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ACTIVE CLEANING TECHNIQUE FOR REMOVING CONTAMINATION FROM OPTICAL SURFACES IN SPACE

Quarterly Progress Report No. 8

By

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1.0 SUMMARY

This report describes work accomplished during the ninth quarter of NASA Contract NAS8-26385. The program is aimed at developing an active cleaning technique (ACT) for removing contaminants from optical surfaces in space. During this reporting period in-situ contamination/cleaning experiments were conducted on gold and platinum coated mirrors. These mirrors were contaminated by exposure to U.V. radiation in a 1,3 butadiene environment. Both argon and oxygen plasma exposures cleaned the mirrors equally well. The U.V. reflectance of the gold mirrors was nearly restored and that of the platinum mirror was more than completely restored. Exposures to ambient and vacuum conditions had little effect on the contaminant film or subsequent plasma cleaning.

The successful contaminant removal using argon plasma cleaning led to an investigation of carbon removal using various gases. A carbon coated quartz crystal microbalance (QCM) was exposed to helium, oxygen and hydrogen plasmas. The QCM response indicated these gases are equally effective in removing carbon.

Silicone cleaning experiments were also conducted during this reporting period. The outgas products of silicone compounds (RTV3116 and RTV602) were deposited on MgF_2/Al mirrors with the film deposition monitored by use of the QCM. Film depositions in the order of 2×10^{-6} gm/cm^2 produced severe reflectance degradation in the 1200-1400Å wavelength region and increased the reflectance in the 1800-3000Å wavelength region. The contaminated mirrors were exposed to helium, oxygen and hydrogen plasmas. The exposures tended to restore the reflectance at the shorter wavelengths and degrade it at the longer wavelengths. Hydrogen plasma exposure produced the largest restoration at the shorter wavelengths and helium produced the largest degradation at the longer wavelengths.

2.0 INTRODUCTION

The need for developing an in-situ or active cleaning technique (ACT) for use in both space and vacuum chambers has recently become apparent. Manned spacecraft have experienced numerous contamination problems including deposition of volatile organic compounds onto windows, and light scattering from particulate contaminants surrounding the spacecraft. Sources of this contamination include outgassing of organic compounds, waste and water dumps, rocket plumes, and leakage from the life support system. It is believed that contaminant film deposition has also occurred on unmanned spacecraft surfaces. Data from a reflectometer experiment on the ATS-3 spacecraft (Reference 1) has indicated rather severe degradation on reflective surfaces, which may be the result of contaminant film deposition. Also, the TV camera mirror from the Surveyor III spacecraft which resided on the moon for 2-1/2 years was covered with a diffuse coating--presumably the result of contaminant film deposition. Contamination on an unmanned spacecraft has been verified with quartz-crystal thin film monitors on OGO-6 (Reference 2). A recent review of the spacecraft contamination problem has been published in Reference 3.

Contamination can also occur during spacecraft testing in high vacuum chambers. An example of this was the extreme-UV solar spectroheliometer experiment for the Apollo Telescope Mount (ATM) vehicle. A film of back-streamed diffusion pump oil was apparently deposited on surfaces during thermal/ vacuum testing (Reference 4). Another example of contaminant film deposition during environmental testing is discussed in Reference 5. In those experiments it was shown that an extremely stable organic film could be deposited onto telescope mirror surfaces during irradiation with low energy protons in a relatively clean vacuum environment.

Based on existing knowledge, contamination problems anticipated for future spacecraft include: (1) deposition of non-volatile substances onto optical components, sensing elements, and temperature control surfaces; (2) particulate and gaseous contamination near the spacecraft (resulting in light scattering and absorption); and (3) chemical contamination which can interfere with upper atmosphere studies, analysis of interplanetary or planetary matter, and material processing experiments. It is anticipated that contamination effects can be reduced by changes in design, materials, operating procedures, and possibly control techniques. The use of more sensitive surfaces and longer term missions will, however, offset these improvements. Thus, the need exists for developing an ACT for space use.

The specific approach being investigated in this program involves exposing surfaces to a plasma containing excited gas species. Experiments in References 5 and 6 have shown that this cleaning technique is very effective for removing hydrocarbon contaminant films from optical surfaces in vacuum.

3.0 EXPERIMENTAL RESULTS

3.1 In-Situ Contamination/Cleaning Experiments

The in-situ contamination/cleaning experiments were continued using gold and platinum coated mirrors. Prior experiments (Reference 7) used MgF_2 overcoated Al mirrors. The continuation of these experiments had been delayed because of the contaminated optics in the monochromator (Reference 7 and 8). The present experiments utilized two gold coated mirrors and one platinum coated mirror. These mirrors were contaminated by exposure to U.V. radiation in a 1,3 butadiene environment, and subsequently exposed to argon and oxygen plasmas. In addition to the plasma cleaning effects the effects of extended periods (at vacuum and ambient conditions) on the contaminant films were investigated.

Table 1 gives the 'log of events' for gold coated mirror sample Au6. Figures 1 and 2 show the reflectance data for this sample. The primary objective of this experiment was to determine the effect of exposure to an argon plasma. Figure 1 shows that the argon plasma removes the contaminant film. Instead of continuing the argon plasma exposures it was decided to see if an oxygen plasma would clean more rapidly. The data in Figure 1 do not indicate any apparent differences in cleaning rate. Since the reflectance was not completely restored the question is raised as to whether or not the order of exposure (in this case argon first and then oxygen) affects the contaminant cleaning. The tests of this sample also show that a short term (one hour) exposure to ambient air does not change the contaminant film effects on the U.V. reflectance.

TABLE 1 LOG OF EVENTS FOR GOLD COATED MIRROR - Au6

EVENT	COMMENT
Contamination	
Exposed to U.V. radiation at 4 torr 1,3 butadiene for 100 minutes	QCM response indicated film deposition of about 5×10^{-6} gm/cm^2. U.V. reflectance degraded by more than factor of 3.
Sample 'let up to air' for one hour	No change in U.V. reflectance.
Argon Plasma Exposures	Chamber pressure 5μ, plasma tube pressure 4 torr, RF power 38 watts, RF voltage 2000 volts, peak to peak. 'Ignited plume' mode of operation.
1st exposure 7.5 minutes	
2nd exposure 5. minutes	
3rd exposure 22.5 minutes	
	All exposures increased the U.V. reflectance.
Oxygen Plasma Exposures	Chamber pressure 6μ, plasma tube pressure 6 torr, RF power 35 watts, RF voltage 2500 volts peak to peak. 'Ignited plume' mode of operation. First three exposures significantly increased U.V. reflectance. Fourth exposure gave no further increase. U.V. reflectance not completely restored.
1st exposure 7.5 minutes	
2nd exposure 15. minutes	
3rd exposure 15. minutes	
4th exposure 10. minutes	
Sample left under vacuum for 3 days	Chamber pressure rose from about 10^{-5} torr to 3.5 torr in 3 days. U.V. reflectance slightly degraded at shorter wavelengths.
Plasma Cleaned Monochromator Optics	20 minutes oxygen plasma exposure. U.V. reflectance measurements showed some differences, from previous values, below 1400A.
Oxygen Plasma Exposure	15 minute exposure produced small increase in reflectance.

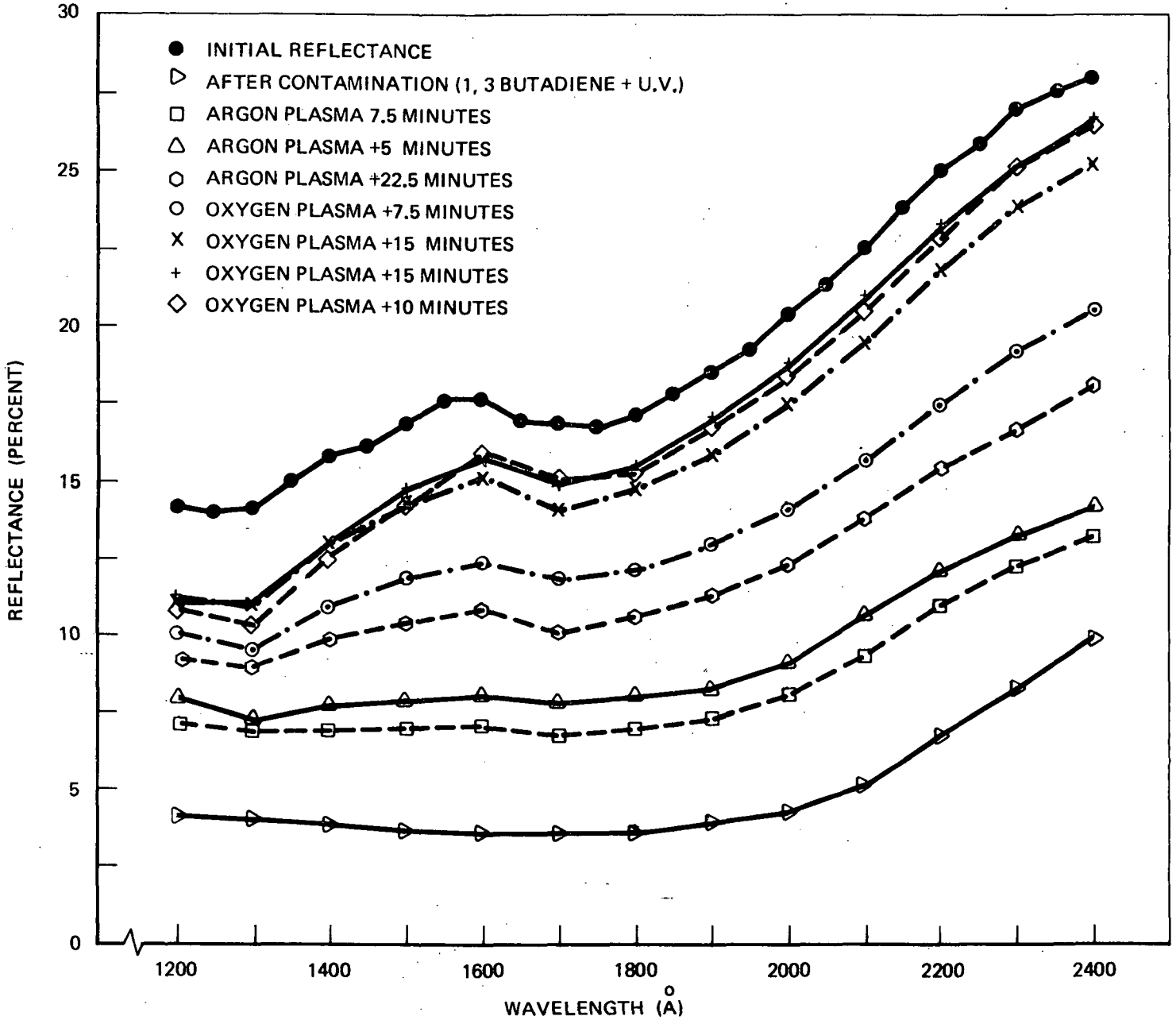

Figure 1: REFLECTANCE DATA FOR GOLD COATED MIRROR Au6

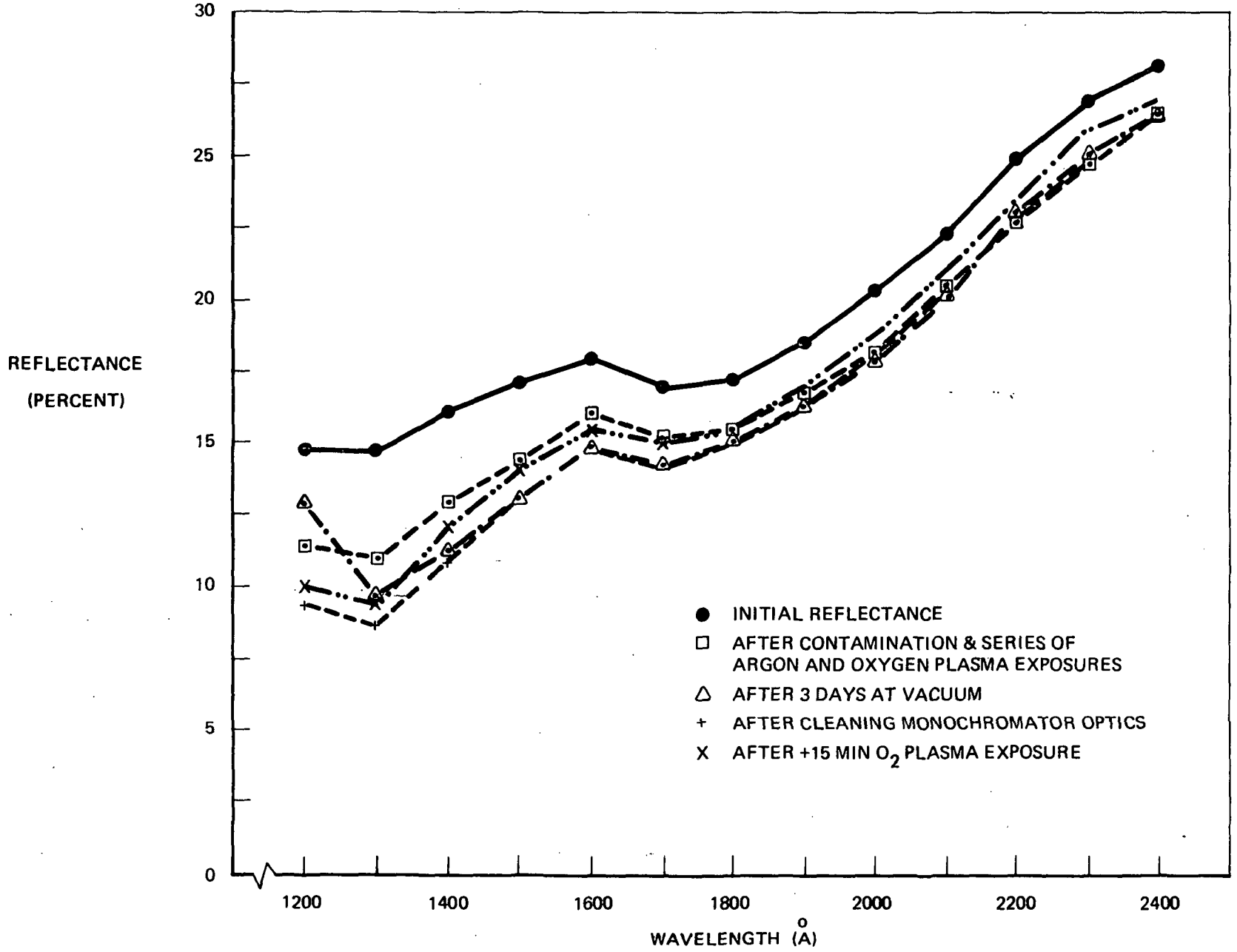

Figure 2: REFLECTANCE DATA FOR GOLD COATED MIRROR Au6

Also shown in these tests is a slight degradation of the U.V. reflectance during an extended period (3 days) under vacuum (see Figure 2). The data in Figure 2 also show that the degradation of the monochromator optics influences the reflectance data measurements in the 1200 to 1400 Å wavelength region.

In order to see if the order of plasma exposure (argon or oxygen first) affects the contaminant film cleaning, experiments were conducted using a second gold coated mirror (Au7). Table 2 gives the 'log of events' and Figures 3-5 show the reflectance data for sample Au7. Figure 3 shows that the U.V. reflectance was nearly restored using two oxygen plasma exposures and that an additional exposure using an argon plasma had little effect on the reflectance. Since the U.V. reflectance of Au7 was more completely restored using an oxygen plasma than was that of Au6 using first argon and then oxygen plasmas, Au7 was recontaminated to see if argon would be as effective as oxygen in plasma cleaning. Figure 4 shows that a 15 minute argon plasma exposure restored the U.V. reflectance to that prior to recontamination. These data indicate that there is little difference between argon and oxygen plasma cleaning. If the cleaning phenomena is, as has been thought, due to the chemical reaction between the plasma and the contaminant film resulting in volatile products, then it seems reasonable to expect much more effective cleaning with oxygen than with argon. Since this does not occur, the cleaning phenomena must be related to the interaction of excited atoms in the plasma with the contaminant film and/or ion sputtering of the contaminant film. Similar conclusions were reached in a recent experimental investigation (Reference 9) which showed that the presence of atomic oxygen was not sufficient for cleaning contaminant films. Figure 4 also shows that the U.V. reflectance of the sample degraded after

TABLE 2 LOG OF EVENTS FOR GOLD COATED MIRROR - Au7

EVENT	COMMENTS
Contamination	
Exposed to U.V. radiation at 4-6 torr 1,3 butadiene for 78 minutes	QCM response indicated film deposition of about 1.7×10^{-6} gm/cm^2.
Continued exposure for additional 18 minutes after leaving at vacuum overnight.	Total contaminant film deposition of about 2.2×10^{-6}gm/cm^2. U.V. reflectance degraded by more than factor of 2.
Oxygen Plasma Exposures	
1st exposure 7.5 minutes	U.V. reflectance nearly restored by these exposures.
2nd exposure 22.5 minutes	
Argon Plasma Exposure	15 minutes exposure produced little change in reflectance.
Contamination	
Exposed to U.V. radiation at 4 torr 1,3 butadiene for 100 minutes.	QCM indicated film deposition of about 1.8×10^{-6}gm/cm^2. U.V. reflectance degraded.
Argon Plasma Exposure	15 minute exposure restored U.V. reflectance to that prior to recontamination.
Let Chamber 'up to air' and cleaned quartz U.V. window	U.V. reflectance degraded slightly.
Contamination	
Exposed to U.V. radiation at 4 torr 1,3 butadiene for 30 minutes	QCM response showed slow contamination rate (film deposition of only about 0.2×10^{-6}gm/cm^2). U.V. reflectance showed very little degradation.
Let up to air and recleaned window. Exposure then continued for additional 99 minutes	QCM showed total film deposition of about 2.9×10^{-6}gm/cm^2. U.V. reflectance degraded by more than factor of 2.5.
Exposure sample to ambient air for 20 hours.	U.V. reflectance remained unchanged.
Argon Plasma Exposures	
1st exposure 22.5 minutes	First exposure nearly restored U.V. reflectance. Second exposure caused significant degradation.
2nd exposure 22.5 minutes	
17 hours under vacuum	U.V. reflectance degraded slightly.
Oxygen Plasma Exposure	25 minute exposure produced further degradation.

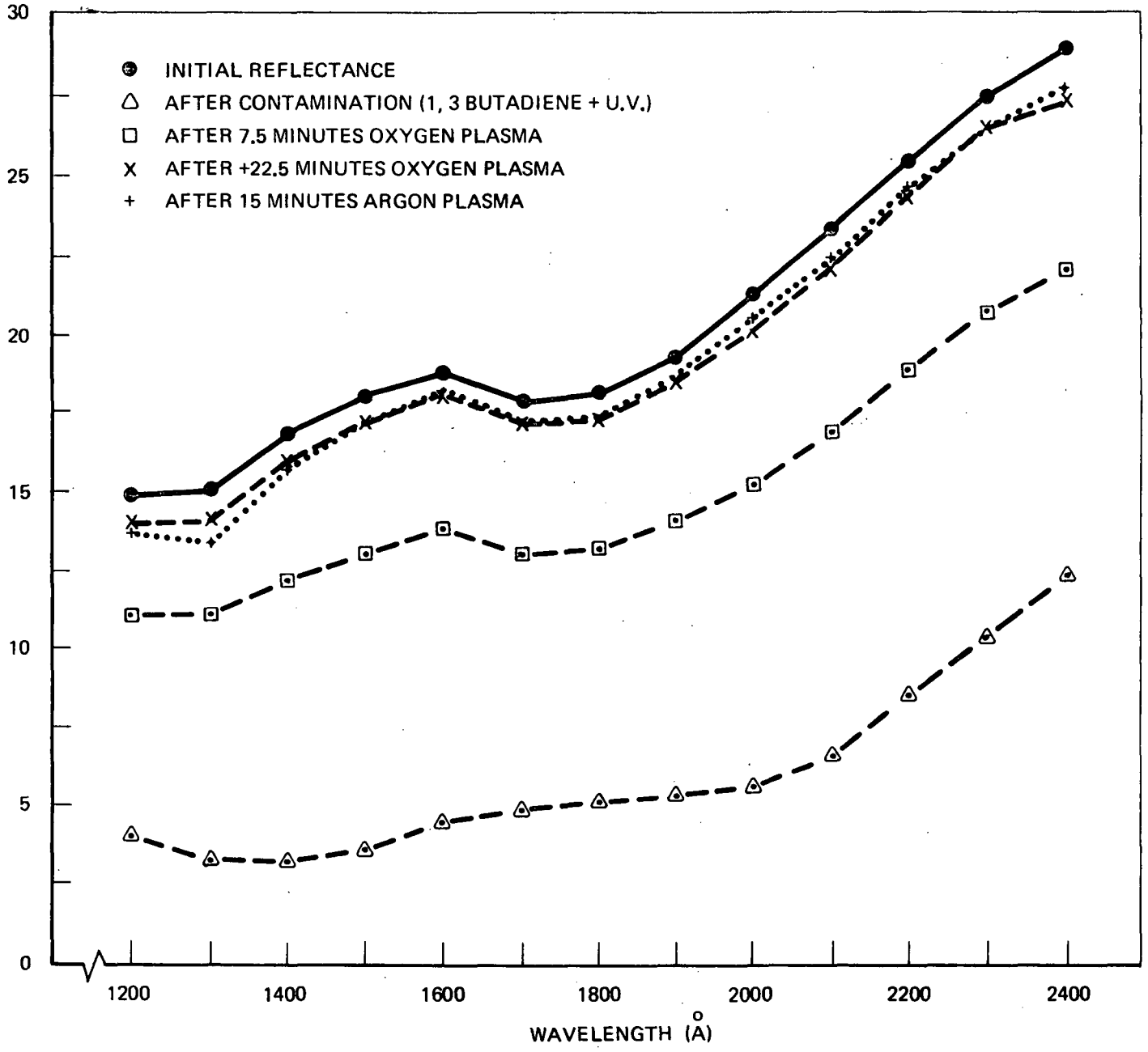

Figure 3: REFLECTANCE DATA FOR GOLD COATED MIRROR Au7

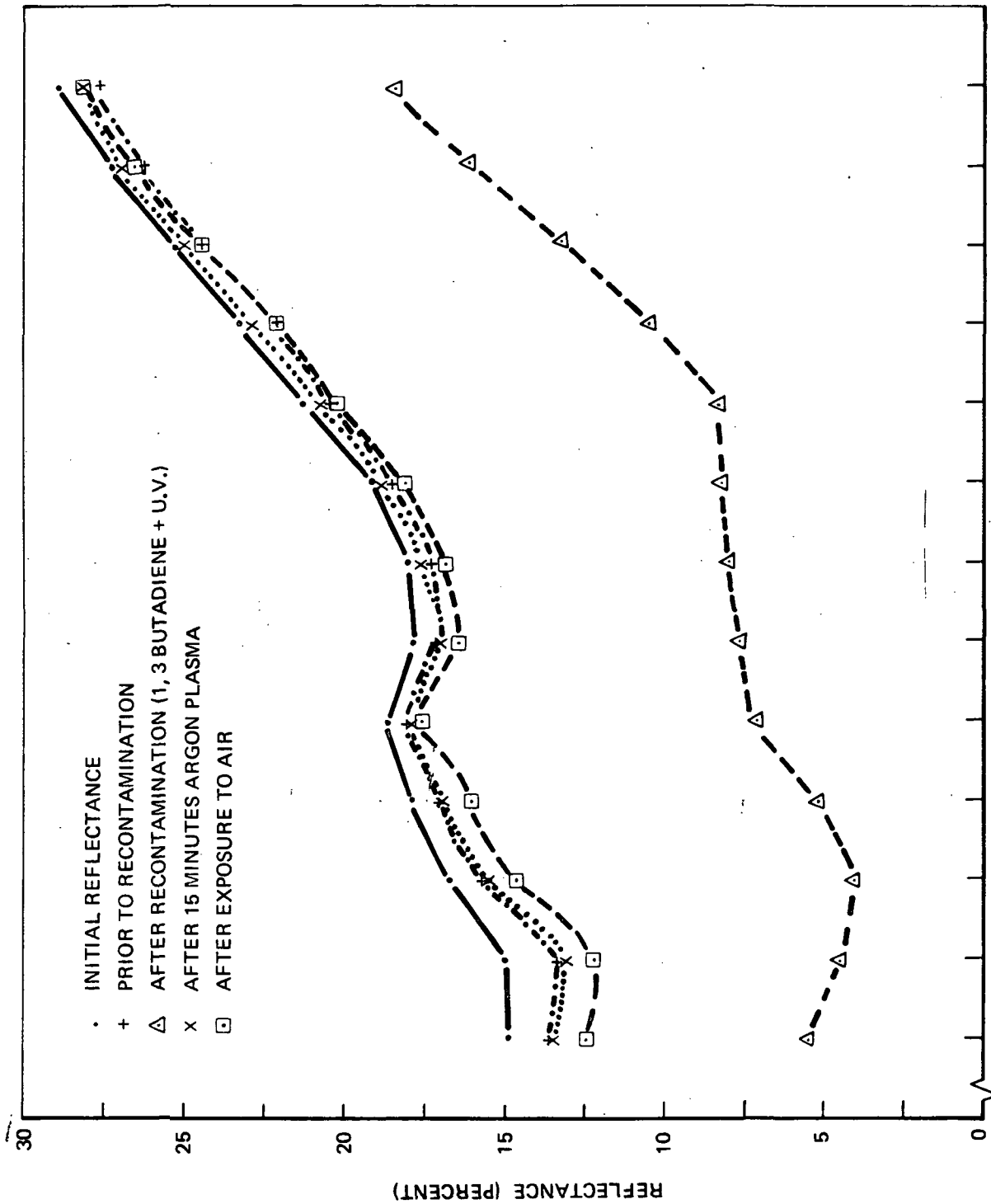

Figure 4: REFLECTANCE DATA FOR GOLD COATED MIRROR Au7

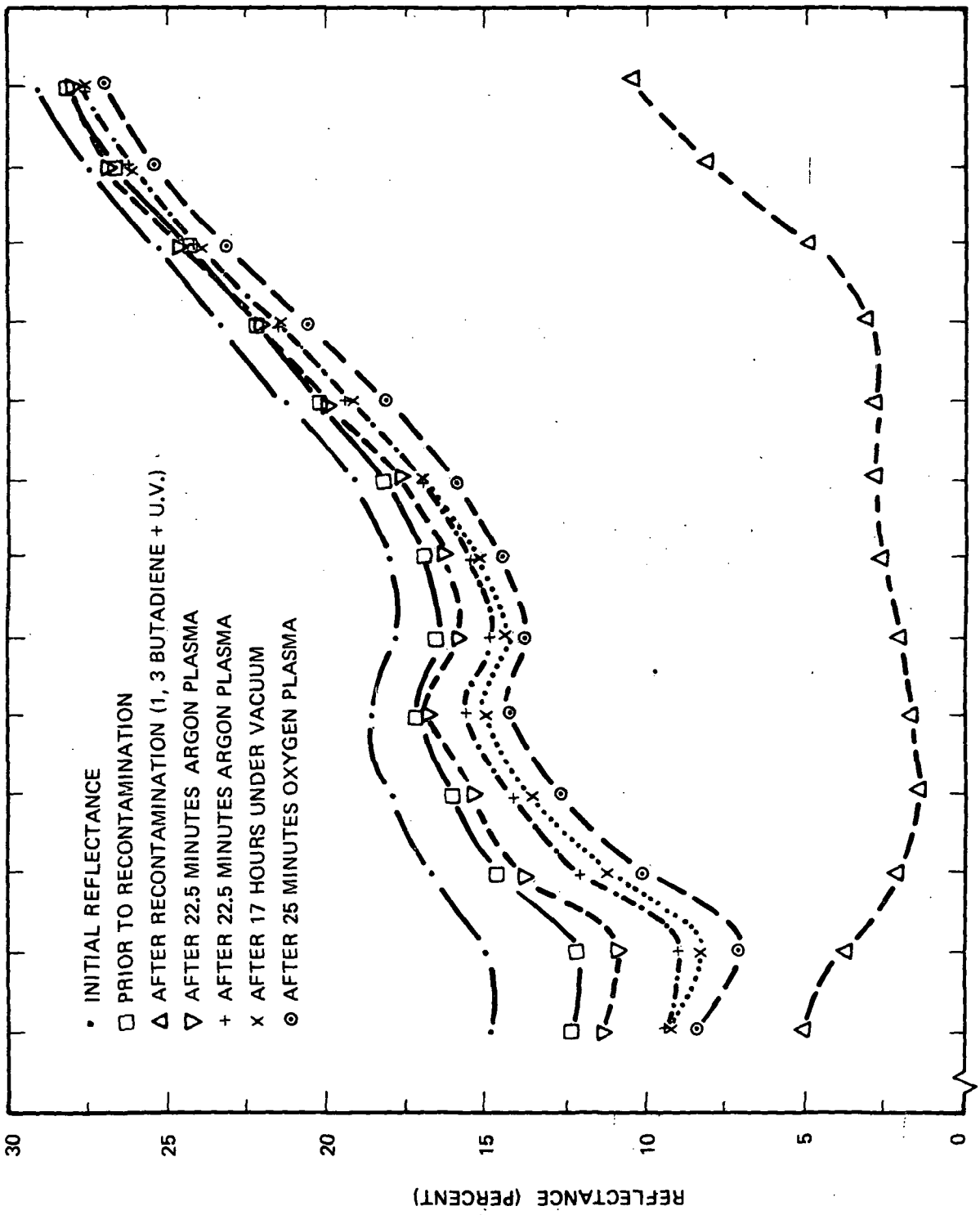

Figure 5: REFLECTANCE DATA FOR GOLD COATED MIRROR Au7

it has been exposed to air while the vacuum chamber quartz U.V. window was being cleaned. This degradation might have been due to contamination from the solvents (ethanol and methylethylketone) used in cleaning the window.

Following the argon plasma cleaning, sample Au7 was recontaminated and exposed to ambient air for 20 hours. This ambient air exposure had no measurable effect on the degraded U.V. reflectance of the sample. Figure 5 shows that a 22.5 minute argon plasma exposure nearly restored the U.V. reflectance to that prior to recontamination. However an additional 22.5 minute argon plasma exposure produced degradation in the reflectance. Leaving the sample exposed to vacuum for 17 hours resulted in slight further reflectance degradation. This result is consistent with that observed for sample Au6. Apparently contaminants from the closed-off vacuum system deposit on the cooled sample to some degree. Figure 5 also shows that an additional 25 minute exposure to an oxygen plasma produced further reflectance degradation. This along with the results of the previous argon plasma exposure indicates 'over cleaning' of the sample.

Table 3 gives the 'log of events' for the experiment using platinum coated mirror Pt 14. The reflectance data for this sample are shown in Figures 6 and 7. Figure 6 shows that the degradation, produced by the butadiene contaminant film, was almost eliminated with a single 7.5 minute oxygen plasma exposure. A second exposure increased the reflectance beyond that prior to contamination. A third exposure produced little effect. The sample was then contaminated again and left under vacuum, with the vacuum system closed-off, for 65 hours. Figure 7 shows the amount of degradation still present after this extended period under vacuum. The degree of degradation is less than that shown in Figure 6. Some of the contaminant film may have been removed by the extended period under vacuum, however, it should be noted that the initial contamination

TABLE 3 LOG OF EVENTS FOR PLATINUM MIRROR - Pt 14

EVENT	COMMENTS
Contamination	
Exposed to U. V. radiation at 5 torr 1,3 butadiene for 100 minutes	QCM response indicated film deposition of about 3.2×10^{-6} gm/cm^2. U.V. reflectance degraded by more than factor of 2.
Oxygen Plasma Exposures	
1st exposure 7.5 minutes 2nd exposure 7.5 minutes 3rd exposure 7.5 minutes	First exposure nearly restored U.V. reflectance. Second exposure restored reflectance beyond original. Third exposure caused very little change.
Contamination	
Exposed to U.V. radiation at 5 torr 1,3 butadiene for 60 minutes	QCM response indicated film deposition of about 0.5×10^{-6} gm/cm^2.
Left at vacuum for 65 hours	Chamber pressure rose to 5 torr.
Oxygen Plasma Exposures	
1st exposure 7.5 minutes 2nd exposure 7.5 minutes	First exposure restored reflectance to that prior to recontamination. Second exposure produced little change.
Plasma Cleaned Monochromator Optics	15 minute oxygen plasma exposure. U.V. reflectance slightly below previous measurement.

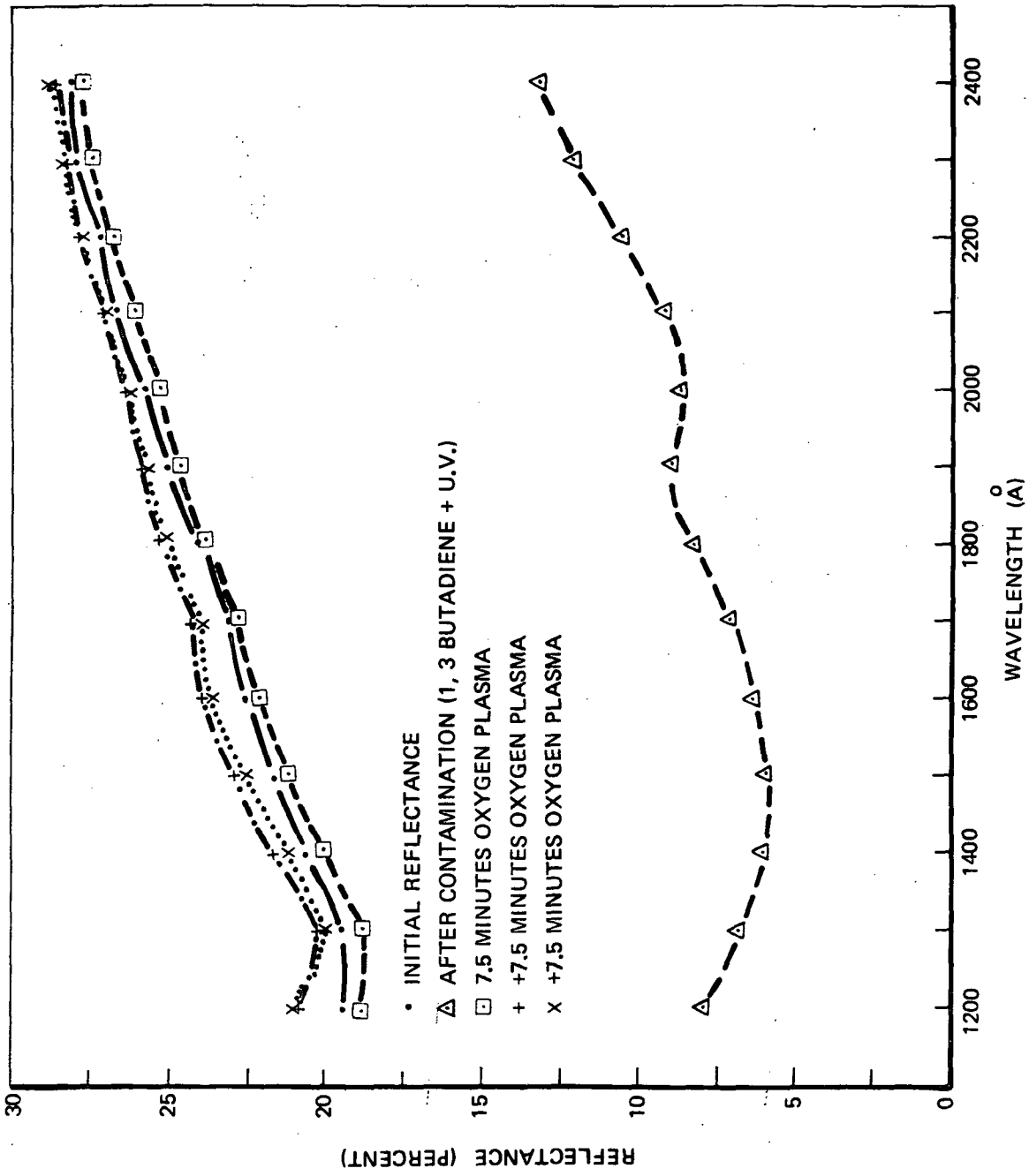

Figure 6: REFLECTANCE DATA FOR PLATINUM COATED MIRROR Pt 14

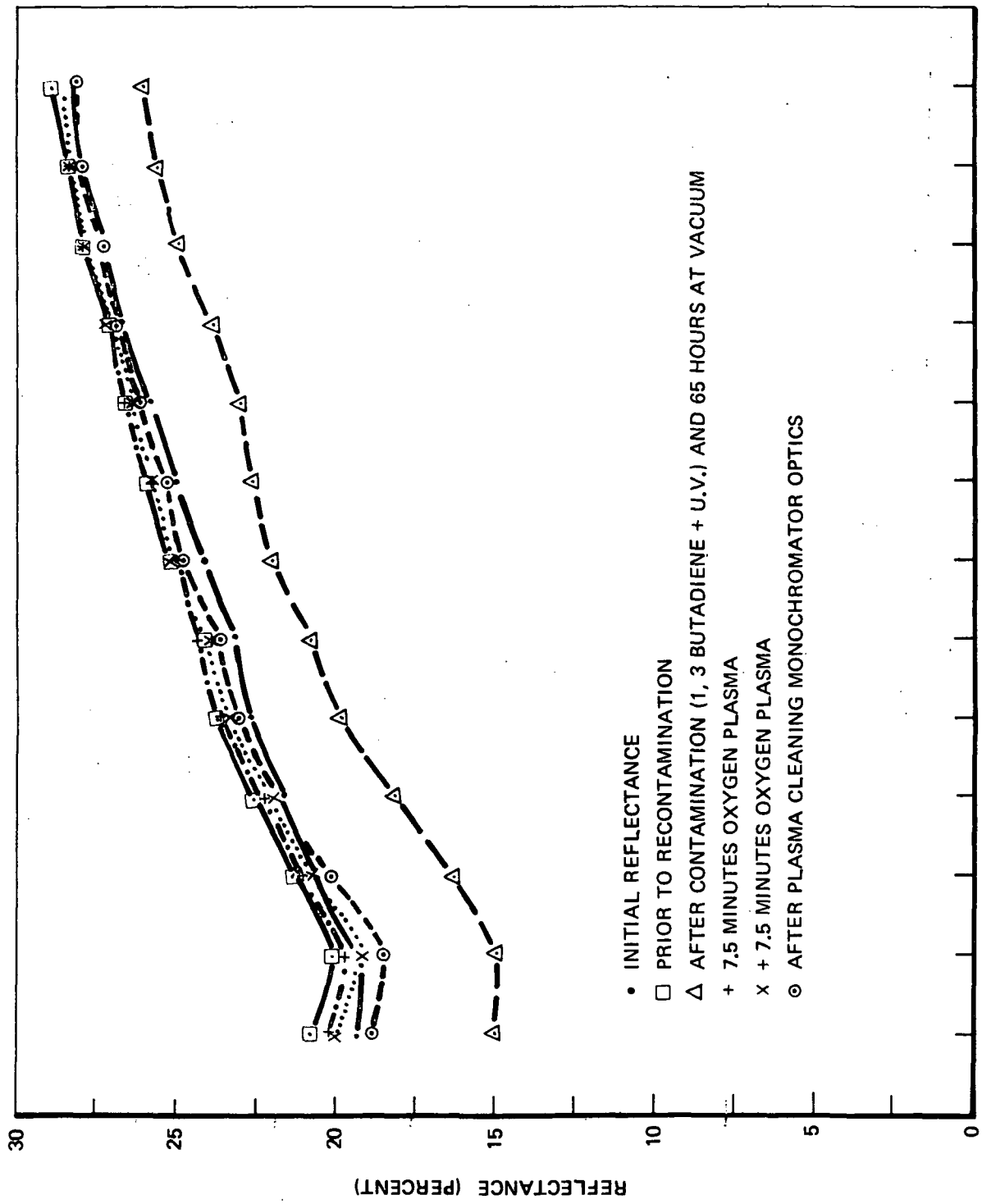

Figure 7: REFLECTANCE DATA FOR PLATINUM COATED MIRROR Pt 14

produced about six times the amount of contaminant film that was produced in the recontamination. Consequently the primary reason for the differences in degree of degradation is probably the amount of contaminant film deposition rather than the effects of the extended vacuum exposure. The reflectance (prior to recontamination) was essentially restored with a 7.5 minute oxygen plasma exposure. A second exposure produced little change in the reflectance. However, after plasma cleaning the monochromator optics, a remeasurement of the reflectance showed slight degradation in the 1200-1500Å wavelength region. This again indicates that the degradation of the monochromator optics influences the reflectance measurements in the shorter wavelength region.

3.2 Carbon Cleaning Experiments

The experiments discussed in the preceding section showed argon to be as effective as oxygen in plasma cleaning butadiene contaminant films. Subsequently experiments were conducted to see if plasma cleaning of carbon deposits would be affected by the type of gas used. These experiments used a Quartz Crystal Microbalance (QCM). The quartz crystal was coated with gold which was in turn overcoated with about 200Å of vacuum deposited carbon.

Figure 8 shows the effects of plasma exposure on the frequency of the QCM. A reduction in the QCM frequency indicates a removal of mass from the crystal (removal of 1 microgram/cm^2 changes the frequency by about 50 Hertz). All plasma exposures used the 'ignited plume' mode of operation. The initial series of tests used helium, oxygen and hydrogen plasmas. Figure 8 (top three curves) indicates that oxygen and hydrogen are about equally effective in removing carbon. Helium appeared to be less effective. In order to verify this result a second series of tests were made using helium and oxygen. Figure 8 (bottom four curves) indicates that helium is as effective as oxygen in

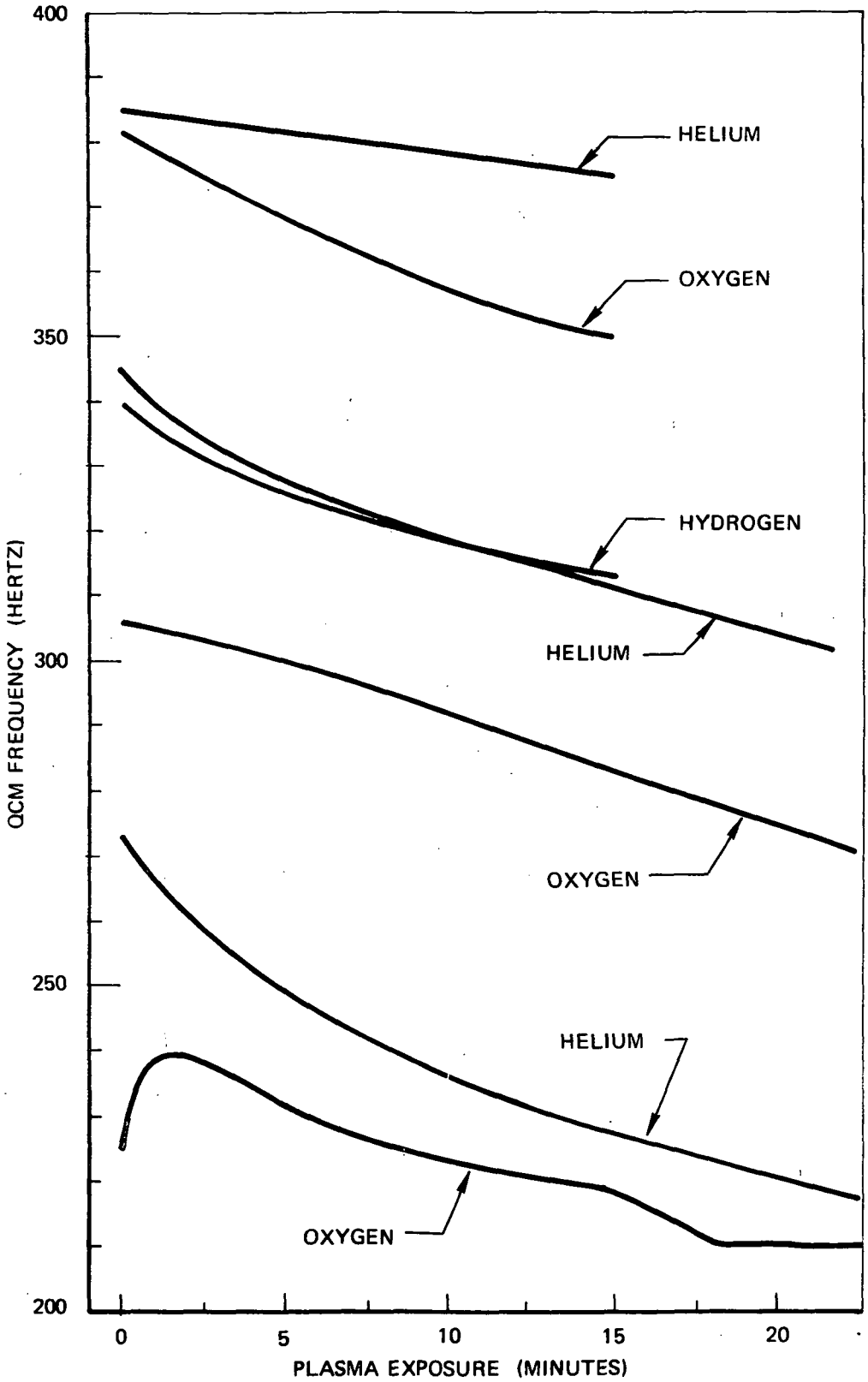

Figure 8: PLASMA CLEANING OF CARBON COATED QCM

removing the carbon. It should be noted that the final plasma exposure, using oxygen, removed the remaining carbon from the QCM. These data show that helium, oxygen and hydrogen plasmas are equally effective in removing carbon. The apparently less effective cleaning during the initial exposure, using helium, may just be an anomaly or may have something to do with it being the first exposure to a plasma. These results add further evidence that normal chemical reaction, between plasma and surface resulting in volatile products, is not the basic cleaning process.

3.3 Silicone Cleaning Experiments

During the present reporting period several silicone cleaning experiments were conducted. Samples were contaminated by outgassing silicone compounds (RTV3116 and RTV602) in a heated tube attached to the chamber contamination inlet port. The contaminant film deposition was monitored using a gold coated QCM. In the initial experiment the QCM was contaminated with the outgassed products from RTV3116. The QCM response indicated a contaminant film deposition of about 1.8×10^{-6} gm/cm^2. The QCM was subsequently exposed to oxygen, argon and hydrogen plasmas. The QCM response during these exposures gave no indication of contaminant film removal. Consequently it was decided to contaminate MgF_2/Al mirrors and use the U.V. reflectance data to assess the effects of plasma exposure.

The first MgF_2/Al mirror (MgF113) was exposed to the outgas products of RTV3116 for 24 minutes. The QCM response indicated a contaminant film deposition of about 2×10^{-6} gm/cm^2. The U.V. reflectance of the sample was severely degraded in the 1200-1400 Å wavelength region and was increased in the 1800-2400 Å wavelength region (Figure 9 shows the reflectance data for sample MgF113). As shown in Figure 9, some of the contaminant film appears to have evaporated during a 17 hour period under vacuum. The sample was then subjected to a series of helium

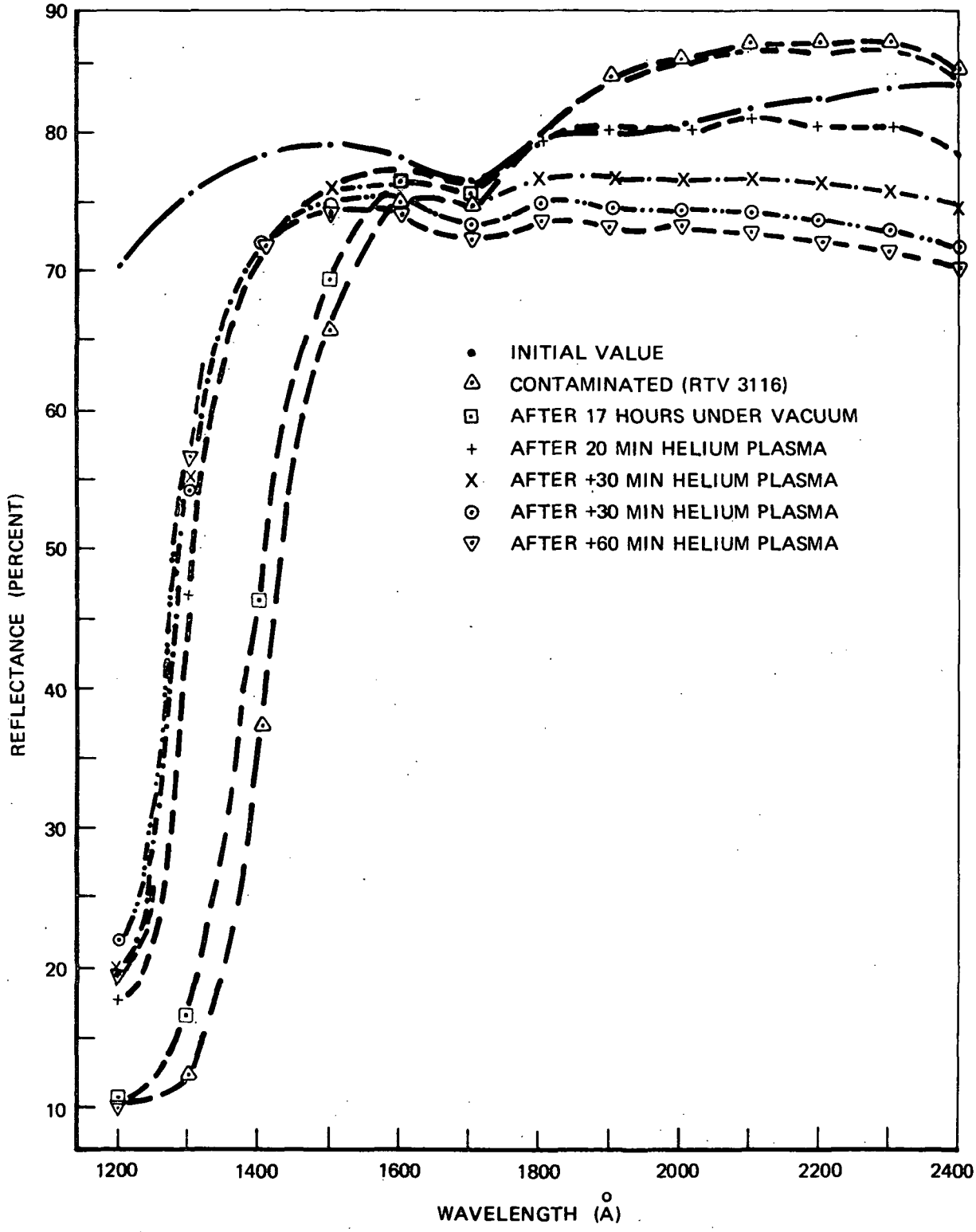

Figure 9: REFLECTANCE DATA FOR MgF_2/Al MIRROR MgF 113

plasma exposures (the first for 20 minutes then four of 30 minutes each). These exposures increased the reflectance at the shorter wavelengths, however, that at the longer wavelengths was decreased (see Figure 9). The restoration at the shorter wavelengths appeared to be limited, whereas, the degradation at the longer wavelengths appeared to be progressive. A subsequent 15 minute exposure to an oxygen plasma (results not shown in Figure 9) resulted in very little change in reflectance except for a slight degradation in the 1200-1400Å wavelength region.

A second MgF_2/Al mirror (MgF114) was exposed to the outgas products of RTV3116 for 30 minutes. The QCM response indicated a contaminant film deposition of about $1.5 \times 10^{-6} gm/cm^2$. The effects of the contaminant film on the U.V. reflectance are similar to those observed for sample MgF113 (see Figure 10). The data in Figure 10 also indicate that some of contaminant film evaporated, as before, during a 19 hour exposure to vacuum. This sample was subjected to a series of hydrogen plasma exposures of 7.5, 10, 12 and 7.5 minutes. As with the helium plasma exposures of MgF113, the hydrogen plasma exposures increased the reflectance of MgF114 at the shorter wavelengths and decreased it at the longer wavelengths. However the hydrogen plasma produces more restoration at the shorter wavelengths and less degradation at the longer wavelengths. Subsequent to this series of plasma exposures the sample was left under vacuum for 18 hours and then subjected to three additional plasma exposures, (12 minutes using hydrogen, 9 minutes using oxygen and 7.5 minutes using helium). Figure 11 shows the reflectance data for these cases. The hydrogen plasma exposure resulted in a slight improvement at the shorter wavelengths and slight degradation at the longer wavelengths. The oxygen plasma exposure resulted in slight further degradation. The helium plasma exposure produced a significant further degradation of the reflectance.

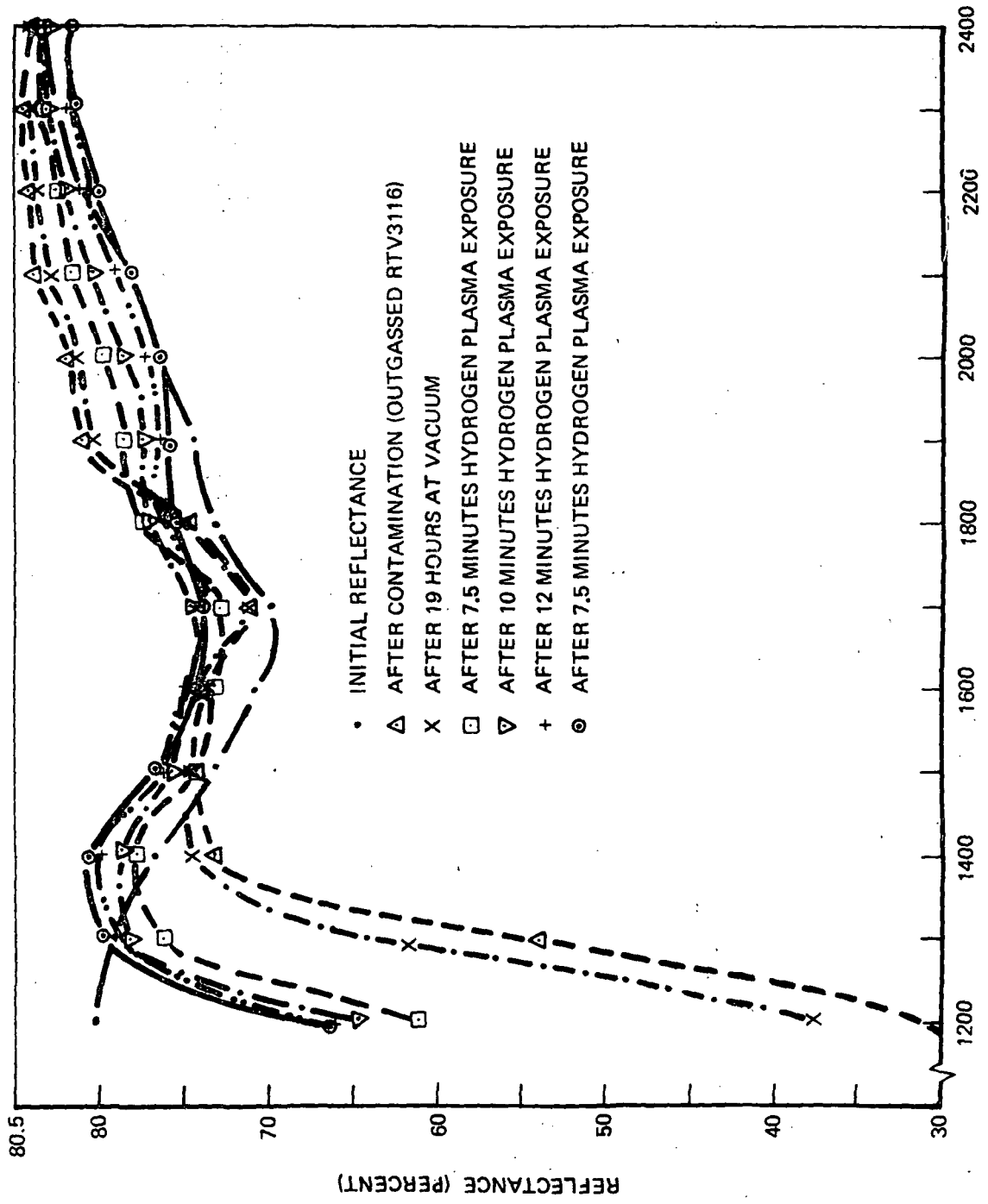

Figure 10: REFLECTANCE DATA FOR MgF_2/Al MIRROR MgF114

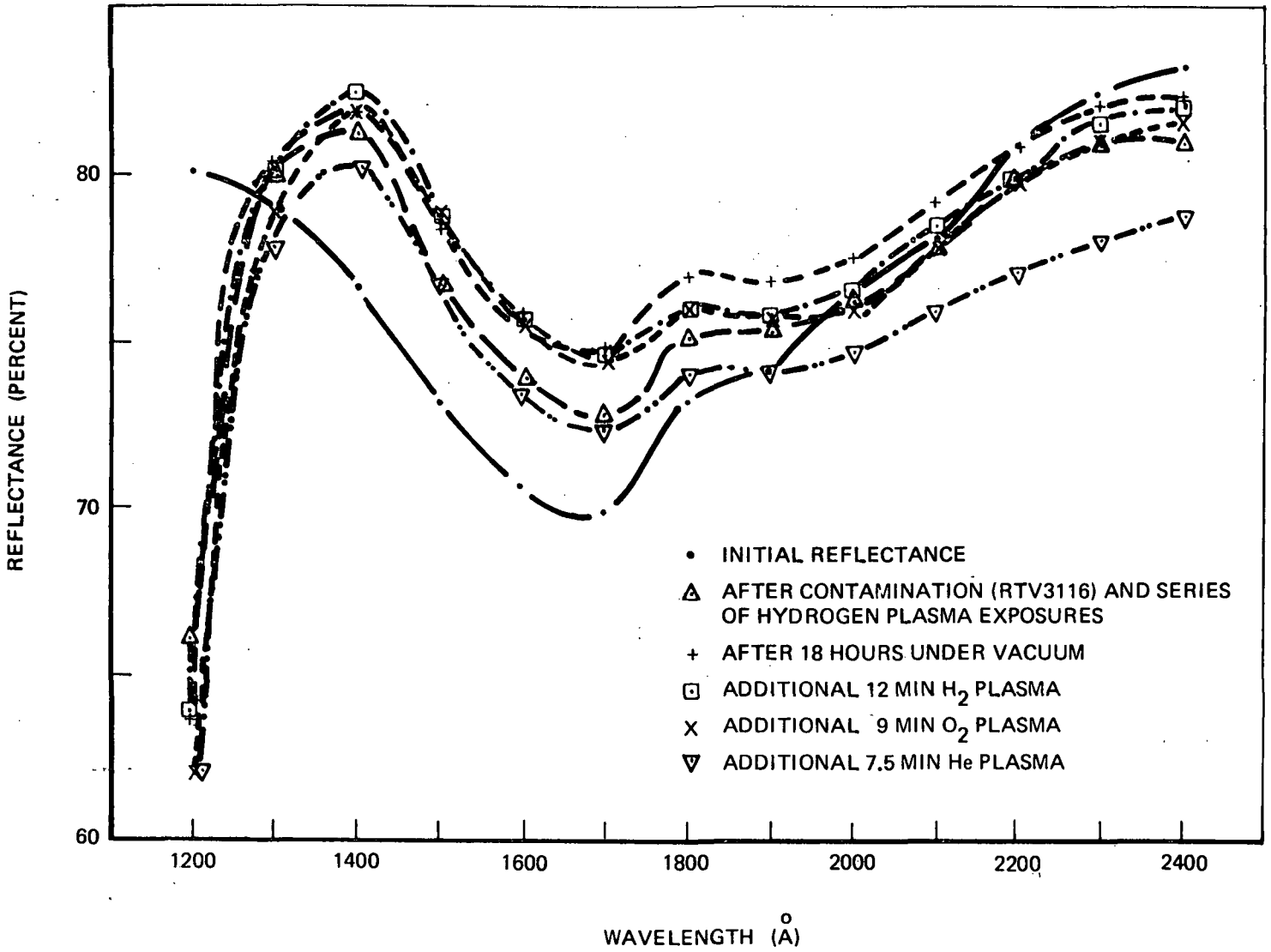

Figure 11: REFLECTANCE DATA FOR MgF_2/Al MIRROR MgF114

These initial experiments showed that the U.V. reflectance of MgF_2/Al coated mirrors, contaminated with the outgas products from RTV3116, could be partially restored using helium and hydrogen plasmas. Further experiments were conducted using the outgas products of RTV602 to contaminate MgF_2/Al coated mirrors. The silicone compounds in RTV602 are similar to those in S-13 white paint (a spacecraft coating). These experiments also utilized a sapphire window in the in-situ monochromator to extend the reflectance measurements to 3000 Å. Two mirrors were used in these tests. The first mirror MgF116 was exposed to the RTV602 outgas products for 27 minutes. The QCM response indicated a contaminant film deposition of about 2.2×10^{-6} gm/cm^2. During this exposure a gold coated glass slide was also contaminated. This slide was placed in the chamber contamination inlet port so that a thick contaminant film would result. Subsequent analysis of this film revealed the presence of dimethylsiloxane. Subsequent to contamination, sample MgF116 was exposed to U.V. radiation for 30 minutes. This exposure produced some restoration of the reflectance. (The reflectance data for MgF116 are shown in Figures 12 and 13). The sample was then subjected to a series of hydrogen plasma exposures. Figure 12 shows that these exposures produced a significant restoration of the reflectance at shorter wavelengths and Figure 13 shows a progressive degradation at the longer wavelengths. Two additional hydrogen plasma exposures totaling 52.5 minutes produced little change in the shorter wavelengths and slight further degradation at the longer wavelengths. (These data are not shown in Figures 12 and 13).

The second mirror MgF117 was first exposed to the outgas products of RTV602 for 75 minutes with a resultant indicated film deposition of only about 0.8×10^{-6} gm/cm^2. A subsequent reflectance measurement revealed negligible contamination. During this initial contamination attempt the chamber pressure

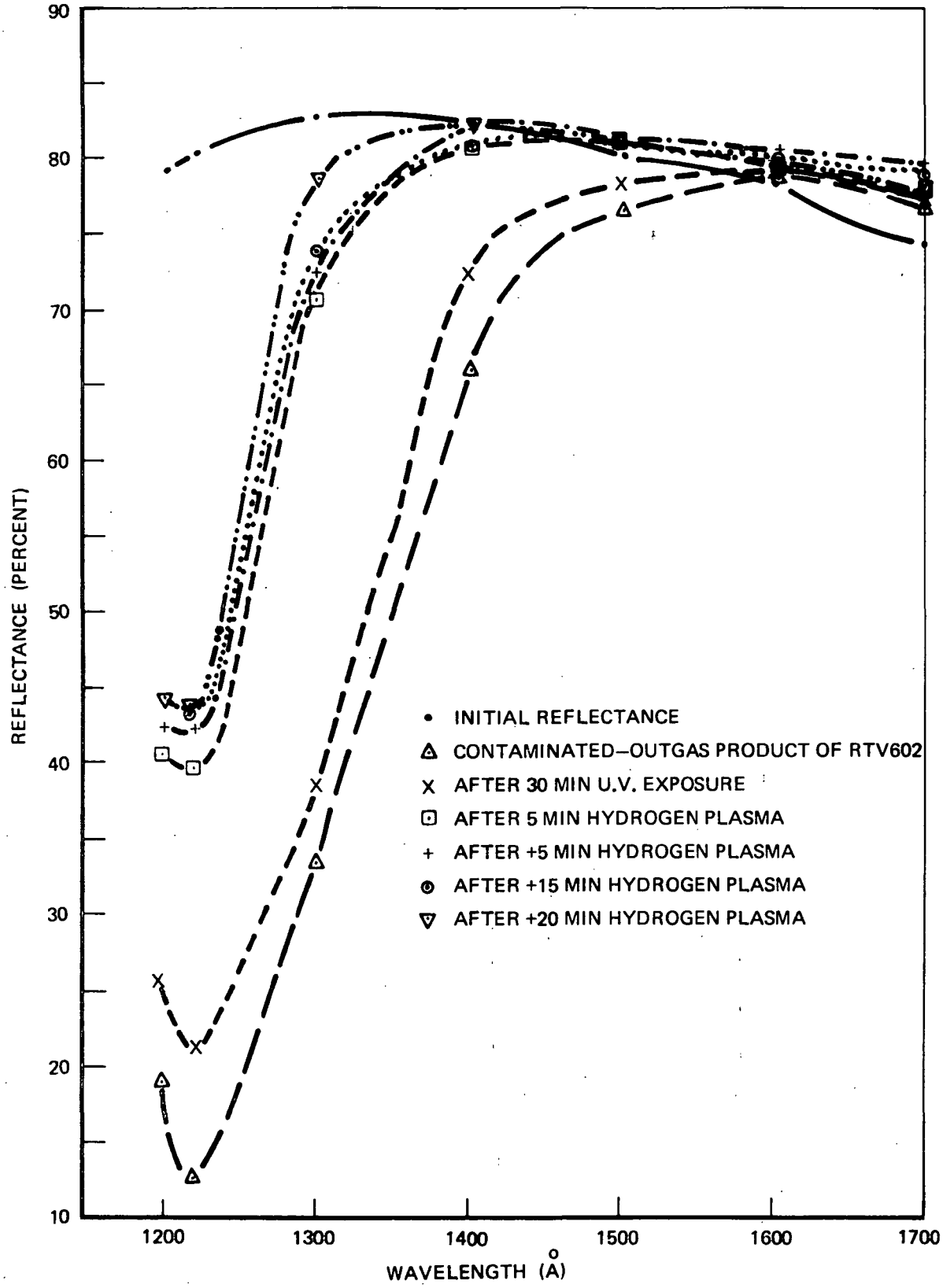

Figure 12: REFLECTANCE DATA FOR MgF_2/Al MIRROR MgF116

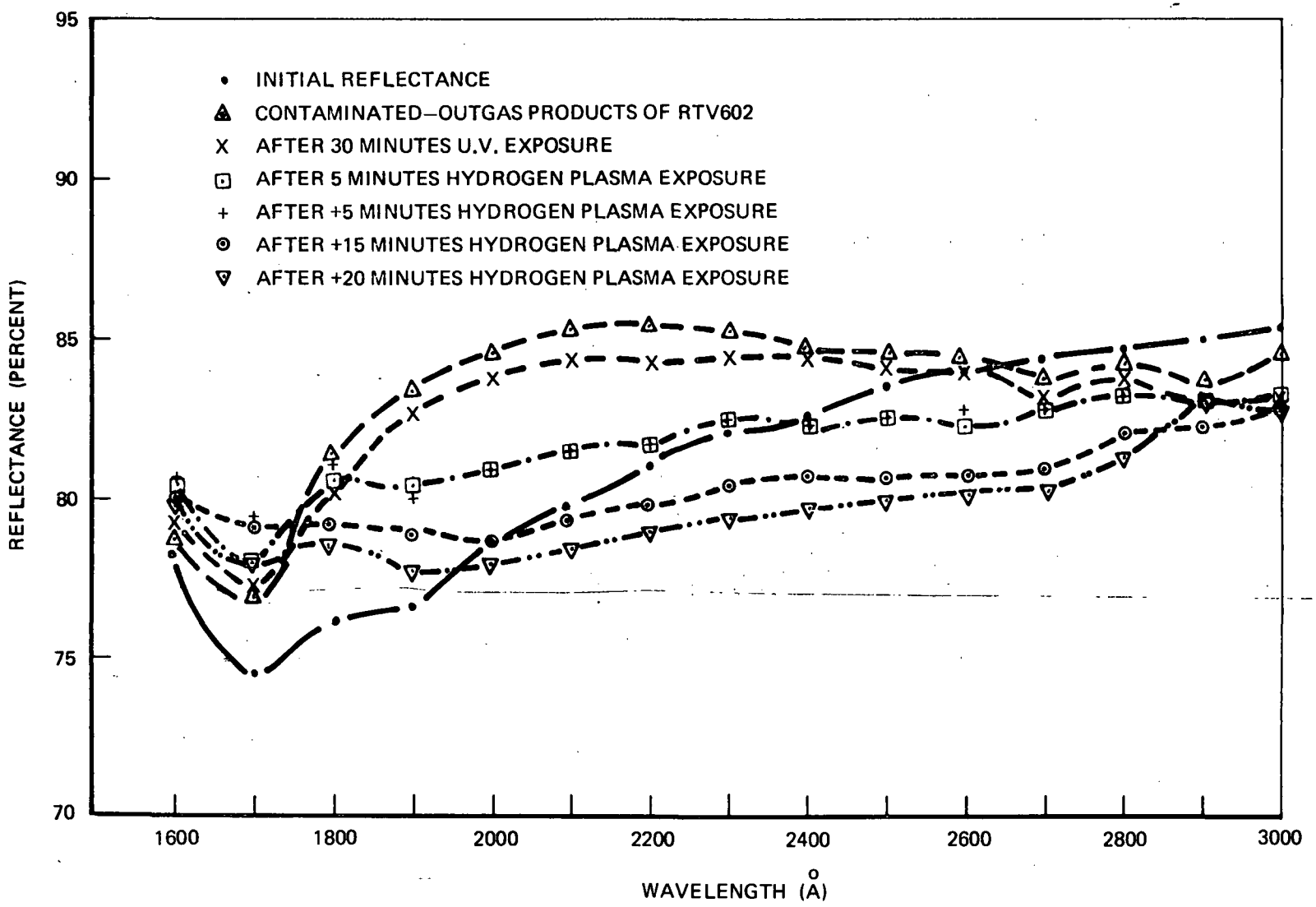

Figure 13: REFLECTANCE DATA FOR MgF_2/Al MIRROR MgF116

was above 1μ. At first this pressure was attributed to outgassing effects, however, a system leak was discovered later. After sealing the leak the sample contamination procedure was repeated. A 30 minute exposure then resulted in a total indicated film deposition of about 3×10^{-6} gm/cm^2. The sample was then exposed to a series of oxygen plasma exposures. Figures 14 and 15 show the reflectance data for mirror MgF117. The degraded reflectance, prior to plasma exposure, shows minimums at about 1220, 1270 and 1320 Å, whereas, that for MgF116 showed only the 1220 Å minimum. This difference might be explained by the fact that, after contamination, MgF116 was left under vacuum and then let up to air prior to the reflectance measurement, or by the fact that MgF117 had a somewhat thicker contaminant film. Figure 14 shows that the minimums at 1270 and 1320Å were removed by oxygen plasma exposure, however, the restoration at 1220 Å was much less than that obtained for MgF116 using hydrogen plasma exposure. The data shown in Figure 14 also show that the reflectance at the shorter wavelengths is initially increased and then progressively degrades with continued exposure to an oxygen plasma. Figure 15 shows somewhat the opposite effect at the longer wavelengths.

The silicone contamination experiments conducted during the present reporting period all show that a contaminant film deposition on MgF_2/Al mirrors on the order of 2×10^{-6} gm/cm^2, produces a strong absorption band in the 1200-1300 Å wavelength region. This degradation was partially removed using the plasma cleaning technique. Helium, hydrogen and oxygen plasma exposures all resulted in some restoration of the reflectance at the shorter wavelengths, however, hydrogen appeared to give the best results. The silicone contaminant film increased the reflectance at the longer wavelengths (1700-3000Å). After contamination, plasma exposure degraded the reflectance at the longer wavelengths, with helium plasma exposure producing the most severe degradation.

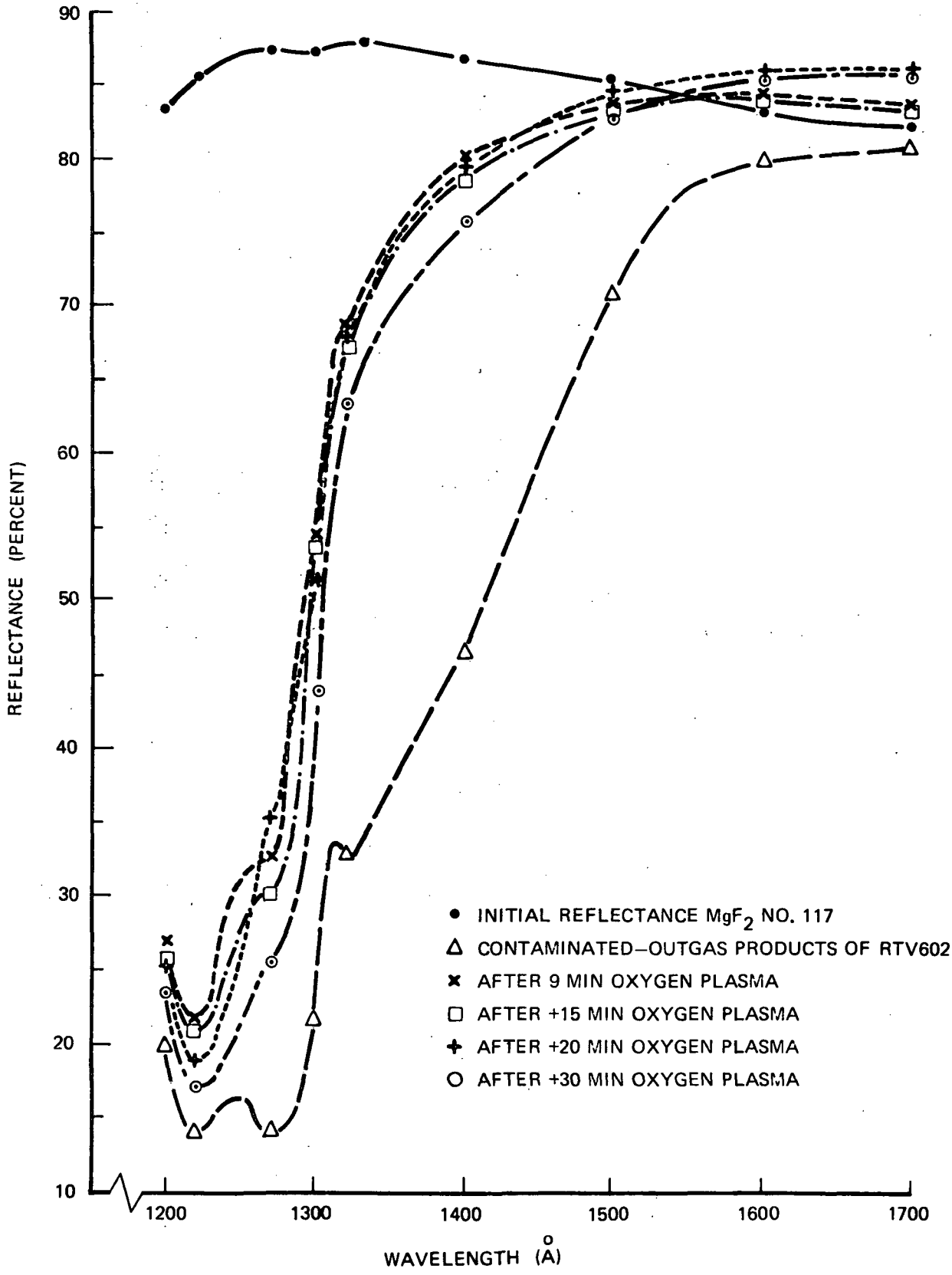

Figure 14: REFLECTANCE DATA FOR MgF_2/Al MIRROR MgF117

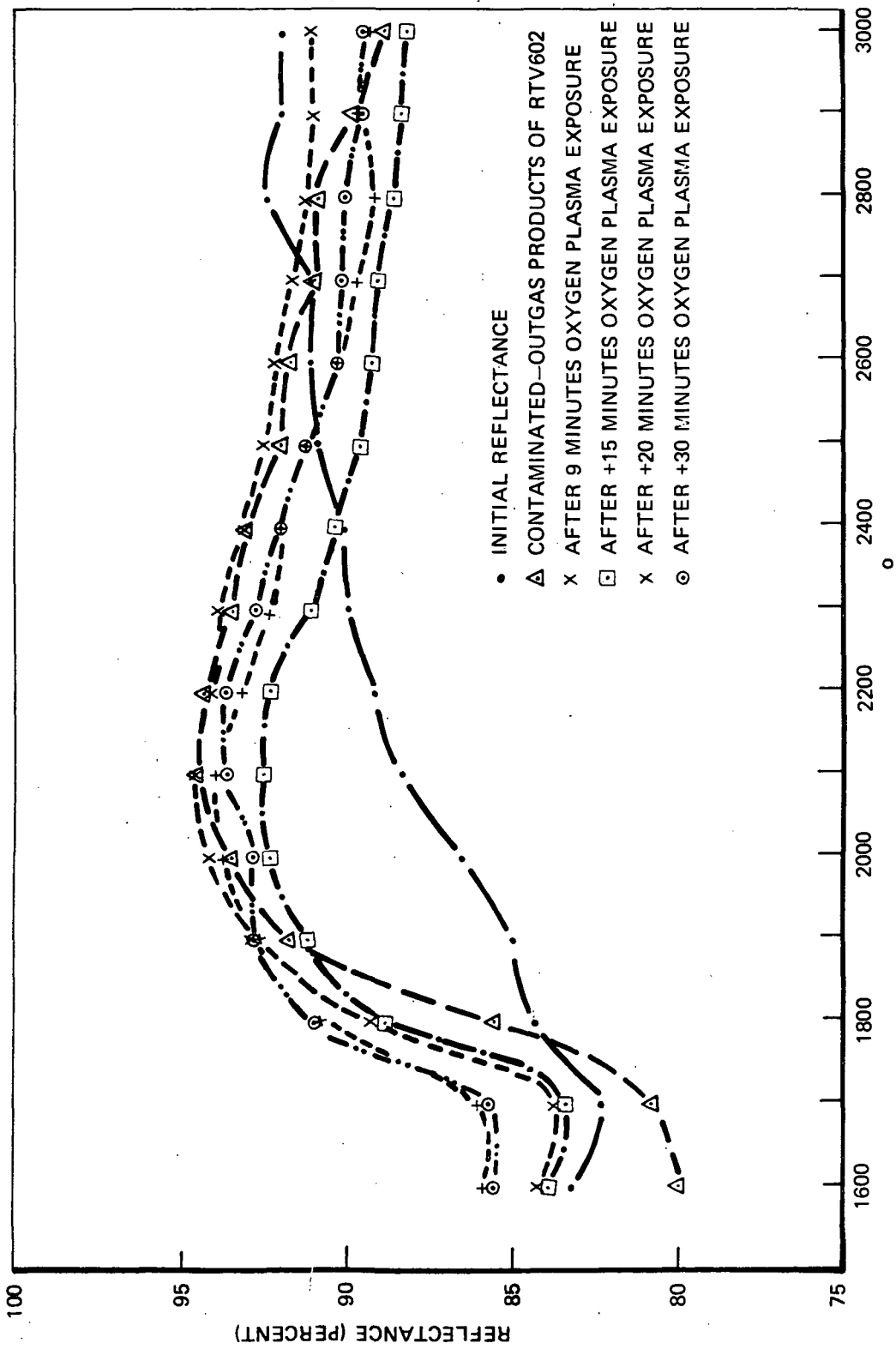

Figure 15: REFLECTANCE DATA FOR MgF_2/AL MIRROR MgF117

4.0 PROGRAM PROGRESS

4.1 Schedule

The program schedule is shown in Figure 16. Solid bars on the schedule indicate work completed and non-solid bars represent work planned. The remaining work requires plasma tube operation for which the 'chamber discharge' phenomena is not present. Consequently this work is scheduled to use the plasma tube developed under companion contract NAS8-28270. No schedule delays are anticipated and the release of the final report is scheduled for August 30, 1973.

4.2 Expenditures

Cumulative expenditures as of June 1, 1973 were about $65,200. Approximately $11,000 will be required to complete the program, including the fee. No cost overrun is anticipated.

4.3 Future Work Planned

During the final reporting period the cleaning rate distribution in a plane perpendicular to the plasma tube will be determined, and the contamination on surfaces adjacent to those being cleaned will be evaluated.

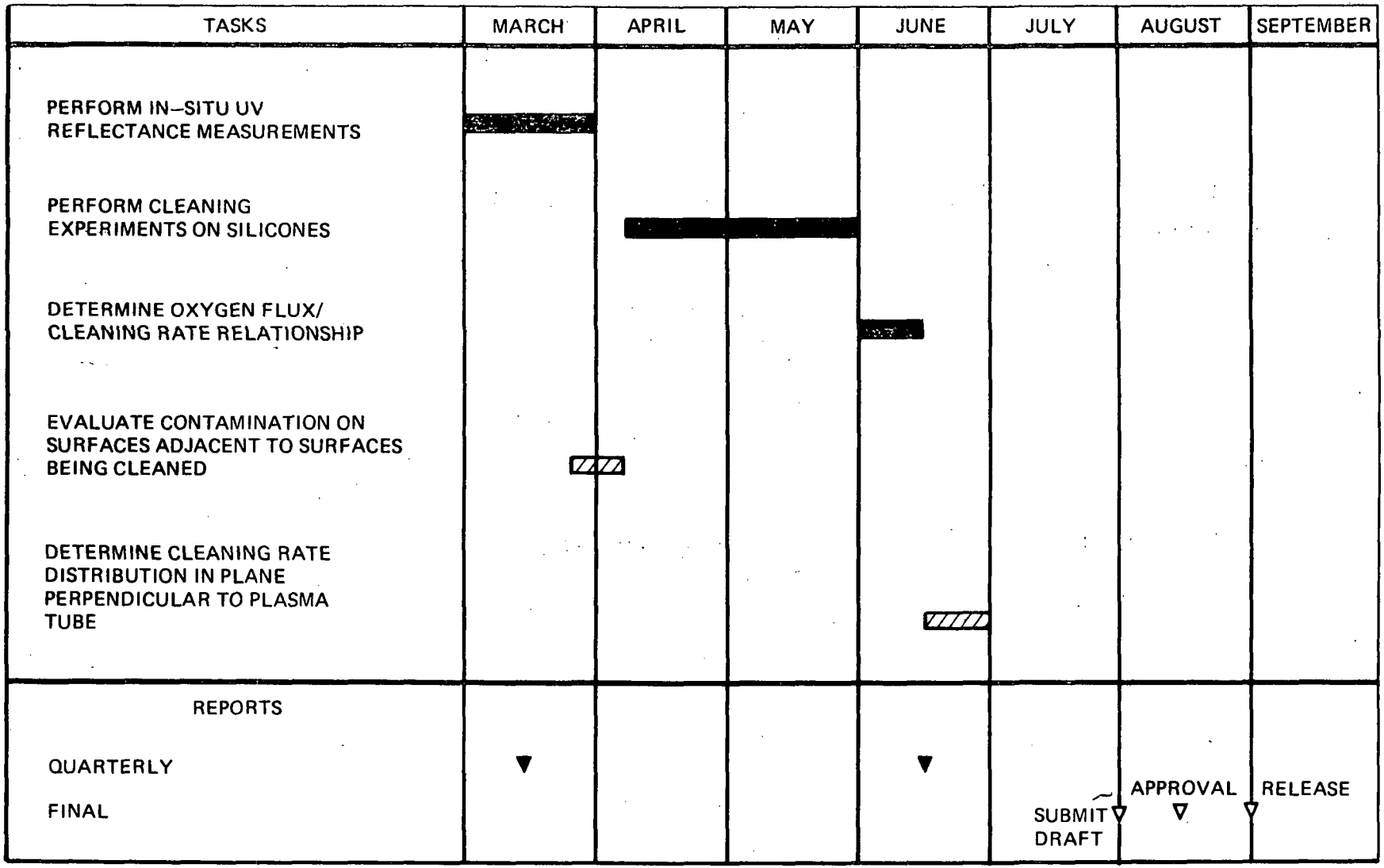

Figure 16: SCHEDULE FOR MODIFICATION NO. 4 TO NAS 8-26385

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