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High-Temperature Joining of Ceramics and Sapphire by Laser-Based Process

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Abstract

Ceramics and sapphire are housing materials that are resistant to extreme stress and used in extreme environments today. The necessary high temperatures in conventional joining procedures are not suitable for encapsulating components owing to the functional requirements. Therefore new technologies are being developed in order to achieve local and time-limited heat input into the joining zone and thus to reach the necessary joining temperature.

This following paper deals with high-temperature joining with laser beams using high-temperature glass filler material e.g. glass solder. Even if the working temperature is above 450 °C, the filler material is referred to glass solder in scientific terminology. Practice-related examples demonstrate the experimental investigations on control of the process control as well as on joint formation and the resulting properties.

Keywords: Alumina, joining, laser, glass solder, high temperature

I. Introduction

Housings in microelectronics, microsystem technology, but also in optics and sensor technology have to protect the components or systems and simultaneously create a connection with the system surroundings. However, the system housing and the system itself must not be considered separately. Increasingly defining factors here are the functional characteristics of the system. For example, hermetically sealed housings are necessary in environments involving aggressive or corrosive media and at temperatures higher than 800 °C. The characteristics of alumina (Al_2O_3-ceramic) and sapphire perfectly meet these requirements as carrier and housing materials. Especially sapphire is, owing to its optical transparency and high-temperature resistance, an ideal material for optical windows, which are also used in aeronautics. At the same time a joining technology is necessary that is tailored to the material, the application conditions and the function of the component.

In the past, several high-temperature brazing applications of glass solders for joining ceramics have been reported [1–8]. Often the aim of these investigations was to produce vacuum-tight and high-temperature-resistant joints between oxide-ceramics (Al_2O_3, ZrO_2) as well as non-oxide ceramics (SiC, Si_3N_4) by means of laser radiation. Often CO_2-laser sources (10.64 μm) and diode lasers (0.808 resp. 0.94 μm) were used. Because of the thermal shock behavior of oxide ceramics, the diode laser was recommended for the laser brazing, especially to heat up the part profile homogeneously. On the other hand, non-oxide ceramics can be heated by CO_2-laser sources as well due to higher heat conduction and thermal shock resistance. Depending on the braze seam, the process time can be in the second and minute range. As a result, the joint is homogeneous, high-temperature-resistant, vacuum- resp. gas-tight and exhibits high strength as well as lower porosity and almost no microcracks. For oxide ceramics, the filler material has to be adjusted resp. modified for the short-time joining process as well as to the coefficient of thermal expansion of the base materials. Different filler material compositions are given in [6] and [7] with regard to softening and flow temperature together with absorption behavior for different laser wavelengths (808, 940 and 10 600 nm). As a result, the short wave lengths (808 and 940 nm) are favored because of the volume absorption. The experiments were conducted with two rotating axes and fixed optical system. For joining, the clamped Al_2O_3- resp. ZrO_2 samples (Ø= 10 mm) were moved synchronously at 1 rpm. In [9], a two-step procedure for laser joining is described. At first, the filler material is applied to the sample and sintered in a furnace process (glazing, formation of crystalline phase). Afterwards, the parts have to be joined by means of laser processing (remelting). The result of another investigation is that the crystallizing glass solder G018 – 358 (made by Schott) can be used for short-time joining processes like laser joining [10]. It was concluded further that the maximum application temperature is lower in the case of laser processing (600 °C) compared to the temperature after long-time furnace joining (1040 °C). Because of the short time at temperature during laser processing, there is no complete formation of crystalline phases.

II. Objective

The aim of the research was to develop a joining technology with a local heat-affected zone for the production of housings made of alumina and sapphire as well as a com-

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bination of both materials. Temperature-sensitive components or media for material tests have to be encapsulated vacuum-tight and electrically insulated. Joining in a furnace, as normally used for glass solder, is not possible in this case. A thermal load of the complete assembly with the joining temperature leads to the destruction of the functional elements to be encapsulated. Rotation-symmetric components are used for applications such as temperature sensors, reaction cartridges and lens systems in analytical processes. The following requirements have to be considered:

– Use of housing and filler material with sufficient thermal shock resistance,
– Adaptation of the heat input to the material and the component geometry to avoid stress in the joint,
– Temperature stability of the filler material joint according to the application conditions,
– Avoidance of thermal overload of the functional elements during the joining process,
– Gas tightness and dielectric characteristics of the fusing zone.

The above-mentioned requirements have to be fulfilled by means of material- and function-related process control. This requires a suitable heating concept with the necessary process parameters. The joint characteristics have to be tested with the help of suitable methods.

III. Experimental Procedure

(1) *Heating concept*

An irradiation of the lateral area in radial direction is necessary for the homogeneous heating of the rotation-symmetric components in the joining zone. Two different system setups were included in the experiments.

In the first setup [11] the relative movement between laser beam and components is characterized by a movement of the laser beam using a laser scanner system. The components, in contrast to the state of the art [9], are not moved so that the soldered joint is not exposed to mechanical stress, vibrations or centrifugal forces during the joining process. An optical element (loop mirror) is necessary for the irradiation of the components. The mirror deflects the laser beam from the scanner in radial direction onto the components (Fig. 1a). For this purpose, the loop mirror has a cone-shaped surface. The components, the cone mirror and other optical elements have to be arranged coaxially to each other. If the laser beam is moved in a circular motion on the surface of the cone mirror at high speed the soldering zone can be irradiated quasi-simultaneously over the whole circumference up to a material-dependent diameter.

The mirror surface is dished and so the laser beam is focused onto the centre of the cone mirror (Fig. 1b), which creates an elliptic beam profile on the component surface (Fig. 1c). An advantage of this beam shaping is that despite defocusing, the whole radiation energy hits the component surface.

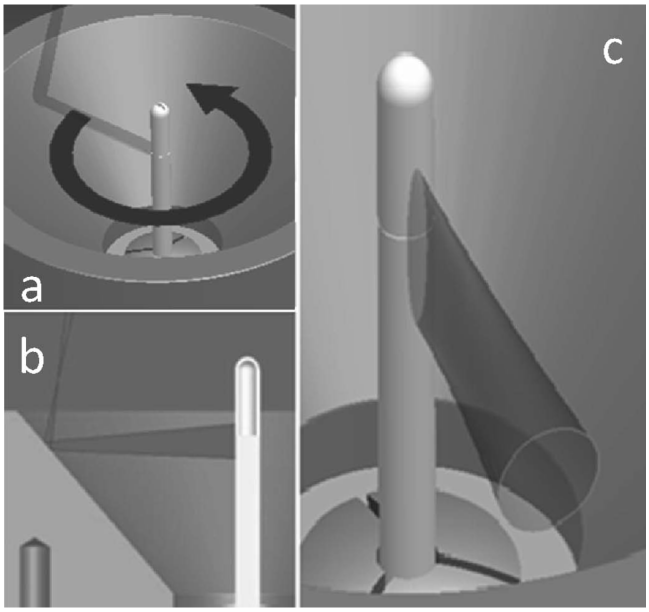

Fig. 1 : Heating of static components by means of a circular-rotating laser beam (a), beam shaping of rotating laser beam with a cone mirror (b, c).

The second setup [14] uses a ring mode laser over the cone mirror without any beam movement and real simultaneous heating (Fig. 2). This setup is useful for tube diameters larger than 11 mm in general and is faster than the scanner set-up because of the permanent circumference heating. But it needs a laser source with higher peak power. The donut mode is easy to realize with the integration of optical elements depending on the application and material. The uniform energy distribution is achieved by a precise adjustment of the optical elements.

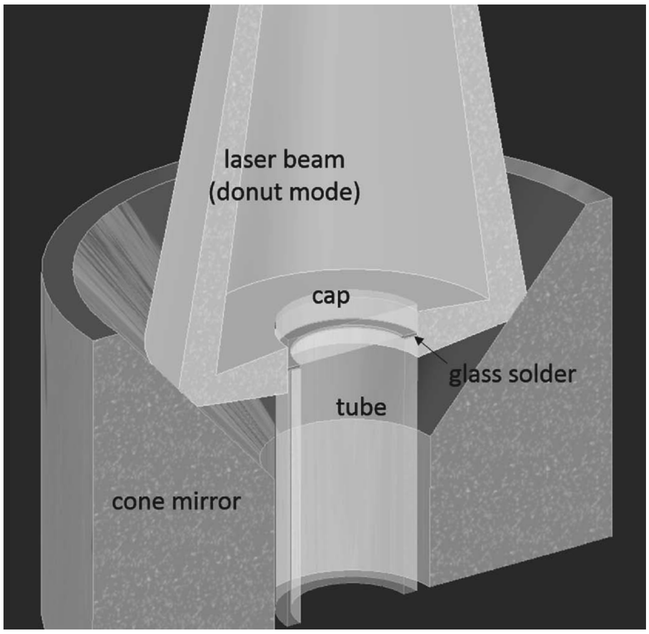

Fig. 2 : Donut mode laser setup.

(2) *Process equipment*

Different CO_2-laser types with a maximum beam power of 3500 W were used for the tests. The emitted laser radiation has a wavelength of $\lambda = 10.64\ \mu m$ and is well-absorbed by the alumina, the sapphire and the glass solder. The laser power can be adjusted manually as well as being program- or temperature-controlled. A pyrometer with a measuring wavelength of $\lambda = 5.14$ µm and a temperature range from 400…2200 °C was used for detecting the process temperature close to the joining area and obtain the temperature for closed loop heating. For this, the pyrometer-measured temperature values were given to a PI-controller, which adjusts the laser power to reach stable process conditions. As mentioned earlier, the laser beam is moved in circular motion on a cone-shaped loop mirror by means of a laser scanner system resp. using a donut-mode laser source (Fig. 3) [11…14].

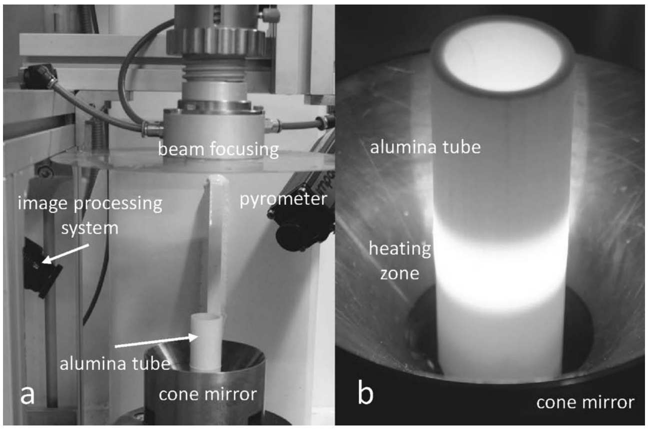

Fig. 3: Experimental setup (a) and heating zone (T = 1450 °C) in the soldering process (b).

(3) Experimental investigations

Rotation-symmetric test pieces from alumina and sapphire with a diameter range between Ø 2.6 and 25 mm and a wall thickness range of 0.3…2.5 mm were used in the experimental investigations. The crystallizing glass solder was used as a solder glass foil in the laser joining process. Therefore a project partner developed a solder glass green foil (thickness = 80 µm) with a high solid content and a low content of adhesive agent. This foil provides the following advantages for the joint creation:

- Simple assembly of the components,
- Easy setting of the soldering gap based on the number and thickness of the foil,
- Easy and residue-free burnout of the adhesive agent,
- Minimal shrinkage of the foil,
- High content of solder material and
- Minimum pore formation in the joining zone.

Radiant exposure tests were conducted on alumina tubes for defining the laser and process parameters, aiming at the definition of the necessary power range of the CO_2-laser, the spot size, the position of the laser beam at the soldering gap as well as the determination of the material-dependent control for a selective low-stress heating of the components. The quasi-simultaneous heating at the component perimeter was carried out at a scanning speed of 300 m/min. The temperature was defined depending on the materials. The process was observed with a camera to obtain information about the joint formation during the heat input by analyzing the behavior of the glass solder. Several parameters were varied in the tests such as thickness of the solder layer as well as the process design in order to guarantee low-stress heating, burnout of the adhesive agent, wetting, and joint creation and cooling down of the glass solder and housing parts. In the following table, the properties of base materials as well as the used solder are given. The solder was developed for furnace joining (furnace solder with retarding crystallization) and because of this, furnace process parameters are included.

A typical temperature/laser power vs. time cycle is shown in the diagram (Fig. 4). Line 1 is the measured temperature and Line 2 is the adjusted laser power. The temperature rate is around 200 K/min. As mentioned earlier, the temperature profile is preset and the laser power adjusted by the system control. After joining, the sample is cooled down at a controlled cooling rate.

IV. Results

The tests showed that in order to achieve the necessary viscosity of the glass solder used, the peak laser power depends on the sample geometry. The solder foil thickness of 80 µm was not sufficient to achieve leak-proof solder joints. Because of this, a solder layer thickness of at least 160 µm was used. Furthermore, the observations showed that the solder begins to flow at temperatures above 1250 °C. At a temperature of 1350 °C, leak-proof joints can be achieved. The process temperature of the solder should not be higher than necessary in order to maintain the solder composition and thus the set strain adjustment to the base materials as well as to avoid increased blistering.

Table 1: Material data

	Alumina	Sapphire	Glass solder GP06
Thermal expansion coefficient α	8.6 x 10^{-6} K^{-1}	8.4 x 10^{-6} K^{-1}	8.83 x 10^{-6} K^{-1}
Absorption $A_{(\lambda = 10.64\ \mu m)}$	75 %	95 %	no value
$T_{thermal\ shock}$	200 K (good)	no value	no value
$t_{working\ (permanent)}$ $t_{working\ (short)}$	-	-	900 °C 1050 °C
$T_{max.\ furnace}$ $t_{furnace}$	-	-	1250 °C 7 h

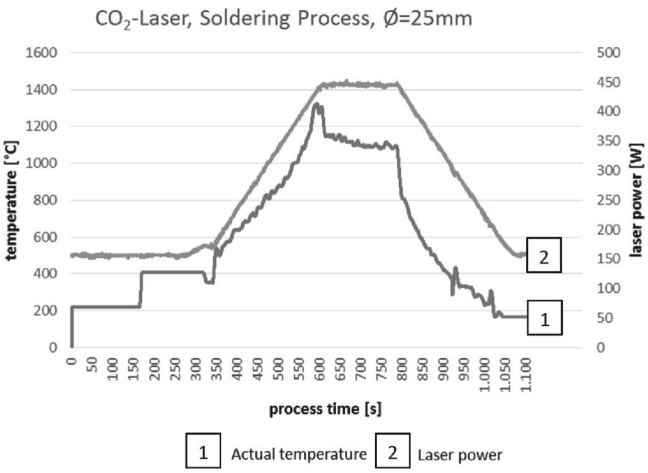

Fig. 4 : Temperature/laser power-time cycle of the CO_2-laser-based joining process.

For the manufacture of test samples from alumina the following laser and process parameters were used (Table 2). Afterwards the laser and process parameters were adjusted to the material sapphire and other tested material combinations.

Table 2: Laser and process parameters for joining alumina samples.

Geometry	3 mm	10 mm	15 mm	25 mm
Max. laser power	50 W	200 W	300 W	600 W
Technology	Scanner	Scanner	Ring mode	Ring mode
Solder temperature	1370 °C	1350 °C	1400 °C	1425 °C
Cycle time	3.5 min	25 min	15 min	30 min

The achieved solder joints were first characterized by means of a light microscope (Fig. 5, Fig. 6) and a scanning electron microscope (SEM). The results are shown in Figs. 5 to 8. Their appearance as well as the cross-sections in the SEM images (Fig. 7, Fig. 8) show a good wetting of the base materials and a homogeneous solder zone. This means an excellent connection of the solder to the alumina or sapphire base material has taken place. Owing to the similar constituents of the joints, no difference in contrast can be seen in the SEM images, whereas the joining zone is characterized by a homogeneous structure with circular pores. The outer and inner solder fillet between the tube and the blank is homogeneous for both materials. The solder zone shows a homogeneous structure over the wall thickness (Fig. 6), which is characterized by finely distributed pores.

The pore sizes and distribution in the glass solder zone could be examined from a micro-computer tomography image of a joined ceramic component. The measured sample was prepared by using the circular-rotating laser beam. The generated model based on the measured data is shown in Fig. 9 as a 3D model which shows the defects in the glass solder zone. It is obvious that the majority of pores are smaller than 0.002 mm^3 and that there is a pore cluster in one area. It is considered that this pore concentration results from the increased energy input at the starting and ending points of the scanner movement. Furthermore, the data provide information on the material volume of the glass solder zone (12.327 mm^3) and percentage of pores within (1.72 %).

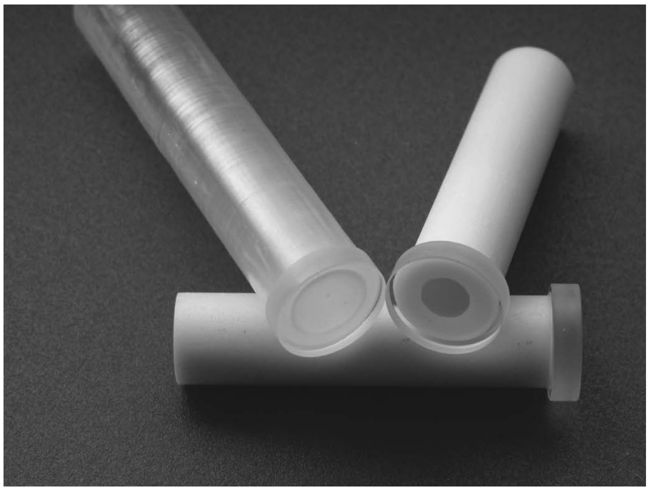

Fig. 5 : Laser-soldered material combinations (alumina tube ø = 10 mm, sapphire tube ø = 11 mm).

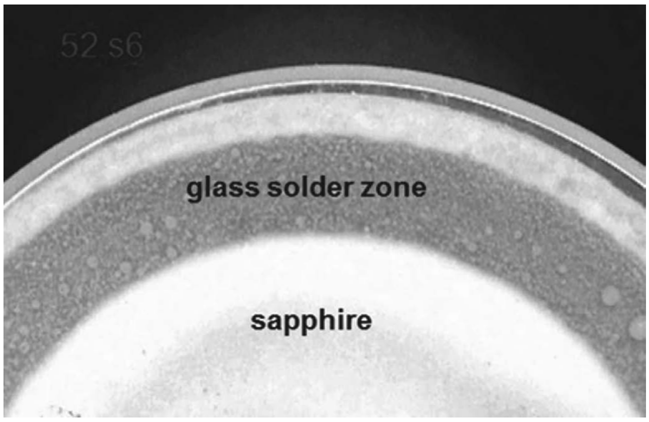

Fig. 6 : Light microscope image sapphire tube/blank joint – view through the sapphire blank.

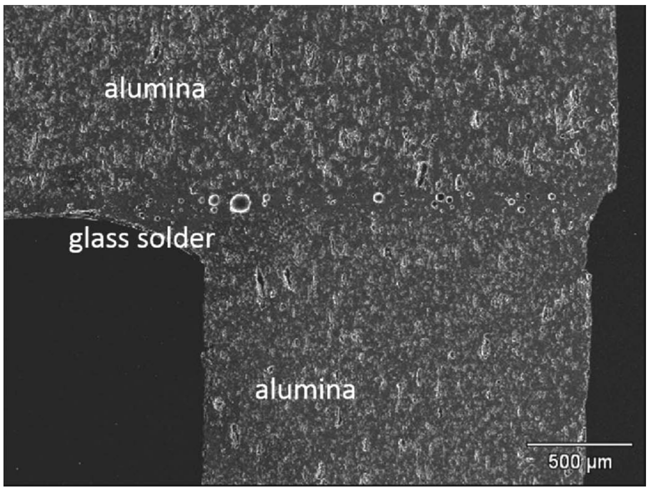

Fig. 7 : SEM image – cross-section of an alumina-alumina-joint.

An essential requirement is to produce gas- resp. vacuum-tight joints. The leak rates of the ceramic and sapphire joints were determined with the help of the vacuum method, where the test samples are evacuated. The achieved vacuum characterizes the quality of the hermetic joint. The determined values were between 1.1×10^{-6} mbar l/s and 1.0×10^{-10} mbar l/s. The required gas tightness of the joint, shown in **Fig. 7** and **Fig. 9,** could be proved by a measuring result of $6.1 \pm 1.9 \times 10^{-7}$ mbar l/s.

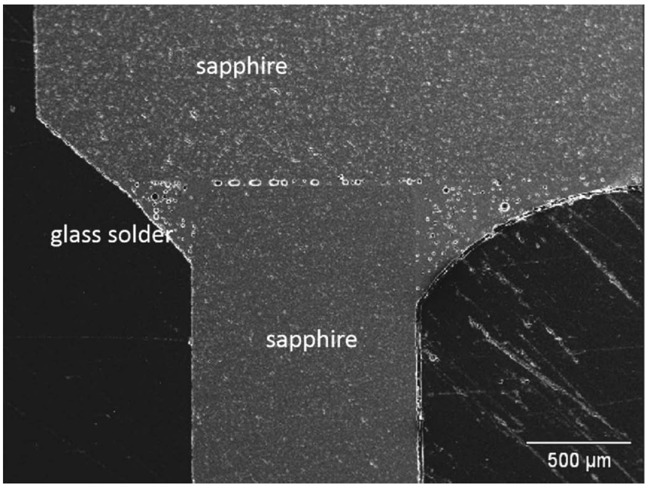

Fig. 8 : SEM image – cross-section of a sapphire-sapphire-joint.

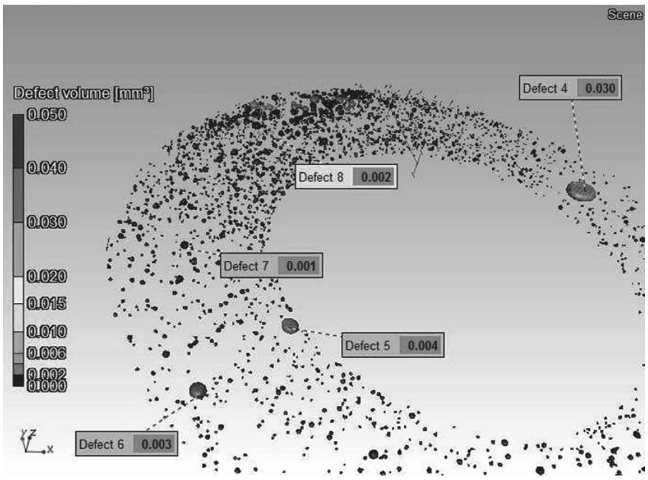

Fig. 9 : mCT analysis of joining area – pores highlighted in the glass solder.

The strength of the joints was also tested using a tension/compression testing machine with a fixture adjusted to the components. For the tests, the components were adhesive-bonded into a clamping device and then placed in the fixture of the test machine. The plunger is inserted through the sample tube down to the blank and a tensile load is applied to the glass solder joint. The average tensile strength of the solder joints is 22.9 MPa with a standard deviation of ± 7.8 MPa.

The selective joining process, in contrast to the conventional furnace process, avoids thermal overload of the functional elements to be encapsulated. This could be proved in an experimental arrangement for the determination of the temperature inside the tube. Therefore, a thermocouple (Pt/Rh-PT) was placed in an axial position at different distances from the heat input zone. After the joining temperature had been reached and the temperature balance had been achieved in the joining zone, the following maximum temperature was measured on the inner tube wall beneath the maximum temperature zone (T = 1250 °C) at a distance of 30 mm – 236 °C and at 40 mm – 165°C.

V. Conclusions

Rotation-symmetric parts made of alumina and sapphire were joined by means of scanner-controlled and donut-mode CO_2-laser technology. The method with the donut mode offers advantages owing to the permanent and homogeneous heating of a local, circular joining zone. As a result the starting and ending points of the rotating beam can be avoided and increased pore formation in this area is prevented. In addition, the total process time can be reduced. Whereas the scanner technology is limited to tube diameters up to 11 mm, the donut mode setup enables a wider range of applications. That could be proved by experiments with tube diameters up to 25 mm.

A special cone mirror arrangement and a fast-moving laser beam were used for the beam shaping and the homogeneous heat input into the components. Joints with an average tensile strength of 22.9 MPa were manufactured and their gas tightness was proved with the help of the vacuum method. The tests also showed that the developed process technology is suitable for encapsulating temperature-sensitive components. A maximum temperature of 236 °C could be measured on the inner tube wall beneath the heat input zone at a distance of 30 mm. This newly-developed joining process prevents damage to the encapsulated components owing to the design of the housings.

Further R&D concentrates on joining by means of laser radiation without additional tempering in order to reduce the time required, because for different reasons, the microstructure of the filler material should be in crystalline state after joining. To achieve this without additional furnace tempering, it is necessary to develop a glass solder type that is suitable for fast crystallization as well as for laser processing.

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