Membrane-delimited multiport high-pressure fluid cells

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Fluid cells have been developed for covering an intermediary pressure domain (3-30 MPa) necessary for studying chemical and biochemical reactions at pressures of interest in industrial synthesis, underground or underwater geochemical processes, underwater life sciences and others. The microfluidic cells developed can function as multi-port micro-reaction chambers allowing *in situ* observation of reaction products using X-rays, electron, photon or various particle beams. A prototype has been developed to include 8 microchannels for feeding/evacuating the fluids, two electrodes for electrochemical or conduction measurements and a possible pressure sensor. The pressure is supposed to be applied from the exterior from a high-pressure fluidic pump, through the capillary connections used for feeding the fluids.

To resist pressure, a combined theoretical and experimental study was conducted using Finite Element Analysis and membrane bulging experiments. Jaccodine and Schlegel [1] provided a formula linking the burst pressure (P_{burst}), membrane thickness h, size L, Young's modulus E, Poisson ratio n and ultimate stress of the material s_{uh} :

$$P_{burst} = 2\sqrt{6}(1-\nu)^{1/2} \frac{h\sigma^{3/2}}{LE^{1/2}}$$
(J-S)

For SiN_x membranes, this gives the lines (purple) shown in the (L,h) domain in Fig.1, supporting the idea that pressure resistance can be increased by making the membranes thicker or smaller laterally. However, both options are not ideal, since membranes must be kept as thin as possible to limit beam attenuation, but also large membranes are preferred for a larger field of observation. Different authors provided statistical results on membrane burst pressures [2-5], some exceeding the J-S predictions and supporting the existence of size effects in very thin membranes. Hereby, we optimized the membrane geometry including a thin doped Si sub-frame to reduce the peak stress values in the pressure-solicited membranes near their edges, which led to burst pressure performances well above the J-S predictions (as marked in Fig.1). Our analysis shows that the sub-frames work, but careful consideration needs to be given to size effects in thin SiN_x membranes manifested under 100 nm thickness. Results of simulations, measurements using a highpressure microfluidic pump and Weibull burst statistics will be presented, along with fabrication and design details (Fig.2).

References

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Fig. 1. Constant burst pressure lines in the membrane size vs thickness space, as provided by the J-S formula (purple lines). Reports of different membrane burst studies from [2-5] are included, along with the point targeted by this study. The inset shows a sketch of the microfluidic pressure cell reported here. The cell is customizable for specific experimental needs, including number of fluid channels, pressure range, intra-cavity electrodes, pressure and temperature sensors, etc.



Figure 2. (a) Assembly of the fluid cell by anodic bonding of two identical dies with a shif to allow th eoverlapping of membranes and exposure of electrical contacts. (b) Assembled fluid cell with glass capillary tubes inserted.