DAMAGE TO SILICATE GLASS CONTAINING IMPURITIES FROM INTENSE RADIATION

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A study has been made of the characteristics of impurities in crystalline quartz and glasses. Threshold values have been determined for glasses in relation to external mechanical loads. The parameters of subthreshold pulses in a damaging series are related to the number of them.

The number, sizes, and characteristics of impurities largely determine the damage to a transparent insulator by high-intensity radiation [1, 2]. Experimental results do not always have a simple explanation from the view point of the thermal theory [3, 4]. Further research is therefore required on impurities in transparent insulators and their role in damage.

We examined specimens of natural quartz crystals (colorless and smoky), K-8 glass, and fused silica. The compositions and amounts of impurities were examined by atomic-absorption spectroscopy (with an instrument of Perkin Elmer 403 type), by spectral analysis (PGS-2, Carl Zeiss), by x-ray spectroscopy (PW-1600), and with an electron probe (Hitachi, XMA-5B). The presence of Ba was determined with an ARL instrument made in the USA. The specimens were prepared in accordance with the requirements for the methods and instruments.

The compositions indicated by spectral analysis and x-ray spectroscopy were in agreement (the averaged results are given in Table 1). The percent compositions of the impurities in the quartz specimens coincided with tabulated data [5], and the minor differences are probably associated with the better apparatus (high measurement accuracy). The percent compositions of the impurities in the quartz were dependent on the deposit, the levels being about 0.08-0.12%. The compositions of the impurities in the glasses were similar to those for the quartz, although there were differences for B and Na at about 1%, K and Ca at about 0.06%, and Ba at about 0.2%, which exceed the contents of these elements in the quartz. The overall amounts of the impurities in the glasses were 2-3%. Electron-probe examination (minimum spot diameter 1 μm) showed that the glasses have uneven distributions: in regions of size about 1 μm, the impurity concentrations might exceed the mean by about a factor 10. Mechanical impurities (or foreign absorbing inclusions FAI) of size 0.5-1 μm were not observed.

According to [5], the impurity elements may enter into the lattice of quartz and also into mechanical inclusions or FAI lying in defect channels in α quartz, whose diameter is 0.02-0.05 μm. These defect channels tend to accumulate foreign components [5]. The glasses usually contain minute crystals of α quartz and α cristobalite of concentration about 10^9 cm^{-3} (incomplete decomposition of the crystalline raw material and the formation of crystallization centers) [6, 7]. These crystals are probably responsible for the nonuniform impurity distributions in the glasses. The impurities thus enter the lattices of the quartz and glasses and may also be present as mechanical impurities of size 0.02-0.05 μm. Foreign impurities of size 0.5-1 μm were not observed. The crystals of cristobalite and quartz in the glasses may also be sites of additional impurity accumulation, which affect the absorption coefficient.

The amounts, compositions, and physicomechanical characteristics of the impurities in K-8 glass were approximately the same as those in fused silica. It might therefore seem that the threshold pulse intensity I_p causing damage should be the same. Measurements have been made on I_p for various glasses at various wavelengths λ and focal lengths F, but the I_p

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TABLE 1. Impurity Compositions and Concentrations

Element	Crystalline quartz			Smoky quartz		Fused silica		K-8 glass	
	spectral method	x-ray method	tabulated data	spectral method	tabulated data	spectral method	x-ray method	spectral method	x-ray method
B	0,003	—		0,002		1	—	1	—
Pb	0,0005	—		0,0005		0,0005	—	0,0005	—
Al	0,003	0,004	0,01	0,003	0,01	0,001	0,002	0,003	0,005
Fe	0,01	0,01	0,01	0,01	0,001	0,02	0,03	0,02	0,01
Cu	0,001	—	0,0001	0,0001	0,01	0,001	—	0,0005	—
Na	0,01	0,001	0,0004	0,01	0,003	1	0,6	1,1	1,1
K	0,01	0,001	0,0002	0,01		0,6	0,7	0,8	0,09
Zn	0,005	0,006		0,005		0,005	0,006	0,005	0,006
Ba	0,03	0,02		0,03		0,2	0,02	0,2	0,3
Sr	0,01	0,01		0,01		0,01	0,01	0,01	0,007
Ti	0,0005	0,0005	0,003	0,0005		0,0005	0,0015	0,0005	0,002
Mg	0,0003	0,0003	0,01	0,0003	0,003	0,001	0,001	0,0005	0,001
Ca	0,05	0,07	0,01	0,06	0,003	0,03	0,03	0,04	0,05

are not comparable. In our experiments, we determined I_p for fused silica and K-8 glass. The radiation (λ = 1.06 and 0.69 μm, $\tau \approx 2 \cdot 10^{-8}$ sec) was focused within the specimen (F = 40 mm) of size 30 × 15 × 15 mm. The threshold was defined as the minimum intensity producing visible damage on a single shot. We found that I_p for fused silica exceeded I_p for K-8 glass by a factor of 3-4 at λ = 1.06 μm, whereas the values of I_p were approximately equal at λ = 0.69 λm.

These results can be explained on the micromechanical damage model MMDM [8]. We assume that α-quartz crystals, not cristobalite ones, are the crystallization centers in fused silica. On heating α quartz to 846°K, there is a phase transition to β quartz, and the volume increases by 4.7% [5]. We assume that the absorption coefficients for α quartz (with mechanical impurities) are identical, as are the concentrations (10^9 cm^{-3}). Then I_p is determined by the heating of the quartz crystals on exposure to λ = 1.06 μm to 846°K (on heating the cristobalite in K-8 glass to 540°K) [8]. Then I_p for fused silica should be larger by a factor 2.2. I_p at λ = 0.69 μm is determined by the thermal destruction of the glass [8], and the thresholds for K-8 glasses and fused silica should be identical.

According to the thermal theory [1, 2], mechanical stress should not influence I_p. The opposite results were obtained in [3, 9]. Here we supplement the results of [3, 9] and find an explanation for them. Hydrostatic compression σ_c was produced up to 10^2 MPa in a specially constructed chamber. There were not less than 40 specimens of K-8 glass and fused silica in each series (three or four specimens for crystalline quartz). The specimens for compression were rounded in shape and of size about 20 mm (about 10 mm for quartz), while for stretching (with a special machine) they were of standard shape with a working part of 20 × 10 × 40 mm. The radiation (λ = 0.69 or 1.06 μm, $\tau \approx 2 \cdot 10^{-8}$ sec) was focused within the specimen by a lens with F = 40 mm. We determined I_p with single shots and damage temperature by the method of [10]. The mean values were derived.

On compression, I_p increases for both λ (Fig. 1). The change in I_p/I_{po}, where I_{po} is the threshold for σ = 0, as σ_c increased at λ = 0.69 μm was always less than that at λ = 1.06 μm. Variation in I_p/I_{po} with σ_c was the same for the different glasses. The thermal destruction temperature increased with $I_p(\sigma_c)$ to 10^3-$1.5 \cdot 10^3$ K. With λ = 1.06 μm the temperature was always higher than that with λ = 0.69 μm. Stretching σ_s had virtually no effect on I_p or the thermal destruction temperature (λ = 1.06 μm). There was a slight reduction in I_p with λ = 0.69 μm. The results were recorded up to σ_s = 20 MPa, since at σ_s = 25-30 MPa the cracks grew under the external load to damage the entire specimen. We determined I_p for quartz crystals only in compression (λ = 1.06 μm). The variation in I_p/I_{po} was similar to that for the glass (at $\sigma_c = 10^2$ MPa by a factor 1.33). The damage occupied the entire focal volume. The temperature varied in the same way as shown in Fig. 1.

An explanation can be given in terms of MMDM [8], where I_p is determined by two processes: the formation of a hazardous defect HD (the quartz or cristobalite crystals are heated to the phase-transition temperature, which corresponds to I_1) and thermal destruction of the glass, $I_p \geqslant I_1$. The HD formation time is determined by the lifetimes of stressed bonds ($\Theta(f, T)$) and by the cristobalite concentration n: $t_{hd} = \Theta/n$. The stress around the cristobalite f is reduced by compression and I_1 increases. Let t_{HD} remain constant. To retain the initial

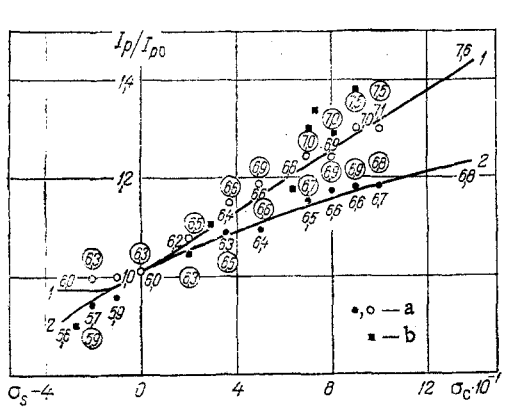

Fig. 1

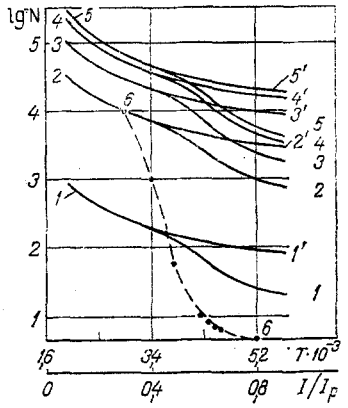

Fig. 2

Fig. 1. Effects of radiations with different wavelengths λ having $I = I_p$. Calculated values of I_p: 1-1) λ = 1.06 μm; 2-2) 0.69. Temperatures in 10^3 K enclosed in circles; a) experimental data, open circles λ = 1.06 μm, filled circles 0.69; b) experimental data of [3]; σ_c in MPa.

Fig. 2. Dependence of the number of pulses on the intensity with various spacings: 1) 10^{-5} sec; 2) 10^{-4}; 3) 10^{-3}; 4) 10^{-2}; 5) $2 \cdot 10^{-2}$ sec; 6) experimental curve of [4], repetition time $2 \cdot 10^{-2}$ sec. Dashed line — calculation without allowance for additional heating.

value of Θ on compression, it is necessary to increase the temperature of the cristobalite by increasing I_1, which leads to an increase in I_p when $I_p = I_1$ (λ = 1.06 μm). Curve 1 of Fig. 1 shows I_p calculated as a function of σ_c. On compression from 0 to 140 MPa, the cristobalite heating temperature increases from 540 to 650°K (we assume that the absorption coefficient remains unchanged), which is below the melting point of the silicate glass (about 1300°K). The thermal destruction temperature is determined by the absorption by the tribobreakdown plasma [8] and the calculated values of I_p and T (Fig. 1) agree with the measured ones. An increase in temperature produces an increased pressure in the crack cavity due to the mass and temperature of the gas. This pressure should be not less than $p + \sigma_c$, where p is the pressure required for the existence of a mobile equilibrium crack of size about 50 μm (at $\sigma = 0$). The calculated and measured temperatures show that cracks can exist. The values of I_1 coincided with I_p for all σ_c. Any reduction in Θ and possible reduction in I_p will be limited by I_1 as required to heat the cristobalite to T = 540°K (or T = 846°K for quartz). The calculated values agree with experiment for λ = 1.06 μm, where $I_p = I_1$ (curve 1 of Fig. 1). When I_p is determined by thermal destruction (λ = 0.69 μm), it is necessary to meet the condition for cracking at the given mechanical loads. An increase in I_p increases the thermmal-destruction temperature and determines the pressure change in the crack. Curve 2 shows the change in I_p for this case. The agreement between the calculated and measured values of I_p and the temperatures is satisfactory.

Curve 1 of Fig. 1 shows the processed I_p from [3] for GLS 1 glass in relation to external load, where the values agree satisfactorily with the calculated curve. In [3] we find a somewhat different explanation based like the MMDM on the kinetic theory of strength. In [9] one finds maximal values of I_p, which were determined by bringing the focal volume into a region with the minimal cristobalite concentration, which leads to an increase in I_p (for σ = 0) [8] and which resulted in no damage for $\sigma_c > 60$ MPa (at the power used). In the experiments described above, the average cristobalite concentration was 10^9 cm^{-3}, which governs the size of the focal volume. In that case, the compression required to prevent damage is about 10^3 MPa or more. In [11], an explanation was given for the dependence of I_p on σ on the basis of the change in the width of the forbidden band in the glass with pressure. Here we have used the MMDM to explain $I_p = f(\sigma)$, but we do not rule out the possibility of invoking the model of [11].

Results have been given [4] on the repeated action of subthreshold intensities, which are difficult to explain within the framework of [1, 2]. We consider this within the framework of [8] on the basis of the cristobalite structure (octahedron), and around the edges of

the crystal the glass is in a state of stress because of the change in curvature. All the exposed crystals are heated by each pulse (linear absorption). There is a linear increase in the volume of the cristobalite by 3.7% between 293 and 539°K, while at T = 540°K the volume increases stepwise by 5.8% [5]. This volume increase produces stresses in the glass. These interact with those already existing around the edges, which leads to microcracking and tribobreakdown. The tribobreakdown plasma may absorb part of the pulse, which heats it and the surrounding glass. We envisage that individual cracks are produced within a time of about 10^{-9} sec. Intensities $I \geqslant 0.7I_p$ produce microcracks around the cristobalite at a concentration of 10^9 cm^{-3}, while for $0.1I_p \leqslant I \leqslant 0.3Ip$ the corresponding value is 0 and for $0.4\ I_p < I < 0.6I_p$ we get a transitional value. The tribobreakdown plasma absorbs part of the pulse, which heats it and also the cristobalite (the heating time is greater than the pulse length). The temperature of the cristobalite is determined by the linear absorption and the heat transfer from the hot plasma. When a pulse train is used, the cristobalite then heats the cristobalite from that temperature. A certain number of pulses is required to attain 540°K, which is dependent on the intensity and the spacing. At T = 540°K, there is a stepwise volume increase, and microcracks are formed over the surface, with tribobreakdown in them [8]. The final heating is determined by the intensity of the last pulse in the series. Damage of scale about 50 μm may not be observed (the temperature is less than the necessary thermal destruction value of $T \approx 6 \cdot 10^3$ K) [8]. Consequently, high-temperature emission occurs after a series of subthreshold pulses, although damage of scale about 50 μm may not occur. However, one cannot rule out the possibility of breakdown in the melted glass [12].

Figure 2 shows graphs relating the number of pulses in a series N to $I < I_p$ when each pulse is of length $2 \cdot 10^{-8}$ sec for various pulse intervals without allowance for the additional heating ((1 — 1') — (5 — 5')) and with such allowance ((1 — 1) — (5 — 5)). We show the maximum temperatures produced by the last pulse in a series, which are less than $6 \cdot 10^3$ K. The experimental data of [4] (curve 6—6) are reasonably close to the calculated values only for I = 0.3Ip and 0.4Ip, although the shape of the experimental curve resembles that of the calculated one. The authors of [4] state that the measurements were inaccurate, particularly near I_p.

It is thus most likely that the impurities are present in the lattice of the quartz and in structures in the glass, while the FAI are present in defect channels in the quartz. The threshold values have been determined for the glasses in relation to external mechanical loads. The number of subthreshold pulses in a damaging series has been calculated. The experimental results are explained within the framework of [8].

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ENERGY CHARACTERISTICS OF A HIGH-TEMPERATURE IR RADIATOR

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Results are presented on the temperatures of the heated body and fused-silica envelope for a high-temperature IR emitter of new design based on a halogen filament lamp.

Infrared (IR) heating is increasingly used in technology. IR heating often not only accelerates the process considerably but also provides higher performance. In particular, specially designed devices are used in the electronics industry as sources of high-temperature radiation for IR heating, which have been called IR modules. Such devices were first described in [1].

The successful use of IR modules has required detailed theoretical and experimental studies on the thermophysical parameters of these in order to indicate the potential of such devices and the scope for making new designs. A model has been given [2] for calculating the heat transfer in an IR module, and the integral and spectral densities of the useful radiation flux have been calculated, while the temperatures of the fused-silica envelope and heated body have been determined. The calculated values were in satisfactory agreement with published data. However, comparison of these results was possible only on extrapolating the energy characteristics of the IR module to the corresponding values for a single halogen filament lamp (HFL). A more rigorous check on the numerical results of [2] is given here from an experimental study of IR module characteristics.

The most general form of IR module consists of a heated body (tunsten spiral 1, Fig. 1) enclosed in a gas-filled cylindrical silica envelope 2, which is enclosed in the hollow metal body 3 within the planar angle $2\pi - \beta$. The body has a reflecting coating of the maximum possible reflectivity on the side facing the silica envelope. The body thus also acts as a reflector. The cooling liquid 4 circulates within the body. Various designs of module are possible in accordance with the value of $2\pi - \beta$ and the air gap between the envelope and the reflector.

This IR module has some obvious advantages (simple design, compactness, and safety), and it also substantially increases the useful radiation flux in a given direction, which is attained by fairly simple means and without the use of expensive focusing reflectors. Numerical calculations [2] show that the temperature of the spiral is increased by 80-200°K in accordance with the angle β.

This effect occurs because the cylindrical heated body is at the focus of the cylindrical reflector. The radiation emitted by the body within the angle $2\pi - \beta$ is returned by the reflector and is partially absorbed (the absorptivity of a tungsten spiral is about 0.4 [3]). As a result, there is self-irradiation, so the temperature rises by ΔT, which leads to the emission of a more powerful useful flux within the given angle β (increase by 25-30%).

The second major advantage of the IR module is that the water-cooled reflector substantially reduces the temperature of the silica envelope, which is important when the module is used in closed IR heating systems.

Particular interest attaches to comparing the numerical calculations with measurements on the temperature of the heated body in the IR module and that in an identical free HFL, as well as to correct temperature measurement for the silica envelope.

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