Glass Frit as a Hermetic Joining Layer in Laser Based Joining of Miniature Devices

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Glass frit as a hermetic joining layer in laser based joining of miniature devices

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Abstract

In this paper we investigate the feasibility of using a laser as the heat source to drive a hermetic joining process based on a glass frit intermediate layer. The laser allows the necessary heat energy to be provided in a localised manner; important either as part of a multi-stage process, or to allow thermally-sensitive materials to be used inside the package. Our study includes an investigation of the impact of rough and grooved surfaces on the hermeticity and strength of the join, demonstrating the robust nature of the process, and its ability to allow feed-throughs to the centre of package. Hermetic sealing is demonstrated, with leak rates of $10^{-9}$ mbar l s$^{-1}$, satisfying the military standard MIL-STD-883G.

Keywords: Laser joining, glass frit

1 Introduction

Many miniature optical, electronic and micro-mechanical systems, for example MEMS (Micro-Electro-Mechanical Systems) require hermetic sealing with long-term stability. The dictionary definition of the term hermetic is a seal that is completely gas tight or impermeable to gas flow. With MEMS technologies it means an air tight seal which will keep gases and moisture out of the package; hence failure of the device due to condensing water vapour inside the package is avoided. Appropriate bonding processes can ensure that a package remains dry and at a high vacuum for years. As no seal is perfect standards have been defined to describe the conditions and leak rates under which seals are still considered to be hermetic. These include the MIL-STD-883G Method 1014.11, JEDEC Standard JESD22-A109-A and ASTM F134-85, all of which are variants of each other. A description of the leak tests according to these standards is given in [1].
A number of different hermetic joining techniques have been developed for miniature devices, typically requiring the whole device to be heated to a high temperature. However, this whole package heating limits the ubiquity of the process, (i) causing problems in manufacturing processes where a number of thermal process steps are carried out in sequence, when a later heating step can cause parts joined earlier to disassemble; and (ii) preventing the use of temperature-sensitive materials inside the device e.g. polymers or magnetic materials. Alternative, more localised, techniques have therefore been developed, including resistive heating [2-5]; microwave heating [6]; and induction heating [7-8]. These require specialised modified components and/or materials; our approach is to instead use a laser to provide this localised heating. Lasers are, of course, widely used in a range of joining processes, including the joining of dissimilar materials such as cemented carbides to steel [9], magnesium and aluminium [10], and borosilicate glass to fused silica [11]. These techniques create a direct bond without any intermediate layer; however they normally require both a high temperature and highly cleaned polished contact surfaces. The use of an intermediate layer has advantages which include a relatively low joining temperature (few hundred degrees Celcius), and less stringent requirements for contact surface smoothness. Suitable intermediate layers include Au/Sn solder [12], Zn [13], Al, Au [14], In [15], Ti [16], thermosetting polymer benzocyclobutene (BCB) [17], or glass frit [18-22]. Glass frits are of particular interest due to the required low bonding temperatures (< 595 K), their ability to deliver a hermetic seal with moderately rough bonding surfaces, and to conform to the surfaces being bonded - even to the extent of allowing metallic feed-throughs to be included in the joining layer [19-20]. However current glass frit-based technologies are based on whole-package heating in a furnace. Laser based joining should allow the advantages of glass frits to be applied to temperature-sensitive processes and/or materials, and in multi-step manufacturing processes; however to our knowledge there has to date been no report on laser-based joining using a glass frit intermediate layer.

Frit is simply broken particles of glass, sorted by various grind sizes, and held in a suitable binder, typically as a paste. Sizes range from finely ground powder to small chunks. The glass frit used in our experiments is DM2700P/H848 from DIEMAT [23]. The paste comprises a low temperature glass powder loaded in an organic system which must be burnt out completely, and the glass glazed, before joining. It can be screen-printed, stencil-printed or dispensed (e.g. from a syringe) onto one of the surfaces to be joined; in our experiments a syringe was used to provide a layer of about 250 μm thickness once glazed. The part with deposited glass frit paste is then heated to 520 K for 30 minutes to provide the organic burn-out before glazing for 1 minute at 620 K. The glazed surface is then mated to the other
component under slight pressure and fired at a peak temperature ranging from 593 to 648 K for as little as 1 minute to achieve a reliable, hermetic seal.

In this paper we investigate the feasibility of this glass frit / laser process with 4 different miniature packages:

(i) LCC (Leadless chip carrier) package. This consists of an alumina-based substrate containing a recess, to which a Kovar™ lid is bonded;

(ii) planar LTCC (low temperature co-fired ceramic), an alumina-based ceramic to which a Kovar™ “top-hat” lid is bonded;

(iii) planar AlN (Aluminium Nitride) to which a Kovar™ “top-hat” lid is bonded;

(iv) planar silicon substrate to which either a Kovar™ or Nickel cap is bonded.

All are realistic packages used in actual device manufacture.

We first of all concentrate on the ability of the laser to provide the required localised heating, but without taking measures to prevent heating inside the devices due to lateral conduction. We subsequently investigate the ability of the laser/glass frit process to bond rough and grooved surfaces, and finally describe a process whereby the temperature inside the device is minimised by the use of appropriate heat-sinking, including both experimental and simulation results.

2 The laser-glass frit joining process

The laser used is a fibre-delivered diode array operating at 940 nm (Laserline, fibre core diameter 200 µm, NA=0.22). A galvanometer scanner was used together with an f-theta lens to provide a focused spot of diameter 1 mm which could be moved at high speed across the workpiece. Alternatively, an axicon lens could be used together with a conventional lens to generate a ring focus, as described in section 4.3 below. A schematic of the laser bonding arrangement is shown in Figure 1(b).
2.1. Laser bonding of LCC (Leadless Chip Carrier) packages

The LCC substrate is made from a high temperature co-fired ceramic, made by companies such as Kyocera and NTK. The ceramic is mostly (90%) alumina (Al₂O₃), with some other glassy materials for binding. The dimensions of the Kovar™ lid and LCC substrate are 12.7 × 12.7 mm and 13.7 × 13.7 mm respectively. The substrate includes a recess 8.9 × 8.9 mm by 1.27 mm deep. Figure 2 shows (a) the LCC substrate and (b) the Kovar™ lid (syringe-dispersed glass frit followed by organic burn-out and glazing with thickness around 250 µm) before bonding and (c) the bonded sample. In this case, to improve the laser beam absorption, a piece of silicon wafer of thickness 380 µm was placed in good thermal contact with the Kovar™ lid as shown in the figure, absorbing 69% of the 940 nm laser light.

Figure 2 (a) LCC substrate and (b) Kovar™ lid (dispersed with glass frit) before bonding and (c) bonded sample

Fine and gross leak tests were carried out for the bonded samples, using a two-part process [1]; the gross leak test is essentially a pass/fail; whilst the fine leak test results are shown in table 1. The acceptable leak rate, as defined by
MIL-STD-883G, is $5 \times 10^{-8}$ mbar l s$^{-1}$ for a package of this volume. It is clear from the results presented in the table that a lower rate than this was achieved for a range of laser powers and bonding times.

Table 1 Fine leak test results of bonded LCC packages

<table>
<thead>
<tr>
<th>Laser Power (W)</th>
<th>Force (N)</th>
<th>Bonding time (s)</th>
<th>Leak rate (mbar l s$^{-1}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>35</td>
<td>2.25</td>
<td>140</td>
<td>$1.3 \times 10^{-9}$</td>
</tr>
<tr>
<td>40</td>
<td>1.96</td>
<td>100</td>
<td>$1.1 \times 10^{-9}$</td>
</tr>
<tr>
<td>60</td>
<td>1.96</td>
<td>40</td>
<td>$2.2 \times 10^{-9}$</td>
</tr>
</tbody>
</table>

Sectioning and polishing of the bonded sample were carried out to visually assess the joint quality across the bond. A typical micrograph of a sectioned sample is shown in figure 3, which indicates that glass frit has excellent contact with both the LCC substrate and Kovar™ lid, with no visible voids; it appears to have conformed well to the locally rough ceramic surface.

Figure 3 Micrograph of sectioned LCC package

2.2. Laser bonding of Top-hat packages

The “top-hat” cap consists of a raised square cap made of Kovar™, coated with a thin layer of nickel and then gold; however there was no coating at the bonding area – see figure 4(a) and (b). This cap is $9 \times 9$ mm, and 4 mm high. Two kinds of substrates were used in our experiments: AlN, $13 \times 13 \times 0.5$ mm (figure 4(c)), whilst the other is LTCC, $18 \times 18 \times 0.6$ mm (figure 4(d)). The LTCC was plated with a 10μm thick Au track in the bonding region.
In this case joining was carried out with the samples inverted; i.e. with the substrate uppermost. Examples of the bonded samples are shown in figure 5.

Successful bonding was achieved, although a higher power laser power of \( \sim \) 100W was required for LTCC, in comparison with 40 - 50 W with AlN. These bonded samples all passed the gross leak test, and met the MIL-STD 883G criteria in the fine leak test (again \( 5 \times 10^{-8} \) mbar l s\(^{-1}\), given the 0.26 cm\(^3\) internal volume). Detailed results are shown in tables 2 and 3 below. The bonding force used in each case was 1.96 N.
Table 2 Fine leak test results of AlN substrate bonded to top-hat Kovar™ cap

<table>
<thead>
<tr>
<th>Laser Power (W)</th>
<th>Bonding time (s)</th>
<th>Leak rate (mbar l s⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>40</td>
<td>40</td>
<td>4.8 × 10⁻⁹</td>
</tr>
<tr>
<td>45</td>
<td>30</td>
<td>1.1 × 10⁻⁹</td>
</tr>
<tr>
<td>50</td>
<td>26</td>
<td>1.3 × 10⁻⁹</td>
</tr>
</tbody>
</table>

Table 3 Fine leak test results of LTCC substrate bonded to top-hat Kovar™ cap

<table>
<thead>
<tr>
<th>Laser Power (W)</th>
<th>Bonding time (s)</th>
<th>Leak rate (mbar l s⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>80</td>
<td>1.4 × 10⁻⁹</td>
</tr>
<tr>
<td>120</td>
<td>50</td>
<td>1.7 × 10⁻⁹</td>
</tr>
</tbody>
</table>

To visually assess the bonded joint, samples were sectioned and polished. Example micrographs are shown in figure 6. Again, the glass frit makes excellent contact with both these samples, conforming well to the joined surfaces.

![Micrograph of section (a) AlN and (b) LTCC substrate to top-hat Kovar™ cap](image)

Figure 6 Micrograph of section (a) AlN and (b) LTCC substrate to top-hat Kovar™ cap

### 3 Influence of micro-machined channels/slots on hermeticity and shear force

Bonding surface roughness is an important issue in most hermetic packaging technology. Many processes require very flat surfaces to be used; however the results presented above demonstrate that the glass frit has the ability to conform well to naturally rough surfaces. In this section we report on our more detailed investigation of the impact of non-planar joining surfaces on hermeticity and shear strengths; specifically surfaces containing micro-machined channels / slots.
In this section we introduce our final sample, which has a silicon substrate, in addition to the LTCC and AlN substrates used in the experiments described earlier. Each substrate was modified by laser machining a series of closely-spaced slots into the surface. The slot depth generated was slightly different in each case, as shown in the micrographs of sectioned samples in figure 7; meanwhile the SEM images shown in figure 8 provide an indication of the extent the surface roughness in each case. The slot depths are shown more clearly in the micrographs shown in figure 8.

![Micrographs of the cross-section of laser machined substrates](image)

Figure 7 Micrographs of the cross-section of laser machined (a) LCC (b) AlN and (c) silicon substrate

![SEM images of laser machined substrates](image)
Figure 8 SEM images of the surface roughness of laser machined substrates (a) LCC (b) AlN and (c) silicon, providing an indication of surface roughness, both of the original sample and in the laser-machined region.

Of interest here is not only the leak rate that can be achieved, but also the shear strength. First of all, non-machined samples were tested. The LCC packages had a shear strength of 62 kg, whilst both the AlN and silicon were only ~ 5 kg. With the laser-machined samples, leak rates of less than $5 \times 10^{-8}$ mbar l s$^{-1}$ were consistently achieved, demonstrating the excellent ability of the glass frit to conform to the joining surfaces. The laser machining did, however, influence the shear strength for both the AlN and silicon substrates; in both cases it was increased to ~ 10 kg provided that the grooves were at least 20 – 30 µm deep. With the LCC substrate, no such increase was observed; however it is clear from figure 8 that the unprocessed surface in this case already has a significant roughness in comparison with the other samples. It is hence clear that one potential drawback of glass frit is a relative lack of strength when joining very smooth surfaces; however in general it is easier to produce a sample with rough surface than a smooth one.

To visually assess the joint line of bonded area, sectioning and polishing of the bonded samples were carried out. Micrographs of the sectioned samples are shown in figure 9; here it is clear that the glass frit has flowed into the laser-machined slot, providing excellent contact in all cases.

![Micrographs of sectioned packages](image)

Figure 9  Micrographs of sectioned packages, (a) LCC with a Kovar™ lid; (b) AlN with Kovar™ “top-hat” cap and (c) silicon with Kovar™ “top-hat” cap.

### 4 Laser based localised bonding technique

In the investigations detailed above, we were not concerned about preventing heating inside the joined packages; indeed for simplicity the samples to be joined are placed on top of a glass slide. However this will heat the whole
sample to a high temperature due to the lateral heat transfer, in particular with the silicon substrate, which has a high thermal conductance. To prevent lateral heating of the centre of the substrate, it is necessary to place a cooled thermally conductive block (in our case copper was used) in contact with the underside of the substrate, to act as a heat sink [24]. To prevent excessive heat loss, and hence reduce the requirement for average laser power, this heat sink was designed to be only in contact with the centre of the package, to cool any temperature-sensitive elements inside, as shown in figure 10.

Figure 10 schematic of localised laser bonding with copper boss cooling unit

To test this cooling technique, we investigated the bonding of a TO5 Nickel cap to silicon; this particular sample was chosen as in this case truly localised heating is particularly difficult to achieve, given the high thermal conductivity of the silicon substrate. It is important to ensure a uniform temperature throughout the glass frit, to ensure that it is fully cured. As a result the approach adopted for laser heating was to scan the focused laser spot on the silicon substrate, just outside the Ni cap, in a ring of diameter 10.4 mm and width 0.35 mm.

4.1 Finite element simulation

In addition to the experiments, a finite element simulation was constructed in FlexPDE to determine the temperature field likely to be generated in this case. The simulation is based on the heat equation in an isotropic and homogeneous medium:

\[
\frac{\partial u}{\partial t} = \kappa \left( \frac{\partial^2 u}{\partial x^2} + \frac{\partial^2 u}{\partial y^2} + \frac{\partial^2 u}{\partial z^2} \right) (1)
\]

where \(u = u(t,x,y,z)\) is temperature which is a function of both time and space; \(\kappa\) is the thermal conductivity; \(c\) is the specific heat, and \(\rho\) the density.

We assumed that the laser heat source has a Gaussian Irradiance profile
where $I(r) = \frac{2P}{\pi\omega^2} (1 - R) \exp(-\frac{2r^2}{\omega^2})$ (2)

Where $P$ is the laser output power, $R$ is the reflectivity of material, $\omega$ is the radius of the laser beam.

This model was constructed for the setup shown in figure 10 and the laser energy was assumed to be absorbed at the surface of the silicon (of thickness 380 µm and reflectivity of 31%). The temperature at the bottom surface of the copper is fixed at 300 K (water cooled), and the temperature at the top layer of glass is also assumed as 300 K and the heat transfer coefficient of ambient air is 40 W m$^{-2}$ K$^{-1}$ [25]. Radiative heat transfer was also included between the closely spaced surfaces in the device and into the surrounding air. To simplify the simulation process, we assume first of all that the temperature has reached a steady state and the laser illumination on the silicon surface is continuous because of its very high scanning speed (1000 mm/s), forming a ring of diameter 10.4 mm and width 0.35 mm. However, a time-dependant simulation was also carried out to investigate the evolution of temperature with time. Results from this model are presented in figure 11(c), for 3 points on the silicon surface, namely in the centre of the silicon chip, within the glass frit ring and directly below the laser illumination track.

We also assume an imperfect thermal contact between the copper boss and silicon, as they are not completely flat, but each have a certain surface roughness; to model this we treat it as a 3 µm air gap. The outer diameter of the TO5 cap is 8.3 mm and it has a ‘lip’ of width 0.5 mm at the base, whilst the substrate is 12 × 12 mm, i.e. the laser illuminates a ring on the substrate slightly larger than the TO5 cap. Figure 11(a-b) shows the calculated temperature field arising from 61 W laser illumination.
Figure 11 Calculated temperature field (a) at the silicon substrate surface and (b) at a cross-section through the centre of the top-hat (at y = 6 mm); (the z-axis is magnified in the figure); and (c) the temporal evolution of temperature at 3 points on the silicon surface, namely in the centre of the silicon chip, within the glass frit ring and directly below the laser illumination track.

It can be seen in figure 11(a) that the temperature distribution expected within the glass frit ring is reasonably uniform. The calculated temperatures at the outer and inner edges of the glass frit ring at the silicon surface are 664 K and 626 K respectively, a difference of 38 K. Furthermore, figure 11(b) shows that the predicted temperatures on the bottom and top of the glass frit at ring centre are 644 K and 590 K respectively, i.e. a difference of 54 K. Meanwhile, the temperature at the centre of the silicon substrate should be kept to around 420 K, so the heating is properly localised.
It can be seen from figure 11(c) that after 10 s laser illumination, the temperatures in the centre of the silicon chip, within the glass frit ring and directly below the laser illumination track are 724, 634 and 418 K respectively; in each case this is at least 97% of the temperature at steady state. This indicates that the steady state model is appropriate for the laser scanning times used here.

It is noted that it is important to include the effect of radiative heat transfer in the model; if this is omitted, the simulated maximum temperature obtained with a reduced laser power of 58W is the same as that with a laser power 61W when radiation is included.

4.2 Experimental – scanning beam

A series of experiments were carried out; however there was found to be a problem on the cooling phase. A few seconds after completing the laser heating phase, a ‘cracking’ noise could always be heard. On inspection it was clear that a thermally-induced crack was occurring in the glass frit. Such a crack is likely to arise from different cooling rates in the silicon and glass frit, due to the high thermal conductivity of both the silicon substrate and Cu support/heat sink. It was found that reducing the laser power in a stepwise fashion, and adding a cooling gas flow to the cap towards the end of the heating process, could reduce the likelihood of this cracking, but not completely eliminate it.

An additional refinement was therefore made to the experimental arrangement, whereby the Cu support was moved away from the silicon substrate immediately after the laser shutter was closed, i.e. at the end of the heating cycle, whilst the cooling gas flow was maintained for a further 15 s. After optimisation of the bonding parameters, a series of 6 samples was bonded, and an improved success rate of 66% (4 out of 6) was obtained. Parameters used were: laser power of 55 W for 150 s, followed by 46 W for 10 s, and the cooling gas flow started at 135 s. Provided no cracking occurred, the leak rate was always less than $2.0 \times 10^{-9}$ mbar l s$^{-1}$. The laser was scanned over a ring of diameter 10.4 mm, on a 12 $\times$ 12 mm silicon substrate.

To determine the maximum temperature reached in the centre of the silicon sample, further experiments were carried out by coating the centre of the silicon with a temperature indicating paint (Tempilaq, Tempil Inc). With an illumination laser power of 56 W for 150 seconds, this paint demonstrates that the temperature in the centre of the
silicon was in the range 505 to 526 K, higher than indicated from the simulation (most likely due to poor thermal contact between the silicon and the copper), but still significantly below the glass frit curing temperature of around 640 K.

### 4.3 Experimental - focused ring

An axicon is a cone-shaped optic, and in combination with a positive lens can create a ‘ring focus’. Figure 12 shows a cross-section through an axicon-lens arrangement, demonstrating this effect, given the circular symmetry of the axicon.

![Figure 12 Schematic figure of an axicon ring](image)

In our setup, fibre-delivered light is collimated with a +100 mm focal length lens before passing through the axicon followed by a positive focal length focusing lens, generating a focused laser ring of diameter 12 mm and width 1 mm. The silicon substrate in this case was 14 × 14 mm, and as with the scanning system, the Ni cap is not heated directly, but rather the silicon substrate immediately surrounding the cap. As with the laser scanning technique, a cooling gas flow was used towards the end of the heating phase, and the silicon separated from the Cu cooling block at the end of the heating phase. An illumination laser power in the range of 60 to 68 W was used for 100 s, and then reduced to 45 W for 10 s. A cooling gas flow was started 85 s after the start of the heating phase. In all cases, the parts were successfully joined (20 parts), with a leak rate of 1.7 × 10^{-9} \text{ mbar l s}^{-1} or less. Measurements with Tempilaq paint demonstrate that the maximum temperature was in the range 505 to 526 K.

### 5 Conclusions and discussions

Knechtel [20] states that the advantages of glass frit bonding are high bonding strength and process yield, low stress at the bonding interface, good process repeatability, hermetic sealing and non-stringent requirements for the flatness of
the surfaces to be bonded. The laser-based process investigated in this paper combines these advantages with localised heating and a high speed process.

In this paper, we have demonstrated the versatility of laser-based bonding using glass frit for a range of miniature packages and materials, where hermetic seals are required. We have demonstrated that the process is highly suited to rough or non-flat surfaces; indeed with a smooth surface it is beneficial to machine micro-slots into the surface before joining. Care must however be taken when using localised heating to join materials of differing thermal parameters; differential cooling can lead to stress across the join and hence failure by cracking of the glass frit. A potential solution has been demonstrated in this paper for a particular sample configuration.

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References


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Prof. Hand is a Fellow of the Institute of Physics, and Consultant Professor of Beijing University of Posts and Telecommunications.