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REDEPOSITION OF VACUUM OUTGASSED PRODUCTS

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TECHNICAL MEMORANDUM X-53801

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ABSTRACT

A quartz crystal microbalance (QCM) with a sensitivity of 10^{-9} gm/cm^2 is used to determine the quantity of material redeposited from an outgassing material source in a vacuum environment. The vacuum system was designed for the QCM and materials testing and is free of organic background. Results of tests on 18 materials are presented in terms of film thickness as a function of temperature to 150°C. Both metallic and non-metallic materials were tested.

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PROPULSION AND VEHICLE ENGINEERING LABORATORY
RESEARCH AND DEVELOPMENT OPERATIONS

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REDEPOSITION OF VACUUM OUTGASSED PRODUCTS

SUMMARY

A quartz crystal microbalance (QCM) is used to determine the amount of material redeposited from an outgassing material source in a vacuum environment. The QCM is used for high temperature applications and still maintains a sensitivity of 10^{-9} gm/cm^2. The influence of the sample heater, titanium sublimation pump, ion pump and residual gases on the QCM is discussed.

A special vacuum system designed for contamination studies is described. The system has a pumping speed of 400 liter/second and an ultimate pressure of 10^{-9} torr. A temperature stabilized holder for the quartz crystal microbalance is shown to alleviate frequency shifts of the crystals due to temperature changes.

Results of seven classes of materials evaluated are presented. Film thicknesses were calculated from the known specific gravities of the materials and the characteristics of the QCM. In addition the maximum amount of oxide present in the metallic films is calculated. Materials evaluated included metals, coatings, potting compounds, elastomers, adhesives, plastics, and foams.

INTRODUCTION

The outgassing characteristics of materials in the space environment is of primary importance in selecting a material that will not be detrimental to the accomplishment of the mission. With the advent of orbiting telescopes and other sophisticated optical systems, it has become necessary to insure that no redeposition of these outgassed products occurs. A film deposited on an optical surface can change the surface's transmission-reflectance characteristics sufficiently to render it useless for the intended measurement. Changes in electrical resistivity sufficient to alter circuit characteristics could occur in instrument packages as a result of vapor condensation between two electrical contacts.

This study was undertaken to determine the amount of outgassed material redeposited in the thermal/vacuum environment. The rate of redeposition was monitored with a quartz crystal microbalance to determine the suitability of a material for use.

BACKGROUND

Material outgassing is of several types. It can be the desorption of surface adsorbed atmospheric gases or the release of solvents of high vapor pressure which are both lost relatively quickly. Gases that have been chemisorbed, i.e., have chemically reacted with the surface interface, outgas less readily than adsorbed atmospheric gases and water vapor. The most tightly bound molecules are those that comprise the structure of the material itself. For molecular bonds to be broken, energy must be admitted to the sample through heating or UV radiation. The additional energy may cause thermal degradation of the material, i.e., breaking of bonds and the release of parent molecules.

Outgassing constituents may recondense on any surface in line of sight of the source. This redeposition process is in effect an uncontrolled thin film deposition. Following impingement on the surface the outgassed molecules may (1) adsorb onto the surface and adhere permanently to the substrate, (2) adsorb and reevaporate (or desorb) in a finite amount of time (3) reflect completely from the substrate. Forces at the interface are sufficient to make a total reflection the most improbable event.

The probability that an adsorbed molecule reevaporates is defined as W the reevaporation probability where

$$W = V \exp \left[\frac{Qad}{KT}\right] \qquad (1)$$

V is the frequency factor for an adsorbed atom, Q_{ad} is the binding energy of the molecule to the substrate, and T is the absolute temperature of the adsorbed molecule [1]. It should be noted that T is usually a temperature that is intermediate between that of the source and substrate.

Q_{ad}, the binding energy, is the negative of the adsorption energy, and is a factor in determining the residence time of the molecule on the substrate. Residence time is inversely proportional to the temperature of the substrate, i.e., a very low temperature substrate can approach a sticking probability of unity. If the outgassed molecule reaches thermal equilibrium with the substrate immediately upon impingement, then the temperature of the molecule is that of the substrate. The ratio Q_{ad}/KT infers that if $Q_{ad} \gg KT$ then reevaporation is very unlikely. This can occur with either a low temperature substrate or a metal redepositing on a metal substrate where Q_{ad} is very large.

Redeposition on an optical surface implies a large degree of chemical inertness between the substrate, usually glass or plastic, and the impinging molecule. In this case, Q_{ad} is very small and W becomes large.

The product of the probability of reevaporation and the number of adsorbed atoms defines the number of atoms returning to the vapor phase per second.

$$N\uparrow = N_{ad}W = N_{ad}V \exp\left[\frac{Q_{ad}}{KT}\right] \quad [2]$$

where N_{ad} is the number of adsorbed atoms per square centimeter. In a controlled evaporation a steady state will be reached where $N\uparrow = N\downarrow$. This state is usually not attained from an outgassing sample since the constituent of lower vapor pressure will soon be depleted.

In the immediate environment of the material, residual gases may be present in sufficient amounts to react with the desorbed molecules. The number of molecules impinging on the substrate per unit time is given by

$$V = 3.51 \times 10^{22} \frac{P}{\sqrt{M'T}} \qquad (2)$$

where M' is the molecular weight of the residual gas, T is the absolute temperature of the gas, and P is the pressure in torr [2]. The number of molecules of outgassing material adhering to the surface is

$$\lambda = \frac{N_o \rho}{M} \left(\frac{dT}{dt}\right) \qquad (3)$$

where N_o is Avagadro's number, ρ = film density, M = molecular weight of the material, and $\frac{dT}{dt}$ = deposition rate in Å/sec [2]. The ratio of $\frac{V}{\lambda}$ is K which is the maximum impurity content from reaction with residual gases.

$$\frac{V}{\lambda} = K = 5.82 \times 10^{-2} \left(\frac{M}{\rho}\right)(M'T)^{-1/2} \left(\frac{P}{dT/dt}\right) \qquad (4)$$

assumes a sticking probability of one.

Single atoms impinging on a substrate will not tend to form a permanent deposit without the formation of aggregates. Aggregates are created through migration and subsequent collision of adsorbed atoms on the interface. An aggregate will tend to reevaporate less quickly than a single atom since the atoms in the aggregate are bound by the condensation energy of that particular constituent. However, a smaller aggregate with a high surface to volume ratio has a greater total surface energy. This makes the aggregate less stable due to a higher vapor pressure than the bulk material and the aggregate may dissociate. That is to say that there exists a critical size of an aggregate for minimum stability. A permanent deposit results when an aggregate of critical size or larger is present on the substrate. On a metal substrate such as that of the quartz crystal microbalance, Q_{ad} is large and may yield a critical size of less than two atoms. This means that a permanent deposit will result in a continuous film after only a few monolayers of material have impinged on the substrate.

EXPERIMENTAL APPARATUS

Vacuum System

An organic-free vacuum system, was designed to insure that no contamination of the microbalance or sample from diffusion pump oil vapor or elastomeric "O" rings could occur. This system is shown in Figures 1 and 2. All materials employed in the chamber were constructed of 304 stainless steel for minimum outgassing. All gaskets and sealing surfaces are OFHC copper.

The redeposition chamber is a 12" high x 12" diameter right circular cylinder sealed with a Wheeler flange. The interior surface is electropolished which aids in rapid desorption of gases from the wall during pumpdown and facilitates cleaning of the surface after a sample has been tested. The chamber is instrumented with a nude configuration Bayard-Alpert gauge, thermocouples to measure temperatures of the sample and balance, and the quartz crystal microbalance. A 20 liter/second getter-ion pump and two cryogenically cooled sorption pumps are used to evacuate the chamber. The use of two separately valved sorption pumps enables the operator to completely trap helium and argon gases, which are pumped very slowly by an ion pump, in the first sorption pump. The second pump can then attain a chamber pressure of 2 millitorr or less where the ion pump may be started.

The effective pumping speed of the 20 liter/second ion pump is effectively increased to 400 liter/second when the internal titanium sublimation pump (TSP) is added. An outboard water-cooled TSP is also available for additional pumping capability if required. The total system has an ultimate pressure of 10^{-9} torr.

All parts used in the experimental set-up are also electropolished, to diminish the initial gas load and permit easier clean-up after a test. Full bakeout of the system is used periodically to insure there is no buildup of organic background molecules which might affect the test results.

Quartz Crystal Microbalance

A specially fabricated water-cooled quartz crystal microbalance was used to monitor deposition of the outgassed material. This is shown in Figure 3. The deposition rate is determined by recording the beat frequency between two AT cut quartz crystals. These crystals are .006 inches thick, one coated on one side with gold and the other with chromium. The crystals are used in selected, matched pairs such that there is a beat frequency of at least 5 KHz between the two crystals in a clean condition at thermal equilibrium. This prevents the two crystals from locking in and oscillating at the same frequency. The AT cut crystals have an extremely low frequency shift at ambient temperature. Therefore, more accurate results are obtained with a water-cooled crystal holder which maintains the crystals at ambient temperature. The metallic coating on the crystals is applied by sputtering in a glow discharge as the sputtered film is more adherent to quartz [3].

A block diagram of the microbalance circuit is shown in Figure 4 [4]. One crystal is exposed to the molecular flux evaporating from the material while the other is shielded. Each crystal is connected to a separate oscillator circuit. The signals from the two oscillators are then mixed to determine the beat frequency difference which is amplified and read out on a 50 Hz - 500 MHz range electronic counter.

An AT cut quartz crystal has the relation between its thickness and resonance frequency

$$f = \frac{1.675 \times 10^{13}}{h}$$

[4], where f is the frequency in Hertz and h is the crystal thickness in angstroms for a 10 MHz crystal. The 10 MHz crystal was chosen due to its high mass sensitivity and lateral dimension to thickness ratio [5]. For small changes in frequency and thickness,

$$\Delta f = \frac{-1.675 \times 10^{13}}{h^2} \quad \Delta h$$

As stated, the QCM utilizes ten megacycle crystals in which case

$$\Delta f = -5.97 \quad \Delta h$$

As molecules impinge on the crystal, the resonant frequency of the crystal changes in a manner that is equivalent to adding the same mass of quartz to the crystal. The ratio of the density of the deposited material to that of quartz must be considered as well as the exposed area of the crystal. The crystal diameter is 0.530 inches with 0.500 inches exposed surface. These factors yield,

$$\Delta f = -5.97 \left(\frac{\rho}{2.655}\right) \left(\frac{.500}{.530}\right)^2 \Delta h = -2\rho\Delta h$$

where ρ is the film density in grams/cc. For frequency changes of 100 KHz or less this relationship is accurate to 0.1%. The QCM can detect a mass change of 10^{-9} gm/cm^2 [5].

EXPERIMENTAL TECHNIQUE

A calibration test was made to determine the magnitude of the variables that affect the output of the quartz crystal microbalance. The sample heater was in position but no sample was present.

During the pumpdown procedure, the oscillator cable of the QCM was disconnected to prevent damage to its electronics package due to glow discharge in the ion pump. At a pressure of 2 x 10^{-6} torr, the sample heater was turned on to duplicate the sample heating cycle of the previous weight loss test. The weight loss test procedure employed a heating cycle of 2°C/minute, so the same thermal environment was chosen for the redeposition test.

The temperature of the sample heater and QCM was monitored by thermocouples located in three positions: (1) top of the heater-sample mount (2) lower plate of the crystal base (3) back plate of the crystal holder as shown in Figure 2. Initial outgassing of the heater created impinging residual gases on the quartz crystal face which was water-cooled. The effect was a short duration insignificant decrease in the output frequency as shown in Figure 5. It was determined that the TSP had very little effect on the beat frequency of the QCM. No film was deposited on the crystals from the titanium filaments.

The pair of quartz crystals are mounted on a common base to eliminate structural effects or orientation differences. The crystals were checked against a 10 MHz standard crystal oscillator to independently measure the frequency of the substrate crystal. To prevent the crystals from locking in and oscillating together, pairs were carefully matched to insure a frequency difference of at least 5 KHz.

The experimental set-up is shown in Figure 6. As shown, the sample is mounted in a shallow cavity directly above the heater. The crystal holder is mounted 2.5 inches directly above the sample, suspended by its cooling water lines. The location of the measuring thermocouples may also be seen. On one side of the crystal holder is a glass slide which is partially masked from the sample. This slide is used in conjunction with a multiple beam interferometer to determine total deposited film thickness as a rough check on QCM performance.

A sample is placed in the holder and the system is evacuated. At about 1 x 10^{-6} torr pressure, or less, voltage is applied to the sample heater to raise the temperature to 50°C. After redeposition has ceased, or a steady state rate of deposition has been achieved, the temperature is again increased, and the process is repeated until 150°C is reached. Changes in the beat frequency are read directly from the frequency counter.

To calculate the film thickness deposited on the quartz crystals, the equation

$$t = \frac{\Delta f}{2\rho}$$

was employed where ρ is the film density of the material. For all materials except the metallic elements and the polyurethane foams, the film density was equated with the bulk density. The densities of the materials examined are shown in Table I.

RESULTS

Redeposition tests were made on seven classes of materials: (1) metallic elements (2) coatings (3) potting compounds (4) elastomers (5) adhesives (6) polyurethane foam (7) nylon synthetic fiber. The results of these tests are shown in Figures 7 through 22. In each case, film thickness and temperature are plotted as a function of time. In Figures 23 and 24, the temperature and change in frequency are shown as a function of time since no density could be formulated for these materials.

DISCUSSION

Redeposition of the metallic elements zinc and cadmium was extensive. In both cases, 60% of the bulk density was used to calculate film thickness as is the practice in metallic film evaporation techniques. The zinc sample, 0.002" electroplated onto 2024 Al, was heated to 500°C during this test. Complete redeposition occurred since the melting point of zinc is 419°C with a vapor pressure of 1.5 mm Hg at 500°C [3]. Deposition began at 150°C, and continued, until a 360 A thick film was deposited. As exhibited in equation 4, the maximum amount of oxidation of the film that can occur as the film is deposited can be calculated. The assumption of a sticking probability of unity is valid since the zinc is being deposited onto a metallic surface of the crystal. Since the total system pressure is 1 x 10^{-6} torr, the partial pressure of oxygen can be 1 x 10^{-8} torr. K was found to be 8.1% ZnO contained in the deposited film.

As shown in Figure 8, cadmium began depositing at 70°C. The film thickness increased to 130 Å and remained at that thickness. The melting point of cadmium is 321°C, however, rapid vaporization occurred before the sample reached 100°C. At the conclusion of the test it was evident that the cadmium that had been electroplated onto a 321 stainless steel cylindrical sleeve had uniformly coated the interior of the vacuum chamber. For the cadmium film the maximum amount of CdO present was calculated to be 5.5%.

It should be noted that for low boiling point metals, such as Zn and Cd, the critical size for nucleation on a metal substrate is less than two atoms. That is to say there is no nucleation barrier to condensation and a permanent deposit. However, for these metals to deposit on a glass substrate, the nuclei must be relatively large before they are stable [2].

Cat-A-Lac black paint failed to redeposit any appreciable film on the QCM, as shown in Figure 10. The film thickness exhibited by the Cat-A-Lac on polyurethane foam in Figure 11 was attributed to the foam substrate but was less than 50 Å thick. Both Micoweld L5X923 and S-13G paint were extremely stable. Redeposition of Kem Lustral flat black enamel occurred when the sample was heated to 100°C and 150°C as shown in Figure 14. This Kem Lustral sample is a medium length alkyd. The sample began redepositing at 80°C, and deposited a film 100 Å thick in 4 hours at 100°C. Redeposition recommenced at about 110°C and reached a final thickness of 335 Å after 6 hours at 150°C.

RTV 118 is a precatalyzed RTV silicone rubber with a room temperature cure. As soon as this potting compound was heated, material was driven off and redeposited. As can be seen in Figure 16, the film thickness increased to 640 Å. Deposition began at 50°C, increased sharply to a constant rate of 25 Å/hr at 100°C, and reached 320 Å total film thickness. The epoxy potting compound Epon 828 was relatively stable, producing a 65 Å film in 25 hours at 100°C. At 150°C, the thickness increased slightly to 75 Å.

Both silicone elastomers, Lord BTR and Cohrlastic R10470 outgassed and redeposited severely. As shown in Figures 17 and 18, a significant breakdown occurred when the samples were heated from 100° to 150°C.

All of the adhesives tested showed poor outgassing characteristics. The heat cured sample of Sylgard 182 deposited a 450 Å film on the quartz crystal. Silastic 140 is a silicone compound whereas PR 1538 is a polyurethane adhesive. In Figure 20 it is evident that the polyurethane molecular bonds were breaking at prolonged periods at 150°C. Film thickness continued to increase, never reaching a steady state condition as did most samples tested.

CPR 11-16 is a 3 lb./ft^3 density polyurethane foam. In Figure 22, it can be seen that some of the redeposited material had been reevaporated from the QCM as the sample was heated to 150°C. Additional material was again driven off the sample such that the deposition rate on the crystal exceeded the reevaporation rate. This implies that $Q_{ad} \approx KT$ for this material at 100°C.

Nylon lacing tape exhibited an initial frequency change at ambient temperature. This could be caused by a flux of outgassing desorbed gases and water from the tape. Stability was recovered before the sample received external heat. After equilibrium was reached, temperature had very little effect on the nylon fiber. This minimal effect was also observed for the nylon net of Figure 22.

CONCLUSIONS

(1) The quartz crystal microbalance is a highly sensitive and useful tool in the investigation of redeposition contamination from outgassing materials.

(2) Very low boiling point metals such as cadmium and zinc should not be employed in high vacuum use or space vehicles.

(3) Silicone compounds exhibit low thermal/vacuum stability and do tend to redeposit.

(4) The adhesives tested showed poor outgassing and tended to redeposit. Redeposition characteristics may vary from those reported when the adhesive is used as a bond between two substrates since less surface area will be exposed.

(5) Nylon materials tested were very stable. The only noticeable response was to the release of adsorbed atmospheric gases and water.

(6) The one epoxy tested appeared stable. A very thin (less than 50 Å) film was formed but this also was attributed to surface adsorbed material.

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3. Holland, L.: Vacuum Deposition of Thin Films, Chapman and Hall, Ltd., London, 1963.
4. Westinghouse Scientific Equipment Department, IL-99-360-1 Model 701A Manual, Pittsburgh, 1965.
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TABLE I

DENSITY OF MATERIALS

MATERIAL	DENSITY (gm/cc)
Zinc	7.14
Cadmium	8.65
Cat-A-Lac Black	1.55
Micoweld Black	1.55
S-13G	2.10
Kem Lustral Enamel	1.60
Epon 828	1.29
RTV 118	1.06
BTR Elastomer	1.26
Cohrlastic R10470	0.623
Silastic 140	1.06
PR 1538	1.05
Sylgard 182	1.06
CPR 11-16	.048 [3 lb/ft^3]
Nylon Lacing Tape	-
Nylon Net	-

FIGURE 1. ORGANIC-FREE VACUUM CHAMBER

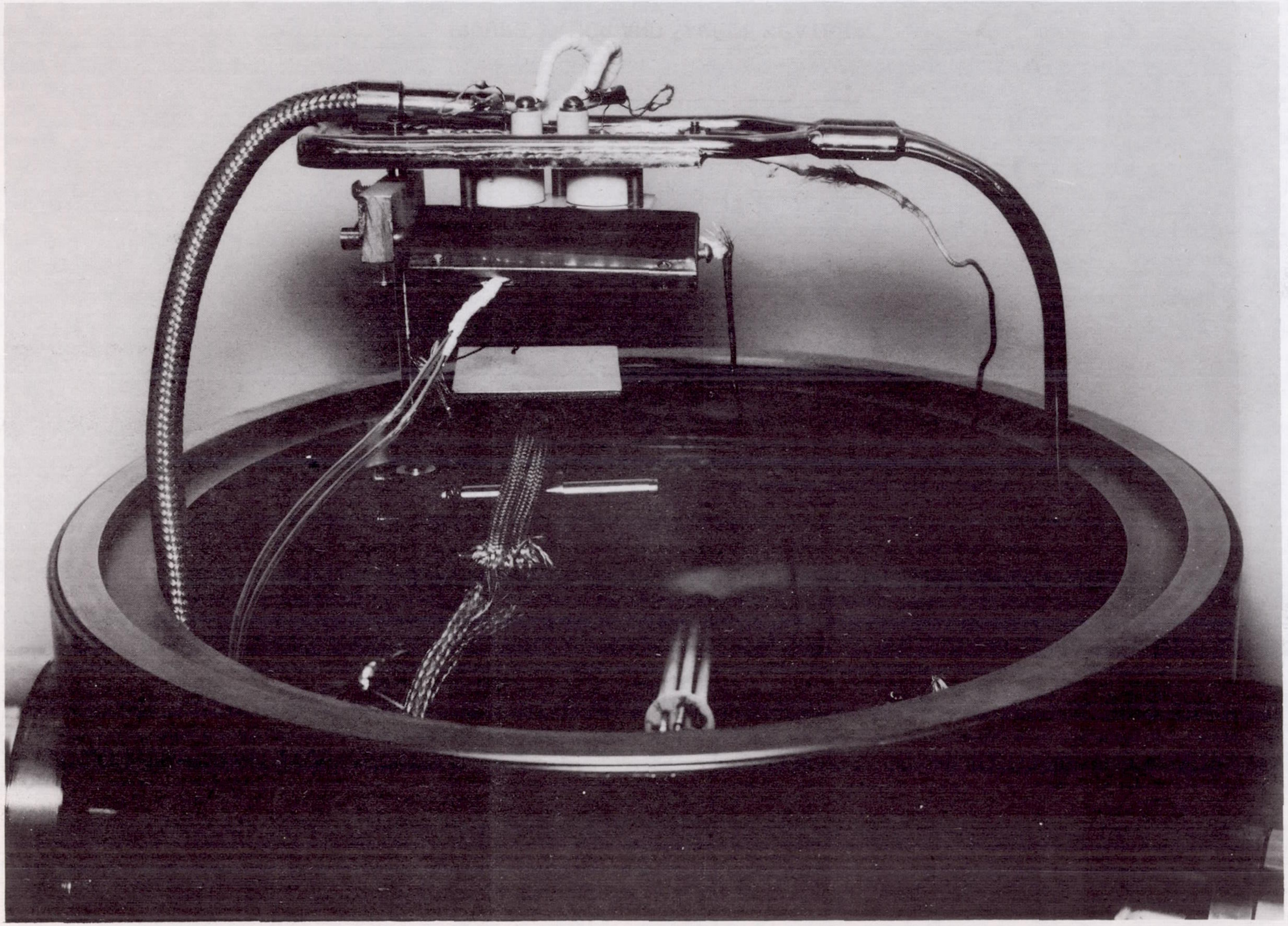


FIGURE 2. INTERIOR OF ELECTROPOLISHED CHAMBER

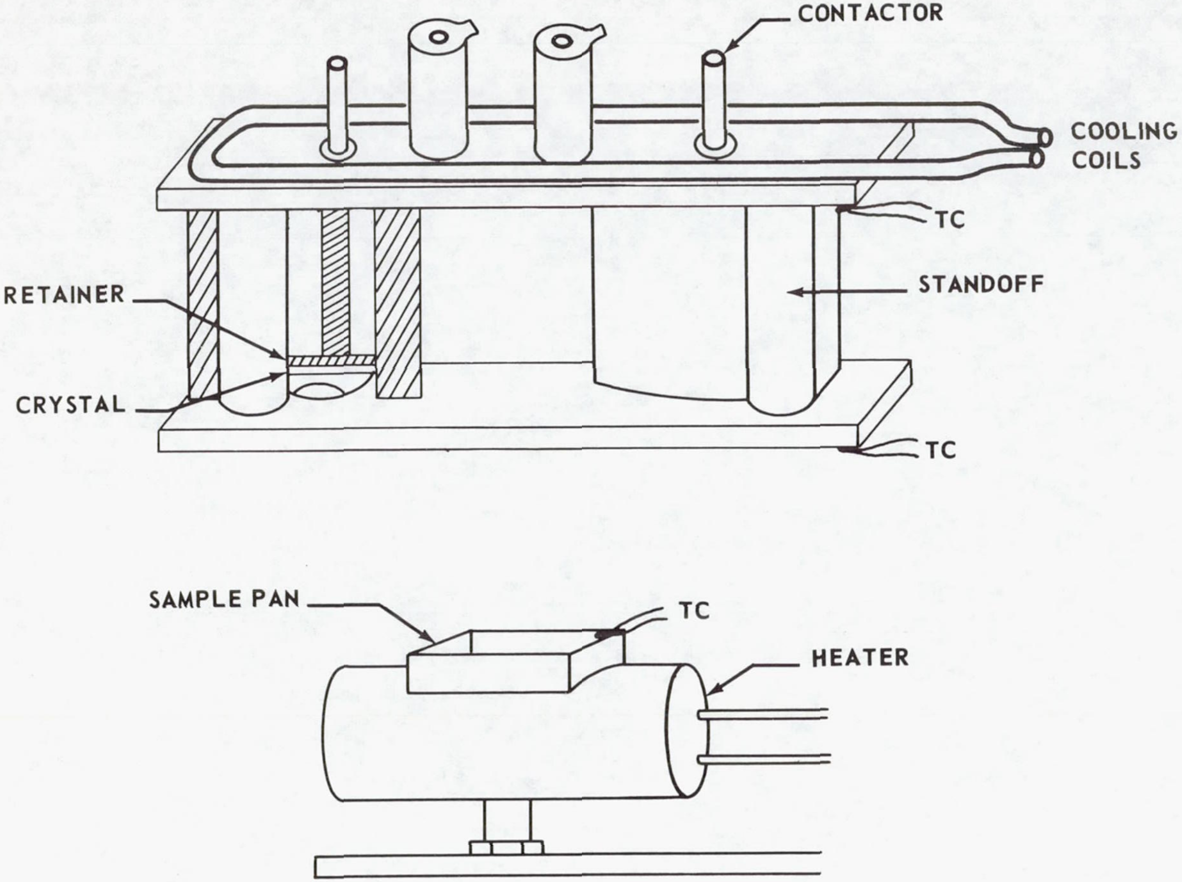


FIGURE 3. QCM AND SAMPLE HEATER

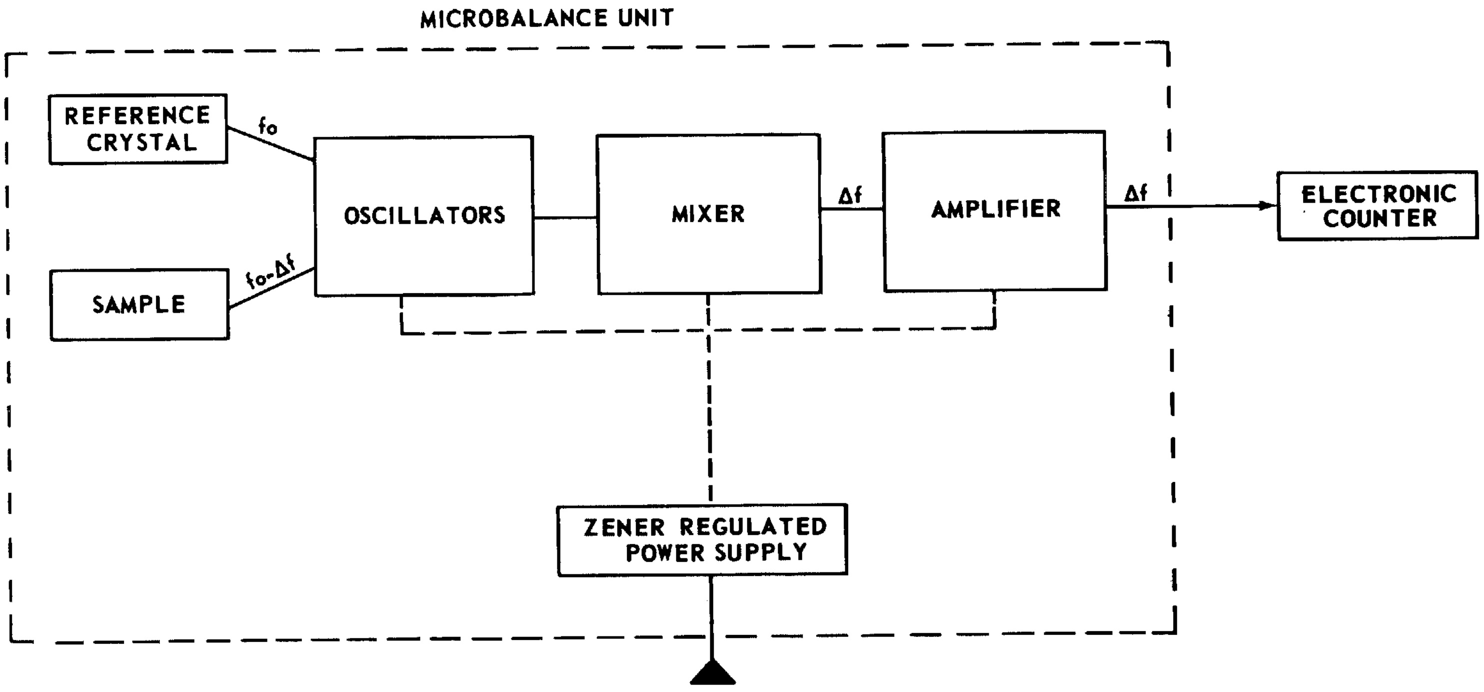


FIGURE 4. BLOCK DIAGRAM OF MICROBALANCE CIRCUIT

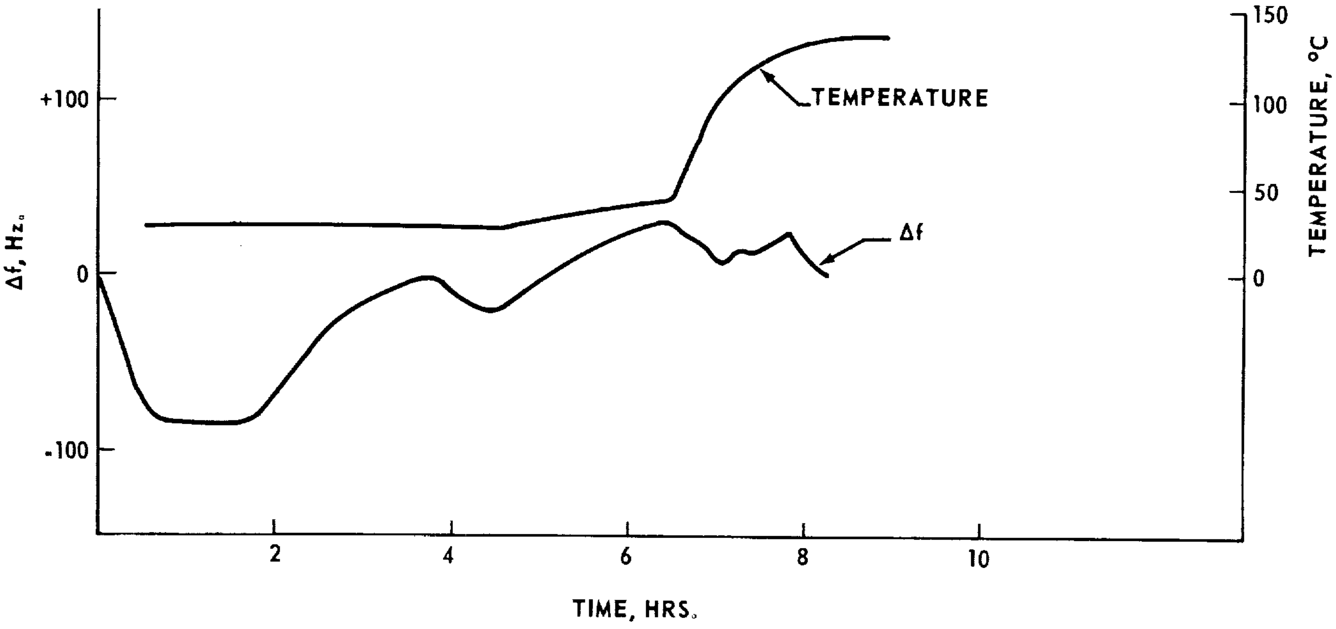


FIGURE 5. SYSTEM BLANK, Δf AND TEMPERATURE VERSUS TIME

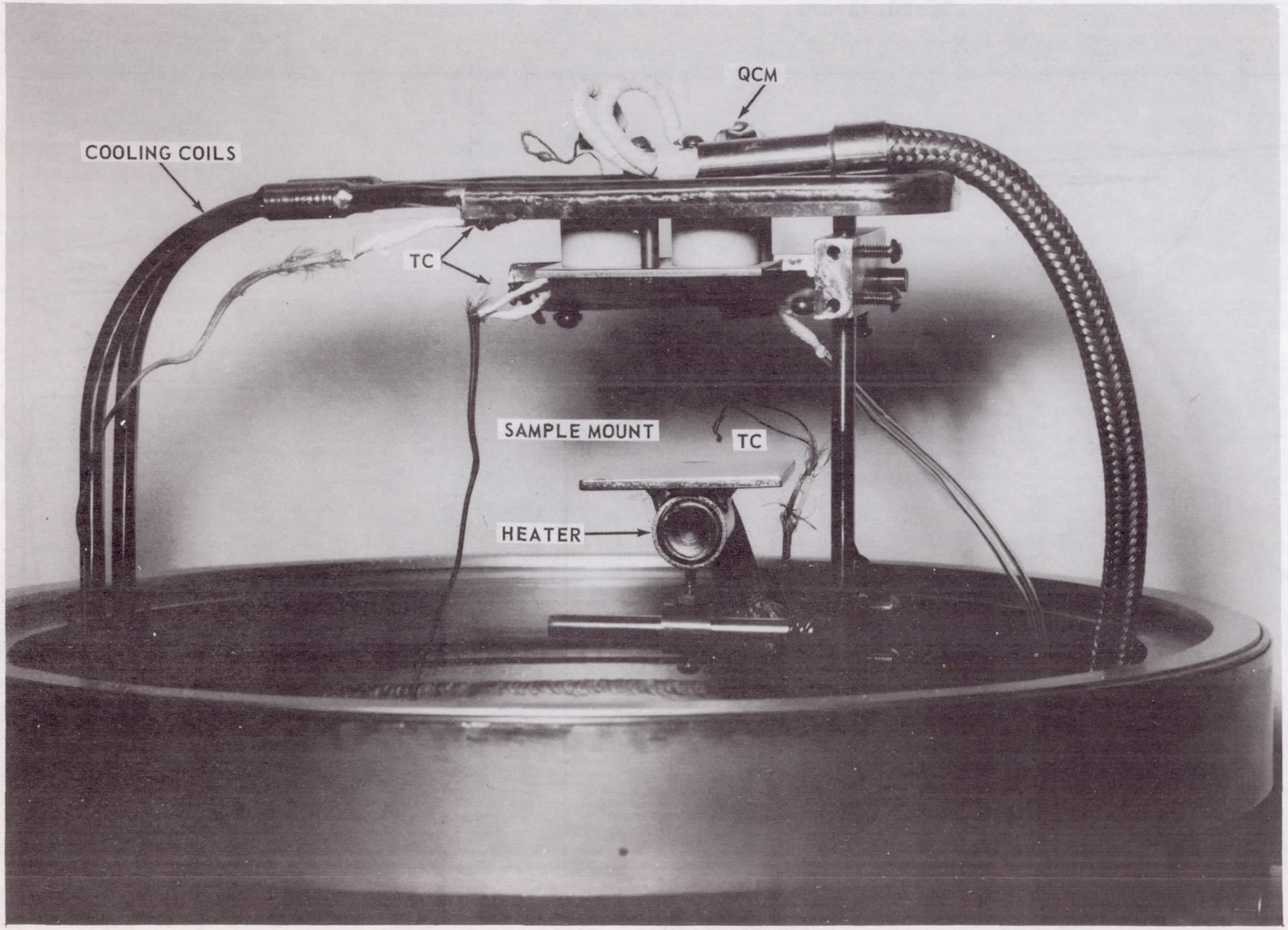


FIGURE 6. EXPERIMENTAL SET-UP OF QCM

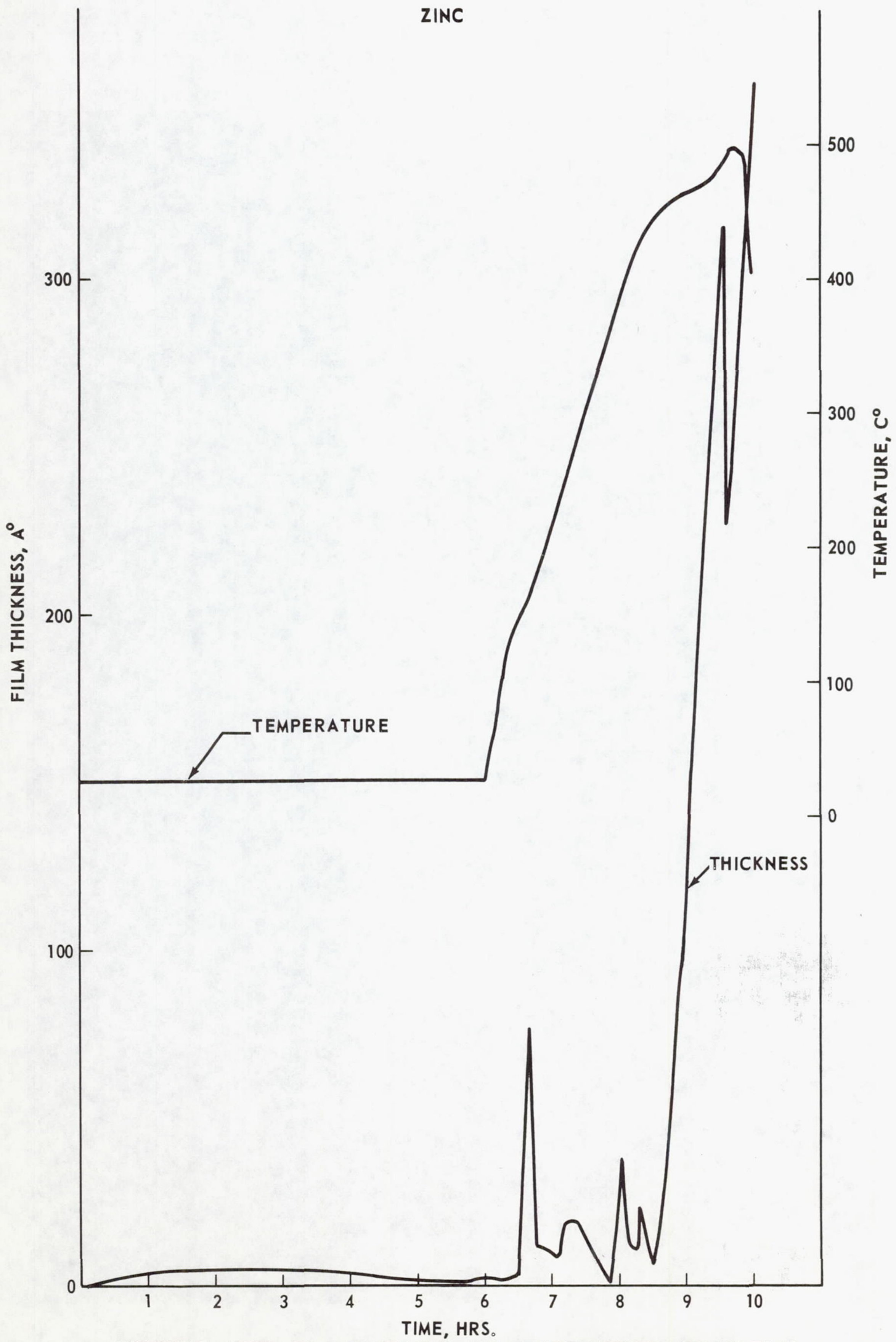


FIGURE 7. ZINC, FILM THICKNESS AND TEMPERATURE VERSUS TIME

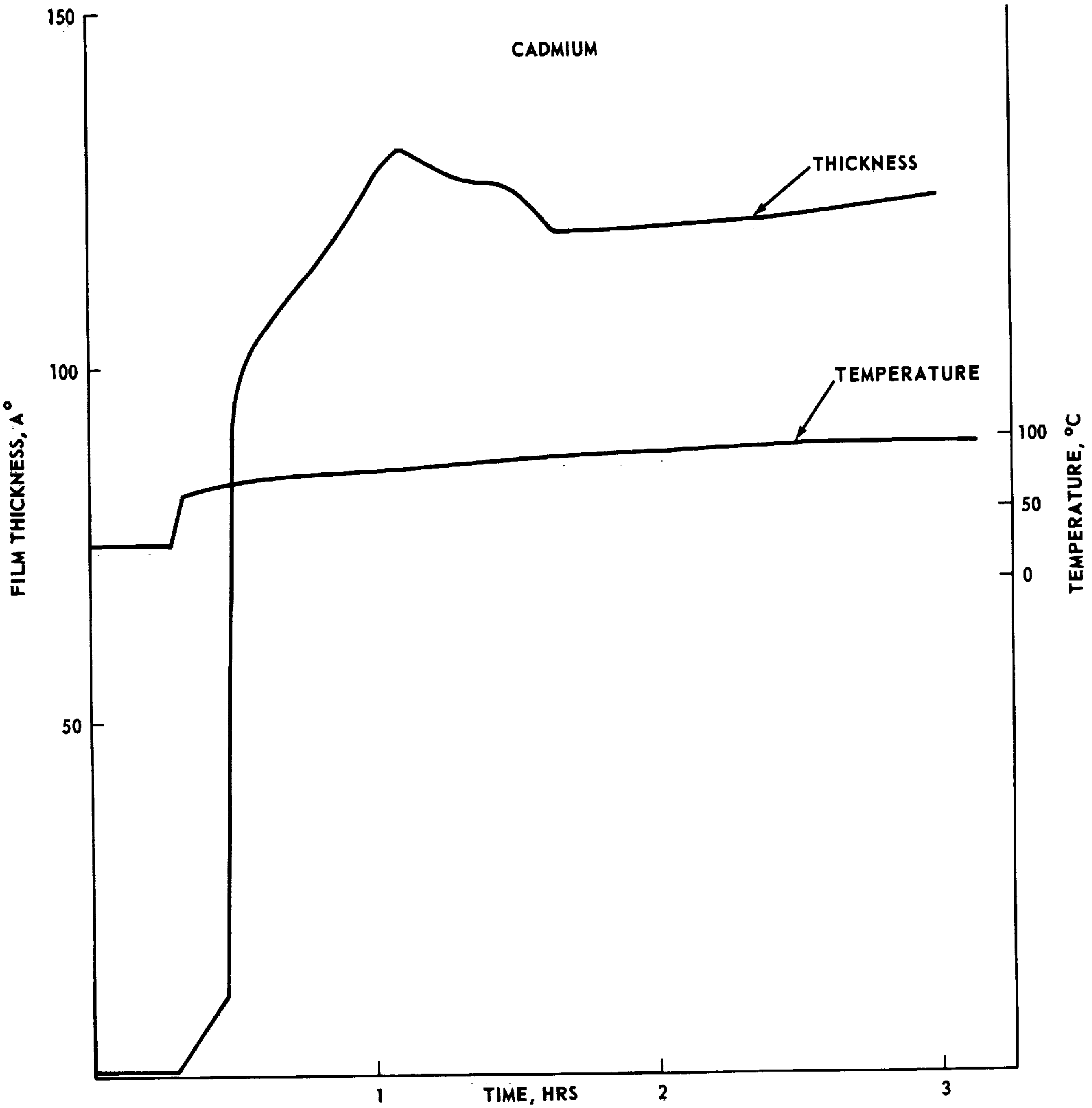
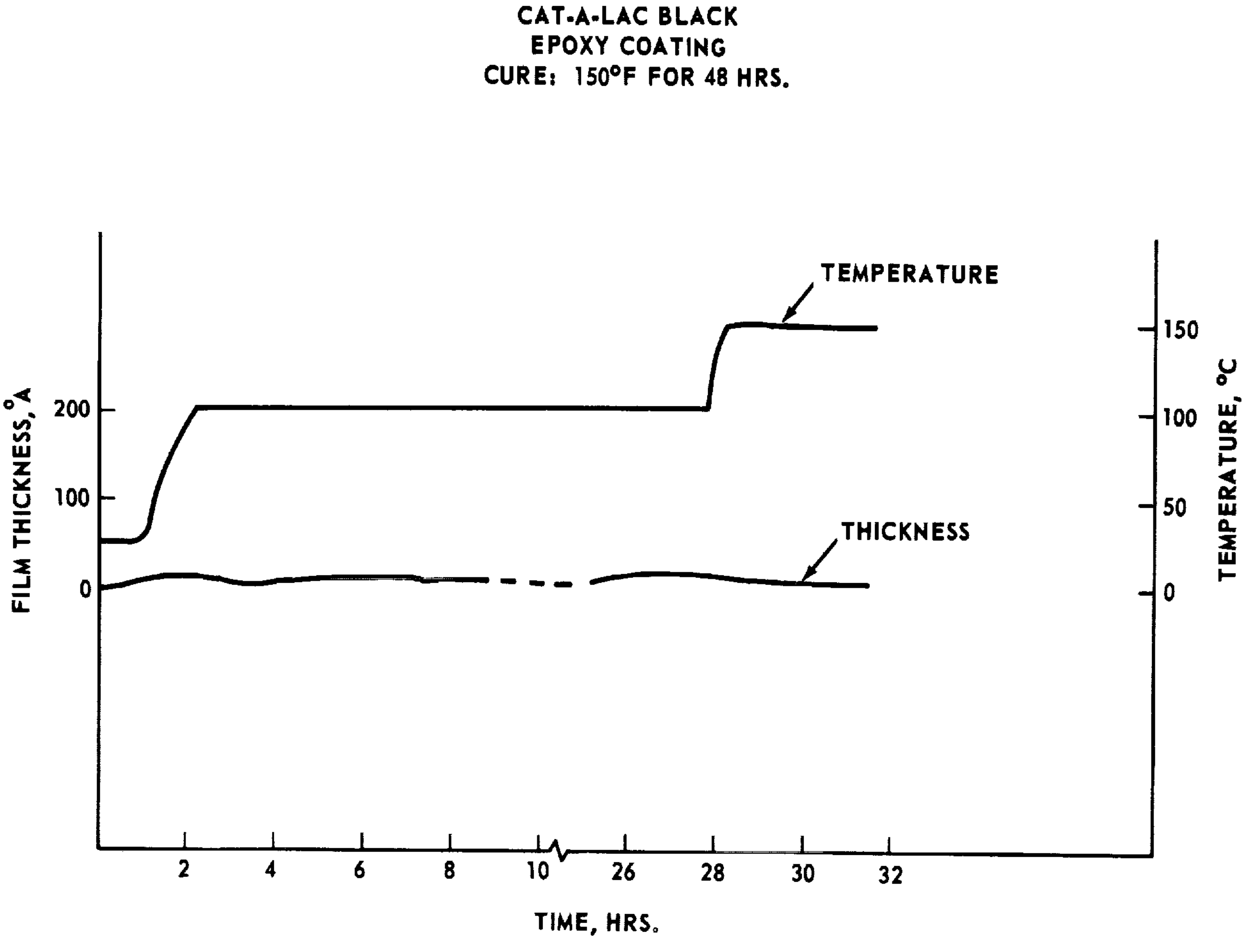


FIGURE 8. CADMIUM FILM THICKNESS AND TEMPERATURE VERSUS TIME



CAT-A-LAC BLACK
EPOXY COATING
CURE: 150°F FOR 48 HRS.

FIGURE 9. CAT-A-LAC BLACK, FILM THICKNESS AND TEMPERATURE VERSUS TIME

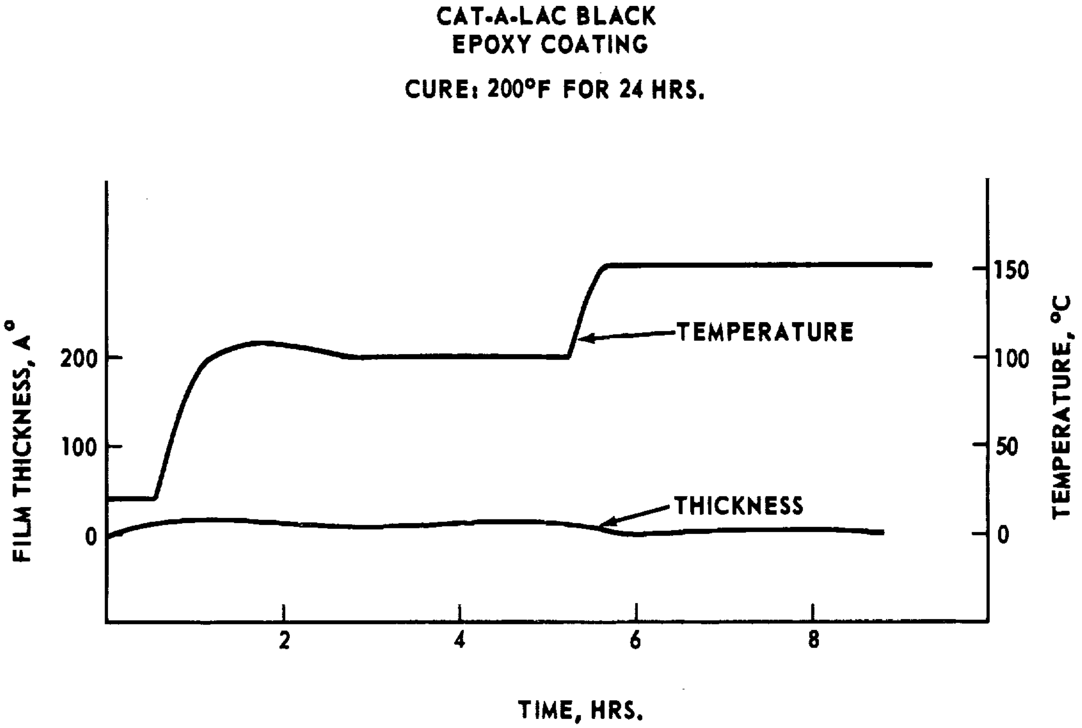


FIGURE 10. CAT-A-LAC BLACK, FILM THICKNESS AND TEMPERATURE VERSUS TIME

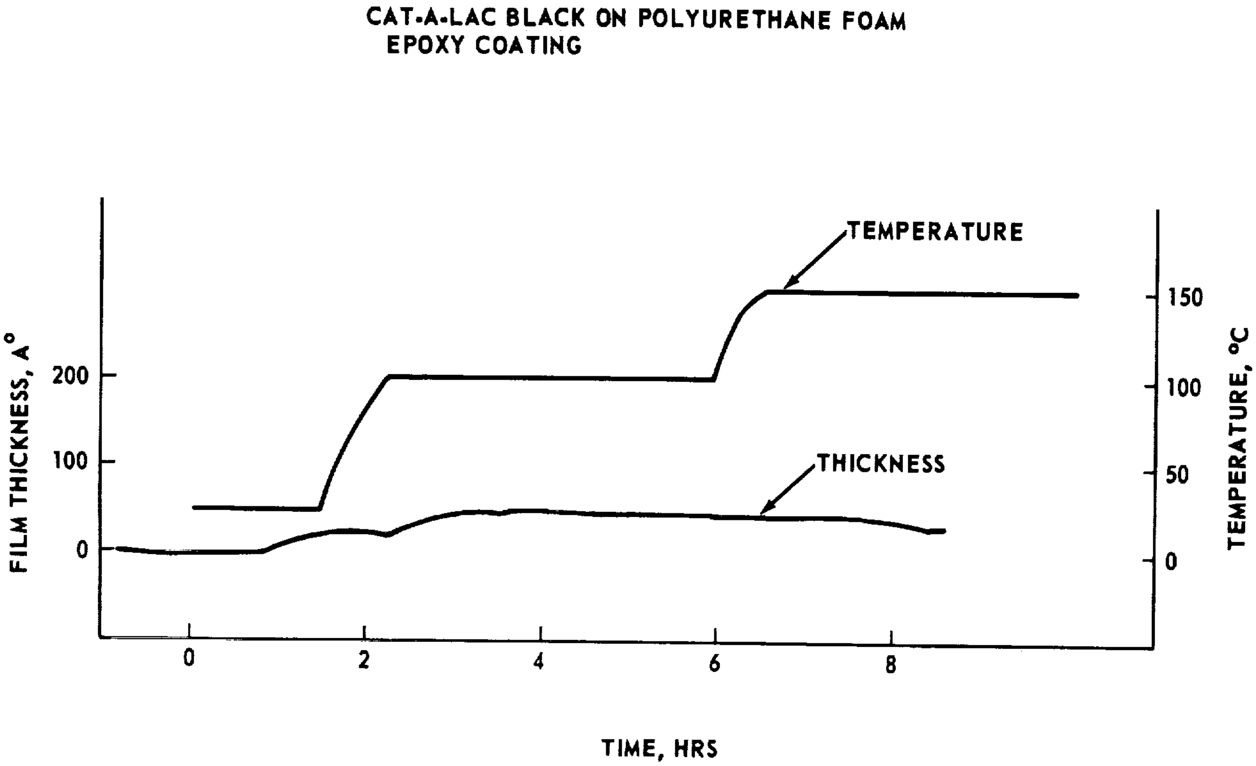
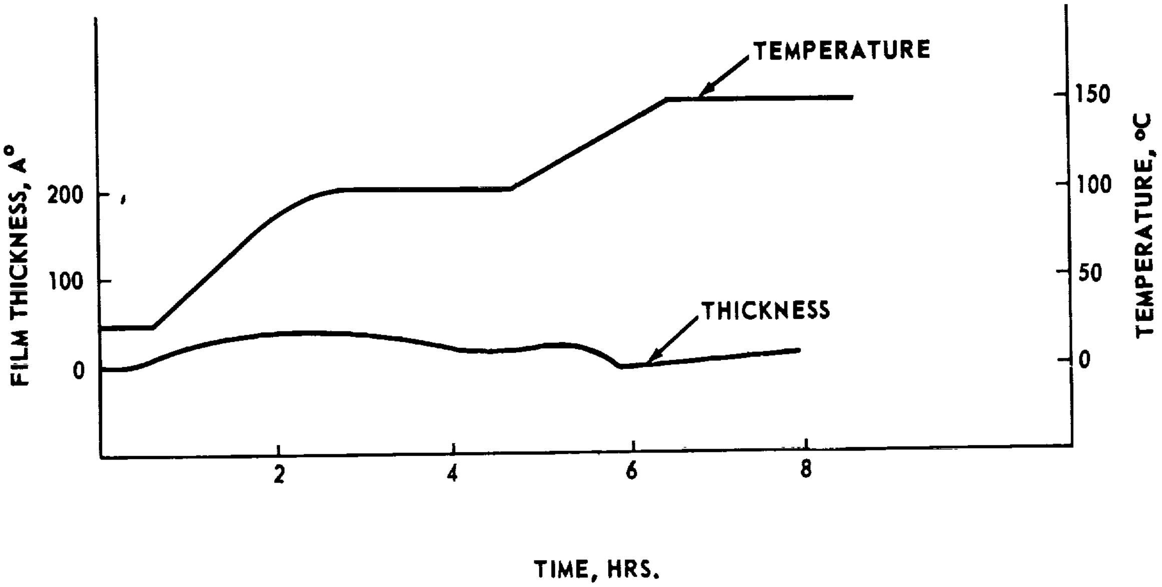

CAT-A-LAC BLACK ON POLYURETHANE FOAM
EPOXY COATING

FIGURE 11. CAT-A-LAC BLACK ON POLYURETHANE FOAM, FILM THICKNESS AND TEMPERATURE VERSUS TIME



MICOWELD L5X923 BLACK PAINT
MODIFIED VINYL PHENOLIC

FIGURE 12. MICOWELD L5X923 BLACK PAINT, FILM THICKNESS AND TEMPERATURE VERSUS TIME

S-13G
RTV 602 WITH ZnO PIGMENT
CURE: 72 HR. @ RT, 24 HR @ 200°F

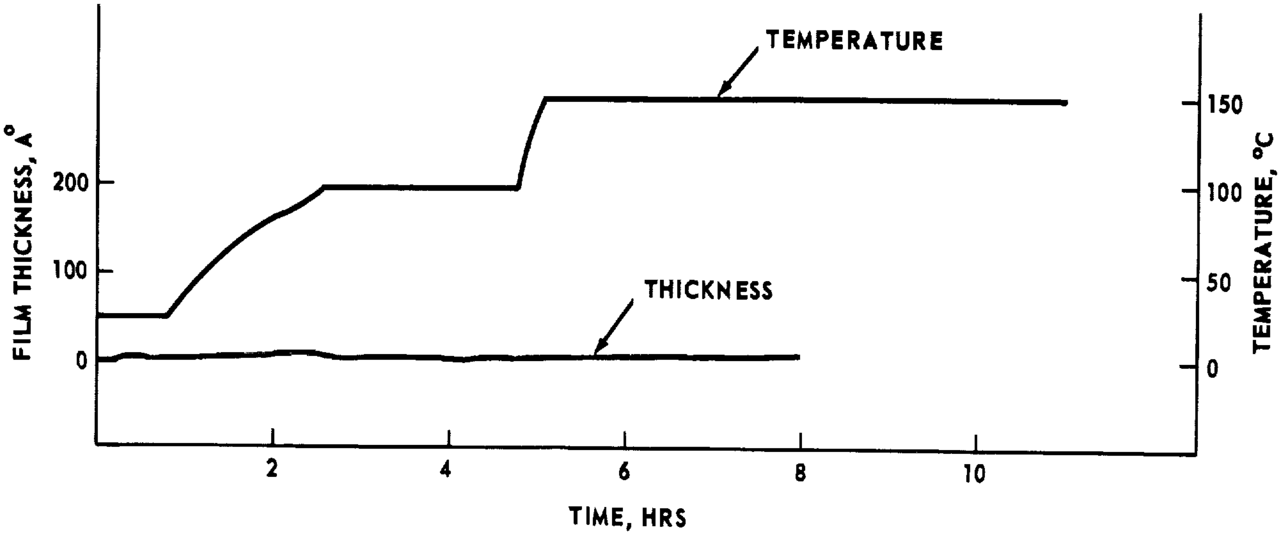


FIGURE 13. S-13G, FILM THICKNESS AND TEMPERATURE VERSUS TIME

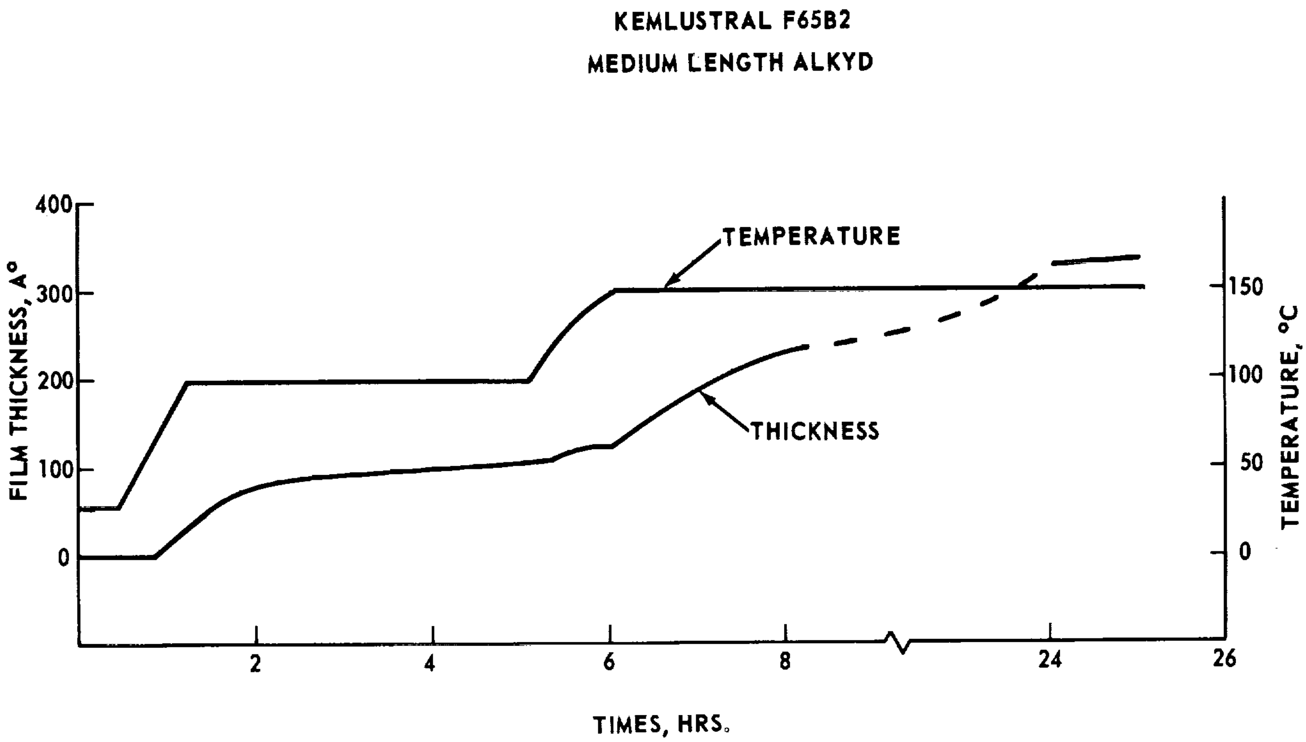


FIGURE 14. KEMLUSTRAL F65B2, FILM THICKNESS AND TEMPERATURE VERSUS TIME

EPON 828 EPOXY

CURING AGENT Z, FLEXIBILIZER 871

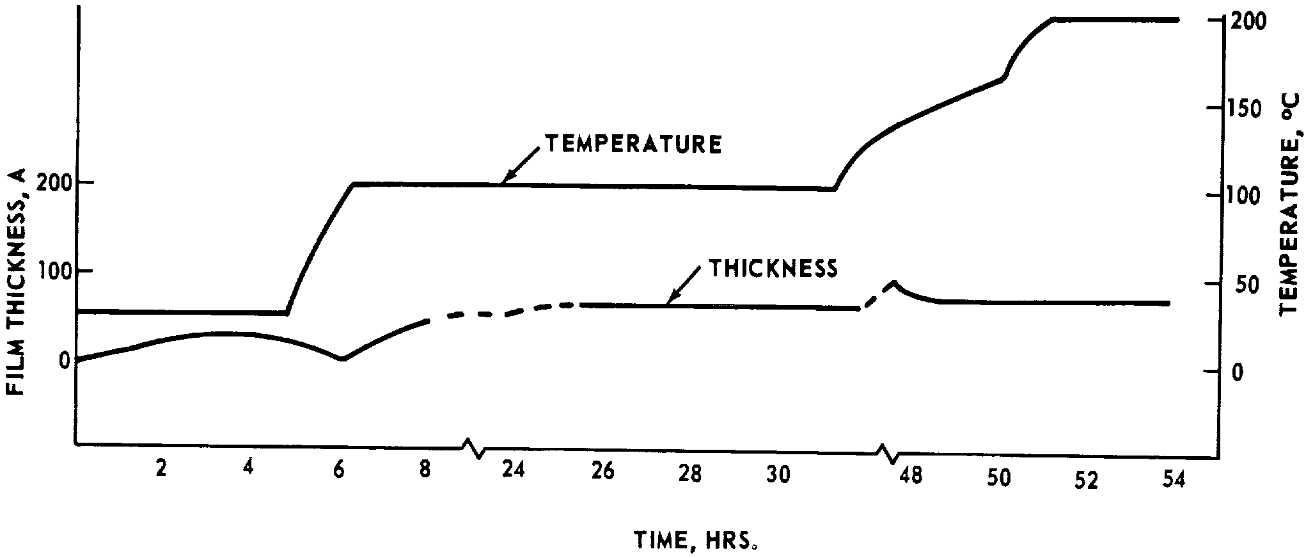


FIGURE 15. EPON 828, FILM THICKNESS AND TEMPERATURE VERSUS TIME

RTV 118 SILICONE

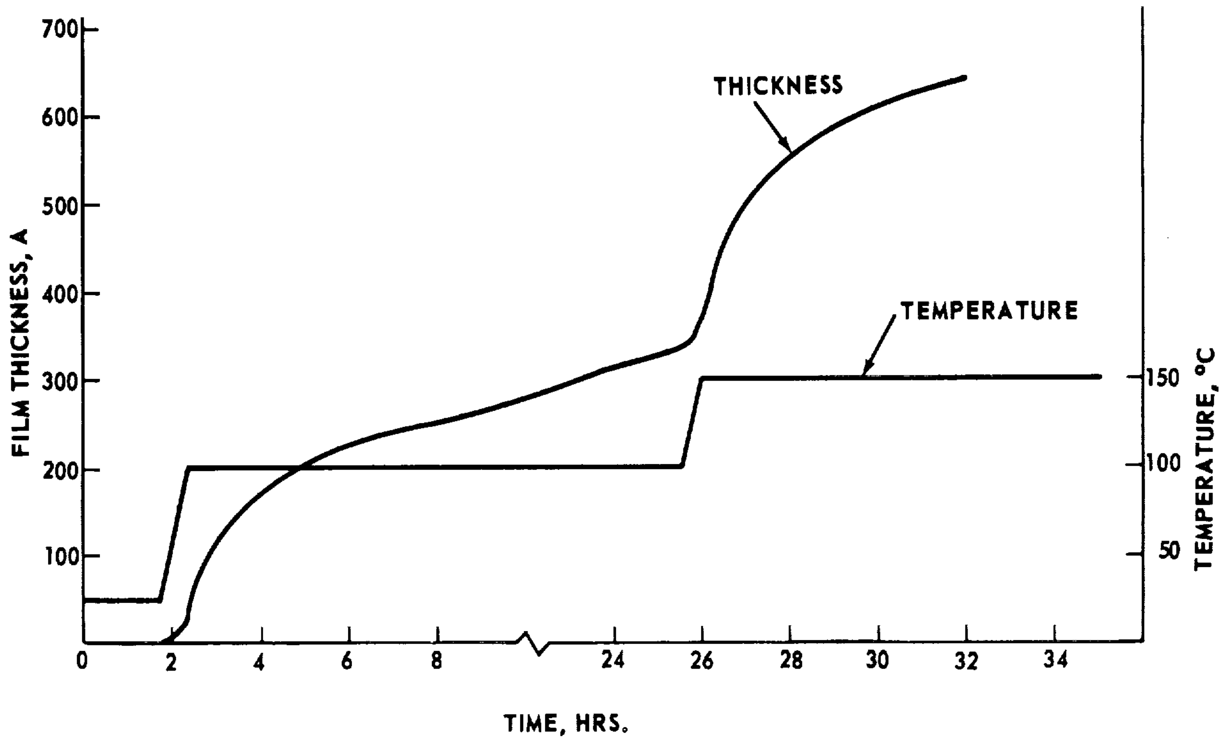


FIGURE 16. RTV 118, FILM THICKNESS AND TEMPERATURE VERSUS TIME

LORD BTR ELASTOMER

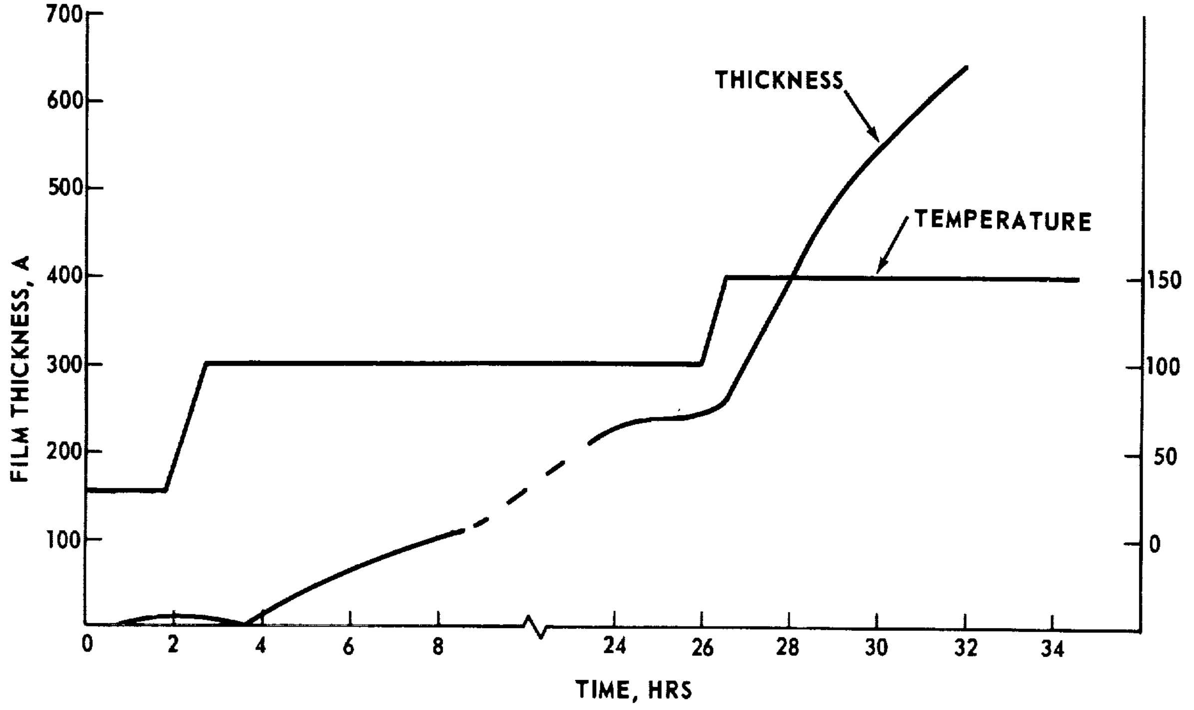


FIGURE 17. LORD BTR ELASTOMER, FILM THICKNESS AND TEMPERATURE VERSUS TIME

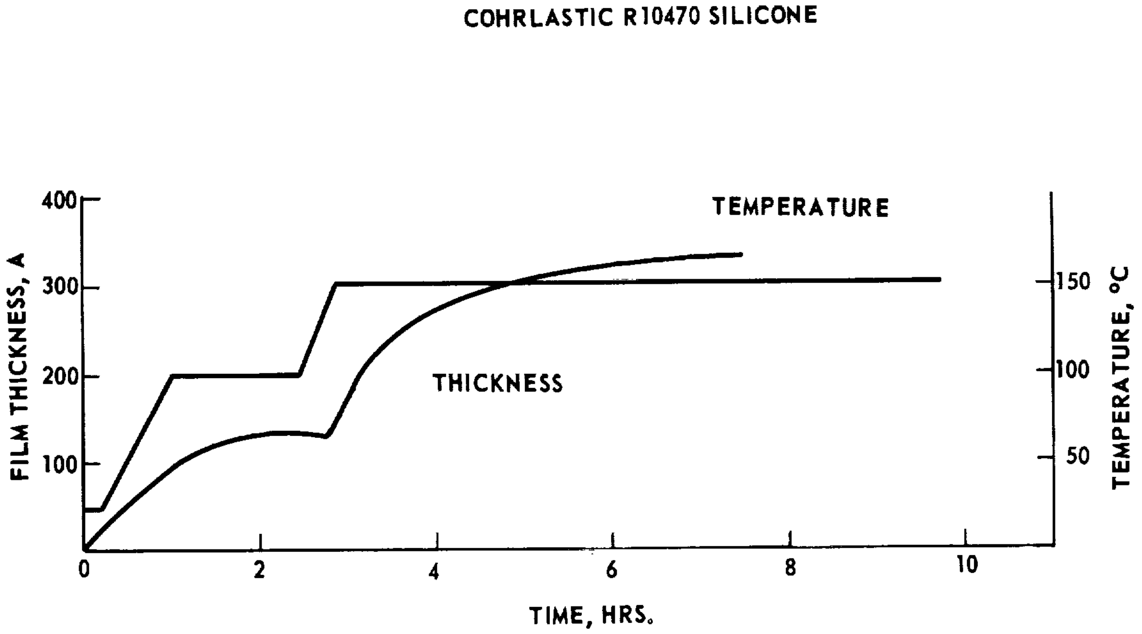


FIGURE 18. COHRLASTIC R10470, FILM THICKNESS AND TEMPERATURE VERSUS TIME

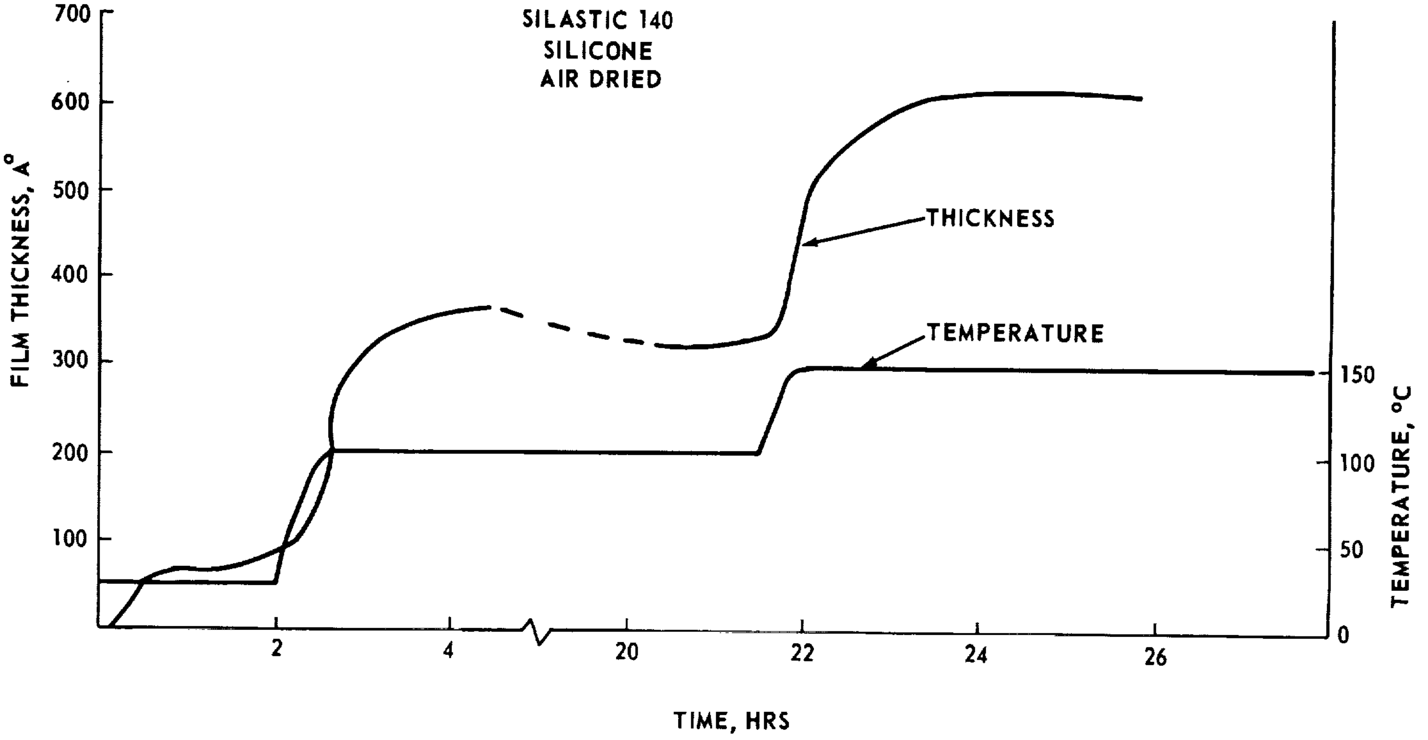


FIGURE 19. SILASTIC 140, FILM THICKNESS AND TEMPERATURE VERSUS TIME

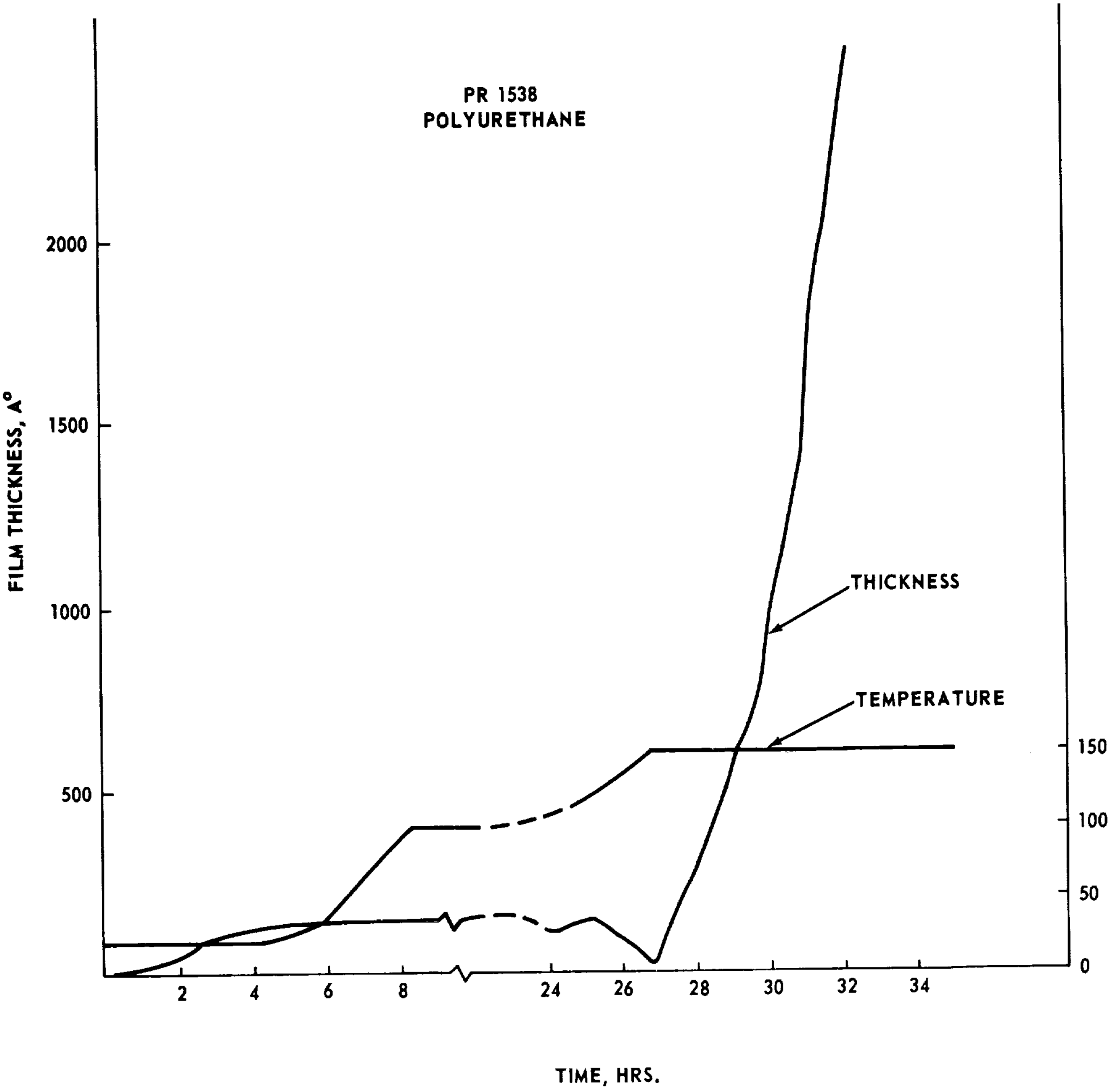
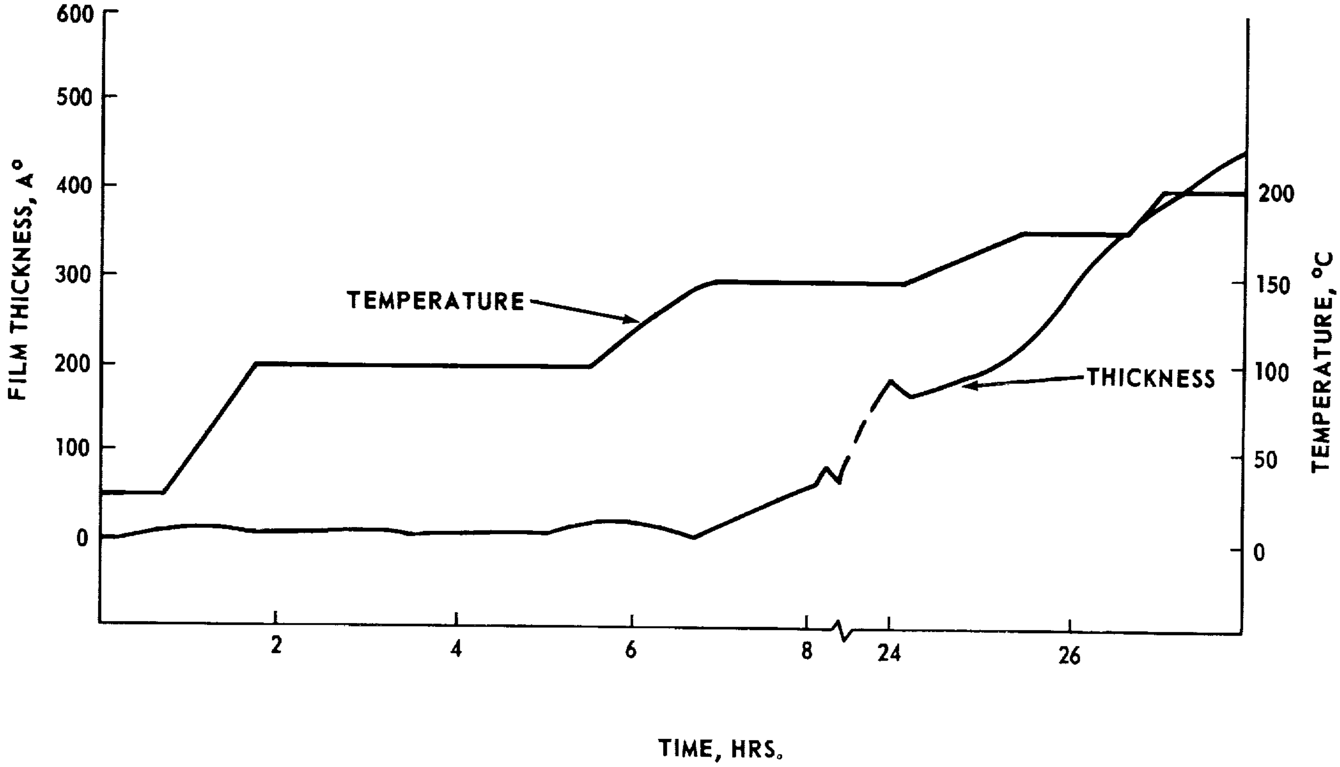


FIGURE 20. PR1538, FILM THICKNESS AND TEMPERATURE VERSUS TIME



SYLGARD 182
SILICONE
CURE: 4 HRS @ 65°C, 1 HR @ 100°C,
.25 HR @ 150°C

FIGURE 21. SYLGARD 182, FILM THICKNESS AND TEMPERATURE VERSUS TIME

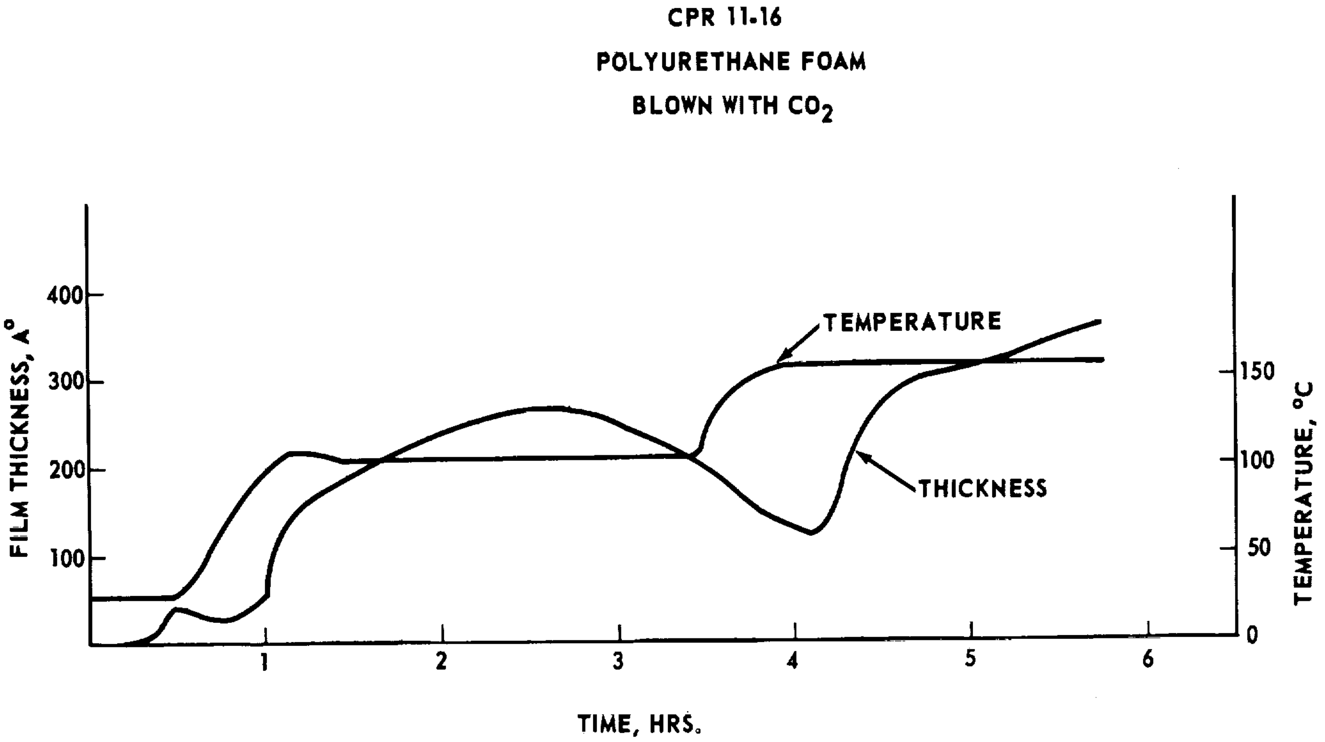


FIGURE 22. CPR 11-16, FILM THICKNESS AND TEMPERATURE VERSUS TIME

NYLON LACING TAPE
STYLE 18B

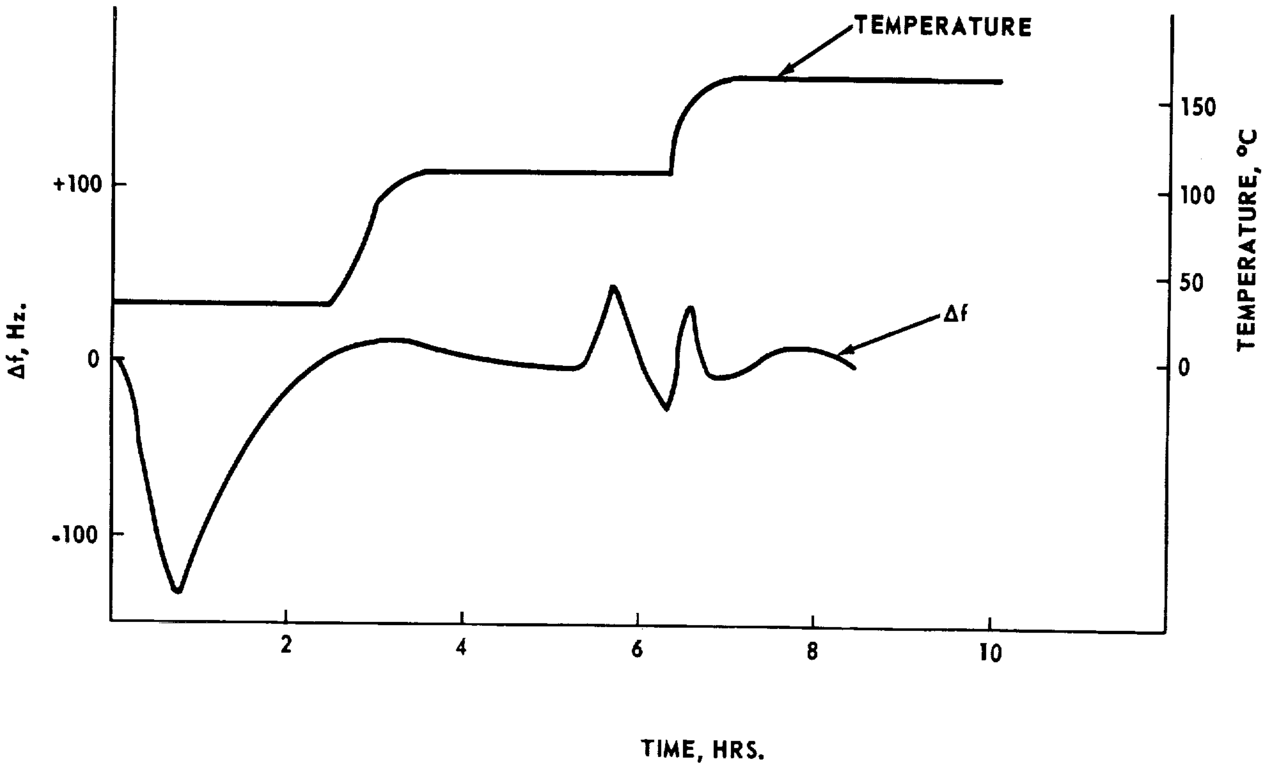


FIGURE 23. NYLON LACING TAPE, Δf AND TEMPERATURE VERSUS TIME

NYLON NET 36K1012

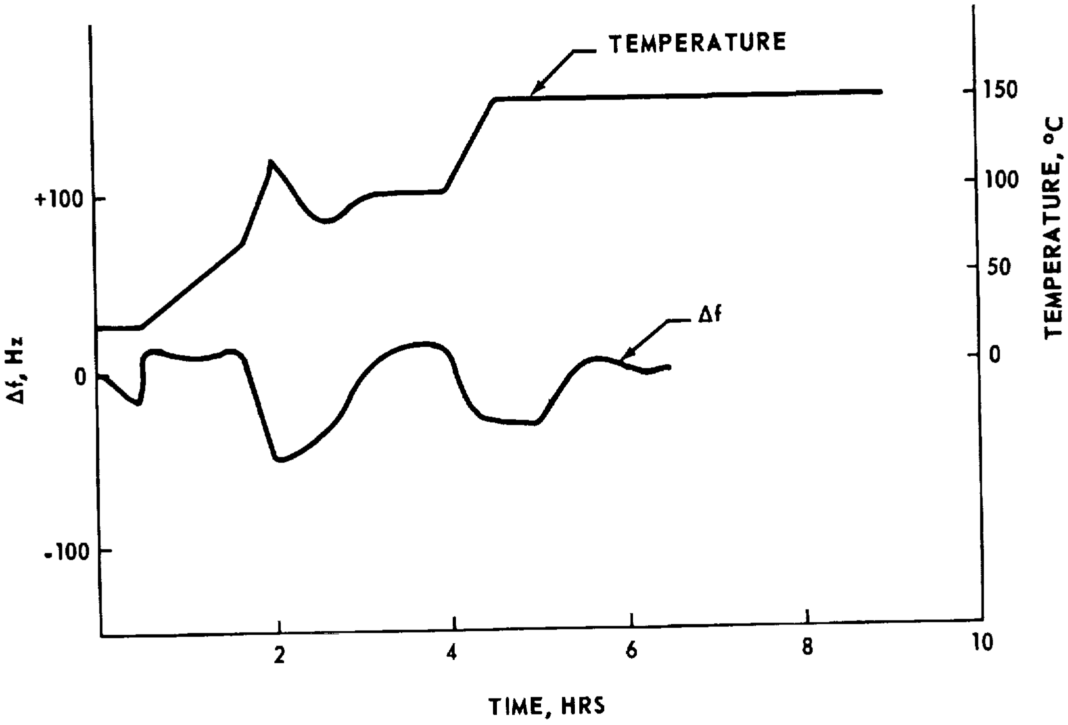


FIGURE 24. NYLON NET, Δf AND TEMPERATURE VERSUS TIME

November 20, 1968 APPROVAL NASA TM X-53801

REDEPOSITION OF VACUUM OUTGASSED PRODUCTS

By Carolyn S. Griner

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This document has also been reviewed and approved for technical accuracy.

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