[redacted]

WAL TR 397/10

TECHNICAL REPORT

WATERTOWN ARSENAL
WATERTOWN 72, MASS.

COPY NO. 2

WAL TR 397/10

AD A951661

WATERTOWN ARSENAL

LABORATORIES

THERMAL CONDUCTIVITY OF TEFLON, KEL-F, AND DUROID 5600
AT ELEVATED TEMPERATURES

BY

A. W. SCHULTZ

~~RETURN THIS MANUAL TO THE LIBRARY~~

~~DO NOT TRANSFER~~

O.O. PROJECT:	HIGH TEMPERATURE THERMAL PROPERTIES OF FLUOROCARBON PLASTICS
D/A PROJECT:	516-05-01
ADMA CONTROL NO.:	ASMA-PBC NO. 9-7-5-01-00/3040/1235 IOD 616-57; SPB 3-113-57
REPORT NO.:	WAL TR 397/10
FIELD TITLE:	FLUOROCARBON PLASTICS, THERMAL CONDUCTIVITY

DISTRIBUTION STATEMENT A
Approved for public release;
Distribution Unlimited

MARCH 1958

DTIC
ELECTE
MAY 27 1982
S D
B

WATERTOWN ARSENAL
WATERTOWN 72, MASS.

82 05 27 036

FLUOROCARBON PLASTICS,
THERMAL CONDUCTIVITY

THERMAL CONDUCTIVITY OF TEFLON, KEL-F, AND DUROID 5600 AT ELEVATED TEMPERATURES

TECHNICAL REPORT

By

A. W. Schultz

A. K. Wong

O.O. Project:	High Temperature Thermal Properties of Fluorocarbon Plastics
D/A Project:	516-05-010
ABMA Control No.:	ABMA-PBC No. 9-7-5-01-00/3040/1235 IOD 616-57; SPB 3-113-57
Report No.:	WAL TR 397/10
Field Title:	Fluorocarbon Plastics, Thermal Conductivity

DTIC
ELECTE
MAY 27 1982
S D
B

WATERTOWN ARSENAL
WATERTOWN 72, MASS.

DISTRIBUTION STATEMENT A
Approved for public release;
Distribution Unlimited

WATERTOWN ARSENAL LABORATORIES

TITLE

THERMAL CONDUCTIVITY OF TEFLON, KEL-F, AND DUROID 5600 AT ELEVATED TEMPERATURES

ABSTRACT

The thermal conductivity values of the fluorocarbon plastics, Teflon, Kel-F, and Duroid 5600, were determined at mean specimen temperatures ranging from approximately 185°F to their respective gel-points. Measurements were obtained utilizing a comparative method technique based on unidirectional steady-state heat-flow theory.

The materials under investigation were of commercial quality and were annealed prior to testing to insure dimensional stability.

For the range of temperatures within which the materials were investigated, it was found that Teflon consistently exhibited higher values than Kel-F and lower values than Duroid 5600. At 350°F, the thermal conductivities obtained were:

Teflon	- 2.37 BTU/hr/ft^2/°F/in.
Kel-F	- 1.72 BTU/hr/ft^2/°F/in.
Duroid 5600	- 3.58 BTU/hr/ft^2/°F/in.

The thermal conductivities were found to increase with increasing temperatures.

Arnold W. Schultz
ARNOLD W. SCHULTZ
Physicist

Anthony K. Wong
ANTHONY K. WONG
Mechanical Engineer

APPROVED:

J. F. Sullivan
J. F. SULLIVAN
Associate Technical Director
Watertown Arsenal Laboratories

Russell F. Scott Jr.
RUSSELL F. SCOTT, JR.
Major, Ord Corps
Director
Watertown Arsenal Laboratories

DTIC COPY INSPECTED 2

Accession For
NTIS GRA&I ☑
DTIC TAB ☐
Unannounced ☐
Justification
1958 PER LETTER
By
Distribution/
Availability Codes
Avail and/or
Dist Special
A

UNANNOUNCED

INTRODUCTION

Recent advances in missile technology have greatly accelerated the search for materials capable of withstanding intense heat for short periods of time. This search involves the study of new materials as well as the re-evaluation of the older, more established ones for the purpose of determining their degree of suitability for use as structural components at high temperatures. Of primary importance in these material evaluation studies is knowledge of the thermal properties and their variation with increasing temperatures.

A literature survey,[1] published recently, regarding physical properties of fluorocarbon plastics indicated a lack of thermal conductivity data at elevated temperatures. Considerable interest has developed in this class of thermo-plastic materials by design engineers during the past few years because of the remarkable combination of physical properties which have resulted in many new applications. Of particular importance, at this time, are two commercially available fluorocarbon plastics identified as Teflon (polytetrafluoroethylene) and Kel-F (polytrifluorochloroethylene). Although the gel-point temperatures of these two plastics are within the 400 to 850°F region and are therefore considered as low-melting-point materials with respect to most metallic structural materials, the large quantity of heat required to disrupt their stable chemical bonds enables them to withstand high temperatures for short periods of time.

The purpose of the investigation described herein was to measure the thermal conductivity of Teflon, Kel-F, and Duroid 5600 (an inorganic fiber-reinforced Teflon-base material) at mean temperatures ranging from approximately 185°F to their respective gel points. The results of these conductivity determinations should provide the missile designer with some of the data required in the selection of compatible materials for use as components in parts subjected to high temperatures for brief durations.

MATERIALS

Test specimens used in this investigation were prepared from commercially available stock purchased from regular suppliers of Teflon, Kel-F, and Duroid 5600 plastics. Consequently, the specimens tested are probably typical of the material currently available on the market. The specific gravities of these specimens in the as-received condition were determined to be as follows:

Teflon	- 2.17
Kel-F	- 2.15
Duroid 5600	- 1.99

A discussion of the properties of these three plastic materials can be found in Appendix A.

APPARATUS

Furnace Design and Construction

The thermal conductivity apparatus designed and constructed for this investigation was based on a modification of the "comparative method" technique of Van Dusen and Shelton,[2] as well as more recent versions attributed to W. D. Kingery,[3] W. J. Knapp,[4] and W. A. Scholes.[5] This technique, utilizing unidirectional steady-state heat flow, involves the specimen to be measured and two calibrated standard specimens. The standard specimens are both of the same material, and their variation of thermal conductivity with temperature is known. The specimen to be measured is placed between the two standards as in Figure 1 (Appendix B), where *a* and *c* denote the standards and *b* the unknown specimen. Heat enters the specimen arrangement through the outer surface of one of the standards as indicated in the drawing. The steady-state unidirectional equation for heat flow is;

$$dQ = -kdA\frac{dT}{dx} \tag{1}$$

where dQ denotes an amount of heat per unit time entering a region of cross-sectional area dA and possessing a temperature gradient $\frac{dT}{dx}$. The coefficient of thermal conductivity is represented by *k*, and the minus sign is introduced to coincide with positive heat flows, i.e., flow from high to low temperatures in the increasing *x* direction.

Extending the steady-state Equation (1) to encompass the comparative method technique, which is essentially unidirectional heat flow through a composite wall, certain assumptions will be made. If it is assumed that the cross-sectional areas of the three specimens are equal, then assignment of the letters *a*, *b*, and *c* to these specimens as in Figure 1, and the subsequent modification of the steady-state equation will result in the following relations:

$$\frac{k_a dT_a}{dQ_a dx_a} = \frac{k_b dT_b}{dQ_b dx_b} = \frac{k_c dT_c}{dQ_c dx_c}. \tag{2}$$

Assuming also that for unidirectional heat flow through the arrangement (which can be accomplished very nearly by proper insulation and external guarding of the furnace interior) an average differential heat-flow value is used, which is within the desired experimental limits of error, Equation (2) may then be simplified further. Allowing dQ_b now to be called the average value of heat flow through the specimen combination and equal to

$$dQ_b = \frac{dQ_a + dQ_c}{2} \tag{3}$$

then the thermal conductivity of the unknown specimen *b* can be expressed as follows:

$$k_b = \frac{1}{2}\frac{dx_b}{dT_b}\left[k_a\frac{dT_a}{dx_a} + k_c\frac{dT_c}{dx_c}\right]. \tag{4}$$

If specimens *a* and *c* are of equal thicknesses, Equation (4) can be further reduced to:

$$k_b = \frac{1}{2}\frac{dx_b}{dx_a}\frac{1}{dT_b}\left[k_a dT_a + k_c dT_c\right]. \tag{5}$$

Assuming, finally, that the thermal expansions of the specimen materials are negligible for the relatively narrow range of temperatures employed in this investigation, there will be effectively no variation in the heat-flow path length. As a result, the thermocouple positions will remain fixed. Thus, for any conductivity run, dx_a and dx_b will be constant and will be indicated as x_a and x_b respectively. Therefore, dT_a, dT_b, and dT_c can be written as ΔT_a, ΔT_b, and ΔT_c respectively. The equation for the thermal conductivity of the unknown specimen, k_b, is then

$$k_b = \frac{1}{2}\frac{x_b}{x_a}\frac{1}{\Delta T_b}\left[k_a\Delta T_a + k_c\Delta T_c\right]. \tag{6}$$

The furnace constructed for this experiment satisfied the conductivity relationship above.

Portability was a feature requirement of the furnace. Due to the nature of the decomposition products of the fluorocarbons it was compulsory to provide appropriate safety precautions. The furnace was to be operated within a hood, thereby requiring the selection of materials that combined minimum space with minimum weight. Of course, a furnace requiring good insulating qualities together with a minimum of weight necessitated large volume. A combination of materials was thus selected that met the necessary thermal and mechanical requirements for mobility.

The method of investigation employed consisted of measuring the axial temperature gradients in the two standard specimens with the unknown fluorocarbon specimen placed between them. These gradients resulted from the system being heated at one end and cooled at the other. The exterior surfaces of the system were protected from heat loss by surrounding guard material. When the steady-state condition had been attained, the heat flux being the same throughout the system, the thermal conductivity at any point within the combination was then inversely proportional to the temperature gradient at that point. A cross-sectional diagram of the furnace, with specimens in place, is shown in Figure 2.

The heaters were made in the laboratory due to the specialized design required, which was not readily available from manufacturers. The main heater was formed from an air-dry refractory cement molded in clay with slightly oversize dimensions to allow for shrinkage and subsequent finish-grinding. The geometrical design selected for the tongue-and-groove pattern on the upper face of the heater was one which would generate a constant heat flux per unit area through the small air gap. A guard heater was constructed at the bottom face of the main heater by cutting a double parallel groove which followed the periphery. Helically wound nichrome heating wire was fitted into the grooves. Between the upper face of the main heater and the heat diffusion plate, where there existed the small air gap, ceramic spacers were placed such that possible buckling and subsequent short-circuiting of the heating elements would be prevented. These spacers were positioned so as to cause a minimum of thermal flow interference.

The vertical guard heating elements, which are shown in Figure 2, were also made from nichrome heating wire. These elements were enclosed within hollow ceramic insulators and selectively positioned around the specimens. The furnace insulation used (Babcock and Wilcox K-23 brick), being structurally adequate, was grooved to accept the guard heater insulators to aid in maintaining their placements. The three decks of guard heaters were independently controlled and their temperatures regulated by differential thermocouple techniques (the values of the exterior guard couples, T_G, being required to be equal to the couples, T_M, the locations of which can be seen in Figure 2).

The thickness of furnace insulation required to inhibit lateral flow was determined to be 4-1/2 inches of B. and W. K-23 brick and 1/2 inch of Transite. The Transite was used mainly for structural support to facilitate portability. This insulating combination reduced the temperature at the outside surface of the furnace to values near the ambient temperature, thereby minimizing heat loss.

Temperature Control

Furnace temperature control was accomplished by means of accurately regulated a-c power. Three separate controlling systems were found to be desirable for satisfactory performance of this apparatus, rather than the more conventional types utilized for experimental work of this nature. Operational temperatures were attained and accurately controlled by means of a thermocouple actuated servo-mechanism system transmitting the available power to the main heater. The bottom guard heater was independently regulated by a thermocouple-actuated thyratron power controller. The servo-system was selected as the most satisfactory type of control for this experiment due to its practically step-free voltage output characteristics, as well as its relatively large power output capacity. Selection of the thyratron controller for use as the guard source was prompted mainly because of its sensitivity to small error voltages. Manually controlled variable transformers were used for the power sources to the vertical guard heaters.

Referring to the schematic wiring diagram of Figure 3, for controlling the temperature of the main heater, it can be seen that comparatively basic design features for temperature control were incorporated into the system. A thermocouple contained in a thin-walled, high conductivity ceramic insulator (to permit rapid detection of temperature fluctuations) was positioned near the main heating element. The couple leads were referenced at a Pace Engineering Thermocouple Reference Junction, and extensions then fed to the recorder-controller. The couple's output voltage was recorded by a Bristol Model 560 Wide-Strip Dynamaster Single-Point D-C Potentiometer Recorder-Controller equipped with a control slide-wire mechanically linked to the recording mechanism. The position of the wiper arm on the slide-wire varied with the deviation of the actual temperature from the control point. The control slide-wire in conjunction with an external feedback slide-wire, which was mechanically linked to a servo-motor-operated General Radio Variac variable transformer by means of a gear assembly and common shaft, formed a bridge circuit. The bridge output voltage was converted to a proportionate amount of a-c voltage and then amplified by an external Minneapolis-Honeywell Servo Converter-Amplifier unit to activate the Variac's servo-motor. The unbalance operating the servo-motor caused the sliding contact on the feedback slide-wire, in combination with the recorder's control slide-wire, to adjust itself so as to restore electrical balance in the bridge circuit. This operation simultaneously moved the sliding arm of the variable transformer, thereby causing a correspondingly adjusted voltage to be impressed upon the main heater. The input power raised the temperature of the heater, making necessary a new balance position of the feedback and recorder's control slide-wires but nearer to the bridge balance position. This action resulted in a lower transformer output and proximity to the value of the power required for steady-state conditions to be reached. Operation, although here described in steps, was smooth and continuous, and proceeded until the couple output was exactly matched by that of the bridge circuit.

The balancing action was initiated with the slightest change in the output voltage of the control thermocouple (the amplifier exhibiting a sensitivity of 2 microvolts). Thus, recording and controlling of the temperature at the main heater was practically instantaneous.

Fine adjustment of the volts per turn ratio of the voltage output from the General Radio Variac (to achieve smooth control) was obtained by imposing minimum and maximum limits on the primary coil of the main heater output transformer. This was accomplished by means of two additional Variacs, the primary coils of which were connected in parallel and the secondary coils of which served as the limiting voltage source for the output transformer.

As indicated in Figure 3, a high-limit control was placed in series with the 110 volt, 60 cycle laboratory source. This control was used as a safety precaution against possible amplifier failure, which would result in values of specimen temperature exceeding that which was desired by the setting of the control slide-wire. The device was employed when personal observation of the apparatus was determined to be impractical. The

thermocouple located in the standard sample and nearest to the diffusion plate was switched from the measuring equipment to a potentiometer. The potentiometer, in conjunction with a modified Minneapolis Honeywell Electronic Null Indicator, was adjusted such that voltage from the couple in excess of that corresponding to the temperature desired would activate a relay, thereby open-circuiting the 110 volt line and thus causing all equipment to be automatically shut-down.

The controller used for the bottom guard heater regulation employed a potentiometer unbalance signal to drive a pair of thyratron tubes connected as in a push-pull amplifier circuit. This error signal was developed by matching the output from a guard thermocouple with a potential developed by an internal d-c voltage supply and corresponding to the specimen couple. The controller output to the guard heater varied approximately inversely to the output from the guard thermocouple. Lag in thermocouple sensing was compensated for by adjustment of a variable throttling band, resulting in oscillatory damping.

Measuring Equipment

The measuring equipment consisted of a Leeds and Northrup Type K-3 Universal (Guarded) Potentiometer, a Minneapolis-Honeywell Electronic Null Indicator (galvanometer), an Eppley Standard Cell, a Willard 1½-volt battery, and a Bristol Model 560 Multiple-Point Wide-Strip Dynamaster D-C Potentiometer Recorder.

The potentiometer, which was accurate to approximately 0.5 microvolts, in conjunction with the galvanometer of one microvolt per mm sensitivity, and the standard cell and battery comprised the measuring system used for obtaining the data values of temperature. The couple leads were referenced directly at the input terminals of the temperature compensated multiple-point recorder. This multi-channel instrument made possible a graphic representation of the furnace interior thermal conditions and, in addition, by modifying slightly its automatic channel switching system, served as a means of transferring the thermocouple outputs to the precision potentiometer.

PROCEDURE

Operating Techniques

The determination of thermal conductivity required the assembly of standards and test specimens, and the accurate control of the different heaters to give satisfactory heat flow and temperature distribution. The standards with wedged-in thermocouples were assembled and aligned, and pressures were applied to hold them in place.

The control slide-wire of the Bristol Recorder-Controller was then positioned at the value corresponding to ambient temperature. This caused no unbalance in the bridge circuit, and thus the furnace input variable transformer, being at its minimum course adjustment position, could not

generate a voltage to the main heater. The control slide-wire was then advanced slowly to the desired operating temperature in order that possible thermal shock to the heater as well as adjoining materials would be minimized. Since the servo-system, governed by the slide-wire, maintained the operating temperature at the bottom standard, the combination was then permitted to approach thermal equilibrium conditions. At this time, the thyratron controller, connected to the bottom heater, was placed into operation, and power was supplied to this heater in proportion to the difference in temperature between the standard thermocouple at the bottom and its corresponding guard couple. Again, equilibrium conditions were awaited, whereupon the vertical guard heaters were activated, producing the same temperature gradients in the guard bricks as occurred in the combination. Control was semi-automatic, and the furnace was then allowed to approach steady-state conditions. Increasing the operating temperature required repositioning of the control slide-wire. Slow advancement of the slide-wire was necessary, since the thyratron controller followed the change increase, although with slight unavoidable time delay.

Initially, the guard heater variable transformers required trial-and-error techniques in order to match the guard and specimen couples. but after some experience with the assembly the guard-brick temperatures could be adjusted quite readily. Use of thyratron controllers for regulating the vertical guard heaters would have been desirable; however, unavailability prevented this.

Experimental Procedure

After the determination of the sample dimensions, the test specimens used were originally cut oversize from sheet stock supplied by the manufacturer and annealed to relieve stresses and to insure dimensional stability. The Teflon specimen was heat treated in a forced convection oven by maintaining a constant temperature of 590°F for 4 hours and then allowing it to be cooled slowly within the furnace after disconnecting the power input leads. It was noted that Teflon's thickness increased approximately 4 percent accompanied by an expected decrease in width and length of about 3 percent. Similarly, the Kel-F specimen was maintained at 390°F for 24 hours and then allowed to cool slowly. For this case, the resultant thickness was observed to have increased by approximately 3½ percent and the width and length to have decreased by about 2 percent. In view of the fact that little information was available concerning Duroid 5600 the same heat treatment described for Teflon was employed. For Duroid 5600, the thickness was observed to have increased by approximately 6 percent but accompanied by no noticeable change in width and length.

Following heat treatment, the specimens were machined to the desired final dimensions. The specific gravities of the heat-treated specimens were found to be:

Teflon	- 2.18
Kel-F	- 2.16
Duroid 5600	- 1.90

The figures indicate a very slight increase in the specific gravity of both Teflon and Kel-F, and a slight decrease in Duroid 5600 as contrasted to the as-received condition.

In Figure 2, the test specimen (S_2) was centered between the two standard samples (S_1 and S_3), which were of 3.79 specific gravity, high purity alumina. The standards and test specimen surfaces were all carefully lapped. Thermocouple holes (0.030 in. in diameter) were drilled by a Sheffield ultrasonic machine tool from one face of the standard to its axis of symmetry, thereby eliminating any lateral extrapolation of the data. These holes were located at equal distances from the top and bottom faces of each of the standards, which were of the same thickness. This conformed to the reduced thermal conductivity Equation (5), where dx_a was set to equal to dx_c. As in Knapp's equipment,[4] thermocouple holes were drilled, only into the standard specimens, and the temperature differential in the unknown test specimen was determined by extrapolation along the axis of symmetry of the combination. This mathematical method was employed only after experimentation with thermocouples* imbedded in the test specimens had shown that interfacial resistance did not, to a noticeable degree, impair values obtained by the extrapolation. Elimination of these two test specimen couples reduced furnace set-up time and provided for fewer sources of error.

Each of the thermocouples located in the standard specimens was held in place by means of alumina wedges. Leads from the couples were carried out laterally from the furnace through alumina insulators imbedded in the guard brick and adjoining insulation brick, as shown by the detail in Figure 4. The use of guard brick material identical to that of the individual specimens assured a miminum of skew temperature gradients.

The area dimension of each of the standards complete with alumina guard brick (flush fitted) was 6 inches by 6 inches. The test specimen with its guard brick of identical material, and of the same area dimensions as the standards, was positioned between the standard specimens. Thin silver foil (0.001-inch thick) was placed at the resulting interfaces in order to insure optimum heat transfer. This combination of specimens was then positioned in the thermal conductivity furnace as indicated in Figure 2. A metal heat sink plate set on top of the specimens completed the assembly. In addition to serving as a heat sink, this plate also exerted a light pressure upon the specimens, thereby minimizing interfacial resistance by providing good surface-to-surface contact. The test specimens used were machined from commercial grades of Teflon, Kel-F, and Duroid 5600.

The ventilating system in the hood in which the furnace was located was turned on prior to the heating of the furnace. Thereupon, power was supplied to the heating coils, as described previously, until steady-state conditions were attained. Temperatures were assumed to have reached steady-state when test specimen average temperature variations of less

Chromel-alumel, used throughout the entire experiment.

than one percent per hour were obtained. The temperatures at which thermal conductivity determinations were made had been arbitrarily selected prior to experimentation; however, some modification was found to be desirable during particular runs. The gel-points of each of the test materials governed the maximum temperature of the individual tests. Thermocouple readings were taken, and the data were then tabulated and reduced.

RESULTS AND DISCUSSION

Figure 5 shows the resulting curves obtained for the thermal conductivities of the three materials tested at elevated temperatures. The value of conductivity for Teflon, as reported by the du Pont Company, is 1.7 BTU/hr/ft^2/°F/in. at room temperature for a thickness of 0.18 inch. The experimentally determined thermal conductivity of Teflon, for a thickness of 0.172 inch, decreased from 2.80 BTU/hr/ft^2/°F/in. at an average temperature of 570.4°F to 2.35 at 331.5°F, and, when extrapolated to a room temperature of 70°F, decreased to approximately 1.84 BTU/hr/ft^2/°F/in. Since the gel-temperature of Teflon is about 620°F, the maximum mean value reached for a conductivity determination was 570.4°F. This limitation was due to the fact that the hot-face temperature of the test specimen was 621.5°F, and obtaining significant results above the gel-point appeared impractical.

The maximum average temperature reached for a Kel-F thermal conductivity determination was 375.8°F. Due to the gel-temperature of Kel-F being approximately 414°F, the maximum average value attained corresponded to a hot-face temperature of 408.7°F. The conductivity of Kel-F, as noted in the M. W. Kellogg Company Technical Bulletin 1-1-55, is 0.418 BTU/hr/ft^2/°F/in. at room temperature. However, information regarding the thickness of the test specimen used in their determination was not available. The experimental values obtained for the conductivity of Kel-F decreased from 1.72 BTU/hr/ft^2/°F/in. at 375.8°F to an extrapolated value of 0.70 at a room temperature of 70°F for a specimen thickness of 0.164 inch. A break in the curve was observed in the vicinity of 225°F.

Duroid 5600, being a fiber-reinforced Teflon-base product, was expected to exhibit slightly higher values of conductivity compared to the virgin Teflon. In view of the lack of thermal data, Teflon's gel-temperature was used to govern the maximum test temperature of the Duroid 5600 material. As can be seen from Figure 5, the slope of the conductivity curve for this specimen is similar to that for Teflon. At 537.5°F, the value of the conductivity for Duroid 5600 was 3.99 BTU/hr/ft^2/°F/in. decreasing to 3.13 at 185°F and 2.80 BTU/hr/ft^2/°F/in. when extrapolated to a room temperature of 70°F, for a thickness of 0.219 inch. The maximum average value of 537.5°F corresponded to a hot-face temperature of 593.9°F making further determinations at higher temperatures infeasible.

The design of the equipment was such that experimental errors were eliminated to as great an extent as possible without sacrificing any more

convenience than necessary. The specimen size used provided satisfactory thermal distribution in the guarding system, as well as good temperature uniformity.

Temperature measurements obtained from the thermocouples located within the standards eliminated the errors and necessary assumptions connected with surface measuring techniques. The additional difficulty encountered in the machining of thermocouple holes in the standards was well repaid by the increase in accuracy and reliability obtained. Improper location of couples results in relatively large output deviations and makes adequate guarding experimentally impossible.

Reproducibility of the data for the three materials tested was good. Temperature control was continuously maintained within ±0.5°F at values between approximately 185 and 570°F for periods of time up to 96 hours. Temperature control for times greater than 96 hours to obtain steady-state conditions was found to be unnecessary. Experience with the apparatus showed that steady-state heat-flow conditions could be maintained to at least within one percent error for periods of approximately 6 to 16 hours. It was found that approximately one hour was sufficient time to obtain steady-state heat-flow conditions to within 10 percent of the final value.

It should be noted that the values determined were for single heats of material as received from the manufacturer and then annealed. Fabrication techniques and additives, such as plasticizers, will undoubtedly alter the thermal conductivity values to some degree depending upon the variation in conditions.

The results indicate that these experimental conductivity determinations for Teflon and Kel-F exceed somewhat the room temperature values reported in the manufacturers' literature. Since interfacial thermal resistance influences conductivity values inversely, poor surface contact necessarily results in larger differential specimen temperatures, thereby decreasing these values. It is conceivable, therefore, that low values of conductivity could be the result of high interfacial resistance, or poor surface contact, as well as differences attributable to conditions of the test materials and experimental techniques.

It is apparent from the resultant curves of Figure 5 that the deviations from the means for the three materials are relatively small when considering the narrow range of the ordinate axis. Comparing these deviations with those obtained by other experimenters for a similar thermal conductivity range, guarding evidently was quite adequate. This is due to the fact that the lateral heat loss by specimens developed in similar apparatus varies inversely as their thermal conductivity. Thus, the results obtained are satisfactory within the limits of error tolerated by comparative method techniques.

Estimated Accuracy

An article by Somers and Cypers[6] indicates that the maximum error due to sample dimensions is 2 percent. The article consists of steady-state heat-transfer calculations by means of the solution of partial differential equations for heat flow in homogeneous materials of rectangular cross section and various thicknesses. One face of the sample is assumed to be at a given temperature, the other face at a temperature greater than the first. The side of the sample is assumed to be at the same temperature as one face, which is more drastic than the actual case. The authors report the ratio of true thermal conductivity to that measured experimentally for various test sections, guard sections, and sample thickness dimensions.

Since careful consideration was given to the instrumentation, errors due to measurements alone should be less than one percent. However, errors produced by slight misalignment of the heaters, variations in the resistance of the heater wires, small inaccuracies in machining and thermocouple location, and other random errors account for the spread in data.

With these points in mind, it is believed that the curves drawn through the experimental data are accurate to within 5 percent.

SUMMARY

1. Compatible results were obtained with the thermal conductivity apparatus designed and constructed for this investigation. A technique commonly referred to as the "comparative method" was employed. Considerable care was given to the selection of instrumentation and design of the circuits for the control of power to the main heater and guard heaters, and for the measurement and recording of temperatures.

2. These experiments were conducted on commercially available material as received from the producer and thereupon annealed. Test control techniques as well as condition of test samples are factors which will affect the thermal conductivity coefficients determined.

3. The thermal conductivities of Teflon, Kel-F, and Duroid 5600 increased as the test temperatures were increased. The conductivity of Teflon increased from approximately 1.84 BTU/hr/ft^2/°F/in, at a room temperature of 70°F to 2.80 at 570.4°F; similarly, the Kel-F conductivity increased from 0.70 at room temperature to 1.72 at 375.8°F; Duroid 5600 values increased from 2.80 to 3.99 at 537.5°F.

ACKNOWLEDGMENT

It is with pleasure that thanks are extended to Mr. Anthony G. Martin, Watertown Arsenal Laboratories, particularly for his design of the servo and thyratron control systems, as well as for his many valuable suggestions and generous assistance during this investigation.

REFERENCES*

1. LEVITT, A. P. and WONG, A. K., "A Survey of the Elevated Temperature Properties of Teflon and Kel-F," Watertown Arsenal Laboratories Technical Note 397/9 (August 1956).

2. Van DUSEN, M. S. and SHELTON, S. M., "Apparatus for Measuring Thermal Conductivity of Metals up to 600°C," *National Bureau of Standards Journal of Research*, 12, 429-40 (1934).

3. KINGERY, W. D. and FRANCL, J., "Thermal Conductivity Apparatus for Determining Thermal Conductivity by a Comparative Method," *Journal of the American Ceramic Society*, 37, 2, 80-4 (1954).

4. KNAPP, W. J., "Thermal Conductivity of Nonmetallic Single Crystals," *Journal of the American Ceramic Society*, 26, 2, 48-55 (1943).

5. SCHOLES, W. A., "Thermal Conductivity of Bodies of High BeO Content," *Journal of the American Ceramic Society*, 33, 4, 111-17 (1950).

6. SOMERS, E. V. and CYPERS, J. A., "Analysis of Errors in Measuring Thermal Conductivity of Insulating Material," *Review of Scientific Instruments*, 22, 8, 583-6 (1951).

7. DOBAN, R. C., SPERATI, C. A., and SANDT, B. W., "The Physical Properties of 'Teflon' Polytetrafluoroethylene," *Society of Plastics Engineers Journal*, II, (November 1955).

8. MADORSKY, S. L., HART, V. E., STRAUS, S., and SEDLAR, V. A., "Thermal Degradation of Tetrafluoroethylene and Hydrofluoroethylene Polymers in Vacuum," *National Bureau of Standards*, 51, 327 (1953).

9. "'Teflon' Tetrafluoroethylene Resin, Health Hazards Involved in Heating," duPont Information Bulletin No. X-59 a (March 1955).

10. HARRIS, D. K., "Polymer-Fume Fever," *The Lancet*, 261, 1008-1011 (1951).

11. SENDROY, J., et al., "Composition and Effects of Vapors Emanating from Insulated Electrical Equipment Under Operation," Naval Medical Research Center Report on Project NM004-5.02.01, Bethesda, Maryland, (1950).

12. STOCKINGER, H. E., "'Teflon' - A Plastic with an Inhalation Hazard," *Occupational Health*, 13, 88, (1953).

13. Haskell Laboratory, "Toxicity of Pyrolysis Products of (Tefion Tetrafluoroethylene Resin," Paper presented before American Industrial Hygiene Association, April 20, 1955.

**References 7 - 15 from Appendix A.*

14. WALL, L. A. and MICHAELSON, J. D., "Thermal Decomposition of Polytetrafluoroethylene in Various Gaseous Atmospheres," *National Bureau of Standards, Journal of Research*, 56, 27 (January 1956).

15. GREENMAN, N. L., "New Fiber-Reinforced Fluorocarbon," *Materials and Methods*, 42, 110 (November 1955).

APPENDIX A

Teflon*

This material is a high polymer of tetrafluoroethylene, $(C_2F_4)_n$, and is the most chemically inert thermoplastic as yet developed. At temperatures below 575°F, there has been no substance uncovered which is capable of swelling or dissolving Teflon. However, chemical attack by molten alkali metals, such as sodium or potassium, chlorine trifluoride, and gaseous fluorine at elevated temperatures, has been observed.

In addition to Teflon's remarkable thermal and chemical stability, it possesses other physical properties which make it attractive for mechanical and electrical applications. At room temperature, the material has a tensile strength ranging from approximately 1,500 to 3,000 psi (to 15,000 psi with oriented films) depending on environmental conditions and has an impact strength of 3 ft-lb/in. increasing to 6 ft-lb/in. at 170°F. Teflon possesses a low coefficient of friction and non-adhesiveness over a wide range of temperatures. It demonstrates good dielectric characteristics relatively independent of temperature from 10^2 to 10^8 cps and very high surface and arc-resistivities. Its water absorptivity is exceedingly small resulting in excellent weathering ability.**

At elevated temperatures, commencing in the vicinity of 530°F, a colorless, odorless, toxic gas is evolved accompanied by a weight loss of material. This rate of decomposition increases with temperature.[7,8] In view of the toxicity[9,10,11,12,13] of the gaseous product, some caution must be exercised in the machining, fabrication, and utilization of Teflon materials under conditions wherein high temperatures are either necessarily or accidently produced. No solid material has yet been found to catalyze or inhibit the decomposition of Teflon.[14]

Above 621°F, the crystalline structure of Teflon is converted into an amorphous, rubbery gel of extremely high viscosity. However, even at temperatures approaching this crystalline melting point, many useful physical properties are still retained.

Kel-F***

Kel-F is the polymer of trifluorochloroethylene, which is characterized by the formula $(CF_2 - CFCl)_n$. Although similar to Teflon in many of its physical properties the substitution of a chlorine atom into the structural ethylene molecule contributes to the transparency, exceptional flow, and rigidity characteristics of the polymer. The chemical inertness and zero moisture absorptivity of Kel-F are attributed to the fluorine atoms. It is

Trade-mark of the E. I. duPont Nemours Company, Inc., which is the only commercial producer of this plastic in the United States at the present time.

***Properties data cited here are from duPont Bulletin A-2069.*

****Trade-mark of the M. W. Kellogg Company. Similar material is produced by the Acme Resin Company and the Bakelite Company under the names of Polyfluoron and Fluorothene respectively.*

impervious to most corrosive chemicals and is highly resistant to most organic solvents. However, slight swelling of the polymer may occur with some highly halogenated and aromatic materials.*

At room temperature, the tensile strength of this material ranges from 4,600 to 5,700 psi (from 30,000 to 50,000 psi with oriented films) and with an impact strength of 3.6 ft-lb/in. A general comparison of some of the room temperature properties of Teflon and Kel-F indicates that Kel-F has a lower coefficient of thermal conductivity, lower specific heat, higher Young's modulus of elasticity, and less ductility. Both plastics are nonflammable and have similar specific gravity values. In general, Kel-F is easier to fabricate than Teflon.

Another unique feature of this fluorocarbon plastic is that it is amenable to heat treatment. Slow cooling from about 390°F results in a crystalline material which is milky in appearance, hard, and resistant to distortion. The rate of cooling controls the quantity and size of the crystals formed. These crystals tend to aggregate as spherulites which form in sheath-like groups and grow until restricted by adjacent crystalline areas. Thus, slow cooling produces fewer and larger crystals and spherulites, resulting in relatively low light transmission, impaired clarity, reduction in solvent absorption, and a harder, more brittle product. However, rapid cooling or quenching produces many small crystals, thereby resulting in a product exhibiting maximum light transmission characteristics, clarity, and a higher degree of flexibility.

Plasticizers (low molecular weight trifluorochloroethylene oils and waxes) are sometimes added to increase the flexibility of the plastic, but the resultant product is limited in its usefulness in high temperature applications.

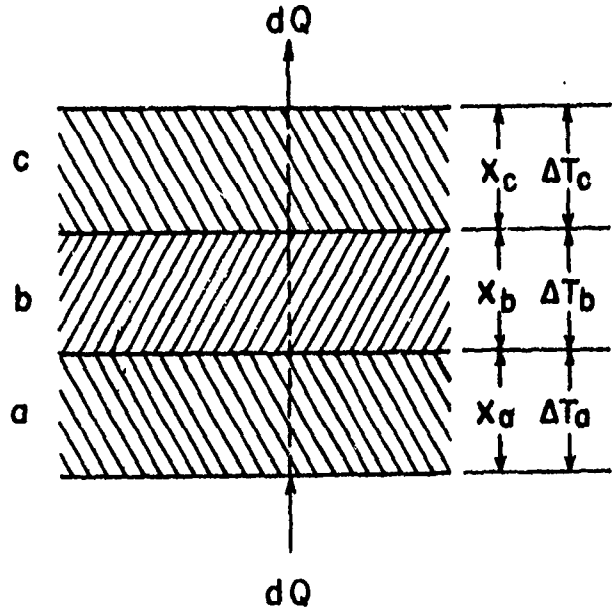
Duroid 5600**

A modification of Teflon plastics has been developed by the Rogers Corporation. This material, designated Duroid 5600, is an inorganic fiber-reinforced Teflon-base product, the purpose of which is to improve the cold flow and high temperature distortion characteristics of virgin Teflon while retaining most of its desirable properties. It is claimed that the heat distortion temperature under a load of 66 psi is 250°F for Teflon and 500°F for Duroid 5600. Limited data available[15] indicate that the tensile strength, impact strength, thermal expansion, deformation under load, short time dielectric strength, and specific gravity of Duroid 5600 are lower than that of Teflon. However, stiffness, coefficient of friction, and water absorption are reported to be comparatively higher. At room temperature, the specific heats of Teflon and Duroid 5600 are similar.

*These and other data regarding relative chemical resistance of unplasticized quenched Kel-F polymer are listed in M. W. Kellogg Co. Technical Bulletin 1-1-55.

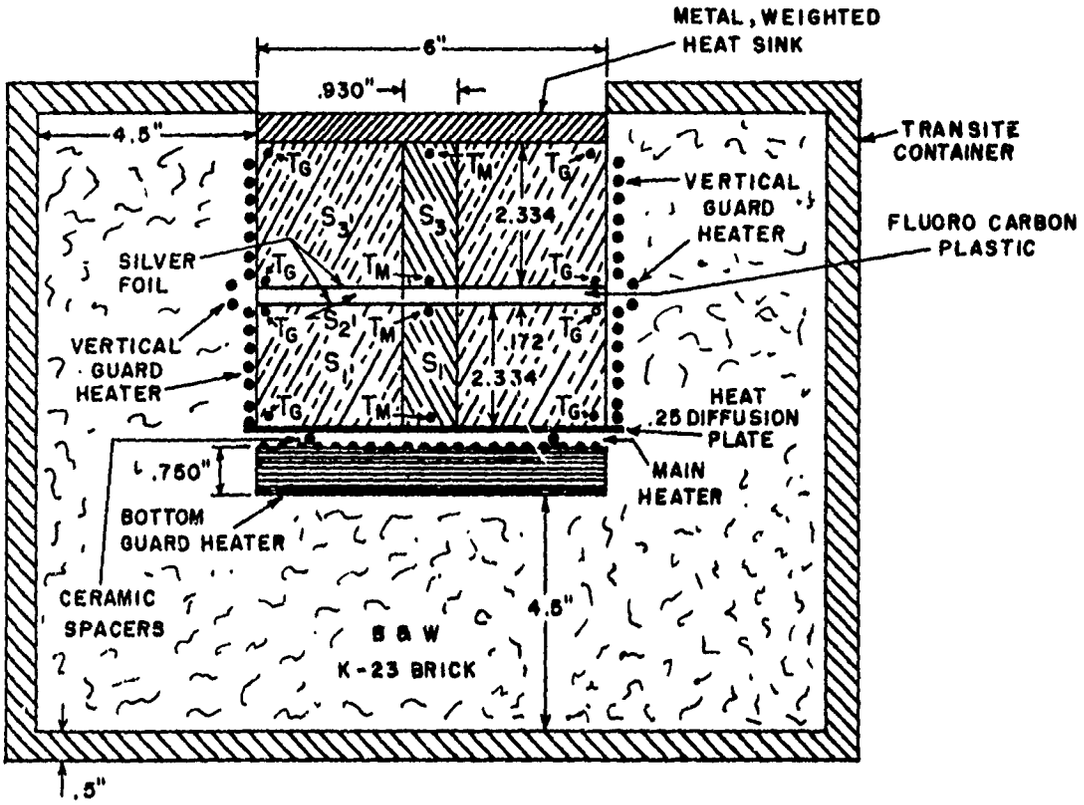
**Trade-mark of the Rogers Corporation, Rogers, Connecticut.

APPENDIX B



HEAT FLOW THROUGH A COMPOSITE WALL

FIGURE I



S_2 = UNKNOWN SPECIMEN

S_2' = SPECIMEN GUARD MATERIAL

S_1 & S_3 = STANDARD

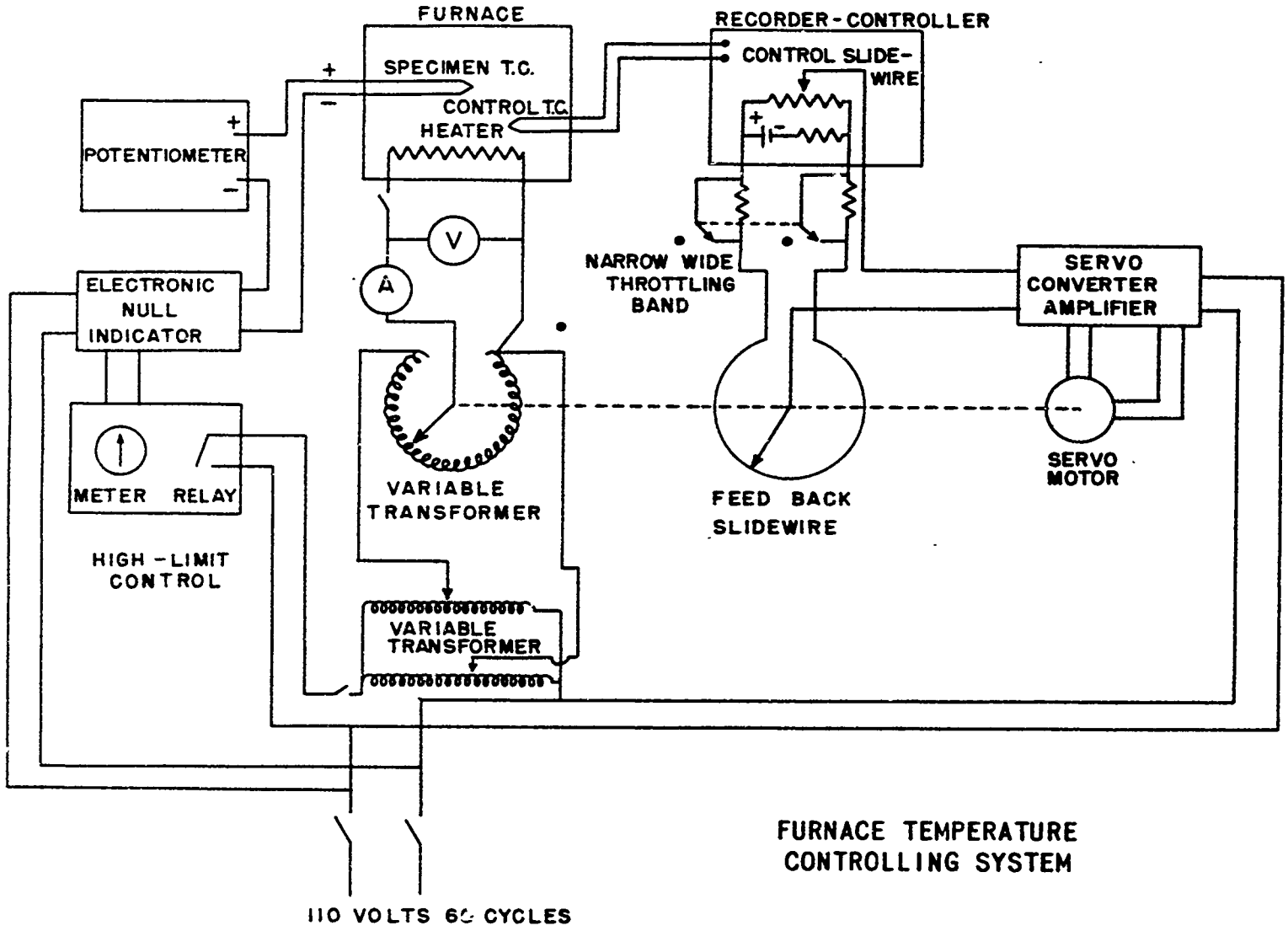
S_1' & S_3' = GUARD BRICK

T_M = MEASUREMENT THERMOCOUPLES

T_G = GUARD THERMOCOUPLES

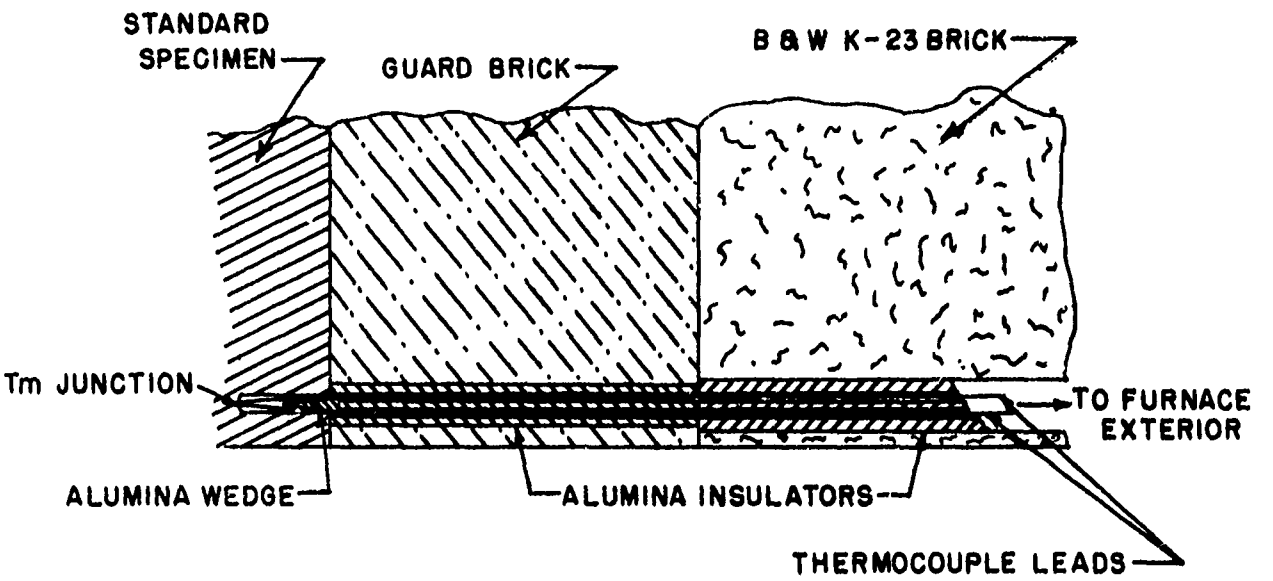
THERMAL CONDUCTIVITY FURNACE

FIGURE 2



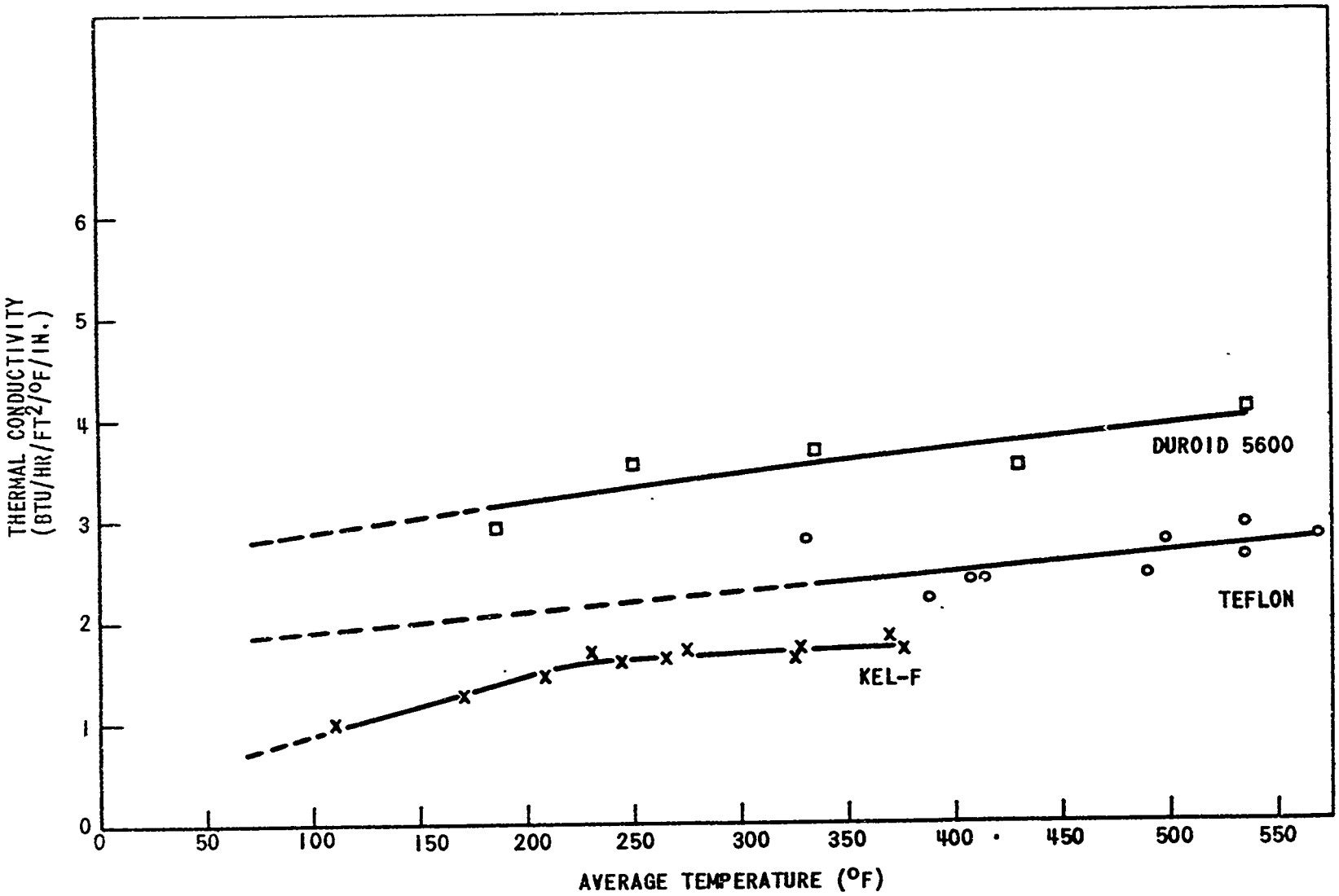
FURNACE TEMPERATURE CONTROLLING SYSTEM

FIGURE 3



THERMOCOUPLE PLACEMENT

FIGURE 11



THERMAL CONDUCTIVITY VERSUS AVERAGE TEMPERATURES OF TEFLON, KEL-F, DUROID 5600

FIGURE 5

WATERTOWN ARSENAL

TECHNICAL REPORT DISTRIBUTION

Report No.: WAL TR 397/10 Title: "Thermal Conductivity of Teflon, Kel-F, and Duroid 5600 at Elevated Temperatures"

To	No. of Copies
Commanding Officer Watertown Arsenal Watertown 72, Mass. Attn: ORDBE-L	5
ORDBE-Z	1
Commanding General Army Ballistic Missile Agency Redstone Arsenal, Alabama Attn: ORDAB-IP	7
ORDAB-DSR	1
Commanding General Ordnance Weapons Command Rock Island, Illinois Attn: ORDOW-TB	2
Commanding Officer Office of Ordnance Research Box CM, Duke Station Durham, North Carolina	1
Armed Serv. Tech. Info. Agency Arlington Hall Station Arlington 12, Va.	5
Commanding General Aberdeen Proving Ground Aberdeen, Maryland Attn: Dev. & Proof Serv.	1
Ballistic Res. Labs.	1
Commanding General Frankford Arsenal Philadelphia 37, Pa. Attn: Pitman-Dunn Labs.	1
Commanding General Picatinny Arsenal Dover, New Jersey Attn: Engineering Division	1

To	No. of Copies
Commanding General Redstone Arsenal Huntsville, Alabama Attn: Structures Branch	1
Commanding Officer Detroit Arsenal Center Line, Michigan Attn: Materials Lab.	1
Commanding Officer Rock Island Arsenal Rock Island, Illinois Attn: Engineering Div.	1
Commanding Officer Diamond Ordnance Fuze Labs. Washington 25, D. C.	1
Commanding Officer White Sands Proving Ground Las Cruces, New Mexico Attn: ORDBS-OM, Tech. Library	1
Commanding General Research and Engineering Command Army Chemical Center Maryland	1
Technical Reports Library SCEL, Evans Signal Corps Lab. Belmar, New Jersey	1
Commanding Officer Signal Corps Engineering Lab. Ft. Monmouth, New Jersey Attn: Director of Research	1
Office of the Chief Signal Officer Engineering and Technical Division The Pentagon Washington 25, D. C. Attn: SIGGD	1
Commanding Officer U. S. Naval Proving Ground Dahlgren, Virginia	1

To	No. of Copies
Chief, Bureau of Ordnance (AD3) Department of the Navy Washington 25, D. C.	1
Commanding Officer U. S. Naval Powder Factory Indianhead, Maryland	1
Director U. S. Naval Ordnance Lab. White Oak Silver Spring, Maryland	1
Director Naval Research Laboratory Washington 25, D. C.	1
U. S. Atomic Energy Commission Technical Information Service P. O. Box 62 Oak Ridge, Tennessee Attn: Chief, Library Branch	1
U. S. Atomic Energy Commission Washington D. C.	1
Commander Wright Air Development Center Wright-Patterson Air Force Base Dayton, Ohio Attn: WCRRL	1
Commanding General Air Research and Development Command P. O. Box 1395 Baltimore 3, Maryland Attn: RDD	1
Commanding General Air University Maxwell Air Force Base Alabama Attn: Air University Library	1
David Taylor Model Basin Department of the Navy Washington 25, D. C.	1

To	No. of Copies
Director National Bureau of Standards Washington 25, D. C.	1
Corona Laboratories National Bureau of Standards Corona, California	1
Commanding Officer Engineering Res. & Dev. Labs. Fort Belvoir, Virginia	1
Chief Naval Ordnance Test Station Inyokern China Lake, California	1
National Adv. Comm. for Aeronautics 1512 H Street, N. W. Washington 25, D. C.	1
Cambridge Research Center A.R.D.C. Lawrence G. Hanscom Field Bedford, Mass. Attn: Mr. Lawrence Sharpe	1
National Adv. Comm. for Aeronautics Langley Field Virginia	3
Office of Naval Research 495 Summer Street Boston, Mass. Attn: Mr. J. H. Flanagan	1
Commander Portsmouth Naval Shipyard Portsmouth, New Hampshire Attn: Mr. E. C. Taylor, Code 251B	1
Navy Office Army Ballistic Missile Agency Huntsville, Alabama	1
TOTAL COPIES DISTRIBUTED	58

ARMY WATERTOWN ARSENAL, MASS.