#### 10

# Self-organizing half-wave gratings on the surface of silica glass

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Received on December 20, 2021 Revised on December 20, 2021 Accepted on December 30, 2021

The interaction of femtosecond laser pulses with the surface of silica glass has been studied. As a result of interference between the incident radiation and surface plasmon polaritons, the formation of self-organizing subwavelength periodic structures with a period of 250 nm was observed. The minimum pulse energy at which recording occurs without surface ablation has been revealed.

Keywords: direct laser recording, femtosecond laser pulses, silica, nanogratings.

DOI: 10.21883/EOS.2022.04.53732.58-21

# Introduction

In recent years, the field of direct laser-optical recording of functional elements in the volume and on the plane of wide-gap dielectrics has been actively developed [1]. In the process of interaction between the laser pulse and the material, nonlinear absorption occurs [2]. As is known, in order to implement nonlinear scattering, the electric field strength of laser pulse must be comparable with the strength of the field that binds electrons in atoms. To achieve this level of intensity, the very high intensity of laser radiation is required. Depending on the radiation parameters, different modifications can be obtained in dielectrics [3-5]: areas of compression or decompression of the material, point defects, etc. The use of laser in dielectric processing is used in fields such as waveguide recording [6], birefringent element fabrication [7], 3D optical memory [8], as holographic recording, 5D optical memory and 3D holographic memory [9,10].

Quartz glass is an excellent material for the production of optical components for the visible and near infrared (IR) regions of the spectrum. Their operation is possible up to a temperature of 950°C provided there is no thermal shock [11]. Quartz glasses are widely used in the production of high-quality optical products from simple lenses to complex elements with multilayer dielectric coatings, such as beam splitters. Possessing sufficient inertness to many substances, including almost all acids, quartz glasses also find their application in aggressive environments. The dielectric properties, together with very high electrical susceptibility and low thermal conductivity over a wide temperature range, help them to be used as thermal and electrical insulators.

In this work, the possibility of creating surface diffraction gratings with a subwave period on quartz glass with direct femtosecond laser recording of arrays of microlines is considered.

# **Experimental units**

The unit used to record surface structures includes the oscillator, multipass amplifier, output compressor, and acoustooptic modulator (Fig. 1). The source of femtosecond laser pulses was a Satsuma [12] laser system based on ytterbium fiber laser pumped by LED radiation with a maximum power at wavelength of 850 nm. The control is carried out through the program on an external computer. The central wavelength of the generated radiation is 1030 nm, the pulse duration is 300 fs. At the output



**Figure 1.** Scheme of the experimental installation used to record surface structures: RM — mirrors, L — lens with focal length 200 mm.



**Figure 2.** Optical images of a section of the quartz surface with recorded lines obtained without polarizers (a) and in crossed polarizers (b). The images show the energy values of the recording pulses: 20, 22, 24 nJ.



Figure 3. SEM images of the results of recording on a quartz surface using femtosecond laser at pulse energy of 48 (*a*), 40 (*b*), 32 (*c*), 24 (*d*), 22 nJ (*e*).

of the system, the maximum energy per pulse can be up to  $10\,\mu$ J.

Series of lines about  $200\,\mu\text{m}$  long were recorded at a rate of  $20\,\mu\text{m/s}$  for pulse energies of  $20-48\,\text{nJ}$  and a pulse repetition rate of  $100\,\text{kHz}$ . The object lens

with a numerical aperture  $NA\,{=}\,0.65$  was used for recording.

To obtain a microscale image of the surface the high vacuum field emission scanning electron microscope TESCAN VEGA 3 with tungsten cathode is used. The study of the chemical and phase compositions of materials with submicron resolution in area and depth was carried out by Raman spectroscopy (RS) using a Confotec MR350 confocal scanning Raman microscope. The surface structures were studied when a 532 nm laser line excited a  $1 \,\mu$ m spatial region specified by the numerical aperture of the object lens NA = 0.75.

### Experimental results and discussion

#### **Optical images**

Optical images of lines recorded on the surface of quartz, obtained without polarizers and in crossed polarizers, are shown in Fig. 2. At the threshold energy of the recording pulses 20 nJ, periodic surface structures without crater are formed. At pulse energies exceeding the surface damage threshold, spall ablation occurs with the formation of a crater and the shedding of glass particles (glow in crossed polarizers, Fig. 2, b).

### Scanning electron microscopy

By scanning electron microscopy was obtained images of periodic self-organizing structures indistinguishable in optical images, written on the surface of quartz using a femtosecond laser (Fig. 3). Spatial period of these structures is about 250 nm. Their appearance is associated with the interference between the incident femtosecond laser radiation and surface plasmon-polaritons  $K \ge 1/\lambda_{\text{las}}$ (lattices with near-wavelength periods) [13,14]. For the appearance of surface plasmon-polaritons, the permittivity must be negative. Since the photoexcitation of quartz results in its metallization, the permittivity of quartz becomes negative [15].

#### **Raman scattering**

Raman spectroscopy is an effective method for determining structural modifications in glass. The main feature of the Raman spectrum of quartz glass is a wide band centered at  $\sim 420 \,\mathrm{cm^{-1}}$ , associated with the rocking and bending of the Si–O–Si bond. Bands at 478, 605 and 805 cm<sup>-1</sup> reflect vibrational motions of oxygen atoms in four- and three-membered ring structures [16].

To assess modifications in glass after femtosecond laser treatment, Raman scanning was performed in the region of surface structures. The  $478 \text{ cm}^{-1}$  band refers to three-membered ring bonds in the quartz lattice (Fig. 3). The change in the ratio between the amplitudes of the peaks at 420 and  $478 \text{ cm}^{-1}$  can be associated with photoinduced breaks in strained bridge bonds Si–O–Si.

# Conclusion

In this work, we have demonstrated the possibility of creating self-organizing structures with a subwave period



Figure 4. Raman spectra of an unprocessed surface and a structure with a period of  $\sim 250\,\text{nm}$  produced by pulses with an energy of 22 nJ.

on a quartz surface using femtosecond laser processing. The period of the resulting submicron structures was about 250 nm. In this work, the threshold modes of structuring of the quartz glass surface were determined, at which spall ablation does not occur. Based on the results of Raman spectroscopy, one can draw conclusions about the change in the concentration of oxygen ring bonds in quartz and the modification region.

#### Funding

The study was supported by a grant from the Russian Science Foundation (project  $N_{\mathbb{P}}$  20-71-10103).

# **Conflict of interest**

The authors declare that they have no conflict of interest.

# References

- R. Woods-Robinson, Y. Han, H. Zhang, T. Ablekim, I. Khan, K.A. Persson, A. Zakutayev. Chem. Rev., **120** (9), 4007–4055 (2020). DOI: 10.1021/acs.chemrev.9b00600
- D.V. Kartashov, A.V. Kirsanov, A.M. Kiselev, A.N. Stepanov, N.N. Bochkarev, Yu.N. Ponomarev, B.A. Tikhomirov. Opt. Exp., 14 (17), 7552–7558 (2006).
   DOI: 10.1364/OE.14.007552
- [3] N. Sanner, O. Utéza, B. Bussiere, G. Coustillier, A. Leray, T. Itina, M. Sentis. Appl. Phys. A, 94 (4), 889–897 (2009) DOI: 10.1007/s00339-009-5077-6
- K.I. Popov, C. McElcheran, K. Briggs, S. Mack, L. Ramunno. Opt. Exp., 19 (1), 271–282 (2011).
   DOI: 10.1364/OE.19.000271
- [5] S. Gräf Clemens, K. Frank, A. Müller. Materials, 10 (8), 933 (2017). DOI: 10.3390/ma10080933
- [6] A.A. Khalil, P. Lalanne, J.-P. Berube, Y. Petit, R. Vallee, L. Canioni. Opt. Exp., 27 (22), 31130-31143 (2019).
   DOI: 10.1364/OE.27.031130

- S.S. Fedotov, A.G. Okhrimchuk, A.S. Lipatiev, A.A. Stepko, K.I. Piyanzina, G.Yu. Shakhgildyan, M.Yu. Presniakov, I.S. Glebov, S.V. Lotarev, V.N. Sigaev. Opt. Lett., 43 (4), 851-854 (2018). DOI: 10.1364/OL.43.000851
- [8] K. Miura, J. Qiu, S. Fujiwara, S. Sakaguchi, K. Hirao. Appl. Phys. Lett., 80 (13), (2002).
- [9] B. Eggleton, B. Luther-Davies, K.N. Richardson. Nat. Photon., 5 (3), 141–148 (2011).
- [10] F. Brückerhoff-Plückelmann, J. Feldmann, C.D. Wright, H. Bhaskaran, W.H. Pernice. J. Appl. Phys., **129** (15), 151103 (2021). DOI: 10.1063/5.0042549
- [11] A.P. Velmuzhov, M.V. Sukhanov, M.F. Churbanov, T.V. Kotereva. Inorg. Mat., 54 (9), 925–930 (2018).
- [12] A.E. Rupasov, P.A. Danilov, M.P. Smaev, M.S. Kovalev, A.S. Zolot'ko, A.A. Ionin, S.I. Kudryashov. Opt. Spectrosc., 128 (7), 928–931 (2020)
   DOI: 10.1134/S0030400X20070188
- J. Wu, Y. Zhang, L.QiLi, Y. Ren, Q. Lu, L. Wand, F. Chen. Results in Physics, 21, 103814 (2021).
   DOI: 10.1016/j.rinp.2021.103814
- [14] S. Kudryashov, A. Levchenko, P.A. Danilov, N. Smirnov, A.A. Rudenko, N. Melnik, N. Busleev, A.A. Ionin. Appl. Phys. Lett., **115** (7), 073102 (2019). DOI: 10.1063/1.5114630
- [15] P.A. Danilov, S.I. Kudryashov, A.E. Rupasov, N.A. Smirnov, E.
  A. Oleynichuk , A.S. Rivnyukc, R.A. Zakoldaev. JETP Lett., 113 (10), 622–625 (2021).
   DOI: 10.1134/S0021364021100052
- [16] S.O. Kucheyev, S.G. Demos. Appl. Phys. Lett., 82 (26), 3230 (2003).