**Process Control of Particle Deposition Systems Using Acoustic and Electrical Response Signals**

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Keywords: Microblast, Spraying, Process Control, Electro-acoustic, Electrostatic, Tribo-charging

The implementation of statistical quality control methods for monitoring and control of powder abrasion/deposition is of increasing importance in a manufacturing environment. For the wider adoption of both current and new powder coating technologies, quality control systems need to be developed, which are easily installed, non-invasive and work in real time. This study evaluates the use of a dual electro-acoustic and electrostatic surface-charge measurement technique as means of realising real-time process control. Simultaneous changes in the signals were obtained under both powder flow -on and -off conditions and also for edge detection of the substrate. It was discovered that the most important variables which governed changes in the acoustic response signal were due to variations in the deposition pressure and stand-off distance, whilst those for the electrostatic response signal came from changes in particle size and deposition stand-off distance. A phenomenological predictive equation was developed based on a two-level full factorial design with five variables for both response factors. The coefficients of determination, *r*2, for the models were 93 and 98%, respectively, with respective χ2 probability values of 99 and 99.5%. This enabled the use of specific limits for any variation of variables amongst those tested to be set up, resulting in the apparatus necessary for the development of a sensitive continuous control system. Examining variations in surface roughness with electrostatic signal was observed to show a linear relationship, increasing at a rate of 0.19 μm per 0.01eV, as the effective particle size of Al2O3 was decreased.

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# Introduction

Continuous feedback process control for particle spraying technologies, such as abrasive shot-peening or particle coating deposition, is an important tool to ensure a high quality end-product and process-reproducibility are achieved [[1](#_ENREF_1), [2](#_ENREF_2)]. These spraying operations are often used in the medical device sector to deposit biologically active coatings and as a result extensive quality process control is required [[3](#_ENREF_3)]. These processes are normally conducted within enclosed chambers, with poor visual inspection capabilities; hence, if a problem arises, operators are often unable to determine any defects until the end of the process. Even if the process is observable, small fluctuations in the process conditions during the operation can significantly affect the overall production effectiveness, leading to substandard end-products and necessary reworks. Although the use of Almen strips is widely used as a quality control technique for these types of powder blasting processes [[4](#_ENREF_4)], they do not provide real time feedback as to whether the process is operating at its optimum functionality. Hence, there is substantial impetus to develop easily incorporated real-time feedback solutions; this forms the motivation for the data-driven approach explored in this study using electro-acoustic and electrostatic signal responses.

Electrostatic particle charging through contact is an important phenomenon which has garnered considerable interest over the last few decades [[5-7](#_ENREF_5)]. This contact charging is often termed tribo-charging and arises from particle-particle and particle-carrier tube interactions, with the charge being transferred due to difference in the work functions of the two materials [[8](#_ENREF_8)]. Tribo-charging is a prominent issue in many industrial sectors which deal with the transportation of particulate material on both large and small scales [[9](#_ENREF_9)]. The charging of particles in pneumatically-fed tubing is known to lead to adhesion of the particulates to the insides of tubing; this in turn can result in blockage and subsequent machinery downtime [[10](#_ENREF_10)]. Furthermore, there is potential for excessive charging of particles to an extent where a discharge could occur that may produce a spark with the potential to initiate an explosion [[11](#_ENREF_11)]. Tribo-charging of particles although problematic in some cases can be advantageous in others, in particular where electrostatic charge is used in applications such as coating deposition systems [[12](#_ENREF_12)] and electrostatic particle separation [[13](#_ENREF_13)].

In this study, the generation of electrostatic charge is used as a means of process control. Previous studies in this area have included work by Michalcioiu et al [[14](#_ENREF_14)], in which particles were charged using a tribo-gun and actively monitored using a Faraday pail and an electrometer connected to a LabVIEW program. The monitoring of electrostatic charge under particle flow conditions has also been examined previously in various guises using both “passive” and “active” charging methods: Masuda *et al* [[9](#_ENREF_9)] for example used a passive method to measure the current produced as the particles impacted on grounded metallic pipes with an electrometer. It was reported that the amount of charge produced was dependent not only on the mass flow rate of the particles, but also on the pipe material and the initial charge on the powder. In contrast, Matsuka *et al* [[15](#_ENREF_15)] actively-charged Al2O3 powder prior to transportation and found that the average charge on a particle increased with pipe length and that the rate of increase of the charge declines gradually with length until the charge reaches an equilibrium state, which is dependent on the material. That study also suggests a means of controlling charge in particle flow situations by alternating pipe material, highlighting the importance of the choice of conveying-pipe material.

In addition, the present study also evaluates an electro-acoustic measurement technique as an alternative and complimentary partner to the electrostatic measurements to monitor the change in deposition variables for the microblast system as a function of process conditions. Electro-acoustic signals are produced by a microphone placed in the vicinity of the process. Acoustic pressure waves created by the process are picked up by the microphone and converted into an electrical signal. Variations in process conditions can be determined through examination of amplitude changes of this signal, at or over a range of specific frequencies. Such electro-acoustic measurements have previously been applied successfully as a means of process monitoring in atmospheric pressure plasmas reel-to-reel systems [[16](#_ENREF_16)] and plasma jets [[17](#_ENREF_17)] and also in the monitoring of mass flow of bulk solids in pneumatic pipelines [[18](#_ENREF_18)]. Albion et al. used the monitoring of acoustic signals along the length of a pneumatically fed system to allow for early detection of particulate build up of fine particles within a metallic pipe [[19](#_ENREF_19)]. Ivantsiv et al. using a set up to accelerate particles similar to the one used in this study, accelerated glass bead of 60 μm and aluminium oxide of 25 μm at the surface of an acoustic emission transducer [[20](#_ENREF_20)]. Mass flow rates were correlated with acoustic signals obtained and variations in flow regimes were actively measured. The numbers of particle impacts on the transducer were also able to be counted; however, the set-up had limitations based on high flow rates. These electro-acoustic studies all involved the monitoring and measurement of powder flow either at the carrier tube or post process.

In this study the aim is to achieve greater particle deposition process control by combining electro-acoustic signals in tandem with electrostatic surface-charge measurements. The investigation is carried out on an abrasion/deposition microblast type system. To the author’s knowledge, this is the first time that the combination of these two measurements has been used for process control measurements. A primary aim of this study is therefore to explore the benefits of using two signal sources, measured in tandem, rather than singularly and to examine which effects dominate each of the measurement responses. A further factor investigated is the sensitivity of these response signals to variations in process parameters. Hence, if one of the process variables is operating outside of its limitations, a critical question is will the other be still able to still pick up variations in the process, thus maintaining a both active and robust control system. Using this dual-measurement approach, the temporal evolution of the process is examined as a function of each process variable. The most common powder abrasion / deposition process variables used in industry are explored in this study, *i.e*, stand-off distance, input pressure, particle size, tube material and substrate type. Key process conditions are identified subsequently, with the goal of creating a phenomenological predictive equation that may be used for process control.

1. **Materials and Methodology**
   1. *The Microblast System*

The microblast process involves the acceleration of micron-sized particles by a dried compressed air source, through a Comco nozzle (type MB2520) using an Accuflo® hopper. This nozzle has a consistent internal diameter with an aperture of 1.5 mm. In addition a custom made de Lavel nozzle was also used, which has a restriction from 2.8 to 2.2 mm within its internal length. These nozzles were affixed using a custom made holder to a Staubli TX40 robotic arm, which controlled the nozzle position in 3 dimensional space. The system’s programmable software ensured precise control of the nozzle movement over the substrate. The standard operating parameters were 20 mm stand-off distance and 620 kPa input pressure using Comco polyurethane tubing; these were used based on previous industrial process conditions [[20](#_ENREF_20)]. All experimental work was performed within an enclosed cabinet at atmospheric pressure and ambient temperature, Fig. 1 (a).

* 1. *Materials*

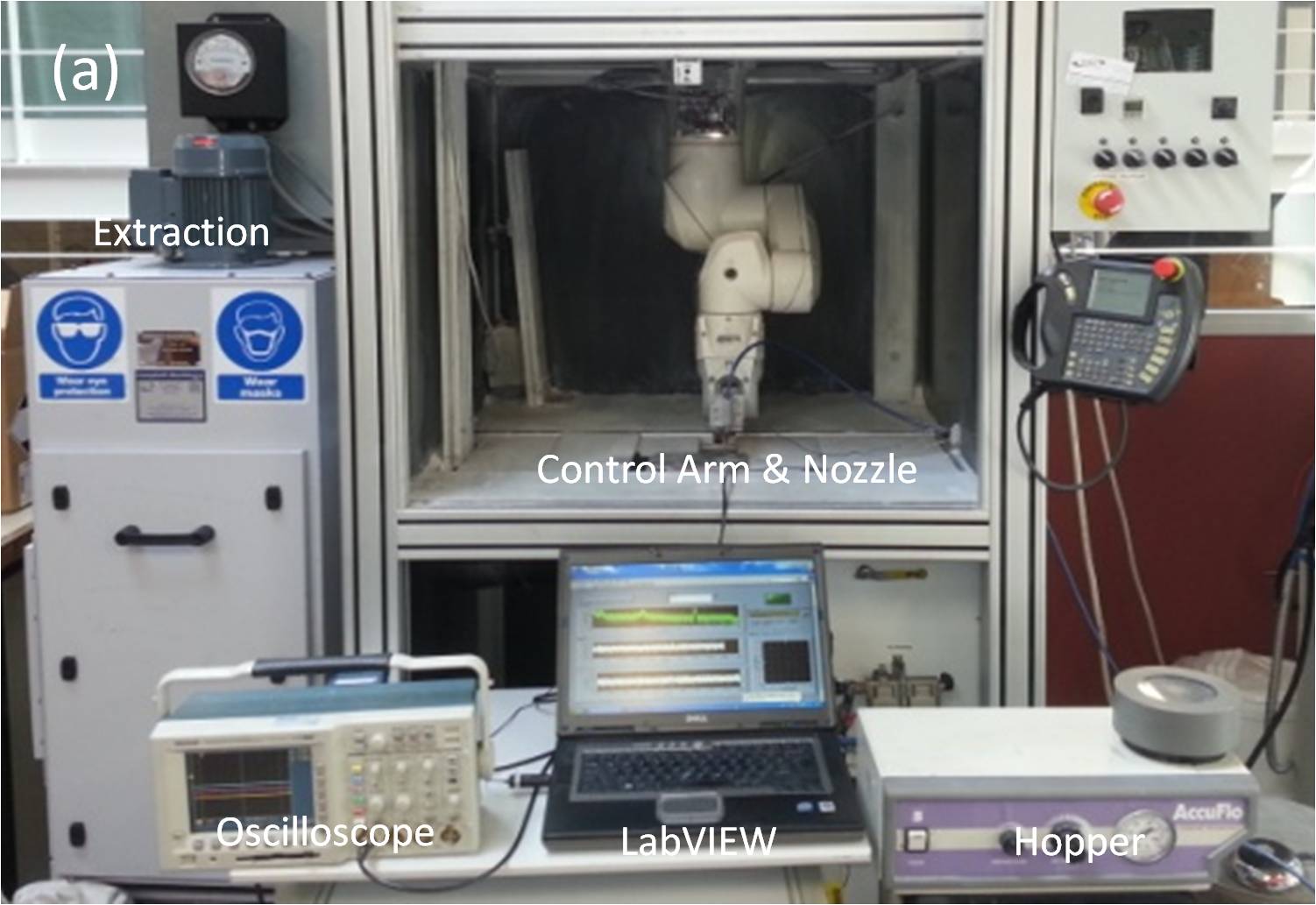
Commercially available aluminium oxide (Al2O3) powder of varying particle sizes (50-500 μm), was used as the main feedstock for the characterisation process of the system. Other powders investigated were Polytetrafluoroethylene (PTFE) (1 μm), agglomerated titanium dioxide (TiO2)nano-particles(40 μm), glass beads (50 μm), silicon carbide (SiC) (50 μm), sodium bicarbonate (50 μm) and hydroxylapatite (HA) (100 μm). Stainless steel (304) of dimensions 155 x 100 x 1 mm was used as the standard substrate for many of the experiments, due to its relatively neutral charge affinity on the tribo-electric series [[21](#_ENREF_21)]. Aluminium, brass and titanium substrates of the same dimensions were also used to examine the effect of altering the substrate type. To study the effect of carrier tube material type between the hopper and nozzle, a number of different tube materials were examined. These were anti-static polyurethane and PTFE tubing as supplied by Radionics, along with an Mb1233 abrasive polyurethane tubing as supplied by Comco.

* 1. *Data Analysis*

Electro-acoustic and electrostatic surface charge measurements were obtained simultaneously using two data collection channels and synchronised using a dedicated National Instruments LabVIEW 2011 program, running on a Dell latitude D830 laptop. The electro-acoustic signal was monitored using an Omni-directional microphone (2428905 Radionics), which has an impedance of 1kΩ at 1 kHz and an acoustic frequency range of 1-18 kHz. The microphone was attached to the deposition nozzle; thus, the measurement reflects the position of the microphone in relation to the substrate. The microphone’s analogue voltage signal was fed into the 1 kΩ impedance jack of the computer’s soundcard where it was converted to a digital signal. The LabVIEW software programme was then used to sample the data stream over a 5- second time period and perform a Fast Fourier Transform (FFT), to present the digital data in the frequency-domain at a resolution of 1 data point per Hz. After a series of iterative experimental measurements, a Savitzky–Golay (SG) digital filter [[17](#_ENREF_17), [22](#_ENREF_22)], with a moving window ranking of ± 3 data points and with a polynomial function (*m* = 1), was chosen to smooth the data by least-squares minimisation. The average amplitude of 500 data points (500 Hz) was used to extract data with the start point set to 10 kHz. This data window was chosen as it was found experimentally to be the portion of the frequency spectra to undergo the greatest change.

The data for the electrostatic measurements was obtained by connecting the central wire of a 50 Ω impedance coaxial cable to the substrate, with the outer ground shield braid left at ground. The resulting analogue electrical signal on the coaxial cable was then passed to Tektronix TDS 3034B oscilloscope, where it was digitised and displayed in the time-domain. The digital signal was also passed via a NI-488 GPIB-USB interface to the same LabVIEW 2011 programme for display and analysis.

The two synchronised measurements were each taken over a 40 second time period, with the initial 20 s undertaken under no powder flow conditions to establish an average reference baseline. The subsequent 20-40 second period was dedicated to a powder flow on condition. Using this approach, both average and standard deviation values for the process were obtained for each measurement run which was repeated at least 6 times. A visual representation of the experimental configuration is shown in Fig. 1 (a) with a schematic of the set up in Fig. 1 (b).



Figure_1.tif

(b)

**Fig 1: Experimental set up (a) with schematic of signal measurement (b); Electric signals from the substrate surface are fed into an oscilloscope prior to input into LabVIEW. Electro-acoustic signals from the microphone are fed directly through the soundcard of the computer to LabVIEW**

* 1. *Design of Experiments*

To understand and evaluate the complex interactions between the process variables a two-level 25 full factorial design of experiments (DOE) strategy was implemented [[23](#_ENREF_23)]. Here the DOE explores the main effects and interactions of five variables: stand-off distance, input process pressure, particle size, tubing and substrate type on both electro-acoustic and electrostatic measurements. Deposition conditions for each of the ‘low’ and ‘high’ levels in the DOE are shown in Table 1. In all cases, robust spacing was chosen between the low and high values for each process variable, in terms of producing very distinct and easily distinguishable differences in both response variables for the successful application of DOE principles [[23](#_ENREF_23)]. Each of the 25 runs was run in random order either 9 or 12 times, with the runs at each condition also taking place non-sequentially to minimise any randomisation issues [[23](#_ENREF_23)]. Following the construction and undertaking of the experiments, an analysis of the variance (ANOVA) of the data was conducted in order to determine the most important factors and interactions for each of the responses [[23](#_ENREF_23)]. Using this information, a predictive linear-regression model was constructed and a thorough examination of the residuals was conducted as a test of the model’s underlying assumptions and validity.

**Table 1: Variables used in the design of experiments along with the value, materials and designations used**

|  |  |  |
| --- | --- | --- |
| Variable | Low (-1) | High (+1) |
| Stand-off Distance | 2 mm | 40 mm |
| Process Pressure | 345 kPa | 690 kPa |
| Particle Size (Al2O3­) | 50 μm | 350 μm |
| Tubing | Polyurethane | Teflon |
| Substrate | Brass | Steel |

1. **Results and Discussion**
   1. *Process feedback*

A preliminary experiment was carried out to determine if the electrostatic and electro-acoustic measurements obtained using the experimental set up shown in Fig. 1 could be used for simple on/off-process control. The results of this experiment are given in Fig. 2. In part (a) of this figure, 100 µm Al2O3 powder was impacted upon a stainless steel substrate using a circular Comco nozzle, from a stand-off distance of 20 mm. It was observed that the electro-acoustic and electrostatic voltage signal exhibited a steady baseline for the initial 20 second ‘off-period’ of the process. Following this, upon commencement of the flow, both signals jump simultaneously in amplitude, indicating a “process-on” scenario. This demonstrated the ability of this measurement technique for on/off-process control. The data in Fig. 2 (b) depicts the electro-acoustic and electrostatic voltage signal results obtained with a mixed Teflon/Al2O3 (50/50) powder feedstock, using the same equipment set-up. This powder mixture was chosen due to its non-flow nature, pre-disposition to block the tubing and for its ease in illustrating the difference between good and bad process conditions. The electrostatic and electro-acoustic signals for the Teflon powder mix exhibit erratic signals during the on-phase, indicative of a blocking of the nozzle due to the intermittent particle flow.



**Fig 2: Process control: Deposition process flow functioning correctly in an off- and on- regime with both electrostatic and electro-acoustic signal responding to Al2O3****powder flow (a), nozzle blockage and flow restriction obtained using the Teflon/Al2O3 (50/50) powder feedstock mixture (b)**

The second fundamental process monitoring scenario investigated was substrate material edge detection. In Fig. 3, both the electro-acoustic and electrostatic signals were observed to react simultaneously to the ‘process off- and on-‘conditions, as observed previously using the 100 µm Al2O3 powder in Fig. 2. In this scenario, however, the deposition stream is moved off the steel substrate after 40 s. At this point the electrostatic measurement drops to the baseline zero-level with the electro-acoustic signal dropping to a lower, but non-zero level (zero value being -140 dB). This is due to the fact that although the powder is no longer impacting upon the solid substrate, it is still emanating from the nozzle and as a result, producing a noise.



**Fig 3: Electro-acoustic and electrostatic voltage signals obtained with the 100 µm Al2O3 powder. The signals obtained demonstrating reaction to On-Off operation and edge-detection**

These preliminary experiments establish proof-of-concept and indicate that a simple on-off process control system could be set in place once any deviation from the baseline reading of either signal response was observed. If no deviation was observed, then the process could be flagged and the operation halted to save valuable materials and machine time. These basic feedback dynamics, although informative, do not infer anything other than as to whether the system is on or off, or if the deposition stream is on the sample. As a result, a further set of experiments was developed to evaluate the behaviour of the electro-acoustic and electrostatic signals, as each of the variables were individually changed, whilst maintaining the other parameters constant. This is often termed a ‘one-factor-at-a-time’ (OFAT) variation and was undertaken before the two-level factorial design, to aid in the choosing of its low and high levels, and to explore any intermediate non-linearity between these extremes of operation. Doing so allows for more precise limits to be imposed for an on-off control regime and thus facilitating a greater understanding of the impact of each deposition variable on the responses.

* 1. *Variation of Individual Process Variables*
     1. *Particle Size*

The effect of varying the abrasive particle size (Al2O3), in the range of 50 to 500 μm, on the electro-acoustic and electrostatic surface charge measurements is shown in Fig. 4 (a) and (b) respectively. Particle size was observed to show an increase in the electro-acoustic signal in the 50 to 350 μm range, with a drop in signal recorded for the largest 500 μm size. This decrease in the amplitude of the signal for the largest powder size could be due to slower impact velocity of these particles and hence a reduced signal. A decreasing trend in the electrostatic signal was noted as the particle size of the feedstock was increased. The larger particles induce a smaller amount of charge on the surface of the substrate. This could primarily be due to the larger particles’ higher mass, leading to a lower substrate impact velocity when compared to particles of smaller mass. This lower velocity may not have the required energy to overcome the work function of the substrate to release an electron. An alternative explanation could be that due to the lower velocity of the larger particles, interaction time between them and the surrounding atmosphere increases as they travel from the nozzle to the substrate and lose any charge they have gained.



**Fig 4: Influence of particle size (50-500 μm Al2O3) upon the measured electro-acoustic (a) and electrostatic voltage (b) signals**

Although the size of the deposition particles is of interest in numerous applications in particular coatings for solar applications [[24](#_ENREF_24)] or for control of surface roughness [[25](#_ENREF_25)], two other common deposition variables easily changed in the majority of powder-blasting processes are the process pressure and stand-off distance. The results of the variation of these process parameters are shown in Figs. 5 and 6, respectively.

* + 1. *Process Pressure*

In Fig. 5 (a), the effect of systematically varying the process pressure on the electro-acoustic signal is examined. An increase in the signal was observed up until a value of 483 kPa, beyond which there is a levelling-off of the signal. Similarly, in Fig. 5 (b), a near-linear increase in the electrostatic voltage signal was observed from 207 to 483 kPa, after which a levelling-out of the signal was observed. The process pressure is linked inherently to the velocity of the particles in the deposition stream and hence the shape of the graph can be explained from a particle velocity viewpoint. Above 483 Pa, the particles have potentially reached their maximum velocity and hence upon impact with the surface no difference beyond the standard deviation of the signals was observed. As expected, due to sound being a pressure wave, an increase in the amplitude of the electro-acoustic signal was observed as the process pressure was increased in the system.



**Fig 5: Influence of process pressure upon the electro-acoustic (a) and electrostatic voltage (b) signals**

* + 1. *Process Stand-off Distance*

Studies involving variation in the stand-off distance are shown in Fig. 6, with the electro-acoustic results in panel (a) and electrostatic voltage measurements in (b). Both graphs show similar trends between 20 - 60 mm substrate-nozzle distances with a slight drop-off in the electro-acoustic signal below this and an increase in the electrostatic measurements. Due to the 90º angle to the surface angle of deposition, particles rebound off the surface readily and impact off the nozzle at low stand-off distances. This could give rise to the decrease in the electro-acoustic signal and an increase in the standard deviation of the electrostatic measurements. Both signals also drop off as the stand-off distance increases. In the case of the electro-acoustic measurements, the signal drops off due to the microphone residing further away from the particle impact zone. For the electrostatic measurement, the increased distance between the nozzle and the substrate gives the particles enough time to dissipate their charge between themselves and the environment and a reduction in the signal is observed at the substrate.



**Fig 6: Influence of the nozzle to substrate stand-off distance on the electro-acoustic (a) and electrostatic voltage signal (b)**

* + 1. *Substrate Type*

The effect of substrate type on both the electro-acoustic and electrostatic signals were evaluated. In addition to steel the substrates evaluated were aluminium, brass and titanium. As before, Al2O3 particles of 100 µm where fed through standard Comco polyurethane tubing at 620 kPa, at a distance of 20 mm from the surface. The signal obtained was observed to vary depending on the substrate material. The higher the electrical resistivity of the substrate material, the lower the associated electrostatic voltage measurement from the surface of the substrate was recorded. This could potentially be due to a greater restriction to the flow of electrons in the material to the measuring setup. For the materials with lower resistivity, such as aluminium and brass, the charge travels with greater ease in the material and hence a larger signal is recorded. No substantial variation in the electro acoustic signal beyond standard deviation was observed for any of the substrate materials tested.

**Table 2: Variation in the electrostatic and electro-acoustic measurements with respect to the substrate material**

|  |  |  |  |
| --- | --- | --- | --- |
| Al2O3 Particles  On Substrate | Electrostatic Voltage  (V) | Electro-acoustic signal  (dB) | Electrical Resistivity  (X10-8Ωm) [[26](#_ENREF_26)] |
| Aluminium | 0.17 ± 0.04 | 52.36 ± 0.66 | 2.8 |
| Brass | 0.14 ± 0.01 | 52.09 ± 3.21 | 6.4 |
| Titanium | 0.09 ± 0.01 | 54.88 ± 2.69 | 42.0 |
| Steel | 0.08 ± 0.01 | 52.60 ± 1.97 | 95.0 |

* + 1. *Particle Type*

In industrial deployment, not only does the type of substrate vary, but attention has to be afforded to the type of material used for the abrading/coating process. Hence, in Table 3, a selection of commonly used abrasives (Al2O3, glass bead, SiC, sodium bicarbonate) and coating materials (TiO2, HA-Al2O3 and PTFE-Al2O3 mix), were explored. Using the standard process conditions outlined previously, the feedstock powder was varied. Within the table, the particle sizes of each of the powders is also indicated as variation of this parameter as seen in Fig. 4 has a substantial effect on the response signals. On examination of the results obtained from the 50 μm particle size, abrasive materials display little variation beyond the standard deviation for the electro-acoustic response signal. There was, however, visible variation in the electrostatic signals with the insulating glass bead abrasive exhibiting a much lower signal, than that obtained for the more conductive Al2O3 and SiC powders. The powders used to deposit coatings show variations in both the electro-acoustic and electrostatic signal response. It was observed that the softer powders, TiO2 and PTFE-Al2O3 mix, had a lower electro-acoustic signal, when compared to that of the harder Al2O3 abrasive and Ha-Al2O3 mix. The softer powder dampens the acoustic signal leading to a lower measured value. The TiO2 powder used was agglomerated nano-particles with a primary particle size of 40 nm. During the deposition process these particles have a tendency to fragment [[24](#_ENREF_24)] and as a result there will be a greater degree of interaction between the nano-sized particulates and the walls of the tubing resulting in a higher electrostatic signal when compared to the other coating media. The PTFE-Al2O3 and the HA-Al2O3 powder mix resulted in a smaller electrostatic signal than the TiO2, but higher than that of the pure Al2O3 powder. Variations in the signal can as a result either be attributed to changes in the material properties of the powder or the slight variation in the particle size. The materials could not be tested individually outside of the mix with this system due to the non-flow nature of both the PTFE and HA.

**Table 3: Variation in the electrostatic and electro acoustic measurements with respect to the feedstock material**

|  |  |  |  |
| --- | --- | --- | --- |
| Particle Type on  Steel Substrate | Mean Particle Size  (μm) | Electrostatic Voltage  (V) | Electro-acoustic signal  (dB) |
| Abrasive Media |  |  |  |
| Al2O3 | 50 | 0.15 ± 0.02 | 48.24 ± 1.02 |
| Glass Bead | 50 | 0.04 ± 0.02 | 46.69 ± 0.82 |
| Silicon Carbide | 50 | 0.13 ± 0.02 | 46.91 ± 0.75 |
| Sodium Bicarbonate | 50 | 0.12 ±0.01 | 49.78 ±0.50 |
| Al2O3 | 100 | 0.08 ± 0.01 | 52.66 ± 1.94 |
| Coating Media |  |  |  |
| TiO2 | 40 | 1.36 ±0.06 | 42.60 ± 1.82 |
| Al2O3-PTFE mix | 100-1 | 0.57 ± 0.01 | 45.94 ± 1.25 |
| Al2O3-Hydroxyapaptite mix | 100-50 | 0.30 ±0.10 | 52.28 ±1.45 |

* + 1. *Tubing Type*

As mentioned previously, the particles in the microblast system are charged not only through particle-particle interactions, but also through particle-tube interactions. Hence, the type of tubing used to carry the powder is of vital importance in the creation of charge in the system, Table 4. It can be observed clearly that the anti-static tubing carries out its function correctly, removing the majority of the charge from the powder before it reaches the surface, with a very small voltage being registered by the detection system. In contrast, if PTFE, a highly electronegative material is used, the measurement at the surface is some 5 times larger than that of the standard polyurethane tubing. It is to be noted that the same length and diameter of tubing was used in all tests, as differing lengths will induce varying degrees of charging [[15](#_ENREF_15)] and different diameters affecting pressure differentials. The standard Comco tubing for this system seems to be midway between the two extremes but still allowing a significant amount of charge to build up on the flowing powder. No variation in the electro-acoustic signal was noted for the varying tube types, this was as expected as tubing type should not affect flow dynamics or particle velocity.

**Table 4: Variation in the electrostatic and electro-acoustic measurements with respect to the carrier tubing material, 100 μm Al2O3 was used as the feedstock**

|  |  |  |
| --- | --- | --- |
| Tubing Type | Electrostatic Voltage  (V) | Electro-acoustic signal  (dB) |
| PTFE | 0.40 ±0.03 | 52.25 ± 0.93 |
| Polyurethane | 0.08 ± 0.01 | 52.60 ± 1.97 |
| Anti static | 0.01 ± 0.002 | 51.70 ± 2.14 |

* + 1. *Nozzle Type*

The choice of nozzle is also an important variable when undergoing any abrasion/deposition process for control and efficiency purposes. In Fig. 7 the Comco nozzle and a custom-made de Lavel nozzle investigated in this study are compared using an experiment in which the process pressure was varied. These two nozzles are both constructed from tungsten carbide, but of differing lengths. The de Lavel nozzle due to a constriction in the flow chamber induces a pressure differential accelerating the particles to a higher velocity, when compared to a standard straight-through variant. The electro-acoustic signature in panel (a) of Fig. 7 was noted to differ between both nozzles with the Comco nozzle producing a larger acoustic signature for each of the pressure settings. From panel (b) in Fig. 7, it was observed that the de Lavel nozzle induced a lower charge on the surface of the substrate when compared to its Comco counterpart. This could potentially be attributed to the variation in the lengths of the nozzles, with increased amount of charge dissipation to the tungsten carbide from the particles occurring over the length of the nozzles. The de Lavel nozzle is roughly five times longer than the Comco nozzle with a 4 fold reduction in the electrostatic signal observed.



**Fig 7: Effect of varying of nozzle type with process pressure. Electrostatic measurements (a); electro-acoustic measurements (b)**

* 1. *Interaction effects and proposed model for process control*

Upon completion of the OFAT experiments, the DOE experimental design was undertaken and an ANOVA statistical analysis was conducted using Minitab for both of the electro-acoustic and electrostatic response signals. The main-factor and interaction effect coefficients were computed and together with the plotting of normal probability plots, and important factors were selected for incorporation into their representative regression equations [[23](#_ENREF_23)]. Normal probability plots are when the experimentally obtained results are plotted against a theoretically defined normal distribution to create a linear relationship. If any of the points lie significantly outside this linear relationship, they are deemed to have the greatest influence on, in this case, the response signals.

Fig. 8 shows the effect (%) on the process that each of the variables has on both the electro-acoustic and electrostatic signal, as determined from examination of the contribution to the sum of squares in ANOVA. These vary considerably for each of the responses measured. For the electro-acoustic response, the process pressure was found to have the biggest influence for variations in signal followed by stand-off distance and particle size, with tubing and substrate type showing little effect when compared to the other variables. This is consistent with results obtained from analysis of the OFAT experiments. Although process pressure was the most influential variable for the variation in the acoustic signal, this is not the case for the electrostatic signal. This response was dominated by the particle size of the deposition powder, followed by tubing type, pressure and stand-off distance. This makes the use of the two response signals in tandem an attractive prospect as they are most sensitive to two very different process variables. If, for instance, an insulating powder of a large particle size is used in a process, the electrostatic response may be low and un-measureable, but the electro-acoustic signal will be active and measurable, keeping the control system’s operation viable. The substrate type, although seen to have an effect on the electrostatic signal in the individual study, was not observed to have a significant contribution in the DOE, when compared to the effects of the other variables.



**Fig 8: Effect (%) of each variable on the response signal for both electro-acoustic and electrostatic measurements**

By examining the effects of the main process parameters and the inter-parameter interactions, normal probability graphs were constructed; Fig. 9. The most important factors were determined visually by noting any outliers from the central linear grouping and numerically through the examination of the relative contributions to the sum of squares in the ANOVA analysis. The numerical values for the coefficients of the factors labelled in Fig.9 are listed in Table 5.



**Fig 9: Normal probability plots for electro-acoustic (a) and electrostatic (b) results with the most significant factors labelled**

**Table 5: List of factor identifiers and coefficient values for equations 1 & 2**

|  |  |  |  |
| --- | --- | --- | --- |
| Label | Factor | Electro-acoustic Coefficient Eqn 1 | Electrostatic  Coefficient Eqn 2 |
| Single Factor Interactions | |  |  |
| A | Stand-off Distance | 1.6603 (A1) | -0.01835 (E1) |
| B | Pressure | 3.6197 (A2) | 0.03698 (E2) |
| C | Particle Size | 1.3341 (A3­) | -0.06377 (E3) |
| D | Tubing | -------- | 0.04035 (E4) |
| Two Factor Interactions | |  |  |
| AB | Stand-off Distance \* Pressure | 1.0816 (A4) | -------- |
| DE | Tubing \* Substrate | -0.5897 (A5) | -------- |
| CD | Particle size \* Tubing | -------- | -0.03906 (E5) |
| BC | Pressure \* Particle Size | -------- | -0.03517 (E6) |
| AC | Stand-off Distance \* Particle Size | -------- | 0.01437 (E7) |
| BD | Pressure \* Tubing | -------- | 0.01333 (E8) |
| Three Factor Interactions | |  |  |
| BCE | Pressure\*Particle Size\*Substrate | -0.3509 (A6) | -------- |
| BDE | Pressure\*Tubing\*Substrate | -0.3022 (A7) | -------- |
| BCD | Pressure\*Particle Size\*Tubing | -------- | -0.0136 (E9) |

Using the data obtained, linear-regression predictive models for both of the response variables was developed, equations 1 & 2 [[27](#_ENREF_27)]. For both response variables each factor (A-E) was denoted with a value of either -1 or 1 in the equation depending on whether the low or high setting, respectively, was used, Table 1.The coefficient value for each of the interactions Ax and Ex can be found in Table 5.

***Equation 1***

***Equation 2***

Using equations 1 and 2, predictions of the expected experimental values for both electro-acoustic and electrostatic signals were calculated and compared with the experimentally measured results. Figs 10 and 11 show respectively the experimental values for the electro-acoustic and electrostatic signals for each of the 25 runs of the DOE, along with their associated standard deviations denoted as error bars. Superimposed upon these results is the predicted value for each of the runs determined from eqns. 1, 2. For the electro-acoustic measurements, a coefficient of determination, *r*2, of 93 % and a χ2 test certainty of 99 % was achieved [[28](#_ENREF_28)]. Under these conditions, a rejection of the null hypothesis (H0) was obtained when comparing the experimental to the predicted values, on a χ2 test with 25-1-8 = 23 degrees of freedom (d.o.f.) [[29](#_ENREF_29)]. Similarly for the electrostatic signals a *r*2 value of 98% was obtained with a χ2 test confidence level in rejecting H0 of over 99.5% confidence on a test with 25-1-9 = 22 d.o.f. The presence of at least 9 separate measurements for each run means that the application of a χ2-test is reasonable, given that around a half-dozen measurements is generally regarded as being needed for each observed value to correspond to the expected one (in this case, that predicted by the model-regression expression, (eqns. 1 and 2). As can be observed, the majority of the predicted values fall within one standard deviation of the experimentally-measured averaged-values. Indeed, this is as one would expect, given that the underlying assumption of normality would be consistent with around two-thirds of values being within one standard deviation [[29](#_ENREF_29)]. It should also be noted that the models produced in equations 1 and 2 are based on a linear relationship between the two extremes of the DOE. From the OFAT experimental work conducted, this relationship is known not to be entirely accurate as some of the variables exhibit slightly off linear relationships. Hence, some of the discrepancies in the predicted results can also be accounted by this simplification. On examination of the residuals, it was found that there was no ordered pattern, and that they were randomly-distributed. Therefore there is potential for an easily-implemented data driven model-based control strategy emerges based on this DOE-determined regression model.



**Fig 10: Experimental values for the electro-acoustic signals respectively for each of the 25 runs of the DOE, along with their associated standard deviations denoted as error bars.**



**Fig 11: Experimental values for the electrostatic signals respectively for each of the 25 runs of the DOE, along with their associated standard deviations denoted as error bars.**

The data within Fig. 12 (a) and (b) represent plots of the experimentally calculated values of the electro-acoustic and electrostatic signals against their theoretically calculated counterparts for each of the 25 runs of the DOE. Both these plots indicate good degrees of fit between the two for both signals with the electro-acoustic and electrostatic signals achieving the aforementioned r2 values of 93% and 98% respectively.



**Fig 12: Plots of experimentally measured values of the electro-acoustic (a) and electrostatic signals (b) against their predicted counterparts.**

* 1. **Process control of Surface Roughness**

Another potential application for the electrostatic response signal lies is in the indirect measurement of the roughness of a substrate post abrasion. It is often necessary, post process, to carry out a profilometry measurement on the ablated surface using either an optical or mechanical process in order to determine the surface roughness. This is often a time consuming, but nonetheless necessary, quality control check. In this study two differing particle sizes of Al2O3 (50 and 150 μm)were mixed in varying ratios in order to change the effective mean particle size of the mixture Fig. 13 (a). A linear increase in both the abraded steel surface roughness (Ra), measured using optical profilometry as well as the electrostatic voltage against powder ratio was observed. A plot of the average roughness against the electrostatic voltage indicates a further linear relationship Fig. 13 (b) from which we can determine from the slope that the surface roughness increases a rate of 0.19 μm per 0.01 V. Hence, when considering variation in particle size for the experimental system examined here, all electrostatic measurements can be deemed have an associated surface roughness. The electro-acoustic signal was not observed to vary beyond an appreciably distinguishable level for the mid-range powder mixes, and hence is not included here for further discussion.



**Fig 13: Plots of electrostatic voltage and experimentally measured surface roughness values against powder-ratio mixtures (150-50 μm Al2O3) (a) and a linear relationship between average roughness of substrate and electrostatic voltage.**

1. **Conclusions**

A statistically significant process control technique for the particle-spraying microblast system, in which both electro-acoustic and electrostatic signals play central roles, has been outlined and developed successfully. Simultaneous changes in the signals obtained from the two measurement techniques were obtained in both flow-on and -off conditions, as well as for sample edge detection. All of the process variables were seen to have some degree of an effect on the acoustic and electrostatic response as they were changed systematically, whilst the others were kept constant. It was determined that the most important variables governing changes in the acoustic signal emanate from changes in the process pressure and stand-off distance, whilst those for the electrostatic signal arose from variations in particle size and stand-off distance. As a result, the use of the two different response signals in tandem is much more beneficial to the user than a single feedback as each are affected to greater degrees by different process variables. Linear regression predictive equations were developed for both responses based on two-level full factorial designs. These equations enable the use of specific limits for any variable variation amongst those tested to be established, resulting in ample scope to develop a sensitive continuous control system. Finally, a potential link between the measured surface roughness and electrostatic signal was explored as an indirect means of measuring the surface roughness with a rise of the average roughness of 0.19 μm per 0.01V observed.

**Acknowledgements**

This work was supported by the Precision Strategic Research Cluster Grant No.08/SRC/I1411. Kevin McDonnell and Niall English would also like to acknowledge support under Science Foundation Ireland (SFI) Research Frontiers Programme (reference No. 10/RFP/MTR2868). The authors would also like to thank EnBIO Ltd for their support and access to equipment and materials.

# References

[1] L. Wagner, Shot Peening, Wiley. com, 2003.

[2] M. Awais, D. Dini, J.M. Don MacElroy, Y. Halpin, J.G. Vos, D.P. Dowling, Electrochemical characterization of NiO electrodes deposited via a scalable powder microblasting technique, Journal of Electroanalytical Chemistry, 689 (2013) 185-192.

[3] J.N. Barry, B. Twomey, A. Cowley, L. O'Neill, P.J. McNally, D.P. Dowling, Evaluation and comparison of hydroxyapatite coatings deposited using both thermal and non-thermal techniques, Surface and Coatings Technology, 226 (2013) 82-91.

[4] H. Fuchs, Defects and virtues of the Almen intensity scale, in: Proceedings of the 2nd International Conference of Shot Peening ICSP, 1984, pp. 74-78.

[5] B. Gady, D. Schleef, R. Reifenberger, D. Rimai, L. DeMejo, Identification of electrostatic and van der Waals interaction forces between a micrometer-size sphere and a flat substrate, Physical Review B, 53 (1996) 8065.

[6] P.M. Ireland, Triboelectrification of particulate flows on surfaces: Part I — Experiments, Powder Technology, 198 (2010) 189-198.

[7] A. Nesterov, F. Löffler, K. König, U. Trunk, K. Leibe, T. Felgenhauer, F.R. Bischoff, F. Breitling, V. Lindenstruth, V. Stadler, M. Hausmann, Measurement of triboelectric charging of moving micro particles by means of an inductive cylindrical probe, Journal of Physics D: Applied Physics, 40 (2007) 6115.

[8] S. Matsusaka, Control of particle tribocharging, KONA Powder Part. J, 29 (2011) 27-38.

[9] H. Masuda, S. Matsusaka, S. Nagatani, Measurements of powder flow rate in gas-solids pipe flow based on the static electrification of particles, Advanced Powder Technology, 5 (1994) 241-254.

[10] F.-J. Wang, J.-X. Zhu, J. Beeckmans, Pressure gradient and particle adhesion in the pneumatic transport of cohesive fine powders, International journal of multiphase flow, 26 (2000) 245-265.

[11] M. Glor, Ignition hazard due to static electricity in particulate processes, Powder Technology, 135 (2003) 223-233.

[12] W. Kleber, B. Makin, TRIBOELECTRIC POWDER COATING: A PRACTICAL APPROACH FOR INDUSTRIAL USE, Particulate science and technology, 16 (1998) 43-53.

[13] J.R. Mountain, M.K. Mazumder, R.A. Sims, D.L. Wankum, T. Chasser, P.H. Pettit Jr, Triboelectric charging of polymer powders in fluidization and transport processes, Industry Applications, IEEE Transactions on, 37 (2001) 778-784.

[14] A. Mihalcioiu, L. Dascalescu, S. Das, K. Medles, R. Munteanu, Virtual instrument for statistic control of powder tribo-charging processes, Journal of Electrostatics, 63 (2005) 565-570.

[15] S. Matsusaka, M. Oki, H. Masuda, Control of electrostatic charge on particles by impact charging, Advanced Powder Technology, 18 (2007) 229-244.

[16] J. Tynan, V.J. Law, P. Ward, A.M. Hynes, J. Cullen, G. Byrne, S. Daniels, D.P. Dowling, Comparison of pilot and industrial scale atmospheric pressure glow discharge systems including a novel electro-acoustic technique for process monitoring, Plasma Sources Science and Technology, 19 (2010) 015015.

[17] V.J. Law, F.T.O. Neill, D.P. Dowling, Evaluation of the sensitivity of electro-acoustic measurements for process monitoring and control of an atmospheric pressure plasma jet system, Plasma Sources Science and Technology, 20 (2011) 035024.

[18] Y. Yong, Mass flow measurement of bulk solids in pneumatic pipelines, Measurement Science and Technology, 7 (1996) 1687.

[19] K. Albion, L. Briens, C. Briens, F. Berruti, Flow regime determination in horizontal hydrotransport using non-intrusive acoustic probes, The Canadian Journal of Chemical Engineering, 86 (2008) 989-1000.

[20] V. Ivantsiv, J. Spelt, M. Papini, Mass flow rate measurement in abrasive jets using acoustic emission, Measurement Science and Technology, 20 (2009) 095402.

[21] C.K. Adams, Nature's electricity, Tab Books, 1987.

[22] A. Savitzky, M.J.E. Golay, Smoothing and Differentiation of Data by Simplified Least Squares Procedures, Analytical Chemistry, 36 (1964) 1627-1639.

[23] D.C. Montgomery, Design and analysis of experiments, Wiley New York, 1984.

[24] K.A. McDonnell, N.J. English, C.P. Stallard, M. Rahman, D.P. Dowling, Fabrication of nano-structured TiO2 coatings using a microblast deposition technique, Applied Surface Science, 275 (2013) 316-323.

[25] C. Aparicio, F. Javier Gil, C. Fonseca, M. Barbosa, J.A. Planell, Corrosion behaviour of commercially pure titanium shot blasted with different materials and sizes of shot particles for dental implant applications, Biomaterials, 24 (2003) 263-273.

[26] G.W.C. Kaye, T.H. Laby, Kaye & Laby Tables of Physical & Chemical Constants, National Physical Laboratory, 2011.

[27] J. Neter, W. Wasserman, M.H. Kutner, Applied linear statistical models, Irwin Chicago, 1996.

[28] D.V. Lindley, New Cambridge statistical tables, Cambridge University Press, 1995.

[29] R.V. Hogg, A. Craig, Introduction to mathematical statistics, (1994).