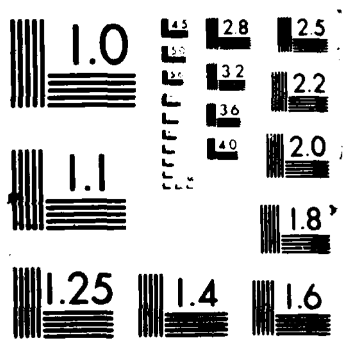
AD-A186 004 DEVELOPMENT OF A LOW NOISE 10 K J-T REFRIGERATION SYSTEM(U) MMR TECHNOLOGIES INC MOUNTAIN VIEW CA W A LITTLE 15 JUN 87 N00014-86-C-0301 1/1

UNCLASSIFIED F/G 13/1 NL


1.0
2.8
2.5
3.2
2.2
3.6
4.0
2.0
1.1
1.8
1.25
1.4
1.6

(12)

Item No. 0001AC

Final Technical Report

Development of a Low Noise 10 K J-T Refrigeration System

June 15, 1987

Contract N00014-86-C-0301

MMR Technologies, Inc.
1400 Stierlin Rd, #A5
Mountain View, CA 94043

Prepared by :

William A. Little
Chairman

87

TABLE OF CONTENTS

1.0 Introduction

This report summarises work done on Contract No. N00014-86-C-0301 in the period from Febrauary 16, 1987 to June 15, 1987 on the development of a low noise Joule-Thomson, microminiature refjrigeration system designed for 10 K operation.

In the previous report we described the calculation of the phase diagram of mixtures of certain hydrocarbon gases with nitrogen using the Benedict-Webb-Rubin equation of state. Based on this data and the experimental observations of the cooling capacity of the mixture in a Joule-Thomson refrigerator it was possible to redesign the three stage refrigerator to make use of the added cooling power of the mixture. This has now been done.

In the past, all the layout of the refrigerators had been done by hand, first by drawing the mask scaled up by a factor of 40 X, then laying it out on Rubylith , photographing the Rubylith and then reducing it photographically to provide the final mask for the lithography. We have now developed the capability of preparing the photo masks using a simple CAD software package. The masks are. laid out on the computer screen at 4 X magnification, they are then laser printed and photo-reduced. This process reduces by almost a factor of ten the time to complete a photo mask. In addition, the cost of the process is reduced by an order of magnitude because of two factors. First, the reduction of only a factor of 4 between the original drawing and the mask reduces the material costs and the labour costs of the photography. Second, the step-and-repeat preparation of the multiple images can readily be done on the computer, rather than by an additional step in the photography. In addition, having the mask on the computer allows one to modify the patterns at minimal cost.

After completion of the layout of the mixed gas stage, the depths of the channels in the different parts of the heat exchanger were calculated using the enthalpy and isobar results calculated earlier in determining the phase diagram of the mixture. The enhanced cooling power of the first stage allows one to increase the capacity of the hydrogen stage by a factor of three and this in turn allows one to increase the capacity of the helium stage. The cooling capacity of the 10 K stage is now estimated to be 30 mW, appreciably greater than the 10 mW originally calculated using argon. Uncertainty in this number remains, however, due to uncertainty in the efficiency of the final stage heat exchanger due to problems which may be encountered there in building a sufficiently thin interlayer.

As reported earlier we have encountered serious problems in fabricating the hydrogen stage of this cooler. The thinner glass substates required for this cooler, together with the larger area and multiple layers of the refrigerator, have demanded performance of the technolgy which is beyond the present state of the art. This has shown up as unsatisfactory bonds between layers, air bubbles in the layers, collapsed channels or deformed portions of the refrigerator. Part of this was found to be due to problems in laying down and controlling the thickness of the adhesive bonding layer. This layer is presently screen printed. In this process it is difficult to control the thickness of the layer to more than $\pm$ 4 microns. In addition, this process gives a relatively thick adhesive layer. This thick layer can extrude into the fine channels of the refrigerator resulting in blockage or reduced flow of the gas. If one attempts to process the refrigerator at higher temperatures to obtain better bonds, the channel walls can soften too much and collapse or run together. These are the problems which have plagued us. We have addressed them as follows.

First, we have redesigned the capillary sections of the refrigerators to provide more land between the channels and have thereby strengthened the channel walls. Once the pattern had been entered on the computer, the redesign was made easier by the use of the CAD system.

Second, we have set up, a magnetron-enhanced sputtering system to allow us to sputter a layer of adhesive on the glass substrates with a much better defined thickness than can be laid down using the older screen printing method. The vacuum system, r.f. Power Supply, sputtering gun and target has been acquired. Low power tests have been made with the system, and thin films have been obtained. Work is now proceeding on determining the firing conditions for obtaining suitable bonds with these films.

Third, in addition to the work with sputtered films, we are working with screen printed films but using lower temperature firing conditions with much higher clamping pressures. Encouraging results have been obtained under these conditions giving very well defined channels with no evidence of deformation of the channel walls. We expect to have solved these problems in the next couple of weeks.

2.0 Redesign of H_2 Refrigerator Test Stage

The H_2 test stage refrigerator has been redesigned to double the width of the "land" between the channels in the capillary section of the refrigerator to strengthen the channel walls during the subsequent firing process. This is where the above problems had been found to occur. In the earlier design there was a 1:1 ratio between the land width and the channel width. In the new design shown in Figure 1 this ratio has been increased to 2:1. To compensate for the change in length of the capillary section, the channels are made less deep. Refrigerators using this design are now being fabricated. This refrigerator allows one to check the performance of the low temperature heat exchangers and to make modifications of the heat exchanger interlayers relatively easily.

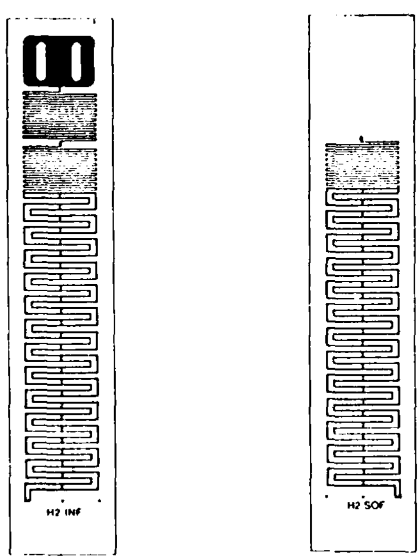


Figure 1. Redesigned in-flow and short out-flow channels for Hydrogen Heat Exchanger.

3.0 Redesign of Mixed Gas Heat Exchanger

The larger Joule-Thomson effect of the hydrocarbon-nitrogen gas mixture allows one to reduce the area of the heat exchanger for the first stage of the three stage refrigerator. This also allows one to increase the flow through the hydrogen stage. The exchanger was laid out on the CAD system , laser printed and photo reduced. The layout of the exchanger is shown in Figure 2. The etch depths were then calculated based on the measured lengths of each of the three sections of the exchanger and capillary sections. These are given in Table I.

Channel	Length	Width	Type
In-flow	27cm	0.0087cm	Double
1st Capillary	8.9cm	0.0087cm	Single
2nd Capillary	15.1cm	0.0087cm	Single

Table I

4.0 Refrigerator Fabrication

As reported earlier we had run in to problems in sealing the multi-layers of the refrigerator together. Air bubbles between the layers were found or if the temperature of the furnace was raised high enough to ensure a good bond between layers, inevitably it resulted in the collapse of the walls of the finer capillaries in the hydrogen portion of the refrigerator or the flooding of the channels with solder glass. The overall yield fell to an unacceptably low level. Part of the difficulty was attributed to the relatively thick layer of solder glass used to bond the different layers of the refrigerator together. The decision was made to explore the possibility of sputtering this film in place allowing a much thinner film to be used. Preliminary results using this technique are now being obtained.

4.1 Magnetron-Enhanced Sputtering System

During the past two months we have assembled and set up a diffusion pumped, vacuum system and magnetron-enhanced sputtering system. Working in collaboration with a group at Stanford University we have re-conditioned an NRC Model 3114 Vacuum Coater, replaced the

a.

b.

Figure 2. Redesigned Mixed Gas Heat Exchanger showing:
a. Inflow Channels
b. Short Outflow Channels

mechanical pump and re-built the vacuum chamber. It has been cleaned and tested. A 2" US' Planar Magnetron sputtering Gun was installed in the vacuum system. This was connected to a Plasma -Therm Inc., 1.5 KWatt, 13.56 MHz r.f. generator with automatic matching network which was brought back in to operation and installed. A Matheson Model 8160 Mass Flowmeter was acquired and set up to monitor the inflow of the sputtering gas. A Sloan Quartz Crystal Deposition Controller was set up to measure the deposited film thickness. A substrate holder was designed and built to hold the glass slides which are to be coated. Glass targets have been made and fabricated into 2.0" diameter disks, which were ground and polished.

The system was completed and tested using a low melting point glass as the target. The first glass disks, which were 3/8" thick were used as received. At 200 Watts input power, local heating by the discharge at the center of the disk caused the first disks to shatter. The remaining disks were then sliced into 1/8" thick pieces, which were ground and polished. Preliminary results have been obtained running the r.f. power supply at low power (50 Watts). A good deposit was obtained of the low melting point glass on the glass substrate. This is where we stand today. We have not yet determined the furnace conditions for firing these films. This we are about to do.

4.2 Screen Printed Films

In parallel with the work on the sputtered films we have also gone back to the screen printed films in an attempt to refine that technology, to allow the thinner glass substrates which are needed for this next generation of refrigerators, to be bonded using the well established techniques used on the heavier refrigerators. To do this we have up-graded the cleanliness of our clean room to eliminate all traces of particulate matter which could impair the integrity of the bonds. We have switched to a lower melting point solder glass and have developed an improved clamping system to provide a stronger clamping force to the glass layers during the fusing cycle. Preliminary results have given good bonds with clear crisp edges at the etched surfaces, showing that no softening of the channel walls has occurred. The process is being refined and we are hopeful that a dramatic improvement in yield will result from these changes.

5.0 Gas Cleaner

As mentioned in our earlier reports it is essential to reduce the level of the condensible impurities in the hydrogen and helium gas circuits to a level of a lew parts per billion if continuous cooling is to be obtained at 20K or 10K. This is difficult to do with a Zeolite absorber at ambient temperatures, however, it is possible to do so with an absorber which reacts irreversibly with the impurity gases which may be present. We have now acquired such a filter and will be using it for tests on the hydrogen stage of the refrigerator. Based on the performance we obtain with this we plan to fabricate a suitable filter for incorporation in the closed cycle system.

6.0 Personnel

The following personnel have been involved in the program:

Redesign of Refrigerator

W. A. Little

Refrigerator Fabrication/Testing

F. Tochez
C. Fuentes
M. Dubois

Fabrication Improvements

W. Shubin

Development of Sputtering System

W. A. Little
W. Shubin

Respectfully submitted,

W. A. Little
President

END
12-87
DTIC