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RESULTS OF TESTS FOR ADHESION IN AIR AND IN VACUUM OF TITANATE COATINGS APPLIED TO 321-STAINLESS-STEEL BRAYTON PARASITIC-LOAD RESISTORS

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ABSTRACT

Tests were run to determine the adhesion of a high-emissivity plasma-sprayed coating of either iron titanate or calcium titanate to the 321-stainless-steel surface of the Brayton Parasitic Load Resistor during operation of the Parasitic Load Resistor in air and in a vacuum in the temperature range of 1700° R to 2000° R. Iron titanate did adhere during operation in a vacuum, but iron titanate did not adhere during operation in air. Calcium titanate adhered during operation both in a vacuum and in air.

RESULTS OF TESTS FOR ADHESION IN AIR AND IN

VACUUM OF TITANATE COATINGS APPLIES TO 321-STAINLESS-

STEEL BRAYTON PARASITIC-LOAD RESISTORS

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SUMMARY

Iron titanate was selected as a coating for the Brayton Parasitic Load Resistor to provide a high-emissivity surface for radiating heat in a vacuum. The Parasitic Load Resistor must operate both in air and in a vacuum. Previous experience with iron titanate as an emissive coating had been limited to small samples in a vacuum.

After the Parasitic Load Resistor contractor had experienced poor adhesion of iron titanate to the 321-stainless-steel surface of the Parasitic Load Resistor during operation in air, the NASA Lewis Research Center initiated a program to re-evaluate iron titanate and to evaluate calcium titanate as candidates for this application.

A resistor element from the Parasitic Load Resistor was coated with iron titanate and heat cycled both in vacuum and in air. Previous results were confirmed in that the iron titanate coating performed adequately in a vacuum and in that it "flaked off" during the first cool-down period in air.

A resistor element from the Parasitic Load Resistor was coated with calcium titanate and heat cycled both in vacuum and in air. The calcium titanate adhered during the heat cycles under both atmospheric conditions.

INTRODUCTION

The NASA Lewis Research Center is currently engaged in a Brayton-cycle space-power technology program. The Brayton power conversion system being developed is expected to have applicability for solar, nuclear reactor, and radioisotope space power systems in the net power range of 2 to 10 kWe at 1200 hertz.

The Brayton rotating unit is a constant-speed single-shaft unit. The 1200-hertz-alternator shaft is supported by gas bearings with a compressor overhung on one end and a turbine overhung on the other end of the single shaft.

The constant rotational speed is maintained by a frequency-sensitive parasitic-load type of speed control on the electrical output of the alternator. As the net shaft input power from the turbine varies or as the demand for useful vehicle load varies, the speed control maintains a constant shaft speed by dissipating the excess generated power into an electrical parasitic load. Therefore with a constant input power to the alternator, the electrical load on the alternator is maintained constant by varying the amplitude of the parasitic load so that the sum of the useful vehicle load and the parasitic load is constant.

The Parasitic Load Resistor consists of nine sections. Each section is capable of dissipating 2 kilowatts of electric power by radiating the heat to space. A complete description of the Parasitic Load Resistor program is available in reference 1.

Iron titanate was selected as an emissive coating to increase the emissivity to 0.88 (ref. 2) from the 0.40 which is the emissivity of oxidized 321-stainless steel (ref. 3). The iron titanate coating "flaked off" during the cool-down period following the first application of electrical power for 5 hours to the iron-titanate-coated Parasitic Load Resistor in air at the contractor's facility.

As a result of this failure of the iron titanate coating to adhere to the 321-stainless-steel casing of the Parasitic Load Resistor, tests were run at the NASA Lewis Research Center to reevaluate iron titanate and to evaluate calcium titanate as candidates for this application.

BACKGROUND

The Parasitic Load Resistor is shown in figure 1. The selection of a unit which radiates its heat to space is a departure from SNAP-8 for which the heat is removed by immersion in a liquid coolant.

The basic resistive element used in the Parasitic Load Resistor consists of a Nichrome V wire inside a 321-stainless-steel casing with magnesium oxide packed between the wire and the casing. The casing has an outside diameter of 0.500

inch (1.27 cm), a wall thickness of 0.065 inch (0.156 cm), and a nominal length of 31 inches (78.7 cm). The Nichrome V wire is spiral wound and centered along the length of the casing. Both the centering and the pitch of the spiralled wire are carefully controlled during manufacture. The magnesium-oxide powder is packed into the casing around the wire so that no voids are visible when the completed resistive element is x-rayed in various planes. The ends of the Nichrome V wire are welded to end connections which contain pure nickel terminals. As with all radiating surfaces, the operating temperature of the Parasitic Load Resistor at rated power is dependent upon the emissivity of its surface. At rated power and with the emissivity of 0.40 for oxidized 321 stainless steel, the Parasitic Load Resistor has a maximum case temperature of 1940° R. The addition of the iron-titanate coating would raise the emissivity to 0.88 and lower the maximum case temperature to 1710° R.

Calcium titanate appeared to be an equally appropriate emissive coating, but iron titanate was selected because of its intended use in the heat sources of the Brayton power conversion system.

The properties of the iron titanate as well as the preparation and application procedures were in all instances in accordance with the work performed in reference 2.

A prototype section of the Parasitic Load Resistor was plasma coated with iron titanate by the contractor, and rated electrical power was applied with the prototype section in air at ambient conditions. After five hours application of electrical power, the power was removed and the section was allowed to cool. As the section cooled the iron titanate coating flaked and fell from the prototype section of the Parasitic Load Resistor.

INVESTIGATION AT NASA LEWIS RESEARCH CENTER

The initial step in the investigation at the NASA Lewis Research Center (LeRC) of the problems associated with the adhesion of iron-titanate coating to the Parasitic Load Resistor was to confirm the experience of the contractor under controlled conditions.

Two obvious possibilities were considered which would contribute to the failure of the iron titanate to adhere:

(1) Iron titanate of improper composition and/or purity,

(2) Improper plasma coating technique.

Therefore, a Parasitic Load Resistor element having a 321-stainless-steel casing was coated at Lewis Research Center by a controlled technique using iron titanate of known composition and purity as evolved under NASA Contract NAS3-4174 (ref.2).

Rated electrical power was applied to the iron-titanate-coated Parasitic Load Resistor element in air for 35 minutes. Power was then removed, and the element was allowed to cool by natural convection. Approximately 9 minutes after the electrical power was removed (i.e., after the cooling cycle had been initiated), the iron-titanate coating began to flake from the Parasitic Load Resistor element, the flakes literally jumping from the Parasitic Load Resistor element. Within 19 minutes after initiation of the cooling cycle, very little of the iron-titanate coating remained on the Parasitic Load Resistor element.

These results under controlled conditions substantiated the results obtained by the Parasitic Load Resistor contractor. Therefore, it was decided to investigate other possible high-emissivity coatings for the Parasitic Load Resistor.

Calcium titanate was also a candidate emissive coating for this application because of the previous experience with it (ref. 2). A Parasitic Load Resistor element (element 40) was coated with calcium titanate and subjected to a series of heat cycles both in air and in a vacuum. Another Parasitic Load Resistor element (element 104) was coated with iron titanate to check iron titanate's performance under vacuum conditions.

The Parasitic Load Resistor element has an effective heater length of approximately 21 inches (53 cm) with approximately 4 inches (10 cm) of unheated length at each end where the electrical terminal is attached. Five thermocouples were attached to each element as shown in figure 2. Two thermocouples were placed near the center of the length of each element and the other three thermocouples were placed between this center section and one end of the element. Each element was suspended by its two electrical leads so that the only conductive paths for heat removal were the electrical leads and these were attached to the coldest portions of the element.

A "run" in these tests consisted of a heating period followed by a cooling period. Rated electrical power was applied to the Parasitic Load Resistor element until well after temperature readings had stabilized. The calcium-titanate-coated element in vacuum averaged 18 minutes, the calcium-titanate-coated element in vacuum averaged 16 minutes, and the iron titanate in vacuum averaged 13 minutes in order for the hot-spot temperature to stabilize. In general, electrical power was applied for 35 to 40 minutes. Then the electrical power was removed, and the element was allowed to cool to ambient conditions.

The first six runs were conducted on the calcium-titanate-coated element in air. The calcium titanate adhered for these six runs in air, and these positive results justified a continuation of the investigation of calcium titanate as a candidate for this application.

During the heating portion of the first of these six runs (run 1), the center 19 inches (48 cm) of the length of the calcium-titanate-coated element changed color from its normal charcoal gray to a pale yellow as the temperature surpassed approximately 1260° R. The remaining 5 inches (13 cm) on each end did not reach a temperature of 1260° R, and there was no color change in these end sections. There were no furthur color changes during runs 1 through 6 in air.

Table 1 tabulates the stabilized temperatures on the calcium-titanate-coated element for runs 1 through 6 which were conducted in air. Temperature measurements where color change did not occur (T_1 and T_2) were relatively consistent for the six runs. Where change in color had occurred (T_3, T_4, and T_5), the stabilization temperatures decreased from run 1 through run 4.

The next five runs were made on the same calcium-titanate-coated element but in a vacuum chamber rather than in air. The vacuum level ranged from 10^{-5} to 10^{-7} torr.

On run 7 (the first run in vacuum after six runs in air), the center 19 inches (48 cm) of the calcium-titanate-coated element changed color from the pale yellow back to the original charcoal gray as temperatures exceeded 1260° R. This first run in a vacuum apparently reversed the process which caused the center section of the calcium-titanate-coated element to turn pale yellow on the first run in air. No further color changes occurred during the five runs in the vacuum chamber.

Table 2 tabulates the stabilized temperatures on the calcium-titanate-coated element for runs 7 through 11 which were conducted in the vacuum chamber. Temperature measurements where color change did not occur (T_1 and T_2) were relatively consistent for the five runs. Where a change in color had occurred (T_3, T_4, and T_5), the stabilization temperatures decreased from run 7 to run 9. The emissivity at T_4 and T_5 was calculated for run 11 and found to be 0.85.

After the five runs in the vacuum chamber, five additional runs were made in air on the same calcium-titanate-coated element.

On run 12, the first rerun in air, the pale yellow color again appeared on the portion of the calcium-titanate-coated element that exceeded 1260° R. As in the earlier runs in air, no further changes in color occurred on subsequent runs in air.

Table 3 tabulates the stabilized temperatures on the calcium-titanate-coated element for runs 12 through 16 which were reruns conducted in air. Temperature measurements where color change did not occur (T_1 and T_2) were relatively consistent over the five runs. Where a change in color had occurred (T_3, T_4, and T_5), the temperatures showed less change then in the initial air runs and in the vacuum chamber runs, but this time the changes were upward rather than downward.

X-ray diffraction readings were made for six different conditions of the calcium titanate with results as shown below.

Sample Examined	$CaTiO_3$	TiO_2	CaO	$Ca_2Al_2SiO_7$ and/or $Ca_2Mg(SiO_4)$ 2
1. Calcium titante powder prior to being applied to element.	Strong	Medium	Strong	----
2. Sprayed calcium titanate prior to application of heat (sample scraped from element)	Strong	Trace	----	----
3. Calcium titanate heat changed to pale yellow color after run 1 in air. Sample taken after run 6	Strong	----	Medium	----
4. Calcium titanate that did not change color during runs 1 through 6. Sample taken after run 6	Strong	----	Weak	----
5. Calcium titanate heat changed back to charcoal gray from pale yellow during run 7 in vacuum. Sample taken after run 11.	Strong	----	Medium	Strong
6. Calcium titanate that changed to pale yellow color after run 12 in air. Sample taken after run 16.	Strong	----	Medium	----

The $Ca_2Al_2SiO_7$ and/or $Ca_3Mg(SiO_4)_2$ is probably from the asbestos and glass which were present in the vacuum chamber with the element. These compounds are missing after the succeeding runs in air.

Evaluation of these analyses did not provide any conclusive evidence regarding the change in color of portions of the calcium titanate coating or its significance.

In order to have a complete picture, a Parasitic Load Resistor element (element 104) was plasma coated with iron titanate for three runs in the vacuum chamber. The results tabulated in table 4 verified that the iron titanate will adhere to the 321-stainless-steel element if used only on runs in a vacuum. The emissivity at T_4 and T_5 for run 19 was calculated and found to be 0.79.

In table 5, the results from the final run for each of the four conditions (calcium titanate run in air, calcium titanate run in the vacuum chamber, clacium titanate rerun in air, and iron titanate run in the vacuum chamber) are shown for comparison. The temperatures for the iron-titanate-coated element are higher because more heat was being dissipated by this element. The current used in all tests was held constant, but the resistance of the iron-titanate-coated element was 109 percent of the resistance of the calcium-titanate-coated element.

CONCLUSIONS

The purpose of these limited tests was to determine the adhesiveness of calcium titanate and iron titanate to the 321-stainless-steel surface of the Parasitic Load Resistor elements during a normal operational cycle both in air and in a vacuum.

The following conclusions were reached as a result of these tests:

(1) Iron titanate will not adhere for even one heating and cooling cycle in air. This confirmed the findings of the contractor.

(2) Iron titanate will adhere at least for three cycles in a vacuum chamber. This confirmed previous work on iron titanate coatings in a vacuum as reported in reference 2.

(3) Iron titanate is not satisfactory for this application.

(4) Calcium titanate will adhere at least during limited operation cycles in air and in a vacuum. Further testing under controlled conditions is required on the calcium titanate coating

to assure that it will perform adequately over a five-year period. For example, the significance of the color change when calcium titanate is used in air and in a vacuum must be determined.

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	T_1(°R)	T_2(°R)	T_3(°R)	T_4(°R)	T_5(°R)
Run #1	740	1120	1500	1540	1540
Run #2	740	1120	1500	1530	1530
Run #3	750	1120	1490	1520	1520
Run #4	740	1120	1470	1510	1510
Run #5	735	1120	1480	1510	1510
Run #6	740	1120	1480	1510	1510

TABLE 1 - STABILIZED TEMPERATURES ALONG CALCIUM TITANATE COATED PARASITIC LOAD RESISTOR ELEMENT IN AIR

	T_1(°R)	T_2(°R)	T_3(°R)	T_4(°R)	T_5(°R)
Run #7	960	1270	1590	1640	1650
Run #8	960	1270	1580	1630	1640
Run #9	950	1270	1560	1610	1610
Run #10	950	1270	1560	1610	1610
Run #11	950	1270	1560	1610	1610

TABLE 2- STABILIZED TEMPERATURES ALONG CALCIUM TITANATE COATED PARASITIC LOAD RESISTOR ELEMENT IN VACUUM

E-5143

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	T_1(°R)	T_2(°R)	T_3(°R)	T_4(°R)	T_5(°R)
RUN #12	720	1090	1470	1500	1500
RUN #13	720	1100	1490	1510	1510
RUN #14	730	1100	1490	1510	1510
RUN #15	730	1100	1490	1510	1510
RUN #16	730	1100	1500	1520	1520

TABLE 3 - STABILIZED TEMPERATURES ALONG CALCIUM TITANATE COATED PARASITIC LOAD RESISTOR ELEMENT ON RERUNS IN AIR

	T_1(°R)	T_2(°R)	T_3(°R)	T_4(°R)	T_5(°R)
RUN #17	970	1360	1670	1690	1690
RUN #18	970	1360	1660	1680	1680
RUN #19	970	1340	1650	1660	1660

TABLE 4 - STABILIZED TEMPERATURES ALONG IRON TITANATE COATED PARASITIC LOAD RESISTOR ELEMENT IN VACUU

	T_1 (°R)	T_2 (°R)	T_3 (°R)	T_4 (°R)	T_5 (°R)
RUN #6 CALCIUM TITANATE IN AIR	740	1120	1480	1510	1510
RUN #11 CALCIUM TITANATE IN VACUUM	950	1270	1560	1610	1610
RUN #16 CALCIUM TITANATE IN AIR (RERUN)	730	1100	1500	1520	1520
RUN #19 IRON TITANATE IN VACUUM	970	1340	1650	1660	1660

TABLE 5 - RETABULATION OF FOUR SPECIFIC RUNS OF TITANATE COATED PARASITIC LOAD RESISTOR ELEMENTS

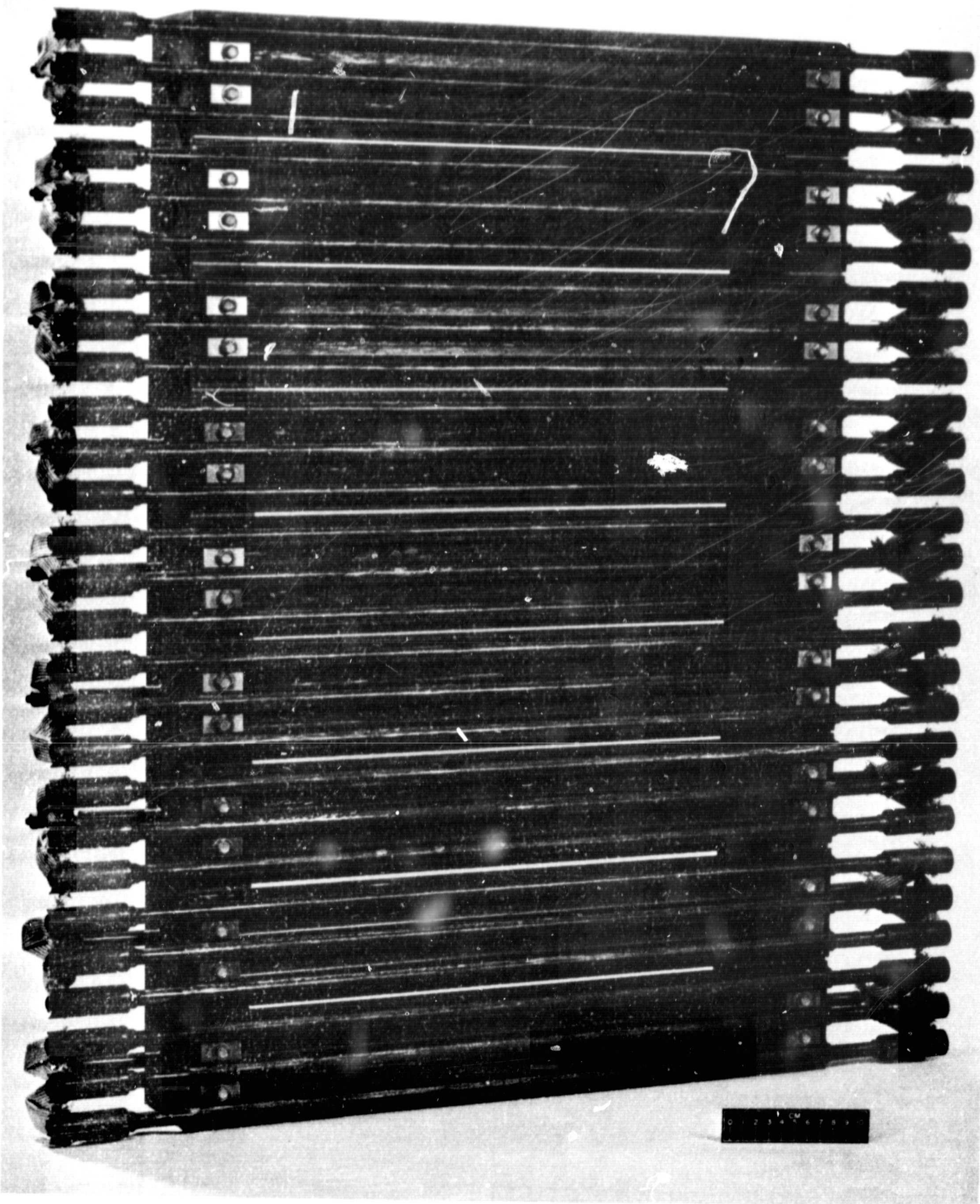

Figure 1. - Parasitic load resistor.

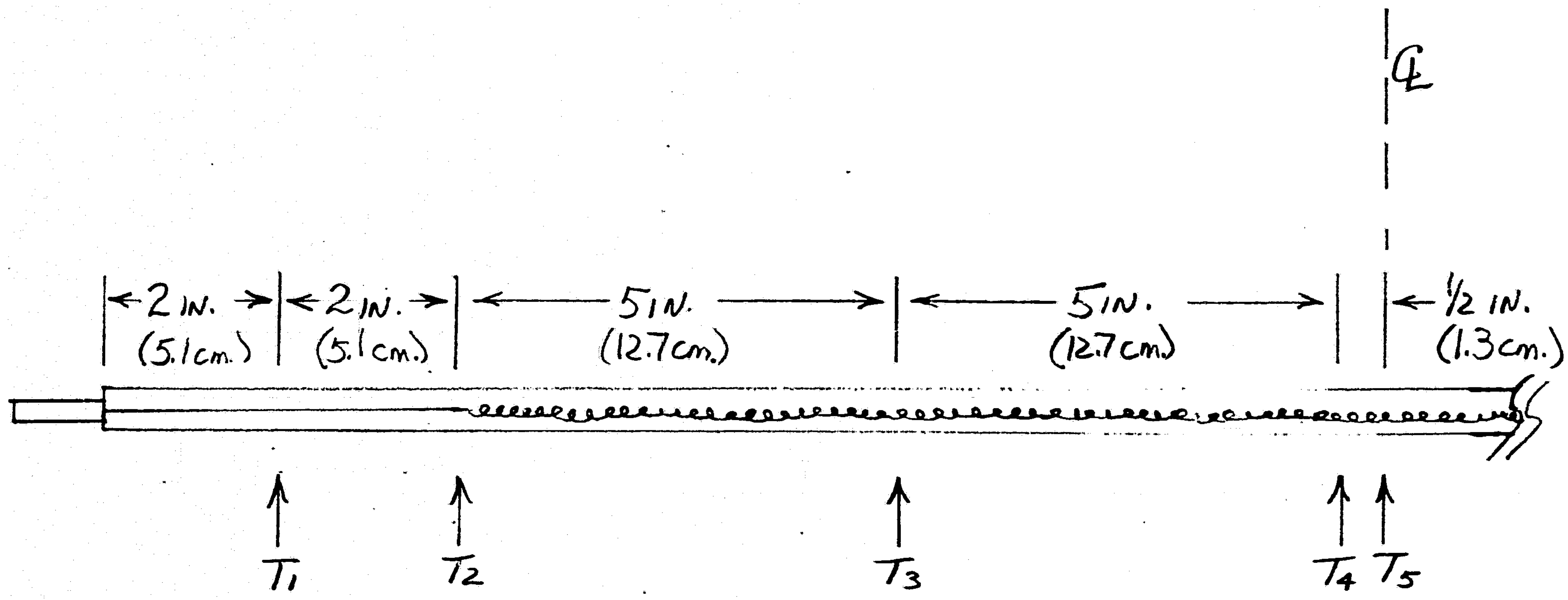

FIGURE 2 – THERMOCOUPLE PLACEMENT ON PARASITIC LOAD RESISTOR ELEMENTS IN LEWIS RESEARCH CENTER INVESTIGATION