On the Reliability of Thermopneumatic Actuators with Silicon Membranes

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ABSTRACT

The application of thermopneumatic actuators to microvalves and microfluidic processing systems continues to attract research and development interest. Yet, as with most microvalve and microfluidic work, little information has been reported with respect to the reliability of microfluidic and microflow systems. In this work, therefore, we extend our earlier discussions of factors affecting silicon membrane reliability, to encompass particular effects of thermopneumatic actuators on such membranes. Specifically, we report experiments demonstrating the effect of cavitation in thermopneumatic actuators on silicon membranes. These experiments show that the nature of cavitation, in a hermetic, thermopneumatic actuator cavity which includes a silicon membrane, is to initiate fast transient pressure pulses in the cavity. The membrane moves mechanically in response to these pressure pulses. If the magnitude of these pressure pulses causes the membrane stress to exceed the silicon yield strength, then fracture of the membrane can occur. The work concludes with a conceptual approach to the design of thermopneumatic actuators, to ensure this failure mode does not occur.

1. INTRODUCTION

Thermopneumatic actuators have been applied to a relatively wide variety of applications using microfabrication techniques [1-16]. Microvalves, micropumps, and integrated flow distribution and control systems (for either gases or liquids) have been researched, developed and deployed commercially. Most of these devices or systems have employed silicon as the main mechanical material, because of its attractive mechanical properties, especially in terms of yield strength [17], although other structural materials have been employed [7, 9, 10, 12, 16]. On the other hand, silicon is brittle, so that stress in excess of the yield strength leads to a rapidly increased probability of fracture. This phenomenon has been the subject of only a few studies; effects related to surface roughness, mechanical cycling, exposure to high thermal temperatures, and the presence of surface oxides or nitrides, have all been studied [18-25], using a variety of conventional methods, as well as more sophisticated means such as micro-Raman spectroscopy [26].

In our own previous work on the reliability of thermopneumatically-actuated silicon microvalves, we have reported on the reliability of systems utilizing such microvalves [27-30], as well on factors affecting silicon microvalve reliability directly [31]. In this work, however, we report a relatively new reliability phenomenon related to thermopneumatic silicon microactuators. The phenomenon stems from the dynamics of the thermopneumatic fluid used in the actuator, as it changes from a liquid-only phase, to a vapor-liquid. Details of the statics related to this phenomenon have been reported earlier [32-37]. In this work, however, the dynamic nature of the phenomenon is crucial to understanding both its nature, and its effect: in particular, its effect on pushing the stress in silicon membranes associated with such actuators, beyond the yield strength of the silicon.

2. CONCEPTUAL DEVELOPMENT AND SUPPORTING MEASUREMENTS

A reliable, silicon thermopneumatic actuator involves understanding all elements of the following chain of reasoning:

- Fundamental material properties (the yield strength of silicon)
- Mechanical device properties, which involve:
  - the relationship between silicon membrane transmembrane pressure, and the membrane stress
  - the relationship between the volume change mapped out by the silicon membrane deflection, and the membrane stress
- Thermodynamic properties of the microvalve actuator fluid, which involve:

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- the phase state of the actuator fluid (liquid only, or gas-liquid, or vapor-liquid)
- the volume change of the actuator fluid as a function of mean fluid temperature
- the possible change in phase state due to changing external conditions of pressure and/or temperature:
    - changing from gas-liquid (or vapor-liquid) to liquid only (bubble collapse, or 'cocking')
    - changing from liquid only to gas-liquid or vapor-liquid (cavitation, or 'snap-on-cold')

- The effects of statistical variation on each of these elements in the chain of reasoning

2.1. Fundamental Material Properties

Silicon is an elastic material, which essentially does not fatigue. However, it can fracture. Membranes manufactured from silicon must therefore be engineered to avoid conditions under which fracture can occur. This requirement is particularly important for silicon microvalves, where a flexible silicon membrane comes into contact with a hard valve seat, potentially with high force and attendant stress.

The first step toward a complete understanding of microvalve devices, which enables them to be so engineered, is embodied in Figure 1. It shows the statistical distribution of silicon microstructure failure as a function of silicon stress (that is, yield strength) [18]. While quantitatively related to specific microbeam structures, this type of curve represents qualitatively the expectations for any silicon membrane. Other factors which may affect the bulk or surface nucleation of silicon fracture events include thermal processing conditions (time, temperature, and gaseous annealing environment), and concentration of defects, including dopants and oxygen precipitates. We have found that control of these parameters can affect somewhat the mean of the yield strength (as determined by subjecting an ensemble of membranes to fracture); whereas such control has a greater effect on the standard deviation of the fracture distribution of the ensemble. This type of statistical distribution must therefore be viewed as a fundamental and immutable property of silicon. Any device fabricated from silicon must account for it.

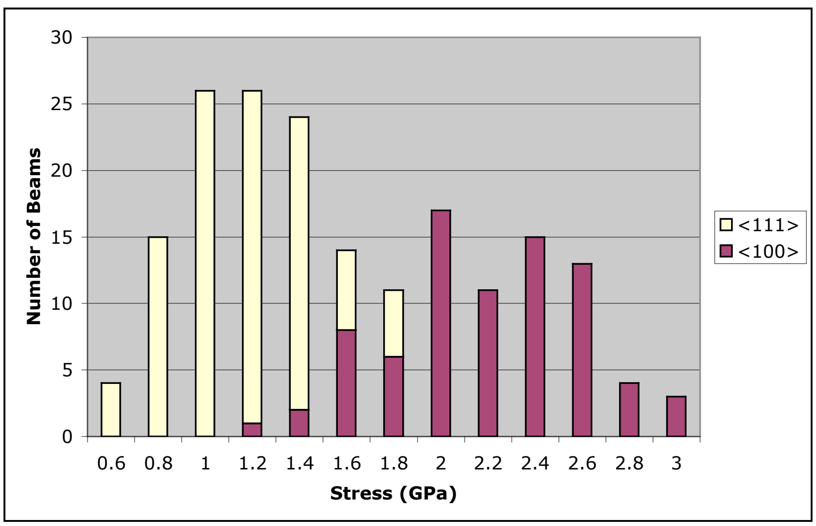


Figure 1: Probability of Silicon Fracture vs. Peak Stress (after Wilson and Beck [9]).

2.2. Mechanical Device Properties

The second relationships which must be defined are the peak stress generated in a silicon membrane, and the volume mapped out by the membrane deflection, both as a function of the transmembrane pressure. These characteristics have been determined for specific microvalve designs, including normally-off shut-off microvalves [38, 39], and normally-open proportional valves [2]. These determinations were made primarily by comparing measured results of membrane deflection vs. transmembrane pressure, with simulated results (using ANSYS) of membrane deflection (and peak stress) vs. transmembrane pressure [28, 34, 35]. We also made an attempt to measure stress directly in our membranes, using micro-Raman spectroscopy [26, 28]. Other relationships of stress vs. transmembrane pressure can be obtained, through the engineering of different membrane geometries. Generally speaking, however, no matter the specifics of the geometry in terms of the presence or absence of one or more boss, or pedestal, components, the measured and modeled behavior in terms of displacement and stress follows the predictions of the non-linear models in ANSYS, and of the analytical equations in references such as Ref. 40.

Figure 2, then, shows the membrane peak stress, as a function of transmembrane pressure, for a representative shut-off valve (SOV) membrane, with a membrane thickness of 35 µm, and two or three pedestals. This figure also shows a schematic cross-section of the SOV, and a top view of the ANSYS structure used in simulations. A different set of characteristics will hold, if the pedestal size or position is changed, or if more pedestals are added; or, if the lateral size of the membrane is changed; or, if the membrane thickness is changed.

Figure 3 shows the peak stress as a function of the volume mapped out by the membrane deflection for this three-boss membrane. The mapped-out volume is related directly to the volume change of the actuator fluid, whether liquid-only or vapor-liquid, as discussed in the next section. 'Pull-in' in the figure title refers to the characteristic that the membrane is pulling in, due to a transmembrane pressure that shrinks the volume of thermopneumatic cavity. The transmembrane pressure leading to this shrinkage is the difference between the external pressure, and the cavity pressure. The cavity pressure is set by the thermodynamic conditions of the thermopneumatic fluid as it is encapsulated in the cavity, and the mean fluid temperature thereafter. The mathematical details relating to the cavity thermodynamics are given in Ref. 35.

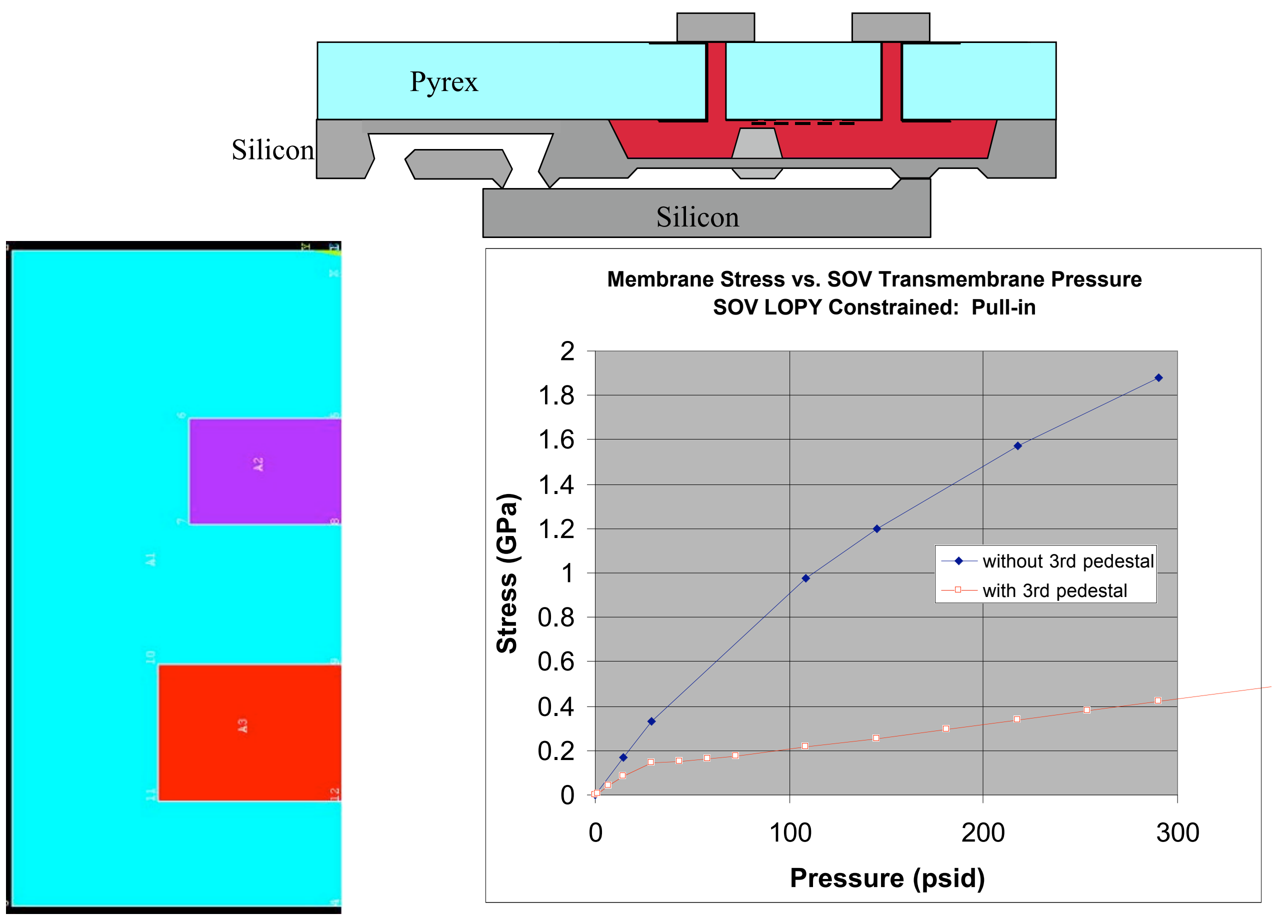
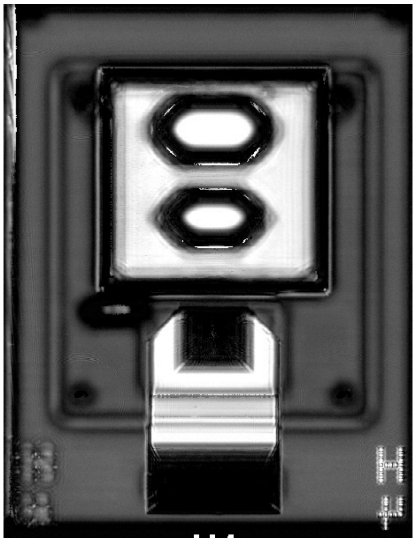


Figure 2: Top: Schematic cross-section of a normally-closed thermopneumatic shut-off valve. The relative positions of the three bosses/pedestals are shown. The third pedestal is the one located in the actuator cavity. The membrane is 35 µm thick, and is about 4.5 mm square. Left: Top view of the two- or three-pedestal SOV membrane (from ANSYS simulation). Only half of the symmetric membrane shown. The third pedestal, or boss, is located on the opposite side, beneath the smaller of the two pedestals. Right: Peak SOV membrane stress vs. transmembrane pressure.



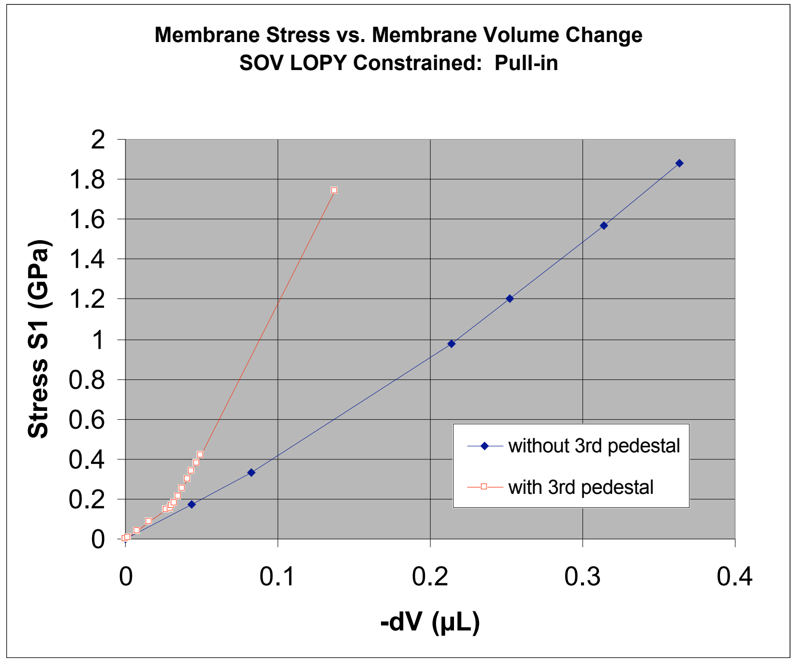


Figure 3: Left: Acoustic microscope image (top view) of a completed SOV, according to the top portion of Figure 2. Right: Peak SOV membrane stress vs. volume change mapped out by membrane deflection.

Figure 4 shows linescans through the center symmetry line of a 50 µm SOV membrane (in this case, with only a single pedestal), for different values of transmembrane pressure. The integration of the displacement across the full extent of the membrane area constitutes the volume mapped out by the transmembrane pressure. In an operating actuator, this volume is filled by the thermopneumatic fluid sealed in the actuator cavity.

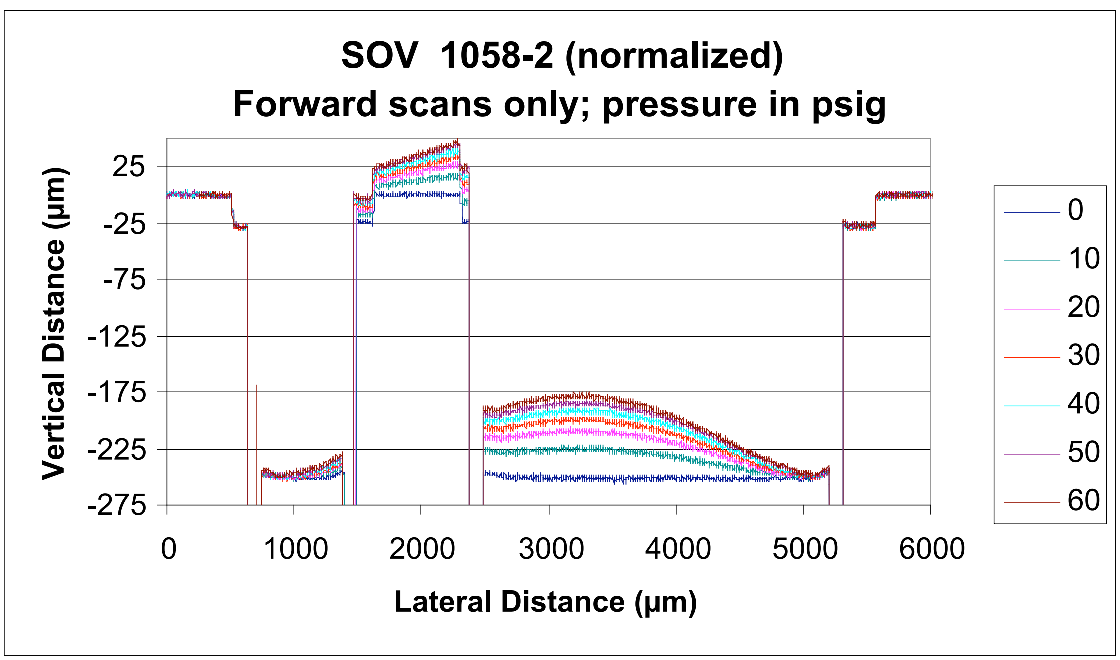


Figure 4: CyberScan™ laser linescans through the center symmetry line of an SOV membrane, showing the deflection of the membrane as a function of transmembrane pressure.

2.3. Thermodynamic Properties of the Microvalve Actuator Fluid I: The Effect of Cavitation (Snap-On-Cold)

The fundamental connection between the thermodynamic state of the actuator fluid, and membrane reliability, occurs through the volume change of the actuator fluid, depending upon whether the cavity is liquid-only, or gas-liquid (vapor-liquid). As shown in Figure 5, the fundamental thermodynamic nature of the actuator fluid – in particular, its pressure and temperature dependence, relative to the phase diagram – will determine whether the cavity is in an all-liquid state, a vapor-liquid state due to boiling, or a vapor-liquid state due to cavitation. The introduction of other gases into solution with the thermopneumatic liquid also can affect the state of the thermopneumatic cavity. With respect to Figure 5, the small circle shows a representative initial state of the thermopneumatic fluid as it is sealed into the cavity. In this instance, that state is liquid. If the liquid is heated enough, a vapor-liquid state will result due to boiling. If the liquid is cooled from its initial state, the liquid shrinks in volume. Because of surface tension between the liquid and the membrane, the membrane is pulled inward. Because of the energy stored in the membrane due to this pull-in effect, the path of the thermodynamic state of the liquid through the phase diagram can cross the vapor dome, resulting in a release of the surface tension force, and creation of a vapor-liquid state due to cavitation. The release of the energy stored in the membrane results in mechanical oscillations in the membrane just after release.

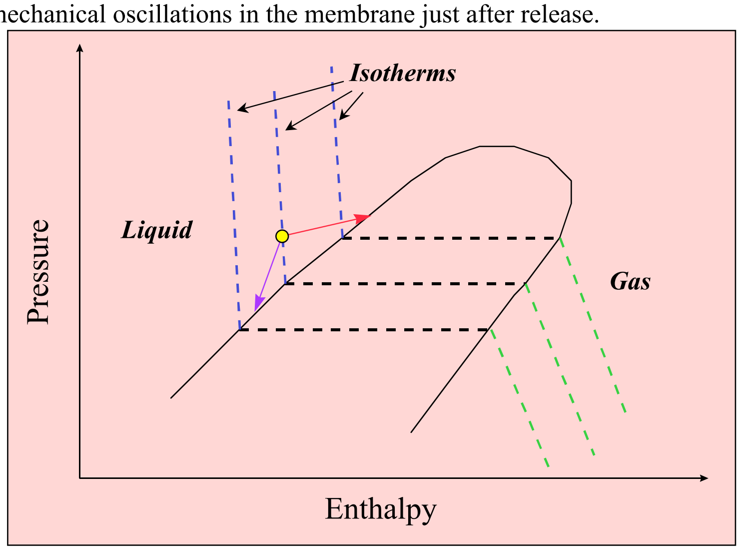


Figure 5: Thermodynamic phase diagram of the fluid in a thermodynamic actuator. The circle shows a hypothetical initial state. The arrow to the right shows the trajectory if the cavity is heated. The arrow to the lower left shows the trajectory if the cavity, and the fluid, are cooled.

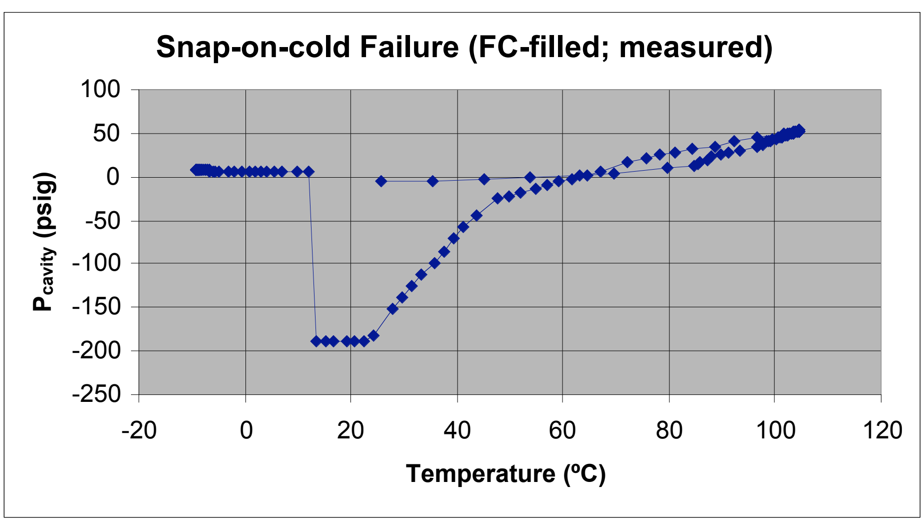


Figure 6: SOV cavity pressure vs. thermal cycling temperature for Fluorinert fluid.

Studies based on thermal cycling from about –20 ºC to about 100 ºC have shown the kind of characteristic represented by Figure 6. This figure shows the cavity pressure, as measured by an externally-mounted silicon piezoresistive pressure sensor (see Figure 7), vs. the isothermal temperature. In this particular example, cavitation occurred at about 14 °C. And, this cavitation event caused the membrane to fracture and fail. For the Fluorinert™ liquids, we have in nearly every instance experienced cavitation, or 'snap-on-cold', over this temperature range. Usually, the cavitation does not cause fracture of the membrane. However, even membranes which have experienced many successive thermal cycles which include cavitation without fracture, can still fracture upon a subsequent cavitation event. This phenomenon is not due to fatigue or work hardening of the silicon membrane. Rather, it is due to the transient cavity pressures caused by the cavitation event. This phenomenon will be detailed in the next section.

We have attempted to reduce the cavity pressure at which cavitation occurs, by introducing surfaces with very small radius of curvature. We have also tried to reduce the intrinsic surface tension of the actuator fluid, so that cavitation occurs at lower pressures. To date, efforts in both these directions have failed.

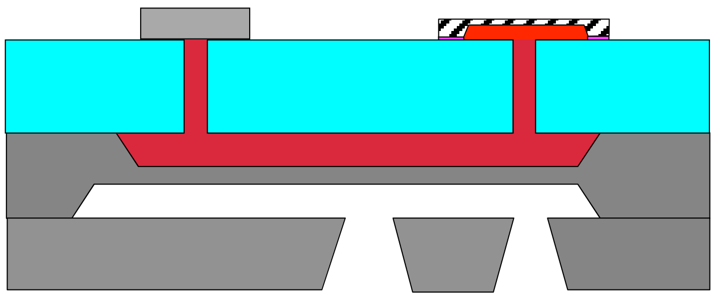


Figure 7: Cross-section of normally-open thermopneumatic microvalve with (cross-hatched) pressure sensor.

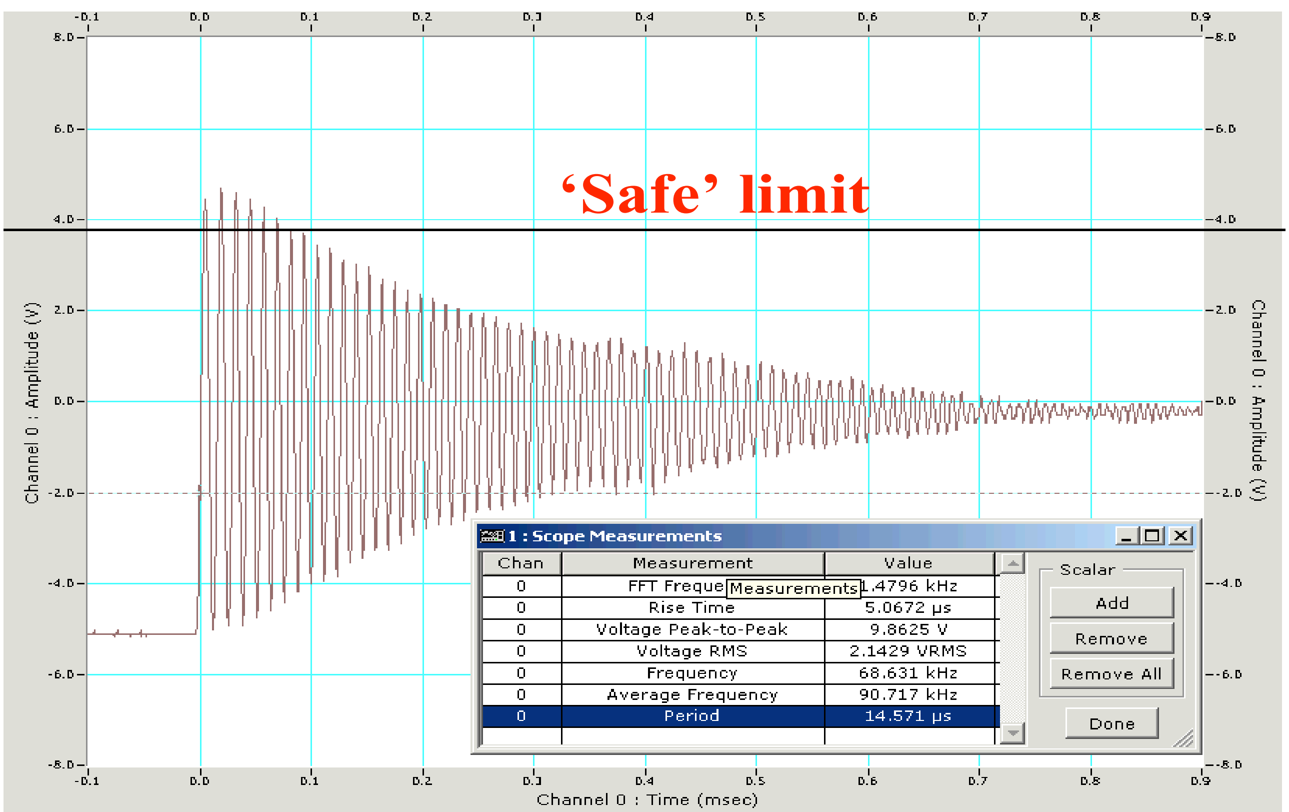


Figure 8: Oscilloscope trace of SOV actuator cavity pressure during cavitation event.

2.4. The Effects of Statistical Variation

Based on the figures in the preceding sections, it may seem a reliable thermopneumatic actuator design has already been achieved: the static cavity pressures caused by the shrinking Fluorinert™ liquids should not cause the valve membrane (with three pedestals, at least) to exceed the silicon yield strength – even if the silicon is from a particularly weak lot in terms of the fundamental yield strength distribution as in Figure 1. So, why do membranes still fail?

The reason is due to the transient effect imposed on the actuator cavity pressure, and thus on the stress in the silicon membrane, as shown in Figure 8. In this figure, the amplitude is proportional to the cavity pressure. The maximum negative static pressure occurs at t=0. In this particular example, membrane fracture did not occur, although the cavity pressure which causes stress to exceed a safe level (labeled 'safe limit' in the figure) has been exceeded. However, this curve is not repeatable. That is: oscilloscope traces captured for successive cavitation events on a single actuator cavity show two important features. First, the frequency of oscillation is not constant, and can vary between about 20 and 50 kHz. Second, the actuator cavity pressure transient response during cavitation can appear to have more than one oscillation frequency (note the amplitude increase after t=0 in Figure 8: evidence of more than once oscillation mode). Third, and most important, the actuator cavity pressure transient response can <u>exceed</u> the maximum static pressure prior to cavitation, by as much as about 30%. *It is the random nature of this transient response, and its effect on cavity pressure, and thus on membrane stress, which makes membrane fracture possible, even in membranes which have survived many thermal cycles and many cavitation events.*

2.5. Thermodynamic Properties of the Microvalve Actuator Fluid II: The Effect of Alternate Cavity Fluids

It is not completely understood why the cavitation event sometimes causes actuator cavity transient pressure to be greater than the static pressure, so that fracture can occur. It may be that the spatial initiation point for the cavitation is not constant from cycle to cycle. It may be that cavitation at one point excites only one cavity oscillation mode, in a safe manner so that fracture cannot occur; while cavitation at another point excites multiple modes, which trade energy back and forth, so that the yield strength can be exceeded, and the membrane may fracture.

Figure 9 demonstrates what is required in the case of the SOV so that reliable operation is achieved. We measured improvements in mean-time-to-fail (MTTF), or mean thermal cycles to fail, afforded by the use of HFE™ instead of Fluorinert™ [41] as the actuator fluid, and those results will not be presented here. However, the MTTF improvement is directly due to the reduced (negative) cavity pressures afforded by the HFE™ liquid, and by the absence of cavitation.

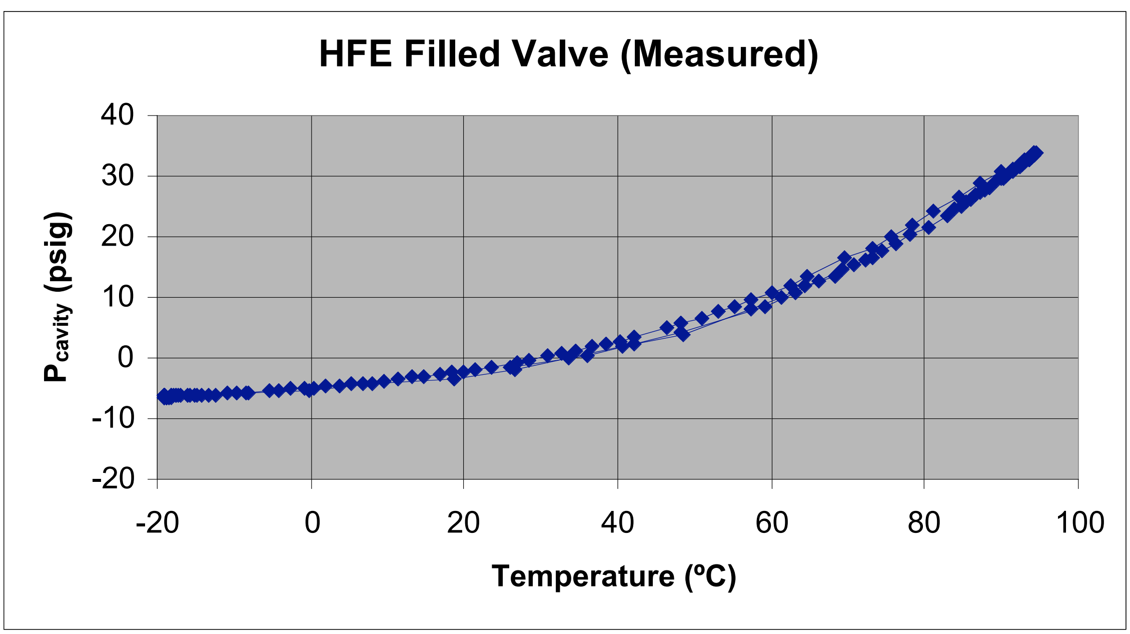


Figure 9: SOV cavity pressure vs. thermal cycling temperature for HFE™ fluid.

The question remains: why does HFE™ succeed, where Fluorinert™ does not? Why does HFE™ have a reduced negative cavity pressure? Why does HFE™ have no cavitation? The answer is given in the comparison of the next two figures. Both are based on established models for Fluorinert™ actuator fluids [35], combined with the mechanical behavior of a dual pedestal SOV membrane. (Note that, if the three-pedestal membrane mechanical behavior had been incorporated, the negative cavity pressures in the liquid-only state for Figures 10 and 11 would be increasingly negative, matching what is measured experimentally in Figure 6.)

Figure 10 plots the actuator fluid cavity pressure for the vapor-liquid state, and for the liquid-only state, for the FC-72/FC-84 mixture shown. In this example, the fill factor is 1.0, which means there are no voids (bubbles) in the cavity after the hermetic seal is made. At low isothermal temperatures, there is a gap in the cavity pressure for the two possible states of the actuator fluid. In practice, the thermodynamic state of the actuator can have either value. See Figure 6 for a measured example of just such a situation. The existence of the two states means that cavitation can occur. So, in this example, when the isothermal temperature rises above 50 °C, a bubble (which may exist at lower temperatures due to cavitation) is collapsed, and the actuator fluid is said to have become 'cocked'. During subsequent cooling below 50 °C, the cavity pressure will follow the lower curve (liquid-only state), until the surface tension of the cavity liquid is exceeded by the negative cavity pressure. At this point, cavitation occurs. And, as we have shown, cavitation (snap-on-cold) can cause membrane fracture, with a non-zero probability.

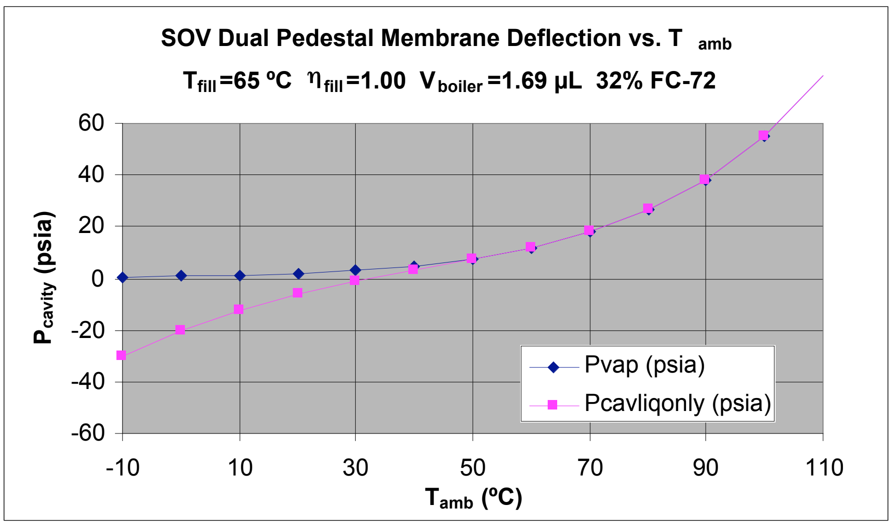


Figure 10: Modeled SOV cavity pressure vs. thermal cycling temperature for Fluorinert™ actuator fluid (mix of FC-72 and FC-84).

The avoidance of cavitation, and the achievement of reliable actuator operation, is described by Figure 11. The modeled actuator fluid is the same as in Figure 10, except that now the fill factor is only 0.97. This means that the actuator cavity has the possibility of retaining a bubble over a much broader temperature range. In Figure 11, the liquid-only actuator fluid pressure is always less than the vapor-liquid pressure, across the temperature range shown. Thus, the mechanical system represented by the actuator fluid in contact with the mechanical membrane never becomes cocked, as long as the microvalve never is subjected to a temperature above approximately 120 °C, in this case.

Now compare the vapor-liquid phase curve of Figure 11 with the measured HFE™ performance in Figure 6. They are qualitatively, and almost quantitatively, identical. This means that the HFE™ achieves reliable actuator operation, and protection from membrane fracture due to cavitation, by not allowing the fluid-membrane mechanical system to become 'cocked' over the given temperature range. This characteristic derives from the presence of a bubble in the HFE™ actuator over the full temperature range.

How does the HFE™ fluid achieve this gas-liquid phase over the full temperature range? We believe it is due to the higher solubility of water vapor in the liquid HFE™, as compared to the Fluorinert™ liquids. The fill procedure used saturates the thermopneumatic liquid at low temperature with water vapor. After the hermetic seal is made, the room temperature state reduces the solubility, and the water vapor creates a bubble. This bubble persists over the full range of temperature to which the microvalve is subjected. The existence and persistence of this bubble ensures the microvalve membrane is never subjected to cavitation, and so will not fracture, similar to the effect shown in Figure 11.

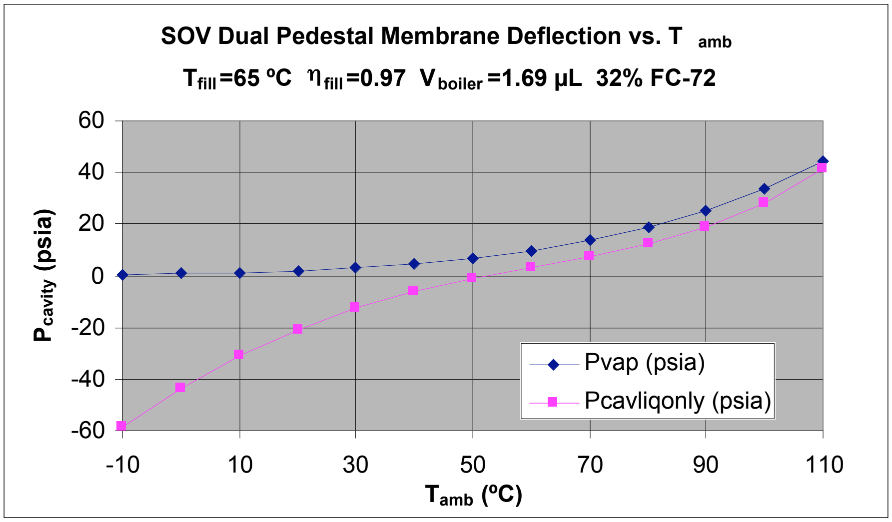


Figure 11: Modeled SOV cavity pressure vs. thermal cycling temperature for Fluorinert™ actuator fluid with fill factor = 0.97.

3. DISCUSSION

It is desirable to make a map of 'safe regions' of inlet pressure and operating temperature (see Figure 12), for a particular thermopneumatic actuator design. This safe operating region map defines where bubble collapse, and 'cocking' of the membrane, may occur, which would then create a possibility for membrane fracture. The map thus defines a specification, not only for actuator operation, but also for actuator storage and shipment, which will eliminate membrane fracture due to cavitation ('snap-on-cold').

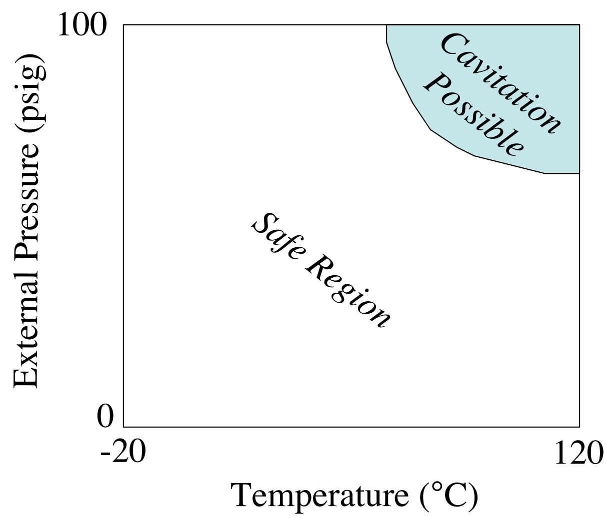


Figure 12: Estimate of safe region for 35 µm membrane, Three-pedestal SOVs with HFE™ actuator fluid.

Several statements are needed for clarification. First, Figure 12 is not derived from measurements. It is an estimate based on one set of measurements. The boundary between the 'safe' region, and the region where cavitation is possible, must be delineated by measurement, not estimation. Second, the map which results is by no means the best which can be achieved. It holds only for the 35 μm thick membrane SOV, with three pedestals, as presently described, for the narrow HFE™ Fluorinert™ conditions which have been measured.

A summary of the unknowns discussed earlier in this work is worthwhile. Reliable actuators are a function of many variables. Membrane material yield strength must be a maximum. So, surface roughness must be controlled, as must the thermal processing temperature and gaseous environment of the membrane manufacturing process. If the membrane is silicon, then defect concentrations (dopants, and oxygen precipitates in particular) must be controlled. The fill process associated with introduction of the thermopneumatic fluid into the hermetic actuator cavity must be controlled, with particular attention given to dissolved gases, fill factor, fill temperature, and fill pressure. Surface tension between the thermopneumatic fluid and the membrane material can be estimated, but for the most part is unknown. Future experiments to explore this issue would be fruitful, in particular to determine if structural features could be fabricated to ensure release of surface tension at low values of stored mechanical membrane energy. Design of membranes to suppress oscillations associated with excess stress during cavitation transients could be helpful. In particular, suppression of oscillation modes which could trade energy (leading to membrane stress in excess of membrane materials yield strength) could be helpful.

5. CONCLUSIONS

Given the understanding and control of materials properties, thermodynamic properties of the thermopneumatic fluid, and structural dimensions of the thermopneumatic actuator, the following must occur to achieve a reliable actuator:

- For a proportional valve (typically, a normally-open valve), the thermodynamic state of the actuator fluid must remain liquid under all possible external conditions of temperature and pressure. Cavitation, or snap-on-cold, must be avoided. This condition can be engineered for virtually every actuator design, by adjusting the mechanical design of the membrane (membrane width and thickness), and by adjusting the thermodynamic state of the actuator fluid during the fill process. Other means of achieving this result (by, for instance: increasing the yield strength of the silicon; or by engineering the surface tension in the actuator fluid; or by engineering to a lower value, the cavity pressure at which cavitation occurs) would require substantial research.
- For the on-off shut-off valve, the thermodynamic state of the actuator fluid must remain gas-liquid or vapor-liquid under all possible external conditions of temperature pressure. Bubble collapse, or ‘cocking’, must be avoided, as it leads to the possibility of sudden cavitation, or snap-on-cold, which can result in membrane fracture. This condition can be engineered for virtually every actuator design. The results reported here with the HFE™ actuator fluid demonstrate just such a robust design.

For other structural materials or dimensions, and other thermopneumatic fluids, the models developed (in Ref. 35) are sufficient to design reliable actuators, provided the structural material properties, and fluid thermodynamic properties, are well-defined. Additions to the model, to include a quantitative description of dissolved gases in the thermopneumatic fluid, are forthcoming. In the meantime, the effects of dissolved gases can be modeled adequately by modulating the fill factor in the model.

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