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PREPRINT UCRL- 82797

Lawrence Livermore Laboratory

BLACK CHROME OUTGASSING STUDY

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September 25-27, 1979

Integrated Contractors Conference, Oak Ridge, TN

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BLACK CHROME OUTGASSING STUDY*

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INTRODUCTION

This report covers why black chrome is of interest, why its outgassing is relevant, how the study was conducted, examples of resultant data, and a conclusion.

Black chrome is of interest in several areas of activity. In the solar energy field black chrome is used predominantly as a collector. In plasma physics experiments black chrome is used as an anti-reflective coating inside the chambers. In optical transport systems black chrome is used as an anti-reflective coating also.

Vacuum outgassing characteristics must be known to establish the pumping speed requirements. How much gas is evolved determines the pump speed required. The species of gas evolved determines the type of pump. The outgassing characteristics must be known to determine needed precleaning steps.

METHOD AND EQUIPMENT DESCRIPTION

The outgassing was conducted by the throughput method using AVS Standard 9.1, in which $Q = \frac{\Delta PC}{A}$ where: Q is the outgassing rate in Torr liters/sec cm², ΔP is the differential pressure across the aperture in Torr, C is the conductance of the aperture in liters/second, and A is the surface area of the sample in square centimeters.

* "Work performed under the auspices of the U.S. Department of Energy by the Lawrence Livermore Laboratory under contract number W-7405-ENG-48."

Figure I is a schematic of the outgassing system used for this study. Shown is a chamber of known surface area with an ion gage, and an aperture with an ion gage on the side away from the chamber. This arrangement allows measuring the ΔP across the aperture. A bypass pumping network of higher conductance than the aperture permits using the gas analyzer in two modes of operation. One mode is when the chamber is at a higher pressure. In this mode the gas analyzer is used on the low pressure side of the aperture. The second mode is when the chamber is at lower pressure. In this mode the analyzer is used on the chamber itself, thereby increasing the sensitivity of the analyzer. The system used was all-metal-sealed above the LN_2 high vacuum trap. This allows a high temperature bake, which decreases the background gases evolving from the system. A turbomolecular pump backed by a mechanical pump was used. Figure II is a photo of the system. Note the close coupling of the gaging with the aperture in between, and the close coupling of the sample chamber to the pumping medium. This was done to minimize the error introduced by conductance limitations other than the aperture itself. Figure III is a schematic of the data acquisition system used. An LSI-11 computer was used in conjunction with a gas analyzer. The computer controlled the gas analyzer. The signal from the analyzer was then fed back to the computer, at which point there were three options: 1) observing a video terminal as the data was produced, or 2) X-Y plotting the data as it was produced, or 3) automatically storing the data on a disc as it was produced. In this study we used the latter option because it allowed us to take data faster. It also made it practical to take data continuously over an extended period of time.

OUTGASSING SYSTEM

FIGURE I

BLACK CHROME OUTGASSING STUDY

DATA ACQUISITION SYSTEM

FIGURE II

FIGURE III

DESCRIPTION OF SAMPLES

The black chrome studied was produced using the "DuPont" process. The coating was approximately ten microns thick, deposited on type 304 stainless steel. The surface area of each specimen was 3000 square centimeters.

OUTGASSING STUDY

The outgassing was studied under three conditions: 1) at ambient temperature, 2) at 350°C, and 3) at ambient temperature after the 350°C bake. The samples were divided into two sets. The first set (sample set #1) was studied under all three conditions. The second set (sample set #2) was studied at ambient temperature only.

Figure IV shows an RGA scan of the empty chamber, at ambient temperature after the 350°C bake. This was done to establish the background prior to the outgassing study. The pressure at this point was 2×10^{-9} Torr with an outgassing rate of 3.8×10^{-12} Torr liters/sec cm^2 . The major constituent was carbon monoxide (mass 28) with a little bit of water (mass 18) and some carbon dioxide. Figure V shows an RGA scan of sample set #1 at ambient temperature after 100 hours of vacuum exposure. The pressure was 3×10^{-7} Torr. The outgassing rate was 3×10^{-9} Torr liters/sec cm^2 . The major constituent was water (mass 18). Also present were hydrogen (mass 1 & 2), oxygen (mass 16 & 32), carbon monoxide (mass 28) and carbon dioxide (mass 44). Figure VI shows an RGA scan of sample set #1 at 350°C. The scan was taken after 100 hours at this temperature. The pressure was 3.8×10^{-7} Torr. The outgassing

CHAMBER

AT AMBIENT TEMP
AFTER 350°C BAKE

FIGURE IV

PRESSURE = 2×10^{-9} TORR

$Q = 3.8 \times 10^{-12}$ TORR-LITERS/SEC /CM²

MASS

AMP (10^{-10})

2

.3

18

.5

28

1.0

44

.4

AMP

AMU

100

SAMPLE #1

AT AMBIENT TEMP
AFTER 100 HOURS UNDER
VACUUM

FIGURE V

PRESSURE = 3×10^{-7} TORR

$Q = 3 \times 10^{-9}$ TORR-LITERS / SEC / CM²

MASS	AMP (10^{-10})
2	85.0
16	54.0
18	420.0
28	60.0
32	57.0
44	62.0

AMP

SAMPLE #1

AT 350°C FOR 100 HOURS

FIGURE VI

PRESSURE = 3.8×10^{-7} TORR

$Q = 1 \times 10^{-8}$ TORR-LITERS/SEC /CM²

MASS	AMP (10^{-10})
2	190.0
12	40.0
18	320.0
28	67.0
44	79.0

AMP

rate was 1×10^{-8} Torr liters/sec cm^2 . The major constituent was still water (mass 18). Hydrogen (mass 1 & 2) had more than doubled. Carbon (mass 12) was now evident. Carbon monoxide and carbon dioxide had risen slightly. Figure VII shows an RGA scan of sample set #1 after cooling and being maintained in vacuum for an additional 80 hours. The pressure is 6×10^{-9} Torr. The outgassing rate is 9×10^{-11} Torr liters/sec cm^2 . The major constituent was carbon monoxide (mass 28). The amount of carbon monoxide (mass 28) was greatly reduced. Figure VIII shows an RGA scan of sample set #2 at ambient temperature after four hours under vacuum. The RGA was on the pump side of the aperture because the chamber was operating in the higher pressure regime. The pressure was 4×10^{-7} Torr. The outgassing rate was 8×10^{-9} Torr liters/sec cm^2 . The major constituent was water (mass 18), hydrogen (mass 1 & 2), carbon (mass 12), oxygen (mass 16 & 32), carbon monoxide (mass 28), and carbon dioxide (mass 44) were also present. Figure IX shows an RGA scan of sample set #2 at ambient temperature after 100 hours under vacuum. The pressure was 2×10^{-7} Torr. The outgassing rate was 2×10^{-9} Torr liters/sec cm^2 . The major constituent was water (mass 18) in approximately the same quantity as in sample set #1. Also present were hydrogen (mass 1 & 2), oxygen (mass 16 & 32), carbon monoxide (mass 28), and carbon dioxide (mass 44). Figure X shows an RGA scan of sample set #2 at ambient temperature, after 250 hours under vacuum. The outgassing rate was 3×10^{-10} Torr liters/sec cm^2 . The major constituent was still water, but the quantity was 100 times lower than in the previous scan. The sample was at equilibrium outgassing rate.

SAMPLE #1

AT AMBIENT TEMP
AFTER 350°C BAKE

FIGURE VII

PRESSURE = 6×10^{-9} TORR

$Q = 9 \times 10^{-11}$ TORR-LITERS / SEC / CM²

MASS	AMP (10^{-10})
------	--------------------

2	1.0
---	-----

18	1.5
----	-----

28	6.0
----	-----

44	1.4
----	-----

AMP

28

AMU

100

SAMPLE #2

AT AMBIENT TEMP
AFTER 4 HOURS UNDER
VACUUM

NOTE: RGA ON PUMP
SIDE OF APERTURE

FIGURE VIII

PRESSURE = 4×10^{-7} TORR

$Q = 8 \times 10^{-9}$ TORR-LITERS / SEC / CM²

MASS	AMP (10^{-10})
2	34.0
12	14.0
16	25.0
18	240.0
28	61.0
32	43.0
44	80.0

AMP

SAMPLE #2

AT AMBIENT TEMP
AFTER 100 HOURS UNDER
VACUUM

FIGURE IX

PRESSURE = 2×10^{-7} TORR

$Q = 2.8 \times 10^{-9}$ TORR-LITERS / SEC / CM²

MASS	AMP (10^{-10})
------	--------------------

2	78.0
---	------

16	50.0
----	------

18	389.0
----	-------

28	61.0
----	------

32	50.0
----	------

44	51.0
----	------

SAMPLE #2

AT AMBIENT TEMP
AFTER 250 HOURS UNDER
VACUUM

FIGURE X

$$\text{PRESSURE} = 3 \times 10^{-9} \text{ TORR}$$

$$Q = 3 \times 10^{-10} \text{ TORR-LITERS / SEC / CM}^2$$

MASS	AMP (10^{-10})
2	4.6
18	4.9
28	4.7
44	3.6

AMP

CONCLUSION

This study indicates that the outgassing characteristics of "DuPont" black chrome allows its use in vacuum applications. The outgassing rate was 3×10^{-10} Torr liters/sec cm^2 unbaked. This is acceptable for use in optical transport networks and solar collectors. The outgassing rate for baked samples was 9×10^{-11} Torr liters/sec cm^2 . This is acceptable for plasma physics ultra high vacuum applications. Although the difference between baked and unbaked samples appears extremely small, one must consider that when dealing with surface areas of millions of square centimeters, this difference greatly influences the pump speed required to achieve a given pressure.

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