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Fabrication of a micro cryogenic cold stage using MEMS-technology

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Abstract

This paper describes the design and production process of a variety of reliable micro cryogenic coolers. The different cold stages are based on an optimized design found during a study which was done to maximize the cold-stage effectiveness. Typical cold-stage dimensions are $30 \times 2 \times 0.5 \text{ mm}$ with an expected net cooling power varying from 10 mW to 20 mW at a tip temperature of 96 K. A cold stage consists of a stack of three fusion bonded D263T glass wafers. The production process has 7 lithography steps and roughly 100 process steps. In order to determine the maximum bend, shear and bond stresses inside a 175 $\mu$m thick D263T glass wafer, several pressure tests were performed.

1. Introduction

Cooling of electronic circuitry to very low temperatures can improve the signal-to-noise ratio and bandwidth of a system. In many cases the system, which is to be cooled, is very small so an accompanying small cooler would be obvious. Several attempts have been made to construct a miniature refrigerator using MEMS technology [1]. This technology is highly suitable for the fabrication of a micro cooler because of the high accuracy, possibility of integrating the system with the electronic circuitry and use of batch processing, which can result in relatively low cost per unit. The next section gives a brief introduction to the cryogenic aspects involved. Section 3 describes the previous work which has been done on the development of a micro cryogenic cooler. The optimization of the cooler design is considered in section 4. Section 5 gives the results of the pressure tests done to determine maximum stresses inside thin glass wafers. Fabrication of the cold stage is treated in section 6 followed by the measurements in section 7.

2. Cryogenics

Cryogenics is the field of physics which is concerned with the production and investigation of extremely low temperatures, typically temperatures lower than 120 K. Cryocoolers can be used to cool down electronic circuitry to improve the signal-to-noise ratio and bandwidth of a system. For superconducting devices it is crucial that they are cooled below there critical temperature in order to operate properly. In many cases the system, which is to be cooled, is very small so an accompanying small cryocooler would be obvious. Cryocoolers are based on a whole range of different cooling principles [2, 3]. For the fabrication of a micro cooler, research was mainly focused on cooling through the expansion of a gas. All micro cooler work previously presented as well as the current project uses a so-called Joule–Thomson (JT) cooling cycle (figure 1). In a JT cold stage, high-pressure gas flows through a counter flow heat exchanger (CFHX) to a flow restriction. Through this restriction, the gas undergoes isenthalpic expansion to the low-pressure side, cools and usually changes its phase to a liquid [2, 3]. By absorbing heat from its surroundings the liquid evaporates and the produced vapor flows back through the CFHX absorbing heat from the warm high-pressure side. In open-cycle systems, the low-pressure gas leaving the CFHX is vented to air, whereas in a closed-cycle system the fluid is re-compressed as is depicted in figure 1.

3. MEMS-based cryocoolers

Since the 1980s, different groups around the world have tried to build small cryogenic refrigerators [1]. Most noticeable
results were achieved by Little and Burger, who fabricated cold stages which had a total volume of about 1 cm$^3$ using MEMS technology.

In the 1980s, Little et al made a range of miniature cold stages [4, 5] using an abrasive etching process. The smallest was $15 \times 2 \times 0.5$ mm ($0.015$ cm$^3$) and used an open-loop JT cycle with nitrogen gas; see figure 2. A cooling power of 25 mW at 101 K was claimed. The smallest cold stages that Little et al have manufactured were not reproduced or turned into an industrial product. Larger coolers of this design, however, were made commercially available by MMR technologies, Inc. [6]. Typical dimensions are $60 \times 14 \times 2$ mm ($1.7$ cm$^3$). For applications where this cooler size is much larger than the device to be cooled (e.g., a low noise amplifier) an even smaller cooler could be preferable. Our research aims at fully optimized, reliable micro coolers ($\ll 1$ cm$^3$) that can be manufactured in large quantities for these purposes.

In early 2000, Burger et al developed a JT cold stage with a total volume of $0.76$ cm$^3$ ($77 \times 9 \times 1.1$ mm, see figure 2). They combined the cold stage with a sorption compressor, thus realizing for the first time a closed-cycle micro cooler [7]. This miniature cooler had a cooling power of 200 mW at 170 K. Burger used MEMS technology to construct most of the components of the cold stage. Counter-flow heat exchangers were made out of tiny glass capillaries. After fabrication, the different cold stage parts were glued together.

Currently, at the University of Twente a project is running to develop a reliable and reproducible micro JT cold stage using MEMS technology only [8]. By using a sorption compressor the gas cycle will be closed in combination with a total exclusion of any mechanical moving parts. The aim is a cold stage with a tip temperature of 96 K and a net cooling power of about 10 mW. In cooperation with Micronit [9], a set of cold stages has been fabricated with a total volume varying from 0.02 cm$^3$ to 0.1 cm$^3$. This paper presents the design approach and production process of these micro cold stages.

### 4. Optimization of the cooler design

The micro cooler is based on a JT-cycle with nitrogen gas at a high pressure of $p_H = 80$ bar, a low pressure of $p_L = 6$ bar and a mass flow of $\dot{m} = 1$ mg s$^{-1}$. The temperature of the cold tip which is 96 K, is determined by the evaporation temperature of the used gas ($N_2$) at the chosen low pressure. Clarification of the chosen parameters and further explanation of the cycle can be found in [10].

The most crucial part of a cryocooler based on the JT principle is the counter flow heat exchanger (CFHX). The CFHX maintains the temperature gradient between the warm and cold ends of the cooler and greatly improves the efficiency of the cooler cycle by exchanging heat between the high-and low-pressure gas flows. Because the amount of energy that is exchanged in the CFHX is typically two orders of magnitude larger than the cooling power in the evaporator, a small reduction of effectiveness results in a large decrease of available cooling power.

To maximize the effectiveness of the CFHX, a geometry that results in an optimal heat exchange between the high-and low-pressure lines is needed. In general, this means that the heat-exchange surface between these lines has to be maximized. Two rectangular channels on top of each other form in this respect a convenient configuration. A very thin layer separates the channels; see figure 3.

In a CFHX, two important loss mechanisms can be distinguished. The first is the loss due to pressure drop in the flow channels and the second is the loss due to conductive heat flow. To reduce the heat flow from the hot side to the cold tip of the cooler, the entire device is fabricated in glass. Glass has a relatively low thermal conductivity ($\lambda \approx 1$ W m$^{-1}$ K$^{-1}$, in
Fabrication of a micro cryogenic cold stage using MEMS-technology

Table 1. Design parameters of the different cold stages. First two dimensions apply to both the high- and the low-pressure channels. The CFHX channel depth is 50 µm for all designs. M1 and M2 in the first row stand for, respectively, multistage cooler 1 and multistage cooler 2. The tip temperature of all stage is calculated at 97 K (using N2), except for the second stage of the multistage which is calculated at 27 K (using Ne).

<table>
<thead>
<tr>
<th>Prototype number</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>M1</th>
<th>M2</th>
</tr>
</thead>
<tbody>
<tr>
<td>CFHX channel width (mm)</td>
<td>2.0</td>
<td>2.0</td>
<td>2.0</td>
<td>2.0</td>
<td>4.0</td>
<td>5.0</td>
<td>4.0</td>
<td>2.0</td>
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<tr>
<td>CFHX channel length (mm)</td>
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<td>15</td>
<td>35</td>
<td>25</td>
<td>25</td>
<td>25</td>
<td>25</td>
<td>25</td>
<td></td>
</tr>
<tr>
<td>Mass flow (mg s⁻¹)</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
<td>2.0</td>
<td>2.0</td>
<td>2.0</td>
<td>3.0</td>
<td>2.0</td>
<td>1.0</td>
</tr>
<tr>
<td>P̄gross theory (mW)</td>
<td>14.74</td>
<td>14.74</td>
<td>14.74</td>
<td>29.48</td>
<td>29.48</td>
<td>29.48</td>
<td>44.22</td>
<td>29.48</td>
<td>9.30</td>
</tr>
<tr>
<td>P̄net calculated (mW)</td>
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<td>7.80</td>
<td>13.60</td>
<td>20.00</td>
<td>24.10</td>
<td>25.50</td>
<td>39.60</td>
<td>25.50</td>
<td>5.50</td>
</tr>
</tbody>
</table>

Figure 3. Left: a 3D-view of a part of the CFHX. Right: a cross-section of the CFHX.

Figure 4. The Femlab model of an isotropically etched channel with a depth of 50 µm. The pressure inside the channel is 80 bar. Left: channel geometry on the left side, the symmetry axis and Femlab-mesh included. Right: stress distribution inside the glass. A peak value of 550 MPa is found in the channel corner.

Comparison with silicon: λ ≈ 1000 W m⁻¹ K⁻¹ [11]. To minimize the losses, and thus optimize the effectiveness of the CFHX, a study was performed on the basis of minimizing the total entropy production in the CFHX. This study was published elsewhere [10]. The fact that the presented cold stage is optimized for maximum cooling power in combination with minimum pressure drop makes the design distinct from that of both Burger and Little et al. The coefficients of performance (COP) of the micro cold stages presented in this paper are approximately doubled compared to the designs fabricated by Little including the smallest (0.5 × 2 × 15 mm). In other words, for the same input power, the net cooling power has roughly been doubled.

The found dimensions of the flow channels were used in the design. Due to fabrication constraints, however, the thicknesses of the used wafers vary slightly from the assumed wafer thicknesses during the optimization study. Also pillars inside the flow channels were added later to the design to reduce stresses inside the glass, see section 6. The relevant dimensions of the optimal design [10] are given in table 1. Besides this optimal design, seven other designs, all based on it, are fabricated. This is done to verify the used optimization model and to investigate the influence of different design parameters such as the dimensions of the CFHX and the value of the mass flow.

When the cold tip is at a cryogenic temperature, there is a possibility that contaminant gases inside the nitrogen (e.g. H₂O) freeze inside the restriction which reduces the mass flow and thus the cooling power. To reduce the chance of clogging, a relatively wide and shallow restriction geometry was chosen. The width of the restriction is chosen to be 1 mm and the height 300 nm. The length is determined from the required design flow of 1 mg s⁻¹ [10] and the equation,

\[
\dot{m} = \frac{1}{12} w h^3 \int_{p_l}^{p_h} \frac{\rho(p, T)}{\mu(p, T)} \, dp, \tag{1}
\]

where \(\dot{m}\) is the mass flow through the restriction, \(w, h\) and \(l\) are respectively the width, height and length of the flow restriction, \(\rho(p, T)\) is the density of the gas and \(\mu(p, T)\) the viscosity both dependent on pressure and temperature. Using this equation and the mentioned parameters, a length of 140 µm results.

5. Pressure tests

As was mentioned in the previous paragraph, the pressure inside the high-pressure micro channel is 80 bar. This relatively high pressure can result in high stresses inside the fabrication material (glass). Maximum stress values for glass found in the literature vary from \(\sigma_{\text{max}} \approx 50\text{ MPa}\) for Pyrex glass to \(\sigma_{\text{max}} \approx 140\text{ MPa}\) for D263T glass [12, 13]. Finite-element simulations (Femlab [14]) show extremely high stresses concentrated in the sharp corner of the pressurized channel (\(\sigma_{\text{max}} = 550\text{ MPa}\)). Figure 4 shows a schematic of a single-channel model which is pressurized to 80 bar. The left figure shows the channel geometry with the chosen mesh. The right figure shows the calculated stress distribution inside
were fabricated that consist of a single channel HF etched in D263T glass as is stated by the manufacturer [13]. Etching results strongly depend on the specific mesh that is chosen. The smaller the mesh, the higher the peak value will be. This is a result of the infinitely sharp corner that is used in the computer model. In practice, this corner will have a finite curvature. Because it is difficult to predict this curvature the simulation results can be used as a qualitative indication only.

Clarity about the maximum allowable stresses inside D263T glass is needed to minimize the chance of a mechanical failure of the micro cold stages. Since simulations give no clear value of the stresses involved, pressure tests were performed to determine the maximum allowable stress in thin D263T glass wafers (thickness = 175 µm). Pressure test samples were fabricated that consist of a single channel HF etched in a 175 µm thick D263T glass wafer; see figure 5. The channel width varies from 110 µm to 3000 µm. The channel depth is 50 µm leaving a membrane thickness of about 125 µm. Another glass wafer with a thickness of 400 µm is fusion bonded to the thin wafer. Gas pressure is applied via a micro hole (diameter 300 µm), powder blasted in the 400 µm wafer. Variability of the glass substrates thickness is standard ±2%. Variability in the channel widths is standard ±10% according to the manufacturer’s specification [13]. Etching depths are accurate to ±2%. Variability in the channel widths is standard ±4 µm. The channel corner has a 90° angle with the top plate which is ensured by the isotropic etching process. SEM observations have been done to ensure proper isotropic behavior. More detailed processing information concerning the pressure test samples as well as the micro coolers is to be discussed by Micronit [9].

To support the connection part, the area surrounding the powder-blasted micro hole is glued between rectangular pieces of stainless steel. The total sample can then be clamped in a connection piece which contains small Viton O-rings; see figure 6. The samples are tested in a relative pressure range of 0–120 bar until they fail. The maximum normal (a.k.a. bend) stress inside the material is defined as

$$\sigma_{\perp, \text{max}} = \frac{p_{\text{max}} w^2}{2 d^2},$$

with $p_{\text{max}}$ the failure pressure of the sample, $w$ the width of the channel and $d$ is the thickness of the membrane. The maximum shear stress inside the material is defined as

$$\sigma_{\parallel, \text{max}} = \frac{p_{\text{max}} w}{2 d}.$$

Equations (2) and (3) are derived for a uniformly distributed load on a rectangular plate clamped at both sides [15]. The total stress inside the material is a combination of the two stresses [15]. The bend to shear stress ratio is given by $w/d$ meaning that for the test samples the bend stress equals the shear stress for a channel width of 125 µm. Therefore, it can be concluded that the bend stress will dominate in the majority of the tested samples. Samples with a channel width smaller than 500 µm did not fail within the applied pressure range. These were two samples with a channel width of 150 µm, two samples with 350 µm and two samples with 450 µm.

Figure 7 shows the results of the pressure tests. These graphs show the different values of the maximum normal and shear stress, determined by substituting the found failure pressure of a sample in equations (2) and (3), with the total stress being a combination of the two. The maximum normal stress increases with increasing channel width whereas the maximum shear stress is decreasing with increasing channel width.

The graphs show that, in almost all cases, the maximum normal stress found in the experiments is equal to or higher than the maximum stress value we found in the literature for D263T glass, i.e. 140 MPa [13]. The maximum normal stress found is determined at $\sigma_{\perp} = 450$ MPa and the maximum found value for the shear stress is $\sigma_{\parallel} = 31$ MPa. The total number of samples tested is 27 (i.e. those shown in figure 7 and the six samples with a channel width smaller than 500 µm). Since glass is a brittle material, there is very little plastic deformation before a failure occurs [15]. Local defects in the glass (e.g., caused by the production process) can be a location for stress concentration and a starting point of a fracture. This property will result in a statistical distribution of the found maximum stresses for samples with the same channel width, explaining the spread in the found maximum stresses.

None of the tested single channel samples (figure 7) showed a failure that indicated a rupture of the bond. In other words, all the samples failed by exceeding the maximum mechanical stress of the material, not by exceeding the maximum bond stress. The samples with a channel width between 640 µm and 1000 µm have a different failure
mode than those with a width of 2000 \( \mu \text{m} \) and 3000 \( \mu \text{m} \). The first always showed a failure where a small piece of the membrane blew off from the edge of the channel (see figure 5 magnification of sample D). At larger channel widths, the entire membrane was destroyed each time (see figure 5 magnification of sample A). It can be concluded that for the first group shear stresses play a larger role in the failure mechanism than for the second group. The samples fail by exceeding the maximum (shear) stress at a location at the channel edge where the stress is concentrated (e.g. a wafer defect or pinhole). In the second group, the bend stress is mainly the bottleneck. Since the channels are much wider, the bend stress in the middle of the channel membrane is relatively much higher. The membrane will bend to its maximum and break from the middle, tearing the entire membrane surface.

Besides determining the maximum normal and shear stresses of the material, also the maximum bond stress was tested. The maximum bond stress of a direct bond found in the literature is about 25 MPa \([16, 17]\). The bond sample is designed such that the force inside the channel is divided over a large area using pillars; see left picture of figure 8. In this way, both the normal and the shear stress are kept at relatively low values (\( \tau \) is very small) and the bond stress will be the limiting factor. Seven of these bond samples were tested. Six of the samples failed at relatively low pressures: 2, 5, 7, 15 and two at 17 bar which corresponds to bonding stresses of about 82 MPa. It can thus be concluded that if the bond is perfect, a relatively high maximum bond stress can be accomplished. However, it should also be noted that imperfections in the bond interface between the pillars and the membrane can introduce more fracture initiation points and thus may result in a lower maximum pressure because of stress concentrations.

All results presented in this paragraph were used in the design of the MEMS-based cryogenic micro cooler. It is seen to it that the maximum calculated stress in the micro cooler prototype does not exceed a chosen maximum allowable stress. For the maximum allowable stress the following values were taken: \( \sigma_{\perp, \text{max}} = 26 \text{ MPa}, \sigma_{\parallel, \text{max}} = 5 \text{ MPa} \) and \( \sigma_{\text{bond, max}} = 5 \text{ MPa} \). Using these relatively low values, the cold stage should be able to withstand the high pressure of 80 bar. The high- and the low-pressure channels as well as the restriction contain pillars to keep the maximum stress within these limits.

### 6. Cold-stage fabrication

The micro cryogenic cooler consists of a stack of three D263T glass wafers. The production process has 7 lithography steps and roughly 100 process steps. Each wafer goes through subtractive etch processes. Figure 9 shows 3D models of a cold stage, few of which are contained on a single wafer. Both sides of the three wafers are shown. A total of 22 cold stages in nine different designs were machined on a single wafer. Discussion on the different design parameters and presentation of the test results of all different designs will be presented in a separate paper. The fabrication process is described using figures 9 and 10. In the top wafer (thickness 175 \( \mu \text{m} \)), the flow restriction with a height of 300 nm is BHF etched ('A' in figure 10).
Next, the high-pressure gas channel with a depth of 50 µm is HF etched (B). This channel contains a high density of micro pillars (diameter 200 µm). The low-pressure channel (depth 50 µm) is HF etched (C) in the middle wafer (thickness 145 µm) leaving a thin wall of only 95 µm between the high- and low-pressure sides. The low-pressure channel is supported by pillars with a diameter of 50 µm. Feed-through holes with a diameter of 140 µm and slit feed-throughs for the evaporator are powder blasted in the middle wafer (D). These slits connect to the evaporator slits that are powder blasted in the bottom wafer (E). Simultaneously, gas in- and outlet holes (diameter 270 µm) are powder blasted half way through the bottom wafer (E). They meet the holes blasted from the bottom side in step (F). Also in step (F) the bottom wafer’s thickness is locally reduced to about 150 µm to minimize heat conduction through the CFHX. Next, the three wafers are fusion bonded to one stack. Alignment of the various plates before fusion bonding was performed using a mask aligner. Fusion bonding parameters for the pressure test samples and the cold stage samples were the same. Finally, the gaps between the actual cold stages and the protective rings are powder blasted (figure 9). In this step, the different samples are also separated from each other. Figure 11 shows four different fabricated micro cold stages. On the right, magnifications of the flow channel, micro pillars, restriction and evaporator are shown.

7. Measurements

As was mentioned before, detailed discussion on the test setup and test results of the different cooler designs will be presented in a separate paper. As an illustration of the successful fabrication, however, a cool-down curve of a cold stage with dimensions 30 × 2.2 × 0.5 mm is shown in figure 12. It can be seen that the cold stage works perfectly but does not reach the predicted temperature of 96 K. The cold tip temperature is measured with a thermocouple that is connected to the outside of the evaporator. It is calculated that the heat resistance of the thin layer of glass and the connection
between the thermocouple and the cold stage can easily give this $\Delta T$ of about 10 K. This specific design is developed to have a mass flow of 2 mg s$^{-1}$ with a theoretical gross cooling power of about 30 mW. Through measurement and estimation of additional losses, its net cooling power is estimated to be about 20 mW at a tip temperature of about 100 K.

8. Conclusion

A set of micro cryogenic coolers, consisting of a stack of three fusion bonded glass wafers, is fabricated using only MEMS technology. An optimization study has been done to minimize the cooler dimensions in combination with an optimal performance. In order to minimize the chance of a mechanical failure of the micro cold stages, the maximum bend, shear and bond stresses inside a 175 $\mu$m thick D263T glass wafer, are experimentally determined. There is a large variation in the maximum bend and shear stresses found as a result of the statistical nature of failure in brittle materials. The maximum value found for the shear stress is $\sigma_\parallel = 31$ MPa and the maximum bend stress is determined at $\sigma_\perp = 450$ MPa. The maximum value found for the bond stress is $\sigma_{\text{bond, max}} = 82$ MPa. A cool-down curve of one of the fabricated cold stages, with dimensions $30 \times 2.2 \times 0.5$ mm, was presented. This micro cooler had a net cooling power of 20 mW at about 100 K.

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