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ROTATING DISK ALUMINA PRECURSOR FIBER FORMATION PROCESS STABILITY DETERMINATION USING IMAGE ANALYSIS

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ABSTRACTS

Precursor fibers are made from the aluminium-hydroxide chloride based sols. The process stability can be influenced both by the fiber formation conditions and the rheology of the sol. The distance from the disk canter is the same, and the rotation speed is changed in one set of experiments. Four different sols are used under the fixed fiber formation conditions in the second set of experiments. Obtained fibers were dried at room temperature. The image analysis program, Lira 98 developed at the Faculty of Technology and Metallurgy in Belgrade, is used to measure fibers diameters. Obtained results are statistically handled. The fibers diameters coefficients of variation are used to evaluate the process stability dependence on fiber formation conditions and sol rheological properties.

Key words: alumina fibers, proces stability, image analysis

1. INTRODUCTION

Alumina ceramic fibers are used in new metal and ceramic matrix composite materials as reinforcement [1]. The other way to use them is as heat isolation [2]. For any purpose, their diameter is required to be less than 10 μm and the fibers should be of the good diameter uniformity. Fibers having these diameters can be produced from aluminium hydroxide-chloride based precursor by rotating disk method [3]. Green fibers are heat treated to obtain α-alumina ceramic fibers [4]. After heat treatment fibers preserve there shape but the diameter is smaller due to shrinkage. Hydrodynamic stability of the process is influenced by rheological properties of the sol [5]. Coefficient of variation of fibers diameters is chosen as a measure for process stability monitoring for different fiber formation conditions for the same sol.

The sol-gel transformation occurs during fiber formation process and the fiber geometrical characteristics are influencing their further use. Two main ways in process parameters evaluation can be used, one starting from deterministic modelling of fiber formation process where governing equations are solved, and main process parameters are established, and the other where the input and output parameters are studied and their dependence is established using statistical methods.

2. EXPERIMENTAL

Aluminium hydroxide-chloride based sol is prepared using the commercial Locron-s produced by Hoechst. Polyvynile-alchohole is added as additive to adjust the viscosity of the sol. To obtain the sol having lower surface tension the surface-active material is added in amount of 0,1% mass to two samples. In the second set of experiments the fiber formation conditions are changed so the influence of fiber formation conditions can be monitored.

The rotating disk method for fiber formation is used in experiments. The sol was dosed on the surface of the rotating disk at the flow rate of 10 cm^3/min. Obtained fibers were collected at approximately 1m from the place where they were formed. The fibers were dried at room temperature conditions prior to examination under the optical microscope attached to the computer using the CCD camera.

The sol of given rheological characteristics is dosed on the surface of the rotating disk at a fixed distance from the disk canter. The speed of rotation used in experiments was 5455 rpm, and the distance from the disc canter was 3 cm.

In order to examine the influence of fiber formation conditions on the formation of the sol of fixed rheological properties was dosed on the surface of the rotating disk using two distances from the disk canter, 3 cm and 6 cm and two speeds of rotation rpm and rpm.

3. RESULTS AND DISCUSSION

Fibers diameters were measured using the program Lira 98 developed at the Faculty of Technology and Metallurgy in Belgrade. Obtained data are used in statistical analysis and coefficient of variation is calculated for every specimen of fibers obtained under different fiber formation conditions. In Table 1 results of statistical analysis of obtained data for different sols used to obtain fibers at same formation conditions are given. In Table 2 results of the influence of formation conditions on fiber diameter are given.

Table 1 - Results of statistical analysis of fibers diameter measurements

	Viscosity 163,8 mPa s Surface tension 33,55 mN/m	Viscosity 2136 mPa s Surface tension 34 mN/m	Viscosity 1591 mPa s Surface tension 51,65 mN/m	Viscosity 163,8 mPa s Surface tension 51,7 mN/m
Mean fiber diameter value, μm	1,6917	3,8702	2,3617	1,6584
Standard Error, μm	0,0722	0,2534	0,1684	0,0839
Standard Deviation, μm	0,3751	1,6226	0,8911	0,4517
Minimum, μm	1,1734	1,8304	1,0064	0,7956
Maximum, μm	2,6304	10,3995	4,3305	2,8392

Table 2 - Results of statistical analysis of fibers diameter measurements

	n=5455rpm r=3cm	n=7273rpm r=3cm	n=5455rpm r=6 cm	n=7273rpm r=6cm
Mean fiber diameter value, μm	3,8702	4,1867	3,7703	4,0145
Standard Error, μm	0,2534	0,1608	0,3052	0,2817
Standard Deviation, μm	1,6226	1,1484	1,7795	1,5167
Minimum, μm	1,8304	1,7271	1,5728	2,1227
Maximum, μm	10,3995	6,7106	11,3239	9,9378

In Figure 1 typical histogram of relative frequency of the fibers diameters obtained from the sol under defined formation conditions is given.

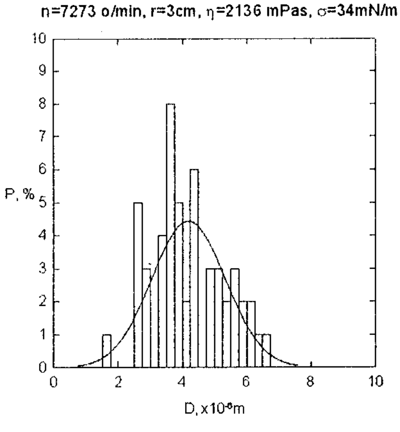

Figure 1 – Typical histogram for fibers diameters distribution for the sol under dosing radius of 3cm and rotation speed of 7273 rpm.

From deterministic modelling of fiber formation process the main process parameters are established. They are rheological characteristics of the sol and those are sol viscosity and sol surface tension [6]. Other parameters are kept fixed as they are determining the process of rotating disk fiber formation and those parameters are speed of disk rotation and distance from the disk center where the sol is dosed on the surface. Obtained fibers were dried at room temperature. The image analysis program, Lira 98 developed at the Faculty of Technology and Metallurgy in Belgrade, is used to measure fibers diameters. Obtained results are statistically handled. The mean fiber diameter value dependence on sol viscosity and surface tension is obtained. The fibers diameters coefficients of variation are used to evaluate the process stability dependence on fiber formation conditions.

The dependence of fiber diameter on sol rheological parameters was established in a linear regression model as:

$$D_{mean} = 2{,}764 + 0{,}0008887\eta - 0{,}02971\sigma \qquad (1)$$

Where η is the sol viscosity in mPa s, and σ is surface tension in mN/m. The regression coefficient R^2 is 0,923 so the model is acceptable. The ex-perimental results compared to the obtained model are given in Figure 2.

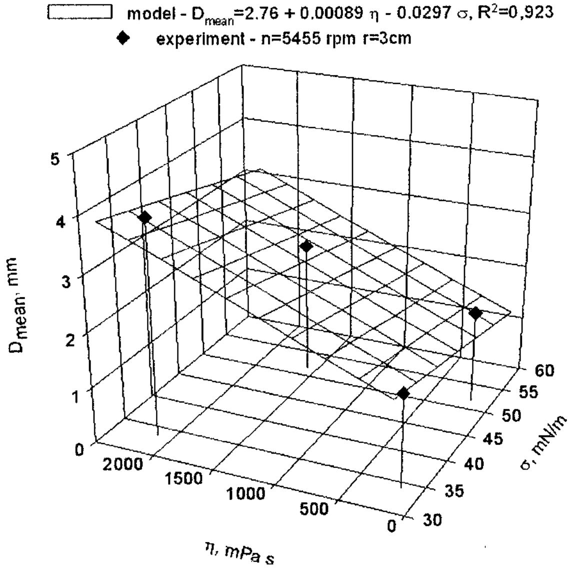

Figure 2 – Experimental results compared to obtained regression model for dependence of mean fiber diameter on sol rheological properties

The coefficient of variation of fiber diameters is used to establish the process stability. The dependence of coefficient of variation CV_D on sol rheological properties is given by the regression model:

$$CV_D = 0{,}161277 + 0{,}0000913334\,\eta + 0{,}00161249\,\sigma \quad (2)$$

Where η is the sol viscosity in mPa s and σ is surface tension in mN/m. The regression coefficient R^2 is 0,981 so the model is acceptable.

Linear regression model of dependence of coefficient of variation of fiber diameter on fiber forming conditions such as distance of dosing and the rotation speed was obtained. The linear regression model is given as:

$$s/D_{sr} = 0{,}687 - 0{,}0000658\ \mathrm{n} + 0{,}0268\ \mathrm{r} \quad (3)$$

Where s is the standard deviation, D_{sr} is the mean fiber diameter value in μm, r is distance from the disk centre where the sol was dosed and n is the speed of rotation in rpm. The regression coefficient is R^2=0,97 and is high enough so the model can be used for data interpretation for the sol having given viscosity and surface tension values. The process is more stable for smaller radius of dosing and for higher values of rotation speed. In Figure 3 the changes of the coefficient of variation with the formation conditions together with the obtained regression model are given.

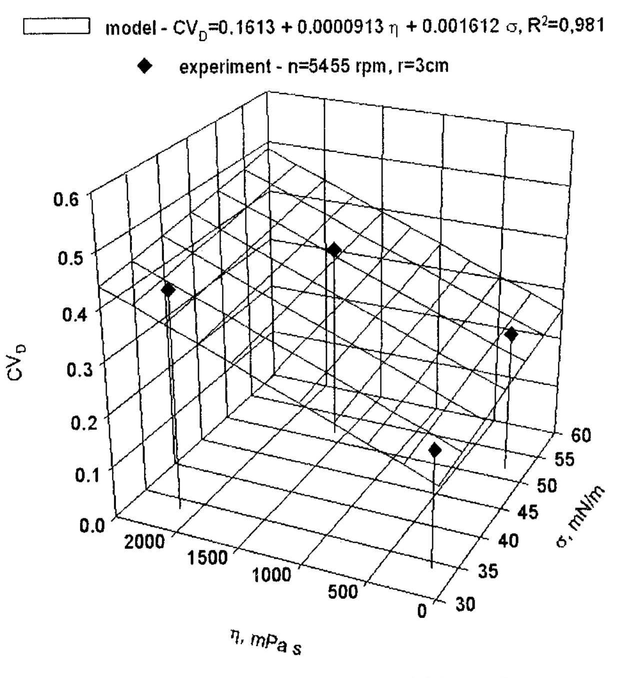

Figure 3 - Changes of coefficient of variation of fibers diameters with speed of rotation and distance of dosing together with the regression model

From the model for mean fiber diameter it can be concluded that fiber thickness is directly proportional to the sol viscosity and indirectly to the sol surface tension. That is expected from the force balance. The lower the viscosity it is easier to draw the fiber. The process stability is measured using the fiber diameter coefficient of variation. The lower value of coefficient of variation means more stabile process and the process is according to the established model more stabile for the lower values of sol viscosity and surface tension. This process is very specific and for the complete analysis more parameters should be studied, but those parameters are the most important for process control.

4. CONCLUSION

The rotation disk method was used to produce alumina ceramic fibers. Green fibers were examined using the optical microscope attached to the computer using the CCD camera. For all fiber specimens the fibers diameters were measured and used in statistical analysis. Regression models of mean fiber diameter and fiber coefficient of variation were derived.

Other important result from this paper is that the use of image analysis to determine essential data for model evaluation. The program Lira 98 was designed to enable the measurements of several image characteristics. One of them is used in this measurement and that is the distance between two points. The object to be studied was one single fiber that could clearly be seen on the image. The distance from two sides of that image was measured on three different points of the fiber and than the mean value for the single fiber was calculated. This process was done on large number of fibers and than data were statistically handled using standard statistical methods. This is the first step in image analysis program development and the algorithm for fiber diameter measurements was obtained. The second step is to make this measurement automatic and statistical data driven from the measurement very easy obtainable and this was done according to these results.

REFERENCES

[1] Kerans R.J., Jero P.D., Parthasarathy T.A., "Issues in the Control of Fiber-Matrix Interfaces in Ceramic Composites", *Composites Science And Technology*, **51** [2] 291-296 (1994)

[2] Leiser D. B., Smith M., and Goldstein H. E., "Developments in Fiborous Refractory Composite Insultion," *A. Ceram. Soc. Bull.*, **60** [11] 1201-204 (1981)

[3] Jančić R., Aleksić R.,"Influence of formation conditions and precursor viscosity on mean fiber diameter formed using the rotating disk method" *Mat. Lett.* **42** (2000) 350-355

[4] Jančić R., Aleksić R., "Dependance of green fibers diameters formed on the rotating disk on formation conditions" in serbian, *Metalurgija*, **5**,[1], (1999) 25-8

[5] Jančić R. Aleksić R, Stojanović D. "Hydrodinamical stability of ceramic fiber precursor spinning", *International conference of south-east europian chemiccal societies, Halkidiki Greece June 1998*. p 832

[6] Jančić R., Aleksić R.,"Mathematical modeling of ceramic fiber precursor spinning", *CHISA 98. CD Book of papers* H1.6