Statistical control in the glass technology research and manufacturing

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It is well known that the mathematical method to investigate and control large populations of objects is the classical statistics. One of the many applications of this predictive method is the control of the technological processes. This application enables the user to save time and money in the perspective of obtaining products with very precise values of properties and good quality. In the present paper two applications are presented of the way to use the statistical control in the glass technology research and manufacturing. One of them refers to the control of the crystalline populations developed on a glass matrix. This approach enables to predict the right time and temperature parameters of the crystallization process in order to achieve a well-defined range of crystalline dimensions. The second application deals with the way of statistical control in developing a small production process for glass capillary tubes. This approach enables to precise detect the moment when some changes which affect the product quality succeeds in the processing machine and consequently the moment when some corrective measures must be applied in order to optimize the process.

1. Introduction

In order to control the technology for making glass based structures, to know the dynamics of the involved structural transformation processes is a necessity. SEM, in the SE mode is a modern investigation tool in the field of microstructure characterization of ceramic and glasses. Statistic treatments of the dimensional and frequencial information concerning the structural entities produce data such as crystal growth speeds and specific densities. In the vitreous partly crystallized structure presented in the first part of this paper, the structural entities are produced in low crystallizable vitreous matrix by different times (t) and temperatures (T) of thermal exposure.

The statistic control is also one of the modern tools of the fabrication processes monitoring and quality estimation. The use of this method requires the following steps to be considered: the primary analysis, establishing/evaluating the statistical indicators correction and new analysis of the fabrication process.

2. Statistical control of the crystallization processes

2.1. Basics

Fig.1 presents the steps of the statistic method for computing the growth speeds of the crystals developed in a low crystallizable matrix at different temperatures. For each thermal exposure temperature T, a plot of maximum crystal's dimensions (dm) versus time t must be performed. The slope of each plot represents the growth speed V at the corresponding temperature T. This treatment is based on the method of Panaite and Munteanu[1] for the statistic control of products.

2.2. Application on a vitreous partly crystallized structure in the SiO_2-B_2O_3-BaO-PbO-Bi_2O_3 system

For a low crystallizable glass matrix from the SiO_2-B_2O_3-BaO-PbO-Bi_2O_3 system, having high Pb and Bi content, crystalline entities was developed on samples' surfaces by heating them for 1- 5 hours at temperatures in the range 775 - 850 °C. The temperature

range for the thermal treatments was chosen from the DTA investigation of the matrix glass. After a previous thin Al film vacuum deposition, the samples were SEM investigated (ex. in Fig.2) and this primary information was then treated like in sect. 2.1. This treatment allow to picture the temperature dependence of crystals' growth speeds V (Fig. 3a), to position the maximum growth speed in the temperature scale and also to find the corresponding isometric chart (Fig.3b).

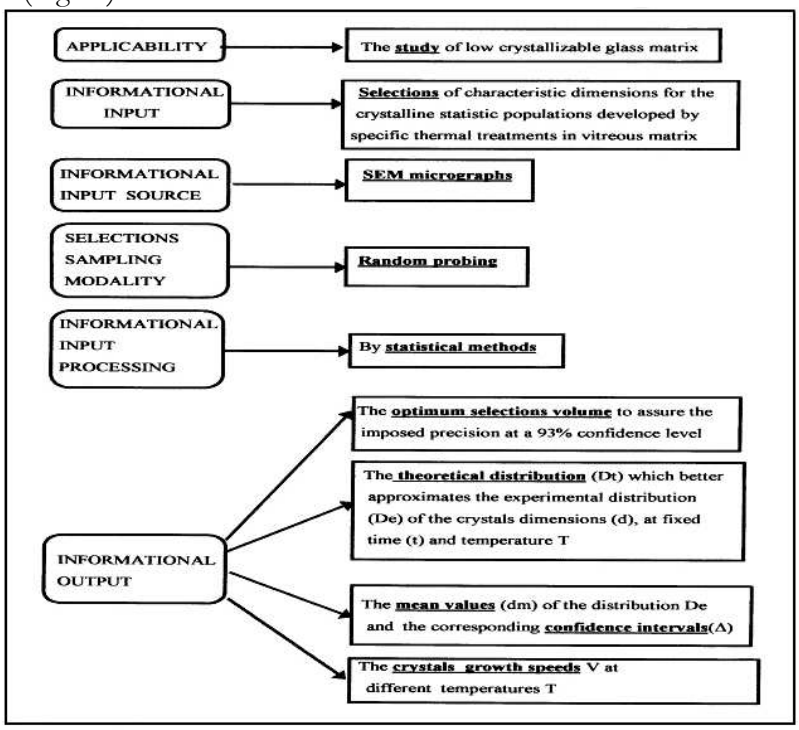
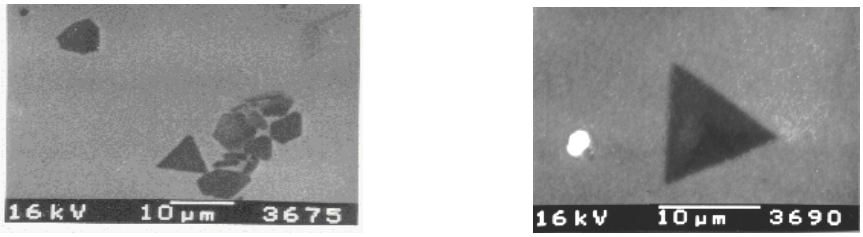


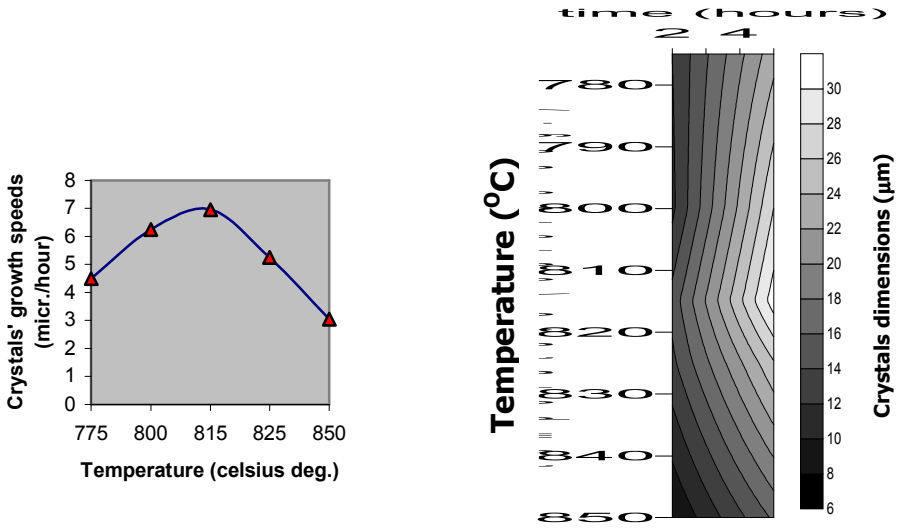
Fig.1 Presentation of the statistic method to process primary SEM data[2]



a) *b)*

Fig.2 SEM photos on a low crystallizable glass matrix from the SiO2-B2O3-BaO-PbO-Bi_2O_3 system, having high Pb and Bi content, heated for 3 hours at 850 °C (the white zones in the photos represents some undesired inclusions in the glass matrix).

This chart represents the curves of equal crystals dimension in the plane TIME - TEMPERATURE and it is indispensable in a controlled size crystallization technology for high quality vitreous crystalline material's synthesis. It enables the user to find the right temperature and time for the thermal exposure in order to obtain a desired dimension for the developed crystals.



a) *b)*

Fig.3 a) Crystals' growth speeds as a function of temperature; b) Isometric computed chart in the T - t plane (μm in the gray scale legend)

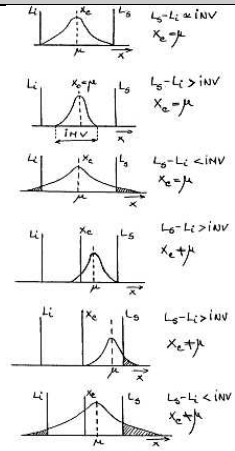
3. Statistical control of the quality in small production processes

3.1. Basics[3]

In the following, there are presented some basically aspects of the measure based control. The primary analysis checks the process static and dynamic stability. The technological process is considered to be statically stable if, for a great volume selection, the values of the tested property present a random variability and the corresponding experimental distribution is the normal distribution. A number of successive selections are studied in order to check the dynamic stability. The adjustment (position of the field scattering) and the precision (scattering amplitude) of the technological process are evaluated using appropriate indicators (the mean value μ and the dispersion σ^2). The dynamic stability is present when the values μ_i and σ_i^2 ($i = \overline{1,k}$) for a number of k inspected selections present small oscillations around a fixed value and don't systematically present an ordering trend (in an increasing or decreasing sequence). A technological process can be static and dynamic stable but not precisely adjusted, e.g., not being in the specified limits of the technical documentation or not having the necessary precision. The mean value μ and the standard deviation σ characterize the adjustment and the precision of the technological process. The adjustment and the precision are estimated in correlation with the position of μ and σ respecting the specified limits (see table 1).

The minimum admissible tolerance field is equal with the natural variation range INV corresponding to a 0.001 signification level, e.g., $INV = 6.58\,\sigma$. It is recommended that the specified tolerance range ITS = L_s- L_i = T, to respect the condition $INV/T \cong 0.6 \div 0.8$. The central value $X_C = T/2$ must correspond with the settled value of μ. The statistical control based on measure checks the localization parameter and the variation parameter (adjustment and precision) with the aid of the following indicator pairs: mean value and amplitude (μ-W), median value and amplitude (Me-W), mean value and standard corrected deviation (μ-s).

Table1. Framing of a production process in the specified limits

Possible cases- Relations between parameters	Type of the production process	Conclusions
	*Precisely, centered, without drop outs*	*Not correcting*
	High precision, centered, without drop outs	*Correcting, if it is economically justified*
	Not-precisely, centered, with drop outs	*Increasing the precision*
	High precision, not-centered, without drop outs	*Centering and reducing the precision, if it is economically justified*
	High precision, not-centered, with drop outs	*Centering and reducing the precision, if it is economically justified*
	Not-precisely, not-centered, with drop outs	*Correcting the process or working on another machine*

** Ls- superior tolerance, Li – inferior tolerance*

For a selection of volume n, the upper indicators are calculated as follows: $\mu = \frac{1}{n}\sum_{i=1}^{n} x_i$,

$s = \frac{1}{n-1}\sqrt{\sum_{i-1}^{n}(x_i - \mu)^2}$, $W = x_{\max} - x_{\min}$, $Me = \begin{cases} x_{\left(\frac{n+1}{2}\right)} \text{..... ..... . for odd } n \\ x_{\left(\frac{n}{2}\right)} + x_{\left(\frac{n}{2}+1\right)} \text{..... for even } n \end{cases}$,

where x_i are the values for the checked characteristic.

The maximum efficiency statistical control is realized using the pair μ-s. For the localization parameter μ, the control limits (superior L_{cs} and inferior L_{ci}) and the monitoring limits (superior L_{ss} and inferior L_{si}) are calculated using specific relations. Equal for the limits of the variation parameter s (L'_{cs} , L'_{ci} ,L'_{ss} , L'_{si}). The monitoring limit is an "advertising" limit and the process must be carefully tracked when this limit is exceeded. The control limit is an "action" limit, it's exceeding indicating the action necessity.

3.2. Application on a small production process for glass capilary tubes

The upper basics were applied on an "in developing" experimental installations of producing hematological capillary tubes, in order to point out the steps to be performed till the optimum functioning. It was checked the three successive days production represented by the lots R3(100pcs.), R4(221pcs.) and R5(228pcs.) having the following prescribed characteristics: external diameter $\varphi_{ext} = 1.5mm$, length L= 60 mm, one point measuring diameter tolerance $\pm 20\mu m$, two points measuring diameter tolerance $\pm 5\mu m$. Because the values of the characteristic $\Delta\varphi$ (deviation from the prescribed diameter, determined by one point measuring) present a random variation, the process was found to be statically stable. For checking the dynamic stability there were used 5 selections of volume 5: one selection from the R3 lot, two selections from the R4 lot and two selections from the R4 lot. Analyzing the data from table 2 it was found a tendency to dynamic instability of the process.

Table 2. Statistical indicators for evaluating the dynamical stability

Selection code	Day	$\Delta\varphi$ value (μm)					Statistical indicators (μm)		
							μ_i	σ_i	s_i
P1	1	+30	+46	+32	+40	+8	31.2	12.94	14.46
P2	2	0	+14	-2	+20	+18	10.0	9.21	10.30
P3	2	+14	+12	+4	+32	-12	10.0	14.31	16.00
P4	3	-10	+14	+12	-12	+30	6.8	15.83	17.70
P5	3	+14	+14	+18	+68	+12	25.2	21.49	24.5

On the basis of the data from table 2 and of the prescribed values, there were found the following values for the interest parameters: L_s=20μm, L_i= -20μm, INV=97.12μm, T=40μm, Xc=20μm, $\overline{\mu}$ =16.64μm, $\overline{s}$ =16.59μm, $\overline{\sigma}$ =14.76μm. The relation between (L_s–L_i) and INV, respectively Xc and $\overline{\mu}$ is: L_s–L_i <INV and Xc≠ $\overline{\mu}$. In conformity with the table 1,it is the case of a not-precisely, not centered, with dropouts - fabrication process. On the basis of this conclusion, an optimization to the capillary drawing machine was performed in this stage. This enabled to obtain precisely, centered, without dropouts fabrication process and to integral eliminate the periods of instability of the adjustment and precision (like in the first and third analyzed days, in conformity with the control diagrams from fig.4).

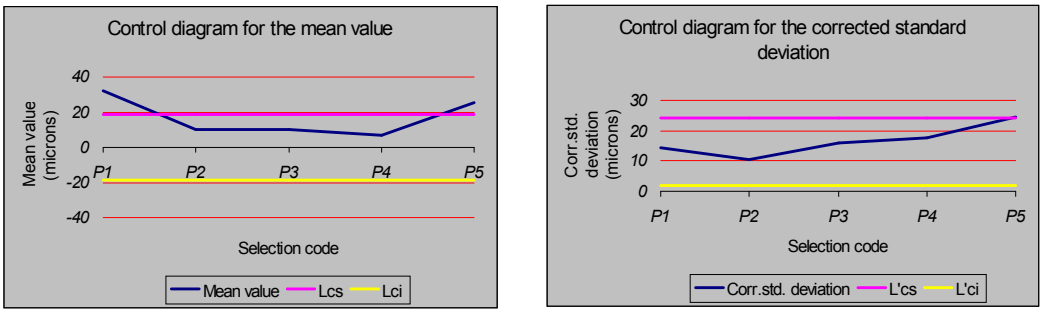


Fig. 4 Control diagrams for μ and s

4. Conclusions

The paper presented the utility of the statistical analysis in monitoring the products and processes quality in the field of glass industry. It was demonstrated that this type of analysis is also a useful tool in the research field because it enables to optimise the products and technologes in their early stages of development.

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[1] Valeriu Panaite, Radu Munteanu, in *Control statistic si fiabilitate*, edited by Editura Didactica si Pedagogica (Bucuresti, 1982), p. 204-209.

[2] Laura Ionescu, Building Materials **4**, p. 298-302 (2000).

[3] Valeriu Panaite, Radu Munteanu, in *Control statistic si fiabilitate*, edited by Editura Didactica si Pedagogica (Bucuresti, 1982), p. 209-218.