

07,04

Correlated accumulation of microcracks in impact damaged surface of α -quartz amorphized by Ar^+ ions implantation

© I.P. Shcherbakov, A.E. Chmel[✉]

Ioffe Institute,
St. Petersburg, Russia

[✉] E-mail: chmel@mail.ioffe.ru

Received July 5, 2021

Revised July 5, 2021

Accepted July 7, 2021

The microcrack accumulation in impact damaged α -quartz plates prior to and after the Ar^+ -ion implantation was studied with the acoustical emission (AE) method. The 40 keV implantation doses of 10^{14} and 10^{16} ion/cm² were applied. The statistical analysis of the energy distribution in impact-induced AE time series detected in unirradiated samples showed a random (poissonian type) accumulation of defects which is specific for mechanical destruction of homogeneous brittle materials. The energy distribution in the AE time series excited when damaging the preliminary implanted samples followed a power law typical for the fracture of heterogeneous solids such as rocks, ceramics, etc. The optical photography evidenced a transition from brittle (prior to implantation) to ductile damage formation caused by the disturbed interconnectivity of the crystalline structure in irradiated specimens.

Keywords: ion implantation, alpha quartz, impact damaging, acoustical emission.

DOI: 10.21883/PSS.2022.14.54340.160

1. Introduction

Modification of a surface layer of alpha quartz (α -quartz) at irradiation with particles (ions, neutrons, electrons) was a subject of multiple studies in the second half of the last century due to rapid development of microelectronics [1,2], and this science and technology direction is in demand now [3], particularly due to development of electromechanical microsensor devices [4], applied under radiation conditions [5]. This study is related to a change of mechanical destruction mechanism under field impact load of α -quartz, preliminary implanted with ions of Ar^+ . Using acoustical emission (AE) method, the statistics of microcracks accumulation in material surface layer depending on irradiation dose (D) was analyzed.

2. Specimens and equipment

Specimens in the form of polished plates of crystalline quartz, made using hydrothermal method, had planes, normal to Z axis of crystal, that were processed at linear accelerator with doses of 10^{14} and 10^{16} ion/cm² Ar^+ with energy of 40 keV. Specimens damaging was performed by striker established of a weight, falling onto a pointed steel peen, mounted on specimen surface. Since α -quartz has strongly pronounced expressed piezoelectric properties, for AE detector we used a plate of high-sensitive piezoceramics $\text{Pb}(\text{Zr}_x\text{Ti}_{1-x})\text{O}_3$, piezoelectric coefficient of which is almost in two orders of value higher than that of higher than for α -quartz. AE signals were coming to analog-digital

converter ASK-3106 input and were saved in digital form on PC. Duration of all signal types collection was 1 ms; time resolution — 20 ns.

3. Results and discussion

Figure 1 shows time-based sweeps of AE signals produced by specimens surface impact at $D = 10^{14}$ and 10^{16} ion/cm². It is seen, that AE generation from implanted layer is significantly shorter, than that from untouched surface. This effect can be explained by surface densifying of SiO_2 and silicate glasses at ions implantation [6–8], that shortens the development of damaging process. Molecular mechanism of compaction induced by ions consists in reduction of angles of $\text{Si}-\text{O}-\text{Si}$ with formation of 3- and 4-membered siloxane rings [9].

Pulse energy (E) is proportional to intensity (amplitude square of amplitude, A^2) of AE signal. Figure 2 shows the distributions a number of pulses depending on energy in pulse constructed from based on time series presented in Fig. 1. Distributions are built as dependencies $N(E > \varepsilon) \propto \varepsilon$, where N — number of pulses, which energy E is higher than value of some „threshold“ ε .

In other words, parameter ε successively takes values of energy in the detect pulses within interval from 0 to 1 ms (horizontal coordinate), and number of pulses, which energy E exceeds the current value of ε that is plotted along the vertical axis.

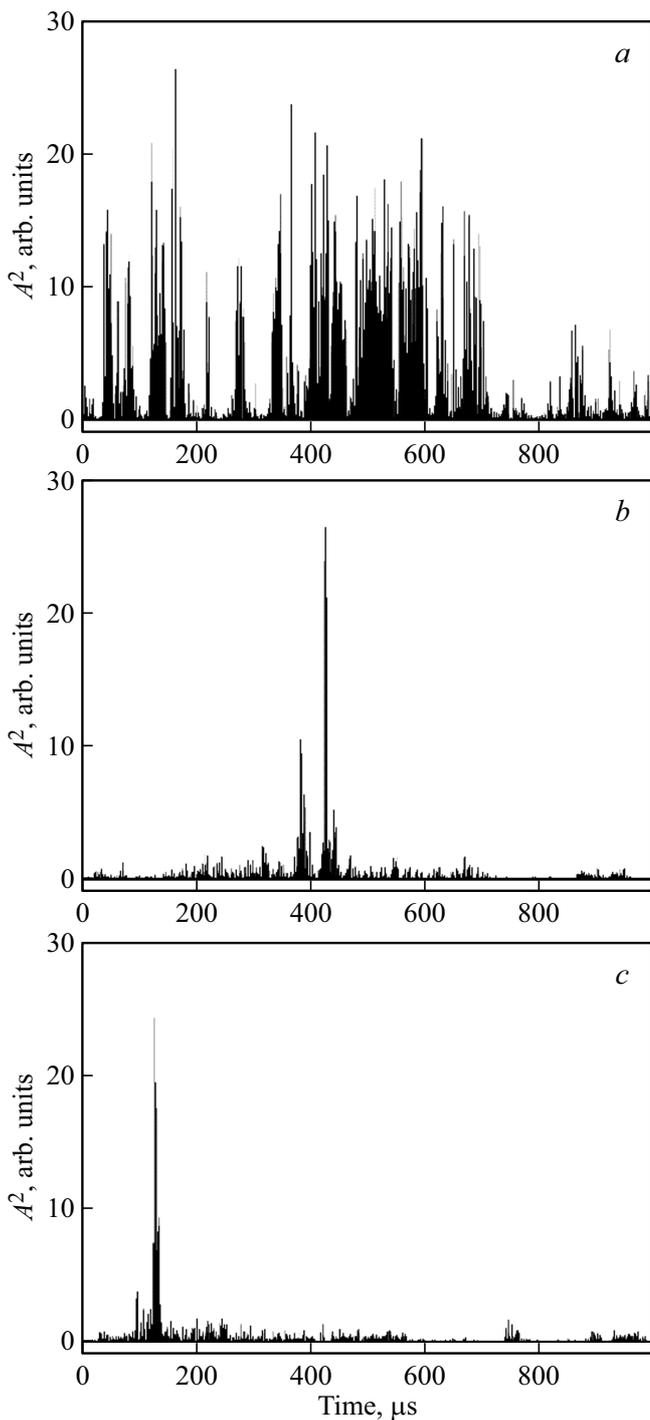


Figure 1. Time bases of AE from α -quartz specimen, stimulated with localized impact; $D = 0$ (a), 10^{14} (b), 10^{16} ion/cm² (c).

The same data are plotted in two coordinates — semilogarithmic (Fig. 2, a, b, c) and double logarithmic (Fig. 2, d, e, f). Pulses distribution by energy in initial specimen ($D = 0$) is presented with a straight line, corresponding to correlation

$$\log_{10} N(E > \varepsilon) \propto -a\varepsilon, \quad (1)$$

where a is the line slope. Correlation (1) is equivalent to the Poisson-type exponential law

$$N(E > \varepsilon) \propto \exp(-a\varepsilon), \quad (1a)$$

which is characteristic for random events. Microcracks nucleation happens independently of each other, since there is no required concentration of potentially „weak spots“ for defects interaction in homogeneous solid. In double logarithmic coordinates the distribution for non-irradiated specimen (Fig. 2, d) can not be expressed by means of some elementary function.

Another pattern of AE signals distribution was observed at impact damaging of specimens after ion implantation. In semilogarithmic scale, the compound curves appeared without semi-log-straight sections and not corresponding to any elementary function. At the same time, in double logarithms the distributions of $N(E > \varepsilon)$ versus ε present log-linear dependencies

$$\log_{10} N(E > \varepsilon) \propto -b \log_{10} \varepsilon. \quad (2)$$

By working out the logarithms from correlation (2), we obtain a distribution of energies in AE pulses in the form of a power law

$$N(E > \varepsilon) \propto \varepsilon^{-b}. \quad (2a)$$

Slowly (as compared to exponent) damped power function reflects the long-range interactions between microcracks, when single defect formation increases possibility of new defects nucleation in surrounding „weak spots“, i.e. damage development happens as per cooperative scenario. Under implantation process, the weak spots are formed in the places of silicon-oxygen carcass ruptures that was earlier called „amorphization“, of α -quartz surface layer [1,10]. This term has a specific nature, since the crystalline structure is destroyed not due to the formation of unordered tetrahedron ring compounds of SiO_4 as in the structure of melted SiO_2 but due to multiple ruptures of interatomic bonds. Thus, a reservoir of interacting „weak spots“ is formed, providing a cooperative accumulation of microcracks under mechanical impact.

In our case, the amorphization effect of the mentioned type was also manifested in destruction morphology change. In optical photography of the specimen before implantation (Fig. 3, a), the localized destruction has a brittle nature with visible cracks along crater perimeter. After implantation with dose of 10^{16} ion/cm² there were no brittle cracks (Fig. 3, b), and crater had more regular round shape specific for ductile fracture of material with low structural connectivity. It should be noted that despite the fact, that the modified layer at argon ions implantation into quartz is only several hundreds angstrom [11], its presence significantly changed the morphology of damage with a scale of ~ 1 mm.

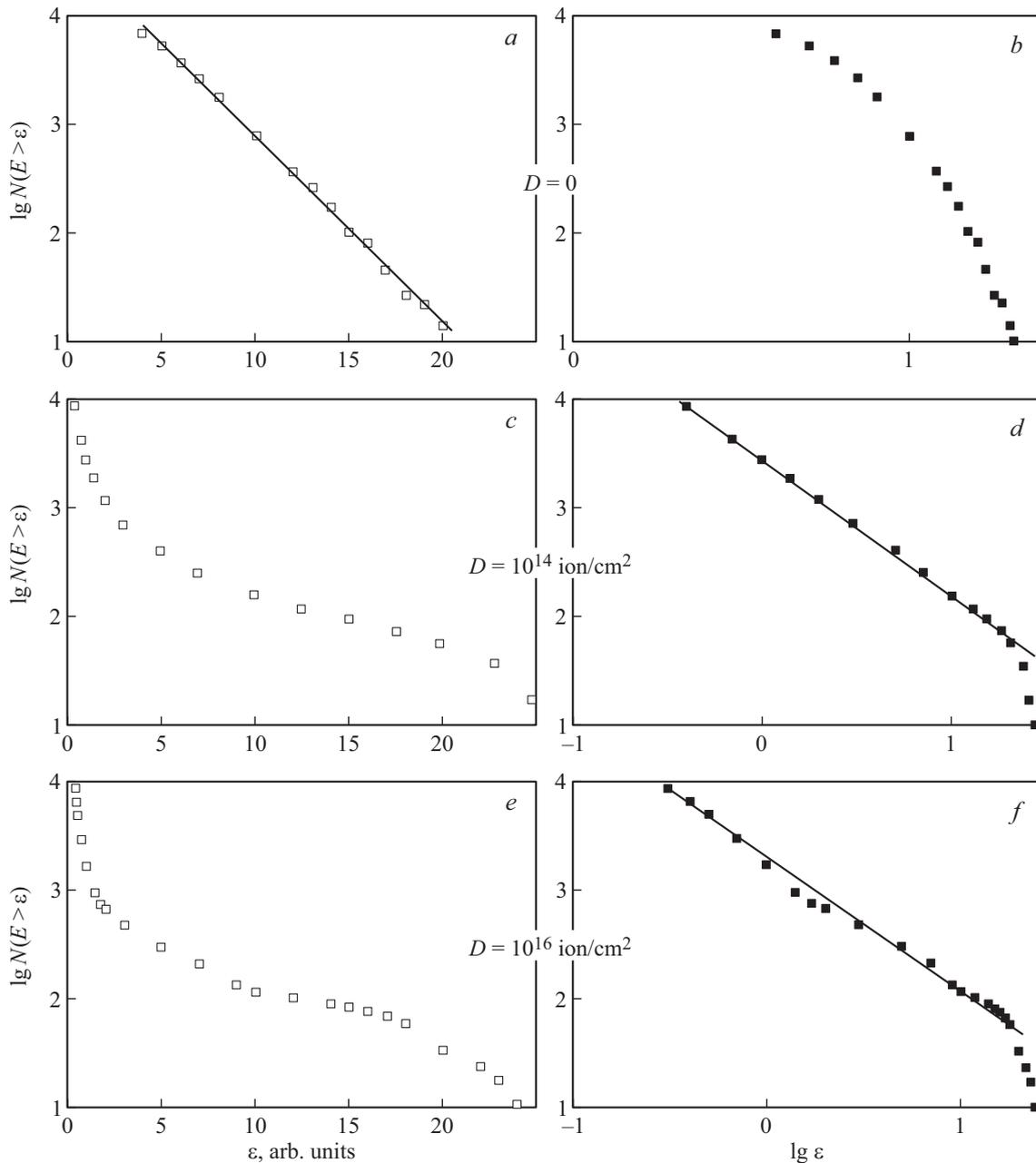


Figure 2. Energy distributions in AE pulses calculated from time series presented in Fig. 1; *a, b, c* — semilogarithmic coordinates; *d, e, f* — logarithmic coordinates.

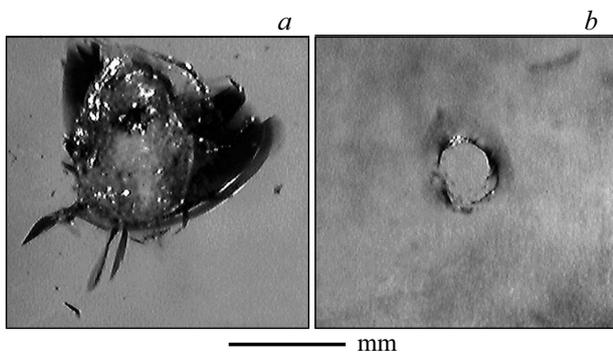


Figure 3. Optical photographs of the damaged surface before implantation (*a*) and after irradiation with a dose of $D = 10^{16}$ ion/cm² (*b*).

4. Conclusion

Implantation of inert argon ions results in degradation of crystalline order of α -quartz in the form of multiple ruptures of interatomic bonds. Analysis of the microcracks accumulation, as a result of mechanical impact, active in acoustical emission, showed that destruction of surface layer, disrupted by irradiation, occurs as per type, characteristic for heterogeneous materials. High concentration of atomic defects contributes to the further interaction of developing microcracks, resulting in energy correlated process of macroscopic damage formation. Despite the small depth of ions path (hundredths of micron), the morphology of

localized damage of irradiated surface significantly differed from initial one.

Conflict of interest

The authors declare that they have no conflict of interest.

References

- [1] H. Fischer, G. Götz, H. Karge. *PSS* **76**, 249 (1983).
- [2] G.W. Arnold *Nucl. Instrum. Meth. B.* **65**, 213 (1992).
- [3] Sh. Zhou, J.W. Ju. *Int. J. Damage Mech.* **29**, 923 (2020).
- [4] B. Li, C. Li, Y. Zhao, Ch. Han, Q. Zhang. *Micromachines* **11**, 724 (2020).
- [5] B. Wang, Y. Yu, I. Pignatelli, G.N. Sant, M. Bauchy. *J. Chem. Phys.* **143**, 024505 (2015).
- [6] B. Li, X. Xiang, W. Liao, S.B. Han, J.X. Yu, X.L. Jiang, H.J. Wang, M. Mushtaq, X.D. Yuan, X.T. Zu, Y.Q. Fu. *Appl. Surf. Sci.* **471**, 786 (2019).
- [7] G. Battaglin, G.W. Arnold, G. Mattei, P. Mazzoldi. *J. Appl. Phys.* **85**, 8040 (1999).
- [8] E. Szilágyi, I. Bányász, E. Kótai, A. Németh, C. Major, M. Fried, G. Battistig. *Radiat. Eff. Defects Solids* **170**, 229 (2015).
- [9] K. Fukumi, A. Chayahara, M. Satou, J. Hayakawa, M. Hangyo, Sh.-I. Nakashima. *Jpn J. Appl. Phys.* 29. Part 1, 905 (1990).
- [10] L. Douillard, J.-P. Duraud. *J. Phys. III France.* **6**, 1677 (1996).
- [11] C.R. Fritzsche, W.R. Rothmund. *J. Electrochem. Soc.* **119**, 1243 (1972).