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**CRADA Final Report
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Micromechanical Structures Fabrication

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ABSTRACT

Work in materials other than silicon for MEMS applications has typically been restricted to metals and metal oxides instead of more “exotic” semiconductors. However, group III-V and II-VI semiconductors form a very important and versatile collection of material and electronic parameters available to the MEMS and MOEMS designer. With these materials, not only are the traditional mechanical material variables (thermal conductivity, thermal expansion, Young’s modulus, etc.) available, but also chemical constituents can be varied in ternary and quaternary materials. This flexibility can be extremely important for both friction and chemical compatibility issues for MEMS. In addition, the ability to continually vary the bandgap energy can be particularly useful for many electronics and infrared detection applications. However, there are two major obstacles associated with alternate semiconductor material MEMS. The first issue is the actual fabrication of non-silicon micro-devices and the second impediment is communicating with these novel devices. We have implemented an essentially material independent fabrication method that is amenable to most group III-V and II-VI semiconductors. This technique uses a combination of non-traditional direct write precision fabrication processes such as diamond turning, ion milling, laser ablation, etc. This type of deterministic fabrication approach lends itself to an almost trivial assembly process. We also implemented a mechanical, electrical, and optical self-aligning hybridization technique for these alternate-material MEMS substrates.

STATEMENT OF OBJECTIVES

The objective of this project was to use the unique ORNL collection of resources to fabricate microstructures made out of exotic semiconductor materials. This fabrication method has previously been successful in microstructures fabrication at ORNL for alternate material. These micro-devices are intended to ultimately be employed as sensor platforms responding to various stimuli. To meet this objective, an experimental design was required to be generated. Next the actual micro-fabrication was to be performed using primarily the ORNL tools of Single-Point-Diamond-Turning and Focused-Ion-Beam processing. Finally the results were to be evaluated and transmitted to EEG, Inc.

BENEFIT TO FUNDING DOE OFFICE

This work was not funded by DOE. The ORNL effort was funded entirely by the Industrial Partner. The Industrial Partner’s funds were not from the Department of Energy. However this type of novel “leap frog” sensor material technology development would obviously be beneficial to various DOE offices and missions. This work has laid the foundation for increased utility in MEMS based chemical and physical sensors technology and may even make completely new approaches to sensing feasible.

TECHNICAL DISCUSSION

Alternate Material Micro-Machining

The traditional IC micro-fabrication processes typically involves substantial wet chemistry to create microstructures of interest. This type of process is not well understood for many alternate materials and the microstructures often collapse due to liquid surface tension effects. Even if dry processing, such as reactive ion etching, is extensively employed the fabrication will be highly material dependent. Thus assuming it is chemically possible, new process parameters would be required for every new material system of interest.

Therefore we have chosen to employ in the execution of this CRADA an essentially material independent and dry fabrication that has been recently developed at ORNL. In the fabrication of micro-devices there are basically two options, precision material deposition and/or removal. We have employed multiple tools for both approaches that have been used in novel ways to directly fabricate micro-devices from material substrates of interest. The precision fabrication tools that have previously used include: single point diamond turning (SPDT); focused ion beam (FIB) milling; laser ablation; focused ion beam chemical deposition; focused ion beam assisted chemical etching; direct write lithography; and molecular beam epitaxy. Since the goal in our fabrication scheme was to maximize material independence of the process, it was important in the material removal mode of this approach to remove the maximum amount of material before proceeding to the next more material dependent fabrication step, if required. Due to the ultimate micro-device sizes needed for this project, and since this was a limited scope effort, two primary tools were employed in this work; SPDT and FIB. Unlike conventional etching processes, precision direct write techniques can be used to rapidly and precisely remove material without many of the problems associated with traditional photolithography-based etching processes used to create silicon features. Even though there is some inherent material dependence associated with almost every process, these effects were negated since more than 99% of the material removal was accomplished with precision SPDT as shown in Figure 1. Furthermore, eliminating or substantially reducing dependence on etchants will substantially decrease the time consuming step of matching a specific etching agent to the target binary, ternary, or quaternary material. From Table 1, the design flexibility of employing material other than silicon becomes clear. The available micro-device design variables often justify the cost of alternate materials.

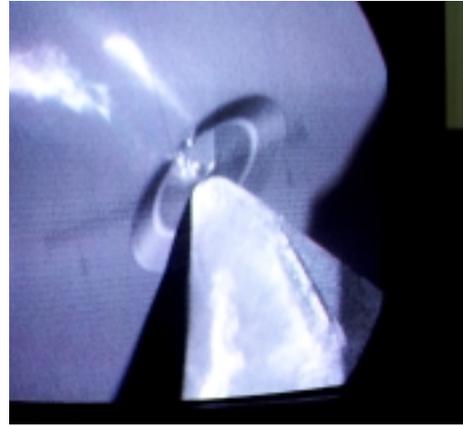


Figure 1. 2 mm diameter, < 10 μm thickness conical semiconductor depression (diaphragm) during single point diamond turning.

Table 1. Potential MEMS Design Variables For Materials Investigated

MEMS Material Studied	Material Parameters						
	Hardness (Knoop #) (Kg/mm ²)	Refractive Index @ $\lambda = 5\mu\text{m}$	Young's Modulus (GPa)	Specific Gravity (g/cm ³)	Thermal Conductivity (W/mK)	Thermal Expansion (10 ⁻⁶ K ⁻¹)	Bandgap Energy (eV)
Si	1100	3.43	130.91	2.33	163	2.6	1.1
Ge	800	4.02	102.66	5.35	59	6.1	0.67
InSb	225	3.29	42.79	5.78	36	5.0	0.17
GaSb	469	3.82	63.32	5.62	44	6.9	0.72
InP	430	3.03			70	4.5	1.35
GaAs	721	3.30	82.68	5.32	55	5.7	1.4
InAs	330	3.46			50	5.3	0.36
HgCdTe	Compositionally dependant values						

ORNL has a premier diamond turning research and development facility, allowing us to explore many exotic semiconductor material systems. The diamond turning research facility at ORNL was initially established to produce challenging high performance optical surfaces such as paraboloids, ellipsoids, conformal optics, and diffractive/refractive hybrid designs¹. These type of precision optical components are typically

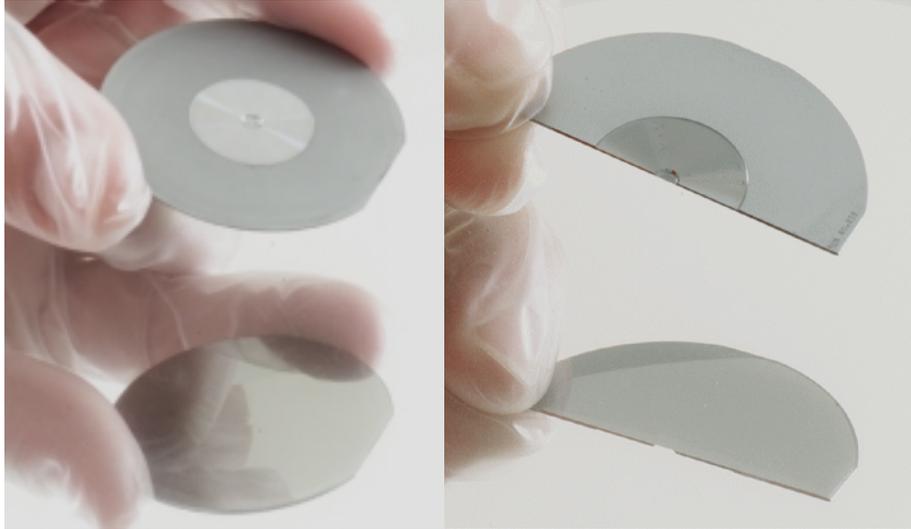


Figure 2. SPDT produced InP and cleaved InAs wafers (2mm diameter and <math><10\mu\text{m}</math> thick diaphragms with a surrounding 20 mm SPDT area before machining of the release ring)

held to tolerances on the order of a micrometer deviation from the perfect design surface across a 100 mm (or more) diameter part. Thus using this equipment for the sub-micrometer precision required in the micro-fabrication and hybridization efforts for this project was not insurmountable since the diameters involved are only a few millimeters. Two diamond turning machines were available for this project; the prototype of the Nanoform 600 class machine, that can diamond turn parts with diameters of 0.6 m with a positioning resolution of 1.25 nm; and the Precitech Optimum 2800 that was particularly amenable to semiconductor micro-machining.

The micro-machining conditions used for all materials for the final surface passes were: rotational speed of 2000 rpm; feed rate of 500 nm/min.; cut depth of 0.5 μm ; and diamond tool radius of 0.508 mm. However, the roughing cut parameters varied according to the individual material properties. Examples of micro-machined alternate material diaphragms are the InP and cleaved InAs wafers shown in Figure 2. Clearly visible in Figure 3 is the release ring that was SPDT around the diaphragm depression to a thickness of less than 30 μm to allow subsequent separation from the wafer. The quality of the micro-machined surfaces was critical since these thin diaphragm surfaces serve as the substrates for the eventual micro-devices. All of the non-silicon starting substrate wafers were polished on the vacuum fixture side and lapped on the diamond tool side. Thus the final micro-devices have a polished side and a diamond turned side.

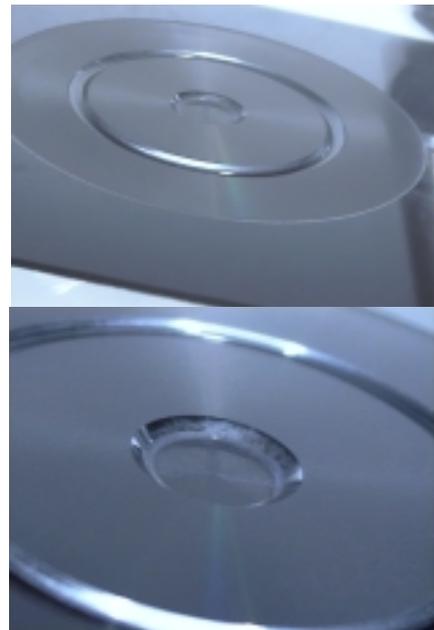


Figure 3. Alternate material release ring (10 mm dia.) and diaphragm (2 mm dia.)

An atomic force microscope image of a silicon micro-machined surface is depicted in Figure 4. Although at first glance this silicon surface appears highly textured, the scale indicates the extreme exaggeration of the vertical (z-axis) dimension in this image. The peak to valley deviation is approximately 5 nm. Two distinct periods can be seen in Figure 4. The larger period of approximately 300 μm

corresponds to the expected values given the feed rate, rotational speed, and depth-of-cut. However, the other pattern has a higher frequency component induced by vibration. Fortunately, most of the important group II-VI and III-V semiconductors are more amenable, to varying degrees, than silicon to this fabrication process. The antimony based materials (InSb and GaSb) were the most ductile and showed the least sensitivity to the fabrication parameters, with properties similar to aluminum. HgCdTe was also readily processed, however the wafer consisted of a 20 μm thick liquid phase epitaxy grown HgCdTe layer on a CdZnTe substrate. The wafer was vacuum mounted on the HgCdTe side and SPDT machined through the substrate into the epitaxial layer to form a diaphragm less than 10 μm thick. The harder arsenic based materials (InAs and GaAs), along with the group IV materials (Si and Ge), required the use of more gentle machining conditions for the “rough” cuts. The final finishing passes for all of the materials investigated were identical, as described above. InP showed intermediate machining properties (ductile/brittle) compared to the other materials investigated. Only the extremely hard materials, such as SiC, were specifically excluded from this study.

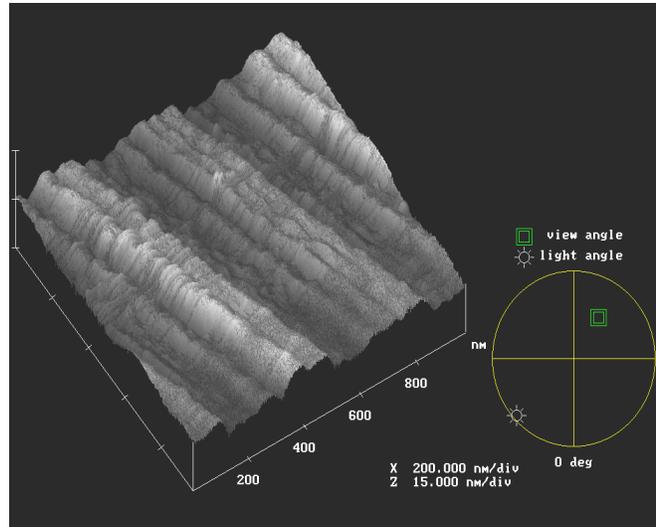


Figure 4. AFM image of a silicon diamond turned surface, 5nm PV

Micro-device Implementation

Also available for our studies was a FIB milling system (FEI 200). The system uses a Ga ion source to produce an energetic ion beam focused onto the working surface^{2,3,4}. The beam energy was 30 keV and the beam current varied (1 pA to 10.5 nA), depending on the focused beam diameter (25 nm to 600 nm). The system utilizes secondary electron emission produced by the scanning ion beam to image the surface in a manner similar to an electron microscope. This allows the region of the surface being modified to be observed before and during the ion milling process. Magnifications achievable are similar to electron microscopes, ranging from 190 to over 100,000. The system is capable of direct write ion beam milling or deposition by rastering over a user-defined pattern superimposed on an image of the surface^{5,6}. ORNL has an extensive background

developing ion beam milling processes for ultra-precision fabrication applied to a variety of non-semiconductor materials⁷. Roughening of metals and non-metals under ion bombardment is an issue of critical importance for applications involving micro-structures and has been extensively studied in our previous



Figure 5. FIB milling of InSb diaphragm produced this simple micro-device used for subsequent IR response testing.

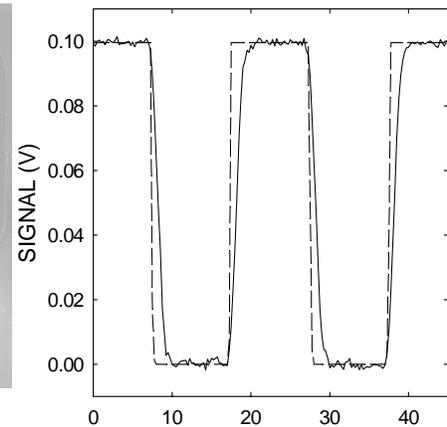


Figure 6. IR deflection response of bulk InSb micro-device

investigations⁸.

The work described in report concentrated on semiconductor single crystal materials that are expected to make the largest impact in micro-devices

applications from alternate (non-silicon) substrates.

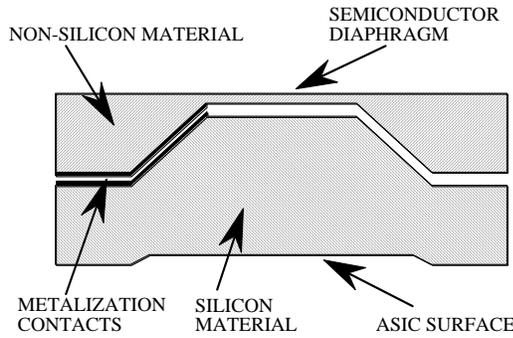


Figure 7. Rapid hybridization (mechanical and electrical) with a matching inverse silicon mate to complete the MEMS.

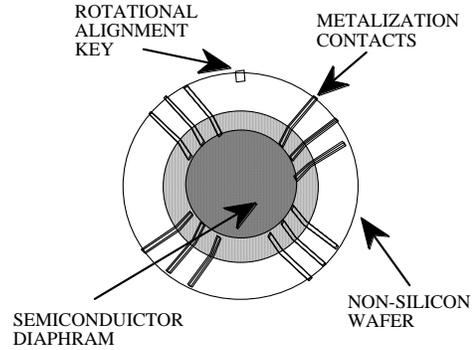


Figure 8. Schematic of diamond turned conical diaphragm with metal contacts in an exotic semiconductor material.

The alternate material diaphragms discussed in the previous sections were the starting substrates for the FIB process. These diaphragms were often further thinned with broad beam ion milling before being directly patterned with FIB. This thinning process was often hampered due to preferential sputtering of one of the constituent elements in the binary material. InSb exhibited the most severe preferential sputtering while GaAs was the most amenable. The group IV single element semiconductors showed essentially no preferential effects. This problem was ultimately overcome with the introduction of non-normal sputtering angles and rotation during sputtering. This eliminated preferential sputtering and produced extremely smooth surfaces.

An example of a simple patterned and released micro-device is shown in Figure 5. This 500 μm long micro-mechanical structure was patterned in the middle of a 6 μm thick InSb diaphragm using FIB. The missing material simply dropped down due to gravity once the pattern was complete, leaving the released micro-device. The deflection response of this micro-device due to exposure to energy from a 500 $^{\circ}\text{C}$ blackbody source ($\lambda > 1.1 \mu\text{m}$) is shown in Figure 6. The experimental arrangement for this deflection measurement has been adopted from atomic force microscopy (AFM) as reported previously⁹. This bulk InSb micro-mechanical IR detector has exhibited superior deflection response (magnitude and frequency) compared to simple thermo-mechanical (bi-material) movement due to photo-induced, rather than thermal, stress in the lattice¹⁰.

Mems Hybridization

In addition to the MEMS / MOEMS fabrication obstacles associated with the use of materials other than silicon, we also addressed the difficult issue of hybridization. The labor intensive, costly, and inefficient (even when automated) process of joining wafers mechanically, electrically and even optically can be substantially simplified. Hybridization is necessary when a material other than silicon is required for a given application. Traditionally an extremely complex method of linking exotic material substrates to silicon processing circuitry had to be reinvented for each specific

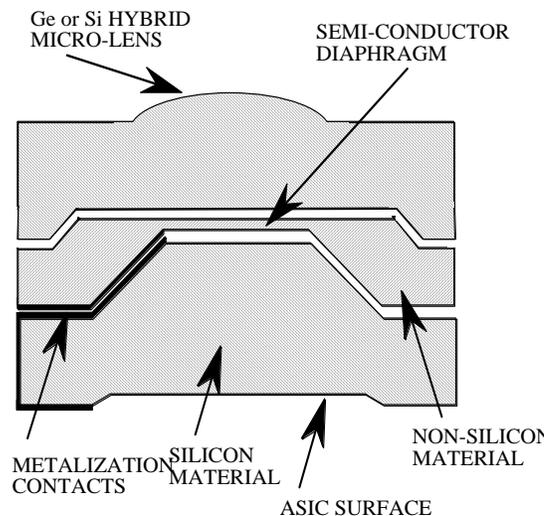


Figure 9. Another possible level of hybridization can be achieved with a micro-lens attached to complete the MOEMS.

application. Instead, hybridization can be addressed by ultra-precision SPDT in a way similar to that used in self-aligning optical systems and is shown in Figures 7 and 8¹¹. The rapid and precise material removal required for this effort was also performed with the sub-micrometer tolerance SPDT machine. When a micro-device was fabricated from an exotic material within a diaphragm (conic depression shown earlier in Figure 3), an exact negative (conic projection) can also be produced in a silicon wafer. When radial metalization contacts are deposited on the micro-device from the diaphragm to the outer edge of the

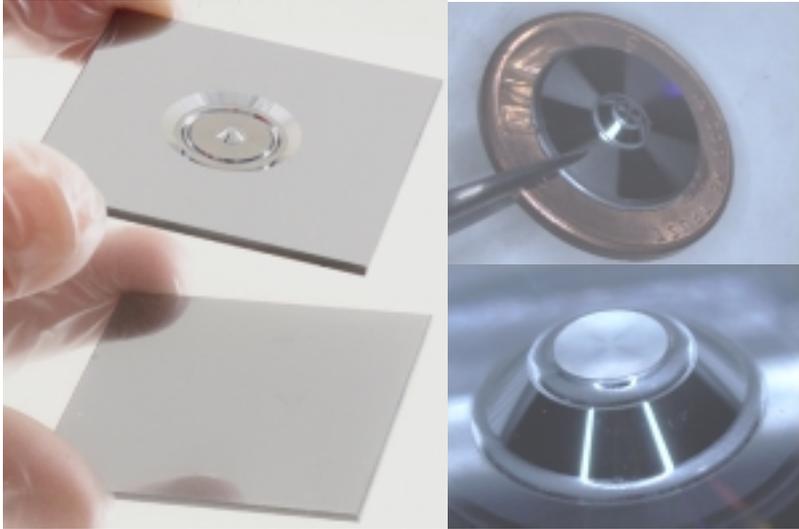


Figure 10. Three views of silicon interface; as machined 50 x 50 mm wafer; released 10 mm diameter substrate; and conic 2 mm diameter projection.

wafer, and onto the corresponding mating surface of the inverse silicon projected cone, there exists a simple means of electrically connecting the two dissimilar wafers. Although not shown in the images in the above figures, the silicon mating wafer can have metalization lines wrapping around the wafer to connect to conventional silicon-based micro-circuitry on the back side of that wafer. Additionally, a simple locating mark was embossed on the wafers to facilitate clocking (rotation) which is the only remaining degree of freedom in such an alignment scheme. If an optical interface is required for a particular micro-device, an additional wafer can be aligned in a similar manner to the other side of the exotic material (Figure 9). This additional wafer can contain a high performance hybrid lensed surface, typically made from Si, Ge, ZnS, or ZnSe, to allow operation in the infrared. The present state-of-the-art in SPDT equipment readily allows substantially sub-micrometer material removal increments forming the basis for this novel hybridization process.

The silicon mating/interface structure required to hybridize the alternate material MEMS is depicted in three images in Figure 10. The image on the left shows a SPDT silicon wafer. The top image on the right shows a subsequently released substrate compared in size to a coin. The final image in Figure 10 shows the detail features of the conic interface projection. The top edge of the cone was relieved to accommodate almost any radius diamond tool that might be used to fabricate the inverse alternate material depression without interference. Thus the 100 μm design gap between the top of the silicon cone and the bottom of the alternate material diaphragm can be maintained by forcing the two mating interfaces to contact only on the 45 degree angled cone surfaces. This eliminates any problem with different coefficients of thermal expansion between the two substrates.

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INVENTIONS

There were no invention disclosure associated with the execution of this CRADA by either party.

COMMERCIALIZATION POSSIBILITIES

There has been an increasing interest in developing MEMS (micro-electro-mechanical systems) and MOEMS (micro-opto-electro-mechanical systems) for a variety of physical and chemical sensing applications. The total monetary investment in MEMS development is reflected by a world market that is well over \$1 billion and may approach \$10 billion in another decade¹². These types of devices are traditionally made using micro-fabrication techniques and processes derived from microelectronics integrated circuit (IC) fabrication. As a result, the material used almost exclusively is silicon since it has been studied and implemented extensively. To date, there is very limited information and research in the area of micro-devices that are made from "exotic" semiconductor materials. In this and previous studies we have explored the following material systems: Ge, InAs, InSb, GaAs, InP, GaSb, and HgCdTe ($\lambda_c = 11 \mu\text{m}$). Although silicon has acceptable mechanical, thermal, and electrical properties for many applications, the next generation of advanced MEMS and MOEMS will greatly benefit from the flexibility of using different material systems. Some of these material dependant design parameters are shown in Table 1 for the materials we investigated. Techniques and processes were needed that are material independent for the fabrication of these micro-devices. In this way future devices will not be limited to any single semiconductor material. The use of binary, ternary and quaternary semiconductor materials will open numerous micro-device applications areas. Applications that will be most impacted by a material independent micro-fabrication approach involve micro-devices requiring engineered properties such as optical (absorption cut-off wavelength), mechanical (Young's modulus and friction coefficient), thermal (expansion and conductivity), chemical (reaction and "sticking" coefficient) and thermo/ferro-electric. In addition to the difficulties associated with the fabrication of these non-silicon micro-devices, the problem of hybridization (mechanical and electrical connection to a silicon processor) has also been a major impediment to the development of these advanced

MEMS and MOEMS. Thus simple and inexpensive alignment schemes are required to successfully hybridize mechanical, electrical, and optical functions which will provide the required commercial viability.

CONCLUSIONS

Although SPDT and FIB are traditionally used for other purposes, these technologies were readily applied to the fabrication of micro-structures, and the hybridization that was required when using alternate materials. The term SPDT micro-machining in this case refers to an actual ultra-precision machining process compared to the more conventional usage associated with MEMS fabrication methods. In this development effort multiple semiconductor wafers with starting thicknesses of approximately 0.5 mm were rapidly thinned to less than 10 microns, producing diaphragms over a central region of the wafers approximately 2 mm in diameter. These diaphragms were selectively ion milled using FIB to produce fully released micro-structures within the diaphragm. Although this is not a mature fabrication process, we have demonstrated the potential power of this technique for most material systems. These precision fabricated alternate-material micro-structures can also easily be mated to silicon structures of inverse shape to produce a relatively simple and rapid hybridization process. These techniques were employed to rapidly demonstrate substrates for alternate-material sensors such as InSb based IR detection MEMS. Since the first and most rapid step in this fabrication and hybridization processes involves SPDT, the cost realism of this approach was also examined. Micro-devices that can be fabricated from silicon will always enjoy a substantial cost advantage over the techniques discussed above. However for applications where particular silicon material parameters are not suitable, there is essentially a straightforward and relatively low-cost alternative. There is a continually growing list of such applications. Most prominent of these is IR detection since silicon becomes a window material for wavelengths above 1.1 μm . Small commercial SPDT machines are now available that are capable of working 50mm parts for under \$100K. In recent consultations with machine manufacturers we have outlined simplified semiconductor based machines that would cost even less. Thus, for a \$1M investment, a MEMS manufacturer could have as many as 15-20 machines running continuously. This suggests a possible parallel fabrication approach with arrays of lower cost machines instead of the traditional serial IC fabrication process for non-silicon based micro-device applications.

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