local temperature, pressure, bulk fluid velocity and wall shear stress along the cooler. A series of coupled ODEs describing mass flow, momentum and heat transfer in the tube were solved simultaneously using the in-built MATLAB ode solvers. The model incorporated the following physics (space restrictions prevent the reporting of equations):

- (i) At the temperatures and pressures encountered in the cooler the chlorine can be modelled as an ideal, compressible gas. The volume fraction of the solids phase is small so the suspension can be modelled as a pseudo-gas, with the solids phase incorporated in the gas properties. Coupling of momentum transfer between the gas and solids was modelled using the drag force treatment reported by Crowe *et al.* (1998). The contribution of particle drag on the gas is added as a body force in the momentum equation.
- (ii) Heat transfer in the gas phase involves forced convection and radiation. The TiO_2 particles and the fouled surface are black, and radiation is significant in the early sections (I and II on Figure 1).
- (iii) The chemical reaction is exothermic and releases significant amounts of heat until 500 K when the reaction reaches equilibrium.
- (iv) The cooler diameter is large enough that the fouling layer can be treated as a thin slab. Heat transfer through the cooler wall is modelled as conduction through an annulus.
- (v) Heat transfer to the cooling water is complex, involving natural convection, forced convection and subcooled boiling at different positions. The contribution from each mechanism was estimated.

In the absence of fouling, the overall heat transfer coefficient for the benchmark flow rate of chlorine and TiO₂ ranged from 500 to 3000 W m⁻²K⁻¹. The dominant resistance was heat transfer from the gas phase to the pipe wall. The distribution of pressure, gas velocity, V_g , temperature, T_g , and wall shear stress, τ_w , are reported in Figure 2. For reasons of commercial confidentiality these parameters and the cooler diameter are plotted here and in Figure 4 in dimensionless form, scaled against the largest value of each parameter.

The temperature decreases rapidly in the first section, causing the conversion of remaining reactant, X_{TiCl4} , to reach a steady level rapidly. As the gas cools its velocity drops from around 60 m s⁻¹ to 20 m s⁻¹, with step changes at the bends. The wall shear stress follows the trend in velocity, with values up to 11 Pa after the initial 30 m.

Knowledge of the local operating conditions allows the deposition rate to be estimated. Particulate fouling was modeled as a linear sum of the growth rate, which was determined to be constant from examining the work of Young and Leeming (1997), and a removal rate proportional to the shear rate:

$$\frac{\mathrm{d}R_{\mathrm{f}}}{\mathrm{d}t} = \frac{1}{\rho_{\mathrm{f}}\lambda_{\mathrm{f}}} (\dot{m}_{\mathrm{d}} - \dot{m}_{\mathrm{r}}) = \frac{1}{\rho_{\mathrm{f}}\lambda_{\mathrm{f}}} (\dot{m}_{\mathrm{d}} - BfV_{\mathrm{g}}^{2}R_{\mathrm{f}})$$
(1)

which, assuming the material parameters and growth rate are constant, in the steady-state gives,

$$R_{\mathbf{f},\infty} = \frac{B^*}{fV_{\mathbf{g}}^2} \tag{2}$$



Figure 2 Variation of key process parameters along the cooler for the clean case (no deposit). Labels correspond to positions shown in Figure 1. Pressure drop, ΔP_{g} , is that relative to the inlet pressure: τ_{w} , V_{g} , D and T_{g} are scaled by the largest value in each case.

The foulant density, $\rho_{\rm f}$, and voidage was determined by measuring the mass and volume of samples recovered from the cooler at a shut down. The voidage was high, ranging from 75-85%. A thermal conductivity, $\lambda_{\rm f}$, of 0.2 W m⁻¹ K⁻¹ (see later) was initially used in the calculations. The deposition regime is determined by the particle relaxation time, τ_p^+ , defined as:

$$\tau_{\mathbf{p}}^{+} = \frac{1}{18} \frac{\rho_{\mathrm{mat}}}{\rho_{\mathrm{g}}} \left(\frac{d_{\mathbf{p}} V_{\mathrm{g},F}}{\upsilon_{\mathrm{g}}} \right)^{2} \tag{3}$$

where ρ_{mat} is the TiO₂ particle material density, ρ_{g} is the gas density, d_{p} is the particle diameter, $V_{\text{g,F}}$ is the frictional gas velocity and v_{g} is the gas kinematic viscosity.

Figure 3(a) reports the values of τ_p^+ estimated for particles of diameter 60 nm and 1 µm (representing the range of sizes of unagglomerated TiO₂) along the cooler. The local operating conditions are those from the fouled state (see Figure 4).

Whereas the smaller particles are transported to the wall primarily by diffusion, the 600 nm particles are chiefly subject to diffusion-impaction, and the largest particles are subject to diffusion-impaction in some sections and inertial impaction in others. Figure 3(b) shows the effect on the dimensionless mass transfer coefficient, V_{dep}^+ , defined as

$$V_{\rm dep}^{+} = \frac{G_{\rm dep}}{\overline{\rho}_{\rm p} V_{\rm g,F}} \tag{4}$$

The plots indicate that the mass transfer coefficient for all particle sizes is highest at the bends, and differs noticeably between particle sizes in the straight sections. There is thus a trade-off between deposit growth and wall erosion, caused by the sand particles, at the bends. The mass transfer coefficient is higher at the bends as the diameter of the cooler is larger there in order to reduce erosion of the wall by the sand. This promotes TiO₂ deposition but is needed to reduce the rate of wear. Inspection of the bend sections at shut-down showed evidence of non-uniform sand impaction, with regions on the outside of the bend scoured almost completely clean by the sand. Detailed simulations of bend wall scouring by particles in a high speed gas flow have been reported by Njobuenwu and Fairweather (2012). Wear of the bend sections thus sets limits on the design and operation of the cooler.

The steady state fouling resistance can be estimated using Equation (2), which also predicts that an asymptotic fouling layer thickness, denoted ∂^* , can be obtained under certain combinations of operating conditions. Here the combined constant, B^* , is set such that the ∂^* values are consistent with the observation that the fouling layer thickness is approximately 1 mm at the inlet to the cooler. This constant is a lumped parameter, calculated using the mean TiO₂ particle size of 285 nm, with contributions from the growth rate, the strength of the deposit and fouling layer material properties, as well as sand removal. It was not possible to separate out contributions from shear-induced removal and erosion resulting from sand impact.



Figure 3 Estimated (*a*) particle relaxation times, and (*b*) dimensionless deposition velocities for different TiO₂ particle sizes, as well as (*c*) asymptotic fouling layer thickness along the cooler under fouled condition (Figure 4).

The δ^* values estimated for the cooler are reported in Figure 3(*c*). These calculations employed the process model operating as a dynamic simulator to calculate the local temperature, velocity and shear stress at each point, as

deposition upstream modifies the rate of heat transfer *etc*. The deposit thickness varies noticeably with position. δ^* reaches 40 mm at the bends but is present at all positions. The effect on heat transfer is significant, as even a 1 mm thick deposit has a resistance of 0.01 m²K W⁻¹.



Figure 4 Distribution of key operating parameters along the cooler for the fouled case with local deposit thickness. in Figure 3(c). ΔP_g is relative to inlet pressure: τ_w , V_g , D and T_g scaled as before (see Figure 2).

Figure 4 shows the impact of the predicted deposition profile on the cooler operation. In this case fouling cannot be quantified by an overall heat transfer coefficient: the presence of a significant fouling layer immediately downstream of the reactor means that the reaction is not quenched so latent heat release continues over the first 10 m. Both V_g and T_g in section I are therefore greater: the former increases the deposition rate. The presence of the fouling layer and extra heat release means that the gas exit temperature approaches 500 K. This is a key operating parameter as there is an upper temperature limit for the sand removal system.

The predicted initial fouling rates are fast, reaching 0.1 mm s⁻¹ in places. Lister *et al.* (2012) showed that the rates likely to be encountered in these systems (of order 10 μ m s⁻¹) are too fast to be monitored reliably using external devices such as heat flux sensors. Attempts to do this on the plant have confirmed this result: variations in heat flux are observed when sand impacts cause deposit spalling, but the noise levels and short times prevent reliable measurement of fouling rates.

DEPOSIT THERMAL CONDUCTIVITY

The deposit thermal conductivity is an important property. The thermal conductivity of the gas is 0.029 W m⁻¹K⁻¹, while TiO₂ has a value of 3.34 W m⁻¹K⁻¹. The thermal resistance of round, flat (where possible) samples of deposit was measured using the device shown in Figure 5(a). The sample is located between two titanium 6-4 cylinders which serve as heat flux meters. The assembly is insulated and the temperature distribution across the titanium cylinders is measured using a series of thermocouples. Heat is supplied by an electrical band heater mounted in a copper block, with a PID controller set to give a constant temperature. Heat is removed by cooling water passing through a second copper block. A clamping system and metal foils ensure good contact between the deposit and the heat flux meters. Contact resistances can be significant and were measured in separate tests.

Figure 5(*b*) shows an example of a temperature profile obtained after initial transients due to conduction through insulation *etc.* had settled. Measurements were typically taken over two hours, to ensure that transients had been eliminated. The linear temperature profile is used to estimate the heat flux, q, across each cylinder. There is good agreement between the two, and the average is used to estimate the thermal resistance, R_t , from

$$R_{\rm t} = \frac{1}{q} \left(T_{\rm s,upper} - T_{\rm s,lower} \right) \tag{4}$$

where $T_{s,upper}$ - $T_{s,lower}$ is the temperature difference across the sample, obtained by extrapolation (see Figure 5(*b*)).



Figure 5 (*a*) Schematic of the apparatus used to measure deposit thermal resistance, based on Tan *et al.* (2006); (*b*) example of temperature profile obtained for a 0.38 mm thick sample, giving $\lambda = 0.08 \pm 0.02$ Wm⁻¹K⁻¹.

Sixteen different samples of deposit were tested and the results are presented in Figure 6. The scatter in the data is expected for plant samples of uneven shape.

The thermal conductivity was calculated from $R_s = \delta/\lambda + R_{\text{contact}}$, giving $\lambda = 0.26 \pm 0.1$ W m⁻¹K⁻¹. This is significantly larger than the values estimated using the parallel, geometric and Maxwell models for spheres in a continuous matrix for a voidage of 0.8. It can be compared with the Reimann Weber prediction for overlapping (*i.e.* sintered) spheres, *viz*.

$$\lambda = \lambda_{TiO2} \left(\frac{d_p}{2a} + \frac{1}{\pi} \ln \frac{d_p}{a} \right)$$
(4)

where *a* is the radius of contact of spheres of diameter d_p . Putting $\lambda = 0.26$ W m⁻¹K⁻¹ into Equation (4) gives $d_p /2a = 0.1$, which indicates that there is significant overlap between particles. This result is consistent with the deposit being a brittle solid with a voidage of 0.8: the largest voidage expected from a packed bed of particles is noticeably smaller. Separate X-ray microtomography studies indicated the existence of large internal channels within some samples, possibly arising from scouring by sand.



Figure 6 Measured thermal resistances. Solid line shows fit to linear trend line. Dashed lines indicate 90% confidence intervals:

SAND EROSION STUDIES

Initial studies of sand as an erodent were based on standard erosion approaches. A metal ball bearing was dropped on a deposit sample and the tendency to break was recorded. These tests indicated that the deposits were brittle and often fractured completely, which would result in spalling. There was noticeable variation in deposit fracture strength, however, so this property was studied in simpler indentation tests using a Texture Analyser (Stable Microsystems) XT+ device fitted with a 1.03 mm diameter spherical indenter. Deposit samples were mounted on a heated stage and the indenter moved into the sample a distance of 0.5 mm at a speed of 0.1 mm s⁻¹, then withdrawn. Figure 7(*a*) shows an example of the indentation force recorded over time.

There is a noticeable discontinuity in the force-displacement profile at 3.4 s, marked I, corresponding to fracture of the deposit. Integrating the force-displacement data up to this point gives the energy for fracture, $E_{\rm f}$, which can be compared to the kinetic energy of a sand particle striking the layer. Separate tests at different velocity indicated that this parameter was not strongly sensitive to indentation velocity. Other tests measured Young's modulus of the deposits. These varied from 5 to 40 MPa, and decreased slightly with increasing temperature over the range tested (40-200°C).

The fracture energy data are plotted on Figure 7(*b*). There is no consistent variation with temperature. Also plotted on the figure is the kinetic energy of a typical sand particle with density 2650 kg m⁻³ and diameter 0.5 mm at impact

velocities of (*i*) 20 m s⁻¹ and (*ii*) 70 m s⁻¹, representing the exit and highest gas velocities in the cooler, respectively. Most of the $E_{\rm f}$ values lie above the former, which is consistent with the deposits being collected from the cooler after prolonged operation.





Since temperature has little effect on $E_{\rm f}$, the data are presented in the form of Weibull distribution in Figure 7(*c*). There is considerable scatter in the fit. 50% of the layers are expected to fail when the sand particles are able to transfer 0.26 mJ of energy, corresponding to a 0.5 mm particle impacting at a velocity of 55 m s⁻¹. The Weibull parameter in this case is 1.29, indicating a mild ageing effect.

These results indicate that smaller sand particles are not efficient removal agents for this application. Figure 8(*a*) shows the size distribution (in terms of circle equivalent diameter, d_{CE}) for the standard sand used on the plant. There is a noticeable fraction of fines but these do not contribute significantly to the volume (mass) distribution. The fines will have little effect on erosion as their kinetic energy is small. The mean sand size of 0.76 mm would give a kinetic energy of 0.26 mJ at a gas velocity of 15 m s⁻¹, which is close to the gas velocity along the straight sections of the cooler. Particle shape also affects the erosion efficiency: this was studied (Lister, 2014) but is not reported here. The effect of shape and other erodent properties is discussed by Bousser *et al.* (2013).

Reduction in the size of the sand particles as a result of collision will have a detrimental effect on their erosion efficiency. This was studied by measuring the particle size distribution of a batch before and after it was passed through a sand blasting gun (Sealey Model SG10E) located directly above a stainless steel plate. The propellant was air at room temperature and pressure at a velocity of 72 m s⁻¹. This velocity is representative of the conditions at the inlet of the reactor where the sand is introduced. The product distribution in Figure 8(*b*) shows the presence of a second maximum around 0.2-0.3 mm, arising from the products of particle breakage. The volume-based distributions were fitted to a bimodal particle size distribution with a breakage factor *p*,

$$f_{\rm V,total}(d_{\rm CE}) = p f_{\rm V,old}(\mu_{\rm old}, \sigma_{\rm old}) + (1-p) f_{\rm V,total}(\mu_{\rm new}, \sigma_{\rm new})$$
(5)

where μ_{new} and σ_{new} are the mean and standard deviation, respectively, of the log-normal standard distribution fitted to the breakage fragments; μ_{new} , σ_{new} and *p* were identified by the use of maximum likelihood estimators and numerical optimization routines in MATLAB.

Table 1 summarises the results from particle size and breakage analysis of several sands and a glass material being considered as an alternative feedstock. The recycled A material has slightly larger particles – the recovery process will have removed some of the broken fraction – but its breakage factor is twice that of the original material. This indicates that there is limited scope for repeated recycling of the sand. Some blending is therefore employed in practice.

Sands B and D had similar size distributions while C had a wider distribution. All three gave similar breakage to recycled A, confirming sand A to be the preferred erodent.



Figure 8 Sand A size distributions. (*a*) fresh sand, showing number and volume fractions; (*b*) after one pass through the sand blasting test.

Table 1. Particle breakage fraction, p, and size distribution* parameters. Before indicates original material; broken relates to non-fines material generated by sand blasting.

Sand	Before		Broken
	Mean*, s.d.*	р	Mean*, s.d.*
	/mm		/mm
Α	0.76, 0.2	0.08	0.30, 0.2
Recycled A	0.90, 0.1	0.18	0.40, 0.4
В	0.82, 0.2	0.19	0.39, 0.3
C	0.87, 0.5	0.17	0.42, 0.3
D	0.85, 0.2	0.15	0.42, 0.3
Glass	1.00, 0.2	0.33	0.49, 0.4

* Log-mean distribution, volume/mass basis: mean is the geometric average and s.d. is the standard deviation in *ln* (size)

The glass feedstock featured larger particles but noticeably greater breakage with a p value $4 \times$ that of A and $2 \times$ B-D.

This result confirmed its unsuitability for this application: it should be noted that this material originated from bottle recycling and would have provided a 'green' option.

CONCLUSIONS

- 1. Particulate fouling is an acute and unavoidable phenomenon in titanium dioxide plasma manufacturing systems. A process simulation including fouling was constructed which allowed the local operating conditions which control fouling, and its impact on process performance, to be quantified.
- 2. Deposition was dominated by larger TiO₂ particles. Initial deposition was rapid and asymptotic fouling levels were predicted to be large.
- 3. Measurements of deposit thermal conductivity highlighted the role of sintering between particles in these high voidage microstructured materials.
- 4. The addition of sand as an erodent to limit fouling buildup was confirmed as an effective strategy. Erodent particle size is important and the tendency for particles to break on impact was compared between four different sands and a recycled glass product.

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NOMENCLATURE

a	radius of contact, m
B,	lumped constant, fouling model, kg ² m K/s
B*	lumped constant, fouling model, K s ² /W
dp	particle diameter, m
d _{CE}	circle equivalent diameter, m
D^*	dimensionless pipe diameter, -
Ef	fracture energy, J
f	Fanning friction factor, -
fv	frequency distribution by volume, -
Fi	indentation force, N
G_{dep}	deposition mass flux, kg/m ² s
\dot{m}_d	mass deposition flux, kg/ m ² s
m,	mass removal flux, kg/m ² s
Р	pressure, Pa
р	breakage fraction, -
q	heat flux, W/m ²
R	thermal resistance, m ² K/W
R _f	fouling resistance, m ² K/W
R _{f,∞}	asymptotic fouling resistance, m ² K/W
t	time, s
Т	temperature, K
Tg	gas temperature, K
T_g^*	dimensionless gas temperature, -
V _{dep+}	dimensionless deposition velocity, -
Vg	gas mean velocity, m/s
$V_{g,F}$	gas friction velocity, m/s
V_{g}^{*}	dimensionless gas velocity, -
х	distance, m
Z	distance along cooler, m
X _{TiCl4}	conversion of TiCl ₄ , -
δ	deposit thickness, m
ε δ*	asymptotic deposit thickness m
λ	thermal conductivity. W/m K
0	density kg/m ³
r V	dynamic viscosity Pa s
v	moon n c d m
μ	mean, p.s.u, m

- standard deviation, p.s.d. σ
- $\tau_w \\ \tau_w^*$ wall shear stress, Pa
- dimensionless wall shear stress
- $\tau_p{}^+$ particle relaxation time, -

Subscript

g	gas
f	deposit
mat	material