Automation of thermal processing of quartz glass with the following computer treatment of the quality of siloxane communication

S M Dolapchi\textsuperscript{1}, O A Denisova\textsuperscript{2}

\textsuperscript{1}Department of Information, Mathematical and Natural Sciences graduate, South Ural Technological University, Chelyabinsk, Russia

\textsuperscript{2}Department of Physics, Ufa State Petroleum Technological University, Ufa, Russia

Abstract. The strength characteristics and isotropic physical properties of quartz glass give broad prospects for its use as a resonator for navigation systems of aircraft, as well as for complex optical systems and automation elements. To obtain a sufficiently hard surface with the desired properties, it is necessary to create conditions for the occurrence of siloxane bonds - silicon and oxygen compounds. During the formation of such bonds, the process of polymerization and annealing of quartz glass, high-strength resonator samples are obtained. The quality of the resulting surface is determined by different methods - fractal geometry, recumbent drop and Vickers. In the presented material, the authors propose to use the recumbent drop method (distilled water was used). To obtain samples of resonators, a resistance furnace has been designed and constructed, which operates in an automated mode using a personal computer control. Also, a computer program "Droplet stacking method" was written in Delphi 7.0, which presents the results of monitoring the annealing process, including the surface annealing temperature and contact angle. The results of measurements and calculations can be recorded as a file and observed on the display. The program interface is intuitive and convenient for controlling the process of obtaining samples. In addition, to form a high-quality connection, a program was also written to determine the radius of the nucleus and the value of the Gibbs free energy, which is necessary for the formation of the nucleus. The experimental results obtained and the calculations performed by the lying drop and fractal geometry methods have demonstrated the agreement between the results within the error limits of the methods.

1. Introduction

Obviously, the presence of vibrations is typical for mechanisms. The great demand for products made of quartz glass in the aviation and aerospace industries as hemispherical mechanical resonators (Figure 1) makes it an indispensable material.

One of the most important properties of quartz glasses KU-1 is the isotropy of physical properties. But this is not enough, it is necessary to strengthen the surface of the product, since during operation the shape of the resonator may change, which leads to a change in the loop inductance and the appearance of an error in the gyros of aircraft [1-9]. The quality of a quartz glass product is one of its main characteristics indicating quality. The quality factor can be estimated by the following formula [10]:

\[ Q = \frac{1}{R} \sqrt{\frac{L}{C}}. \]  

(1)
where $Q$ - is the quality factor of the product; $R$ - active resistance, Ohm; $L$ - inductance of the product, Gн; $C$ - product capacity, F.

Let’s consider how each factor affects the value of the quality factor. Quartz glass is a poor conductor of electric current, since it does not have a sufficient number of free electric charge carriers, therefore, this value is constant. The capacity of the part - the ability to accumulate electric charge, is also a constant value. Inductance will be variable and dependent: magnetic permeability of the medium; product sizes; shape of the product. The magnetic permeability of the medium is a constant, we cannot influence its technical or physical methods. The dimensions of the product can be changed, but often they are defined constructively, and we also cannot influence them [11, 12].

![Figure 1. The appearance of the resonator](image)

The form of the product also sets the designer, but we can affect its quality by physical methods, using the technology of obtaining siloxane bonds on the surface of the product. This will give not only the hardening of the surface, which entails an increase in strength, ductility and brittleness, but these parameters ensure the preservation of the shape of the part in the automation equipment with repeated switching or using it as a gyroscope. During annealing, in the process of creating a siloxane bond with a large temperature gradient, the surface is covered with small globules, which makes the surface even more durable, like a chain mail. The small size of the globules will ensure good coaxiality and small beating of the part, which will increase the inductance of the product, therefore, will directly proportional increase its quality factor. The strength of the surface is directly proportional to the structural structure of the glass, namely the size of the globules. The smaller the globules, the stronger the siloxane surface. The smallest size of the globules is achieved by experimental selection of the annealing temperature.

2. Objects of study and 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. The technology of formation of the siloxane surface on a quartz glass product KU-1 includes annealing. The annealing operation took place over 11 hours: 3 hours of heating, 5 hours of annealing, and 3 hours of tempering for slow stress relief. In order to avoid cracking of the samples during calcination, they were placed in a furnace and heated at a rate of 300°C/h to a predetermined temperature.

The furnace was designed and constructed so that the temperature gradient covering the surface of the product was uniform from all sides, this was ensured by the winding of the heating element 4 and the body of the chamber 3, which was provided by a large volume of the working space of the furnace compared to the dimensions of the product (Figure 2). The working temperature was maintained by thermal sensors 6 and monitored 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 grounds 1. Products 10 in the amount of 5 pieces were loaded into the furnace body 2 through the loading
window 9. According to the principle of operation, it is an indirect-action furnace, since heat is released in heating elements uniformly covering the heating chamber. Heat is released in accordance with the law of Joule - Lenz. The heating chamber is lined with refractory lining and heat insulation, placed in a metal casing. During the experiment, 8 resonators were annealed, none of them had any defects.

![Diagram of a thermal resistance furnace for annealing quartz glass products](image)

**Figure 2.** Diagram of a thermal resistance furnace for annealing quartz glass products: 1 - furnace base; 2 - case; 3 - heating chamber; 4 - winding of the heating element; 5 - timer; 6 - thermostat; 7 - thermocouple; 8 - one of two thermocouple sensors; 9 - boot window; 10 - product

For the processing of the results, the lying drop method was used [13]. In (Figure 3) there is a micrograph of a drop obtained using an IBM PC computer and a MII-4M microscope equipped with a video eyepiece, connected by a USB bus.

![Micrograph of a drop of distilled water on quartz glass (× 500)](image)

**Figure 3.** Micrograph of a drop of distilled water on quartz glass (× 500)

The height of the drop h and the diameter d of the base were measured by determining the number of pixels in the micrographs obtained using the TSview v7.3.1.7 program. In figure 4 shows the interface of the recumbent drop method [14], while figure 5 input data input window. In figure 6 presents the results of the calculation. All calculated parameters can be output to an external file.
**Figure 4.** The interface of the program “Method of a lying drop”.

**Figure 5.** Data entry interface

**Figure 6.** Results of calculations at a temperature of 900 °C
3. Results and its discussion
After a thorough analysis of the experimental results and calculations, we can conclude that the optimal temperature range for annealing is from 1050 to 1080°C. As confirmed by Table 1.

| Annealing temperature, °C | Angle of wetting, 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 lowest, and this indicates that the surface of quartz glass has maximum strength, since the tetrahedrons that make up the globules stretch the molecules of distilled water. Eight samples of high-quality mechanical resonators underwent heat treatment in our laboratory and showed a high Q-factor. The size of the globules on the quality of the mechanical processing of the surface of quartz glass plays a significant role. With a decrease in the size of the globules, the “tearing” of the surface decreases (Figure 7), since the removal of the layer during machining occurs with the destruction of the bonds between them [15, 16].

![Figure 7](image_url)

**Figure 7.** The effect of globule size on the quality of machining.
Optical microscope MII-4M (× 500)

The theoretical tensile strength is calculated by the formula [17]:

\[ \sigma_{\text{theory}} = \sqrt{\frac{\gamma_s E}{a}}, \]

where \( \gamma_s \) – specific surface energy, \( E \) – Young’s modulus of elasticity, \( a \) – distance between globules.

We have compiled a computer program to calculate the radii of the formed nuclei and the Gibbs free energy, which is necessary for its formation [17, 18]. Both programs are patented. The initial data for the program are specific surface energy \( \alpha \) and specific volume energy \( \gamma \) (Figure 8).
Figure 8. Computer program interface for calculating the radius of the globule nucleus and Gibbs energy

Calculation by the formula (2) for glass gives the value $\sigma=8 \cdot 10^9$ Pa, but the real strength is $\sigma=8 \cdot 10^7$ Pa [16]. This is due to the fact that there are microcracks in a solid body, which concentrate large stresses around them. One of the most effective ways to check the surface quality, which gives good results and does not require large costs, while the surface quality is estimated by one number, is the method of fractal geometry [19]. We used the method of maximum load. The values of microhardness were determined by the formula (3) [20]:

$$H_V = \frac{1.854 P}{d^2},$$

where $P$ - is the load in g; $d$ - is the print diagonal length in μm; $H_V$ – microhardness value, kg·s /mm².

The microhardness value for each sample was determined as the average value of 10 measurements. The measurement error of the diagonal length by the method of difference of two diagonals was determined by the formula [21]:

$$\sigma = \pm \sqrt{\frac{\sum (d - d_{\text{aver}})^2}{n-1}},$$

$d$ - is the print diagonal length in μm; $d_{\text{aver}}$ - the average value of the diagonal length for all measurements in microns; $n$ - number of measurements.

The obtained results of the method of limiting load of a glass sample are reflected in Table 2. The load was measured 10 times for each annealing temperature. The standard deviation is 0.022. The standard error of the sample is equal to 0.005. At a confidence level of 95%, the student's-rate is 2.21.

| Annealing temperature, °C | Microhardness, kg·s/mm² |
|---------------------------|--------------------------|
| 20                        | 890                      |
| 900                       | 882                      |
| 950                       | 920                      |
| 1000                      | 968                      |
| 1050                      | 1023                     |
| 1080                      | 1077                     |
| 1100                      | 996                      |
Conclusion
Comparing the results of the tables, it can be concluded that in the process of mathematical processing of the annealing results, the optimum temperature range is 1050-1080°C. 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 State Pedagogical State University, which in the factory increased the quality factors from (10-22)·10^6, which met the technical requirements.

References
[1] Lyklema J T 2018 Langmuir 14 5659
[2] Maekawa H 2011 J. Non-Cryst. Solids 127 53
[3] Mysen B O 2012 Rev. Geophys. and Space Phys. 20 353
[4] Brawer I A 2005 J. Phys. Chem. 63 2921
[5] Mysen B O 2012 Ibid 67 686
[6] Matson P W 2013 J. Non-Cryst. Solids 58 323
[7] McMillan P 2014 Amer. Miner 69 622
[8] Piriou B 2013 Amer. Miner 68 426
[9] Dolapchi S M, Denisova O A 2017 Electrical and data processing facilities and systems 13 106
[10] Voronkov M G 1976 Siloxane bond (Novosibirsk: Science) p 413
[11] Bykov V N 1990 Melts 2 31
[12] Lyklema J 2008 Langmuir 14 5659.
[13] Dolapchi S M 2016 Study of the structure of the surface of quartz glass by the drop method Certificate of state. reg. computer programs № 2016619827
[14] Bryzgalov A N 2016 Modern high technologies 6 19
[15] Griffith T 2008 The strength of quartz glass (Moscow: Science) p 127
[16] Dolapchi S M 2017 Calculation of the theoretical strength of quartz glass Certificate of state. reg. computer programs № 2017661254
[17] Dolapchi S M 2016 Program for calculating the radius of the embryo of the globule and Gibbs energy Certificate of state. reg. computer programs № 2016613497
[18] Sanditov D S 2019 Journal of technical physics 79 150
[19] Vladimirova T V 2019 OMP 9 31
[20] Glazov V M 1961 Microhardness of metals (Moscow: Metallurgizdat) p 224
[21] Dolapchi S M, Denisova O A 2017 Electrical and data processing facilities and systems 13 115