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FABRICATION OF TUNING FORK GYROSCOPE RESONATOR USING DIRECTED CHEMICAL ETCHING OF FUSED SILICA

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Abstract

The article presents the results of the experimental and practical work devoted to the study of directed chemical etching of fused silica locally exposed to ultra-short pulsed laser radiation as a way to produce sensitive elements of precision instruments of orientation, stabilization and navigation as resonators of tuning fork gyroscopes. The essence of the directed chemical etching method is presented. The created experimental setup structure and components are purposed and the laser processing and chemical etching operational modes are described. The laser processing mode obtained as a result of the experiment was used to fabricate the tuning fork gyroscope resonator. The etching rate of exposed fused silica in 5 % hydrofluoric acid is 27.5 to 68.8 μm/hour. That is much faster than the etching rate of the non-affected material. Based on this results the application of the method in the precision instruments fabrication area can be considered promising and subjected to further study

Keywords

Fused silica, hydrofluoric acid, chemical etching, laser, structuring, gyroscope, resonator

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Introduction. The unique combination of the most important structural properties of fused silica (SiO_2) has been attracting the attention of technical specialists from all countries of the world for the long time. The material, which has high transparency in the ultraviolet, visible and near infrared spectral regions, low sensitivity to thermal shock and excellent dielectric properties, has been widely used in the manufacture of optical systems. Nowdays, pure and doped fused silica is actively used in various fields of science and technology: nuclear power, chemical engineering, electronics, space technology, lighting, precision instruments manufacturing, etc.

One of the important applications of fused silica is the manufacture of sensitive elements of precision mechanics devices, such as accelerometers [1] and vibratory gyroscopes. The latter include various types of Coriolis Vibratory Gyros (CVG) with shell resonators in the form of a ring, a hemisphere, a cylinder, and other shapes (for example, birdbath) [2–6], as well as other solid-state gyroscopes with disk, rod, and beam resonators, etc. including the ones in the form of a tuning fork [7–11]. For such devices, the isotropy of the mechanical properties of fused silica and its ability for low dissipation of mechanical energy are of high priority [12].

A certain complexity is the processing of samples of fused quartz during the formation of elements of small parts. Thus, during the manufacture of the resonator of the tuning fork gyroscope (Fig. 1), it is necessary to make long through grooves 100 μm wide and 1 mm deep.

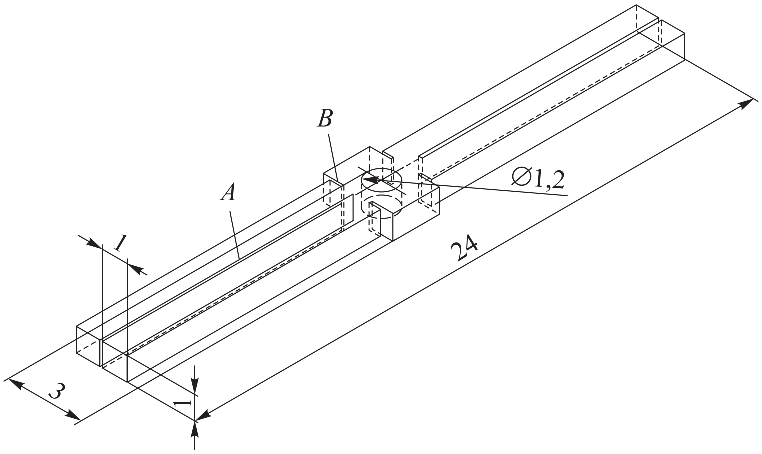


Fig. 1. Tuning fork gyroscope resonator:
A and *B* Through grooves 100 μm wide

Depending on the technical equipment of production, the problem can be solved by various methods: by chemical etching in a solution of hydrofluoric acid (HF); ion-chemical etching; laser treatment; ultrasonic treatment; machining by miniature diamond cutter.

We’ll consider the first of them more deeply. It is known that when a sample of fused silica is immersed in a container with hydrofluoric acid solution, a chemical reaction takes place, which is most often represented as follows [13]:

$$SiO_2 + 6HF \rightarrow 2H^+ + SiF_6^{2-} + 2H_2O \qquad (1)$$

The above equation describes the process of chemical etching only approximately. In fact, the reaction mechanism can vary significantly depending on the concentration of the solution, its temperature and other factors, and can be quite complex. In particular, one of the reaction products can be silicon tetrafluoride SiF_4, existing in the form of crystalline hydrate $SiF_4(H_2O)_2$. Such compounds can prevent uniform etching when they are formed on the surfaces of samples. Removal of the reaction products from the etching zones can be achieved by intensive mixing of the solution.

The typical rate of fused silica etching in a solution of hydrofluoric acid does not exceed several micrometers per hour as a rule. It should be noted that, due to the isotropy of the properties of fused silica, the etching of the sample in all directions proceeds approximately uniformly. Therefore, to obtain grooves of the required shape and size, it is necessary to use masking layer applied on the material, for example, by spraying or by other methods [14, 15]. To obtain groove walls with minimal deviations of the shape, the lateral surfaces formed as the process goes deeper into the material should also be protected by applying additional masks (Fig. 2).

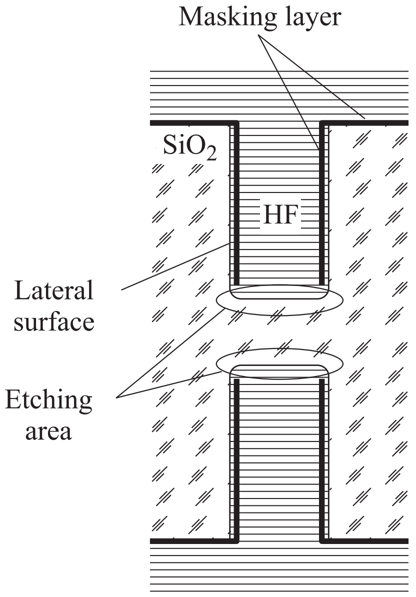


Fig. 2. The chemical etching of the fused silica sample in the solution of hydrofluoric acid

The described process is characterized by a long cycle of production and high labour intensity. Besides, there is a great risk of appearance of defects, due to the masking layer disturbance. Nevertheless, this approach is widely spread in precision instruments industry.

Let’s note that in this research there was no task of the detailed analysis of advantages and shortcomings of application of various methods of fused silica processing. Anyway, they concern such factors as labour intensity and productivity of process, existence and type of the arising defects, observance of sizes and form tolerances, saving critically important properties of material, tool and equipment runout, etc. The presented material, however, is necessary for further exposing of the directed etching method.

It is known from a number of sources [16–22] that there can be various structural changes in fused silica under the influence of ultra-short pulse laser radiation. The nature of these changes depends on the modification mode: density of radiation energy, duration and repetition rate of pulses, etc. Besides,

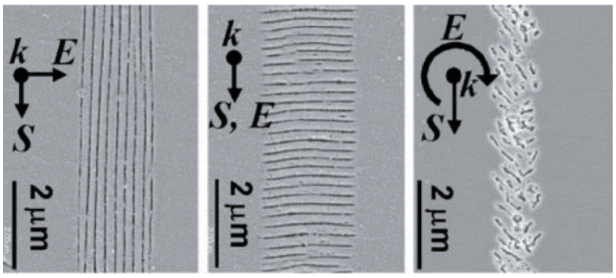


Fig. 3. The structures obtained in fused silica under the laser radiation with linear (at the left and on the center) and circular (on the right) polarization: *E* is the direction of polarization; *S* is the direction of the sample movement during processing

the polarization-depended ordered, or disordered structures can be obtained. At linear polarization the structures are oriented perpendicular to its direction, and at circular — in a random way that can be seen at Fig. 3 from the work [18].

The formation of structures in fused silica is possible at rather high density of energy of laser radiation which in certain cases reaches 20 J/cm^2 [16]. Such density is reached when the radiation is focused by lenses with a numerical aperture (NA) not less than 0.2. The structural changes of material at the same time have local character and are observed in areas, sizes of which at single influences are comparable to diameter of a focal spot and are about 1 μm. To achieve the necessary result the parameters of radiation are set experimentally. For example, pulse energy can vary from the two decimal places to units of microjoules.

The effect of glass structuring has various applications. So, in works of Russian scientists glass modification with femtosecond laser radiation is considered as a way of data recording [23] and creation of fiber Bragg grating for sensor systems, fiber lasers, fiber-optical communication systems [24].

This is an important fact that besides the change of optical properties of the modified material, in some cases the increase of activity of its interaction with hydrofluoric acid and other reagents is also observed [19–22]. This effect is the cornerstone of a method of production of details from fused silica which in some sources received its name of "laser-induced chemical etching". It is possible to achieve selective etching of fused silica in the necessary directions without application, or with limited application of masking layer, using this method. In this regard the term "the directed etching" will be used for designation of the method further in this work.

What defines change of chemical activity of the modified material — appearance of internal stress resulting from the laser exposure, formation of emptiness, micro-cracks or a laser-induced densification — is a subject of discussions for the present moment [17]. Despite the existence of controversial issues, a method has been already adopted widely. The directed etching is applied, for example, for the creation of microfluidic devices of “lab-on-chip” type [21], biochips [22] and other devices. At the same time the attention is not paid to application of a method for production of parts of accelerometers and gyroscopes.

The task to define possibilities of the directed etching method in the field of precision instruments industry was set within this work. For this purpose the experimental setup was created, the work on identification of the modes of interaction of laser radiation with substance for receiving local modifications of fused silica with necessary properties was carried out, the mode of etching of samples in solution of hydrofluoric acid was fulfilled, the technological process was developed and the sample of the resonator of a tuning fork gyroscope was made as an example.

Materials and methods of the solution of the task. ***Experimental setup.*** The femto-second laser system Pharos by Light Conversion with the built-in harmonics generator is used for generation of optical radiation with the ultrashort pulse duration in the experimental setup (Fig. 4). The settings of the system are so that output radiation has following characteristics:

– wavelength 515 nanometers (the second harmonic);
– the pulses repetition rate 100 kHz;
– energy of pulses 2.5 μJ;
– pulse duration ~ 300 fs.

The translation of radiation from a source to a working zone is performed by the system of system of two mirrors transferring a beam from horizontal plate to vertical. An objective lens with NA 0.25 is used to focus the radiation. Besides, the optical path contains the motorized radiation attenuator allowing to vary output energy in limits from 40 to 100 % of entrance value without losses. The excess of energy is taken away on the device built-in radiator when radiation is weak.

The precision motorized three-coordinate table working under control of the computer numerical control system (CNC), equipped with the industrial personal computer with the software, the CNC-controller and the motor driver are used for the sample positioning in the horizontal *XY* plane and adjustments of distance from objective lens focus to the working plane on vertical axis *Z*. Besides the solution of a positioning task of the sample the CNC system also operates the weakening of laser radiation and the shutter of laser system. The contol of the setup is carried out according to the program written in *G*-codes.

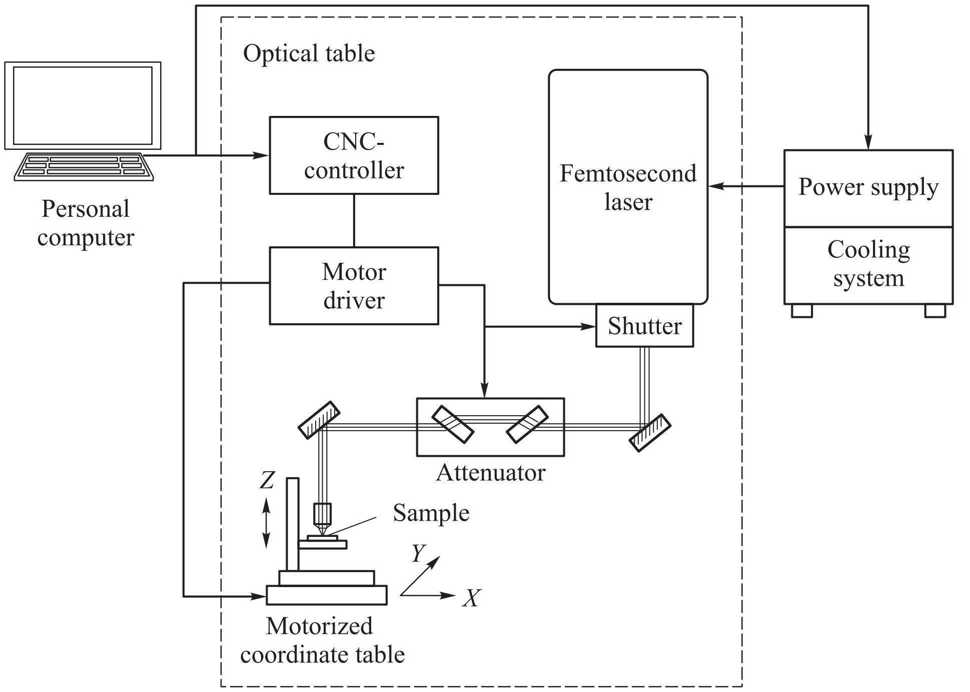


Fig. 4. Structure of the experimental setup

The mentioned setup elements, except for the personal computer, the power supply unit and the cooling system of femtosecond laser system, are fixed on a plate of an optical table with active vibration insulation.

Technological process. Process of the samples production by the directed etching method includes two main stages:

– structuring the fused silica at influence of laser radiation with the ultrashort duration of pulses. As modification of material happens under the influence of the radiation of optical range, further we will call this process as "exposing";

– removal of the modified material when etching a sample in solution of hydrofluoric acid.

It is necessary to make the following explanation to the exposing process. This process has to be carried out layer-by-layer, at movement of focus from the lower surface to top and with overlapping of layers for the modification of material on all depth of preparation (Fig. 5).

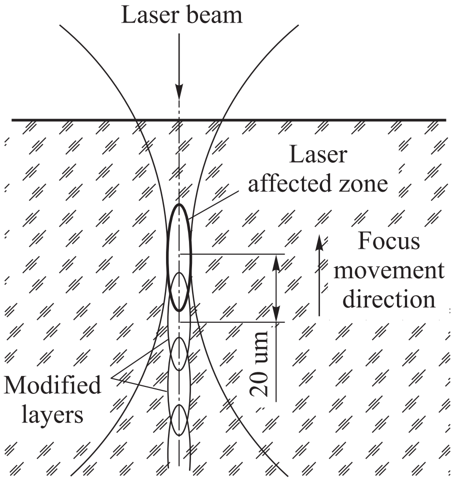


Fig. 5. The focus movement with layers overlapping

It is established in practice that under the considered conditions the necessary effect is reached when focus is moving on axis Z with the step equal to 20 μm. The choice of the step size depends on characteristics of laser radiation, the focusing objective lens and optical properties of initial material.

Results. ***Determination of the interaction mode of laser radiation with substance.*** During the experiment on a plate made of fused silica with thickness ~ 1.1 mm at various modes of laser radiation interaction with substance 25 multilayered contours of 2.5 × 2.5 mm in size has been produced in total (Fig. 6). Width of the contours line in each layer was ~ 100 μm that has been provided with repeated passing of a beam with a step of 20 μm. The exposing rate has been varied from a contour to a contour from 0.5 to 2.5 mm/s with a step of 0.5 mm/s, and the pulse energy has been varied from 1.0 to 2.0 μJ with a step 0.25 μJ.

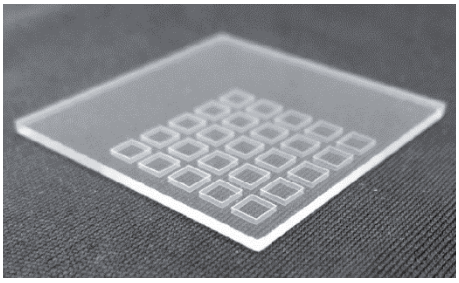


Fig. 6. The plate of fused silica with the zones modified at various modes of laser radiation interaction with substance

After exposing the sample was submerged in 5 % solution of hydrofluoric acid for the long period of time. The solution has been continuously mixed up during etching. Each two hours the sample removed from solution, washed out in water and dried, after that the visual estimation of the modified areas condition was carried out by means of a microscope.

After 8–10 hours through grooves were formed in a number of zones, at the same time their faster formation was observed along axis Y. Full pickled contours were obtained in 18–20 h.

The obtained difference in the etching rate can be connected with the differences in orientation of the modified structures due with the direction of radiation polarization (the laser system has linearly polarized output radiation, respectively, in the considered case the polarization is directed at a zero angle to axis X and 90° to axis Y). This assumption is confirmed by the data provided for example in [16, 20]. Besides, practically in all considered works devoted to the directed etching of fused silica the significant influence of orientation of structures on quality of process is noted: the most effective etching is observed for cases when modification of fused silica is made by the radiation with circular polarization, or the linear polarization focused at an angle 90° towards the movement of the sample [18].

The problem can be solved, for example, by changing the polarization of radiation from linear to circular with a quarter-wave (λ/4) plate, or by introducing a rotating half-wave (λ/2) plate into the experimental setup, which changes the direction of linear polarization according to with movement of the coordinate table.

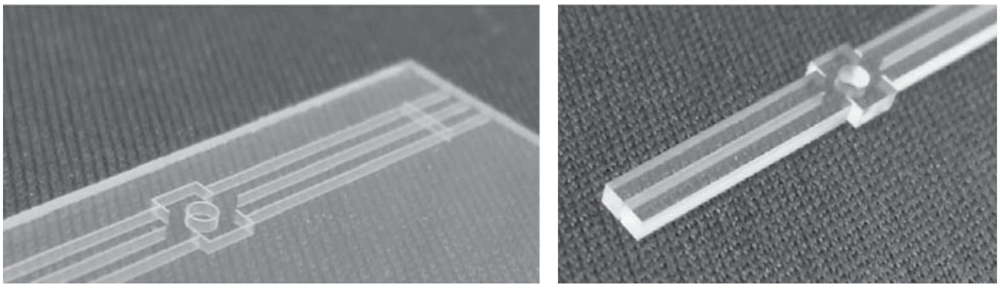
The observations made during the experiment showed that it is necessary to change the radiation energy depending on the position of the focus during exposure. This is due to the fact that at too high values of energy near the upper boundary of the sample, destruction and ablation of the material [25] can occur, or the formation of a layer of material that is poorly etched, and when the values are too small, there is no material modification at the lower boundary. To avoid such effects, one can follow the method, according to which the process of exposure is divided into two stages [17]. For each stage, the corresponding value of the laser pulse energy is determined. Switching the processing mode is carried out at the boundary located inside the sample.

Fabricating resonator of tuning fork gyroscope. The following processing parameters have been chosen for exposing the resonator contour based on the data obtained in the course of the experiment: speed 1.0 mm/s; the maximum pulse energy (on the lower plane of the sample) is 1.5 μJ; the minimum pulse energy (on the upper plane of the sample) is 1.0 μJ.

A program was developed in the *G*-code and the simulation of the technological process in the CNC-software was performed before the production.

The total eposing time of the resonator contour was ~ 10 hours. Processing was carried out in two stages: at the depth of focus from the bottom plane of the sample to 400 μm with a maximum energy, and from 400 μm to the surface with a minimum energy.

The obtained sample was etched under conditions similar to the described experiment. The results of the processes of exposure and etching are presented at Fig. 7.



a *b*

Fig. 7. Production of the tuning fork gyroscope resonator. The exposed resonator contour (*a*). The resonator after chemical etching (*b*)

Later the similar results were obtained at simultaneously increasing the speed of movement of the coordinate table to 2.5 mm/s and the repetition rate of pulses to 200 kHz and maintaining other processing parameters. Thus, the exposure time was reduced to 4 hours. It have to be noted that the performance of the process in this case is mainly limited by the capabilities of the mechanical coordinate table used in the experimental setup. The process may be accelerated by 1–2 orders if high-speed coordinate tables and laser beam scanning systems are used, such as, for example, a 3D microscanner [25].

Discussion of the obtained results. The through grooves by depth about 1.05 mm were obtained during the process of fabricating of a resonator sample. Taking into account that process of chemical etching proceeded along with two sides of a plate, the etching rate of the modified material r_s in 5 % solution of hydrofluoric acid was from 27.5 to 68.8 μm/h. The estimation of thickness of initial sample and the obtained sample of the resonator allows to conclude that the etching rate of the initial material r_0 was about 1.2 μm/h at the same time.

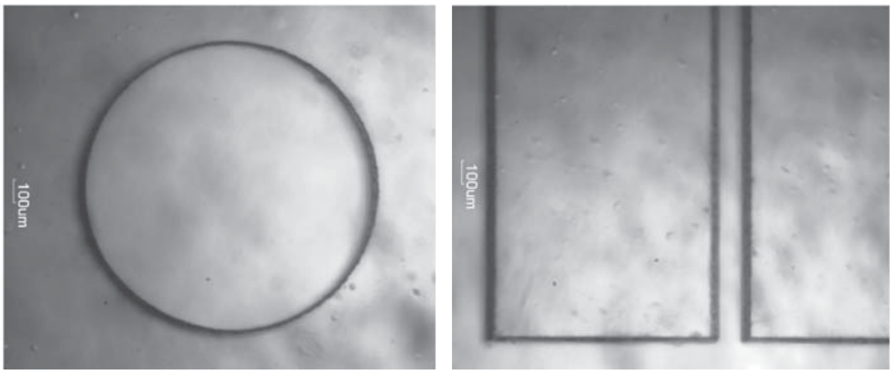
The quality of the selective etching process is estimated on selectivity of s — the dimensionless parameter characterizing the efficiency of etching of the modified material in comparison with the initial one. The etimation of selectivity can be obtained from the expression [16, 22]:

$$s = \frac{r_s + r_0}{r_0}. \tag{2}$$

On the basis of the beforesaid, within the offered technological process the average value of selectivity is in range from 22.9 to 57.3 that in general correlates with the results received under comparable conditions in [16].

The observed imprecision in results can be explaned by errors at measurements of sample thickness and the obtained detail, distinctions of the modes of interaction of laser radiation with substance and etching, properties of the used fused silica grades, influence of other factors. In particular, in this work the objective lens with NA 0.25 was applied to focusing of laser radiation, whereas in the considered sources there were high-quality lenses with NA not less than 0.4, as a rule.

The fabricated sample of the resonator was studied using an optical microscope equipped with a 3-megapixel Motic Moticam 3 camera. Based on the obtained images of the resonator elements (Fig. 8), it can be concluded that the maximum deviations of the sizes from the nominal values (~ 50 μm) have been observed near the upper and lower boundaries. Thus, the slope of the walls of the through openings and grooves does not exceed 3°.



a *b*

Fig. 8. Resonator elements under a microscope. The central opening (*a*) and resonator legs (*b*)

Conclusion. Taking into account that the resonator of a tuning fork gyroscope of the satisfactory sizes and form has been obtained as a result of fabrication, the conducted researches confirmed a possibility of application of the directed etching of fused silica method for production of sensitive elements of precision instruments. Besides, the received estimates of etching rate of the modified fused silica in 5 % solution of hydrofluoric acid (from 27.5 to 68.8 μm/h) and selectivity of process (from 22.9 to 57.3) correlate with the results stated in other researches well.

Nevertheless, the full application of a method in practice requires the further study of technological process including specification of the modes of fused silica modification for obtaining the best selectivity, the solution of the questions connected with ensuring uniformity of etching, exposing process acceleration, etc. Studying of the obtained samples on existence of defects and undesirable changes of initial material in boundary zones, definition of their influence on quality of a product is also necessary.

It is also necessary to note that the most perspective continuation of the work from the practical point of view is the production of quartz accelerometers pendulums as one of the most mass products of precision instruments industry.

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