

MEASUREMENTS OF MICRO-DEBRIS DEPOSITS USING A QUARTZ CRYSTAL MICROBALANCE

Don Evans*, Aaron Wilkosz, Milt Triplett, and Andy Hollerman
Nichols Research Corporation
4040 South Memorial Parkway
Huntsville, Alabama 35802

ABSTRACT: The Nichols Research Corporation (NRC) recently completed a series of x-ray tests at Blackjack 5 (BJ5) in San Diego, California. The tests consisted of placing selected optics samples inside BJ5, conductively cooling them to about 89 K, and exposing them to selected x-ray fluence levels. A series of measurements were made using a quartz crystal microbalance (QCM) to determine the accumulation rate of surface debris in the BJ5 environment under conditions of poor vacuum, multiple contamination sources, and prolonged cryogenic cooling. The results of these tests are discussed in this paper.

INTRODUCTION

Nichols Research Corporation (NRC) completed a test series using the Blackjack 5 (BJ5) soft x-ray (SXR) source at Maxwell Laboratories in San Diego, California in December, 1989. This test consisted of placing selected optics samples inside a BJ5, cooling them to cryogenic temperatures, and exposing them to x-rays. The optics samples had to remain clean during the entire experimental sequence, which involved assembling the samples in a clean room and mounting them inside a BJ5.

Unfortunately, some mass build-up occurred due to cryogenic cooling and the presence of debris sources inside the BJ5. As a result, NRC performed a series of tests using a quartz crystal microbalance (QCM) to determine the mass of sample surface deposits accumulated in the BJ5 environment. The results of those tests along with the results of a series of tests completed at NRC simulating the vacuum and environmental conditions of the BJ5 are the subject of this paper.

MATERIALS AND METHODS

When samples were cooled to 123 K, the entire experimental assembly, and especially assembly parts linked directly to the path of the liquid nitrogen flow, showed traces of cryogenic deposits. A QCM Research model MK9 quartz crystal microbalance was used to determine the magnitude of the cryogenic deposits. The MK-9 sensor, with the associated low temperature oscillating chip, was used with the QCM Research model 1900 controller to monitor sensor output.

A QCM operates using two vibrating quartz crystals. One of the crystals collects contaminants, while the other functions as a standard. The change in frequency between the two crystals corresponds to the amount of mass deposited. For the 10 MHz crystals used by NRC, a frequency change of 7.14 Hz indicated a mass deposit of 1×10^{-8} g. The frequency range for the exposed crystal can vary from 10 MHz to 15 MHz, and the accuracy in digital mass readout is 1×10^{-8} g. QCM crystals can collect mass deposits composed of almost any substance, when exposed to an unknown environment. In order to determine the debris composition, the vacuum environment must be examined for all possible contamination sources.

* Present address: Jaycor, Incorporated, 4950 Corporate Drive NW, Huntsville, Alabama 35806.

Several equations were used to calculate the results discussed in subsequent sections. The QCM controller has the capability to display total mass, mass accumulation rate, temperature, and frequency. Only mass accumulation rate, temperature, and frequency were recorded using the QCM. Recording total mass was not very practical, because the QCM can record total mass only over a limited range. A more effective method of determining the total deposited mass is the use of the following equation:

$$M_{\text{QCM}} = \left[\sum_{i=1}^N m_i \Delta t_i \right] \cdot 10^{-10}$$

where M_{QCM} equals the total accumulated mass (g), m_i equals the mass accumulation rate (g/s), Δt_i equals the time increment (s), and N equals the number of measured data points. The assumption is made that the rate of mass deposition varies linearly with time. Therefore, instead of integrating the mass accumulation rate for each individual time interval, a simple summation of the product of the mass rate and sample interval yields the total accumulated mass.

The areal density for the mass deposit was calculated as:

$$\mu = \frac{M_{\text{QCM}}}{A_{\text{QCM}}}$$

where μ equals the areal density (g/cm²), M_{QCM} equals the total accumulated mass (g), and A_{QCM} equals the surface area of the QCM sensor, 0.3167 cm².

The thickness for the mass deposits was calculated as:

$$s = \frac{\mu}{\rho_t}$$

where s equals the deposit thickness (cm), and ρ_t equals the total deposit density (g/cm³). The assumption is made that the mass deposit consists of a single constituent and that the mass distribution is equal over the entire QCM surface. For the temperature and pressure ranges considered at the NRC and for the BJ5 vacuum systems, water was the compound most likely to condense. Materials, such as diffusion pump oil, alcohol, machine oil, kimfol, paralyene-C, kapton, and other hydrocarbons, can be found in the BJ5 vacuum system in amounts up to a few percent by mass.

NRC QCM TEST RESULTS

Several different approaches were used at NRC to model debris deposition in the BJ5. The initial goal was to show that mass build-up is possible in a working vacuum system by establishing a lower and an upper limit on the total mass deposited at NRC and then extrapolating the values relevant to the BJ5. Only the definition of a lower and upper limit was possible, because of limitations imposed by the NRC vacuum system. The maximum BJ5 operating pressure, 2.5×10^{-4} torr, is reached using mechanical roughing and diffusion pumps. The NRC vacuum system uses a combination of mechanical roughing and turbo molecular pumps, which produce a maximum vacuum pressure of approximately 1×10^{-7} torr. The NRC vacuum chamber is also several hundred times smaller than either the super-dome or mini-dome vacuum chambers used in the BJ5. Finally, the BJ5 vacuum chamber had a continuous internal source of debris. Therefore, the exact experimental conditions in the BJ5 could not be duplicated at NRC. NRC implemented a three phase series

Table 1. NRC QCM test results.

NUMBER	TEST TIME (min)	TEST MASS (μg)	TOTAL DENSITY ($\mu\text{g}/\text{cm}^2$)	AREAL TEMP (K)	FINAL PRESSURE ($\times 10^{-4}$ torr)	TEST SET-UP
7	36	-	-	93.3	450.	CT
1	36	-	-	296.0	0.014	SH
2	97	2.99	13.18	206.3	503.	SH
3	12	1.63	5.15	227.0	0.022	SH
4	66	3.56	11.24	150.9	0.009	SH
5	75	6.41	20.24	99.2	0.009	SH
6	75	5.81	18.35	115.0	449.	SH
8	86	6.82	21.53	100.7	0.007	SH
9	57	5.44	17.18	103.6	0.007	SH
10	70	6.67	21.06	109.9	409.	SH
11	65	6.07	19.17	110.0	356.	SH
12	42	0.65	2.05	293.2	18.1	SH
13	51	5.31	16.77	139.3	15.3	SH
14	91	8.69	27.44	93.0	11.3	SH
AVE SH	61	5.00	16.11	129.0	157.3	-
15	45	16.43	51.88	90.2	0.015	SE
16	45	10.03	31.67	98.6	353.	SE
17	47	8.39	26.49	102.0	50.	SE
18	70	12.03	37.99	105.2	226.	SE
19	26	4.80	15.16	89.6	0.015	SE
AVE SE	47	10.34	32.64	97.1	125.8	-

CT = Cooling test.

SE = Sensor exposed.

SH = Sample holder.

of QCM tests to study the cryogenic build-up and micro-debris accumulation problem.

Phase One. The first testing phase was designed to determine the cleanliness of the NRC vacuum system. The QCM sensor was mounted on the back surface of the holder in the position normally occupied by the sample. Then, this assembly was attached to a vacuum flange, which was mounted in the NRC vacuum chamber. Liquid nitrogen was used to cool the holder, and the chamber was evacuated to the operating turbo pump pressure of approximately 1×10^{-7} torr. The QCM was cooled to an average temperature of 100 K, and data were collected for about 45 minutes. Table 1 shows the results of the NRC QCM vacuum tests. Insignificant amounts of cryogenic and micro-debris deposits were observed on the QCM sensor.

In a second set of tests, the above procedure was repeated, except that only the mechanical roughing pump was used to reduce system pressure. No significant differences in the total accumulated debris mass resulted from this change. A larger mass accumula-

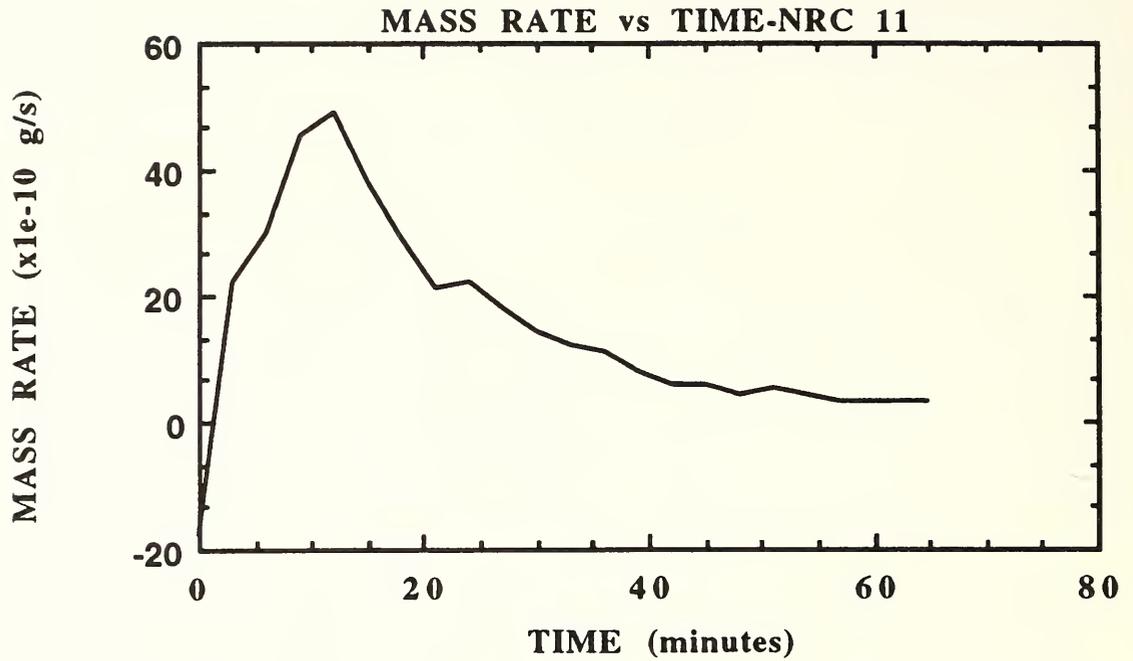


Figure 1. NRC QCM and sample holder results.

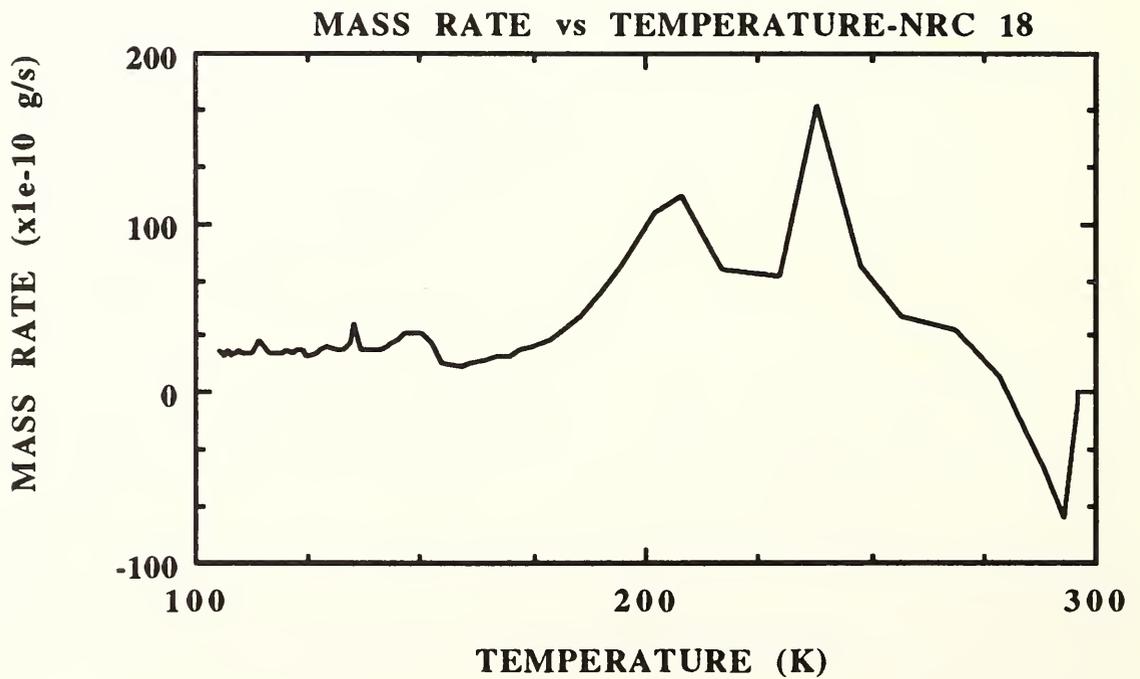


Figure 2. NRC exposed QCM results.

tion was expected for the second test series, because the working pressure was 4 to 5 orders of magnitude higher (approximately 1×10^{-2} torr). Larger amounts of mass deposition were expected, because of higher particle flux. Conversely, a smaller amount of mass deposition was expected at lower pressures, because fewer debris particles were present in the system.

Tests were also performed in which the NRC vacuum chamber was artificially contaminated with alcohol, oil, and grease. These tests showed no significant increase in total accumulated debris mass. These results suggest that the face plate of the holder effectively prevented the flux of incoming debris from reaching the QCM sensor by decreasing the exposed surface area of the sample to approximately 0.1 cm^2 . For the contaminants to reach the sensor, they had to traverse the length of the aperture, which was being cooled. The aperture acted as a cryogenic trap, significantly reducing the number of particles reaching the sample surface.

The phase one tests showed that the QCM sensor detected a total average mass accumulation of $5.0 \mu\text{g}$ (Table 1). This average mass was deposited on the surface of the QCM sensor in an average of 61 minutes. A typical deposition rate versus time plot for the QCM sensor mounted on the sample holder is seen in Figure 1. The average thickness deposited on the QCM was approximately $0.2 \mu\text{m}$.

Some unusual observations were made during the first five minutes of these tests. The readout on the QCM frequency controller intermittently disappeared and reappeared with small abnormal readings. The rapid cooling created a temperature gradient between the QCM crystals during the initial phase of each test. This gradient made the QCM electronics unstable, resulting in the display of inaccurate data. The readings ceased 5 to 7 minutes after the start of the cooling cycle. Data collected by the QCM after the instability period were considered to be accurate. Only these data were used for mass build-up calculations.

Phase Two. In the second phase of the NRC tests, the holder was removed and the stainless steel case containing the QCM was attached directly to a plate in contact with the copper cooling tubes. The goal of this phase was to determine whether or not appreciable mass deposition occurred, when the QCM was totally exposed to the NRC vacuum environment. The amount of mass accumulated on the exposed QCM increased by about a factor of 2 (Table 1). However, the increase was much less than expected. The error was attributed to a possible miscalibration of the QCM unit. To verify that the QCM was functioning properly, a third set of tests was required (see below).

A plot of the mass accumulation rate versus temperature for the exposed NRC QCM is shown in Figure 2. This plot resembles the pattern observed in tests, where the sensor was installed behind the holder. A careful analysis of the data can lead to a reasonable conjecture about the elements condensing on the surface of the QCM sensor.

Elements present in the surface mass deposits can be identified by establishing operating pressure and temperature limits. Once these limits are defined, the elements that are likely to condense can be identified using elemental vapor pressure and temperature data. The examination of the vapor pressure characteristics of common gases over a temperature range from 150 K and 300 K and a pressure range from 1×10^{-7} to 1×10^{-1} torr indicated that water was the dominant contaminant condensing on the QCM. If the temperature falls below 150 K, CO_2 may also condense. In a clean vacuum environment, only mass deposits composed of water and CO_2 are possible. However, most working vacuum systems also contain various types of hydrocarbons (grease, oil, hydrocarbon derivatives, etc.).

Table 2. BJ5 QCM test results. Measured thickness was calculated from the corresponding total mass values.

TEST NUMBER	TEST LENGTH (min.)	TOTAL MASS (μg)	MEASURED THICKNESS (μm)	AREAL DENSITY ($\mu\text{g}/\text{cm}^2$)	FINAL TEMP (K)	FINAL PRESSURE ($\times 10^{-4}\text{torr}$)
B1	40	7.34	0.231	23.18	111.2	2.2
B2	38	8.02	0.253	25.32	115.5	2.0
B3	12	33.11	1.050	104.55	85.9	1.55
B4	11	15.69	0.495	49.54	91.1	2.64
B5	31	8.24	0.260	26.02	106.0	1.62

Phase Three. A third group of tests was used to verify that the QCM sensor and controller were functioning properly. First, the vacuum chamber was completely filled with clean, dry CO_2 gas. Then, the vacuum chamber was evacuated with the mechanical roughing and turbo molecular pumps. Finally, the QCM was cooled below 100 K. Under these conditions, a significant accumulation of debris was expected. However, even though more debris was deposited in this test than was observed in previous tests (Phase 2), the amount did not fall within the calculated range. A mathematical model, based on the kinetic theory of gases, indicated that the mass accumulation rate should have been three orders of magnitude higher than the mass accumulation rate shown in Figure 2.

Several additional tests were performed using only the roughing mechanical pump and CO_2 contamination source. Once more, the experimental values were much lower than the theoretical predictions. The QCM sensor collected mass without any conspicuous anomalies, but the QCM controller consistently displayed values that were lower than expected. Based on these observations, the QCM unit seemed to be functioning properly. Therefore, we concluded that the NRC vacuum system could not be used to model the BJ5 environment, because of differences in size and cleanliness. QCM measurements would have to be made in the BJ5 vacuum environment at the Maxwell facility in California.

BJ5 QCM TEST RESULTS

At the Maxwell facility, the QCM unit was connected to a Macintosh SE/30 personal computer running LabVIEW software using an ACRO 900 data acquisition and control system. Every attempt was made to duplicate the sample handling and mounting procedures that were used at NRC. A total of five tests were completed on the BJ5 in the superdome vacuum configuration (Table 2).

Test B1 was performed with the QCM attached to a holder having a kapton debris shield mounted to the face plate over the SXR transmission aperture. In tests B2 and B5, the debris shield was removed from the holder. In tests B3 and B4, the holder was removed so that the QCM sensor was totally exposed.

Test B1 was designed to determine whether or not the kapton debris shield had any effect on the build-up of debris on the sample surface. Cold surfaces greatly enhance pressure reduction by cryogenic pumping. As the debris shield was cooled, it acted as a cryogenic pump, removing particles from its vicinity. Test B1 was designed to show the magnitude of this phenomenon. Tests B2 and B5 were used to determine whether the debris shield acted as a cryogenic pump for and a physical barrier to the incoming debris par-

Table 3. Average BJ5 QCM Data.

TEST NUMBER	TEST DESCRIPTION	TIME (min)	MASS (μg)	THICKNESS (μm)	DENSITY ($\mu\text{g}/\text{cm}^2$)	TEMP (K)	PRESSURE ($\times 10^{-4}$ torr)
B1	DS,SH,S	40.0	7.34	0.231	23.18	111.2	2.25
B2 & B5	WDS,SH,BS	34.5	8.13	0.257	25.67	110.8	1.85
B3 & B4	SE	11.5	24.4	0.773	77.05	88.5	2.10

DS = Debris shield.

WDS = Without debris shield.

SH = Sample holder.

SE = Sensor exposed.

BS = Baffle stack.

tests. Tests B3 and B4 were designed to determine the net cleanliness of the BJ5 vacuum environment. The NRC sample holders were detached and removed for these tests, leaving the QCM sensor entirely exposed to the vacuum environment.

Table 3 shows the average values computed from the corresponding entries in Table 2. An important component of the assembly is the baffle stack, which limits the amount of debris falling onto the sample surface by deflecting debris particles arriving at an angle with respect to the vertical or with horizontal velocity components. Debris particles with horizontal velocity components will land on one of the oblique plates and are effectively diverted from the sample surface.

The use of the kapton debris shield reduced the amount of mass deposited on the QCM by approximately 10.8% (Table 3). When the QCM was exposed directly to the BJ5 environment, the accumulated mass was about 3 times greater than when the sensor was placed behind the baffle stack and holder assembly.

The debris shield acted as physical barrier that stopped particles from striking the QCM surface (Figure 3). By placing the debris shield over the fluence aperture, an isolation region was created between the sample surface and the debris shield. The QCM sensor was only able to acquire particles present in that small isolated volume. Calculations showed that mass deposits contributed by this region alone were negligible.

The mass accumulation rate consistently increased at temperature values ranging from 140 K to 160 K during test B1. Experiments performed at NRC did not show this phenomenon. Mass deposition from an undefined source was taking place. One possible explanation was the presence of hydrocarbons in the BJ5 vacuum system. The total mass accumulated in this test was 7.34 μm over an elapsed measurement time of 40 minutes. This mass corresponds to a uniform layer of water-ice 0.23 μm thick.

Changes in the mass deposition rate for the BJ5 without the debris shield (tests B2 and B5) are shown in Figure 4. A total mass of 8.13 μg was deposited on the QCM over 34.5 minutes. This mass corresponds to a water-ice thickness of 0.26 μm . The holder and the baffle stack reduce mass accumulation on the sensor surface.

In tests B3 and B4, the debris shield and the baffle stack were both removed, completely exposing the QCM to the system. Both tests showed a sharp increase in mass accumulation over a short time period. An average total accumulated mass for the two tests was 24.4 μg over 11.5 minutes. This mass corresponds to a uniform layer of water-ice 0.77 μm thick.

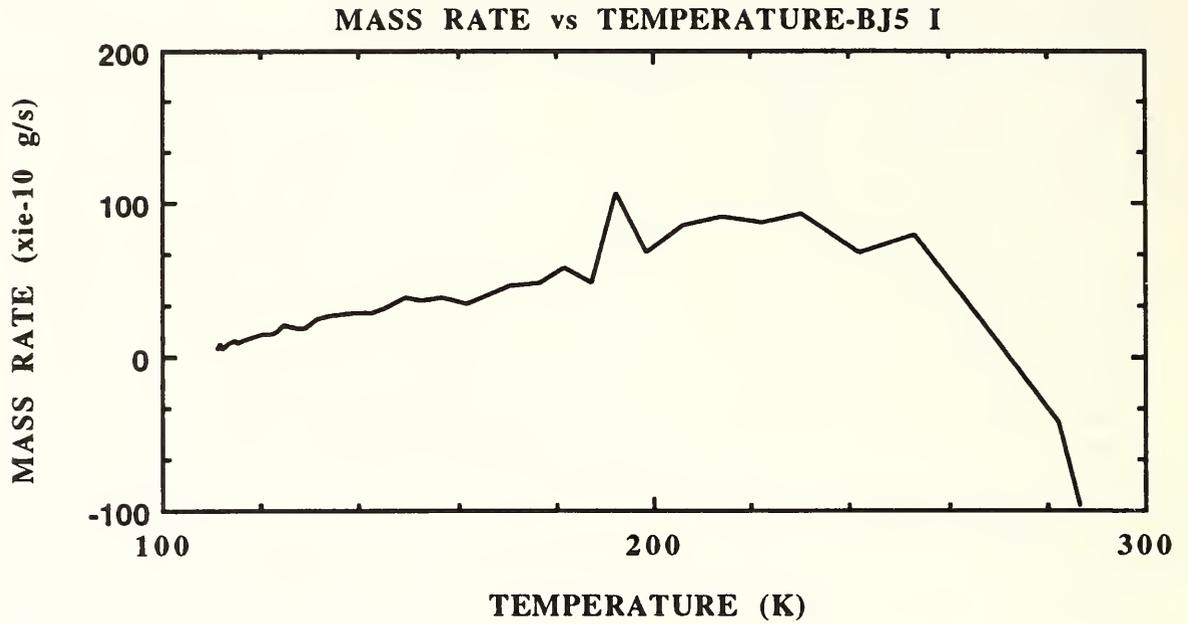


Figure 3. BJ5 QCM debris shield results.

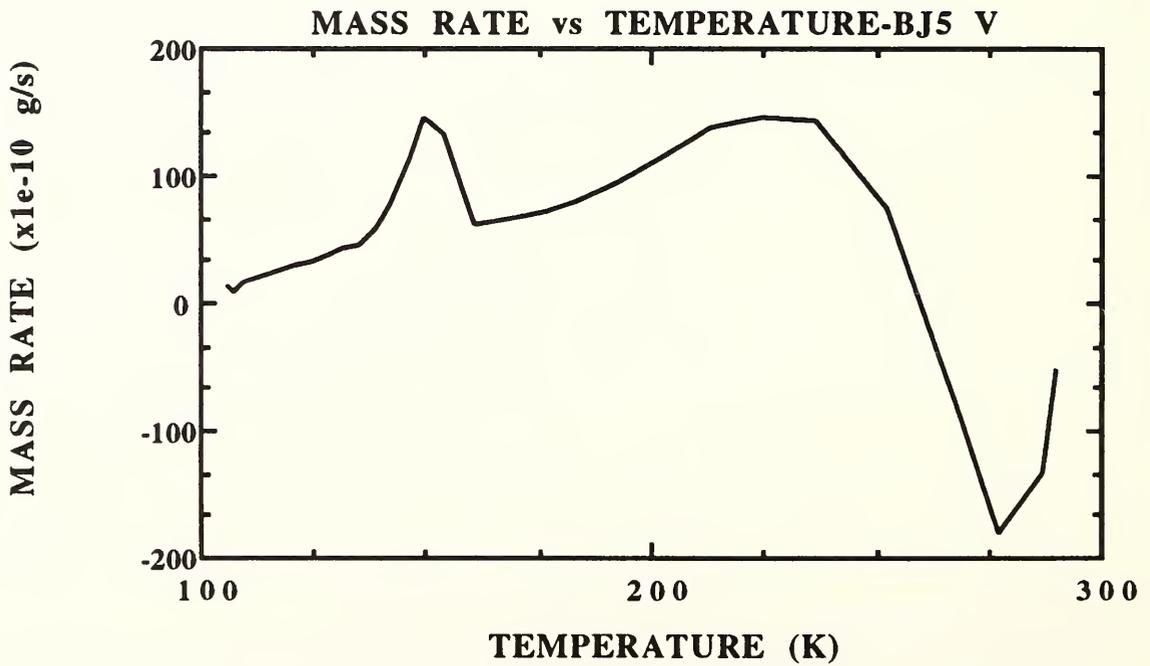


Figure 4. BJ5 QCM without debris shield results.

CONCLUSIONS

Measurements performed in the BJ5 super-dome mode indicate that an accumulated mass thickness between 0.2 μm to 0.5 μm is possible during a 40 minute run, when the sample is mounted on the holder with the debris shield and baffle stack. Less accumulated mass is expected in the BJ5 mini-dome, because the sample is well protected from the vacuum environment. For samples directly exposed to the BJ5 environment, the accumulated mass deposit ranges in thickness between 0.8 μm to 3.5 μm during an exposure of 40 minutes. The BJ5 environment is clearly contaminated. The data suggest that water is the dominant contaminant. Grease, oil, and other hydrocarbon derivatives are also assumed to be present in small quantities. The kapton debris shield reduced mass deposits on the QCM by 10.8%. The shield not only acted as a physical barrier, but also as a cryogenic trap.

ACKNOWLEDGMENTS

This effort was sponsored by the United States Army Space and Strategic Defense Command (USASSDC) Wide Field of View Program (WFOV).

LITERATURE CITED

- Barron, R. 1966. Cryogenic systems. McGraw-Hill Book Company, New York, 659 pp.
- Hollerman, W.A., D.R. Evans, M. Ward, G.A. Shelby, and A. Wilkosz. 1990. Silicon carbide composite mirror SXR test final report. Nichols Res. Corp., NRC-TR-90-097, Huntsville, Alabama, 112 pp.
- QCM Research. 1983. Quartz crystal microbalance. QCM Sensor Mark 9 Manual, QRM Research, Palo Alto California, 50 pp.
- QCM Research. 1983. Model 1900 user's manual. QRM Research, Palo Alto, California, 42 pp.

