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CHARACTERISTIC STATE-TRANSFORMATION TEMPERATURES OF A QUARTZ GLASS-CERAMIC WITH UNIDIRECTIONAL HEATING

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This article examines characteristic ablation temperatures of a quartz glass-ceramic. The emissivity and transparency of materials are evaluated.

Quartz glass-ceramic has been widely used in many areas of technology in recent years. The working temperatures encountered in some of these applications are significantly higher than the melting point. In particular, a device made of quartz glass-ceramic and designed to afford protection from heat at such temperatures is very effective because of the large amount of heat generated by all of the physical-chemical changes that take place during ablation. According to [1], the total heat release from glassy materials changes relatively little and may be about 11,000 kJ/kg for quartz glass. The high viscosity of melts of materials based on quartz glass makes it possible to determine the fraction of evaporation [2]. The rate of destruction of a quartz glass-ceramic is nearly independent of the amount of oxygen in the incoming gas flow, and it usually does not contain a low-temperature component (binder). All this provides grounds for the conclusion that quartz glass-ceramic is the most suitable material for studying the processes of heating and destruction during high-temperature unidirectional heating.

However, a pure quartz glass-ceramic has partial transparency, especially at temperatures above the melting point. Thus, its optical properties depend on the size of the heated volume. Moreover, a thermocouple installed in such a material will give exaggerated values of temperature due to radiative heat transfer.

Unfortunately, there is presently almost no data on absorption coefficients above the softening point not only for quartz glass-ceramic, but also for quartz glass. This makes it impossible to calculate emissivity with destruction of the surface of the material and to evaluate the error of the readings of a thermocouple inserted in a partially transparent medium.

Thus, the above-noted advantages of quartz glass-ceramic are partly offset by a serious shortcoming — its partial transparency at high temperatures.

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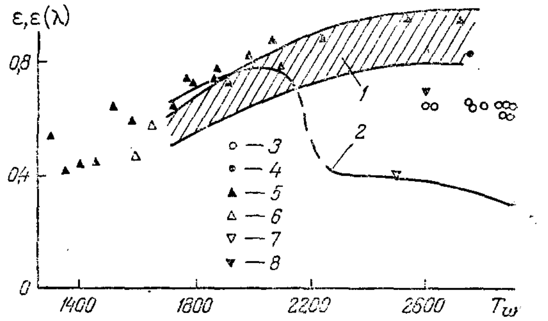

Fig. 1. Spectral and integral emissivities of a quartz glass-ceramic: 1, 4, 5, 8) alloyed quartz ceramic (up to 2.5% Cr_2O_3); 2, 3, 6, 7) pure glass-ceramic; 1, 2) integral emissivity, data from [7]; 3, 4, 7, 8) spectral emissivity, $\lambda = 0.65 \cdot 10^{-6}$ m; 3, 4) electric-arc heater, data from [5]; 7, 8) gas generator; 5, 6) spectral emissivity, $\lambda = 1.39 \cdot 10^{-6}$ m. T_W, K.

Here we pose the problem of evaluating the emissivity of a surface above the softening temperature of a quartz glass-ceramic, establishing the temperatures corresponding to structural changes in this material, and determining the dependence of the latter on the heating conditions. The problem is solved by conducting comparison tests of specimens of quartz glass and pure and alloyed quartz glass-ceramic for different types of heating, experimentally and theoretically determining the rates of destruction of these materials, evaluating spectral emissivity by methods developed previously, and studying structural changes in the specimens by means of petrographhic and metallographic analyses.

Studies were conducted on specimens of pure quartz glass-ceramic and a quartz glass-ceramic alloyed with chromic oxide (0.5–1%) prepared from a melt of a transparent quartz glass by the technology in [3]. Other tests were performed on grade-KV quartz glass. The porosity of the ceramic specimens was within the range 10–12%. Heating was done in the subsonic air jet of an electric-arc gas heater, in the supersonic jet of a gas generator operating on kerosine–oxygen fuel, and on a radiant heater. The heating time was set with a timer and ranged from 0.3 to 60 sec, depending on the test regime and the purpose of the experiment. The brightness temperature of the surface during convective heating was measured in the visible spectral range $0.65 \cdot 10^{-6}$ m with an FÉP-4M photoelectric pyrometer, while the same quantity was measured during radiant heating by a special heliopyrometer in the infrared range $1.39 \cdot 10^{-6}$ m corresponding to the "trough" in the solar radiation spectrum [4]. The allowable main error of the pyrometric systems is no greater than ±0.6% of the top measurement limit in the range 1500–2500°K and ±0.8% in the temperature range 2500–3500°K.

The methods in [5, 6] were used to determine emissivity. The error of measurement by the method in [5] for a confidence level of 95% was 12% for emissivity and 6% for surface temperature, with allowance having been made here for the random error. The error of brightness temperature was 2.2% when determined by the method in [6], while the methological error of emissivity measurements was 2.6%.

The emissivity of the specimens tested in the jet of the gas generator was determined by comparing thermocouple measurements and readings of the FÉP-4M pyrometer. Temperature was measured with VR 5/20 tungsten–rhenium thermocouples with electrodes $0.2 \cdot 10^{-3}$ m in diameter. The thermocouples were butt-welded and placed in the specimens at a depth of about $2 \cdot 10^{-3}$ m in such a way that the length of the isothermal section l/d was equal to about 75. A groove $0.5 \cdot 10^{-3}$ m wide was cut in the specimens at the prescribed depth. The groove was covered by a plate of the same material after the thermocouple was inserted.

The surface came closer to the thermocouple as the material was carried off, and eventually the thermocouple was exposed and burned. Recording of this process on an N030A loop oscillograph made it possible to realiably establish the moment the thermocouple reached the surface and to therefore determine its temperature even in the case of a semi-transparent material, since the radiation from the generator jet could be ignored. The change in the brightness temperature of the surface of the specimens in all of the tests was recorded on the oscillograph.

Figure 1 (curves 1 and 2) shows that, in accordance with the data in [7], the integral emissivity of an alloyed (1–2.5% chromium oxide) quartz glass-ceramic increases monotonically, while melting leads to a sharp change in the emissivity of the pure quartz ceramic. Figure 1 also shows results of measurements of the spectral emissivity of alloyed and pure quartz glass-ceramic for the visible $0.65 \cdot 10^{-6}$ m and infrared $1.39 \cdot 10^{-6}$ m regions of the spectrum obtained here and in [5]. Despite the different heating conditions and methods of measurement, the emissivity of the alloyed quartz glass-ceramic in these spectral regions is 0.83 ± 15% at tem-

TABLE 1. Experimental and Theoretical Rates of Ablation of Quartz Glass and Pure and Alloyed Quartz Glass-Ceramic (I – experiment; II – calculation)

Material	q_c, kW/m²	I_e, kJ/kg	T_w, K	Γ	$\bar{V}_\infty \cdot 10^3$, m/sec	
					I	II
Alloyed glass-ceramic (0,5—1% Cr_2O_3)	7650	8600	2620	0,57	0,1	0,12
	11500	12300	2800	0,82	0,18	0,17
	14700	4700	2600	0,1	0,18	0,17
Pure glass-ceramic (100% SiO_2)	7650	8600	2610	0,61	0,17	0,19
	11500	12300	2710	0,83	0,28	0,24
	14700	4700	2450	<0,1	0,29	—
Quartz glass	7650	8600	2590	0,58	0,22	0,21
	11500	12300	2660	0,80	0,29	0,24
	14700	4700	2360	<0,1	—	—

peratures above 2000°K. Some of this scatter can be attributed to variations in the chromic oxide content of the specimens.

Since the emissivity of the alloyed ceramic changes negligibly, is slightly dependent on the heating conditions, and has a value close to unity, as a first approximation the alloyed ceramic can be considered a nontransparent material in this spectral region. This conclusion is well supported by comparison of experimental and calculated values of the ablation rates of the quartz glass and pure and alloyed ceramic (Table 1). It is evident from the table that given the same heating conditions, the ablation rate for the quartz glass is twice as great as the rate of destruction of the alloyed ceramic. The error of the determination of linear ablation rate in the tests was no greater than 15%. It was found by a least-squares analysis of the results of specimen tests with different heating times.

Since the term accounting for radiation should not be included in the heat balance on the outside surface in the case of a transparent material [1], comparison of experimental and theoretical data for such materials can serve as indirect proof of the degree of their transparency. The expression obtained in [1] to calculate the ablation rate can be written in the form

$$\bar{V}_\infty = \frac{q_0 - n\varepsilon\sigma T_w^4}{\rho\Gamma[\Delta Q_w + \gamma(I_e - I_w)] + \rho c(T_w - T_0)}.$$

In the calculations, n was given a value of 0 for quartz glass, 1/2 for the pure glass-ceramic and 1 for the alloyed glass ceramic. Thus, we assumed in the calculations that quartz glass is a transparent material, the pure glass-ceramic is translucent, and the alloyed glass-ceramic is opaque. The integral emissivity of the pure and alloyed ceramics was determined from the data in [7] (Fig. 1). The density of the quartz glass was taken equal to 2200 kg/m³, while the value for the quartz glass-ceramic was 2000 kg/m³. Mean-integral heat capacity was taken to be 1.3 kJ/(kg·K) [8]. The vapor fraction was found by the method in [2].

Although the calculations are approximate, they offer a qualitative estimate of the effect of the degree of transparency of a material on the rate of its destruction. The appearance of the surface and the fusion zones of the specimens after the tests are shown in Fig. 2.

In the case of the pure glass-ceramic, we can establish only a certain effective emissivity for the thickness of the entire molten layer. The emissivity of such materials is greater the more of the material located in the zone of maximum temperatures. This conclusion is well supported by the record of brightness from [5] (Fig. 3). If we use this data to calculate emissivity, then for the pure glass-ceramic it increases from 0.35 to 0.64 as the size of the molten layer increases. Brightness remains nearly constant for the alloyed glass-ceramic and has a value of about 0.8. It was shown in [1] that at the given surface temperature, a body being destroyed at a high rate should radiate less intensively than a body melting at a lower rate, i.e., the emissivity of translucent materials depends on the conditions under which the material is being destroyed. It is evident from Fig. 1 (points 3 and 7) that the effective emissivity of the pure quartz glass-ceramic is 1.5 times greater in the jet of the arc heater than in the jet of the gas generator. At the same time, the results from [5] (Fig. 1, points 3) show that effective emissivity remains almost constant within a relatively broad range of temperatures (2600-2800°K) for the quartz ceramic. Under these conditions, the

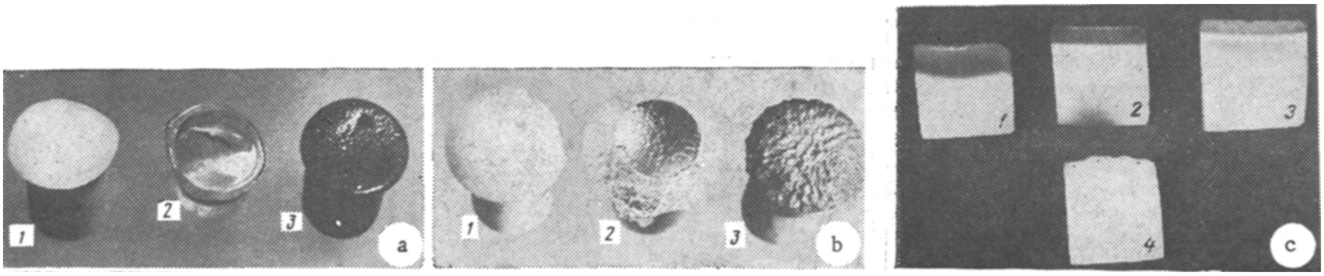

Fig. 2. Appearance of the surface (a, b) and fusion zone (c) of specimens after the tests: a) specimens tested in the jet of the arc gas heater (1 — pure quartz glass-ceramic, T_W = 2610°K; 2 — quartz glass, T_W = 2590°K; 3 — alloyed glass-ceramic (0.5-1% Cr_2O_3), T_W = 2620°K; b) specimens tested in the jet of the gas generator (1 — pure glass ceramic, T_W = 2450°K; 2 — quartz glass, T_W = 2360°K; 3 — alloyed glass-ceramic, T_W = 2600°K); c) fusion zone (1-3 — alloyed ceramic, T_W = 2490, 2620, 2800°K (arc heater); 4 — pure glass-ceramic, T_W = 2450°K (gas generator)).

thicknesses of material through-heated to temperatures above 2000°K were greater than $1.5 \cdot 10^{-3}$ m, while the vapor fraction was greater than 0.5. Material is carried off mainly in liquid form (vapor fraction about 0.1) from the surface of specimens heated in the generator jet. Analysis of the heated layer of the specimens after the tests showed that the difference in the effective emissivities of the specimens tested in the jets of the arc heater and gas generator could be attributed mainly to the different porosities and thicknesses of the layer of material heated above 2000°K.

Thus, as a first approximation, a quartz glass-ceramic alloyed with chromic oxide can be considered an opaque material, and the emissivity of its surface can be assumed to be independent of the heating conditions. In the absence of data on absorption coefficients above the softening point for the pure glass-ceramic, it is evidently most expedient to examine the effective emissivity and to assume a value of 0.35-0.4 for the wavelength $0.65 \cdot 10^{-6}$ m for the specimens destroyed mainly by fusion. Under heating conditions when the vapor fraction of the material exceeds 0.5, a value of 0.6-0.65 can be taken for effective emissivity at this wavelength.

Despite the relatively large amount of data on the absorption coefficients of quartz glasses [9÷11], there are serious problems with using this information to determine spectral emissivity during destruction of the material. It is evident that the emissivity of quartz glass depends heavily on its composition and the thickness of the specimen. The emissivity is also significantly influenced by the geometry of the specimen, such as the ratio of the width of a cylindrical specimen to its diameter. The literature data on absorption coefficients is generally limited to temperatures up to 1700°K.

In connection with this, we proceeded as follows to evaluate the emissivity of quartz glass specimens. Using different heating times for specimens of pure and alloyed quartz glass-ceramic in the jet of the gas generator, we established that the temperature at which material begins to be carried away from the surface coincides with the melting point of quartz and is equal to about 2000°K. On oscillograms depicting the change in surface temperature, this temperature corresponded to the characteristic point of inflection of the curve. The path of the temperature curve changed even more at the moment a melt was formed on the surface of the quartz glass specimens. Comparison of readings of the pyrometer for the wavelength $0.65 \cdot 10^{-6}$ m at this moment and the temperature of melt formation (~2000°K) allowed us to evaluate the effective spectral emissivity. For specimens of quartz glass 25 mm in diameter and 30 mm in length, it was equal to about 0.25.

A quartz glass-ceramic is an amorphous material. It therefore has no distinct melting point. The temperature of the transition from the solid state (when viscosity is greater than 10^{13}-10^{14} P) to the softened state is taken as the state-transformation temperature of both quartz ceramics and quartz glasses. According to the data of different authors, this transition temperature ranges from 1410 to 1640°K.

Preliminary information on the temperatures corresponding to structural changes in a quartz glass-ceramic can be obtained from the phase diagram of pure silica determined by Pryanishnikov [12]. It follows from this diagram that beginning at the temperature 1700°K, α-quartz undergoes two transformations simultaneously: into α-cristobalite and into a melt of

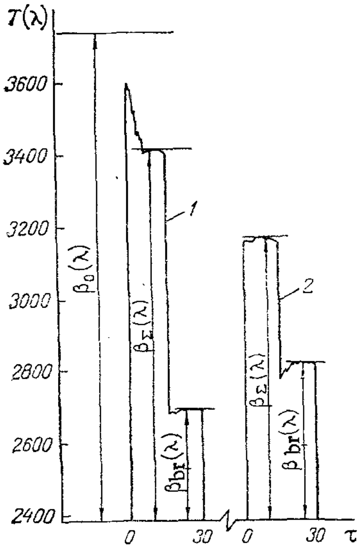

Fig. 3

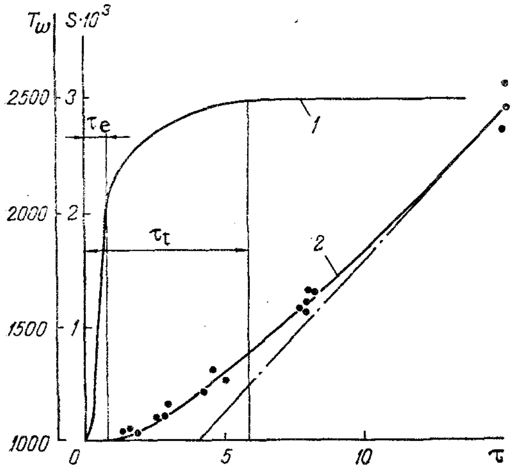

Fig. 4

Fig. 3. Change in the brightness of specimens of quartz glass-ceramic during destruction of their surface (according to the temperature on the pyrometer scale): 1) pure glass-ceramic; 2) alloyed glass-ceramic. T(λ), °K; τ, sec.

Fig. 4. Dependence of surface temperature and linear ablation of a pure quartz glass-ceramic on heating time: 1) surface temperature; 2) linear ablation. S, m.

variable density which becomes fully molten at 2000°K. At high heating rates and short heating times (about 1 min) for quartz glass-ceramic, cristobalite can be ignored and it can be assumed that the material formed above 1700°K consists wholly of an amorphous phase of variable density which becomes fully molten at 2000°K. Quartz glass-ceramic is a porous white material. When heated above 1800°K, its increase in density is accompanied by an increase in transparency and a change from white to a frost color [3]. The change in color at high temperatures is even more characteristic of alloyed quartz glass-ceramic. The addition of only 0.5-1% chromic oxide makes it possible to obtain a distinct boundary evidently corresponding to the beginning of melting of the material (see Fig. 2c). Explanation of the reason for the formation of this boundary and reliable determination of its temperature make it possible to use the laws established in [13, 14] for determining the thermophysical properties of quartz glass-ceramic at high temperatures under unidirectional heating conditions.

To determine the temperature of the structural transformations corresponding to the change in specimen color and its dependence on the heating regime and conditions, we visually inspected the surface of specimens tested at heating times from 0.3 to 1 sec in convective heating and up to 60 sec in radiative heating. To calculate the temperature at the moment of appearance of the intense green color for the alloyed quartz glass-ceramic and the increase in transparency due to melting of the surface of the pure ceramic specimens, we used oscillograms of brightness temperature and values of spectral emissivity in Table 2. It was found that this temperature is roughly 1800°K in the heat-flux range 6000-14,700 kW/m² under all of the heating conditions examined.

Petrographic analysis of the specimens showed that the structural changes occurring at the given moment of time correspond to the beginning of melting of the quartz glass-ceramic. At this temperature, the particles approach each other, and there is a significant loss of shape and a reduction in the porosity of the material. The chromic oxide in the glass partially dissolves [15] and is found mainly in the form of unevenly distributed inclusions. In general, no modified transformations were observed in the materials. This includes cristobalite. Such a conclusion is supported by measurements of permittivity and the loss tangent in the quartz glass-ceramic.

At a temperature of about 1800°K, there is a sharp change in these parameters evidently connected with an increase in the density of the material due to its melting [3].

TABLE 2. Values of Emissivity for the Wavelength $0.65 \cdot 10^{-6}$ m Used in Calculating the Surface Temperature of Specimens

Material	T_w, K	$\varepsilon(\lambda)$ $\lambda = 0,65 \cdot 10^{-6}$ m	
		arc heater	gas generator
Pure glass-ceramic (100% SiO_2)	1500—2000	0,4	0,4
	2000—$\overline{T}_w$	0,64	0,4
Alloyed glass-ceramic (0,5—1% Cr_2O_3)	1500—2000	0,65	0,65
	2000—$\overline{T}_w$	0,83	0,7
Quartz glass	1700—$\overline{T}_w$	0,25	0,25

Metallographic analysis showed that the zone which differs from the original material can be subdivided into two regions: a melt region and a softening region. In the softening region, the porosity of the material is reduced by a factor ranging from two to ten just as a result of the approach of the particles toward one another (the form of the pores is the same as in the original material). The thickness of the softening region is 0.3-0.5 mm and is independent of the heating conditions. The porosity of the melt region is due to the presence of bubbles. Both the porosity and the thickness of the melt region depend on the heat flux and the type of heating. The porosity of the melt increases substantially with an increase in heat flux. Melt porosity is characteristically high (up to 40%) for specimens of the alloyed glass-ceramic tested in the jet of the gas generator and is connected with frothing of the melt by the gas flow. The presence of chromic oxide increases the viscosity of the melt and somewhat reduces entrainment of material in liquid form. At the same time, the porosity of the surface layer of specimens of pure glass-ceramic tested under such conditions is lower by roughly a factor of ten. In this case, evidently the entire foamed film of molten material is carried off by the gas flow.

The high enthalpy and low dynamic head characteristic of the subsonic jet of the arc heater qualitatively change the pattern of destruction of the quartz glass-ceramic. The differences in the viscosity of the melt are no longer important in this case, so the thickness and the porosities of the layers of alloyed and pure glass-ceramic nearly coincide under these conditions. The sharp difference in the character of destruction of the pure glass-ceramic leads to a situation whereby the emissivity of this material is 1.5 times lower in the generator jet than in the heater jet.

The high density of the layer directly adjacent to the melt region is connected with intensification of sintering by the mechanism of viscous flow, which in turn is the result of a reduction in the viscosity of the glassy material and an exponential increase in temperature. In the general case, such softening (increase in flow) of glassy materials is seen in the temperature range corresponding to 0.7-0.8 of the absolute melting point [16]. Thus, the boundary between the softening region and the original material corresponds to 0.75 of 2000°K, i.e., to 1500°K.

Chromic oxide is introduced into the ceramic in the form of a fine powder with particles in the micron range. The solubility of Cr_2O_3 in SiO_2 is negligibly low at the sintering temperatures and increases exponentially with an increase in temperature. The high temperature of the melt region leads to an increase in the solubility of chromic oxide and an increase in the intensity of coloration of this region and a reduction in its transparency.

The temperature corresponding to the beginning of entrainment of material from the surface of the quartz glass-ceramic specimens is generally below the surface temperature in the quasisteady destruction regime and is determined mainly by the parameters of the incoming gas flow. To evaluate this temperature, we compared oscillograms showing the change in surface temperature with the dependence of the linear ablation rate on heating time (Fig. 4). For the specimens tested in the generator jet, this temperature nearly coincides with the melting point of crystalline quartz (vapor fraction $\Gamma \sim 0.1$). In the vaporization regime ($\Gamma > 0.5$; $P_e \sim 1 \cdot 10^5$ Pa), it can be assumed as a first approximation that this temperature is equal to 2250 ± 100°K.

Thus, along with the temperature corresponding to the beginning of ablation and the surface temperature corresponding to the quasisteady regime, in studying the destruction of a quartz glass-ceramic it is expedient to also examine the temperature at which melting begins (~1800°K) and the temperature at which the melt forms (~2000°K). Here, the temperature of the initiation of melting means the temperature at which the particles come closer together with a significant loss of their original shape. At the temperature of melt formation, the pores acquire a bubble shape.

In contrast to the ablation initiation temperature and the surface temperature in the quasisteady destruction regime, the temperatures at which melting begins and the melt forms do not depend on the type and regime of heating. The temperature at which melting begins corresponds to an increase in the transparency of the pure glass-ceramic and a significant increase in the intensive coloration of the ceramic alloyed with chromic oxide.

NOTATION

ε, $\varepsilon(\lambda)$, integral and spectral emissivities; T_w, $T(\lambda)$, true and brightness temperatures of the surface; $\bar{T}_w$, temperature of surface in the quasisteady destruction regime; $\beta_\Sigma(\lambda)$, $\beta_{br}(\lambda)$, spectral brightness of the total and background radiation from the surface; $\beta_0(\lambda)$, spectral brightness of the incident radiation from the heat source; λ, wavelength at which measurements were made; τ, heating time; q_c, calorimetric heat flux; Γ, vapor fraction; S, linear ablation; τ_e, time of initial entrainment of surface of material; τ_t, time of establishment of quasisteady surface temperature; $\bar{V}_\infty$, quasisteady linear ablation rate; q_0, heat flux to the hot surface; σ, Stefan–Boltzmann constant; ρ, density of material; ΔQ_w, total heat release from phase and physical-chemical transformations on the surface; γ, injection parameter; P_e, stagnation pressure; I_e, stagnation enthalpy; I_w, enthalpy of gas at surface temperature; c, heat capacity; T_0, temperature of unheated material.

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