Part One Technologies

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# A. Adhesive and Anodic Bonding

# 1 Glass Frit Wafer Bonding

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Glass frit wafer bonding is widely used in industrial microsystems applications where fully processed wafers have to be bonded. This end-of-process-line bonding must fulfill some very specific requirements, such as: process temperature limited to 450 °C to prevent any temperature-related damage to wafers, no aggressive cleaning to avoid metal corrosion, high process yield since wafer processing to this stage is expensive, bonding of wafers with certain surface roughness or even surface steps resulting from metal lines electrically running at the bonding interface to enable electrical connections into the cavity sealed by the wafer bonding, as well as a mechanically strong, hermetically sealed, reliable bond. All of these requirements are fulfilled by the glass frit wafer bonding process, which additionally can be very universally applied since it can be used to bond almost all surfaces common in microelectronics and microsystem technologies.

## 1.1 Principle of Glass Frit Bonding

The basic principle of glass frit bonding is the use of glass as a special intermediate bonding layer. This glass must have a low-temperature melting point. In the bonding process, the glass between the wafers to be bonded is heated, so that its viscosity continually decreases until the so-called wetting temperature is reached, at which point the glass is soft enough and liquid enough to flow and wet the wafer surface. In this flowing process, the glass comes into contact with the surface to be bonded at the atomic level, and flows into surface roughnesses and around surface steps. As a result, perfect sealing behavior is achieved by the liquid glass; hence, this bonding process is also called seal glass bonding. On cooling the glass in the wafer stacks, it re-solidifies, to finally form a mechanically strong, hermetically sealed bond. Due to its excellent bonding and flowing behavior, glass frit bonding is very often used to cap microelectromechanical systems (MEMS) at the wafer level by simultaneous sealing under vacuum or low pressure, which is required for resonant devices such as gyroscopes. Figure 1.1 shows an example of a glass-frit-bonded gyroscope. The flowed glass frit is highlighted by the materials

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**Figure 1.1** Example of glass frit bonding as a process for MEMS capping: flowing and sealing behavior at signal metal lines into a sealed cavity.

contrast of the scanning electron microscope (SEM), so that its sealing behavior is well illustrated.

### 1.2 Glass Frit Materials

Essential for glass frit bonding is the glass material which ultimately forms the bond between wafers. Low-melting-point glasses which can reflow in the temperature range of 400 to 450 °C are very special glass materials with a rather unstable glass matrix. Such materials are very difficult or nearly impossible to deposit by classical layer deposition techniques such as sputtering, spin-on deposition, or chemical vapor deposition (CVD). Additionally, a layer thickness of 5 µm or greater is required to allow a good reflow of the glass during bonding, to ensure excellent sealing by the glass flowing into surface roughnesses and around surface structures. Since glasses can be transformed into pastes, screen printing is the best method for bringing the glass frit onto one of the wafers to be bonded. To prepare such pastes for screen printing, a low-melting-point glass, typically a lead zinc silicate glass or lead borate glass, is ground to particles with a size less than 15 µm. This glass powder is mixed with an organic binder, to form a paste. In order to achieve optimum mixing, the mixing is done with a milling tool. Additionally, the paste contains solvents for tuning the viscosity to obtain the best screen printing results (300-600 P (30-60 Pas)). Because the thermal expansion of the low-meltingpoint glasses is typically significantly higher than the glass surfaces to be bonded, filler particles are added to tune the thermal expansion of the final glass frit. These filler particles have a much higher melting temperature than the active glass component, so they are built into the refused glass frit and reduce the thermal expansion. For example, by adding barium silicate glass ceramic fillers to lead zinc silicate glass frits, the thermal expansion coefficient can be reduced from  $10 \times 10^{-6}$ to  $8 \times 10^{-6} \,\mathrm{K}^{-1}$  which finally gives a stress reduction in the glass-frit-bonded wafer stack. Glass frit pastes are offered by various vendors of glass or electronic materials, such as Ferro Corporation, Schott Glass, and DIMAT, typically as ready-to-use pastes. For high-temperature glass frits, there is a wide range of different materials

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Figure 1.2 Main composition of Ferro FX-11-036 glass frit paste.

available, but for the low-temperature range (<450 °C) that is required to bond processed wafers, only a few materials are available. The most commonly used paste is Ferro FX-11-036 [1], which is also upon which the following exposition is based. The reason for the popularity of this material is that it does not tend to crystallize. This facilitates the thermal processing of the glass frit, since, otherwise, crystallization would change the glass dramatically by increasing the reflow temperatures, which would make final bonding impossible. The composition of Ferro FX-11-036 is shown schematically in Figure 1.2.

In addition to the ready-to-use commercial glass frit pastes, there is also the option to mix special glass frit pastes based on glass material from special melts or special glass powders. However, the mixing of a screen-printable glass paste is very difficult since the binder has to have a rather high viscosity.

From the environmental protection point of view, the lead in glasses used to reach very low melting points is critical. In the RoHS directive there is still an exception for lead in glasses of electronic components, and the total amount of lead in glass-frit-bonded chips is too small to be in conflict with automotive limitations. Nevertheless, the lead in glass frits should be replaced in the medium term. For example, at Ferro, lead-free glass frits for wafer bonding are in development and should be considered for any new product or process developments.

#### 1.3

#### Screen Printing: Process for Bringing Glass Frit Material onto Wafers

For glass frit bonding, glass layers that are several micrometers thick are required to allow a reflow of the material in the wafer bonding process, which ultimately allows the planarizing and sealing of wafer topographies. Such thick layers are relatively easy to deposit using screen printing technology, which offers the additional benefit that the material is structured directly with the deposition. Using this method, glass frits can also be printed on structured wafers, for example on cap wafers with through-holes. During screen printing, the structures to print are realized as openings in a screen film layer which is supported by a mesh structure stretched onto a supporting frame. The film structure is realized by photolithographic methods. The thickness of the screen wires and the screen defines the thickness of the print. The mesh width is related to the paste to be printed, and should be three times the size of the particles. The minimal structure width should



Figure 1.3 Principle of screen printing for glass frit bonding.

include three meshes, to allow a safe screen printing process. For typical glass frit materials, with particle sizes up to  $15 \,\mu$ m, the mesh width is  $45 \,\mu$ m at a wire thickness of about  $35 \,\mu$ m and a film thickness of about  $10 \,\mu$ m. Polymer meshes are mainly used. Structures of width in the range  $150-300 \,\mu$ m at a wet thickness of about  $45 \,\mu$ m can be realized with glass frit pastes. Details of screen materials construction and selection are available at www.koenen.de. For screen printing, the wafer to be printed on is placed underneath the mesh. The wafer is aligned to the mesh by an automatic vision system using special marks on both the wafer and the mesh. In the first step, a squeegee floods the mesh with the glass paste, to fill the mesh openings with the paste. In the second step, the wafer is printed by pressing the mesh to the wafer with the squeegee, and moving it over the mesh area. Figure 1.3 illustrates the screen printing process.

The high-precision screen printing that is required for glass frit bonding (the bond frames should be as small as possible and exactly positioned, since the bond area increases the chip size and, as a result, the cost) is a very sophisticated process. Structure size, mesh travel, squeegee force and speed, as well as paste properties such as viscosity and particle size have a strong influence on the structure definition and placement accuracy. To define general design rules is very difficult, but in practice the following tolerance chain should be considered:

- mesh opening as designed;
- increasing of structure at printing (underflow of paste to mesh);
- misalignment at screen printing and misalignment by screen stretching.

Based on this, an area of up to  $400\,\mu$ m could be required for printing minimal designed frames of  $150\,\mu$ m width on unstructured silicon wafers. For this, the tolerances of the bonding process (glass out-flow and misalignment) must be additionally considered, which finally leads to an area requirement of up to  $500\,\mu$ m in width for microsystem wafers which should be capped by glass frit bonding. Of course, this is much too high an area consumption, so that a self-aligned screen printing process is recommended. Here, structures etched into the wafer to be printed on-mostly such wafers have to be structured anyway, so that this mask layer can also be used for this-define the bond frames. Since screen printing is based on wetting of the surface with the paste, the printing is limited by the structures, even if the mesh opening is wide. With this approach (Figure 1.4), very



Figure 1.4 Principle of self-aligned screen printing.



Figure 1.5 Influences of structure width on printed glass frit thickness.

well-defined structures with minimal width and without screen alignment influences can be realized, and the total area consumption on the system wafer is reduced to about  $250-300\,\mu$ m in width.

Finally, the printed glass frit frame must be considered as a three-dimensional object. The thickness of the glass is mainly defined by the mesh construction (wire and film thickness), but is also influenced by many other factors. Therefore, the final thickness after bonding typically varies in the range  $5-15\,\mu$ m, and should not be used to define functional gaps in microdevices. For the bonding, the actual glass thickness is not very critical, as long it is above  $5\,\mu$ m, to ensure the sealing of surface profiles. More critical is the variation of the glass thickness within the structures which should create a hermetic bond. Even if the glass flows during the bonding process, the planarizing behavior is limited. Since the main influence of the printed structure thickness is the width (Figure 1.5), particularly for small bond frames, it must be ensured in the design that all structures on one wafer have one and the same width. Even corners of rectangular structures show increased thickness, but these are within the limits of the glass flow, and no special

design measures such as corner rounding have to be considered, since they would rather introduce risk for structure integrity, due to the limited resolution of the screen.

### 1.4

# Thermal Conditioning: Process for Transforming Printed Paste into Glass for Bonding

When bond frames are printed with glass frit paste onto one of the wafers to be bonded, the material is not already usable for bonding. The material is a compound of glass and filler particles held in a binder. If this configuration were be bonded at the process temperature at which the glass compound is melted and wets surfaces, a bond would occur, but it would be neither strong nor with hermetic sealing, because the organics from the binder and solvents would cause large bubbles in the glass, as shown in Figure 1.6.

Therefore, a thermal preprocess to burn out the primer and to transform the paste glass into real pre-melted glass is essential for a high-quality bond. This thermal process consists of three steps. First, and directly after screen printing, the glass paste has to be dried and stabilized at 120 °C. In this step, most of the solvents disappear and the polymer binder is internally crosslinked and stabilized; with this step the glass paste becomes stable enough for storage, easy handling, and shipment, if required. Second, the binder is burned out at 360 °C. At this temperature the glass is not really melted, but its viscosity is significantly reduced, so that the glass particles take on a round, droplet-like shape and form an initial adhesion to each other and to the base of the wafer on which they are situated. This microstructure is very important for two reasons: (i) it is an open sintered-like structure, which allows complete burning out of the binder and (ii) it has a certain mechanical stability to maintain the shape of the printed structure. Third, after



**Figure 1.6** Bubble generation in glass frit band if binder is not completely burned out: (a) cross section; (b) after debonding.

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the paste is completely burned out, which takes about 10 min, the temperature is increased to 450 °C; the glass particles are completely melted at this temperature and fuse into solid glass. If filler particles for thermal expansion coefficient management are in the paste, these are enclosed by the glass but not fused into it, which is not required for the targeted effect. Table 1.1 illustrates the thermal conditioning process; the actual temperatures must be cross-checked with the glass frit paste specification, but temperature tolerances of  $\pm 10$  K are not critical.

In thermal conditioning, the glass paste is not only transformed into a bulk glass, but also the structure is densified. Thus, the structure thickness after screen printing is reduced by a factor of about three, but the structure shape and width are not changed. Even if the viscosity of the glass in the pre-melt is low enough to wet the surface, it is not low enough to allow the glass to flow. As a result, the lateral structure is finally defined during the screen printing. Due to the surface tension, the free surface of the glass frit is bowed. Figure 1.7 shows the densification during thermal conditioning.

This multiple-temperature glass-conditioning process can either be done at one profiled temperature step or as three separate processes. It is recommended to keep the wafers horizontal during the thermal processing in order to prevent gravitation-related flowing of the glass structures. Some glass frit materials have a tendency to crystallize. Glass crystallization must be prevented during the conditioning through the use of precise temperatures and defined rates of temperature change, because the glass properties are dramatically altered by crystallization. In particular, after crystallization, the processing temperatures are increased, so that the glass cannot be re-melted in the bonding process, the result being that finally the glass frit is no longer bondable. Crystallization, or deglazing, occurs if on cooling the glass reaches a viscosity range in which the amount of internal glass nucleation and crystallization speed are maximal. Figure 1.8 shows a glass crystallization diagram [2] and an example of a crystallized glass frit. For glass frit materials deposited as glass paste, nucleation is increased due to organic residues coming from the binder, so the probability for crystallization is increased. The crystallization of the glass can be prevented by the glass chemistry: if the number of nucleation points and the crystallization speed do not have their maxima in the same viscosity range, crystallization practically does not occur. This explains why some glasses have a tendency to crystallization and others do not. If crystallization is observed during processing of glass frit materials, the cooling rate of the thermal processing should be varied experimentally. Normally faster cooling should prevent crystallization, but provides a risk of thermal cracks in the glass. On the other hand, glass crystallization can be actively used in the later bond process: if the glass crystallizes on cooling after bonding, a thermally much more stable bond is created. However, the process window for such bonding appears quite narrow.

After this multiple-step thermal treatment, the wafers with the glass frit are finally ready to bond.

Table 1.1	Overview	of	glass	frit	conditioning	process

Thermal conditioning step	Process temperature (°C)	SEM images	Schematic images	Remarks
Drying and binder polymerizing	120	Flown in order at Printed structure ecce 24 CH 24 CH 24 CH 24 CH 25 CH 2	Glass frit paste Glass frit paste Si-Wafer Printed paste Paste with polymerized bin der	<ul> <li>Solvent drying</li> <li>Binder</li> <li>polymerizing</li> <li>No change in</li> <li>glass or binder</li> </ul>
Binder burn-out	360	Ender remuved by burning out Call 20 T dark out out, 35 var 20 da Glace paticles start melling Filer particles mol meller	Binder burn-out	<ul> <li>Sintered-like structure</li> <li>Glass melting starts, but with high viscosity</li> <li>Fixing glass particles to substrate and to each other</li> <li>No change in filler particles</li> </ul>
Glass pre-melting	450	Cline pericips in the molecular discussion Edge of sources particle discussion Edge of sources particle discussion Edge of sources and particle discussion (the molecular discussion) (the molecular discussion) (	Start of pre-melting Completed pre-melting	<ul> <li>Binder burn-out</li> <li>Complete pre-melting of the glass</li> <li>Starting from wafer as heat carrier</li> <li>Void-free glass</li> <li>Filler particles sealed but not fused into the glass</li> </ul>



Figure 1.7 Changes in screen-printed glass frit structures during thermal conditioning.



Figure 1.8 Critical glass frit crystallization: relation to glass processing and SEM image of crystallized glass frit.

## 1.5 Wafer Bond Process: Essential Wafer-to-Wafer Mounting by a Glass Frit Interlayer

The actual bonding process is comparable to other wafer bonding technologies. Since glass frit bonding is typically done at structured surfaces, both of the wafers to be bonded must be aligned before they are loaded into a standard wafer-bonding chamber. In the chamber, the wafer pair is heated to the wetting temperature of the glass frit, which is normally in the range 440–450 °C. At that temperature, the glass flows and wets the surfaces to be bonded. The bonding process, which is, from the physics, driven by temperature, has to be supported by mechanical pressure on the wafers, in order to compensate for wafer bow influences and screen printing inhomogeneities. In the bonding process there is another geometrical transformation of the glass, where it really flows from the bond frame as shown in Figure 1.9. When the glass has flowed and wetted the surfaces to be bonded, the wafer stack can be cooled. The glass frit solidifies and forms a mechanically strong bond.

The parameters of temperature and pressure for this bonding process must be very carefully adjusted. If the temperature and pressure are too low, there is no or just local bonding. If one of these parameters is too high, the glass may flow too much and reach functional structures, where it may block or destroy them. This tuning process has to be done for each bonding machine, and when its



Figure 1.9 Schematic of progress of glass frit bonding (cross section).



Figure 1.10 Parameter field evaluation of glass frit bonding process.

configuration is changed. An essential requirement here is that the chuck and tool plate are very parallel to ensure homogeneous bonding and glass flow. Figure 1.10 illustrates this kind of parameter optimizing process. Here, the silicon wafer with a glass frit is bonded to a glass wafer, which allows very easy inspection of the glass frit flow.

The flowing glass allows hermetic bonding of rough or structured surfaces, even if they also have surface steps such as from metal layers. However, to ensure hermetic bonding, the glass must be hot enough to flow well.



Figure 1.11 Fusing effect of surface layers: (a) CVD oxide; (b) silicon.

The physics of glass frit bonding is related to the glass chemistry [2, 3]. When the glass wets the surface to be bonded at high temperature, thin layers of the material to be bonded are fused into the glass, so that finally after cooling, a continuous material interaction occurs which forms a strong bond. This layer fusing can be investigated using SEM cross sections (Figure 1.11). For CVD oxides (e.g., tetraethylorthosilicate), layer fusing can reach several tens of nanometers and can be evaluated if there is any masking material (e.g., metal lines in the bonding interface). On bonding silicon this kind of fusing cannot be observed, but lead precipitates (ball shaped) indicate that a redox reaction has taken place for which some silicon has to be fused into the glass frit. These lead balls are not critical for bonding strength [4], but could cause leakage currents if unpassivated metal lines are sealed with the glass frit.

It is very critical in glass frit bonding to maintain the wafer-to-wafer alignment accuracy during the bonding process. As mentioned earlier, the wafers are slightly pressed together when the glass is soft. Any sideways force can cause a shift of the wafers very easily. To prevent this, the following measures are recommended:

- The glass bonding should be done with closed fixture clamps to fix the wafers until bonded.
- The bonder chuck and tool plate should be as parallel as possible to prevent wafer-to-wafer sliding.
- Chuck and tool plate should be made from silicon or silicon carbide to reduce thermal mismatch with the wafers to be bonded for preventing lateral forces which can cause wafer-to-wafer shifting and thus poor post-bond alignment.

If all of these points are considered, wafer alignment accuracies of better than  $5\,\mu m$  can be achieved as state of the art for glass frit bonding, as confirmed in Figure 1.12.

If bonding under vacuum or defined gas pressures is required, it is not necessary to use spacers. The glass frit shows a surface topography before bonding that allows the passage of gasses when the wafers are in contact at the cold stage.



Figure 1.12 Alignment accuracy after glass frit bonding.

### 1.6 Characterization of Glass Frit Bonds

The bonding interface must be strong enough to withstand subsequent processing steps such as dicing and packaging. Furthermore, the bonding has to ensure good protection of the active structure during its lifetime. It has been shown that almost all relevant layers for microsystem processes can be bonded with high yields using glass frit bonding. Stud pull tests have shown that the bonding interface is strong enough to fulfill these requirements. Figure 1.13 illustrates the results of pull tests on diced chips. The fracture load varies a little, but is high enough for most applications and comparable to the bonding strength that can be achieved with anodic bonding.

Hermetic sealing is often required to ensure the correct functioning and sufficient reliability of capped micromachined components. Therefore, hermeticity investigations were performed on glass-frit-bonded devices. Because of the lack of MEMS hermeticity testing standards, resonant structures were chosen, whose frequency behavior depends on the gas pressure in the space surrounding the structures. The structures we used show only resonant behavior when moving in vacuum (Figure 1.14) and therefore provide a very sensitive pressure indication. Testing of several resonant structures has shown that a vacuum of about 1–5 mbar can be sealed using glass frit bonding. The minimum sealing pressure is related to glass frit outgassing of remaining organics. After storage of several weeks, no shift of the sealed cavity pressure was observed.

Besides these general wafer bonding requirements and, further, bonding strength and yield, hermetic bonding, and universal usability, glass fit bonding shows some additional advantages:

- bonding of CMOS wafers is possible without adversely influencing them;
- direct sealing of unpassivated metal lines is possible;



**Figure 1.13** Results of bonding of various surface layers. (TEOS, tetraethylorthosilicate; ITO, indium tin oxide.)

- very good reliability;
- very low mechanical stress on bonded mechanical components;
- very safe, reproducible process;
- bonding of structures with through holes is possible;
- no surface activation is required.

### 1.7 Applications of Glass Frit Wafer Bonding

Many different applications for glass frit bonding are possible, because of the universal characteristics of this bonding technology. The main application for glass frit bonding is the encapsulation of surface micromachined sensors such as





Figure 1.14 Hermeticity evaluation using resonant structures.



Figure 1.15 Examples of wafer bonding applications.

gyroscopes (Figure 1.15) and acceleration sensors. With these sensors, the main advantages of glass frit bonding, such as hermetic sealing, metallic lead-throughs, narrow bonding frames, and high bonding yield, play an important role. Furthermore, fully processed cap wafers with cavities above the sensor structures and holes for wire bond pad access can be bonded, so that the cap bonding is the last wafer process step. This sensor technology using glass frits has been introduced into production and is available as a MEMS foundry process. Glass frit bonding can also be used successfully for several additional applications, such as bulk micromachined sensors, the sealing of absolute pressure sensor cavities, the mounting of optical windows, and the capping of thermally active devices.

### 1.8 Conclusions

Glass frit bonding technology provides a wide range of possibilities for the bonding of wafers at process temperatures below 450 °C. The screen printing of the bonding glass as a paste allows *in situ* deposition and structuring on processed cap wafers.

The structured bonding layer protects moveable structures from parasitic bonding. Following this, organic materials are eliminated from the paste by temperature cycling. The bonding is a thermo-compressive process. The bond is formed at 430 °C with a slight pressure applied. Almost all surface layers commonly used in silicon micromachining can be bonded using glass frits. The main advantages of glass frit bonding are hermetic sealing, high process yield, low mechanical stress at the bonding interface, possibility of metallic lead-throughs, high bonding strength, and good reliability. For various applications, the technological potential and the universal use of glass frit bonding have been shown. Critical for some applications may be the high process costs related to the glass frit material and the complex processing.

### References

- Ferro (2002) MEMS and sensor materials, 11-036 sealing glass specification, Rev. 1200, Ferro Electronic Materials, Santa Barbara, CA.
- 2 Zinke, A. (1996) Technologie der Glasverschmelzungen, Technischphysikalische Monographien Band 12, Akademische Verlagsgesellschaft Geest und Portig K-G, Leipzig.
- **3** HVG (1995) *Fügen von Glas: HVG Fortbildungskurs*, Verlag der Deutschen Glastechnischen Gesellschaft, Frankfurt am Main.
- **4** Boettge, B., Dresbach, C., Graff, A., and Petzold, M. (2008) Mechanical characterization and microstructure diagnostics of glass frit bonded interfaces. *Electrochem. Soc. Trans.*, **16** (8), 441–448.