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HEAT FLUX SENSOR RESEARCH & DEVELOPMENT: THE COOL FILM CALORIMETER

Final Report
25 January 1990
Corrected 30 June 1990

Prepared for:

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Hampton, Virginia

In Response to:

Task Assignment 6
Contract No. NAS1-18570

LOCKHEED AERONAUTICAL SYSTEMS COMPANY-BURBANK
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ABS: The goal was to meet the measurement requirement of the NASP program for a gauge capable of measuring heat flux into a 'typical' structure in a 'typical' hypersonic flight environment. A device is conceptually described that has fast response times and is small enough to fit in leading edge or cowl lip structures. The device relies heavily on thin film technology. The main conclusion is the description of the limitations of thin film technology both in the art of fabrication and in the assumption that thin films have the same material properties as the original bulk material. Three gauges were designed and fabricated. Thin

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film deposition processes were evaluated. The effect of different thin film materials on the performance and fabrication of the gauge was studied. The gauges were tested in an arcjet facility. Survivability and accuracy were determined under various hostile environment conditions.

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1.0 INTRODUCTION

The work described in this report has the basic goal of meeting the measurement requirement of the NASP program for a gauge capable of measuring heat flux into a "typical" structure in a "typical" hypersonic flight environment. The basis of this work is a report on the state of the art of heat flux measurement technology, NASP report No. 445 titled "Instrumentation development for the National Aerospace Plane." This report described conceptually a device that had fast response times and was physically small enough to fit in leading edge or cowl lip structures. This device was called the "cool film calorimeter" and relied heavily on thin film technology.

In this report a great deal of effort was expended to adapt the embryonic technology of thin films to the sensor concept. Some limited success is reported. The main conclusion is the description of the limitations of thin film technology both in the art of fabrication (it is often a black art) and in the assumption that thin films have the same material properties as the original bulk material. The development process involved three specific attempts to design and fabricate the gauges, each iteration being a significant improvement on the previous one. Contract resource limitations, however, limited the concept development, and the lessons learned will be applied to future development efforts. A section of the report specifically deals with this aspect.

Existing heat flux gauges suffer from several drawbacks. They are generally complex in their construction; thus, it is not possible to easily manufacture very small units. In addition, most conventional designs are large and therefore, suitable only for steady-state applications. While being quite accurate and robust in the steady-state mode, the existing gauges are entirely unsuitable for applications in which response time is critical. The reason for this is not only due to the complex construction, but also due to the fact that, in almost all cases, the concept underlying the design of the gauge itself is based on steady heat conduction.

To overcome these and other shortcomings of current heat flux sensor technology, a project was launched to develop a new heat flux gauge based on a concept that is ideally suited for fast response-time applications. The new concept is simple enough to permit the manufacture of units that are smaller than the state-of-the-art units available commercially.

Figure 1 is an applicability plot for the various types of heat flux gauges. Commercially available gauges, except for the null point calorimeter,have response times of the order of 50 milliseconds. Maximum heat flux capabilities are between 50 and 100 BTU/ft^{2}sec. The null point calorimeters can be used for applications where a 1-millisecond response time is required and for high heat-flux environments. These gauges, however, suffer from the same time limitations as the cool film calorimeters. It is not advisable to use these devices in experiments that take longer then 50 milliseconds because the gauges are based on unsteady heat conduction and cannot operate properly when the gauge reaches steady-state conditions internally. The cool film calorimeter can be built to have response times down to 1 microsecond and heat flux capabilities to over 10,000 BTU/ft^2 sec. This extends heat flux measurement capabilities well beyond current response-time limitations. Other concepts, such as the thin-film differential gauge, and the boron carbide differential gauge can further extend the heat-flux measurement capability envelope for slower speed, high heat-flux applications; but they are not addressed here since they are beyond the scope of this project.

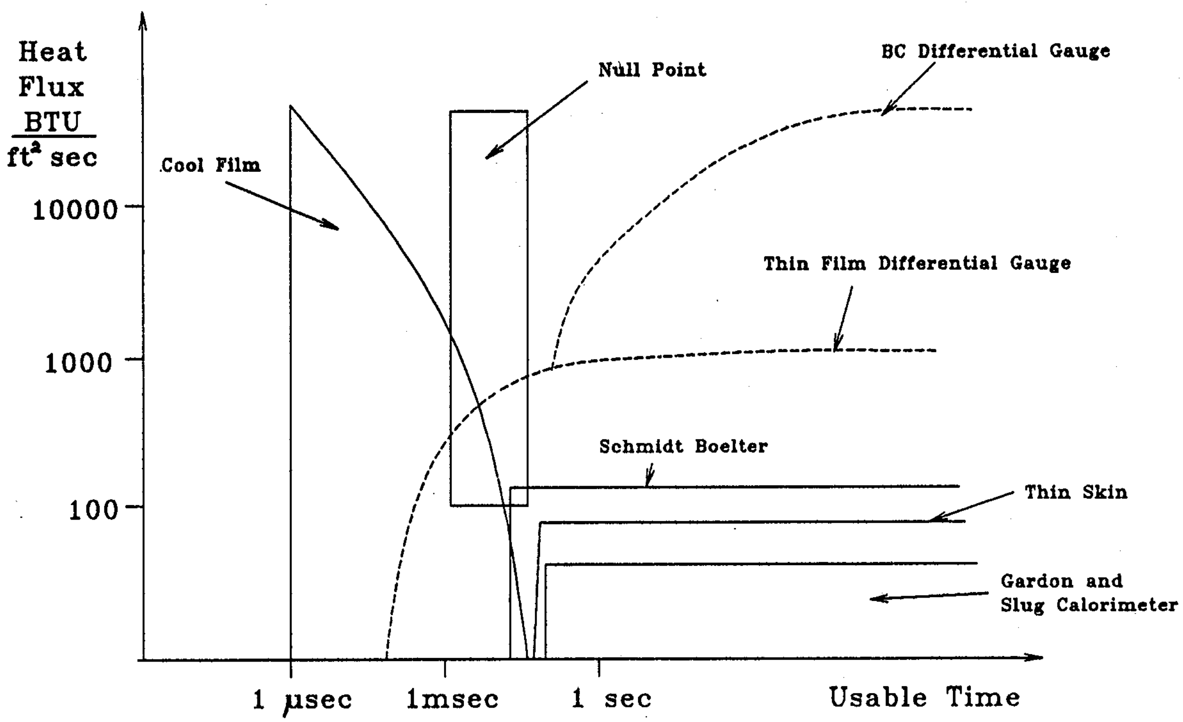

Figure 1: Heat Flux Gauge Applicability

The concept used was this project is that of measuring the surface temperature of a semi-infinite solid exposed to heat. The proposed system consisted of a small diameter rod with a suitably designed thin-film platinum RTD (Resistance Temperature Detector) at the exposed face. This system is thus called the <u>cool film calorimeter concept</u>. If insulation is installed around the rod, the conduction of heat away from the face is one-dimensional and unsteady. The key to the fast response times of this type of probe is the fact that the probe itself is based on an unsteady phenomenon; therefore, this concept did not rely on reaching a steady-state condition, which takes time. Theoretically (in the absence of the RTD at the face) the response time would even be zero. Figure 2 shows a sketch of the proposed system.

Currently, when high-speed response is required, the approach used is that of painting a thin platinum layer onto parts of the model. This thin platinum layer serves as an RTD for surface temperatures. However, even the thinnest painted layer cannot approach the response time of a true vacuum-deposited thin film. Also, the model itself is never a semi-infinite solid; therefore to compute the heat flux from surface temperature data, a complicated numerical scheme must be employed.

It is necessary at this point to consider the question of "why does one want to measure heat flux anyway?", since the rationale of end use can impact the design philosophy.

There are two basic reasons for measuring heat flux. The first concerns the desire to estimate the structural heat loads, while the second reflects a desire to assess the thermal environment of the external fluid flow.

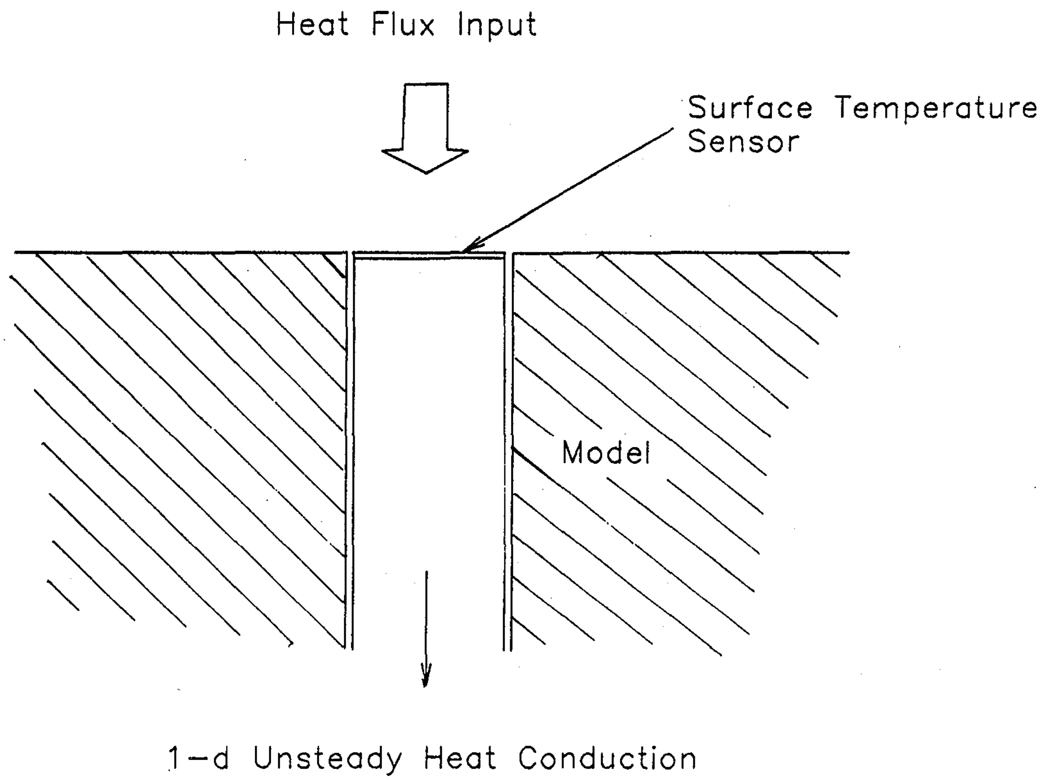

Figure 2: Basic Principle of the Cool Film Calorimeter

Structural heat loads generated by external fluid motion/combustion are either radiative of convective in nature, or some combination of both. If radiation dominates then the energy radiation balance of the gauge must be matched as closely as possible to those of the surrounding body. This means that the absorbtivity must be matched and the re-radiated energy, which depends on probe face temperature and emissivity, must be similar. If the face temperature of the gauge in normal operation is significantly higher then that of the surrounding body the emissivity must be lower in order to match re-radiated heat. If convection dominated the heat transfer then the gauge must have similar thermal conductivities as the surrounding material, thus maintaining similar surface temperatures to ensure heat transfer coefficient similarity. If a combination of effects is expected, one must try to match both requirements in the gauge design if the results are to have any meaning at all.

These effects are also important when the basic desire is to assess the external environment since local gauge "hot spots" can disturb the boundary layer or induce catalytic effects that are not representative of the surrounding structure heat transfer characteristics.

The basic requirements for this heat flux gauge are operation at face temperatures of up to 2000 degrees F and heat flux levels of 4000 Btu/ft^2-second. In addition to the thermal requirements above the sensor was required to be small enough to accurately measure heat flux in small radius structures such as inlet cowl lips and and wing leading edges.

2.0 OBJECTIVE

The primary objective of this project was to explore the possibility of building a heat-flux gauge based on the cool film calorimeter concept, using thin film technologies. The use of thin-film technology in the fabrication of heat-flux sensors creates the possibility of building a gauge with response times in the microsecond range; and, if used in conjunction with other technologies, like photolithography or laser etching, it creates the potential for fabrication of extremely small sensors with a face diameter of less than 1mm.

After the design and fabrication of prototype probes, the study of the function and characteristics of these systems was a secondary objective, as was the determination of suitable data reduction and calibration schemes. In particular, the response times and survivability of the gauges were assessed. Also the influence of the various design parameters and materials used to fabricate the gauge were determined.

3.0 APPROACH

A simple analysis, based on the solution for the heat equation in the case of a semi-infinite solid was performed first. This was a necessary precursor for the development of an effective design and in the selection of materials and fabrication processes.

A suitable data reduction technique was a by-product of this analysis effort. The resulting data-reduction algorithm was kept simple, since it was not the main design objective. This algorithm simplicity assisted in the rapid reduction of data, which allowed more time to be spent on the design, fabrication, and test phases.

The next phase, after the analysis, was that of design and fabrication. In this phase an initial design was developed, and an attempt was made at fabrication. After gaining some valuable information about fabrication, changes were required in the design.

A test phase followed after a design was successfully fabricated. Initial tests were performed in house, and several evaluation tests were performed at two different arcjet facilities.

The design work was preceded by a study of the literature on the state of the art in heat flux gauge technology. This included some of the work done on room temperature, fast response thin film based gauges (1-3) and survey papers describing all currently available types of gauges and their characteristics (6) as well as papers describing specific types of gauges in detail (4,5).

4.0 PROBE DESIGN

4.1 Response Times

The response time of the ideal sensor is ideally zero. The ideal sensor is one where the rod is made of a single material, and the temperature measured is that of the actual face. The real sensor, in actual fact, has the RTD on the face, which changes the heat conduction characteristics in that region. Additionally, the sensor cannot be placed right at the face of the probe, thus, the temperature measured by the sensor is not the true face temperature. One of the main objectives of this project was to investigate the application of thin-film type sensors at the face of the rod to minimize the response time. The main reason for the finite response time in the type of sensor described here is the thermal inertia of the face thermometer assembly.

G.S. Ambrok (1) offers an excellent treatise of the thermal inertia of thin coatings. The results of his analyses are cast in terms of the time it takes for the conduction of heat through a thin coating to closely approximate a steady state. If the flow of heat is abruptly started into the coated surface for a very short time, the conduction of heat through the coating is an unsteady problem. After this, while the substrate still is conducting the heat in an unsteady fashion, the conditions within the coating are such that the temperature distribution and heat transfer correspond closely to a steady-state process. The time required for the temperature difference across the thin coating to reach 95 % of its steady-state value can be computed by the following simple equation :

$$t = \frac{x^2}{0.04a} \qquad \text{Eqn. 1}$$

where x is the thickness of the coating and a is the thermal diffusivity of the coating material. Figure 3 shows the characteristic time as a function of coat thickness for a variety of materials. While it is conceivable to use knowledge of the coating transient characteristics during the data reduction to achieve a zero response time, it is not advisable to use the sensor for phenomena that are shorter than the characteristic time of the combined coatings that make up the RTD sensor at the face of the probe and its protective coating.

The temperature development on the two sides of the coating is shown in Figure 4. After the initial period ($t<t_o$), the temperature difference across the film became constant and roughly equal to the value for steady-state heat conduction. This value is given by :

$$\Delta T = \frac{qd}{Ak} \qquad \text{Eqn.2}$$

where q is the heat flux, A is the area of the coating, d is the thickness of the coating, and k is the thermal conductivity. Figure 5 shows this temperature difference as a function of film thickness for three different heat-flux levels and various different coating materials.

In general, data reduction routines presume that the temperature measured by the thin-film RTD is the true face temperature of the rod. However, to protect the RTD from both electrical charges in the flow and abrasion or particle impact, some hard dielectric layer must be added on top of the RTD. This causes the measured temperature to differ from true face temperature,which creates a potential source of inaccuracies.

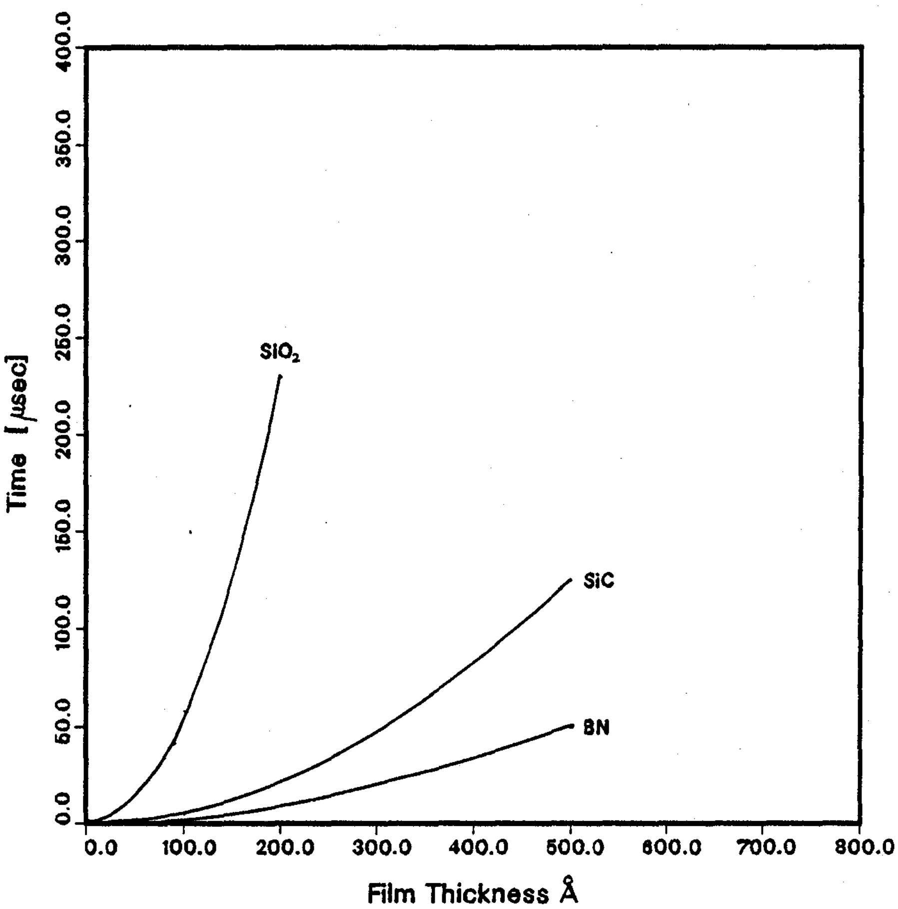

Figure 3: Film Characteristic Time vs. Thickness

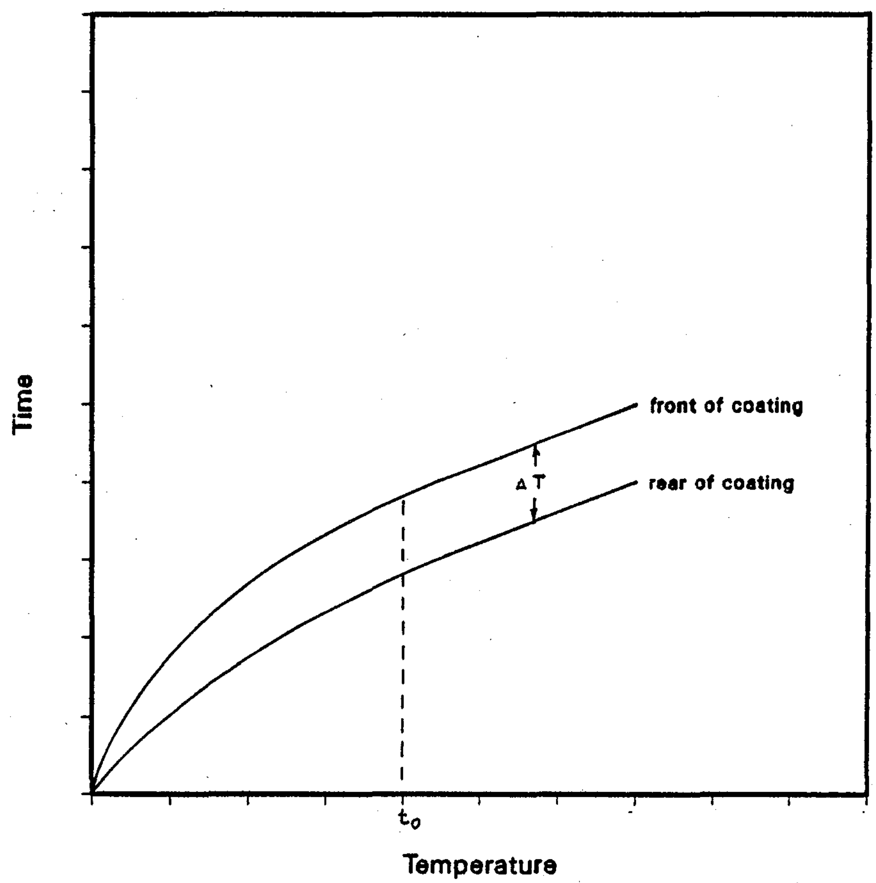

Figure 4: Temperature in Front and Rear of Thin Film

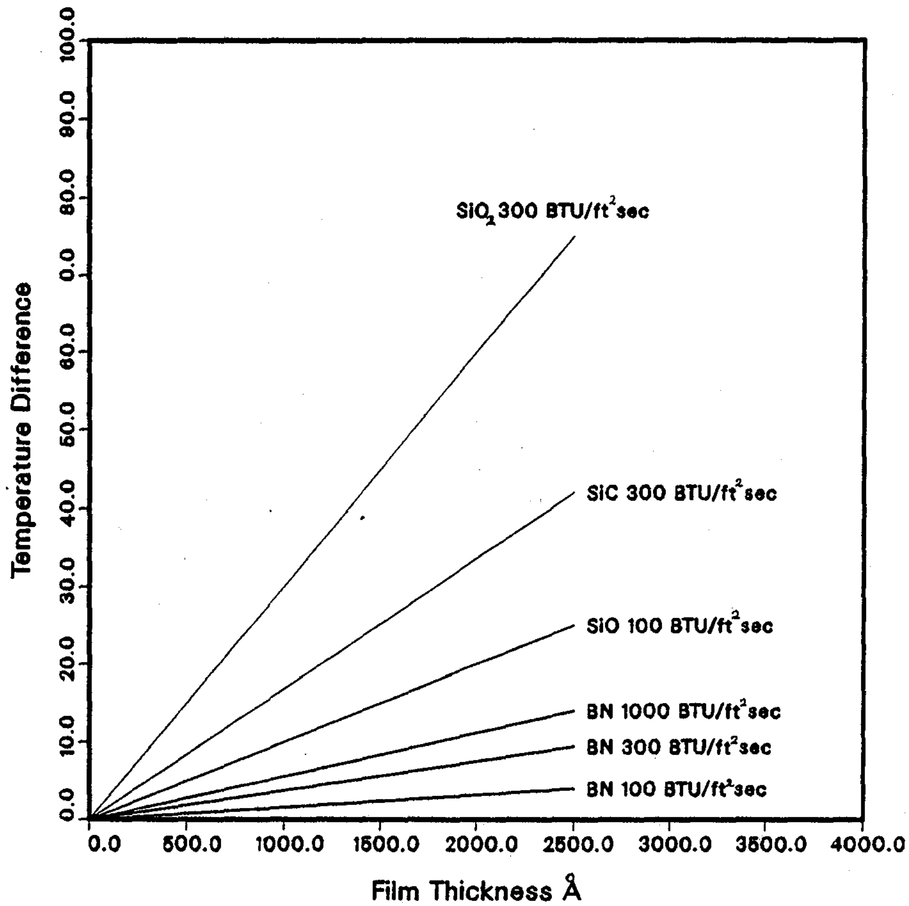

Figure 5: Temperature Drop Across Film vs. Thickness

Because the thermal conductivity of the conducting rod should be high, the materials of choice for the rod are usually electrical conductors. Using an electrical conductor for the rod makes it necessary to isolate the RTD from the conductor by means of another dielectric coat. This coat has an additional temperature drop and additional thermal inertia is thus associated with it. The effect of the various coats on the rod face is an overall reduction in the face temperature of the actual heat-conducting rod.

The effect of the various coatings can be compensated for during data reduction. Two fundamentally different approaches can be chosen with the effect of compensating for the two different types of inaccuracies the films introduce.

In the first approach, it is presumed that the conduction of heat in the film can be described by the equation for steady heat conduction. A numerical procedure, using this approach, is simply to base the temperature drop, in all the films involved, on the computed heat flux into the face. This temperature drop can then be used in the next time step to obtain the actual face temperature that is to be used to find the new heat flux value. While this procedure will theoretically yield an exact result, it cannot be used when the input heat flux rate of change is high. This means that this approach is not usable when the input heat flux changes significantly during one response time of the combined film coating, as developed earlier. In essence, this approach can be used when response time can be finite; but, the heat flux value must be determined very accurately.

The second approach uses the thermal capacity of each of the films within the numerical data reduction scheme. Here the films have to be treated as unsteady regions, since the computational schemes are much more difficult. However this approach can supply a much more accurate face temperature history of the sensor and, thus, reduce the response time of the sensor below the value needed to reach a steady-state in the film. Details of the numerical inaccuracies are discussed in the data reduction section (4.3) of this report.

There are two other sources of inaccuracies that exist in the sensors discussed here. Both stem from the fact that the real situation in the probe is not exactly that of one-dimensional heat conduction in a semi infinite solid. Some of the heat will be conducted "sideways" out of the rod, which generates a design requirement for some type of thermal insulator to surround the probe shaft. This problem will be more significant if the temperature of the probe is much higher than the temperature of the surrounding body; it also becomes more severe with rods that are of smaller diameter.

The other potential source of inaccuracy is due to the finite length of the rod. While this can be a significant problem if one is trying to compute the heat-flux using the solutions to the heat equation for a semi-infinite solid, the use of numerical schemes eliminates this problem altogether. It is, in fact, easier to include a finite boundary condition in the numerical scheme rather than the infinite one.

4.2 Concept Limitations

The underlying concept of this sensor is unsteady heat conduction into a semi-infinite solid. Accordingly, the sensor measures the heat flux into a semi-infinite solid. This has several implications for the usable data time and accuracy. Since the probe is embedded in a body that is not a semi infinite solid and inadvertently has significantly different heat conduction and capacity characteristics from the probe, the heat flux into the body is only identical to the heat flux into the probe at the instant the temperature of the environment is first raised. This means that in a typical experiment the heat-flux data provided by the probe match the heat flux into the body for only a short time at the beginning of the experiment. Thereafter the sensor face temperature is different from the surrounding surface temperature. Additional problems may be generated by this, due to the influence of the hot spot (or cold spot depending on relative thermal conductivity between shear and surrounding material) generated by the sensor on the thermal boundary layer. This limitation becomes more important for cooled structures since the sensor is inherently not externally cooled.

In addition to the concept limitation described above, certain material integrity limitations do exist. In a high heat flux application, the temperature of the face of the probe will rise rapidly. Figure 6 shows the face temperature as a function of time for a variety of input heat flux values. These curves are based on the solution of the heat equation for a semi infinite solid with constant heat flux into the face. Assuming that the materials constituting the face of the probe are able to withstand higher temperatures, then the substrate, the substrate melting temperature becomes the limiting temperature of the probe (for copper, this is about

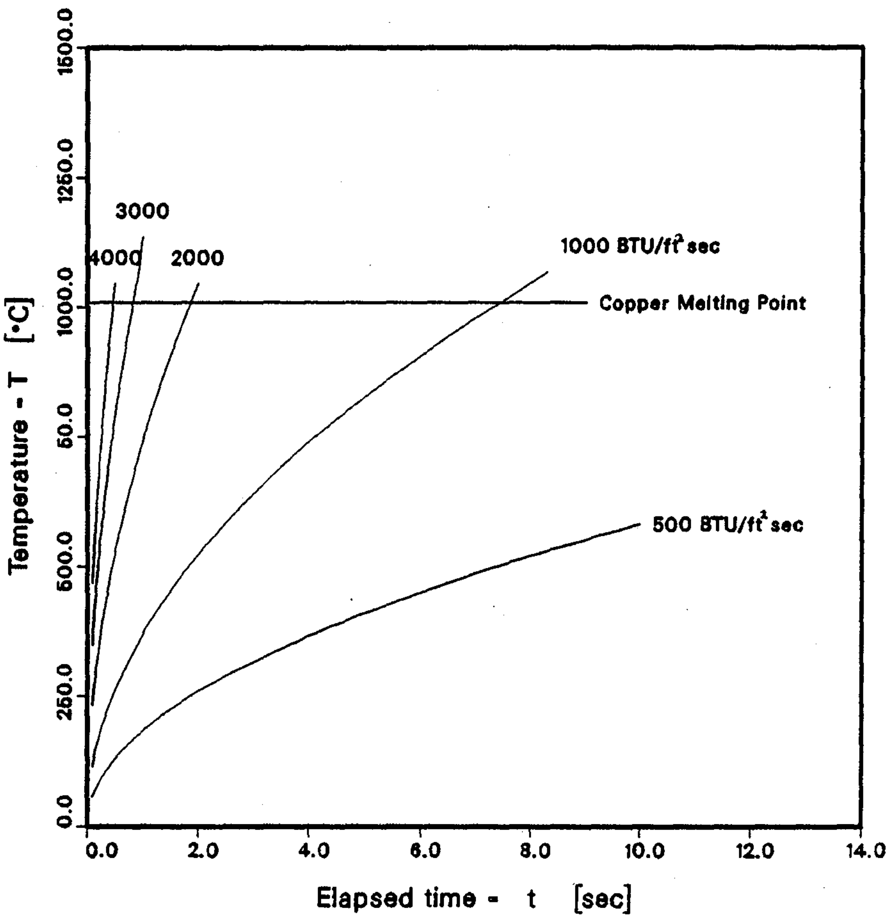

Figure 6: Probe Face Temperature vs. Time for Semi-Infinite Rod

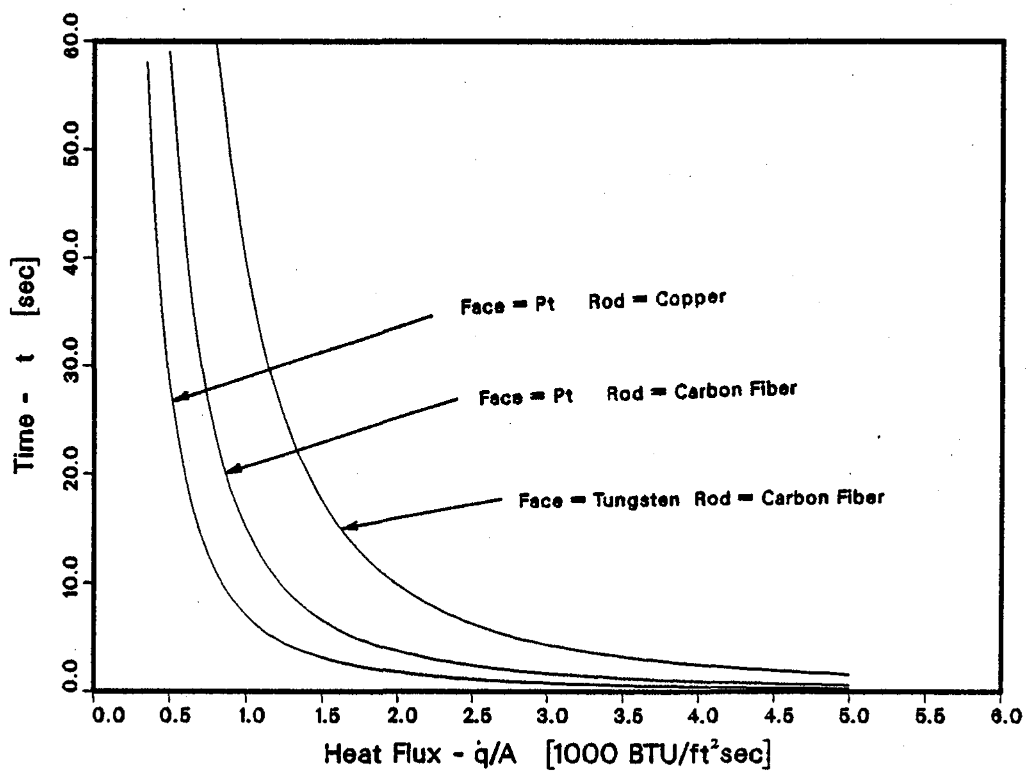

Figure 7: Time for Face to Reach Melting Temperature

1,000° C). This limit is shown in Figure 6. Figure 7 shows the survival time of a probe with a platinum RTD and a copper body as a function of constant input heat flux. This curve was obtained from the solution to the heat equation as well. In order to study the effects that the selection of materials for the substrate and face have on the survival times of this type of sensor a "survival time constant" can be defined as follows :

$$K = \left(\frac{T_m}{2k} \sqrt{\frac{\pi}{\alpha}} \right)^2 \qquad \text{Eqn.3}$$

where Tm is the melting temperature, k the thermal conductivity and α the thermal diffusivity of the substrate material. This constant has units of heat flux squared seconds. The survival time, t, of the probe becomes:

$$t = \left(\frac{A}{q} \right)^2 K \qquad \text{Eqn.4}$$

From this it is determined that the constant K has a strong influence on the survival time. Analyzing the constituents of K it is concluded that the thermal conductivity has a larger effect on the survival than the melting temperature.

Based on this the possibility of using a material other than copper for the rod was investigated. For example, there are recent developments of new carbon fibers that have better thermal conductivity than copper and also have a higher melting point. In conjunction with a tungsten RTD, these fibers could be used to build a probe with extremely high heat flux capabilities and survival times. A comparison of survival time versus input heat flux is shown in Figure 7. It should be noted that these survival times are based on the assumption that all thin films properly withstand thermal shock and the mechanical stresses that are imposed on them.

The effect of thin film parameters have on survival times will be discussed in more detail in the Section 5.0.

4.3 Data Reduction

The first step in the data reduction is the determination of surface temperature from the resistance output of the face RTD. Several standard calibrations exist for platinum RTD's. In this reduction a constant temperature coefficient of 0.00300 (the value for pure platinum) was used in conjunction with the linear temperature resistance relationship.

Most of the analyses described previously are based on the very simple solution of the heat equation for a semi-infinite solid. However, this equation cannot be used to reduce actual data because it is based on constant heat flux into the face. This assumption obviously cannot be made for a probe, since heat flux will never be constant. It is, therefore, necessary to use a numerical scheme to compute actual heat flux input from the measured temperature history.

A large variety of algorithms is in existence for the heat equation. Most are based on the need to compute temperature distributions from given temperature time boundary conditions. The algorithm required for this purpose must, however, compute heat flux from a temperature time and a heat flux boundary condition.

A very simple implementation was chosen to reduce computational expense. The user of these types of probes, based on this project, can use the algorithm given here, or any other derivative one.

A simple finite difference scheme is used. In order to minimize computational complexity time and length steps are chosen such that the temperature at each node is given by the average of the temperatures at both adjacent nodes for the previous timestep. The boundary condition are given at the face by the measured temperature histories and at the cool end by the zero heat flux condition there. The latter specifies that the temperature gradient at the cool end is zero. Appendix A is the source code used for these reductions and based on dividing the 2.5" long probe rods into 140 distance steps. The time steps are 1/10th of a second. Since the data are available only every second the temperatures for the other 9 timesteps inbetween are obtained by linear interpolation.

The scheme used herein assumes that the temperature is given at the face of the probe and that the thermal conductivity is constant. This is adequate for demonstrating the viability of the technique, but cannot be done in a case where actual data must be obtained. Here the computational scheme must be more complex, in that it must account for the variations in thermal conductivity that occur due to temperature changes.

As mentioned earlier, some variations of the data reduction scheme can minimize response times or maximize accuracy. In general, the more realistic the model on which the computations are based, the better the results in terms of accuracy.

The most accurate computations would include the effects of variable thermal conductivity, thermal capacity of the various thin films, and the fact that the temperature sensor is located a small distance away from the face of the probe.

5.0 FABRICATION

While the concept of the cool film calorimeter is very simple, (Figure 2) its implementation in a working sensor is a formidable problem. Several design requirements exist:

(1) The rod must have high thermal conductivity.

(2) The sensor at the face of the probe must withstand high temperatures and thermal and mechanical stresses.

(3) Low resistance leads to the sensor face must be provided in an appropriate manner.

(4) The sensor at the face must be isolated electrically from the rod.

To satisfy the requirement that the rod have high thermal conductivity, copper was chosen. Copper is readily available as wire and can be shaped easily.

A variety of probes for temperature, heat flux, and pressure or strain have been manufactured using thin film technology. Virtually all of them use a non-electrically conducting substrate to carry the resistive sensing element. With this construction, the sensing element can be applied directly to the substrate. To maximize the heat conducted away from the probe face and, thereby, maximize useful data times and survivability of the cool film calorimeter, it is necessary to use a conductive substrate. Therefore, another thin film must be used between the sensing element and the substrate.

The thin films making up the face of the probe must have good adhesion properties. In general, thin films do not behave the same as the bulk material from which they are composed, so the selection of materials is not a trivial task. Also, there are great variations in thin film properties, depending on the manner in which the film was deposited. This applies mainly to the adhesion properties.

The first thin film applied to the copper rod must electrically isolate the sensor from the rod, and still have good thermal conductivity. For these characteristics, boron nitride is the best material. It has a high thermal conductivity and is still a good electrical insulator. Therefore boron nitride was chosen initially for the first face material.

Platinum is the material chosen most frequently for RTD, because of its good resistance versus temperature characteristics and its imperviousness to environmental influences. Both platinum and gold RTDs were used and neither metal showed a clear advantage. However, due to the fact that film resistance is a critical issue in miniaturizing the film and because gold has a lower resistance than platinum, it was decided to use platinum only in the later designs.

All probes were manufactured with a solid face resistor. This means that a large portion of the face was covered uniformly with the thin film resistor. This resistance is quite low and falls rapidly with decreasing face area which generates a severe problem of accuracy with miniaturized probes. The resistances of the face RTD are of the order of 10 ohms at room temperature. Since the leads are exposed to high temperatures as well as the face of the probe their resistance will change also. If the room temperature resistance of the leads is of the same order of magnitude as that of the face sensing resistor, it becomes impossible to tell exactly what was the resistance change of the face sensor due to temperature. This fact generates a requirement for lead resistance to be about two orders of magnitude lower then face resistances and therefore fairly massive leads are necessary.

The obvious solution is to increase the face resistance by micromachining or laser etching in a "zig-zag" pattern. However, the desired simplicity of probe construction would be lost and implementation costs would be excessive.

If the probe face resistor is inserted in a standard resistor bridge circuit as shown in Figure 8, the output of the bridge is given by the following equation:

$$\Delta V_p = \left(\frac{\Delta R_p}{R_p}\right) V_p \qquad \text{Eqn.5}$$

For a given relative resistance change, the output voltage is directly proportional to the excitation voltage of the probe. A high input voltage is, therefore, desirable in order to maximize the output. Since the substrate is electrically conductive and the RTD is separated from this substrate only by a non-conductive thin film, the insulator breakdown voltage severely limits excitation voltage that may be applied to the probe. The maximum voltage allowable is twice the breakdown voltage of the thin film. Above this value, the thin film insulator looses its insulating capabilities at the attachment point of the leads.

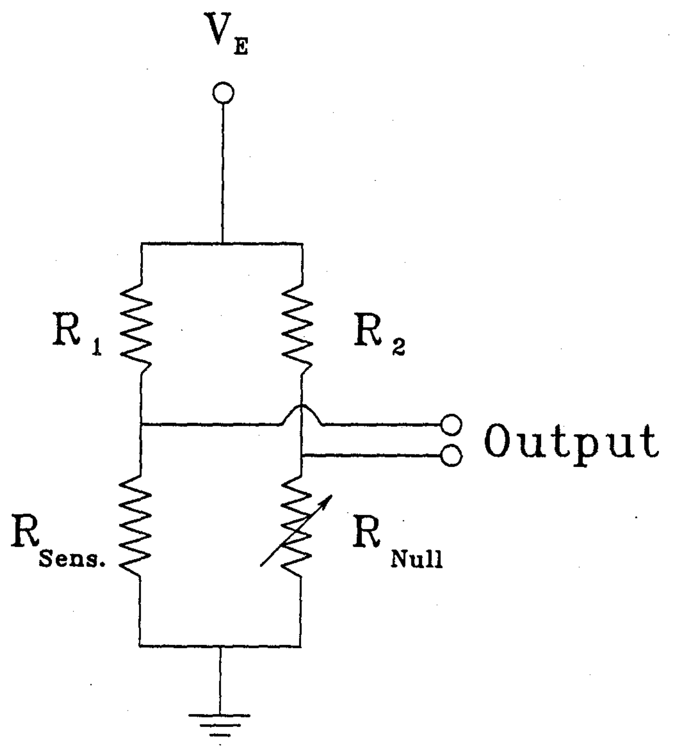

Figure 8: Typical Resistance Bridge Diagram

Another factor that limits the applied voltage for low resistance probes is the heat generated by the current through the probe. This heat can raise the temperature of the probe sufficiently to alter temperature readings and degrade accuracy.

During manufacture of the various probes it became obvious that it is quite difficult to achieve complete coverage of the entire substrate rod face for the chosen material. Several problems, inherent to thin films (for example "pinholing"), allowed minute connections of the RTD to the substrate. The insulating thin films must therefore be chosen to be quite "thick". Figure 5 allows that these "thicker" films can result in relatively high temperature drops across the insulating coatings, especially if materials different from boron nitride have been chosen. Thus, it becomes necessary to include the insulating thin film in any model used for actual data reduction.

It is not necessary to worry about thermoelectric effects as long as the connection points for the leads are at equal temperatures due to the law of intermediate metals. In all of Lockheed's designs, this was the case since they were all axi-symmetric configurations.

The RTD was protected from environmental influences by a hard insulating layer applied on top of the conductive material. Silicon carbide was chosen for convenience. This layer prevents electrically charged particles in the flow from influencing readings of the RTD and also helps in preventing small particles impinging on the face from eroding the RTD. Again, a tradeoff is necessary between accuracy and sensor robustness, since the thicker protective coatings cause fairly large temperature drops. Silicon carbide is a very hard material and appeared suitable for this purpose.

5.1 The First Design

The initial design was conceived (Figure 9) based on the points and requirements described above. In this design the copper rod itself is used as leads. This provides for very low resistance leads. A copper wire is used as the central rod. Onto this, a layer of boron carbide is deposited using thin film sputtering techniques. This thin film would provide electrical insulation to the outer copper layer which will be copper plated again. Thus the inner rod provides one lead to the face of the probe, while the outer annular layer provides the other, the boron nitride thin film separating the two electrically. A thin film of boron nitride is then applied to the face of the probe. This layer is punctured in the center of the face and around the periphery. Following this the platinum thin film resistor is applied by sputtering and is electrically connected to the two leads through the punctures in the boron nitride film. The entire assembly is then covered with silicon carbide, to environmentally protect the device.

This design fulfills all the performance requirements, however, in practice, the plating of a thick outer annulus could not be accomplished successfully. Plating processes apply mostly to a few mils of the material. A much thicker coat was required for which the plating process proved unsuitable. The thickness of one of the leads was thus insufficient to provide for the small electrical resistance required for optimum performance.

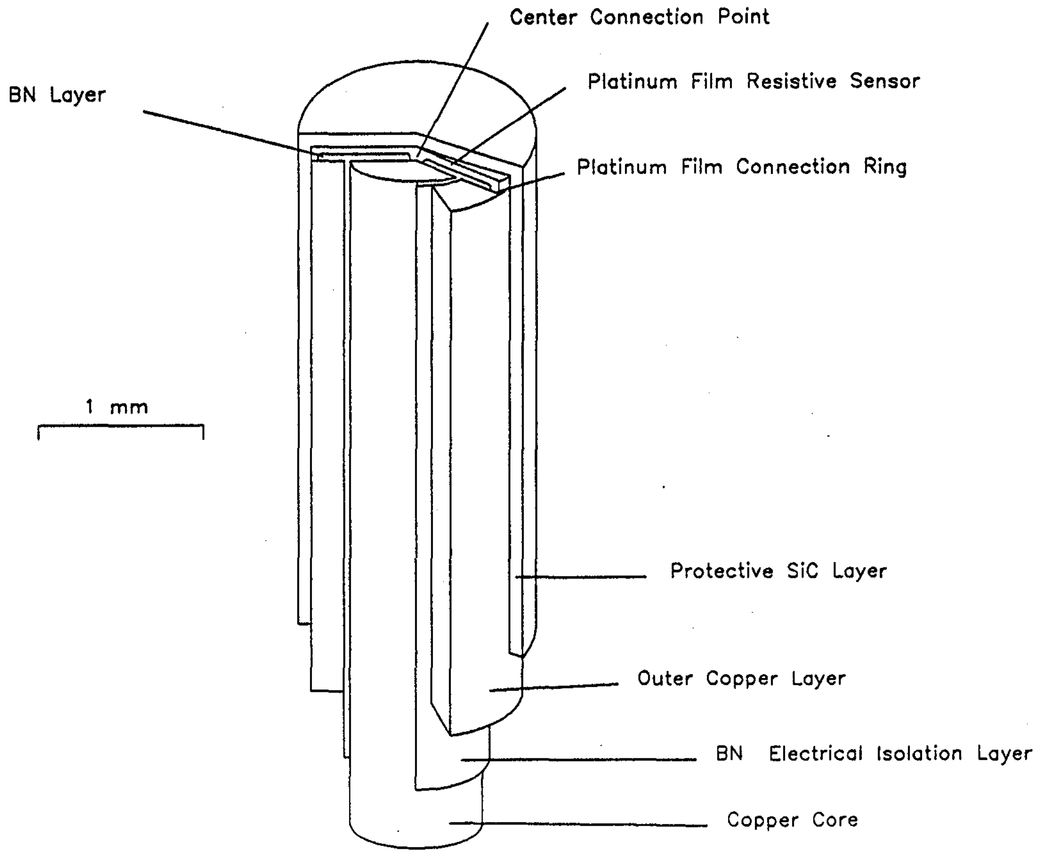

Figure 9: The First Design

5.2 The Second Design

A second design was developed with the goal to circumvent the plating process problems. This design is shown in Figure 10. The annular construction of the leads is now eliminated, and the rod is split in two, instead with each half serving as one lead. One of the experiences gained with the first design was that the boron nitride thin films are extremely sensitive mechanically. That is, adhesion was poor, the coating soft and extremely fragile. This fact ruled out the use of boron nitride thin films to separate the two halves of the rod. It was decided to use a ceramic adhesive coating instead.

After the two leads separated by the ceramic coating were assembled inside a ceramic tube (for support), a thin film of boron nitride was applied to the face of the probe. This film was punctured in two places at the far end of the lead face to provide a connection for the platinum thin film that was next applied.

During low temperature testing, these probes operated well. Their cold resistance was satisfactory, and their response to a jet of hot air good. However, upon applying high temperatures (from a blowtorch), the resistance of the probe started to show sudden changes. Generally, the resistance increased, but not in the expected continuous fashion. Figure 11 is a photograph of the face of the probe after the high temperatures have been applied. The ceramic adhesive insulator is protruding between the two copper leads and has mechanically destroyed the platinum film. The lesson learned from this design is that thin films cannot bridge non thin film insulators. The different thermal expansion coefficients of the leads, and the ceramic material in between, causes the ceramic material to expand out of the gap and to generate a mechanical and electrical discontinuity in the RTD.

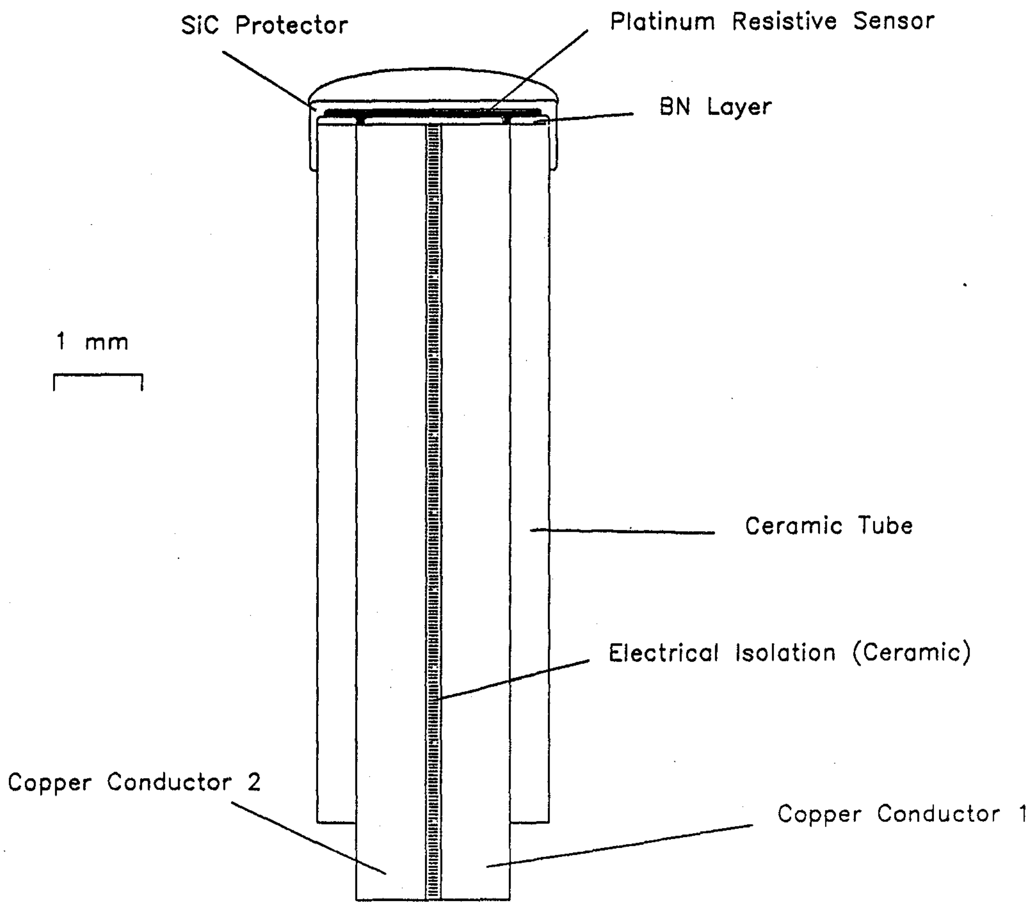

Figure 10: The Second Design

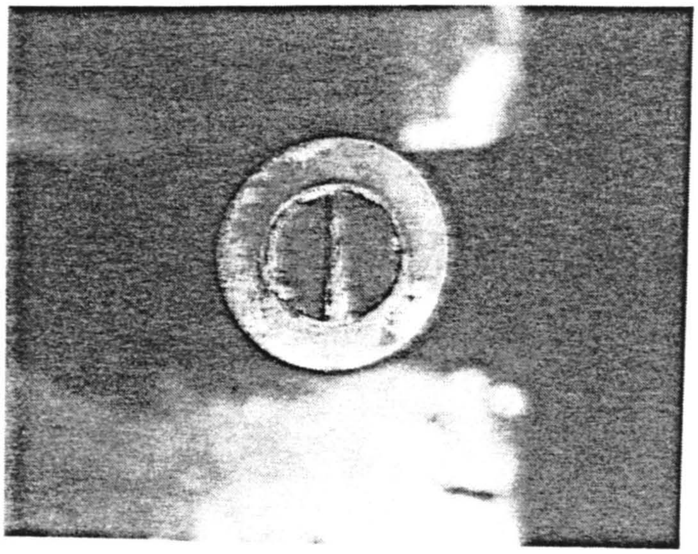

Figure 11: Photograph of the Second Design after Heating

5.3 The Third Design

The analysis of the problems encountered with the two designs described above, resulted in the realization that it is not possible to use the rod itself as lead wire(s) of any kind. If the separator is a thin film, one of the leads must be applied by plating, which is unable to deposit a sufficiently thick layer of material. In addition, if the separator is a layer of ceramic, the different thermal expansion coefficients ultimately cause the destruction of the RTD during the first heating cycle.

This insight resulted in a return to a design that uses separate leads. Figure 12 shows a drawing of the third design made by Lockheed. In this design the central rod does not serve as a lead. It is simply covered with an insulating thin film. A strip of platinum is then sputtered onto the face and down the sides with two specially shaped leads connected to the sides of the platinum thin film, to provide a low resistance path. The entire assembly is housed in a ceramic tube and the face is coated with a thin film of silicon carbide.

For the prototype of this gauge, a 2.5" piece of 14 gauge wire was shaped properly at the ends to provide for a flat smooth face. The entire piece of wire was then coated with a 500 Å thin film of boron nitride. After this coat, a mask of thinned rubber cement was applied to the sides of the rod such that the platinum (which was sputtered on next) would cover a strip of approximately 1 mm width across the face of the rod, and extend about 5 mm down both sides of the rod. After this operation, a thin coat of ceramic adhesive was applied around the rod from the hot end to the point where the platinum thin film stops. The purpose of this coat was to separate the leads from the copper rod, since the thin film of Boron

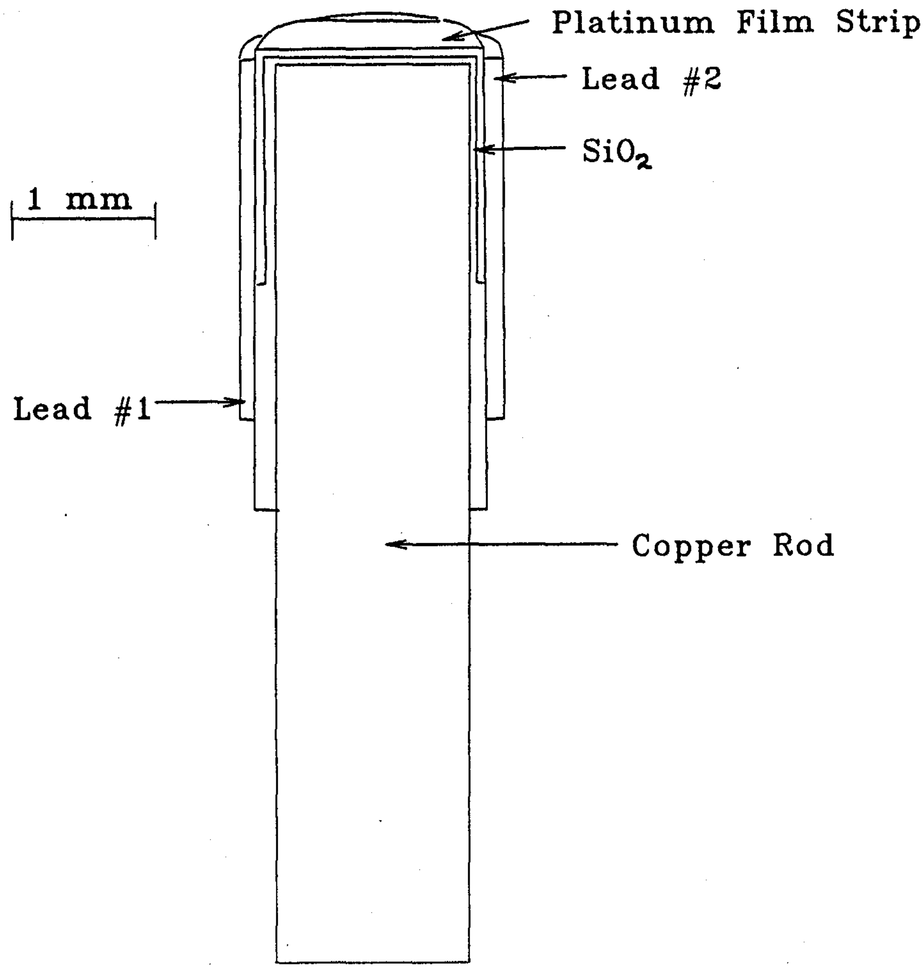

Figure 12: The Third Design

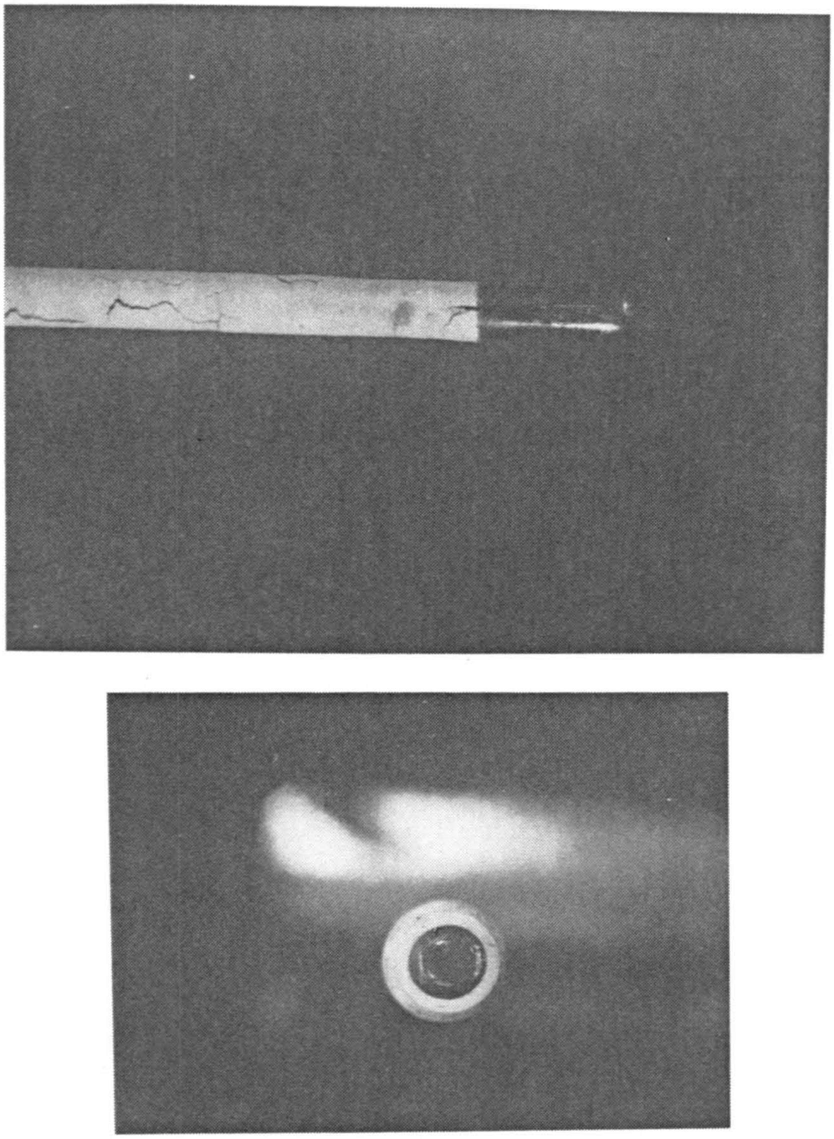

Figure 13: First Stages of Probe Fabrication

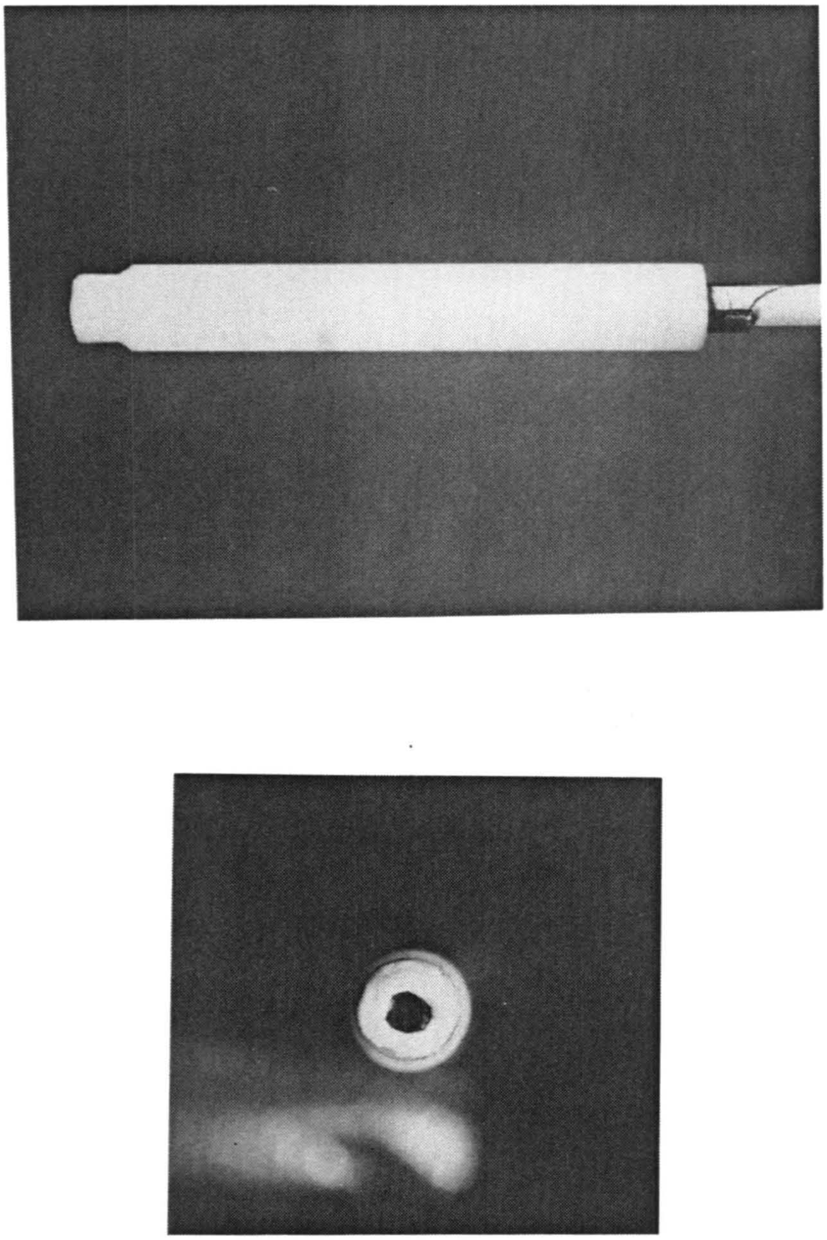

Figure 14: Finished Probe

Nitride is unable to do that properly. The rod is shown at this stage in Figure 13.

The two leads (which are made by cutting small copper tubing in half) were then applied to the sides. In order to assist in making proper contact a small amount of silver epoxy was applied to the side portions of the platinum film. The leads were held on the rod by a ceramic tube with a 3/16" inner diameter. A front view of the probe face in this state can be seen in Figure 13. When the probe was heated up the copper expanded against the ceramic tube causing an increasing contact pressure at the interface between the leads and the platinum film. The hot gases were prevented from streaming by the leads and damaging the rear portions of the probe by a ceramic filler applied around the face of the probe. After this step, the probe was ready for calibration and use.

The interface contact pressure, while necessary to maintain good electrical conductivity caused the leads to damage the thin films and thus the survivability was iteratively improved by increasing progressively the thickness of the boron nitride film was to 2000 Å. The damage to the boron nitride thin film due to the leads remained, however, up to a film thickness of 2000 Å. It was not possible to use a thicker film because of adhesion problems, so it was decided to search for a different material. After some experimentation, silicon dioxide, which has excellent adhesion characteristics and is relatively hard, was chosen. The insulating film was therefore made with a thickness of of 1500 Å of silicon dioxide.

5.4 Lessons Learned

It should be noted here that the application of dielectric thin films to the face of a small rod has proven to be an extremely difficult problem. Most thin film laboratories are accustomed to apply films to relatively large flat surfaces. Several techniques exist to analyze thin films applied to these flat surfaces but these techniques cannot be used in this case, so that we have to rely entirely on the technicians "feel" for the processes involved. This involves a considerable amount of experimentation to evoke a "cook book" of procedures! During the course of our experimentation it became obvious that the number of parameters involved in setting up a run is so large that finding the ideal combination of material and thin film parameters constituted a monumental resource consuming task not available for this project.

These problems of repeatability and measureability of the mechanical characteristics of ceramic thin films made a careful study of the effect of the various thin film parameters on their characteristics impractical.

Metallic thin films on the other hand pose much less of a challenge. These can be readily examined under an electron microscope and their adhesion was found to be excellent on any of the substrates that were used. The numerous successes that have been achieved with thin film RTDs, have all been achieved by applying the metallic film to a dielectric substrate, rather than a conducting one (that is only coated with a dielectric thin film).

The limitations for miniaturization were mentioned briefly earlier. Sufficient accuracy with standard electronics requires a face resistance value of about 10 ohms. To achieve this resistance the face length of the platinum thin film must be of the order of 1 mm with a film thickness of 200 Å. It is not advisable to use film thicknesses below 200 Å because of problems with ensuring film coverage and repeatability. This design cannot be miniaturized any further without compromising accuracy, integrity and survivability.

A series of 16 probes was built, after all the design parameters had been optimized and the prototypes tested showed that this design was close to optimum. Problems do exist with the thin film coatings, since only 50% of the first batch showed good repeatability. Of the 8 inadequate probes 7 were short circuited, because of damaged or improperly applied dielectric thin films and one showed a resistance of 15 K ohms (and noisy temperature response) due to a bad connection between one of the leads and the RTD.

Of the remaining probes, two were used for electrical testing. The dielectric strength of thin films is quite low, and it was predicted that the maximum applied voltage of 0.56 volts was the limit. Thus, two tests were conducted. The applied voltage was slowly raised until the device broke down causing some current to flow from the thin film to the center rod. For both probes breakdown occurred at 0.52 volts. This indicates that while some imperfections are present in the dielectric thin film these are not severe.

Six probes remained for two further sets of tests. Three probes were allocated to the test at the Accurex arcjet facility and three were allocated to tests at the NASA Marshall arcjet facility.

6. TESTS AND CALIBRATION

The basic method chosen for calibration of these probes was to expose them to heat flux, is parallel with known heat flux gauges and then compare the responses. The heat flux derived from these gauges is the result of a numerical solution of the heat equation such that there are no "calibration factors" anywhere in the data reduction. Thus this phase is not a calibration phase, but a validation phase, where the output from the gauges is compared to the result obtained from a known calibrated gauge. If the data deviates significantly from the expected results, some of the assumptions that were used to arrive at the data reduction scheme (like 1-D heat conduction) will have to be modified. In effect, this would mean that the modeling used does not correspond to the phenomena occuring in the probe, and that the modeling has to be chosen differently.

6.1 Arcjet Tests

The initial set of tests and calibration runs were conducted in the Accurex arcjet facility. Figure 15 is a sketch of the test setup and fixture. The flow direction is parallel to the surface in which the probes are embedded. The test fixture that contains the probes is a watercooled block of copper. A set of three off-the-shelf Gardon gauges (Type # 8-250-120-20903, 250 BTU/ft^{2}sec range) was used, in parallel to our probes, in order to supply reference data.

The calibration of probes of this nature can be accomplished in several ways. The probes were exposed and the data reduced according to the theoretical model described earlier. Thus, it was possible to compare the results with data from other tested and calibrated probes (the Gardon gauges). The output of Lockheed probes matches that of the Gardon gauges if both probes are physically working and our modeling is correct. Since the data reduction in this case does not involve any simple input/output relationship, it would be difficult to correct any discrepancies just by changing 'calibration constants'. Thus the classical approach to defining a set of constants for each probe cannot be applied to these probes, because there is no place for constants in the data reduction. If discrepancies between the output from the probes, and the expected values occur, this can only be due to the inadequacy of the model used in the data reduction. Such inadequacies could stem from: (1) significant two dimensionality of heat conduction, (2) incorrect or inaccurate thermal conductivity data, and (3) improper or inadequate treatment of the effect of the thin film coatings on the face.

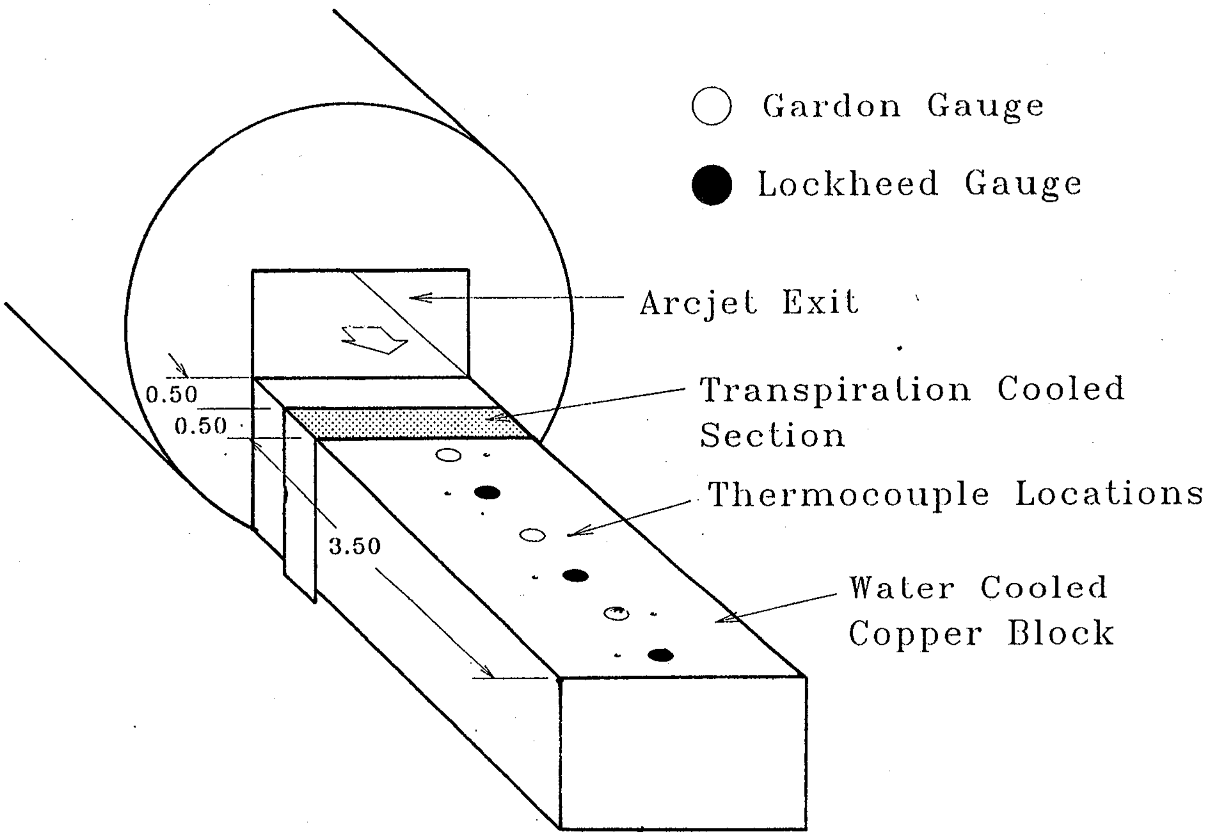

Figure 15: Sketch of Test Setup at Accurex

Figure 16 is a photograph of the copper block installed in the arcjet facility. Since Gardon gauges are in direct thermal contact with the fixture, they accurately measure the heat flux into the watercooled model. Our probes, however, are thermally insulated from the water cooled block, and therefore measure the heat flux into a semi-infinite solid. The two results are identical for the first instant of the experiment and diverge thereafter. The heat flux into our probes diminishes due to the heating up of the face of the probe. Video thermography images obtained from these tests show that the probe face quickly reaches temperatures of 1000^{o} F, while the rest of the block maintains surface temperatures of 350^{o}. In our case the influence of the hot spot due to the probes was not important, but it may be necessary to study the effect of the probes on the flow in some special flow situations.

The Accurex arcjet tests consisted of a series of runs using different gases injected into the flow from the transpiration cooled section. These gases caused variations in heat flux and also showed the effect that corrosive gases have on the probe face. This provided an assessment of survivability. Five runs were made. Three of these used nitrogen as an injectant, one used hydrogen and one methane.

Of the three Gardon gauges, one failed before the first run, while the two remaining ones worked during all further runs. Lockheed's gauges also had one failure immediately during the first run. This gauge simply did not respond at all to any temperature. Since the gauge did not survive all the runs, it was not possible to determine the failure mode.

Figure 17 shows the temperature evolution of the face of the two surviving working probes.

The heat flux levels obtained from the Gardon gauges are plotted in Figure 18 together with the computed heat flux levels from Lockheed's

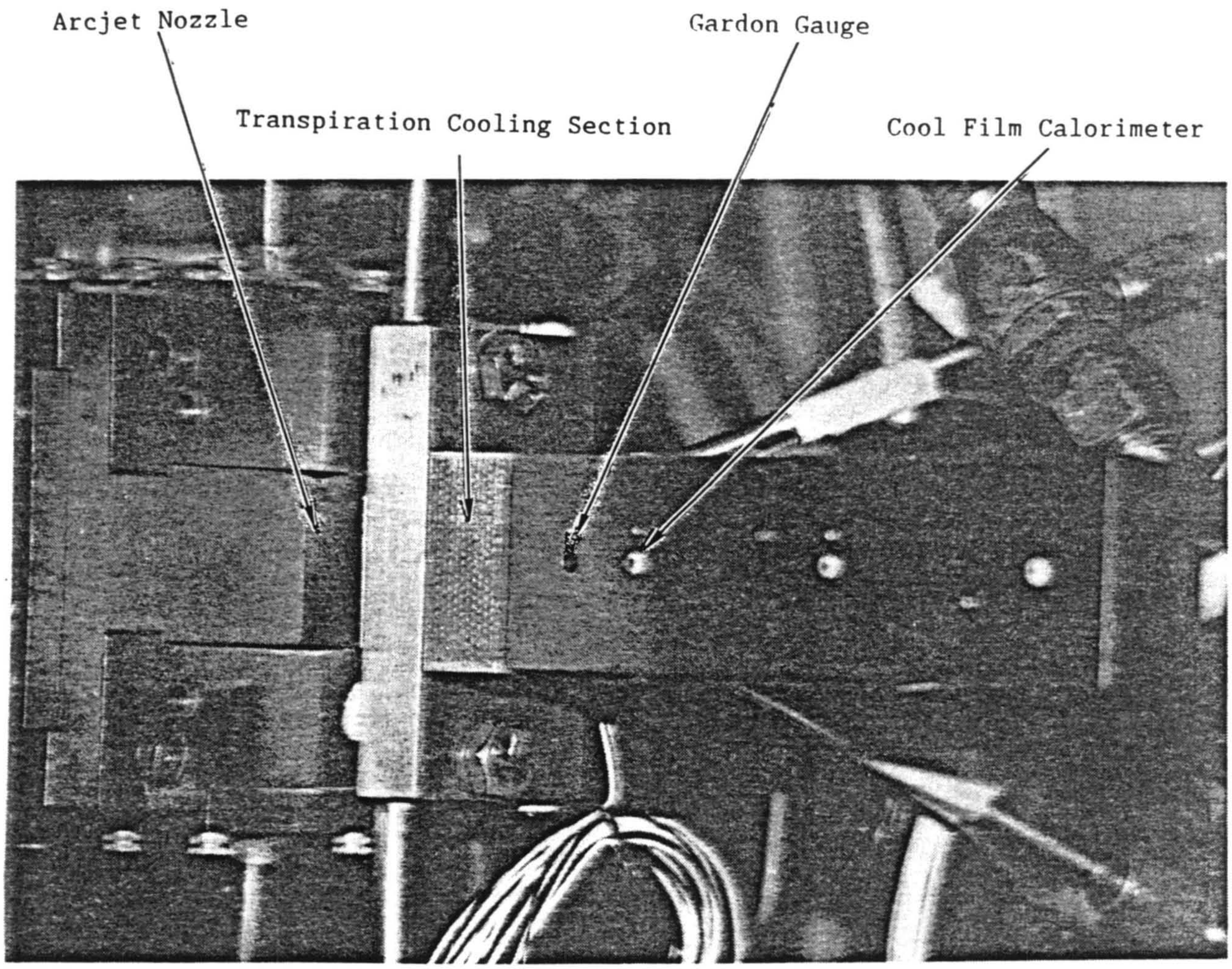

Figure 16: Photograph of Test Setup at Accurex

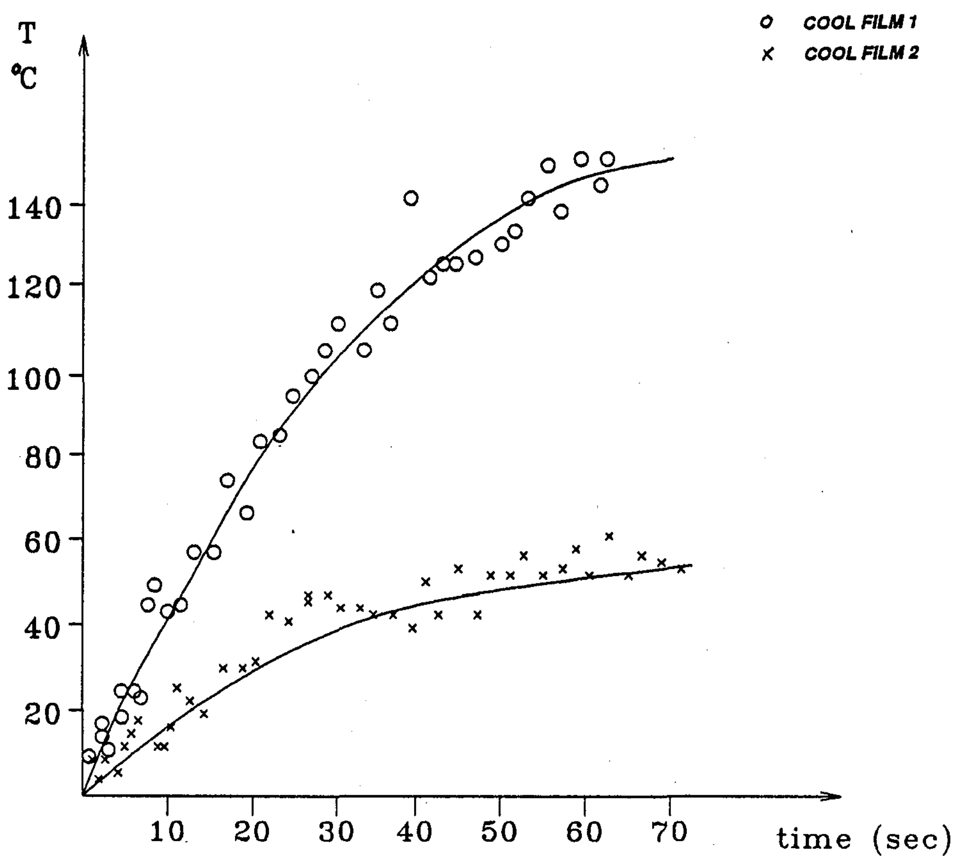

Figure 17: Temperature vs. Time for Run N 1

probes. The initial heat flux reading from Lockheed's gauge matches the Gardon gauge. After that the heat flux levels are significantly lower than expected, and drop off to zero quickly. This is the result of the face temperature rising to the temperature of the gases over the gauge. As soon as the face temperature reaches the temperature of the gases, the heat flux into the rod goes to zero, and the face temperature becomes constant. The data reduction scheme correctly indicated zero heat flux into the probe. We can conclude that, initially, the heat flux is obtained correctly but then a fundamental difference between the Gardon gauge (linked thermally to the watercooled body) and Lockheed's cool film calorimeter (isolated thermally from the cooled body) arises. Both gauges supply the correct heat flux, but it is a different value. Nevertheless, the data during the initial period (when the cool film calorimeter face temperature is close to that of the body) allows a good comparison.

The noise in the temperature data requires that the data be smoothed, in order to prevent oscillations in the heat flux calculations. This implied some uncertainty in the resulting heat flux results, since the smoothing strongly influences the results. The noise in the raw data was mainly due to the extremely low output voltages of the probe itself. These, in turn, were due to the relatively low resistance of the RTD and the severe excitation voltage limitations. One major problem with this technology that was encountered at this stage was the fact that after the probes had cooled back to their original temperature, the resistance did not return to its original value. This change in cold resistance continued to occur in all the runs and, as will be seen later, rendered the probes unusable after only three runs. This by itself does not constitute a major problem however, since it must be assumed that, during the entire run, the resistance of the probe is changing, not only due to temperature, but also

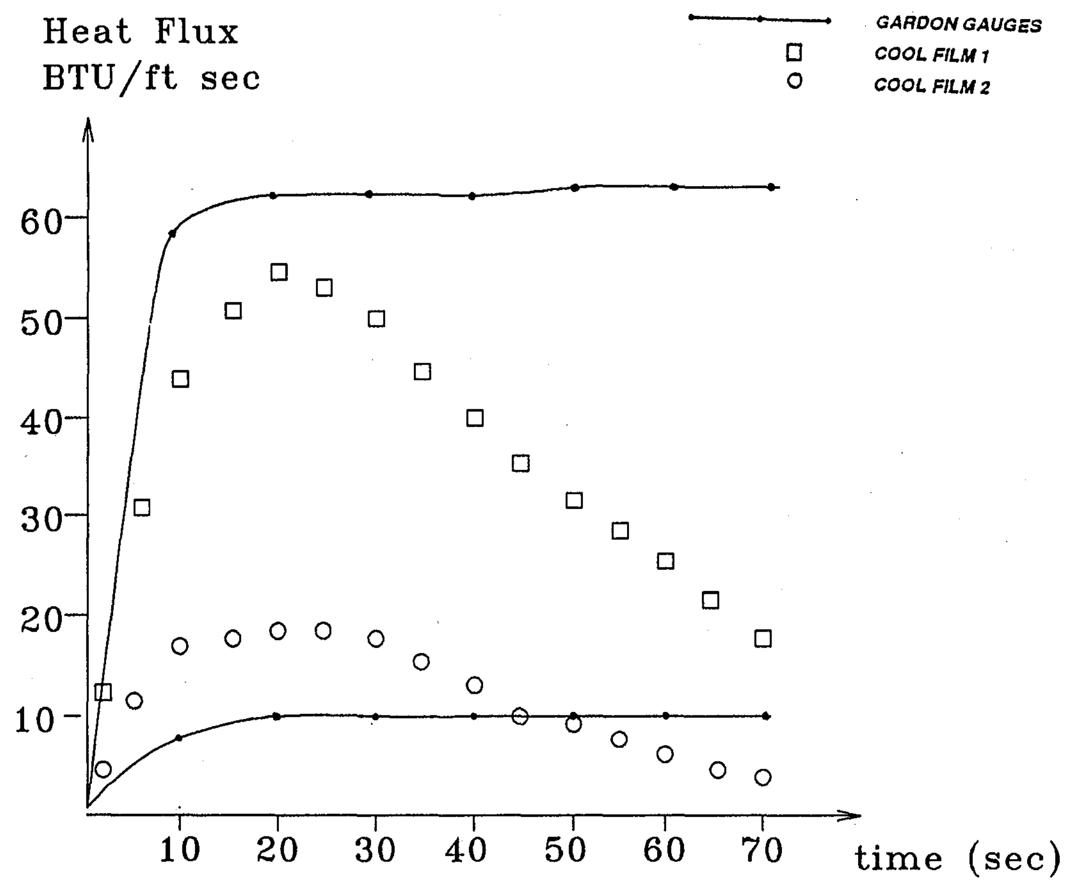

Figure 18: Heat Flux vs. Time for Run N1

due to whatever it is that causes the cold resistance change. It is, therefore, impossible to find the actual face temperature accurately. It was not possible to find the cause for this phenomenon with any degree of certainty. An initial guess was, that particles in the flow would impact on the surface and thereby cause small "shorts" into the copper. Close examination of the probe faces after heating them up, however, showed no traces of impact or damage. The only remaining explanation is that changes must take place in the dielectric thin film (or the platinum thin film) due to diffusion of one or both materials that cause the resistance to change. Since similar changes have not been observed in other RTD devices, it must be, that the cause was in the dielectric film deposition process.

Figure 19 shows the temperature evolution for the second run with a nitrogen injectant. Probe face temperatures at the end of run were over 300^{o} centigrade (600 F). The remainder of the water cooled block, on the other hand, (including the Gardon gauges) never exceeded 160^{o} centigrade. The result of this severe temperature difference can be seen in Figure 20, which shows the heat flux levels for both the Gardon gauges and Lockheed probes. Again, the heat flux levels drop off, soon after the beginning of the run.

Figure 21 shows the temperature/time relationships for the third nitrogen run. It can be seen that the noise has become significant since the decrease in probe face resistance has occurred with each run.

Figure 22 shows that the correlation between the data from Lockheed's probes and the Gardon gauges has become worse. The noise in the data is simply too high so that not enough information remains to arrive at any representative smoothed curve. This trend continued during the two next

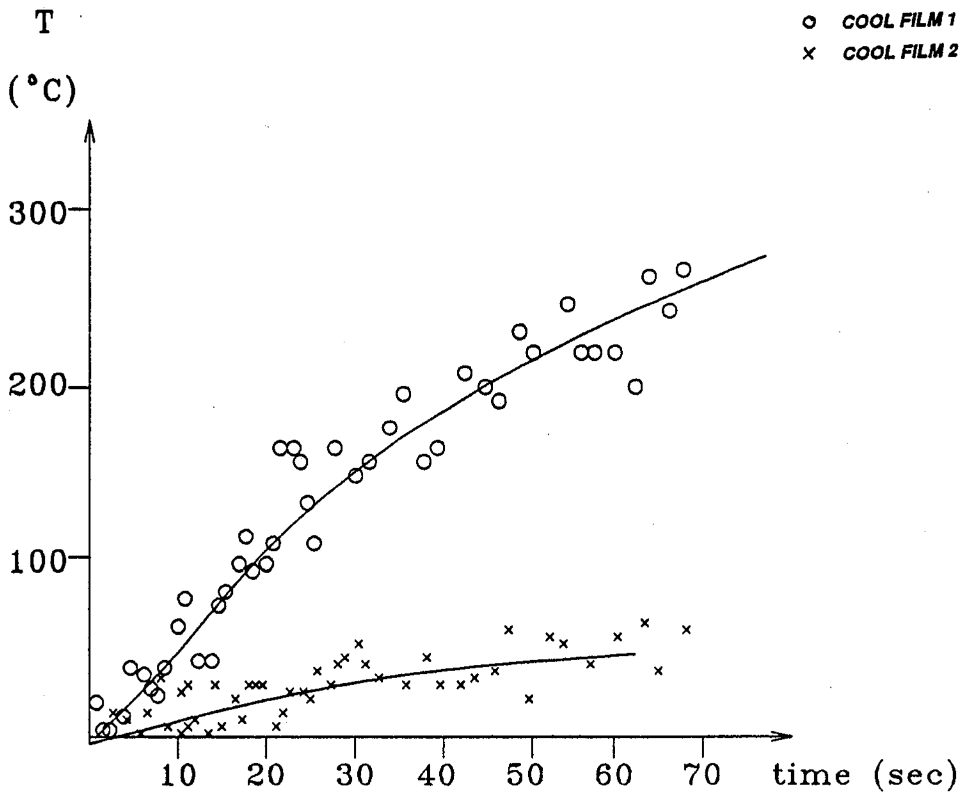

Figure 19: Temperature vs. Time for Run N2

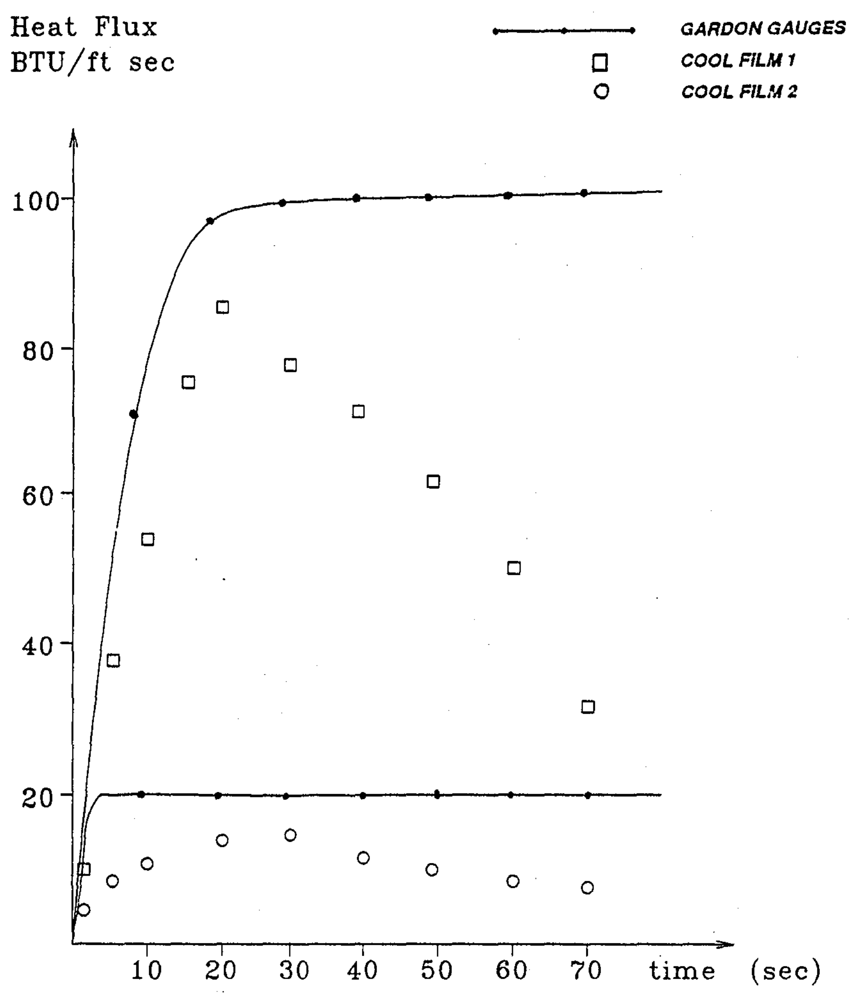

Figure 20: Heat Flux vs. Time for Run N2

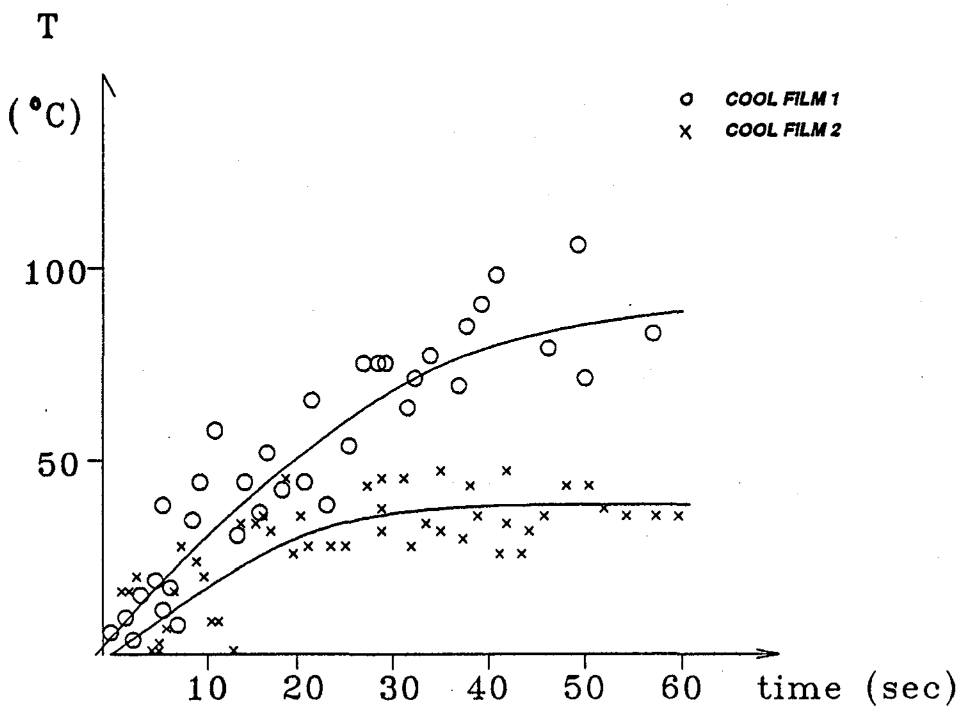

Figure 21: Temperature vs. Time for Run N3

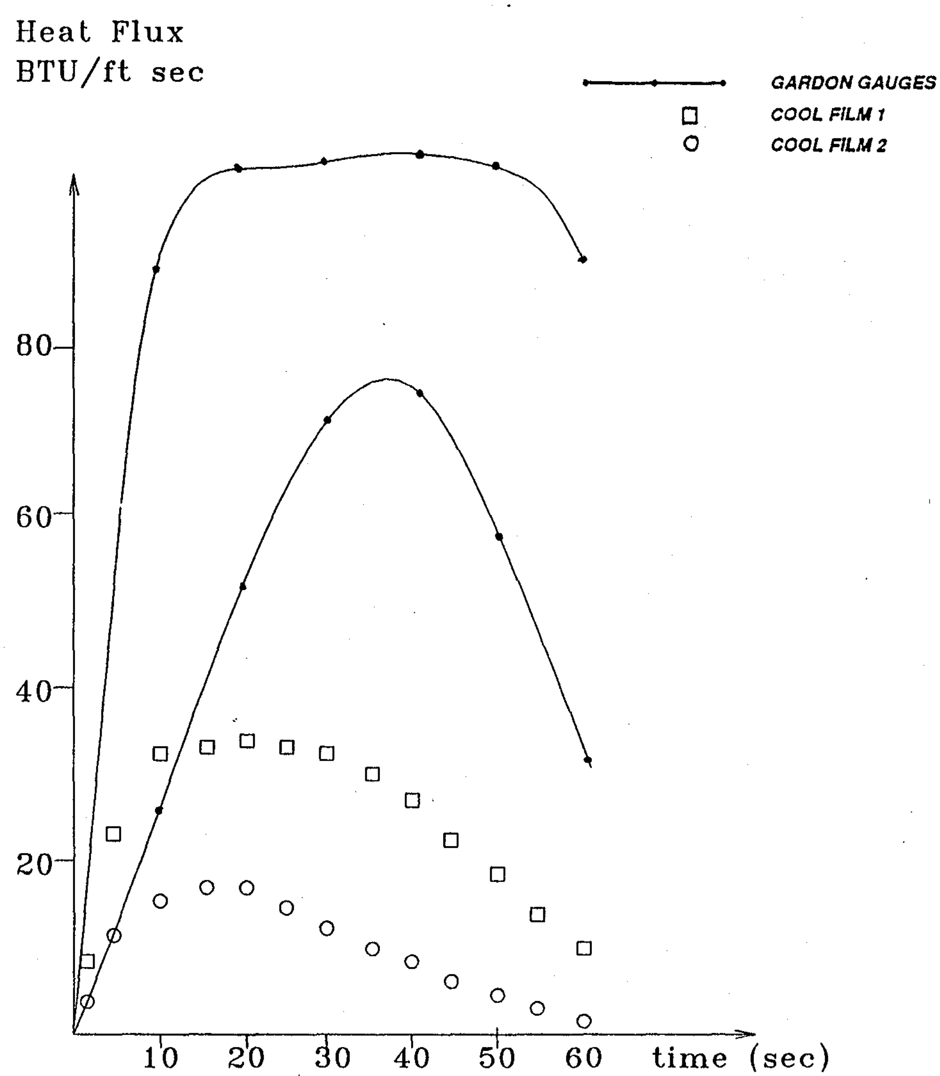

Figure 22: Heat Flux vs. Time for Run N3

runs (Figures 23 through 26) and in the final hydrogen run it was impossible to even attempt a heat flux calculation on the data.

A second evaluation test series was conducted at the NASA Marshall arcjet facility. In these tests the probes were exposed to much higher heat flux levels. Here the probes were installed in small carbon phenolic wedges within the stagnation region. The expected heat flux levels were of the order of 2000 BTU/ft^2 sec. The sampling frequency was increased threefold over the Accurex runs, however, the first datapoint obtained was already too late. All the heat flux gauges, including the Gardon gauges, were immediately destroyed. In fact, past test observation of the probes revealed that complete melting had occurred (including the ceramic tube) because the operator had left the arcjet running for almost one minute, thus far exceeding the planned survival time.

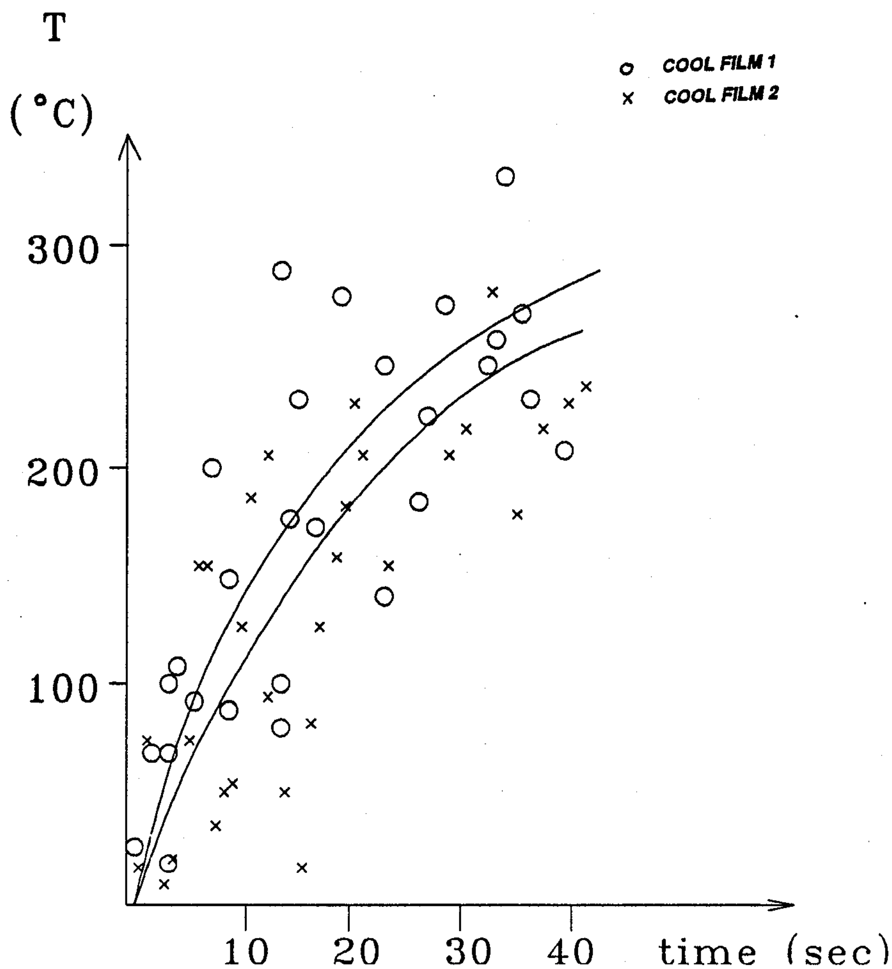

Figure 23: Temperature vs. Time for Run Methane1

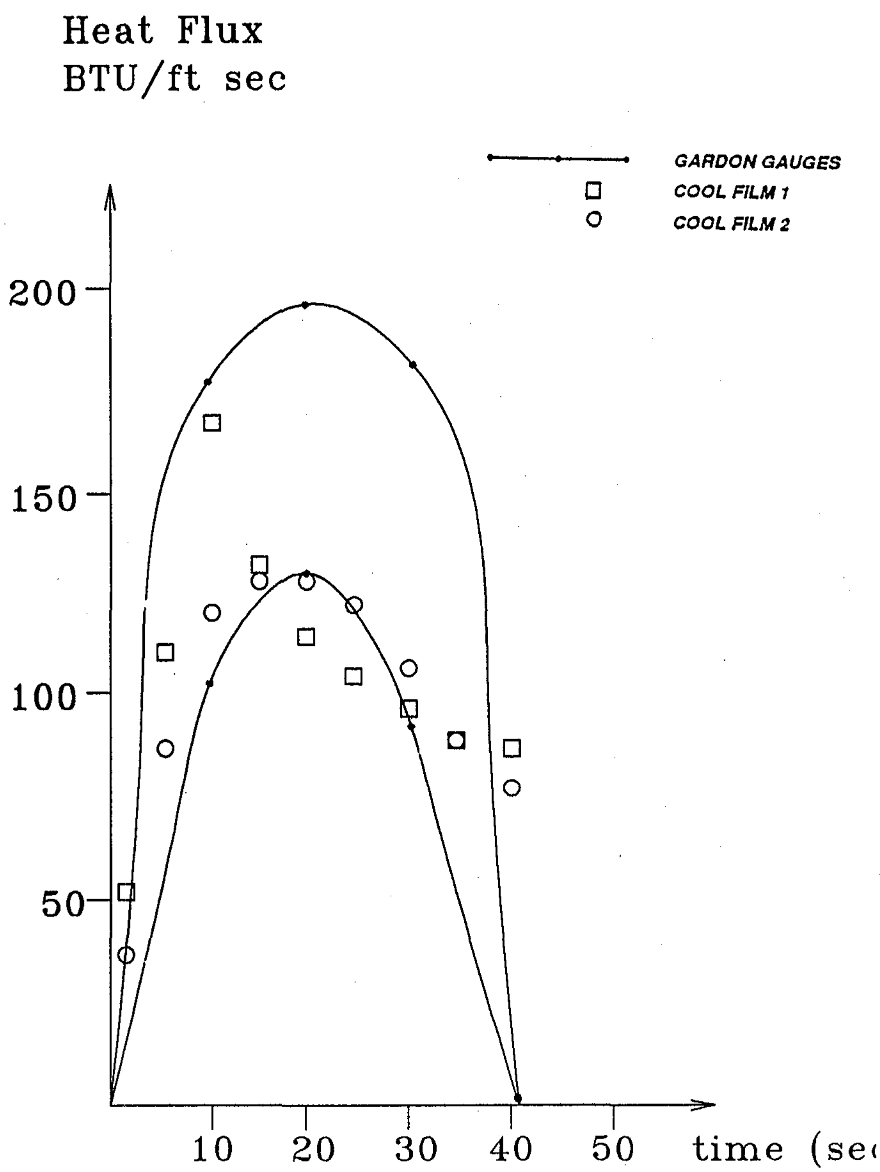

Figure 24: Heat Flux vs. Time for Run Methane1

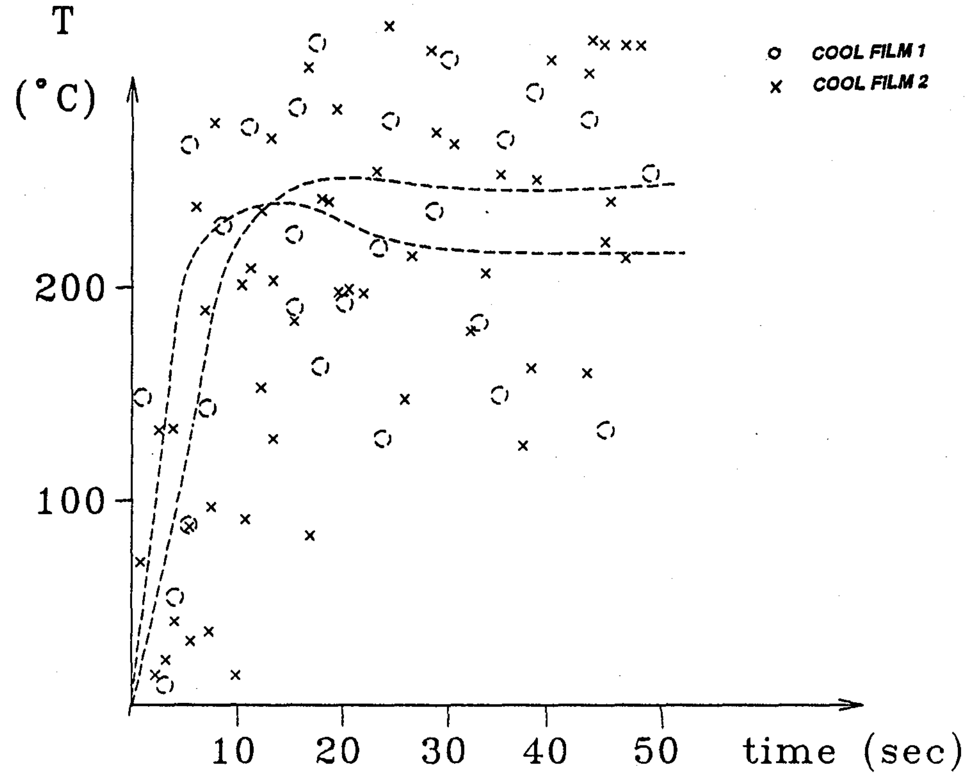

Figure 25: Temperature vs. Time for Run H1

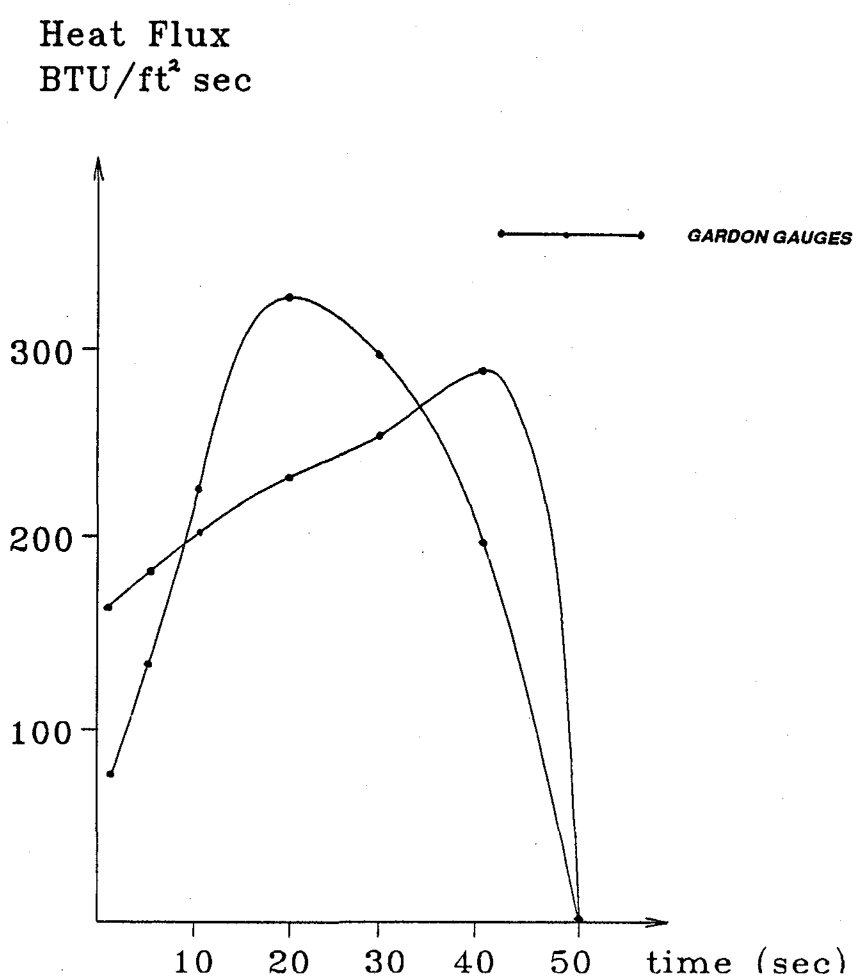

Figure 26: Heat Flux vs. Time for Run h1

6.2 Lessons Learned :

Our work with the third design resulted in some important insights.

(1) The dielectric film separating the RTD from the thermally conductive substrate caused several problems. First, the material of choice for this function (Boron Nitride) could not be successfully applied to the substrate.

Second, even with a different material (silicon dioxide) this film caused a intolerable drift in the signal.

Third, the film introduced a severe limitation on the excitation voltage, which in turn led to problems with signal to noise ratio. The material we had to use for this film had low thermal conductivity, resulting in inaccuracy and increased response times. The lesson learned from this is that we must eliminate this film altogether. This can be done by using a non conductive substrate. The best candidate appears to be a Boron Nitride rod.

(2) A protective thin film had to be applied to the RTD to protect it from the flow environment, especially the impingement of particles. This film causes increased response times and erodes quickly. An idea that would result in the elimination of the requirement for this film would be the use of a thermoelectric temperature sensor instead of the RTD. The resulting cool film calorimeter would consist of a boron nitride rod with two vacuum deposited metallic thin films at the face. Since the metallic thin films would have high thermal conductivity, the response time of this sensor would be extremely small. The "self healing" tendency of the thermoelectric sensor would also eliminate the need for another protective coating in most situations.

Our tests were directed towards obtaining data on longer runs. The cool film calorimeter can supply data only for a relatively short time, so that it will be necessary to revise testing procedures to emphasize shorter duration tests. The physical survival times of the gauges built by Lockheed, proved to be of the order of our predictions, however, the useful data times were much shorter.

In summary, our experience proved that it is possible to successfully build thin film based heat flux gauges. The basic knowledge required to arrive at a good design was obtained from the numerous lessons learned with our various designs. Using this knowledge it will be possible to extend the envelope of heat flux measurement well beyond the state of the art.

7.0 RESULTS

Initially, the various theoretical aspects of the operation of a cool film calorimeter were studied. These studies included analysis of response times, accuracy, survivability and probe applicability regions. It was found that the response time of the cool film calorimeter can be extremely short provided it is recognized that the accuracy of heat flux measurements diminishes very rapidly (due to the fact that the probe represents heat conduction into a semi-infinite solid). These probes are perfect for experiments that are extremely short in duration. The main design factor is that the total amount of energy transferred from the fluid to the model and probe must be small.

The thermal effect of the various thin films was modelled together with the temperature drop and thermal inertia across each film. These data proved useful in guiding the design effort, but since thin film material properties are significantly different from bulk properties, it was not possible to analyze survivability and thermal shock resistance. These parameters had to be found by experiment.

The inability to directly measure the thickness of the dielectric thin films after deposition meant that it was not possible to predict exactly what the film characteristics were. Instead, one had to rely on variations in the numerous setup parameters of the deposition process to, at least approximately, point to the thickness and quality of the resultant thin film.

A simple data reduction scheme was proposed. This scheme utilizes a numerical procedure to compute heat flux from the given surface temperature boundary condition. It assumes one dimensional heat conduction and a constant value of thermal conductivity was used throughout. Depending on the accuracy and application, the user can use his own data reduction methods since the operation of the probe is really independent of the data reduction scheme. If the temperature of the probe increases significantly beyond that of the surrounding body during the run it may be necessary to include the effects of two-dimensional heat conduction. This, however, is not the domain where this type of probe is most applicable, and should be avoided. Other designs based on different concepts will perform better in this regime.

After this analysis effort, design work was started on actual probes. This step presented the most difficulty since the most straightforward design (the first) could not be successfully manufactured. While each thin film deposition run requires a long time at our facility (due to heavy scheduling of the various deposition machines) we had to experiment with various film thicknesses and materials. The second design was easier to manufacture but proved to be faulty. Some basic physical effects had been overlooked that caused total probe failure at elevated temperatures. This failure occurred because a thin film was asked to bridge the interface between a ceramic adhesive coating and the copper substrate. Since the ceramic coating is extremely large relative to the dimensions of the thin film, thermal expansion of this ceramic destroyed the platinum thin film completely.

The third and final design utilized a more traditional approach in that it used separate leads to connect to the thin film, so that the heat conducting central rod was a separate entity. In this design, the platinum RTD was not required to bridge any discontinuities. Since the central heat conducting rod was still made of copper, a dielectric insulating thin film had to be used between the RTD and the substrate. Satisfactory deposition of this film turned out to be a major problem. The material used initially (boron nitride) was too soft, and could not adhere to the substrate effectively. In an effort to make this film thick enough to withstand the mechanical pressures of the leads, the adhesion properties of this material became so bad that even a slight touch with a soft cotton swab would remove the coating completely. Further, internal stresses in the coat were such that once the coat chipped off in one place, it would separate almost from the entire surface.

Thus an attempt was made to use a different material for the dielectric. None of the other materials, however, can match the thermal conductivity of boron nitride, so that fairly high temperature drops across the thin film had to be tolerated. The material chosen was silicon dioxide. This material had adequate adhesion and hardness. The dielectric strength was low, however, such that the signal output from the gauge had to be amplified strongly.

A platinum strip was then laid across the face of the probe to serve as the RTD. Leads were connected using silver paste and a protective coat of silicon carbide applied over the RTD.

This design was tested in two arcjet tests and limited success was achieved. The first series of tests at the Accurex arcjet facility provided useful data. It was possible to measure surface temperatures at least during the first runs and a heat flux calculation could be made. The heat flux results are only correct for a short time of about 1 second. After that, levels measured by our probes are significantly lower then those measured by Gardon gauges. Even though the first computed heat flux level is close to the expected value the data from the probe cannot be used yet for qualitative measurements because of an internal resistance change that is not due to the temperature change. This is entirely due to changes in the dielectric film between the copper substrate and the RTD.

Several lessons were learned from the results of these tests; (1) the useful data time for the cool film calorimeter is very short, (2) useful data can only be extracted while the amount of total heat transferred to the gauge and the surrounding surface is small. This means that the higher the heat flux the shorter the useful data time will be.

The reason for using a high thermal conductivity material like copper for the substrate was to maximize survival times. However, the use of an electrically conductive substrate requires using a dielectric thin film between the RTD and the substrate. This dielectric thin film has been the source of all the major manufacturing problems, and has also introduced some inaccuracies. The concern with survival times becomes secondary. Since the useful data time for these short duration experiments . This points to the use of a different approach in the choice of materials for the substrate. One could, for instance, use boron nitride rods and use a platinum thin film RTD. This would mean a smaller total survival time, but would not affect the useful data time significantly. On the other hand, the manufacture of these probes would be much simpler.

The face resistance of the probe placed a limitation on how small these probes could be made. In fact, the sizes used by Lockheed were probably the smallest possible with a straight platinum film RTD. The rod diameter could only be reduced by lengthening the path of the RTD using laser etching or photo-lithographic techniques. The dielectric thin film would also need to be eliminated for this process to be successful.

8.0 Conclusions

The cool film calorimeter concept was analysed. A set of parametric studies was conducted to guide design efforts and fabrication specifications.

Three gauges were designed and fabricated. Each design eliminated the problems encountered with the previous one. The last design produced a working gauge.

Thin film deposition processes were evaluated. The effect of different thin film materials on the performance and fabrication of the gauge was studied.

The gauges were tested in an arcjet facility. Survivability and accuracy were determined under various hostile environment conditions.

Based on the experiences gained from the analysis, design, and testing of the cool film calorimeter new types of gauges were defined.

9.0 RECOMMENDATIONS FOR FUTURE DIRECTIONS

An alternative, but very promising approach for a heat flux gauge is the use of thermoelectric effects to measure face temperature. Probes based on this concept would use a thin film type thermocouple at the face of the rod. A majority of the manufacturing problems would be eliminated with this approach while the short response times would remain. Several probes using this concept have been developed and tested under internal funding. While these sensors are exceptionally robust the accuracy suffers somewhat due to two dimensional heat conduction effects.

The use of a dielectric rod as a substrate, while diminishing the maximum heat flux capabilities somewhat, would result in the elimination of all the the thin insulating film manufacturing problems, and would lead to strikingly simple designs. The problem of face resistance remains, however, and has to be dealt with by using laser etching or photolithography. This technology is most promising for high speed applications that require very small physical size of the sensor.

The concept of the cool film calorimeter is based on the unsteady heat conduction and therefore suffers from limitations in terms of applicability to longer duration experiments (Figure 1). One can adapt some of the thin film technology investigated here, to differential temperature gauges. These heat flux gauges measure the temperature difference across a thin slice of material and thus arrive at the heat flux directly. This concept enables the gauge to provide accurate data without any usable time limitations.

In the differential temperature heat flux sensor category, the use of extremely high temperature materials like boron carbide and carbon (as a thermocouple pair) would make it possible to build gauges with temperature and heat flux capabilities that far surpass current technology as shown in Figure 1. The work on this type of sensor was performed as a continuation to the cool film calorimeter development under IRAD funding. Figure 27 depicts the basic components of this device. This sensor does not make use of thin film technology as yet and therefore does not have the superior response time characteristics associated with that technology, however, the temperature capabilities (maximum gauge temperatures in excess of 4000 degrees F) are impressive. A prototype of this sensor has been fabricated and tested successfully. Design improvements on this type of sensor will be continued and could possibly include response time improvements by using thin film technology.

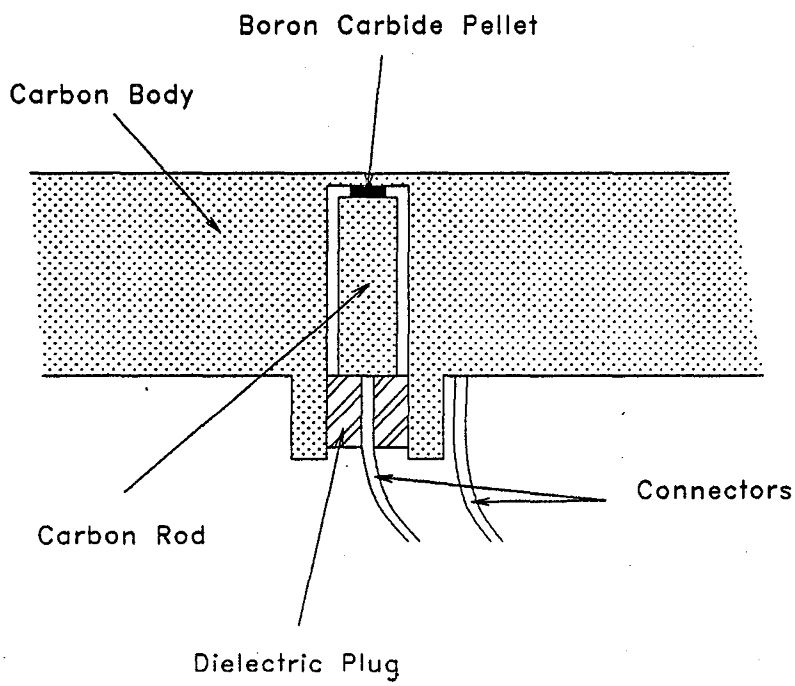

Figure 27 : Boron Carbide / Carbon Differential Heat Flux Gauge

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APPENDIX A

```
/* SOURCE CODE FOR DATA REDUCTION OF LOCKHEED HEAT FLUX GAUGE
INPUT = FACE TEMPERATURE HISTORY IN DEGREES C (1 READING EVERY SECOND)
OUTPUT = HEAT FLUX INTO PROBE FACE EVERY 10TH SECOND
WRITTEN BY A. ABTAHI,  LANGUAGE=C , NO NONSTANDARD EXTERNAL LIBRARIES REQUIR
INPUT AND OUTPUT MUST BE ON STANDARD (USE REDIRECTION)
*/
#include <stdio.h>
main()
{
int N,k,n;
float dx,t[200],tp[200],q,Tf,Tfn,Ti;
/* SETUP OF STEPS AND OTHER PARAMETERS */
N=134;
dx=0.001551;
/* READ INITIAL TEMPERATURE AND SET ALL NOTES TO THAT VALUE */
scanf("%f",&Ti);
for (k=0;k<=N;k++) t[k]=Ti;
Tfn=Ti;
/* THIS IS THE LOOP FOR THE TIME STEPS */
while (Tfn!=0)
{
Tf=Tfn;
/* READ NEW FACE TEMPERATURE */
scanf("%f",&Tfn);
if (Tfn!=0)
printf ("\n\n\n");
for (k=1;k<=1000;k++)
{
*tp=(Tf + (float)k*(Tfn-Tf)/1000.0 + t[1]) / 2.0;
/* FIND NEW TEMPERATURE DISTRIBUTION */
for(n=1;n<N;n++) tp[n]=(t[n-1]+t[n+1])/2.0;
tp[N]=tp[N-1];
/* FIND HEAT FLUX AT THE FACE */
q=(Tf + (float)k*(Tfn-Tf)/1000.0 - *tp)/dx * 0.1114999;
/* OUTPUT */
printf ("Tf= %f  Tn= %f     q=%f  \n",Tf + (float)k*(Tfn-Tf)/1000.0,tp[N],q)
/* PREPARE FOR THE NEXT TIMESTEP */
for (n=0;n<=N;n++)  t[n]=tp[n];
}
}
}
```