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ELECTRICAL PROCESSES IN ENGINEERING AND CHEMISTRY

Activation of Melts by the Energy of Ultrasonic and Infrared Fields

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Abstract—The local introduction of ultrasonic vibrations parallel to the processed surface combined with IR heating in melts allows one to concentrate the activation energy in a small volume. This reduces the mechanical action on the processed surfaces, lowers the melt oxidation, and increases the durability of the soldered connections.

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INTRODUCTION

Conductive pastes on the basis of noble metals, such as silver, platinum, palladium, etc., are widely used for metallization of various components of ceramics; piezo-, glass-, and ferroceramics; ferrites; and other nonmetallic mineral materials. The limited natural resources of noble metals and the growing demand for them in industry define the necessity and practical value of the research directed at the development of such metallization methods that can completely exclude the application of these highly rare metals. Ultrasonic metallization by melted solders is one of the methods that allows one to save precious metals, to improve the productivity of the metallization, and to increase the durability of the seam with nonmetal materials [1].

[redacted] (US) metallization is one of the promising directions in the technology of electronic devices, since mechanic elastic vibrations with a frequency of 18–70 kHz and intensity of 0.1–1.0 MW/m^2 dramatically intensify the majority of physicochemical processes, such as wetting, spreading, and the capillary flow of the solder and the solder diffusion into the materials being soldered [2]. Lead-free solders on the basis of Sn with the addition of Ti and Ce as active components are used for the US metallization of ceramics on the basis of Al_2O_3, SiO_2, and TiO_2 [3].

For US activation, various kinds of mechanical vibrations, which are introduced in the liquid phase using radiators, can be used; these vibrations are elastic waves by their nature (longitudinal, shear, torsional, or their combinations). In the longitudinal waves, the vibrations of the particles of the medium occur in the direction of the wave propagation; in the shear waves, the particles of the medium move perpendicular to this direction. Since liquids and gases do not possess elasticity of their form, only longitudinal waves can propagate in them. However, in melts with high viscosity, the formation of viscous shear waves is possible, which decay at short distances from the radiator.

When longitudinal vibrations, whose intensity nonlineary diminishes with the increasing of the distance to the radiator, are introduced in the melted solder, it is necessary to maintain a constant spacing in the limits of 0.2–2.0 mm. This excludes the appearance of macro- and microcracks in the surface layers of brittle nonmetal materials owing to the shock action of the US waves directed normally to the surface. The dynamic action on the soldered material can be reduced by the variation of the angle of introduction of the vibrations into the solder from 90° to 30°–40° [4].

However, the process of soldering or metallization at small spacing between the end of the US radiator and the surface of the component (on the order of 0.1 mm) is rather complicated, since the spacing value should be maintained with high precision. The smallest inaccuracy can lead to the hard contact of the radiator with the ceramic or glass-ceramic surface with the appearance of micro- and macrocracks owing to the microshocks of the radiator against the surface or in some cases, when internal defects are present, to the destruction of the component.

The heating of melts in the metallization area by IR radiation possesses several technological advantages, though their realization depends on the correct construction of the installation for IR heating. Nowadays, two kinds of IR heating have became widespread in the technological processes of soldering: local focused and precision dissipated. As dependent on the specified conditions, reflectors with various geometry are used, which form the heat field in the area of the heating. For IR heating, a narrow range of the wavelength (from 1 to 5 μm) is usually used, which can be divided into the short-wave range (from 1 to 2.5 μm) and the medium-wave range (from 2.5 to 5 μm) [5]. The short-wave range of the IR radiation allows one to

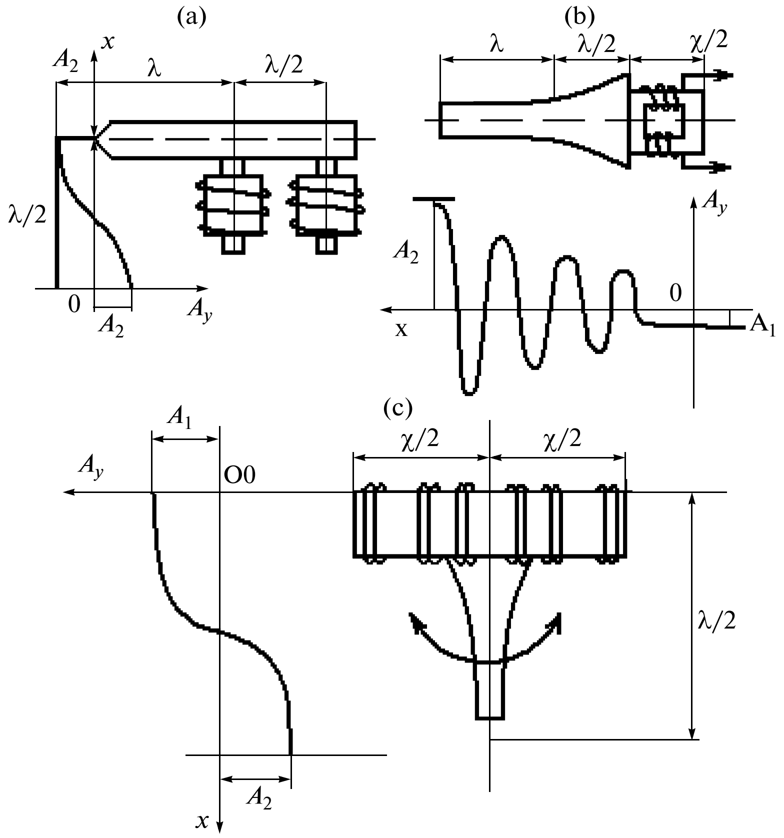

Fig. 1. Schematic view of the generation of US vibrations by waves of different types.

heat the object at a greater rate, since, according to the Wien displacement law, the maximal value of the spectral intensity of the radiation shifts in the direction of shorter waves with the increasing of the temperature.

IR heating is highly productive, invariant with respect to the type of the soldered component, ecologically friendly (does not produce environmental pollution), and allows one to program the regimes of heating in any controllable atmosphere, e.g., in a neutral or protective gaseous.

EXPERIMENTAL

Various kinds of US vibrations were produced using magnitostrictive transducers generating transverse vibrations with a frequency of 41 kHz (Fig. 1a), lontitudinal vibrations with a frequency of 44 kHz (Fig. 1b), and torsional vibrations with a frequency of 22 kHz (Fig. 1c). The amplitude of the vibrations of the radiative end of the waveguide was 8–10 μm. The spacing between the radiative end of the waveguide and the metallized surface was varied using a micrometric mechanism for the displacement of the radiators. The longitudinal pulse vibrations were generated by the pulses of the magnetic bias current; the working point was displaced to a steeper portion of the transducer. The amplitude of the pulse vibrations A_1 exceeded the amplitude of the continuous vibrations A_0 by a factor of 1.5–3.5. The pulse vibrations with a pulse-to-pulse duration of 2–6 were introduced to the transducer from a special generator. A VSA-10 direct current source was used to increase the direct component of the magnetic bias current (Fig. 2).

When shear waves of finite amplitude are excited in a thin melt layer under the condition that the layer's thickness is much less than the US wavelength, viscous waves are generated in the layer. Their wave vector is directed perpendicular to the side surface of the radiator. These waves are quickly absorbed in the direction from the vibrating surface and enter to a depth of

$$\delta = \sqrt{\frac{\eta}{\pi f}}, \qquad (1)$$

where η is the melt viscosity and f is the frequency of the vibrations.

The calculated depth of the penetration of the shear waves in the melts of solders at a frequency of 44 kHz amounts to 170–210 μm; therefore, their influence on the melt is insignificant for spacing exceeding 0.2 mm.

When longitudinal vibrations are excited on the interface between the media, the US wave is partially reflected, interferes with the incident wave, and par-

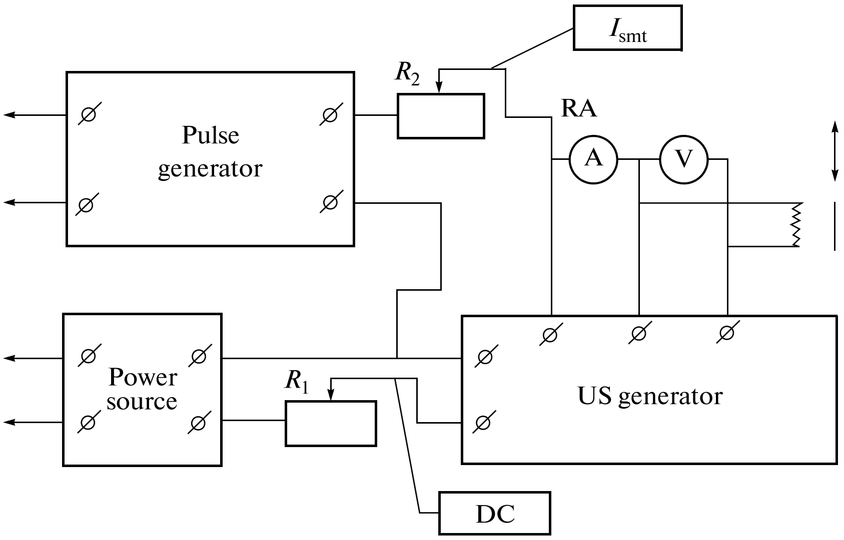

Fig. 2. Schematic view of the generation of pulse longitudinal vibrations.

tially enters the second medium. The pressures for the incident, transmitted, and reflected along the axis waves are expressed, respectively, as

$$P_1 = \rho_1 c_1 \nu_1; \quad P_2 = \rho_2 c_2 \nu_2; \quad P_3 = -\rho_1 c_1 \nu_3, \tag{2}$$

where ρ_1 and ρ_2 are the densities of the media; c_1 and c_2 are the speed of the ultrasound in the media; and ν_1, ν_2, and ν_3 are the speed of the incident, transmitted, and reflected waves, respectively.

Taking into account that the reflection coefficient K_{ref} and the transmission coefficient K_{trans} can be determined using the pressure as [6]

$$K_{ref} = \frac{P_3}{P_1}; \quad K_{trans} = \frac{P_2}{P_1}, \tag{3}$$

$Z_1 = \rho_1 c_1$; $Z_2 = \rho_2 c_2$; for $x = 0$, the following expression is valid

$$P_1 + P_3 = P_2, \qquad \frac{(P_1 - P_3)}{Z_1} = \frac{P_2}{Z_2}. \tag{4}$$

We obtain from (3) and (4)

$$K_{ref} = \frac{Z_2 - Z_1}{Z_2 + Z_1}; \quad K_{trans} = \frac{2Z_2}{Z_2 + Z_1}. \tag{5}$$

The analysis of Eq. (5) shows that the transmission and reflection coefficients substantially depend on the acoustic properties of the media. For $Z_1 = Z_2$, the reflection coefficient is zero, and the interface is acoustically transparent. When the specific wave resistances of the US radiators are greater than the specific wave resistances of the melts, in the case of vibrations parallel to the surface, up to 54% of the energy enters through the radiator–solder interface. This energy activates the cavitation processes, micro-, and macroflows. When the vibrations are directed normally to the surface, the US wave entering the melt undergoes decay spreading within the separation δ; a considerable part of the US wave enters through the surface since $K_{trans} > 1$. Therefore, only 20–25% of the US energy is spent for the activation of the melt.

The calculated K_{ref} and K_{trans} at the radiator–melt ($X = 0$) and melt–treated component ($X = \delta$) interfaces for various materials of the reflector and components are presented in Table 1.

In the case of the radiation of US vibrations into liquid media, it is energy advantageous to use materials with lower density (aluminum, titanium, etc.). At longitudinal vibrations of the radiator, the US waves undergo decay in the melt spreading within the spacing δ and, to a considerable degree, enter through the surface of the treated component at $K_{trans} > 1$.

The measurements of the US effect in a solder bath have shown that the transmission coefficient to a considerable degree depends on the length of the radiative waveguide, the chemical composition of the solder, and the distance between the radiative and receiving waveguides, but it is independent of the melt temperature [7].

The US metallization of glass-ceramic materials on the basis of T-80, T-150, and T-260 ceramics and glass was performed in the chamber of the experimental setup (Fig. 3) with a residual rarefaction of 1–10 Pa. For the metallization, a low-melt tin–zink solder POTs 10 and an experimentally developed Pb–Sn–Zn–In solder on the basis of lead and tin with the addition of zink and indium were used. US vibrations with an amplitude of 10–15 μm and a frequency 22 kHz were introduced into the melt using a radiator in the form of a concentrator of the Fourier type. The melt was heated by IR radiation from two halogen lamps with a

Table 1. Coefficients of the transmission and reflection of US waves according to the pressure

Materials of media	$X = 0$		$X = \delta$	
	K_{trans}, %	K_{ref}, %	K_{trans}, %	K_{ref}, %
Steel-melt	63	37		
Titanium-melt	79	21		
Aluminum-melt	105	5		
Melt-ceramics			90	−17
Melt-aluminum			95	−5
Melt-titanium			121	21
Melt-steel			137	37

power of 1 kW located in parabolic reflectors. The voltage at the IR lamps was supplied from a stabilized power source (SPS) controlled by a control block (CB). The rate of the IR heating amounted to 10–15°C/s. The temperature of the melt was measured in the working zone using a V7-40 device with ~~a KhK~~ thermocouple. The cavitation pressure in the melted solders was evaluated according to the spectral density of the cavitation noise in the frequency band of 100–250 kHz using a cavitometer [8]. The pressure in the cavitation region was registered by a measuring probe with a working area of 1.0 cm^2 connected to a piezoelectric transducer by an elastic waveguide. The sensor was supplied with a heater, which allowed us to maintain the necessary temperature of the receiving surface of the measuring probe. The durability of the connections of the solder with the glass-ceramic surface was evaluated according to the tear strength; the experiments were repeated at least five times. With the aim to increase the accuracy of the measurements of the tear strength and to eliminate shock loads, the samples were loaded into an RP-100 tensile machine in two stages; the preliminary loading was performed at a rate of 1.5–1.8 kN/min and the main loading at 8.8 kN/min.

EXPERIMENTAL RESULTS AND DISCUSSION

The influence of the type of US vibrations on the value of the cavitation pressure in the melted solders and on the durability of the connections with glass-ceramic materials was studied. The values of the cavitation pressure in the melted solder for various types of US vibrations introduced in the thin solder layer with

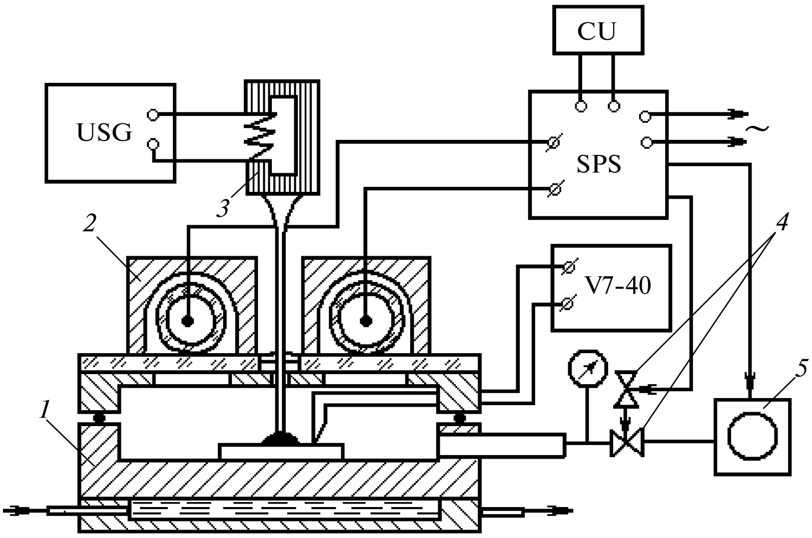

Fig. 3. Schematic view of the activation of the melt by the energy of US and IR fields. (*1*) Chamber, (*2*) IR heater, (*3*) US transducer, (*4*) valves, (*5*) compressor.

the thickness of 0.1 mm between the radiator and the probe are presented in Table 2.

The analysis of the experimental data has shown that, when the vibrations are introduced in the melted solder parallel to the soldered surface and the separation between the radiator's end and the surface amounts to 0.1 mm, the value of the cavitation pressure in the solder increases by 25% on average; this allows one to increase the productivity of the metallization process and to improve the quality of the connections.

The analysis of the dependence of the cavitation pressure in the melted Pb-Sn-Zn-In solder versus the frequency (Fig. 4) at the US activation has shown that, when the exciting frequency is close to the resonance frequency of the US transducer of 43.5 kHz, the output voltage of the US generator steeply falls. This occurs since the amplitude of the resonance current in the exciting coil increases; the amplitude of the displacement of the concentrator end is maximal and reaches 8–10 μm. The greatest value of the cavitation pressure in the melt (3.0–3.5 kPa) was registered at frequencies exceeding the resonance frequency by 0.2–0.5 kHz. This is associated with the generation in the acoustic transducer–matching element–radiator system of a set of harmonics and subharmonics of the main frequency, which facilitate the development of the cavitation processes in the melt.

Three regions can be distinguished in the frequency dependence of the cavitation pressure in the melted solder: (1) the precavitation region; (2) the region of the developed cavitation; and (3) the after cavitation region, which is insignificant in the soldering process. The dependences of the cavitation pressure in the 0.68Pb–0.1Sn–0.1Zn–0.1In–0.02Sb (1) and ~~POTs~~ melted solders versus the angle of insertion of the radiator into the solder α and the separation between the radiator's end and the measuring probe δ are shown in Fig. 5. For these dependences, the nonlinear falling of the cavitation pressure is characteristic owing to the dissipation and absorption of part of the US energy by the melted solder. The highest cavitation pressure was registered for longitudinal vibrations when the separation diminished to 0.2 mm and the angle of insertion the radiator was close to zero. The value of the cavitation pressure in the tinning bath with POS61 solder depends linearly on the output voltage; it nonlineary changes with the frequency and falls when the distance to the radiator (which is the bath bottom) increases. At a frequency close to the resonance one of the bath transducer of 20.5 kHz and at the amplitude of the output voltage of 240 V, the cavitation pressure reaches 3.5–3.6 kPa near the bottom of the bath.

Table 2. Cavitation pressure in the melt for various types of vibrations

Type of vibrations	Frequency, kHz	Amplitude of the vibrations, μm	Cavitation pressure, kPa
Longitudinal	44	8–10	2.5–3.0
Transverse	41	8–10	3.5–3.8
Torsional	22	10–15	5.0
Longitudinal pulse	44	10–15	3.5–3.7

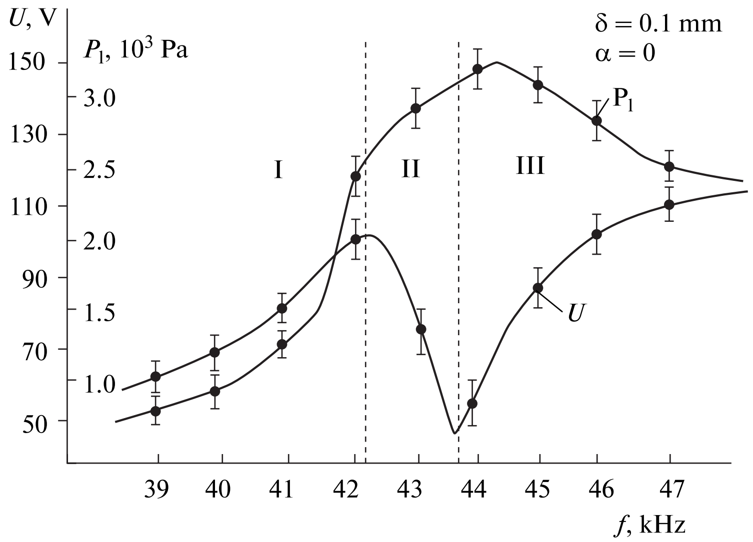

Fig. 4. Frequency dependencies of the US voltage on the transducer and of the cavitation pressure in the melts.

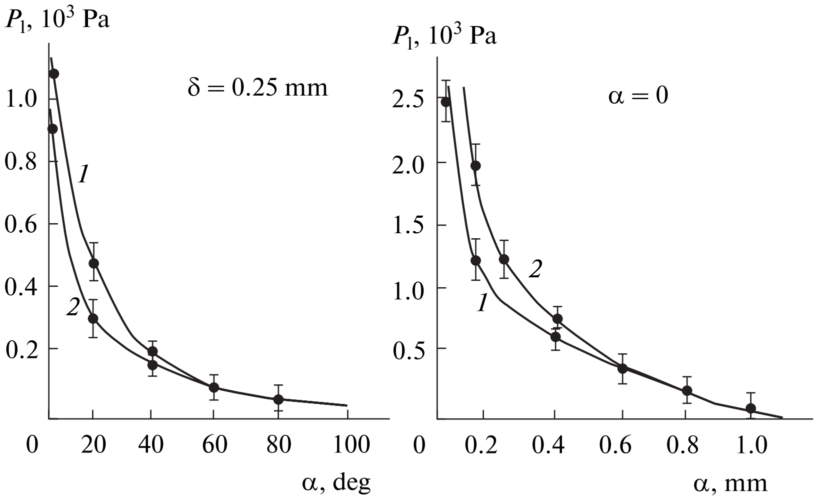

Fig. 5. Dependences of the cavitation pressure in the melt versus the angle of introduction of the vibrations and on the separation between the waveguide end and the surface.

The cavitation pressure in the melt was increased by using pulse vibrations with an amplitude that exceeded the amplitude of the continuous vibrations by a factor of 1.5–2.5[~~TaSh1~~]. The pulse vibrations were directed normally to the surface of the component and introduced into the melted solder together with continuous vibrations. After the US pulse entered, a pause followed whose duration was equal to or somewhat less than the pulse duration. The continuous vibrations introduced into the solder with the intensity that generates the cavitation, during the pause, produce a damping of the dynamic pulses arising under the action of ultrasound and in this way prevent the destruction of the substrate.

The investigation of the dependence of the cavitation pressure in the melts versus the pulse-to-pulse duration of the longitudinal pulse vibrations and the magnetic bias current has shown that the pulse-to-pulse duration of 1.5–5 and the magnetic bias current of 1.0–1.5 A are the optimal working parameters. For the higher magnetic bias currents, the magnitostrictive transducer enters the saturation regime, and the amplitude of the vibrations does not increase because the transducer overheats.

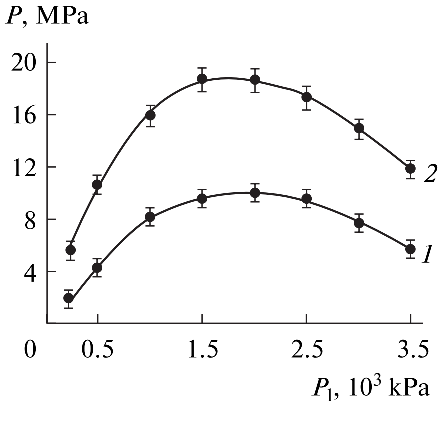

Fig. 6. Dependences of the durability of the soldered connections with glass ceramics versus the cavitation pressure in the melted solders. (*1*) POTs 10, (*2*) Pb–Sn–Zn–In.

The durability of the connections of the POTs 10 solders and the experimental Pb–Sn–Zn–In solder with the glass ceramics depends on the cavitation pressure in the melt (Fig. 6). The maximal value of the durability of the connections at longitudinal vibrations corresponds to the cavitation pressures of 1.5–2.5 kPa. For the higher or lower levels of the cavitation pressure, the durability of the connections decreases. This can be elucidated by the fact that, in the former case, when the cavitation bubbles collapse, the arising dynamic pulses induce microcracks in the pits of the microrelief and in the near-surface layers; this leads to the local destruction of the surface. The destruction of the metalized surface observed at intensive US vibrations is close in its physical sense to the cavitation erosion of the soldered metal. In the latter case, at the low levels of the cavitation pressure, the development of the cavitation processes does not reach the threshold of the wettability characteristic for the melted solders.

The introduction of vibrations in the solder parallel to the soldered surface of the glass-ceramic material allowed us to increase the durability of the connections of the Pb–Sn–Zn–In solder with the surface by at least 1.5 times. Therefore, it is preferable to generate this kind of vibrations in the processes of US soldering and metallization.

The influence of the pulse vibrations normal to the surface on the durability of the connections with glass ceramics was studied for the POTs 10 solders and the Pb–Sn–Zn–In system; in their melts, US vibrations were introduced with an amplitude exceeding the amplitude of the continuous vibrations by 1.5–3.5 times. The US metallization of the glass-ceramic materials was performed with a pulse frequency of 0–2.5 Hz. The average value of the magnetic bias current of the magnetostrictive transducer with a resonance frequency of 44 kHz was maintained at the level of 4.5 A, which by 1.5 times exceeded the magnetic bias current in the usual regime. At the optimal pulse frequency of 1.5–2.5 Hz and the amplitude of 10 μm, the durability of the connections increased by a factor of 1.7–1.8. The subsequent increasing of the pulse frequency reduces the durability due to the inertia of the initiation and the development of the cavitation processes in the melt (Table 3).

Table 3. Durability of the metallization in MPa for various pulse frequencies

Solder	Frequency of the pulses, Hz				
	0	1.0	1.5	2.0	2.5
Sn–10Zn	10.0	16.9	17.6	18.6	17.0
Pb–Sn–Zn–In–Sb	16.0	17.9	18.8	20.7	18.9

The adhesive strength of the metal coatings prepared using melted solders with various ceramic and glass-ceramic materials, which are used for the manufacturing of capacitors of constant capacity, depends on the glass phase content in the material. When its content is large (70–90 mass %), the durability changes insignificantly, since the tearing of the metalized coating occurs over the glass-ceramic material.

According to the tear tests of the metal-coated regions of the samples metalized with the POTs 10 and Pb–Sn–Zn–In–Sb solders, the destruction occurs mainly over the body of the glass ceramics (Fig. 7). The durability of the connections of these solders with the glass ceramics exceeds the adhesion strength of the coatings prepared by burning-in of a silver paste by 3–3.5 times. The tin–lead ~~POS61~~ solders do not provide sufficient durability of the connections; an adhesive character of the destruction is observed at the tearing tests. The investigations of the physicochemical properties of the compounds prepared by US metallization have shown that the adhesion strength with glass ceramics to a greater degree depends on the cavitation pressure in the solder than on the surface roughness.

The investigations of the dependence of the durability of the compounds on the amplitude of the US vibrations proved that it reaches its maximal value (20 MPa) at the amplitude of the vibrations of 10–12 μm and the treatment duration of 15–20 s. For lower amplitudes of vibrations, the development of the cavitation processes does not pass the threshold of the wettability characteristic for the melted solders; the oxide films are destroyed incompletely. The amplitude of the US vibrations equal to 3 ± 0.5 μm is the threshold for the cavitation processes in the melt of the tin–lead solder. At the amplitude of the US vibrations exceeding 15 μm, the generated dynamic pulses initiate the degradation of the near-surface layers; the solder melt dusts and oxidizes intensively.

The electrophysical studies of the soldered connections formed under the action of US vibrations on the melt have shown that the transient electric resistance reduces by 10–25% as dependent on the power and duration of these actions [10]. This is determined by the acceleration of the diffusion processes between the components of the solder and the soldered material and the increasing of the interface width from 1.5–2 to 5–7 μm.

CONCLUSIONS

The regularities and regimes of the metallization of the ceramic and glass-ceramic materials under the action of US vibrations on melted solders were found. The following parameters of the US vibrations were found and allow to one manufacture durable and reliable connections: the frequency, the amplitude and type of vibrations, the intensity of the cavitation pressure in the melt, the temperature and temporal parameters of the process, and the solder composition.

At the local input of US vibrations into the melt combined with IR heating, it is possible to concentrate the US energy in a small volume and to reduce the oxidation of the melt. To increase the durability of the soldered connections, ensure the stability of the metalli-

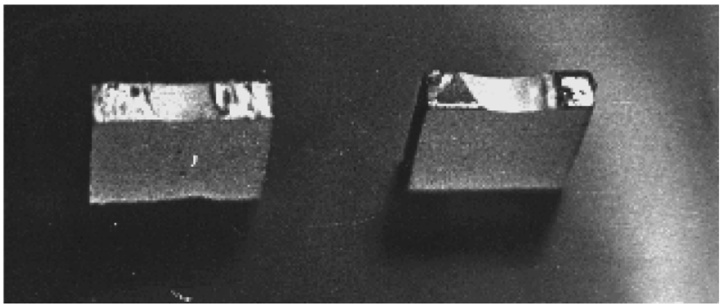

Fig. 7. Glass-ceramic samples after their metallization subjected to the tear test.

zation processes, and reduce the mechanical action on the treated surfaces, it is preferable to use the US vibrations parallel to the surface. These vibrations also increased the cavitation pressure in the local volume of the melt by a factor of 1.4 on average; this facilitated the formation of more durable connections.

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