Characterization of Kovar-Pyrex anodically bonded samples – a new packaging approach for MEMS devices

Stephen E. Vargo*[a], A. A. Green[a], J. Mueller[b], D. P. Bame[b] and R. H. Reinicke[c]

[a]Device Research and Applications Section
[b]Thermal and Propulsion Engineering Section
Jet Propulsion Laboratory, California Institute of Technology
4800 Oak Grove Dr., M/S 302-231
Pasadena, CA 91109

[c]Moog Space Products Division
27281 Las Ramblas, No. 207
Mission Viejo, CA 92691 USA

ABSTRACT

The ability to anodically bond Kovar to Pyrex 7740 significantly expands the packaging approaches available for MEMS devices. This technique greatly simplifies and reliably interconnects micropropulsion MEMS components (thrusters, valves) with the attached propellant system. Experimental bonds of Kovar plates and fixtures have been made to numerous Pyrex samples in order to investigate the strength and failure modes of these bonds. An emphasis on experimentally bonding at low temperatures (~ 200 °C) using large voltages (< 2000 V) was also a important parameter of this research and a current microvalve project at JPL. Bond strength measurements have been made using calibrated pull and burst tests with their results being comparable to typical silicon to Pyrex anodic bonds. Detailed bonding conditions for the tested samples have been included in this manuscript to aid the MEMS designer in using this approach to satisfy their packaging needs.

Keywords: anodic bonding, packaging, Pyrex, Kovar

1. INTRODUCTION

The next generation spacecraft for NASA and defense missions will likely be small microspacecraft. These miniature craft, which will typically be in the tens to few tens of kilograms, are generally considered to be simple, non-redundant and highly-focused for specific tasks.[1] Microspacecraft will dramatically lower mission costs by sharply reducing manufacturing, launching and operational costs of today's very large (thousands of kilograms) multi-purpose spacecraft. The development of microspacecraft will greatly depend upon the capabilities enabled by the use of microelectromechanical systems (MEMS) fabrication techniques and components which can meet stringent power, size and integration requirements. Small thrusters[2] (attitude-control, orbit raising), valves[3] (proportional, isolation) and instruments (gyroscopes, star trackers) are some of the components that are in various stages of MEMS development.

There is a definite increase in the number of MEMS devices that require internal pressurization. Examples of such components can be found in the areas of micropropulsion, including thruster and valve components required for microspacecraft, as well as in other microfluidic areas, such as miniature *in situ* diagnostic devices that perform gaseous analysis. Both of these areas are of increasing interest to NASA due to the wide range of future interplanetary missions currently being studied. In most cases, the MEMS structures are being fabricated by bonding chips together to form the internal cavities required to manage the fluid flow. The strength of these bonds, especially when subjected to internal pressurization, thermal cycling, and vibration, is of crucial importance in the design of these components. Bond strengths will determine maximum pressure handling capability, or, conversely, the minimum bond width required at a given pressure level, thus potentially dramatically affecting the degree of miniaturization achievable for such a device. In particular, for

* Correspondence: Email: Stephen.E.Vargo@jpl.nasa.gov; Telephone: (818) 354-2824; Fax: (818) 393-4540

micropropulsion components, maximization of internal pressures is of importance as it will increase allowable propellant tank pressures (reducing tank volume and overall spacecraft size) and thruster performances (thrust and specific impulse).

The aim of this research is to investigate a new packaging approach using a unique material system (Pyrex 7740 to Kovar) which can enable and enhance current packaging techniques for MEMS devices. Kovar is a Fe-Ni-Co alloy (53% Fe, 29% Ni, 17% Co, 1% trace by weight) that has a thermal expansion coefficient of about 5 ppm/°C in the range of 25-400 °C. The anodic bonding of a machined and polished Kovar part on the exposed Pyrex face of a Pyrex-silicon bonded chip makes direct, non-adhesive packaging possible. Anodic bonding, which results in a permanent chemical bond, is typically achieved between silicon and Pyrex substrates in MEMS devices through the application of a large voltage (~800 V) across them at roughly 350 °C. The bonding of Kovar manifolds to devices also greatly reduces the cost and time required for packaging and efficiently interconnects other components. The characterization of bonds made anodically between Kovar and Pyrex have been made using calibrated "pull" and burst tests. These results will lead to improved reliability for the packaging of micropropulsion and other MEMS devices.

2. EXPERIMENTAL DETAILS

In order to determine the feasibility and strength of anodic bonds made between Pyrex 7740 and Kovar several bonded samples and bond strength testing schemes were utilized. Anodic bonding is a method of electrostatically bonding two dissimilar materials to form a strong, hermetic seal that involves little alteration in the shape, size and dimensions of the materials forming the joint.[4] This method, which was first described in 1969 by Wallis and Pomerantz,[5-7] is a commonly used method for joining glass to silicon for MEMS applications. A typical anodic bonding system is shown schematically in Fig. 1.[8] Bonding is accomplished between a conductive substrate and a sodium-rich glass substrate. In this project the conductive substrates are the Kovar pieces and the glass substrates are the Pyrex pieces.[9] The application of a voltage potential across the parts for a few minutes, with the Pyrex held at a negative potential, causes mobile positive ions (mostly Na^+) in the Pyrex to migrate away from the Kovar-Pyrex interface towards the cathode, leaving behind fixed negative charges.[8] The electrostatic attraction between the positive charges in the Kovar and negative charges in the Pyrex holds the two materials together and facilitates the chemical bonding of Kovar to Pyrex. Only one Kovar and Pyrex piece are bonded at a time to ensure consistent experimental conditions. Compared to silicon fusion bonding or thermocompression bonding, Madou notes that anodic bonding has the advantage of being a relatively low temperature process with a lower residual stress and less stringent requirements for the surface quality of the part faces.[10] Low residual stress levels are derived from the bonding of materials that have similar thermal expansion values over the operating temperature range (i.e. Kovar and Pyrex 7740, or silicon and Pyrex 7740). In the temperature range of 25-400 °C Kovar (5 ppm/°C) and Pyrex (3.25 ppm/°C) have good thermal expansion matches (see Fig. 2). Large differences in the thermal expansion values of two materials at bonding temperatures will cause unacceptable levels of stress during the sample's cooling process. These stresses can induce premature failure of the bond, thus materials with similar thermal expansion values are desired for anodic bonding.

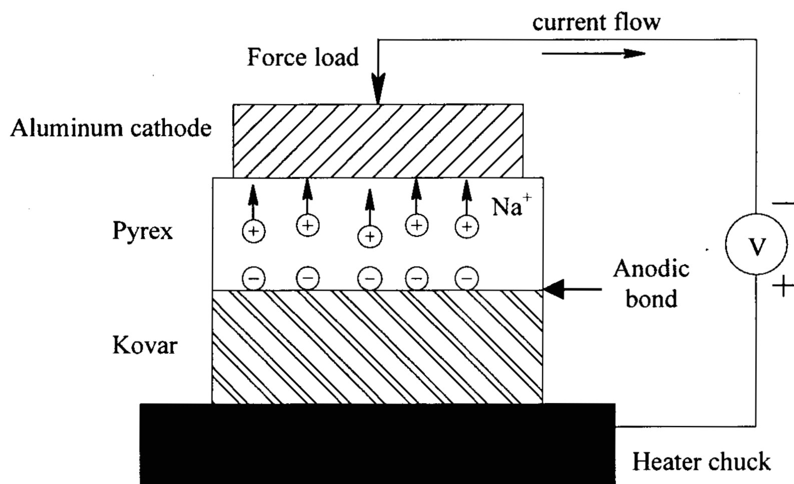

Figure 1. Schematic of the anodic bonding apparatus.[8]

An Electronic Visions AB1-PVS anodic bonding system available in the Microdevices Laboratory at JPL is used to perform the anodic bonding. This bonder is normally capable of providing 0-1200 V, 0-50 mA, 20-500 °C, 0-50 N and high-vacuum (pressures < $1x10^{-5}$ Torr) conditions for anodic bonding. An external Spellman high voltage DC power supply with 0-20 mA and 0-5000 V output has been added to the AB1-PVS to provide bonding voltages up to 2000 V (bonder head has a maximum voltage rating of 2000 V). The samples reported here were all performed under vacuum (system pressures < $5x10^{-5}$ Torr), which prevents oxidation of the Kovar when heated, heater temperatures of > 210 °C, voltages ≥ 1200 V and force loading ≥ 20 N. Sze found that the application of a temperature in the range of 350-450 °C is sufficient to make the sodium ions in the glass mobile under typical applied voltages (< 1200 V) for anodic bonding of silicon to Pyrex.[11] However, at temperatures greater than about 400 °C the thermal expansion values between Kovar and Pyrex become quite large (see Fig. 2). In order to avoid this issue bonding for the Kovar-Pyrex samples was performed at temperatures below 400 °C. As shown and discussed by Sim the consequence of performing lower temperature (< 400 °C) bonds is that the applied voltage to the sample must exceed 1200 V.[12] This results since at lower Pyrex temperatures an increase in applied potential is necessary to compensate for the lower Pyrex ion mobility. Another goal for this project was the determination of the lower temperature limit that successful anodic bonding could be attained between Kovar and Pyrex. Successful low temperature (~ 250 °C) anodic bonding enables the use of lower melting point materials (e.g. elastomers and plastics) in MEMS devices that need packaging. Moog, Inc. and JPL are especially interested in this aspect so that soft seat materials can be utilized in a microvalve project.

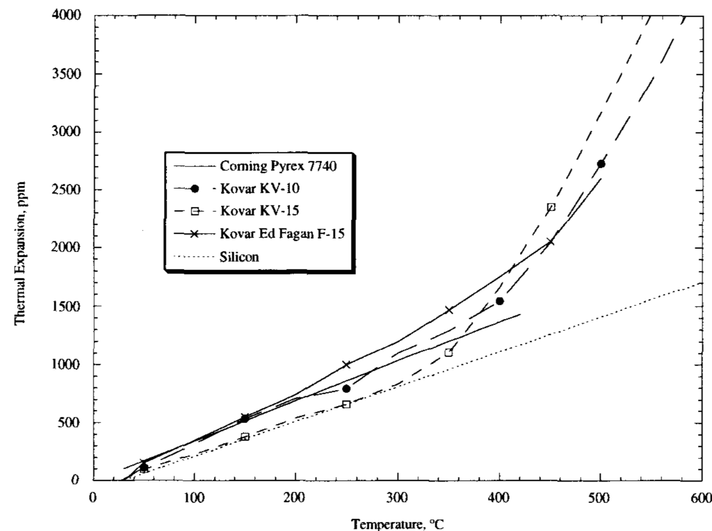

Figure 2. Thermal expansion of Kovar, Pyrex and silicon as a function of temperature (adapted from manufacturer's data).

2.1 Pull Tests

The calibrated pull test is a simple and convenient method for evaluating the strength of bonds between two materials. This test is generally performed by securing attachment fixtures to the exposed material faces (see Fig. 3) using a strong adhesive and applying a pulling load away from the bonding interface (i.e. direction perpendicular to the material faces). The adhesive used was Torr Seal, which is a two-part epoxy, that can provide a bonding (tensile) strength of 5000 psi. Pull tests using an Instron 1331 servo-hydraulic test machine have been performed on Kovar and Pyrex anodically bonded samples. This machine measures both load and deflection of the samples during testing and can provide a full load capacity of about 90,000 N tension or compression with adjustable resolution. The samples, which are designated as flat-wise tensile samples, are adhesively bonded to attachment fixtures to maintain flatness during the entire test. The Kovar material, which was purchased from Ed Fagan Inc. (Ed Fagan F-15), has a thickness, width and length of 1.02 mm, 50.8 mm and 50.8 mm, respectively. One face of the Kovar was highly polished (average surface roughness < 50 Å) in order to provide an ideal

bonding surface for the Pyrex. The Pyrex pieces (average surface roughness < 30 Å) are all 550 μm thick and have a size of 25.4 mm square or 75 mm diameter. The load applied to each sample was increased continuously until bond failure.

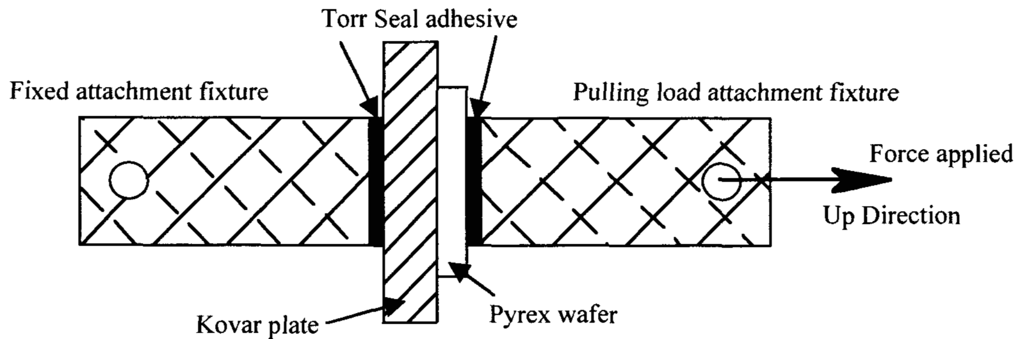

Figure 3. Pull test configuration during the Instron 1331 bond strength measurements.

2.2 Burst Tests

The use of propellant stored at high pressures (> 500 psi) is a requirement for most micropropulsion systems currently under study and development. Bonded elements in the system have to be able to withstand pressurized environments without failure. The burst test involves pressurizing a cavity directly beneath the bonded Pyrex sample (see Fig. 4) with gas in order to simulate this condition. A Kovar test fixture was machined in order to resemble a worst-case MEMS device internal configuration. The Kovar material was purchased from Sumitomo Special Metals America, Inc. (KV-15) and was chosen instead of Ed Fagan material in order to provide lower thermal expansion values. The Sumitomo KV-10 material used by Sim, which is no longer commercially available, is shown in Fig. 2 for comparison. Two thicknesses of Pyrex, which have a size of 15.2 mm square, have been bonded to fixtures under different conditions in order to investigate the bond strength of the test samples. Once bonded the fixtures were pressurized with either N_2 or He until a failure occurred with the sample. Helium was used in some tests in order to get qualitative leak indications using a helium leak detector while the fixture was pressurized at 150 psi (no leaks were noted during these tests).

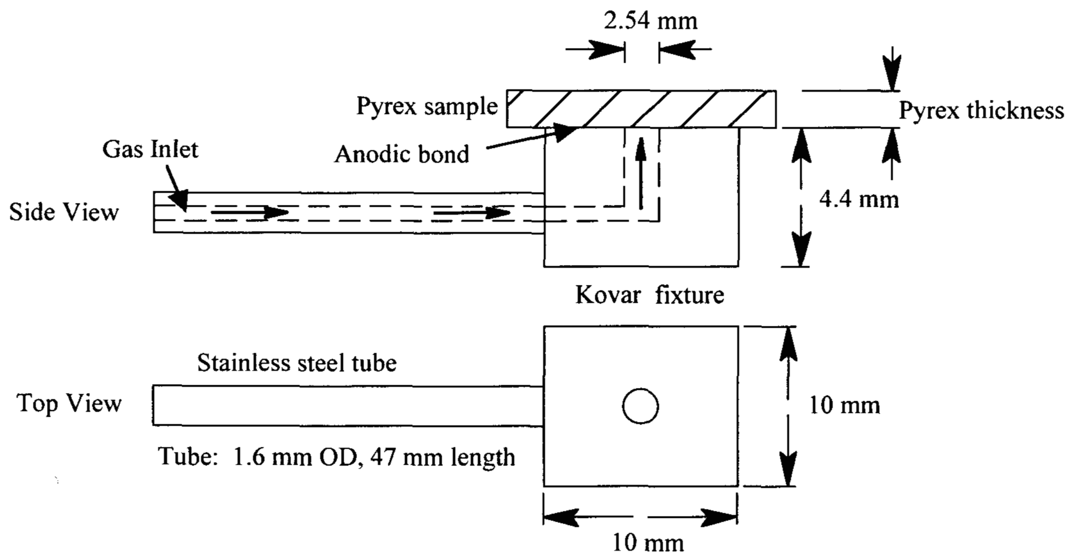

Figure 4. Dimensions and layout of the Kovar burst test fixture.

3. RESULTS AND DISCUSSION

Experimental estimates of bond strengths between Kovar and Pyrex anodically bonded samples have been obtained using the calibrated pull and burst tests outlined earlier. The pull tests were performed on nine samples under various bonding conditions. Their results are outlined in Table 1. It is worth noting that anodic bonding was achieved for a bond temperature as low as 211 °C. However, the actual bond strength was quite low (179 psi). The failure modes during testing were classified as adhesive, anodic or both. Adhesive failure implies that the Torr Seal did not adhere to the glass or Kovar with no damage occurring to the sample. A limitation of using adhesive as a securing method to these samples was that the adhesive could not make ideal bonding to the very smooth surfaces. More surface roughness on the exposed sample faces would have increased the adhesive bonding strength. However, "roughening" the Pyrex surface would have introduced weaknesses in the glass that may have caused premature failure. Anodic failure describes the result of the entire Pyrex piece being removed intact. The case of both bond failures resulted in damaged Pyrex with many Pyrex areas remaining bonded to the Kovar.

Table 1. Summary of the anodically bonded pull test samples.

Label	Pyrex size, thickness	Bonding Conditions	Pull Strength (psi)	Failure mode
KP-1	25.4 mm x 25.4 mm, 550 µm	- 379 °C, 50 N, 1200 V - 21mA peak, 4.5 min bond	1486	Adhesive
KP-2	25.4 mm x 25.4 mm, 550 µm	- 283 °C, 50 N, 1400 V - current N/A, 10 min bond	1468	Anodic
KP-3	25.4 mm x 25.4 mm, 550 µm	- 283 °C, 50 N, 1400 V - current N/A, 10 min bond	1312	Both
KP-4	25.4 mm x 25.4 mm, 550 µm	- 235 °C, 50 N, 1500 V - current N/A, 11.5 min bond	456	Anodic
KP-5	75 mm diameter, 550 µm	- 235 °C, 50 N, 1500 V - 5 mA peak, 10 min bond	445	Both
KP-6	75 mm diameter, 550 µm	- 225 °C, 50 N, 1800 V - 4.5 mA peak, 11min bond	289	Adhesive
KP-7	75 mm diameter, 550 µm	- 215 °C, 50 N, 1800 V - 3.2 mA peak, 10 min bond	533	Anodic
KP-8	75 mm diameter, 550 µm	- 211 °C, 50 N, 1900 V - 2.5 mA peak, 11 min bond	179	Anodic
KP-9	75 mm diameter, 550 µm	- 379 °C, 50 N, 1200 V - >20 mA peak, 4 min bond	634	Both

The results of the pull tests are also shown in Fig. 5 for reference. Note that as the bonding temperature decreases the anodic bonding voltage has to increase in order to perform bonding due to lower mobility of the Pyrex ions. Trends based on the pull strength measurements are difficult to determine since the sample size at each bond condition is small. For example, the KP-1 and KP-9 bond conditions are nearly identical but the KP-1 pull strength was more than twice that of the KP-9 sample. The failure modes were also different and the KP-1 bond strength is likely to be higher than measured since the adhesive failed. In general, bonds made at about 250 °C and 1600 V will result in sufficient bond strengths for most MEMS devices.

Burst tests have been performed to simulate conditions that pressurized micropropulsion components will experience during operation. A total of eight Kovar burst fixtures (see Fig. 4) were individually bonded with Pyrex at various bonding conditions. The bonding conditions and experimental results for the burst tests are outlined in Table 2. The sample size is also small for these tests but acceptable bond strengths for MEMS devices can be inferred. The bonding voltage does not seem to make much difference in the bond strength in this testing method. All pressurized fixtures failed due to the Pyrex breaking in the unsupported area of the fixture and these failures tended to have a slight dependence on the time spent at a given pressure. The KB-6 and KB-8 tests failed at their maximum pressures after waiting at this pressure for 30 sec. Figures 6 and 7 show the results of burst tests with the center of the Pyrex piece having a large section melted and blown out during the instant of failure. As might be expected the thinner Pyrex (525 µm) broke at a lower pressure than did the thicker (1.5 mm) material. Actual measurements of the anodic bonding strength of these bonded samples are difficult to obtain using this

method but they provide indications of bond strengths in excess of 1000 psi. Smaller openings in the test fixture would lead to more realistic measures of bond strength between the Pyrex and Kovar but were not available at this time. Future work with smaller fixture openings are needed to obtain better anodic bonding strength values. The tubing plugged condition of KB-3 and KB-5 were directly attributed to the brazing process that secures the tubing to the fixture. Although burst measurements could not be made with these fixtures they were used to perform anodic bond repeatability checks. The KB-7 sample was a previously anodic bonded Pyrex and silicon sample that was subsequently anodically bonded to the Kovar test fixture. It is worth noting that this sample also had a fairly high burst pressure. The silicon backing for this sample considerably strengthens the attached Pyrex preventing a failure at lower pressures.

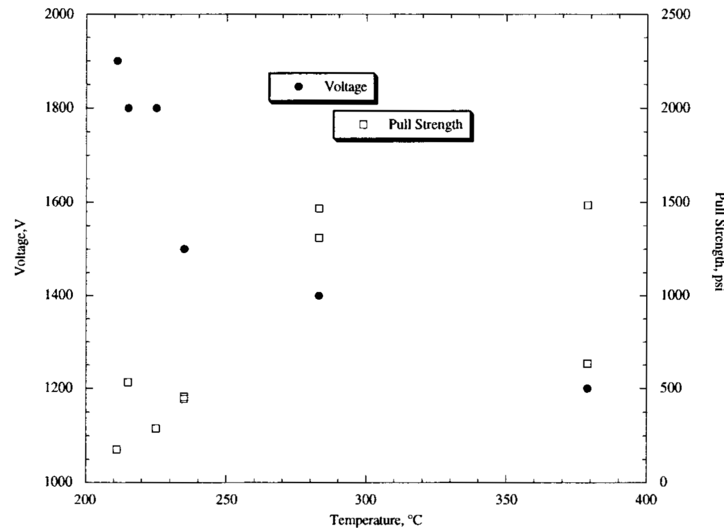

Figure 5. Kovar to Pyrex bonding results for the pull tests.

Table 2. Summary of the anodically bonded burst test samples.

Label	Pyrex thickness (μm)	Bonding Conditions	Failure mode	Burst Pressure (psi)
KB-1	1500	- 476 °C, 50 N, 1200 V - 0.32 mA peak, 4.5 min bond	Pyrex broke	870
KB-2	525	- 354 °C, 20 N, 1600 V - 3 mA peak, 5 min bond	Pyrex broke	460
KB-3	1500	- 325 °C, 20 N, 1700 V - <1 mA peak, 6 min bond	No test	Tubing plugged
KB-4	525	- 331 °C, 20 N, 1700 V - <1 mA peak, 5 min bond	Pyrex broke	525
KB-5	1500	- 341 °C, 20 N, 1800 V - <1 mA peak, 6 min bond	No test	Tubing plugged
KB-6	1500	- 341 °C, 20 N, 1800 V - <1 mA peak, 6 min bond	Pyrex broke	900
KB-7	525 Pyrex, 550 silicon	- 426 °C, 20 N, 1200 V - 3 mA peak, 1 min bond	Pyrex broke	900
KB-8	1500	- 456 °C, 20 N, 1600 V - 3 mA peak, 2 min bond	Pyrex broke	700

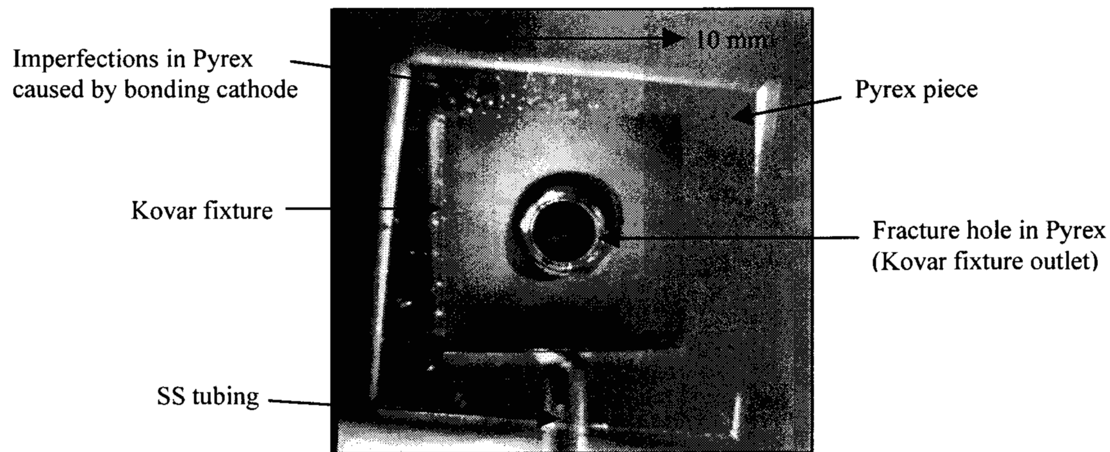

Figure 6. Typical Pyrex failure mode during the burst tests.

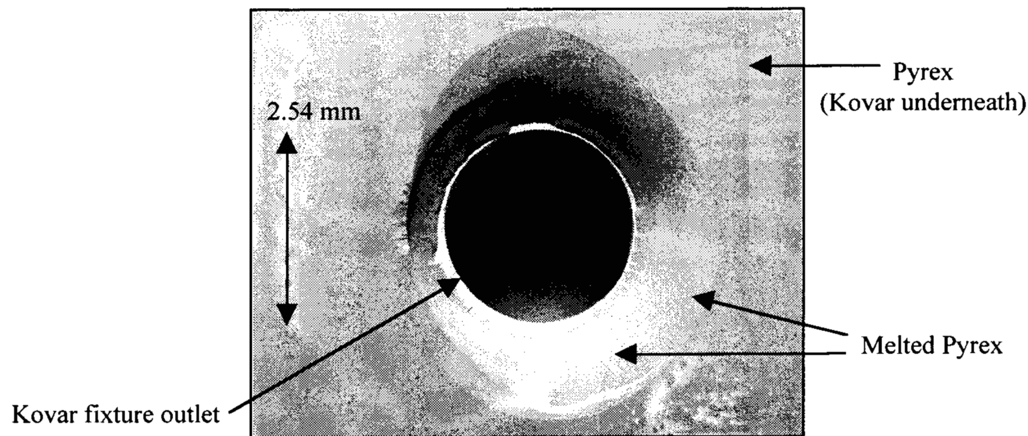

Figure 7. Close-up view of the Kovar fixture outlet on a burst tested Pyrex sample.

4. CONCLUSIONS

Anodic bonding of Pyrex to Kovar affords the MEMS designer the opportunity to package their devices in a convenient and hermetic manner. Stainless steel tubing that is brazed to a machined and polished Kovar part provides a connection between the pressurized system and the MEMS device. The future of micropropulsion systems will most likely depend on techniques that enable very dense integration, miniaturization and reliable methods for packaging their systems. The discussed method of bonding metals to fabricated MEMS chips is one promising approach to satisfying these requirements. Anodic bonding of Kovar to Pyrex has been demonstrated at temperatures as low as 210 °C using 1900 V. Stronger bond strengths were obtained using higher bonding temperatures (< 400 °C) but sufficient bond strengths for current MEMS devices are anticipated using 250 °C and 1600 V bonding conditions. More experimental testing will further strengthen the approach that has been detailed in this manuscript.

ACKNOWLEDGEMENTS

The research described in this paper was carried out by the Jet Propulsion Laboratory, California Institute of Technology, under a contract with the National Aeronautics and Space Administration. Portions of this work are funded by a grant from

the JPL Director's Discretionary Research Fund (DRDF) and Moog, Inc. internal research funds and support is gratefully acknowledged.

Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not constitute or imply its endorsement by the United States Government or the Jet Propulsion Laboratory, California Institute of Technology.

REFERENCES

1. A. D. Ketsdever and J. Mueller, "Systems considerations and design options for microspacecraft propulsion systems," 35th Joint Propulsion Conference, AIAA-99-2723, Los Angeles, 1999.
2. J. Mueller, "Thruster options for microspacecraft: a review and evaluation of existing hardware and emerging technologies," 33rd Joint Propulsion Conference, AIAA-97-3058, Seattle, 1997.
3. J. Mueller, "A review and applicability assessment of MEMS-based microvalve technologies for microspacecraft propulsion," 35th Joint Propulsion Conference, AIAA-99-2725, Los Angeles, 1999.
4. A. Hanneborg, M. Nese and P. Øhlckers, "Silicon-to-silicon anodic bonding with a borosilicate glass layer," *J. Micromech. Microeng.*, **1**, pp. 139-144, 1991.
5. G. Wallis, "Direct-current polarization during field-assisted glass-metal sealing," *J. American Ceramic Society*, **53**, pp. 563-567, 1969.
6. G. Wallis and D. I. Pomerantz, "Field assisted glass-metal sealing," *J. Applied Physics*, **40**, pp. 3946-3949 1969.
7. G. Wallis, "Field assisted glass sealing," *Electrocomponent Science and Technology*, **2**, pp. 45-53, 1975.
8. N. Maluf, *An Introduction to Microelectromechanical Systems Engineering*, Artech House, Inc., Boston, 2000.
9. G. Wallis, J. Dorsey and J. Beckett, "Field assisted seals of glass to Fe-Ni-Co alloy," *Ceramic Bulletin*, **50**, pp. 958-961, 1971.
10. M. Madou, *Fundamentals of Microfabrication*, CRC Press, New York, 1997.
11. S. M. Sze, *Semiconductor Sensors*, John Wiley & Sons, Inc., New York, 1994.
12. D. Y. Sim, T. Kurabayashi and M. Esashi, "A bakable microvalve with a Kovar-glass-silicon-glass structure," *J. Micromech. Microeng.*, **6**, pp. 266-271, 1996.