Automation of the process and mathematical processing of the results of annealing the hemispherical resonators

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Abstract. Isotropy of physical properties and strength of quartz glass makes it a good material for use as resonators in aircraft navigation systems, in complex optical systems and automation elements. To form a solid surface, it is necessary to create siloxane bonds, consisting of silicon-oxygen compounds forming the structural unit of quartz glass of the SiO₄ tetrahedron. For this purpose, an automated resistance furnace operating under the control of a personal computer was developed, and for processing of the results of surface quality, a computer program «Method of lying drop» written in Delphi 7.0 was used. The results of twelve measurements for each temperature are as follows: annealing temperature, mean square deviation, mean square sampling error, the value of the wetting angle, confidence interval, confidence probability, student coefficient. The calculation results can be displayed on the screen or in an external file. The program interface is convenient and clear.

1. Introduction
The great demand for products made of quartz glass in the aviation and aerospace industries producing hemispherical mechanical resonators makes quartz glass an indispensable material [1, 2]. One of the most important properties of KU-1 glasses is the isotropy of physical properties. But this is not enough, it is necessary to strengthen the surface of the product, as in the process of the operation the shape of the resonator can change, which entails a change in the inductance of the circuit and the appearance of errors in the gyroscopes of aircraft. The strength of the surface is directly proportional to the structure of the glass, namely the size of the globules. The smaller the size of globules, the stronger the siloxane surface. The smallest size of globules is achieved by experimental selection of the annealing temperature.

2. Experimental technique
The objects of study were hemispherical resonators having a diameter of 30 mm and a thickness of 2 mm in the amount of 8 pieces. Technology of formation of siloxane surface on the product of quartz glass KU-1 includes annealing.

The annealing operation took place for 11 hours: 3 hours of heating, 5 hours of annealing and 3 hours of vacation for slow stress relief. In order to avoid cracking of the samples during calcination, the samples were placed in an oven and heated at a rate of 300 °C/h to a predetermined temperature. The furnace was designed in such way (figure 1) to ensure that the temperature gradient covering the surface of the product is uniform on all sides. This is provided by the winding of the heating element 4 and the housing of the chamber 3, which has a large volume of the working space of the furnace compared to...
the dimensions of the product. The operating temperature was maintained by temperature sensors 6 and controlled by thermocouples 7 using sensors 8 under the control of a personal computer. The annealing time was controlled by timer 5. The furnace was located on the base 1. Products 10 in the amount of 5 pieces were loaded into the furnace body 2 through the loading window 9.

![Schematic diagram of the thermal resistance furnace for annealing of quartz glass products](image)

**Figure 1.** Schematic diagram of the thermal resistance furnace for annealing of quartz glass products:

1 – the base of the furnace; 2 – housing; 3 – the heating chamber; 4 – heating element winding; 5 – timer; 6 – thermoregulator; 7 – thermocouple; 8 – one of two thermocouple sensors; 9 – boot window; 10 – product.

According to the principle of operation, this apparatus is called an indirect furnace, since heat is released in the heating elements, evenly covering the heating chamber. Heat is released in accordance with Joule – Lenz law. The heating chamber is lined with refractory lining and thermal insulation, placed in a metal casing. During the experiment, 8 resonators were annealed, none of them had defects.

The method of lying drop was used to process the results [3]. For figure 2 shows micrograph of a drop, obtained by means of a PC computer, and microscope MII – 4M is equipped with a video eyepiece connected to the USB bus. The drop height $h$ and the base diameter $d$ were measured by determining the number of pixels on the obtained microphotographs using the program TSview v7.3.1.7. Micrographs for each sample were taken after 1, 3, 5, 10 minutes. The height of the drop and the diameter of its base were taken as the average of three measurements. The measurement error left no more than ± 0.3 degrees. In order to increase the measurement accuracy, we used fluids with different surface tensions $\sigma_w = 71.96$ MH/m for distilled water and $\sigma_g = 59.4$ MH/m for glycerol.

The wetting angle or its cosine is a characteristic of the surface tension forces. It is defined as the angle between the tangent drawn to the surface of the wetting fluid and the wetted surface of the solid, and the angle is always measured from the tangent in the direction of the liquid phase. The tangent is conducted through the point of contact between the three phases: the solid phase (membrane), the liquid (distilled water) and the gas (air). Three phases are involved in wetting: 1) a solid body; 2) wetting fluid; 3) the “precursor” phase, which was in contact with a solid surface before the supply of liquid. In accordance with the Young – Laplace theory, the contact angle is determined by the competition of two forces acting on the three-phase contact line.
The determination of the wetting angle was made by the formula (1) from the condition of mechanical equilibrium on the line of a three-phase contact droplet deposited on a solid surface:

$$\cos \theta = \frac{(\frac{d}{2})^2 - h^2}{(\frac{d}{2})^2 + h^2},$$

(1)

where $\theta$ is the wetting angle; $d$ is the average diameter of the base of the drop; $h$ - the average value of the height of the drop, which is calculated by the formulas of the average value:

$$d = \frac{\sum d_i}{n}, \quad h = \frac{\sum h_i}{n},$$

where $n = 12$ is the number of tests; $d_i, h_i$ - current values of the diameter and height of the drop, in pixels.

During the experiment, we obtained the following results: the program «Method of a lying drop» [4] was developed, which, using 12 values of the height and diameter of a drop, calculates the main parameters: standard deviation; mean square sampling error; angle value (in degrees); confidence interval; confidence level; student coefficient.

In figure 3 the program interface is shown «The method of lying drop» [4], and figure 4 shows the input window.
Figure 4. Data entry window interface.

Figure 5. The results of the calculations at a temperature of 900 °C.

Figure 5 shows the results of the calculation. All calculated parameters can be output to an external file.

3. Results and its discussion
After analyzing the results of calculations it can be concluded that the optimum annealing temperature was the temperature range of 1050-1080 °C. This is confirmed by table 1.

Table 1. Dependence of the wetting angle of the surface with distilled water on the annealing temperature of the quartz glass sample

| Annealing temperature, °C | Wetting angle, degrees |
|---------------------------|------------------------|
| 20                        | 39.33                  |
| 900                       | 32.85                  |
| 950                       | 35.50                  |
| 1000                      | 32.32                  |
| 1025                      | 32.04                  |
| 1050                      | 31.85                  |
| 1100                      | 32.78                  |
| 1125                      | 32.33                  |

The table shows that at an annealing temperature of 1050 °C the wetting angle is the minimum, and this indicates that the surface of the quartz glass has the maximum strength, since the tetrahedra that are
part of the globules stretch the molecules of distilled water. Eight samples of high-quality mechanical resonators were heat-treated in our laboratory and showed a high quality factor.

4. Conclusion
Comparing the results of the tables, it can be concluded that in the process of mathematical processing of the annealing results, the optimal temperature range is 1050 – 1080 °C, since it is in this process that the formation of globules with the smallest size occurs. This leads to the hardening of the quartz glass surface. An important role belongs to the size of the globules and the quality of the surface machining of quartz glass. With a decrease in the size of the globules, the "evenness" of the surface decreases, since the removal of the layer during machining occurs with the destruction of the bonds between them, which leads to an improvement in the inductance of the resonator circuit.

Similar results were obtained when determining the hardness of the surface of quartz glass by Vickers methods with a diamond indenter.

Eight resonators were annealed in the laboratory of the Pedagogical State University, which in the factory increased the quality factors from (10 to 22) · 10⁶ and which met the technical requirements.

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