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Guidelines for the Scale-Up of an Aqueous Ceramic Process: a Case Study of Statistical Process Control

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Abstract
Process-scale up is the change from a feasibility study in a laboratory to a full-scale prototype production process. It is an important issue for the ceramics industry, but has been the subject of relatively little systematic research. This paper will show how certain manufacturing concepts - used in a number of industries - can be applied to the scale up of a feasibility study level, aqueous tape casting process. In particular, it examines the elements of process standardisation using the example of the Statistical Process Control (SPC) technique applied to a sub process of an aqueous powder process. It illustrates that these elements are an essential step in moving from conceptual design to the process prototype.

Introduction

The design of any new process develops through four main stages which are widely described in the existing literature [1, 2]. Before the scale up stage, the process needs to be attuned to meet the final requirements (specifications), within a narrow interval of deviation (tolerance). Variation in processes causes waste and consequently increases costs. Whilst this variation is intrinsic in every production process, it can be minimised. The methodology to reach this target is to develop the design into a regime of 'Total Quality'.
The methodology to improve design is based on the use of statistical methods for decision making. The Statistical Process Control (SPC) technique has become a standard tool in many manufacturing sectors to implement, analyse, assess and control process variation, since its application has been proved to enhance problems and reduce costs [3, 4, 5, 6]. It is mainly used to control industrial process variation during production. However, the application of control charts provides a simple yet powerful tool for presenting and studying results in process scale up. For this reason, SPC has been chosen to help in the passage from the conceptual design phase to the definitive design. This technique can be applied either to entire processes or to parts of them. SPC has four main stages: the recognition of process variables, the identification of which process parameters should be monitored, the collection and analysis of the process data and capabilities and finally the control of the process through charts.
This paper presents an application of such a technique to an aqueous ceramic process. The SPC procedure was applied to a sub-process activity on a laboratory scale, as a case study to show an approach to the problem of defining the capability of a process to meet the requirements, and to defining the settings and controls for an operation.

Method

Navarro et al. developed an aqueous laboratory tape casting process for use with PZT powders. The experimental conditions developed for this process are described elsewhere [7, 8].
A study [9] assessed the warm pressing technique as a suitable method for the lamination of green tape. A side-effect of this process is an increase in the green density of tape cast ceramic up to 65% of the theoretical density, improving the quality of the final sintered microstructure. A green density of (5.00 ± 0.1) g/cm^3 (60-62% of the theoretical density) was decided to be the optimum compromise for a satisfactory sintered microstructure, since 65% was considered very close to a maximum random packing factor [10]. These values were taken as the process specifications and

tolerances. Formally, for ± 0.1 g/cm^3 the upper and lower specification limits were: USL= 5.1 g/cm^3 and LSL = 4.9 g/cm^3. Hence, the specifications tolerance limit was 0.2 g/cm^3.

A G.E.Moore warm press was used. The pressing settings were 50 MPa for 20 minutes at 50 ^{o}C. Pressings were performed in a stack of 5 (n); the tapes were separated by a polypropylene sheet to avoid lamination, so that individual layers could be analysed. The experiment was performed through 4 consecutive days and each day the pressure settings were re-inputted.

The sample thickness was taken as the process parameter to be monitored. 100 samples of 16 cm^2 area were pressed (s = 20 stacks). The thickness was measured using a micrometer gauge with a tolerance of 5 μm. The density of each sample was calculated by measuring the dimensions (area and thickness) and mass. The pressed density was used as the variable for the process charts. Basic tools of the SPC technique (such as individual observation, mean and range charts) were used to plot the green density figures. The data were assumed to approximate a normal distribution. The statistics were analysed and compared with the specifications and the required tolerances.

Results and Discussion

Fig.1 shows the individual observations of sample density together with the upper and lower control (at 3σ from the mean line) and warning limit lines (at 2σ from the mean line). The pressed green density of a single tape varied between 4.4 and 5.0 g/cm^3. The average (mean) was 4.70 g/cm^3 while the standard deviation (σ) was 0.15.

The means and ranges of the sample densities were recorded for each stack. The relevant mean ($\bar{\bar{X}}$) and range (R) charts were consequently produced with the calculated values of the mean of the means ($\bar{\bar{X}}$) and the mean of the range ($\bar{R}$) of the stacks. The UCL and LCL lines were placed respectively at $3\sigma\,(n)^{-1/2}$ above and below the average values of the mean of the means ($\bar{\bar{X}}$) and the mean of the range ($\bar{R}$) as shown in Fig. 2. Table 1 reports the values of the main figures used for the analysis.

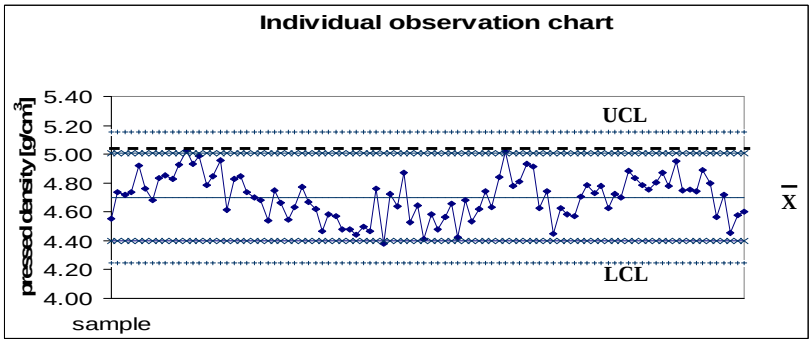
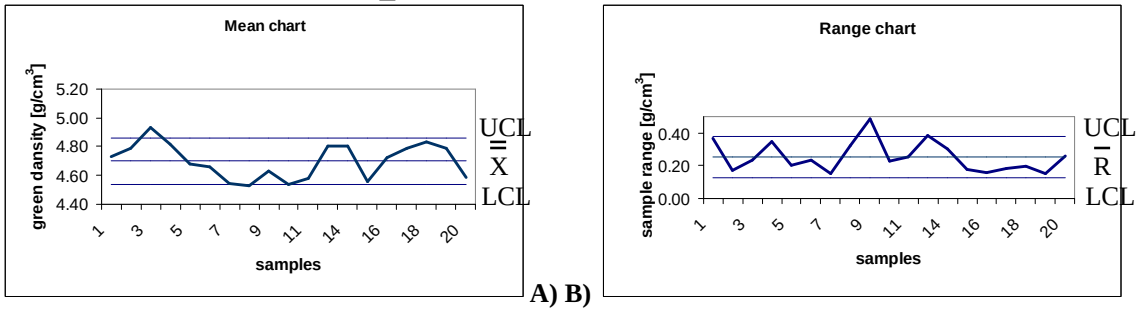


Fig 1: Individual observation chart: upper and lower control limits (UCL, LCL) lines, mean line $\bar{\bar{X}}$, and specification line
Light dots: UCL and LCL
Uniform: $\bar{\bar{X}}$
Heavy Dots: Warning lines
Dashed: Specification

UCL($\bar{\bar{X}}$)	[g/cm^3]	4.86	**UCL R**	[g/cm^3]	0.38
LCL($\bar{\bar{X}}$)	[g/cm^3]	4.54	**LCL R**	[g/cm^3]	0.13
$\bar{\bar{X}}$	[g/cm^3]	4.70	**$\bar{R}$**	[g/cm^3]	0.25

Table 1: Calculated values for UCL, LCL, and means for the mean and range charts



A) B)

Fig 2: A) Mean chart with calculated UCL, LCL and mean $\bar{\bar{X}}$ lines
B) Range chart with calculated UCL, LCL and Mean $\bar{R}$ lines

The individual observation chart (Fig. 1) gives the first indication of process control. It is not, however, as accurate an instrument as the mean and range charts are to perceive small variations in

the mean. The individual observation chart indicates that no values were out of the LCL to UCL range. However, in some circumstances densities were found outside of the 2σ warning lines. Moreover, rows of points (greater than 8 in succession) were located on the same side of the mean. These observations are the first indicators of an 'uncontrolled' process. Figs. 2A and 2B confirm this. The mean density and range show sometimes scatter outside of the UCL to LCL range. Five or six points in succession were observed with either consecutive increasing or decreasing trends.
It can be noted that the specification value of green density, 5.00 g/cm^3, was rarely achieved. In fact, this value was the highest density attained. Useful indexes to compare the process results with the specifications are Cpk_u defined as $(USL-\bar{\bar{X}})/3\sigma$, and Cpk_l defined as $(LSL-\bar{\bar{X}})/3\sigma$. For the studied process Cpk_u was 0.889 and Cpk_l as 0.44. Both of the values are far below 1, which indicates that the process is not capable at any time to achieve the requirements. An easy to obtain measure of the process 'capability' is Cp defined as *(specifications tolerance interval)/6σ*. This index compares the variability of the process (the interval 6σ) with the tolerance of the specifications. For the process analysed in this work, Cp was 0.44, so the variability of the process is greater than the specifications tolerance interval.
These remarks lead to the conclusions that the process:
- was not 'in control'
- did not meet the specification required (i.e. it was not centred on the specification value)
- had variations greater then the tolerance interval.
The 'not-in-control' situation was not unexpected, since the process was at the early stages of its development. However, the analysis conducted highlighted the importance of a further study in order to define the conditions for an optimal design. Furthermore, without such an analysis, variability in final sintered density may have been assumed to be associated with variability of the sintering process, rather than control of laminate green density.
On the basis of the above data, a 'cause-effect' analysis was conducted to elicit all contributing factors to a variation in the pressed density. The results are shown in Fig 3.

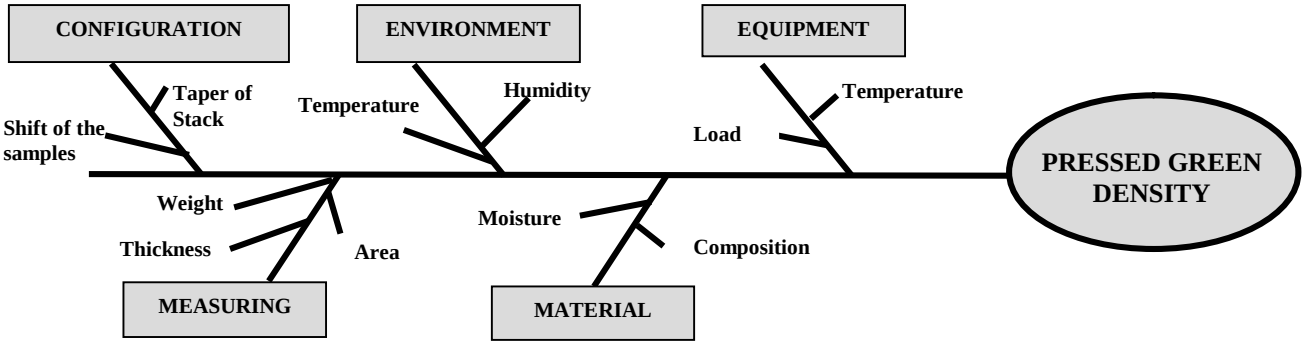


Fig.3: Fishbone analysis: contribution factors to pressed green density

Each of the listed factors noted in Fig.3 was ranked:
- The material used was obtained from the same cast tape for all the samples. Therefore it was unlikely to be the dominant factor in the density variability.
- The exposure of the samples to external conditions was negligible; hence environmental contribution was taken as minimal.
- The measurement techniques were assessed. Two different techniques were compared for the evaluation of the sample areas (geometrical and digital), the thickness (two micrometers) and the mass (two balances). A maximum error in density of 0.6% was detected. Therefore, the influence of the measurement technique was insignificant compared to the process variations.
- The configuration of the samples in the press was checked. Some samples shifted postion on mounting into the press, introducing a taper to the pressed samples. If the density data of those samples was eliminated from the chart analysis as assignable causes no improvement in process

control was found. It was therefore concluded that this factor was not the main cause of the variability.

- The equipment pressure setting was considered an important variation factor, since the applied load was reconfigured each day.

As a result of the above analysis, control charts plotting day by day control limits were drawn showing the variation with pressure re-setting, i.e. the variability each day. These are presented in Fig 4 A and B. In the new charts the means line is usually contained within the band of the control limits. This is an indication of a day by day 'controlled' state

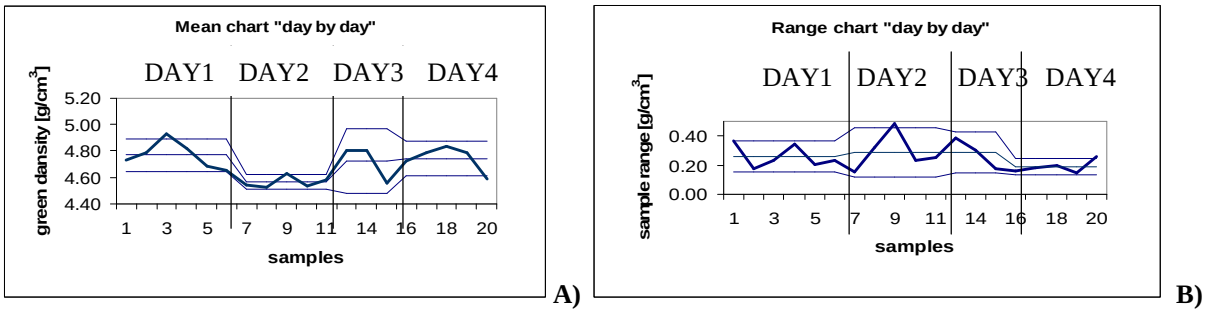


Fig 4: A) Mean chart with day by day variation B) Range chart with day by day variation

Hence the variability in the setting of the load on the equipment was evaluated to be the assignable cause of density variation. On investigation it was noted that the equipment used for the experiment was oversized compared to the sample size, producing difficulties in accurately controlling load.

Larger sample sizes or redesigned load controls could bring the process back within acceptable tolerances. Secondly, the analysis suggested that the pressing conditions of 50 MPa were an underestimate, since the required final density was rarely obtained. An increase in pressure is therefore suggested. However, the density value required (5.0 g/cm^3, 61% of the theoretical density) is close to the achievable maximum.

Conclusions

A warm-pressing process for laminating aqueous tape-cast ceramic tape was analysed with the SPC technique in order to assess its capability of meeting the required process specifications. The process was found incapable of meeting the requirements and was in an 'uncontrolled regime'. Problem solving techniques were applied to find the causes and investigate the possible solutions. The problem solving analysis yielded the conclusion that the most significant factor for the lack of process control lay in the control of pressure on the equipment. Moreover, the designed conditions for the process were judged insufficient for the achievement of the targeted specifications. Suggestions for further development of the process were given.

References

[1] S. Rosenthal: *Effective product design and development*, (Irwin; UK1992), pp.18-36

[2] K.T.Ulrich: *Product Design and Development*, (Mc Graw Hill, USA 2000), p.14-30

[3] J. S. Oakland: *Total Quality Management* (Nichols Publishing Company, New York 1989) p. 50

[4] J.S.Oakland: *Statistical Process Control* (Butterworth Heineman, Oxford 1999): p.14

[5] D.F. Kehoe: *The fundamentals of Quality Management* (Chapman & Hall, UK 1996), p.135-136

[6] C.M. Creveling et al.: *Design for six sigma* (PH PTR, New Jersey 2003), p. 707-21

[7] A. Navarro, J.R. Alcock, R.W. Whatmore: J. Eur.Ceram. Soc. (2003), in press.

[8] L.Mortara, A.Navarro, R.W.Whatmore and J.R.Alcock: J. Eur. Ceram. Soc. (2003): in press

[9] L.Mortara, A.Navarro, R.W.Whatmore and J.R.Alcock: Key Process Engineering (2003): in press

[10] M.R. Rahaman: *Ceramic Processing and Sintering* (Marcel Dekker, New York 1995), p. 270.