Chem. Eng. Comm., 194:1259–1270, 2007

ISSN: 0098-6445 print/1563-5201 online
DOI: 10.1080/00986440701294007

Application of Controlled Thermal Expansion in Microlamination for the Economical Production of Bulk Microchannel Systems

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Diffusion bonding has been widely used within microlamination architectures for the fabrication of micro energy and chemical systems (MECS). MECS are microsystems with the ability to process bulk amounts of fluid within highly parallel microchannel arrays capable of accelerated heat and mass transfer. Thus far, diffusion bonding of microchannel arrays is commonly done in a vacuum hot press system. The use of the hot press greatly restricts the production rate due to vacuum pump-down time and heating-up and cool-down periods. Furthermore, larger substrates are gaining interest in the system design of MECS devices, and it is not apparent that uniaxial pressing within a hydraulic vacuum hot press will provide the bonding pressure uniformity necessary for large substrate bonding. This article presents a novel fabrication approach for high-volume thermal bonding of MECS devices with the use of controlled thermal expansion. A thermal bonding fixture based on the principle of differential thermal expansion was developed with a focus on controlling the bonding pressure magnitude, the pressure timing, and its sensitivity. The application of such a fixture within a conveyorized furnace system could be the key to a continuous thermal bonding approach for the mass production of MECS devices.

Keywords Bonding; Differential thermal expansion; Microchannel arrays; Microlamination

Introduction

Today, the function of many bulk microfluidic devices has been validated in the laboratory (Martin et al., 1995; Martin et al., 1998a, b; Martin et al., 1999a, b; Paul et al., 1999; Kaemper et al., 1997; Loewe and Ehrfeld, 1998; Bachman et al., 1999; Pua and Rumbold, 2003). Currently, the majority of these devices are produced by microlamination (Paul and Peterson, 1999) through the patterning and bonding of thin layers of material including metal shimstock or polymer films. Diffusion bonding or diffusion brazing are typical bonding processes used in metal microlamination. These methods are typically conducted with the use of a vacuum hot press (VHP).

The future development of bulk microfluidic devices made of metal such as microchannel heat pumps or microchannel fuel reformers will require the integration of many different unit operations at low cost and high production volume. The full

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commercial potential of bulk microfluidic systems will be realized through the integration of multiple highly paralleled unit operations into one single device (Wegeng et al., 1997, 1998). The differential thermal expansion (DTE) bonding unit presented in this article could be a key technology for the mass production and commercialization of such microlaminated devices.

DTE has been used to align layers in the lamination of multilayer integrated circuits (IC) in the electronics industry (Pommer, 2004) and also in the microlamination of MECS devices (Paul and Thomas, 2003). DTE has also been used in processes where the clamping of a part is needed during a thermal cycle such as in physical vapor deposition (Nulman and Davenport, 1995) or in the thermal joining of conveyor belts (Willis and Willis, 2001). The use of thermal expansion as a driving force for bonding pressure application during microlamination was first suggested by Pacific Northwest National Laboratory (PNNL) in 1999 (Martin et al., 1999b) in an article on the lamination of ceramic microchannel components, although no work on this concept was ever reported. More recently, IBM has filed a patent application that claims the method for application of pressure to a workpiece by thermal expansion (McHerron et al., 2003). This application relates to the high-volume fabrication of multilayer thin film structures in the semiconductor industry and is considered for low-temperature applications used for the lamination of polymer circuit boards. The sections below explain the general concept of a DTE bonding unit and help to differentiate the present work from prior efforts.

Theoretical Concept and Pilot Study

The general concept of a bonding fixture based on the principle of differential thermal expansion for the application of bonding pressure consists primarily of a frame, two bonding platens, and, in the simplest case, an expansion block interposed between them, as shown in Figure 1. The rigid frame is composed of a low thermal expansion material. The expansion block (expander) has a significantly higher coefficient of thermal expansion than the frame of the fixture. All things being equal, the

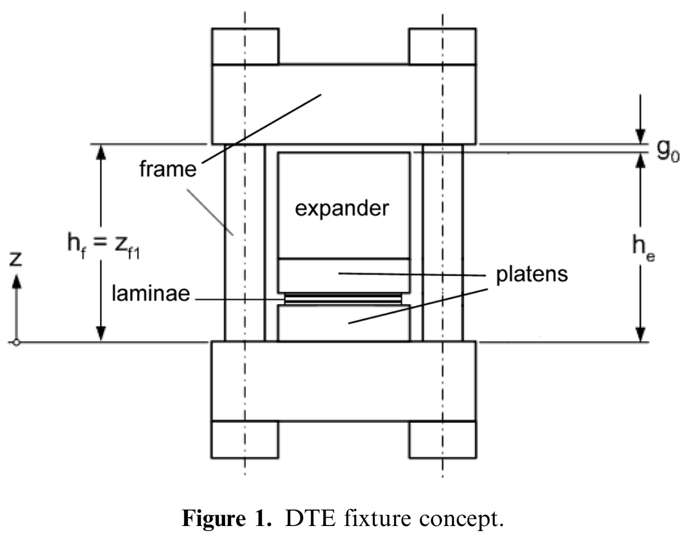


Figure 1. DTE fixture concept.

height of the expansion block is directly proportional to the amount of clamping pressure to be delivered. Between the expansion block and the frame are placed the bonding platens between which the laminae are aligned and stacked. In this case, the bonding platens serve to prevent solid-state diffusion to the fixture parts and may be made of graphite or some other suitable material. As a guideline, the coefficients of thermal expansion should preferably differ at least by a factor of two to guarantee functionality of the fixture.

When the bonding unit is heated up to the bonding temperature, the expansion block and the platens inside the frame expand relative to the frame by the difference in the sum of their coefficients of thermal expansion scaled by the product of the height of the expansion block and the change in temperature. An initial gap (g_0) can be designed into the fixture assembly to scale and time the application of the bonding pressure. The gap is equal to:

$$g_0 = h_f - h_e \tag{1}$$

where h_f is the height of the frame standoffs and h_e is the height of the expander, platens, and laminae combined.

As soon as the initial gap is consumed (i.e., inner parts come into contact with the frame), compression is applied to the laminae and increases with increasing temperature. An initial test fixture, shown in Figure 2, was developed to demonstrate the feasibility of bonding laminae by the use of differential thermal expansion. Prior to the experimental pilot test the generation of bonding pressure was quantified by a finite element model. The model has shown a bonding pressure of 7 MPa at a temperature of 800°C between the bonding platens. The expansion block was conically shaped to uniformly distribute the pressure to the graphite platens. Highly expanding stainless steel 321 was selected as the material for the expansion cone. The threaded core and base were built of a lower expanding Inconel™ nickel alloy.

Five doughnut-shaped copper (alloy 110) layers were bonded successfully with this test fixture and proved the feasibility of diffusion bonding with the use of thermal expansion. This basic concept is the same as the aforementioned IBM patent

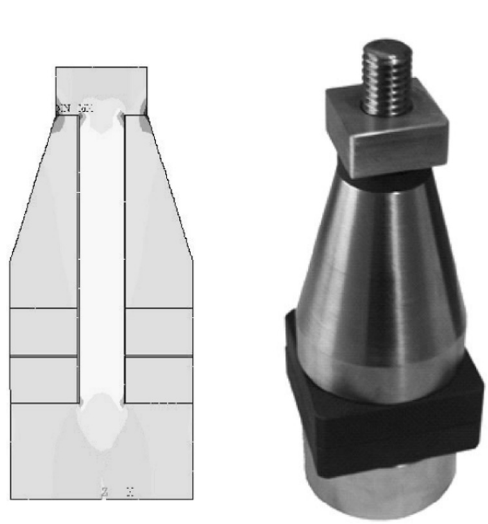


Figure 2. FE model and initial test fixture for bonding pilots.

application. Using the finite element analysis (FEA) model developed, it was found that small changes in either the bonding temperature (±5°C) or initial gap (±5 μm) can result in large variations in bonding pressure (more than ±100%). This is due to the fact that changes in the bonding pressure within the stack are regulated mainly by the elastic modulus of the materials within the stack. Further, the IBM method makes no allowances for strains produced laterally on laminae during bonding due to DTE between fixture components (e.g., platens) and the laminae. These transverse stresses have been known to produce fin warpage within microlaminated structures produced from thin laminae (i.e., < 100 μm thick) and are normally taken care of within a VHP by applying bonding pressures at the final bonding temperature. The purpose of this article is to demonstrate a novel method for better controlling thermal expansion within DTE fixtures.

Novel Fixture Design to Control Bonding Conditions

Experimental and theoretical studies of DTE-bonding fixtures have shown that it is difficult to control the resulting pressure distributions within DTE fixtures. To avoid bonding pressure distribution issues that can lead to device leakage, fin warpage, and/or flow maldistribution within bulk microfluidic devices, it is necessary to (1) dampen the amount of pressure caused by DTE at the bonding temperature to minimize pressure variations due to thermal fluctuations or gap adjustments, and (2) avoid putting pressure onto the stack until just before the bonding temperature. The second requirement extends from any DTE behavior between the laminae and the bonding platen in the lateral dimension. One way to control the timing of applying bonding pressure is to set an initial gap to offset the point of contact until just before the final bonding temperature. However, to be effective, the pressure must be applied as a step function with low-pressure sensitivity to temperature or dimensional fluctuations.

A more sophisticated design of the DTE bonding fixture uses high-temperature spring elements to decrease the pressure sensitivity of the bonding fixture while allowing for rapid application of bonding pressure closer to the intended bonding temperature. The resulting bonding pressure is the quotient of the force delivered by spring compression over the cross-sectional area of the laminae. By introducing spring elements into the fixture design, additional DTE beyond the bonding temperature will be consumed by the springs and the magnitude of the bonding pressure will no longer be dependent on the modulus of fixture materials. For this application, disc (Belleville) springs were used due to the need to provide high loads with minimal compression. In addition, disc springs are available in many different materials and alloys, some of which, for instance, are capable of working at high temperatures. Taking this concept a step further, the spring elements can also be "pre-loaded" to administer the desired final pressure level, as shown in Figure 3. The springs are pre-loaded by screws in the base plate of the bonding unit. The appropriate amount of pre-load force can be applied with a hydraulic press and the screws made snug during the pre-load procedure to secure the appropriate amount of spring compression. During a bonding cycle, as soon as the expansion block comes in contact with the frame of the fixture, the pre-loaded force is transmitted into the laminae stack applying the pre-adjusted pressure. The timing of the force release can be controlled by a setscrew in the fixture frame. The initial

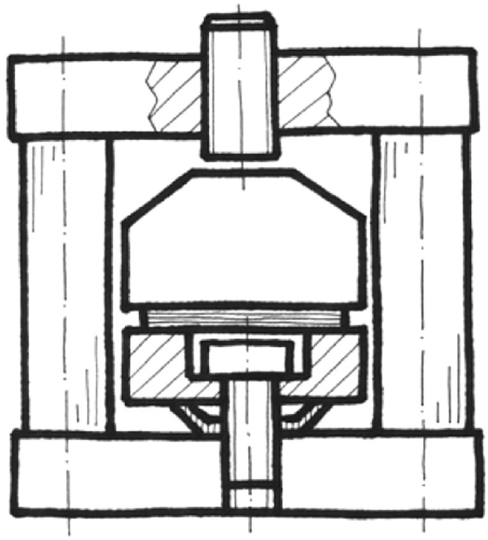


Figure 3. DTE fixture concept with pre-load mechanism.

gap has to be adjusted so that contact is made a few degrees below the final bonding temperature to guarantee release of the pre-loaded force onto the laminae.

During the bonding procedure, the DTE behavior of the fixture follows fluctuations in temperature and can be modulated by the spring constant. Consequently, a thermal expansion fixture with spring elements will be less sensitive to changes in bonding temperature than prior methods. However, the present concept involves some sophistication in the selection of appropriate materials and components to allow the fixture application at high temperatures without diffusion bonding structural components together or reaching the limits of the maximal service temperature.

To provide a more fundamental understanding of this fixture concept for design purposes, a finite element model was created in ANSYS as shown in Figure 4. A theoretical assessment of the bonding pressure, timing of the pressure, and its sensitivity based on changes in bonding temperature, material dimensions, and material properties was of interest in the design and optimization of a prototype fixture for experimental studies. The prototype fixture was composed of multiple high-temperature materials selected depending on their thermal expansion potential. The base and the top of the thermal expansion fixture are made of molybdenum connected by four ceramic (Aremcolox 502-1100) standoffs to guarantee a rigid and low expanding fixture frame. The test articles were placed between isotropic graphite (ISO-63) bonding platens and aligned by three tungsten alignment pins. On top of the graphite bonding platen the engagement block was placed and centered. The engagement block was fabricated out of high-temperature stainless steel (AISI 321) with a high coefficient of thermal expansion to drive the main displacement of the load cell sitting on top of it. The load cell consists of two molybdenum platens with four integrated Inconel™ 718 high-temperature disc springs. The device is held together by four high-temperature fasteners and is mainly responsible for controlling the magnitude of the bonding pressure. The initial gap between the load cell top and the fixture frame was adjusted with a finely threaded setscrew in the top plate of the fixture frame to control the

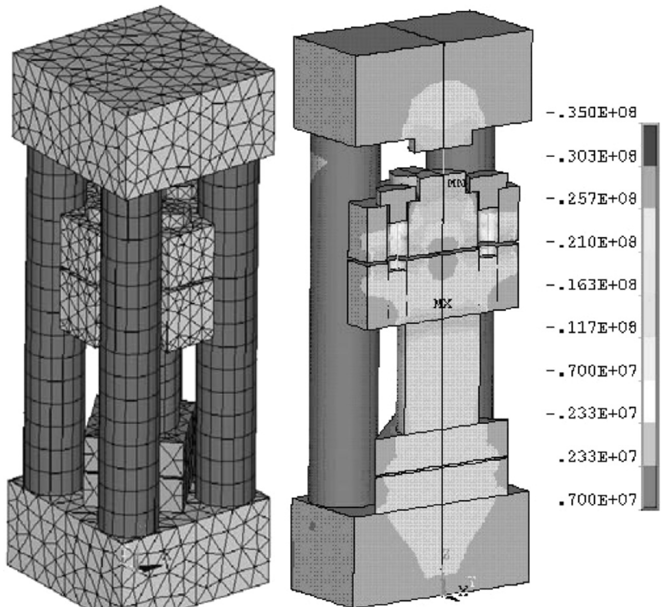


Figure 4. FE model of the DTE fixture prototype.

timing of the bonding pressure. The setscrew is made of graphite to prevent diffusion bonding to the molybdenum top plate.

The gap size can be adjusted by fitting a shim or assembly of shims according to the calculated gap size. The use of a feeler gauge with a variety of precise shims is favorable for the adjustment of the initial gap and therefore for setting the timing of the bonding pressure. Based on the initial gap size, the timing of the pressure can be controlled as shown in Figure 5. As soon as the load cell contacts the fixture frame, the pre-loaded force is distributed throughout the bonding fixture. The remaining portion of the target bonding pressure will be provided by the additional compression of the load cell depending on the difference between contact and bonding temperature. For lower temperature contact, the pre-loaded force is adjusted according to this additional compression due to DTE. The slope of the additional pressure increase is suggestive of the pressure sensitivity of the fixture and is mainly dependent on the total spring constant of the load cell and the DTE behavior. The finite element simulations reached a final pressure of 4.07 MPa, which is only 1.75% off from the target bonding pressure of 4 MPa. These results provided evidence for the feasibility of the DTE fixture design concept. Consequently, a prototype fixture was built as shown in Figure 6.

Experimental Results

Several experiments have been conducted to validate the functionality of the proposed bonding fixture. Initially, Fuji pressure-sensitive film was used to evaluate pressure uniformity, magnitude, and timing in the low temperature regime

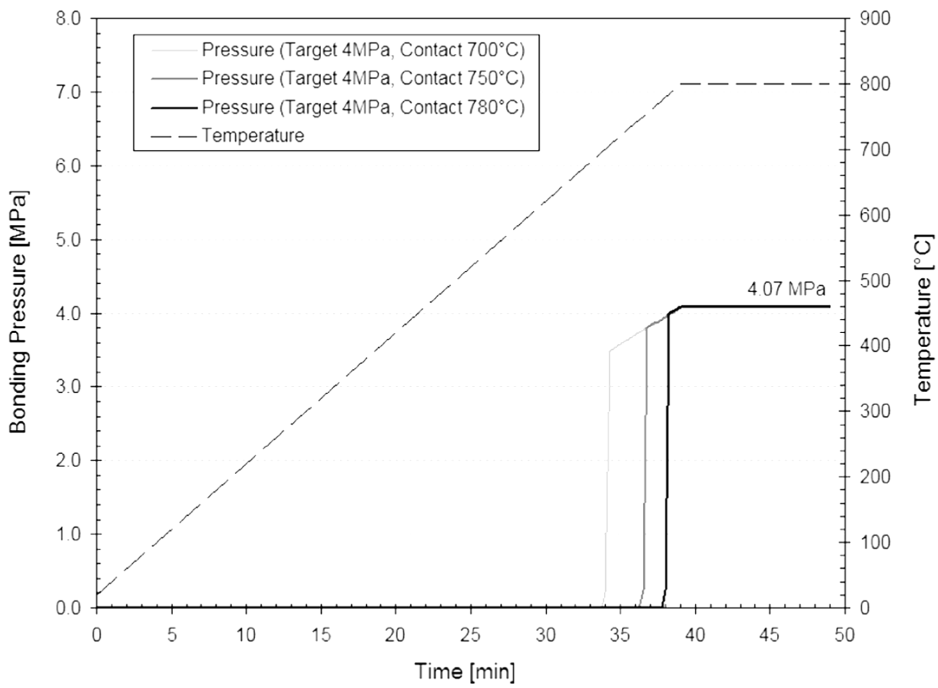


Figure 5. Theoretical pressure engagement of FE model DTE fixture.

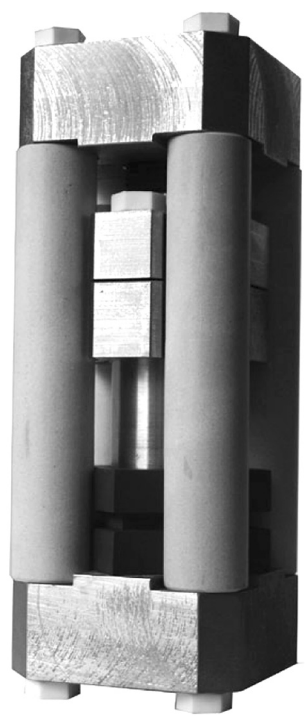
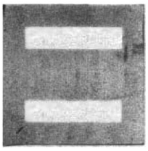


Figure 6. DTE fixture prototype.

Table I. Low-temperature pressure film readings for fixture validation

			
$g_0 = 0$ μm	$g_0 = 30$ μm	$g_0 = 50$ μm	$g_0 = 70$ μm
$T_C = 20$°C	$T_C = 100$°C	$T_C = 150$°C	$T_C > 180$°C
$T_B = 180$°C	$T_B = 180$°C	$T_B = 180$°C	$T_B = 180$°C

(< 180°C). An experiment was conducted by varying the initial gap settings of the fixture (g_0) (Paul and Pluess, 2006). The temperature and the pressure were set to 180°C and 4 MPa respectively. Based on theoretical calculations, for an initial gap setting greater than 63 μm, no contact should be established. Results of this experiment are shown in Table I.

It can be seen that pressure was clearly transmitted in the first three film readings, while the last sample does not show any pressure transmission. These findings confirm the ability to control the contact temperature of the fixture. The color density of the first sample compared with reference samples of the hot press at 4 MPa shows an equivalent level of pressure. Furthermore, it can be observed that the pressure was uniformly distributed across the bonding area. The film readings for the gap settings of 30 and 50 μm, however, show lower pressure readings. A possible source of error can be found in the relaxation of the load cell due to the use of high expanding stainless steel bolts, although the additional expansion of the bolts was considered in the amount of pre-load of the load cell. Nevertheless, the physical functionality of the DTE fixture was confirmed with these results and showed good agreement with theoretical fixture settings, even at low temperatures.

At high temperatures, the extent of fin warpage and bond line voids in copper test samples was used as a reference for evaluating the quality of the bonding device. A total of twenty copper (alloy 110) test articles were bonded, each having three 1-inch × 1-inch laminae. Ten test articles were bonded using the DTE fixture and

Figure 7. DTE-bonded sample showing minimal fin warpage.

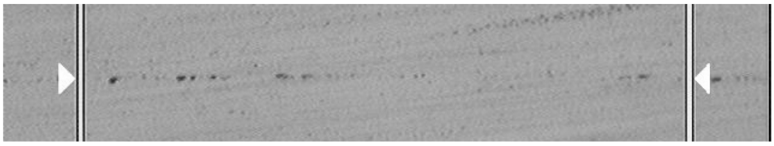


Figure 8. Bond line DTE fixture (800°C/6 MPa/30 min).

ten using a VHP. Bonding conditions chosen were 800°C and 6 MPa for 30 minutes. These conditions were at typical diffusion bonding temperature and pressure conditions for copper. Bonding time was shortened to provide some level of void fraction for comparison. Fin warpage was measured on all twenty test articles by scanning an exposed fin with a Dektak[3] surface profiler. Eight of the twenty test articles were cross-sectioned in two separate areas and analyzed using optical microscopy to evaluate bond lines. Figure 7 shows a low magnification optical micrograph of a cross-sectioned test article produced in the DTE fixture showing minimal fin warpage. Figures 8 and 9 show higher magnification cross sections of test article bond lines bonded in the DTE fixture and the VHP, respectively.

Table II summarizes some of the statistics for the two sets of data. Simple t-tests were used to compare the fin warpage and void fraction means of the VHP- and DTE-bonded samples. The t-tests show that there is significant statistical difference in the fin warpage and void fraction of two sample sets. The tests suggest that there is a 15% chance that the two sets of fin warpage data are from the same population and that there is a 10% chance that the void fraction data are from the same population. It is interesting to note that the DTE fixture yields less warpage but more void fraction than the VHP. Like the pressure film results above, this suggests that under certain conditions the DTE fixture yields lower pressures than expected. While statistically different, the difference in pressure at high temperature to cause these results is estimated to be minor (<0.5 MPa), based on previous bonding results. It is expected that the room temperature pre-loading could be adjusted to provide higher levels of bonding pressure. More important is the indication that the DTE fixture is somewhat repeatable. Additionally, the lower fin warpage may be indicative of the fact that the loading of the bonding pressure in the DTE fixture is more gradual. This could become important with heightened efforts to increase layer-to-layer registration precision and reduce fin warpage. In any event, these results suggest that the administration of bonding pressure below the bonding temperature as in the case of the DTE fixture does not cause additional fin warpage. Additional testing is necessary to validate these claims.

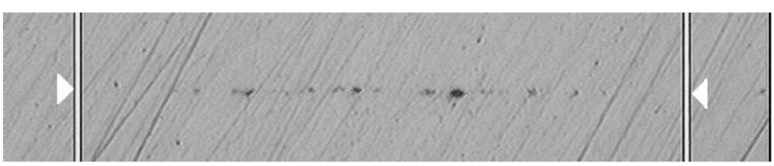


Figure 9. Bond line hot press (800°C/6 MPa/30 min).

Table II. Simple t-test for comparing the effect of VHP and DTE fixture on both fin warpage and void fraction

	VHP	DTE
Fin warpage		
Mean	8.4	6.3
Variance	25.3	3.5
Standard deviation	5.0	1.9
Observations	8	8
Hypothesized mean difference	0	
df	9	
t Stat	1.11	
P(T< = t) one-tail	0.15	
Void fraction		
Mean	15.5	19.5
Variance	13.4	72.1
Standard deviation	3.7	8.5
Observations	10	10
Hypothesized mean difference	0	
df	12	
t Stat	−1.37	
P(T< = t) one-tail	0.10	

Conclusions

A novel diffusion bonding fixture capable of operating within a continuous furnace has been presented. The use of disc springs can significantly reduce the pressure sensitivity of the bonding fixture compared with the prior state-of-the-art as well as provide for the step loading of the pressure magnitude, which is important in the lamination of bulk microchannel devices. The feasibility of the final device concept was theoretically validated by a finite element model, and experimental investigations have proven the viability of the proposed bonding fixture.

The validation of a DTE bonding fixture opens the possibility for high-volume microlamination of MECS devices within a continuous furnace system, which will reduce cycle times and costs associated with diffusion bonding cycles compared with traditional VHP processing. However, it must be realized that before these advantages can be realized, efforts will be needed to better understand increases in fixture costs and inert gas costs. The fixture has the potential to also improve device quality compared with VHP processing. Based on this study it can be concluded that the diffusion bonding of devices within a DTE fixture is possible with less side loading and warpage of lamina fins than with a VHP. This is significant, especially in light of the fact that the DTE fixture has to engage below the bonding temperature of the diffusion bonding cycle, which could lead to more side loading and warpage of laminae. This could be due to the more gradual loading of the laminae associated with the DTE fixture. However, these potential improvements in device quality must be contrasted with the effects of operating in an inert gas environment.

The demonstrated DTE fixture has several other major advantages over existing DTE bonding fixture designs in the state-of-the-art. One major advantage of this fixture is the ability to decouple the bonding pressure magnitude from the process temperature through the concept of pre-loading and force storage within a load cell. Initial gap settings are set independent of the pressure magnitude, and, therefore, any level of bonding pressure can be applied at any level of bonding temperature by design. This flexibility makes the demonstrated DTE bonding device not only interesting for diffusion bonding, but also for other thermal bonding processes. A second major advantage is the ability to control the timing at which the bonding pressure is applied during the temperature cycle, which has been demonstrated in this study. A third major advantage is that the fixture is much less sensitive to fluctuations in bonding temperature.

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