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Development of Thin Film Ceramic Thermocouples for High Temperature Environments

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August 2004

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The maximum use temperature of noble metal thin film thermocouples of 1100 °C (2000 °F) may not be adequate for use on components in the increasingly harsh conditions of advanced aircraft and next generation launch technology. Ceramic-based thermocouples are known for their high stability and robustness at temperatures exceeding 1500 °C, but are typically found in the form of rods or probes. NASA Glenn Research Center is investigating the feasibility of ceramics as thin film thermocouples for extremely high temperature applications to take advantage of the stability and robustness of ceramics and the non-intrusiveness of thin films. This paper will discuss the current state of development in this effort.

I. Introduction

To create the capabilities for long duration, more distant human and robotic missions for the Vision for Space Exploration, instrumentation and material technologies are being developed by NASA in its mission to enable safer, lighter, quieter, and more fuel efficient vehicles for aeronautics and space transportation. The Sensors and Electronics Technology Branch of NASA Glenn Research Center has an effort to develop thin film sensors for surface measurement in propulsion system research. The sensors include those for strain, temperature, heat flux and surface flow.

The use of thin film sensors has several advantages over wire or foil sensors. Thin film sensors do not require special machining of the components on which they are mounted, and, with thicknesses less than 10 μm, they are considerably thinner than wire or foils. Thin film sensors are thus much less disturbing to the operating environment, and have a minimal impact on the physical characteristics of the supporting components.

Four areas in the state-of-the-art thin film sensor technology are targeted for improvement as part of NASA's instrumentation research:

- Further development of electronics packaging and component testing of specialized sensors;
- Further development of fabrication techniques on curves and complex surfaces;
- Improved leadwire and film durability;
- Address needs for higher temperature applications exceeding 1000°C.

The Ceramics Branch at Glenn Research Center has an ongoing research effort to develop structural and functional ceramic technology for aero and space propulsion needs in conjunction with the Air Force Office of Scientific Research. The application of ceramics as thin film thermocouples can have the potential to meet the demands of advanced aerospace environments, as well as to the establishment of fundamental space operations capability.

The maximum temperature of noble metal thin film thermocouples of 1100 °C (2000 °F) may not be adequate for the increasingly harsh conditions of advanced aircraft and next generation launch technology. Ceramic-based thermocouples are known for their high stability and robustness at high temperatures, but are typically found in the form of rods or probes.[1] This investigation studies the feasibility of ceramics as thin film thermocouples for extremely high temperature applications thus taking advantage of both the stability and robustness of ceramics and the non-intrusiveness of thin films.

II. Ceramic TFTC Elements

The need to consider ceramic sensing elements is brought about by the temperature limits of metal thin film sensors in propulsion system applications. Longer-term stability of thin film sensors made of noble metals has been demonstrated at 1100 °C for 25 hrs.[2,3] Our previous experience indicates that noble metal thin films may be able to withstand 1500 °C for less than a minute in oxidizing environments. The capability for thin film sensors to operate in 1500 °C environments for 25 hours or more is considered critical for ceramic turbine engine development.[4,5] For future space transportation vehicles, temperatures of propulsion system components of at least 1650 °C to 3000 °C are expected.[6]

Ceramic materials can survive extreme temperatures. The borides, carbides, nitrides, and silicides of metals show high heat-resisting properties as well as metal-like electrical properties that make them attractive for use as sensing elements at high temperatures. An overview of these ceramics as thermocouples indicates that the silicides and carbides have the largest thermoelectric power.[7] Silicides have the added benefits of forming a passivating oxide coating in air, and carbides are able to be used in extremely high temperatures in inert and reducing environments. The ability of a particular ceramic to survive in harsh environments will influence its usefulness as a sensor. Tables 1 and 2 outline some bulk properties of silicide and carbide thermocouple elements.

Table 1.—Bulk Properties of High Temperature Carbide Thermocouple Elements.[7]

Carbide	Melting Point (°C)	Oxidation Temperature (°C)	Bulk Thermoelectric Power at 20 °C (μV/°C)	CTE (10^{-6} °C^{-1})	Absorption Cross Section for Thermal Neutrons (barns/molecule)[8]
WC	2720	800	–23	3.84	18.3
VC	2810	900	+3.7	7.20	5.08
TiC	3147	1200	–11.2	7.74	6.09
ZrC	3530	1200	–11.3	6.73	0.709
TaC	3880	1000	–5	8.30	20.6
HfC	3890	1200	–11.8	5.60	104.1

The three carbides with the highest melting points in table 1 are HfC, TaC, and ZrC, and ZrC has the added benefit of high radiation resistance. Previous investigations at Virginia Tech tested TaC films to 800 °C in vacuum and found them to have decreasing thermoelectric power with temperature. They also found good electrical and thermal stability when subjected to several thermal cycles.[9]

Table 2.—Bulk Properties of High Temperature Silicide Thermocouple Elements.[10]

Silicide	Melting Point (°C)	Oxidation Temperature (°C)	Bulk Thermoelectric Power at 20°C (μV/°C)	CTE (10^{-6}°C^{-1})	Absorption Cross Section for Thermal Neutrons (barns/molecule)[8]
$TiSi_2$	1510	1200	+9.4	12.42	6.43
$CrSi_2$	1770	1371	+360	12.81	3.39
$ReSi_2$	1980	1800	+313	-	145.1
$MoSi_2$	2049	1650+	–5.4	8.8	2.82
WSi_2	2116	1650	–0.4	8.3	18.6
$TaSi_2$	2371	1650	+25	8.82	20.9

From table 2, three silicides stand out. $CrSi_2$ has the highest thermoelectric power and good radiation resistance; $TaSi_2$ has the highest melting point; and $MoSi_2$ has the best oxidation and radiation resistance. An investigation at NIST demonstrated $MoSi_2$ and $TiSi_2$ as feasible thermocouple elements with protective overcoats to high temperatures in air. However, $TaSi_2$ and WSi_2 with protective overcoats and uncoated $ReSi_2$ failed before they reached 1000 °C due to oxidation effects.[11]

III. $CrSi_2$/TaC Sample Fabrication

In this investigation we report on the study to assess the higher temperature capability of TaC and $CrSi_2$ as thin film thermocouples in air. This decision was based on the high temperature potential of these systems in reducing environments where noble metal films are reactive.

The film depositions were conducted in the Class 1000 Microsystems Fabrication Cleanroom facility at Glenn Research Center using plasma sputtering PVD. The sputtered films are shown in figure 1. The deposition parameters are given in table 3. The thermocouple test sample was fabricated on a 127 mm × 38 mm × 1 mm alumina substrate, as shown in figure 2. Alumina substrates provide readily available test beds that remain intact for the severe heating required of these films, up to 1700 °C if necessary. After surfactant cleaning, the test shims were rinsed in acetone and methanol. The platinum was deposited first, followed by the platinum-rhodium alloy, the silicide, and ending with the carbide film. Platinum pads were added to connect leadwires to the sample.

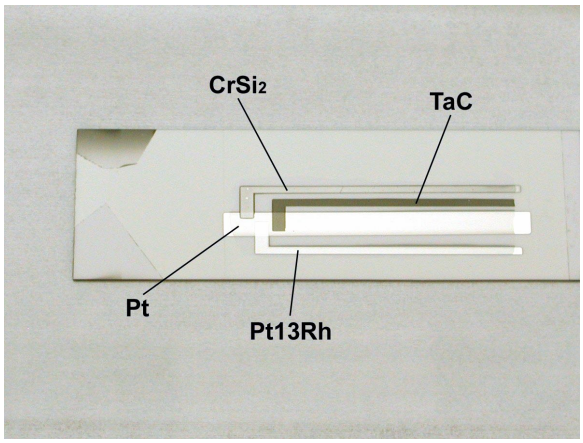

Figure 1.—The $CrSi_2$/TaC thermocouple test sample before the application of platinum connection pads.

Table 3.—Deposition Parameters of Films for the $CrSi_2$/TaC sample.

Film Deposited	Pressure & Gas	Power Density	Time	Film Thickness
Pt	8 mTorr Argon	250 Watts RF /182cm²	110 min.	3.4 µm
Pt-13%Rh	8 mTorr Argon	250 Watts RF /182cm²	120 min.	3.0 µm
$CrSi_2$	8 mTorr Argon	250 Watts DC /46cm²	462 min.	3.0 µm
TaC	8 mTorr Argon	250 Watts DC /46cm²	347 min.	3.0 µm

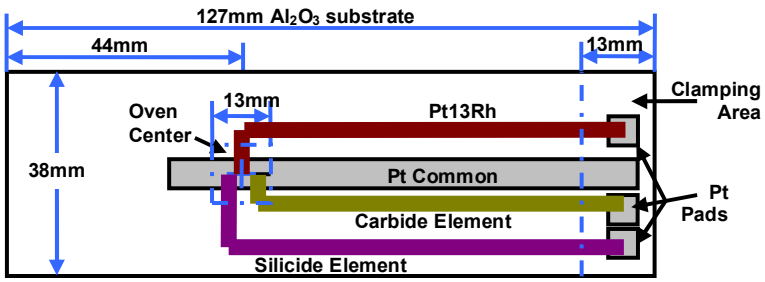

Figure 2.—Diagram of TFTC test sample.

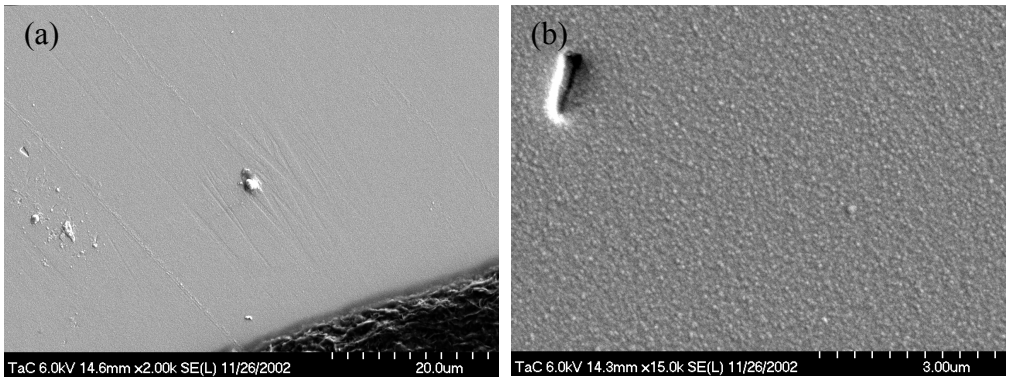

Figure 3.—SEM images of the as-deposited TaC film under (a) 2000× power, and (b) 15,000× power.

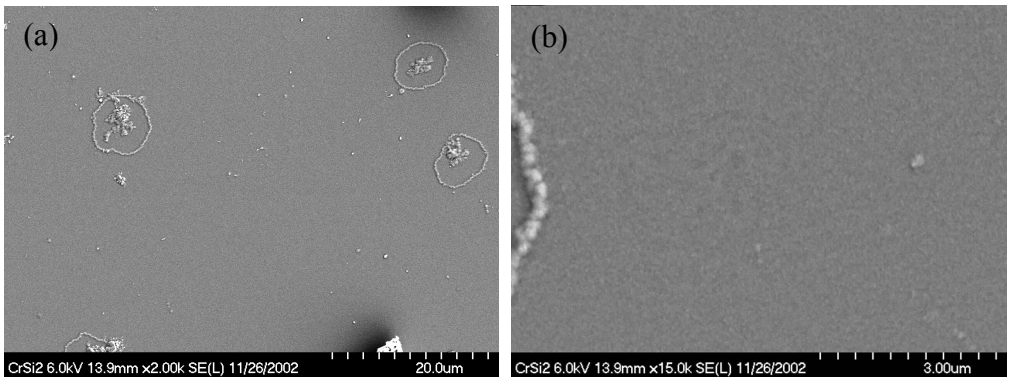

Figure 4.—SEM images of the as-deposited $CrSi_x$ film under (a) 2000× power, and (b) 15,000× power.

The deposited carbide and silicide films were extremely smooth, as shown by the SEM images of figures 3 and 4. Under XRD analysis, test films of TaC target material deposited on glass slides revealed that the deposited film was TaC, as shown in figure 5. No additional phases were observed. The deposited silicide film appeared to be amorphous. Different regions of the $CrSi_2$ target were analyzed by XRD to give insight of the deposited film phase and composition, as shown in figure 6. The main peak of $CrSi_2$ at $2\theta = 43°$ is observable in the target spectrum, as well as a slight secondary "bump" observed in the deposited film spectrum. These microstructure characteristics suggest that the deposited film was amorphous $CrSi_2$.

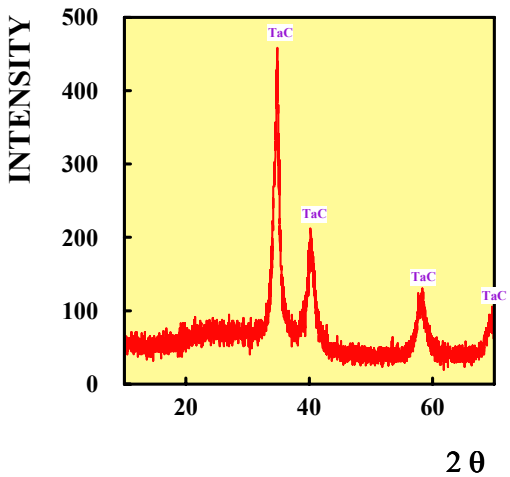

Figure 5.—X-Ray characterization of TaC as-deposited film.

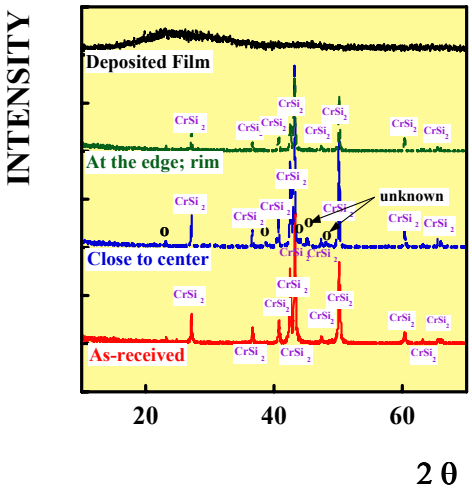

Figure 6.—X-Ray characterization of $CrSi_x$ target and as-deposited film.

IV. $CrSi_2$/TaC Sample Test

The sample was loaded into a clam-shell air furnace as shown in figure 7. The spatial heat distribution of the hot zone of the furnace formed a thermal gradient across the sample. A Type R thermocouple at the platinum common pad provided a signal to define the cold junction reference temperature. The signals were amplified and read on a computer acquisition system, which recorded the signal voltage in time. The furnace setting was set at 56 °C intervals starting with 93 °C. The resulting signals are shown in figure 8. This was done twice to determine repeatability.

The cold junction reference thermocouple signal and the thin film platinum-13% rhodium vs. platinum (Pt13Rh/Pt) thermocouple signals were converted to temperature using the ITS–90 inverse polynomials for Type R thermocouples after data acquisition was complete. Past studies of thin film Pt13Rh/Pt thermocouples fabricated at Glenn Research Center have shown them to be accurate to 3% of the temperature gradient for temperatures under 1200 °C.[12] The room temperature was measured to be consistently 26 °C, and was included in those calculations.

The TaC film failed during the first ramp. The thin film Pt13Rh/Pt thermocouple on the sample indicated a sample temperature of 455 °C. This is shown on figure 8. With only the $CrSi_2$ element still active, the sample was also tested over a period of 180 hours at the 815 °C setting, the upper limit of the furnace, giving a temperature of 670 °C on the sample. The output is shown in figure 9. The sample did not return to a zero-millivolt output after the end of the run, and held a charge for several days before being discharged. The attempt to gain a higher temperature reading destroyed the sample substrate due to thermal shock (see fig. 10).

Figure 7.—Thermocouple sample in clamshell oven.

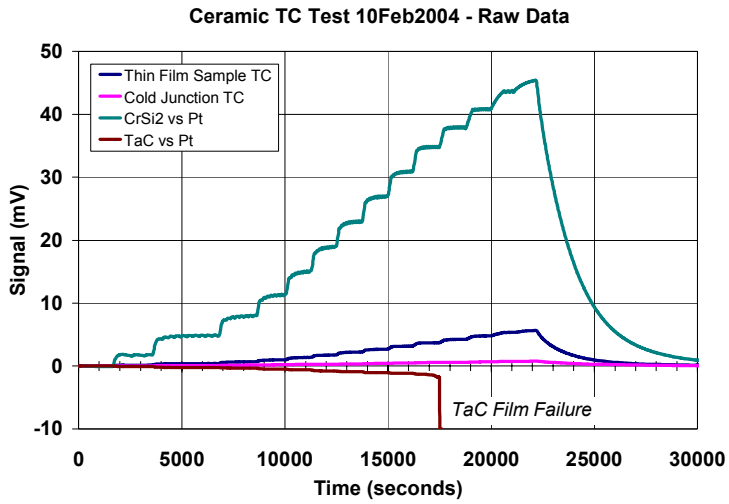

Figure 8.—$CrSi_2$/TaC thermocouple test sample output over time.

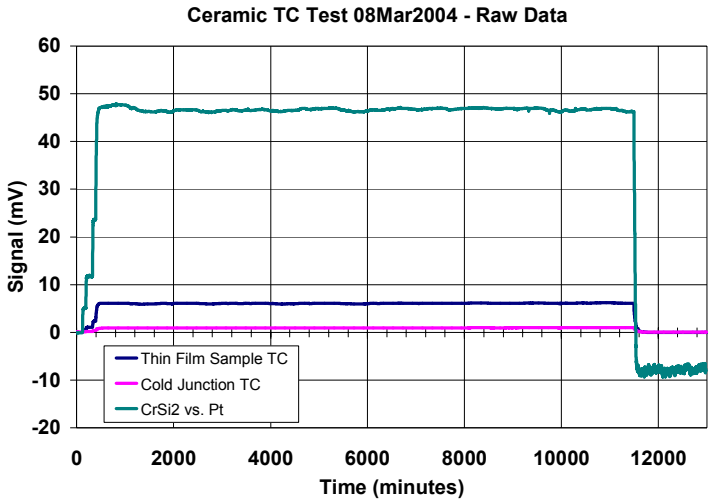

Figure 9.—Long-term test of $CrSi_2$ thermocouple.

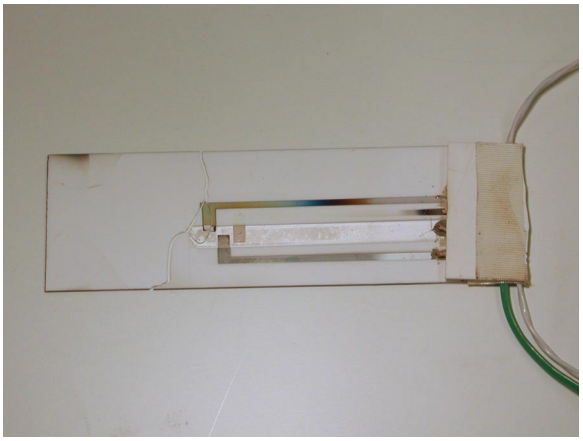

Figure 10.—Post-testing condition of $CrSi_2$/TaC thermocouple test sample.

V. $CrSi_2$ and TaC Thermoelectric Power

The signal output from a thermocouple is related to the Seebeck coefficient as:

$$\varepsilon = \int_{T_1}^{T_2} S(T) \cdot dT$$

where ε is the thermoelectric voltage generated by the thermocouple, $S(T)$ is the Seebeck coefficient, T_1 the low temperature of the gradient, T_2 the high temperature. Typically, the charts for the thermoelectric voltage are given with reference to a temperature of 0 °C or 20 °C so that as the Seebeck coefficient changes with temperature, the correct temperature can be calculated from knowing the actual T_1 of the gradient. In our case, T_1 is floating, not tied to a reference. The linearity of the Seebeck coefficient at lower temperatures is used to determine the thermoelectric voltage relative to a fixed reference temperature.

The resulting plot for $CrSi_2$ vs. Pt thermoelectric voltage vs. temperature referenced to 0 °C is shown in figure 11. The curve follows the cubic relation $\varepsilon(T)_{T_1=0} = -6.494\times10^{-8}*T^3 + 3.4205\times10^{-5}*T^2 + 9.831\times10^{-2}*T$ to within 1.4%, which is also shown in figure 11. For TaC vs. Pt, the thermoelectric voltage vs. temperature referenced to 0°C is shown in figure 12. The resulting curve follows the cubic relation $\varepsilon(T)_{T_1=0} = 3.3558\times$

$10^{-9}*T^3 - 6.2915\times10^{-7}*T^2 - 4.2835\times10^{-3}*T$ to within 1%, which is also shown in figure 12. It should be noted that the deviation from linearity is seen to be 10% immediately before failure of the films. Based on the trends shown in figures 11 and 12, these thermocouple elements may be linear to at least 900 °C for $CrSi_2$ and 600 °C for TaC in non-oxidizing environments. The constant Seebeck coefficients relative to platinum are +102 μV/°C for $CrSi_2$ and –4.3 μV/°C for TaC, accurate to ±3% from the uncertainty of our temperature measurement.

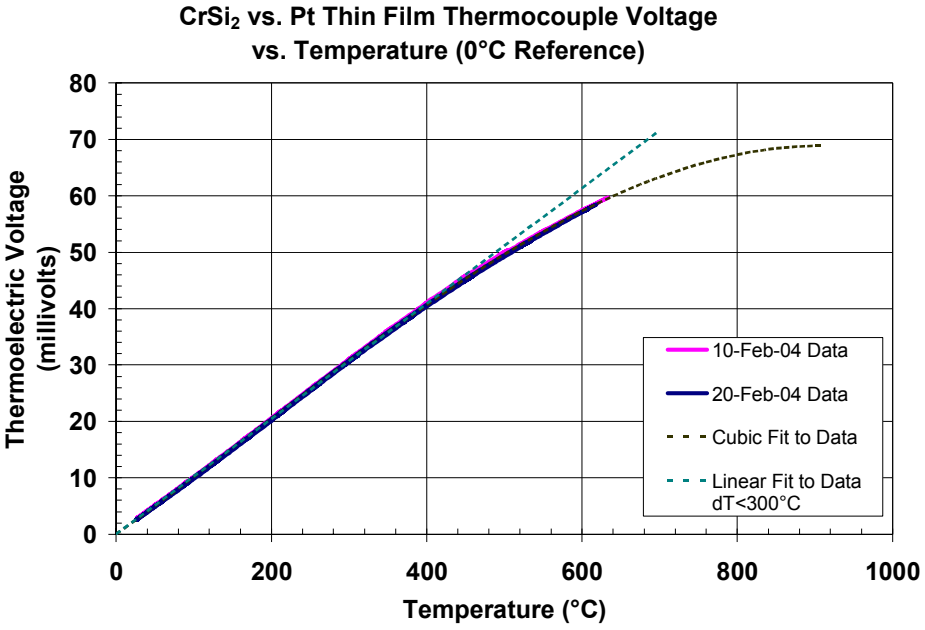

Figure 11.—Thermoelectric voltage output determined for $CrSi_2$ vs. Pt referenced to 0 °C.

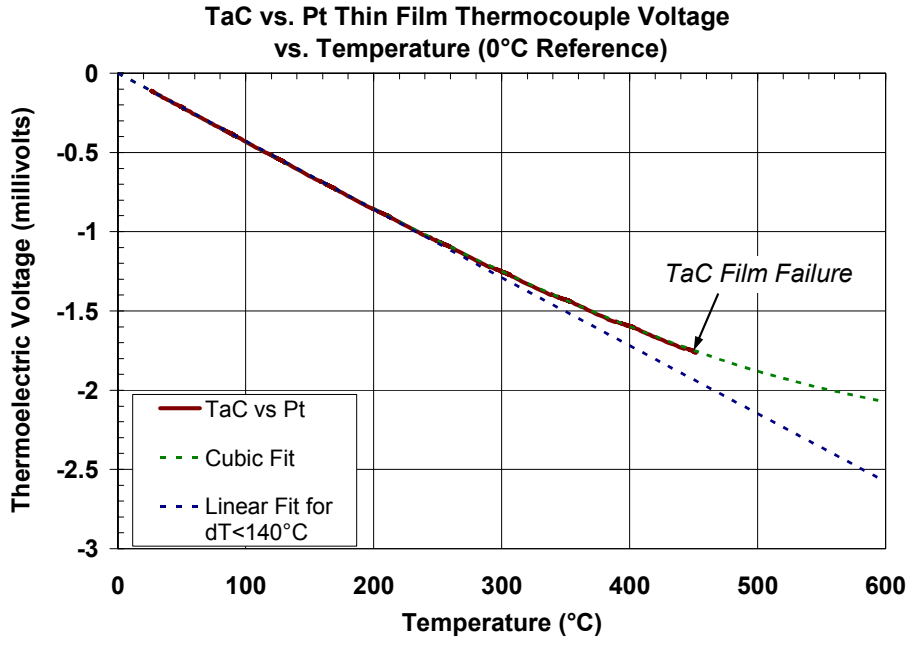

Figure 12.—Thermoelectric voltage output determined for TaC vs. Pt referenced to 0 °C.

VI. Conclusions and Future Work

Thermoelectric data on $CrSi_2$ and TaC was gathered for temperatures up to 650 °C for $CrSi_2$ and 450 °C for TaC. Extrapolating the data for environments where oxidation is not an issue, $CrSi_2$ vs. Pt appears to be linear to 900 °C and TaC vs. Pt linear to at least 600 °C. It is suspected that the oxidation contributed to the deviation from linearity for both cases. The quantitative amount of this contribution and therefore the upper use temperature for both of these systems under repeated use conditions could not be estimated from our study and requires further work. On the other hand, the present investigation showed significant promise of this class of materials as a ultra-high temperature thermocouple pairs provided that the oxidation challenge is addressed. The possibility exists to utilize these classes of materials at very high temperatures in reducing environments for next generation launch vehicles. The paradigm for the use temperature, time and the environmental conditions of application requires further study.

Based on the low oxidation temperatures of these thin film thermocouple elements, additional research needs to be conducted into protective overcoats for the films if they are to be practical in oxidizing environments. Also, testing of the thin film ceramic thermocouples in an inert atmosphere or vacuum will be necessary to gain an understanding of their performance and applicability in space relevant environments.

The merging of the high temperature capabilities of ceramics with the non-intrusiveness of thin films is on-going. It appears that a new class of ceramic thin films can be used as high temperature thermocouples, and we intend to apply this technology to strain gauges as well. This research advances the effort to develop a complete sensor package to enable the use of ceramics as thin film sensors in environments where standard metal sensors would not survive.

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The maximum use temperature of noble metal thin film thermocouples of 1100 °C (2000 °F) may not be adequate for use on components in the increasingly harsh conditions of advanced aircraft and next generation launch technology. Ceramic-based thermocouples are known for their high stability and robustness at temperatures exceeding 1500 °C, but are typically found in the form of rods or probes. NASA Glenn Research Center is investigating the feasibility of ceramics as thin film thermocouples for extremely high temperature applications to take advantage of the stability and robustness of ceramics and the non-intrusiveness of thin films. This paper will discuss the current state of development in this effort.

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