# Compatibility Test and Residual Gas Analysis on 4130 Steel and Methanol Thermosyphons

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Testing has been carried out to determine the material compatibility of methanol and 4130 steel. This fluid/material combination is of interest for rotating heat-pipe applications associated with cooling of electrical generators. Thermal soaking at 150°C of four methanol thermosyphons made of type 4130 steel was accomplished. Two of the thermosyphons have nickel-brazed joints and two have laser-welded joints. The temperature profiles indicated the generation of noncondensable gases (NCGs) in all four thermosyphons for a thermal soak test time exceeding 14,000 h. The analysis showed that the NCG-generation rate accelerated up to 7.4  $\mu$ l/h initially and then dropped to a nearly constant rate of 2.5  $\mu$ l/h. Three NCG-generating regions were identified: accelerating, decelerating, and stable. The qualitative residual gas analysis using a mass spectrometer after 4000, 8700, and 14,000 h of thermal soak indicated the existence of the NCGs: hydrogen, formaldehyde, methane, and carbon monoxide. The corrosion and oxidation theory-based passivating film growth model and methanol catalytic decomposition model were qualitatively supported by the decelerating and stable regions defined in this experiment. It was also shown that burping is not a viable option to prolong the contaminated heat-pipe's life. Data obtained will provide important information for determination of the life and reliability of a rotating methanol heat pipe made of 4130 steel, and also, for the evaluation of the assembly processes used for fabricating rotating heat pipes.

#### **Nomenclature**

$P1E1, \ldots, P1F2$	=	thermocouple position $E1, \ldots, F2$ of
		pipe 1 (thermosyphon 1), etc.
$Q_{ m in}$	=	heat input to thermosyphons
$Q_{ m out}$	=	heat rejected from thermosyphons
q		gas-generating rate, μl/h
$q_{ m uncertainty}$	=	uncertainty in gas-generating rate,
		$\mu$ L/h
T	=	temperature, °C
t	=	time, h
$t_{\text{temp\_fall}}$	=	time at which thermocouple
-		temperature begins to fall, h
$t_{ m uncertainty}$	=	uncertainty in time, h
V	=	total volume of generated
		noncondensable gas, ml
$V_c$	=	internal volume of condenser, ml
$V_f$	=	internal volume of fill tube, ml
$\Delta t$	=	time interval, h
$\Delta V$	=	internal volume of thermosyphons

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between adjacent thermocouples, ml

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Subscripts							
$C1, \ldots, C6$	= thermocouple position on condenser						
E1, E2	= thermocouple position on evaporator						
F1. F2	= thermocouple position on fill tube						

## Introduction

IGH-POWER density rotating machines, such as the switched reluctance and permanent magnet types used in advanced integrated power units, and starter—generators being developed for the more electric aircraft, generally operate at elevated temperatures as a result of their inherent heat generation. Effective cooling is crucial to increase the reliability and life of these rotating machines, and also, to reduce maintenance cost. A high-speed rotating heat pipe has been proposed for cooling applications in advanced electrical machines, where the shaft of the rotating machine is designed to function as a heat transport device.<sup>1</sup>

Commonly used materials for electrical motor and generator shafts are carbon or alloy steels, and commonly used nontoxic heat-pipe working fluids are water and methanol. For the specific fabrication of shaft heat pipes in an alternator cooling application here, a magnetic material 4130 carbon steel was selected as the rotor shaft material, and methanol was chosen as the working fluid. Material compatibility data between the shaft and the working fluid are required for a proper design of the shaft heat pipe and its incorporation into the rotating machine. Any noncondensable gas (NCG) generated inside the shaft heat pipe affects the heat transport capability and the operating life of the heat pipe.

Some heat-pipe research literature has shown that water and stainless steel may generate hydrogen, whereas methanol and stainless steel are compatible.<sup>2-4</sup> However, other investigations have reported that NCG is generated for the methanol and stainless-steel combination.<sup>5-7</sup> Anderson et al.<sup>5</sup> carried out an experimental and theoretical study on the gas generation in 16 methanol/304 stainless-steel heat pipes for the purpose of es-

tablishing a scaling law for the accelerated testing of such heat pipes at higher temperatures. It was stated that this combination was compatible up to 63°C, with gas generation observed at higher temperatures. An NCG accumulation equation based on the ideal gas law for the working fluid vapor was proposed to calculate the NCG evolution. It was observed that the gas evolution in methanol/stainless-steel heat pipes depended on the amount of gas generated per unit area. Two models of gas generation were developed based on the corrosion and oxidation theory with parameters determined by an experiment. The model of passivating film growth with a parabolic time dependence from the corrosion of stainless steel in methanol was presented for the initial exposure. Beyond a critical time, the gas-evolution model was the catalytic decomposition of methanol with a linear time dependence to formaldehyde and hydrogen on the surface of the film, which is the corrosion product from the initital exposure.

Kaufman and Tower,<sup>6</sup> after evaluating 30 commercially available heat pipes in 10 groups of different design and material combinations through 11,000 h of testing, found that for all of the combinations of methanol and stainless steel, ammonia and aluminum, and ammonia and stainless steel, considerable NCG was present in some groups, whereas other groups showed no gas, depending on the processing schedules. It was concluded that the high-temperature vacuum baking and cleaning of the heat pipes reduced gas generation. Reyes et al.<sup>7</sup> continued the life test on the same heat pipes by Kaufman and Tower to 70,000 h. Their findings were similar to those of Kaufman and Tower.

Munzel and Savage<sup>8</sup> carried out life tests over a 3-year period on heat pipes with various combinations of aluminum 6061, stainless-steel 304, and unhydrous ammonia. It was found that the combination of aluminum 6061 as the wall material, stainless-steel 304 as the capillary structure, and unhydrous ammonia as the working fluid is not compatible with long life requirements. The mechanism of the gas generation was considered to be the electrolytic action between the two dissimilar metals. The two combinations, stainless steel and ammonia, and aluminum and ammonia, were found to show long-term compatibility. Eninger et al.,9 after an eight-monthlong life test on 37 heat pipes from the combination of ammonia, stainless steel, and aluminum, drew a similar conclusion. These compatibility tests showed that the improved manufacturing techniques, such as baking out the heat pipes in vacuum at high temperatures and using proper cleaning procedures, would slow down gas generation.

Although there are a vast number of studies on the compatibility of stainless steel and methanol, no data were found on this specific 4130 carbon steel. Furthermore, the available test results were closely tied with the assembly procedures of the tested heat pipes, and the gas-generation mechanism is still not fully understood. To obtain reliable information on the chemical compatibility between 4130 steel and methanol, four thermosyphons made of this material combination were manufactured and subjected to continuous thermal soak tests. Residual gas analysis experiments were carried out using a mass spectrometer (MS).

# **Objectives**

The purpose of this research was to investigate the reactivity and chemical compatibility of 4130 carbon steel and methanol combination, to provide information on life and reliability of the rotor shaft heat pipes, and to provide data suggesting improvements of the fabrication and assembly process for improved performance and reliability of the heat pipes.

# **Experimental Work**

#### Thermosyphon Test Configuration

Four thermosyphons (also referred to as pipes in this paper), made of type 4130 steel with nickel-brazed joints (pipes 1 and

2) and laser-welded joints (pipes 3 and 4), and filled with methanol, were manufactured and subjected to evaporatingcondensing thermal soak cycles. A laboratory fill-station available at Wright-Patterson Air Force Base was used to perform both the final cleaning by vacuum baking of the pipes' interior and the filling with the required amount of methanol, deaerated 99.99% pure analytical grade supplied by Fisher Scientific, using a standard heat-pipe filling procedure. The filling station consists of two vacuum pumps, a graduated fill burette, control valves, a liquid reservoir, a heater for deaerating, and a nitrogen overpressure line. The final cleaning was done by pumping the pipes to 10 mtorr or better, then flowing alcohol (unhydrous isopropyl alcohol) into the pipes' interior and then draining it after a few minutes. Pipes 1 and 2 were baked at 360°C and  $0.9 \times 10^{-7}$  torr for nearly 120 h, and pipes 3 and 4 were baked at 355°C and  $1.1 \times 10^{-7}$  torr for nearly 120 h before filling. Each pipe was filled with ~15 cm<sup>3</sup> of methanol at a temperature of 20°C by a vacuum suction transfer process involving the glass burette and the methanol reservoir. This is 20% of the internal volume at 20°C and 28% at 200°C. The exact fill was determined by weighing the pipes before and after the fill process. The net methanol fills for pipes 1-4 were 12.2, 12.8, 12.4, and 12.6 g, respectively.

Figure 1 shows the thermal soak test setup. Each thermosyphon was heated by a coil heater wound around the evaporator. The heaters were regulated by relays to keep the evaporators at a constant temperature of 150°C. Heat removal was accomplished by a cold shroud surrounding the test containment chamber containing the four thermosyphons. The inlet temperature of the shroud cooling water was 20°C.

Figure 2 is the schematic diagram of the thermosyphons and the temperature measurement locations. Ten copper-constantan (type T) thermocouples with welded-bead exposed junctions were embedded and epoxied to the pipe wall. The thermocouples were used to monitor the temperature history hourly during the life test. The fill tube attached to the top of each thermosyphon collects any noncondensable gas that is generated. The collected gas can be vented through the fill valve to a residual gas analyzer (RGA). If there was any NCG generation inside the pipes, it would accumulate first in the fill tube at the top portion of the pipe that was connected to an MS for gas-component analysis. A gradual temperature drop at the fill tube of the pipe could be an indicator of NCG generation.

The RGA was used for the analysis of the constituents of the gas mixture generated in the thermosyphons. It consisted of an MS, AERO VAC 1000, by Vacuum Technology, Inc., and a turbomolecular pump in series with a mechanical pump. The MS system can be pumped to a pressure level of 10<sup>-8</sup> torr. The RGA system, including all connections, was checked for leakage using a helium leak detector. The RGA MS was calibrated with standard calibration leaks of hydrogen, argon, carbon monoxide, and methane.

The time history of the temperature profile along each pipe was used to determine the gas-generation rate. The NCG samples in pipes 1 and 3 were drawn for component analysis, whereas the NCG samples in pipes 2 and 4 were kept unvented for the purpose of evaluating the thermosyphon life.

## **MS Test Procedure**

The gas constituent analysis procedure consisted of three steps: preparation of the RGA system, preparation of the thermosyphon, and gas sampling. Immediately prior to each sampling, the RGA system was baked at 150°C for 8 h and then pumped overnight at room temperature to a pressure level of  $10^{-8}$  torr. At the same time, the heater to the thermosyphon's evaporator section was turned off, and the thermosyphon was allowed to cool to the condenser control temperature of 20°C. The primary purpose of cooling the thermosyphon and the NCG to 20°C during sampling was to reduce the pressure inside so that when the fill valve was open, there would not be any large pressure gradient between the thermosyphon and the

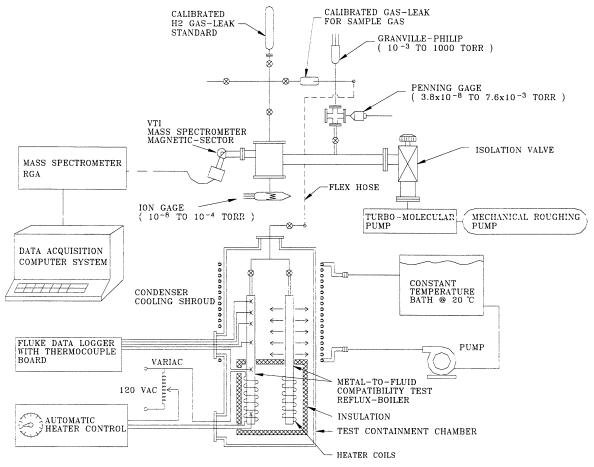


Fig. 1 Material compatibility thermal-soak test setup.

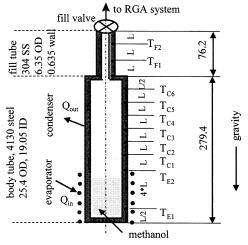


Fig. 2 Schematic diagram of thermosyphons. L = 25.4 mm.

RGA system. The second purpose was to reduce the chance and amount of methanol vapor coming out of the fill tube with the NCG during the releasing of the NCG. Because the ratio of methanol's saturation pressure to its saturation temperature decreases with the decrease of its temperature, and its vapor mass is directly proportional to this ratio, the mass of methanol vapor at a lower temperature is less than that at a higher temperature within the same volume. The next step is to introduce the accumulated gas into the RGA system through the fill valve, as shown in Fig. 2, and to analyze it. In the RGA system, there were three intermediate sections separated by valves between the thermosyphon fill valve and the MS detector chamber. These three holding sections were used to reduce the NCG gas pressure gradually before it was finally introduced

into the MS through a capillary leak. The gas sample analysis data were then recorded in a personal computer and compared with the library of standard gases.

#### **Results and Discussion**

The material compatibility life test continued for more than 14,000 h. Figures 3-6 show the temperature histories of all 10 thermocouples on the four thermosyphons, numbered P1, P2, P3, and P4. It is obvious from Figs. 3-6 that a considerable amount of NCG was accumulating in all four pipes. For pipes 1, 2 and 4, the NCG had reached the location of thermocouple C2 at ~8200, 11,100 and 9500 h, respectively. For pipes 3, before burping, it took 7600 h for NCG to reach the location of thermocouple C3; after burping, it took only 1650 h. The sharp temperature changes in pipe 1 at the time of ~8700 and 14,100 h and in pipe 3 at the time of ~9200 h were a result of the release of the NCG for residual gas analysis.

#### **NCG-Generation Rate**

Based on the temperature histories of the thermosyphons, a gas-generation rate was calculated for each pipe. The instant when the temperature began to decrease from 150°C was used as the time that the NCG had accumulated up to the position where the thermocouple was located. For the purpose of this investigation, a flat-front interface between the methanol vapor and the NCG inside the thermosyphons has been assumed. Although mixing of the methanol vapor and the NCG could affect the precise calculation of the NCG rate, it is believed by the authors that the pipe temperature profile, with a controlled saturation temperature and the fraction of methanol vapor that is mixed with the NCG remaining constant, shows that the increase in NCG is clearly demonstrated by the temperature profile. Numerical model investigations by one of the

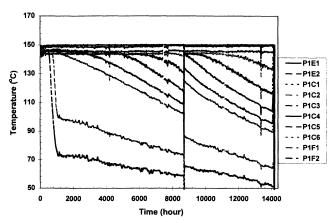


Fig. 3 Thermosyphon 1: temperature vs time.

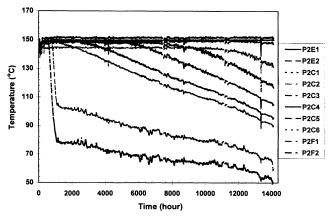


Fig. 4 Thermosyphon 2: temperature vs time.

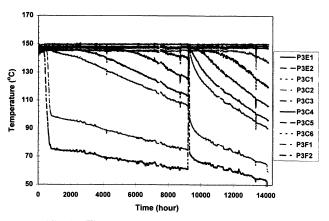


Fig. 5 Thermosyphon 3: temperature vs time.

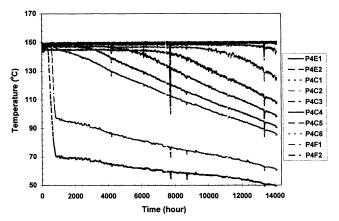


Fig. 6 Thermosyphon 4: temperature vs time.

authors for the temperature profiles with an assumed flat-front interface supported this assumption. Unfortunately, it was more difficult to observe the temperature change farther down the pipe near the assumed front (going from the near saturation temperature to a lower temperature). This could have affected the growth term; however, the change was observed with the same level of difficulty at all points.

The total volume of the NCG generated was calculated as  $V = V_f + V_c$ , where  $V_f$  and  $V_c$  are the volumes of fill tube and the condenser, respectively, occupied by the NCG. The gasgenerating rate was calculated as  $q = \Delta V/\Delta t$ , where  $\Delta V$  is the internal volume in the thermosyphons between two adjacent thermocouples and  $\Delta t$  is the corresponding elapsed time interval marking the onset of temperature decreases from 150°C. The results are shown in Table 1 and are plotted in Fig. 7.

It is obvious from Fig. 7 that the gas-generating rate can be divided into three regions: accelerating, decelerating, and stable. In the first region, the gas evolution was accelerating until it reached a maximum, then the second region began, decelerating until stabilizing at a constant rate. The maximum rate corresponded to the time when the fill tube was totally filled with the NCG.

The NCG in pipe 3 was accidentally released into the atmosphere at ~9200 h, when an MS sampling was attempted. It took much less time for the NCG to generate in pipe 3 to block the fill tube after first burping, as shown in Fig. 7; plot pipe 3, which was the initial gas-generating rate, and plot pipe 3\_A, which was the gas-generating rate after the burp (Fig. 7). The cause for the extremely high NCG generation rate after complete NCG venting is not clear to the authors. What can be deduced from these experimental results is that venting of NCG does not seem to be a feasible means to prolong the life of the contaminated thermosyphon. All four pipes showed a similar trend in the gas-generating rate, except that pipe 1 had a higher stabilized gas-generating rate than the other three pipes. This may be caused by the partial burping of the NCG in the pipe at about 4000 h. It was observed in pipe 3 that after burping, the gas generation was much faster than the initial period.

Another observation from the gas-generation rate was that pipes 3 and 4 had a faster initial gas-generating rate than pipes 1 and 2. The NCG in pipes 3 and 4 blocked thermocouple F2 more than 200 h earlier than in pipes 1 and 2. But the settling gas-generating rates in pipes 2-4 are nearly the same. Pipe 1 was excluded from this observation because of its early partial burping. The difference between the two pipe groups is that the joints in pipes 1 and 2 are nickel brazed, whereas the joints in pipes 3 and 4 are laser welded. However, currently, there is not enough information to conclude that it was the joints that made the difference. A further investigation of the assembly and cleaning processes used in the four pipes is necessary.

#### Residual Gas Analysis with MS

To obtain the constituents and the percentage of each constituent in the gas mixture generated in the material compatibility test, an MS was incorporated into the compatibility test setup, as shown in Fig. 1. To use the MS for the quantitative gas analysis, knowledge of what components exist in the gas mixture is required, which is the objective of this research. Also, the spectrum and relative sensitivity of each component in the specific MS system are necessary for the quantitative analysis of the NCG.<sup>10</sup>

The spectra library and relative sensitivity for common gases provided by the MS manufacturer was used for the gas component identification.<sup>11</sup> Because the spectra library was obtained in a different MS system, four standard leaks, H<sub>2</sub>, CO, Ar, and CH<sub>4</sub>, were used to calibrate the present MS system and verify the validation of the spectra library. Table 2 shows the calibration results. The reading at mass 1 (atomic mass unit) was not recorded because the MS can only detect the ion

Table 1 NCG generation rate

Th	Tab				***************************************
Thermocouple position	$t_{\text{temp}_{-}\text{fall}}$ , h	Interval	$\Delta V$ , ml	$\Delta t$ , h	$q, \mu l/h$
	F	Pipe 1	·		
Start	0				
F2	505	F2-F1	0.515	215	2.394
F1	720	F1-C6	1.613	340	4.744
C6	1060	C6-C5	7.240	1100	6.581
C5	2160	C5-C4	7.240	1610	4.497
C4	3770	C4-C3	7.240	2380	3.042
C3	6150	C3-C2	7.240	2070	3.497
C2	8220	C2-C1			
C1		Total/average	31.086	7715	4.029
		Pipe 2			
Start	0	-			
F2	600	F2-F1	0.515	190	2.710
F1	790	F1-C6	1.613	320	5.040
C6	1110	C6-C5	7.240	1390	5.208
C5	2500	C5-C4	7.240	1990	3.638
C4	4490	C4-C3	7.240	2910	2.488
C3	7400	C3-C2	7.240	3700	1.957
C2	11,100	C2-C1			
C1		Total/average	31.086	10500	2.961
		Pipe 3			
Start	0				
F2	330	F2-F1	0.515	190	2.710
F1	520	F1-C6	1.613	230	7.012
C6	750	C6-C5	7.240	1900	3.810
C5	2650	C5-C4	7.240	2090	3.464
C4	4740	C4-C3	7.240	2910	2.488
C3 .	7650	C3-C2			
C2		C2-C1			
C1		Total/average	23.846	7320	3.258
		Pipe 3_A			
Start	9200				
F2	9223	F2-F1	0.515	22	23.401
F1	9245	F1-C6	1.613	40	40.320
C6	9285	C6-C5	7.240	25	289.583
C5	9310	C5-C4	7.240	446	16.232
C4	9756	C4-C3	7.240	1094	6.618
C3	10,850	C3-C2	7.240	2095	3.456
C2 C1	12,945	C2-C1 Total/average	31.086	3722	8.352
		_	21,000	5,22	3,002
	_	Pipe 4			
Start	0		0.515	100	2.661
F2	360	F2-F1	0.515	192	2.681
F1	552	F1-C6	1.613	218	7.398
C6	770	C6-C5	7.240	1440	5.027
C5	2210	C5-C4	7.240	1570	4.611
C4	3780	C4-C3	7.240	2820	2.567
C3	6600	C3-C2	7.240	2950	2.454
C2 C1	9550	C2-C1 Total/average	31.086	9190	3.383
<u> </u>		10tat/average	31.000	7170	J.J0J

current at a mass higher than 1.2. The limited number of standard leak calibrations on the RGA showed that the spectra library could be used for this MS system with known uncertainty, at least for qualitative analysis.

The gas generated in pipe 1 was sampled three times. The first sampling was carried out after 4000 h of thermal soak, the second sampling was made after 8700 h of life test, and the third sampling was performed after 14,100 h of life test. Before each sampling, the MS system was baked for 8 h at about 150°C, and a background sampling was taken. Figure 8a shows the background before the first time NCG sampling, and Fig. 8b shows the first time sampling result. Figures 8c and 8d show the second time sampling output.

Utilizing the spectra from both the calibration and the spectra library for the residual gas analysis on the data shown

in Fig. 8c, it was found that hydrogen, methanol, water, formaldehyde, methane, and carbon monoxide exist in the gas generated in pipe 1. The third time sampling, an instant analysis performed after 14,100-h life test, showed that hydrogen, carbon monoxide, methane, formaldehyde, and oxygen were the first five major components in the NCG from pipe 1, as shown in Fig. 9. This instant analysis used a matrix inverse technique for mass spectrum in the range of 2–45 amu and a relative sensitivity database with respect to nitrogen covering gases of hydrogen, helium, methane, water, neon nitrogen, oxygen, carbon monoxide, pump oil, argon, carbon dioxide, and formaldehyde. As a result from both analyzing methods, it can be concluded that hydrogen, carbon monoxide, methane, and formaldehyde were generated as NCG in pipe 1.

Table 2 Comparison of standard leaks spectra to library spectra

Mass, amu	Library	Measured
	Methane	
16	100	100
15	86	85.3
14	16	8.8
13	8	3.8
1	4	
12	3	1.2
	CO	
28	100	100
12	5	3.1
16	2	0.6
29	1	0.8
14	1	1.6
	Argon	
40	100	100
20	15	17.8
	Hydrogen	
2	100	100
1	2	

<sup>a</sup>Relative ion current peak. The largest peak height is arbitrarily given the value of 100.

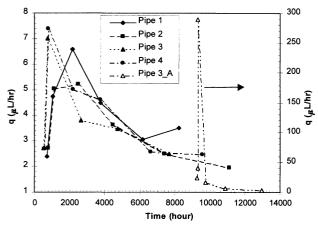


Fig. 7 NCG-generation rate.

#### **Uncertainty Analysis**

The NCG-generating rate was calculated based on the assumptions that the interface between the NCG and the methanol vapor was planar, and the time when a thermocouple's reading began to fall was taken as the time when the NCG reached the location of that thermocouple along the thermosyphons. The reality is that the temperature drop may not be the exact indicator of the interface between the methanol vapor and the NCG. Unlike the sharp temperature drops indicated by  $T_{F1}$  and  $T_{F2}$  in the fill tube, the condenser temperature exhibited a transition period between the beginning of temperature decreases and the linear temperature drop region. The interface between the methanol vapor and the NCG was not clear during this period of time. The uncertainty of the time was about ±100 h. Converting the time uncertainty to the NCG-generating rate by equation  $q_{\text{uncertainty}}/q = t_{\text{uncertainty}}/t$ , the maximum uncertainty for q was 7% and the uncertainty for the average gas-generating rate was about 1.5%.

For the residual gas analysis, in addition to the uncertainty caused by the use of the spectra library, the assumption of the gas constituents introduced another source of uncertainty. However, an exclusion method was used in this analysis for all possible gas candidates (101 gases), at each principle

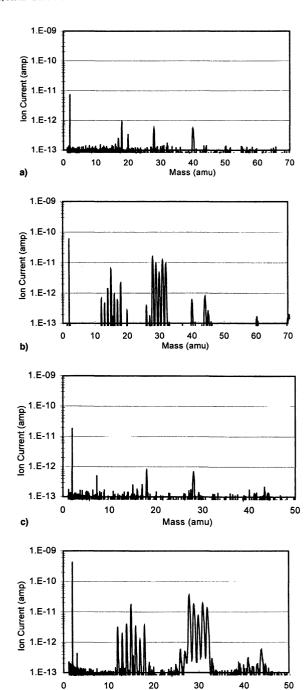


Fig. 8 MS analysis results on gas generated in thermosyphon 1: a) RGA background sampling at 4000 h, b) NCG sampling in thermosyphon 1 at 4000 h, c) RGA background sampling at 8700 h, and d) NCG sampling in thermosyphon 1 at 8700 h.

Mass (amu)

d)

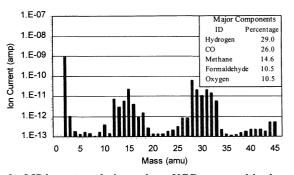


Fig. 9  $\,$  MS instant analysis result on NCG generated in thermosyphon 1 after 14,000 h.

atomic mass. In addition, an instant analysis was performed based on a matrix inverse technique and the relative sensitivity of 20 common gases with respect to nitrogen. The results of both methods supported each other, except for water and oxygen.

The current design of the sample gas inlet system made the release of the NCG from the thermosyphons into the MS vacuum system different from the standard leaks that are crimped capillary restrictions. Fractionation (change in composition of a sample) may have occurred during the NCG sampling. Because the ion current is proportional to the partial pressure of the gas constituent, additional uncertainties for the RGA arise as a result of the pressure reduction during the sampling and the pressure difference between the background sampling and the NCG sampling processes. The pressure variation during sampling in this RGA was the result of the current design of the MS inlet system.

#### Discussion

After the analysis of the gas-generating rate and the gas component analysis, the next logical step was to find the mechanism of the formation of the NCG. The NCG could have been generated either by the chemical reaction between the 4130 steel and methanol, or could be a result of any contamination that occurred during the filling and sealing of the thermosyphons, or both.

Two models of methanol/stainless-steel gas generation were developed by Anderson et al.,<sup>5</sup> based on the corrosion and oxidation theories. The models assumed that at elevated temperatures, uniform corrosion occurred at all stainless-steel surfaces by the operation of a great number of microscopic galvanic cells. In initial exposure, a passivating film would grow with a parabolic time dependence from the corrosion of the stainless-steel surface in the presence of methanol accompanied by the evolution of a proportional amount of NCG. Once the film was formed, the corrosion product may then act as a catalyst to enact the dehydrogenation of methanol to form formaldehyde gas and hydrogen gas, giving a linear time dependence. If oxygen was present, formaldehyde and water would be yielded from the reaction of methanol and oxygen, and oxidation of formaldehyde would produce formic acid. The corrosion rate of stainless steel in formaldehyde is slightly greater than in methanol, but the rate increases by more than an order of magnitude in the presence of formic acid.5

The gas-generation rate found in this study supported qualitatively these two models with respect to the time dependence if the initial accelerating region was neglected. The decelerating region fits to the film growth model, whereas the stable region fits to the model of catalytic decomposition of methanol. The MS analysis result on the gas sample from pipe 1, the existence of hydrogen and formaldehyde, also supported these models indirectly, although carbon monoxide and methane were also detected. The mechanism for the initial accelerating region of gas generation is uncertain at this point.

The detection of water in the sample indicated the possibility of contamination of the thermosyphons. The contaminants could have been introduced during methanol filling, the cleaning process, or the welding or brazing. Further evaluation of the assembly processes used for the thermosyphons may be helpful to identify the source.

#### **Summary**

Four 4130 steel and methanol thermosyphons were fabricated and their compatibility life test experimental setup was commissioned. A high vacuum MS-based RGA was success-

fully established. The life testing continued for more than 14,000 h. The residual gas analysis indicated the existence of hydrogen, formaldehyde, methane, and carbon monoxide. Based upon the temperature histories of the thermosyphons, an NCG-generation rate was calculated. The NCG-generation rate for the 4130 steel and methanol combination, varying from 2.5 to 7.4  $\mu$ l/h, was divided into three regions, acceleration, deceleration, and stabilized regions. The second and third regions fit the passivating film growth and methanol catalytic decomposition models based on the oxidation theory. Some major conclusions are as follows:

- 1) It was shown that venting of NCG does not significantly prolong the contaminated thermosyphons' life.
- 2) Data have been collected that impact the assessment of the service life of shaft rotating heat pipes used for cooling electrical machines.
- 3) Further studies are needed on the mechanism of the NCG generation. This is especially necessary in the initial acceleration region, along with an explanation of the difference in gas generation between the two thermosyphon groups with different joints and the large difference in gas-generation rate before and after complete burping. Both the oxidation and corrosion theories and the fabrication process are proper study directions. Detailed data reduction using the film growth model and the catalytic decomposition model and derivation of parameters for these two models based on this experimental result may provide a practical means to quantitatively predict the service life of the shaft rotating heat pipes.

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# References

<sup>1</sup>Ponnappan, R., Leland, J. E., and Beam, J. E., "Thermal Management Issues of Rotors in Rotating Electrical Machines," Society of Automotive Engineers, Paper 942184, Oct. 1994.

<sup>2</sup>Heat Pipe Design Handbook, Vol. 1, Rept. N81-70112, B&K Engineering, Inc., Towson, MD, June 1979.

<sup>3</sup>Dunn, P. D., and Reay, D. A., *Heat Pipes*, 4th ed., Pergamon, New York, 1994.

<sup>4</sup>Chi, S. W., *Heat Pipe Theory and Practice—A Sourcebook*, Hemisphere, New York, 1976.

<sup>5</sup>Anderson, W. T., Edwards, D. K., Eninger, J. E., and Marcus, B. D., "Variable Conductance Heat Pipe Technology—Final Research Report," NASA CR-114750 (N74-21589), March 1974.

<sup>6</sup>Kaufman, W. B., and Tower, L. K., "Evaluation of Commercially Available Spacecraft-Type Heat Pipes," A Collection of Technical Papers, 3rd International Heat Pipe Conference, AIAA, New York, 1978, pp. 88–95.

<sup>7</sup>Reyes, A. S., Brown, J. R., Chang, W. S., and Ponnappan, R., "Gas Generation Test Data and Life Tests of Low Temperature Heat Pipes," AIAA Paper 90-1756, June 1990.

<sup>8</sup>Munzel, W. D., and Savage, C. J., "Lifetests of Artery Heat Pipes for Low Temperature Range," *Proceedings of the 2nd International Heat Pipe Conference* (Bologna, Italy), ESTEC, Noordwijk, The Netherlands, 1976, pp. 383–392.

<sup>9</sup>Eninger, J. E., Fleischman, G. L., and Luedke, E. E., "Heat Pipe Materials Compatibility," NASA CR-135069 (N77-12182), Jan. 1976.

<sup>10</sup>Willard, H. H., Merritt, L. L., Dean, J. A., and Settle, "*Instru-mental Methods of Analysis*," 6th ed., D. Van Nostrand Co., New York, 1981, pp. 592, 593.

<sup>11</sup>AERO VAC 1000 Instruction Manual, Version 2.6, Vacuum Technology, Inc., Oak Ridge, TN, April 1992.