RE-224

RESEARCH IN GAS-SURFACE INTERACTION 1964-65

Part III

Surface Contamination

August 1965

Grumman

RESEARCH DEPARTMENT

GRUMMAN AIRCRAFT ENGINEERING CORPORATION

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PART III

Surface Contamination

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Part III of Final Report on Contract NASw-1027
Fluid Physics Branch
Research Division
Office of Advanced Research and Technology
NASA

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Charles E. Mack, Jr.
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FOREWORD

This report, in three separate parts, summarizes the research conducted over the past year on the interaction of gas molecules and solid surfaces under NASA Contract NASw-1027. Previous work in this field (under NASr-104 and NASw-709) was reported in Refs. 1 through 4, and the work described herein relies heavily on the earlier results. We expect to continue this research along the lines suggested in the following discussion.

Part I of this report describes the additions and modifications that have been made to our computer program for calculating molecule-surface interactions, gives results which represent the early findings from these modifications, discusses parametric correlation work that has resulted in a much improved understanding of the results of some of our previous calculations, and presents the plans we have for further application of all of these approaches. Part II describes our molecular beam apparatus in its present form and gives a summary of our research on the generation of high intensity molecular beams. Remaining problems and plans for experiments which are now getting under way are also described. Part III is a progress report on the experimental development of methods for the preparation of atomically controlled surfaces to be used for testing in the molecular beam. The high vacuum apparatus for those experiments was described in a previous report (Ref. 4). (Part I, Part II, and Part III are identified as RE-222, RE-223, and RE-224, respectively.)

ACKNOWLEDGMENT

I would like to thank Gerard Connell for his invaluable aid in designing and acquiring the equipment and in helping to make it work. Robert Gunderson, our laboratory technician, has also been very helpful. I am also grateful for the support and encouragement of our colleagues in the Grumman Research Department and of Mr. Alfred P. Gessow and his staff in the Fluid Physics Branch, Research Division of the NASA Office of Advanced Research and Technology.

ABSTRACT

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Preliminary experience on the performance of the surface preparation and characterization apparatus is described. The vacuum system is being modified to increase its reliability and ease of performance. The electrooptical experiments that have been performed indicate that the techniques and equipment to be used will be adequate. In the very near future we expect to begin our major effort toward performing the necessary experiments leading to useful surface preparation techniques.

Author

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INTRODUCTION

The apparatus with which we are to conduct experiments leading to a means of preparing and characterizing sample specimens for our molecular beam has been constructed (Ref. 4). Our primary aims are to determine under what conditions an atomically clean metallic surface can be obtained and maintained, and to determine when these conditions are fulfilled. This knowledge is then to be used to allow us to perform molecular beam experiments on specimens that have been preconditioned in a reproducible way (Ref. 3). This work is necessary because the state of the surface is of utmost importance in understanding and reproducing gas-surface interaction experiments.

We are attempting to prepare our specimens by a combination of high temperature heating, ion bombardment, and annealing. The experiments will be performed in an ultrahigh vacuum environment of 10^{-9} torr. The cleanliness of the surface will be inferred by measurement of its work function and the spectral distribution of the photoelectric yield. In addition a mass spectrometer residual gas analyzer will be employed to monitor the changes in the gases evolved during the cleaning procedure.

SYSTEM MODIFICATIONS

Figure 1 is a photograph of the surface preparation apparatus. During the course of the present development of the system it has become advisable to make several modifications of the original version described in Ref. 4. These include:

1. The replacement of the Alpert type copper foil traps by a Biondi zeolite trap.

2. Construction and installation of an auxiliary demountable pumping system. This allows us to pump the inlet portion of the gas handling to 10^{-6} torr, and also provides a lower working fore pressure.

3. Replacement of the sapphire insulated feedthrough by a less troublesome tungsten to glass seal, which is now in the process of being installed. During the temperature cycling of the system, the former feedthrough developed gas leaks when not carefully handled and several tests indicated this replacement was feasible.

4. A bakeable partial pressure analyzer with a Nier-type electron-bombardment ion source, and a 90-degree sector magnetic analyzer, which can detect partial pressures as low as 10^{-13} torr at mass numbers up to 150 atomic mass units, has been purchased. This is to be installed to the vacuum system before the end of the next quarter. For our experiments this partial pressure analyzer will serve a dual purpose. First, at present the processing of the vacuum system to achieve ultimate pressures is done more or less by trial and error, as we have

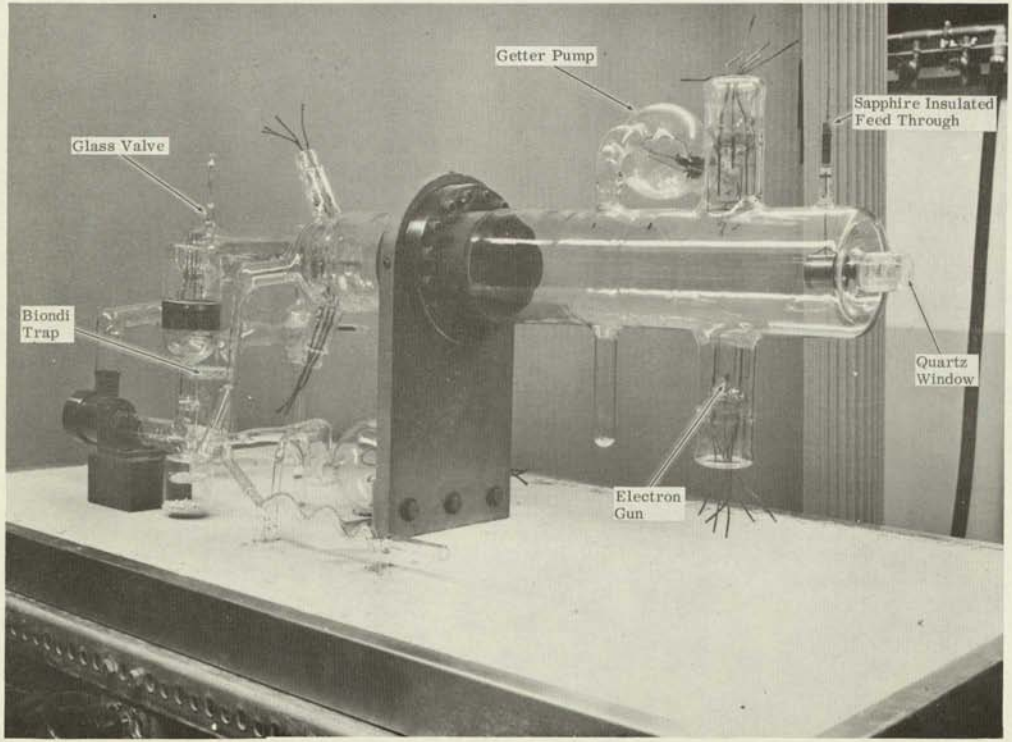

Fig. 1 Photograph of Present Apparatus

practically no idea of the changes of the gas composition with treatment parameters. This makes the attainment of ultrahigh vacuum conditions highly empirical and usually nonreproducible. The second purpose will be to analyze the gas during surface cleaning, as discussed in a later section of this report.

RESULTS OBTAINED TO DATE

A brief calculation indicates that at a pressure of about 10^{-7} torr a significant fraction of a monolayer will form on a surface within the order of about 10 seconds. It appears that in order to perform the necessary experiments to estimate the cleanliness of the surface we should have an ambient pressure of at least as low as 10^{-9} torr. In order to obtain and maintain pressures in this range we have found it necessary to subject the vacuum system to a rigorous preconditioning. This conditioning requires the continuous cycling of the system from room temperature to about 400°C several times over a period of days with intermittent high temperature heating of internal metallic parts. During this treatment it has been necessary to isolate the system from the diffusion pump with a trap cooled to the liquid nitrogen temperature (-195°C). It has also proved important to maintain the liquid nitrogen level to within about 90 per cent of maximum, in order to prevent excessive outgassing of the trap walls. These factors (extremes of temperatures and intermittent flow of liquid nitrogen) coupled with the fact that the system is constructed of several different materials, each of which responding differently to temperature gradients and fluctuations, has required the expenditure of a significant effort toward careful control of the pumpdown procedures. In addition, we have designed and constructed an automatic safety system that properly sequences the shutdown of the system in case of emergency during periods when it is left unattended.

In principle it appears feasible to expect the system to reach an ultimate pressure in the 10^{-10} torr range. At this pressure

there will be sufficient time to carefully perform the necessary experiments before significant surface changes can take place. However, to date this expectation has not been achieved. In addition we have not yet been able to obtain our lowest pressures consistently and in a predictable manner. In this area we expect the mass spectrometer partial pressure analyzer to be of invaluable assistance.

At present the usual fore pressure for the diffusion pump is about 1×10^{-5} torr. The pressure at the low vacuum side of the pump is about 3×10^{-8} torr with liquid nitrogen in the cold trap. The pressure in the test chamber can be maintained at about 6×10^{-9} with the zeolite trap pumping at maximum capacity. However to maintain this requires an excessive amount of manipulation and attention. We hope to avoid using the zeolite as a pump by flashing the getter and sealing the test section from the pumping system. This has proved advisable as it appears the pumping section of the system acts as a source of contamination in the 10^{-9} torr range. This procedure has been used successfully by other investigators (Ref. 5).

The specimen surface is to be treated by a combination of argon ion bombardment, high temperature heating, and annealing. The ion bombardment scheme appears to work properly, although a much better evaluation of its effectiveness will be possible after installation of the partial pressure analyzer. The main area of difficulty is associated with the nonlocalization of the plasma discharge. Visual examination of the specimen mounting arrangement currently being employed showed no signs of the effects of sputtering. The side arm containing the gun structure, which is used to initiate and maintain the discharge, appeared to have a film deposited on it. This could be an indication that a different set of operating conditions are necessary. A more definite conclusion

will await the installation of the partial pressure analyzer. Under the present conditions the discharge can be maintained at about 400V with a current up to 200ua/cm^2 at a pressure of about 2×10^{-4} torr, a pressure low enough to make backscattering negligible.

The results of the heating experiments are very preliminary. It was felt that the heating should not be carried to more than about 150°C, as there was a serious risk that the specimen could loosen from its holder and strike the system walls possibly shattering them. It was observed that with the filament at about 2000°K, the vacuum conditions were not seriously affected. At present a thermocouple has been attached to a dummy surface specimen for temperature calibration and in addition a secondary fastening device will be employed to minimize the risk of damage to the system.

The photoelectric experiments used to estimate specimen surface conditions were attempted in order to assess the performance of the associated equipment. It appeared that the equipment was functioning properly, but the results obtained have not been completely evaluated. It seemed that even though the system pressure was at a reasonable level, there was a gas leak through the sapphire feedthrough (which has since been replaced). This caused the photoelectric response of the specimen to vary with time after ion bombardment. After a short time there was no measurable photoelectric current. This may have been due to gas adsorption (probably oxygen) by the surface.

In order to perform the ion bombardment, heating, and photoelectric experiments, it is necessary that the specimen be translated to each of three different positions by a device which is operated external to the system. We are attempting to do this magnetically, with the specimen riding on a track supported by the

inner walls of the test chamber, the specimen axis being maintained colinear with the axis of the test chamber and the hollow cylindrical anode. The present device appears to work well in the 1×10^{-8} torr range, but there are several improvements to be made that will optimize its performance.

PRESENT CONCLUSIONS AND STATUS

There are several changes currently in progress:

1. The sapphire feedthrough is being replaced by a more reliable tungsten to glass seal.
2. An adjustable optical table is being constructed.
3. Several automatic control and safety devices are being constructed.
4. The residual gas analyzer has been ordered, and provision for its installation is being made.

The first three items should be completed shortly, the last may take two months due to the vendor's shortened summer schedule.

We expect to have begun experiments on the (100) face of a pure nickel single crystal within three months. The results of these experiments should allow us to design practical preparation and control techniques for use in our molecular beam apparatus.

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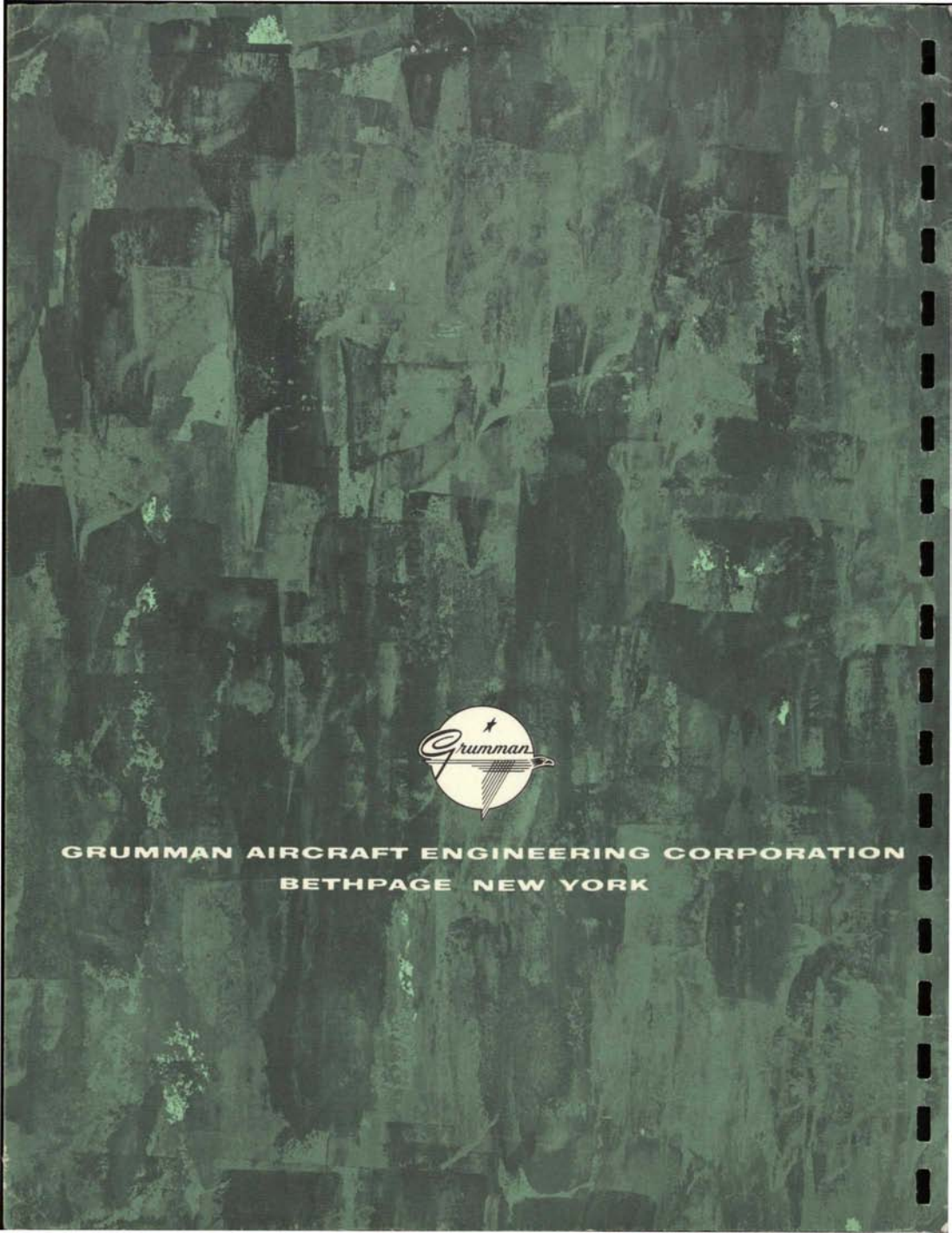

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