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EMITTANCE VALUES OF HAYNES-25 FOR A SELECTED REENTRY ENVIRONMENT

AEROSPACE NUCLEAR SAFETY DEPARTMENT, 9310

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A SELECTED REENTRY ENVIRONMENT

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ABSTRACT

Measurements of total normal and total hemispherical emissivities were performed on specimens of Haynes-25 having various thicknesses of oxide. This program was undertaken to develop emissivity values for use in Aerospace Nuclear Safety reentry analyses where the oxide is developing during reentry.

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SUMMARY

In the reentry heating analyses of SNAP isotopic power sources, it is important to know the emittance values of the isotopic generator, the exterior surfaces of which are oxidizing as it reenters. This report outlines a method of oxidizing Haynes-25 specimens with respect to time, temperature, and pressure from typical SNAP trajectories for pre-orbital abort and orbital decay reentries. Total normal emissivity measurements ranged from 0.10 at 1500°R to 0.90 at 2800°R for orbital decay and from 0.28 at 1500°R to 0.90 at 2800°F for preorbital abort. The emissivity of unoxidized samples was 0.10 at 1500°R.

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EMITTANCE VALUES OF HAYNES-25 FOR A SELECTED REENTRY ENVIRONMENT

CHAPTER I -- INTRODUCTION

The Aerospace Nuclear Safety Program, which has a direct interest in the reentry of isotopic fuel capsules into the earth's atmosphere, conducted this study to obtain data for use in aerodynamic heating analyses. In these analyses, it is important to determine the amount of heat which will be reradiated during reentry or abort. The magnitude of this reradiation is dependent upon the emittance of the surface of the vessel.

Sandia Laboratory selected Atomics International, a division of North American Aviation, to prepare specimens and obtain emittance measurements on Haynes-25 specimens oxidized to simulate reentry conditions. Since the emittance properties of a surface are dependent on surface finish, the quality of the oxide, and the test temperatures, extensive effort was made to simulate actual reentry conditions. The time, skin temperature, and stagnation pressure for both preorbital abort and orbital decay were calculated by Sandia and are presented in Figures 1 and 2. The total normal emissivity measurements made at the oxidation temperature were performed at the Los Angeles Division of North American Aviation.

Figure 1. Fuel capsule temperature and pressure during preorbital abort trajectory (D67-14498)

Figure 2. Fuel capsule temperature and pressure during orbital decay trajectory (D67-14499)

CHAPTER II -- MEASUREMENT OF TOTAL NORMAL EMITTANCE OF HAYNES-25

Preparation of Specimens

Specimens of the Haynes-25 sheet stock used in the fabrication of isotopic generators were obtained from Martin Marietta Corporation. This sheet stock was cut at Sandia into 1-inch diameter discs with a thickness of 0.022 inch. These specimens were then furnished to Atomics International for oxidation and emittance measurements. The surface finishes of representative specimens were measured by a Brush analyzer and found to be approximately 17-microinch RMS.

During the initial checkout of equipment, it was discovered that the oxide formed on the surface of the Haynes-25 was nonadherent, with flaking of the oxide taking place immediately. This was attributed to a contaminant (e.g., grease) present on the surface in the "as-received" condition. An ethyl-alcohol rinse was then used for each specimen prior to the oxidation treatment; this eliminated the flaking problem.

Oxidation of Specimens

For purposes of oxidation treatment, the specimens were heated by radiation from a platinum susceptor which was heated by a high-frequency induction unit. Air was introduced into the system by means of a needle valve, while a mechanical pump continued to evacuate the system. The pressure was monitored by means of a linear pressure transducer.

The specimens were suspended in the platinum susceptor by a platinum-10 percent rhodium thermocouple (Figure 3). The vacuum system used for the controlled oxidation cycles is shown in Figures 4, 5, and 6. The thermocouple was spot-welded to the back side of the specimen to insure correct temperature response.

A pressure-vs-time curve was plotted on the recorder chart paper prior to each run. The pressure transient was programmed by manually

adjusting the needle valve to follow the chart paper program. At the end of the desired oxidation cycle, a solenoid valve was opened, and the system was purged with argon.

The temperature transient was programmed in a similar manner. The induction unit was manually adjusted so that the specimen temperature followed a predetermined program on the temperature recorder chart paper.

The specimens which were heavily oxidized were a dark gray color. X-ray fluorescence analysis performed on an oxidized specimen revealed the oxide to be composed of 27 percent chromium oxide, 8 percent nickel oxide, and 65 percent cobalt oxide. It appears that the composition of the oxide was not appreciably different from the composition of the base material, with the exception of the tungsten. The Haynes-25 composition is 19 to 21 percent Cr, 14 to 16 percent W, 8 to 11 percent Ni, 2 percent maximum Fe, and the balance Co.

Figure 3. Oxidation technique
(D67-14500)

Figure 4. Oxidation control equipment (D67-14501)

Figure 5. Oxidation vacuum system
(D67-14502)

Figure 6. Oxidation heating system
(D67-14503)

Emittance Measurements

There are several techniques used in measuring total normal emittance; the more common technique is to place the reference blackbody and the test specimen in identical optical paths from the detector and compare the radiated energy of the specimen to the radiated energy of the blackbody when both are at the same temperature.

A refinement to the above technique, called the "sliding specimen method," was used to avoid errors in the optical path lengths and temperature differences. In this method, the optical paths are combined into one when the specimen is placed within the reference blackbody and both are at the same temperature. Using the furnace which heats the specimen as the reference blackbody permits this refinement (Figure 7). Once the energy level radiated by the blackbody is determined, the sample is moved rapidly to the position occupied by the blackbody aperture, and the detection system then records only the energy radiated by the sample. The sample acts as a shield and prevents blackbody radiation from reaching the detector. Once the blackbody radiated energy (E_b) and specimen radiated energy (E_s) are determined, a simple ratio yields the total normal emissivity (ϵ_{t_n}) of the material:

$$\epsilon_{t_n} = \frac{E_s}{E_b}$$

As the sample is moved forward from the rear of the furnace cavity to the aperture, the detection system records a decrease in energy radiated from the blackbody level to that of the specimen level. As the specimen reaches the aperture, radiative cooling of the sample takes place, this provides an additional decrease in the energy level sensed by the detection system. This energy level change is monitored on a high speed strip chart recorder.

There is a marked difference in the slopes of the recorded energy levels: (1) as the sample moves from the rear of the cavity to the aperture, and (2) while the sample dwells at the aperture. The intersection of the two curves on the chart identifies the energy level corresponding to the emittance of the sample. The 100 percent energy level of the blackbody cavity and the zero energy level of the system are recorded. The zero energy level is determined by placing a cold wall in the optical path between the detection system and the blackbody cavity.

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Figure 7. Emittance measuring system (D67-14504)

A typical chart showing the blackbody and specimen trace is presented in Figure 8.

The total normal emittance of the Haynes-25 alloy was determined by inserting a specimen into the specimen change compartment of the push rod assembly (Figure 9) and roughing down to 10^{-3} Torr by a vacuum pump. Meanwhile, Compartment A (test chamber) was held at the required temperature and pressure. The test conditions were maintained with a separate vacuum system, including diffusion pumping capabilities. To meet the goals of this program, Compartment A was maintained at 4×10^{-4} Torr.

After stabilizing, the gate valve separating the compartments was opened, and the specimen was introduced into the hot furnace zone by the push rod assembly. Timing began at the instant the specimen reached its test position at the rear of the cavity.

The specimen was held in position for 2 to 3 minutes, depending on furnace temperature, to reach thermal equilibrium with the furnace. The zero and 100 percent energy levels of the furnace were meanwhile recorded on the constantly running strip chart recorder. The specimen

Figure 8. Typical hi-speed strip chart record (D67-14505)

was then pushed rapidly to the aperture where the detection system recorded the cooling rate. The specimen was then returned to its test position at the rear of the cavity where it was reheated prior to repeating the test procedure. The time for each test point was recorded and the final data presentation is a plot of emittance versus time at temperature. This type of representation was used to avoid the question of oxide stability under vacuum conditions. From this plot, the emittance of the Haynes-25 alloy specimen can be determined at time zero by extrapolating from the measured values and known times.

Upon completion of the specimen test interval (usually four minutes in duration), the specimen was returned to the specimen change compartment where it was cooled and discharged.

For use in reentry heating analyses, emittance values at four temperatures are required to establish a total normal emissivity curve.

Figure 9. Specimen test chamber
(D67-14506)

Test Equipment

Detection System

A Barnes Engineering Company Model R4F2 dual channel radiometer system, equipped with lead-sulphide and bolometer detectors (Figure 10), was used for radiation detection purposes. This particular model has a tripod-mounted radiometer head which is focusable and is filterable if narrow band pass measurements are desired. The bolometer detector was used because it has a flat spectral response. With this detector, the data are used directly as printed by the recorder, instead of applying the spectral response correction that is necessary when employing a lead-sulphide detector. The information sensed by the radiometer is converted by a synchronous rectifier amplifier to a signal form usable in a Sanborn Model No. 152-100BP strip chart recorder.

Test Furnace

The test furnace (Figure 11) consisted of a long, cylindrical central test zone, heated to desired temperatures by means of an induction-heated concentric susceptor. A water-cooled aperture at the front (or viewing) end of the furnace shielded the detection system from spurious radiation while providing a cold mount for a KBr transmission window. This assembly of aperture and window comprised the front pressure or vacuum seal for the apparatus.

The primary structural tube at the rear was welded to an adaptor flange and connected to a gate valve by Ladish 2-inch quick-disconnect ferrules. The gate valve was, in turn, connected by quick-disconnect ferrules to the push rod and specimen change compartment. Vacuum seals were obtained by welding this tube directly to the viewing aperture and to the rear adaptor flange.

The primary test zone was pumped by roughing and diffusion pumps to a pressure of 4×10^{-4} Torr, while the specimen change compartment was pumped by a roughing pump to 1×10^{-3} Torr. This compartment was connected to a separate vacuum system so that a specimen could be changed without disturbing the test cavity environment. The vacuum of the test cavity was constantly monitored by thermocouple and ionization gauges, and its temperature was monitored by thermocouples below 2450°R and by a color-matching optical pyrometer above 2450°R . Wall emittance errors

Figure 10. Emittance detection equipment
(D67-14507)

Figure 11. Emittance vacuum and specimen change chamber
(D67-14508)

normally associated with optical pyrometers were eliminated because the cavity simulated a blackbody. Comparisons of thermocouple and optical pyrometer temperatures consistently exhibited agreement to within 15 degrees.

During the course of the program, four different furnaces were used. These furnaces differed in test cavity structural material. Table I, which relates specimen number to measured emittance, also specifies the test furnace in which this value was determined. These furnaces are described below and are designated Furnace A, B, C, and D.

Furnace A -- The test cavity structural material of Furnace A was a Type 304 stainless-steel tube with a tantalum susceptor. Vacuum seals were obtained by welding this tube directly to the viewing aperture and to the rear adaptor flange. Its use was negated when a vacuum failure oxidized the inner walls of the tantalum susceptor comprising the test chamber. This oxidized wall liberated sufficient oxygen under test temperature vacuum conditions to severely alter the surface of the test specimens. Use of this furnace was discontinued.

Furnace B -- The primary test chamber structural material of Furnace B was pure alumina. This furnace had been constructed under a different contract and a nickel-chromium-cobalt spinel coating lined the inner surface of the test cavity. It was recognized that evaporation of this coating under vacuum conditions could alter the emittance characteristics of the test specimens, but at lower temperatures this effect would be negligible. The deposition rate of the spinel coating was slight at 1500°R, and meaningful data were obtained. At 2100°R, the deposition rate of the interior coating was too high to permit reliable extrapolation, and use of this furnace was discontinued until it could be modified.

Furnace C -- The primary test cavity structural tube in Furnace C was fabricated of Inconel-600 nickel-base alloy (Figure 11). The furnace performed satisfactorily to a maximum temperature of 2500°R.

Furnace D -- Additional alternations to Furnace B permitted 2800°R measurements to be conducted. Modified Furnace B is designated Furnace D (Figure 9) in this report. An alumina ceramic tube was the primary test cavity tube member in Furnace D. This furnace differs from Furnace B in

that the interior coating was removed from the ceramic, and the internal structure of the furnace was modified. This furnace was calibrated with National Bureau of Standards samples and has an effective cavity emittance of 97 percent. This slightly lower performance figure necessitated correcting measured values by 3 percent.

Results

The corrected (extrapolated) emittance values are presented in Table I. The emittance values as a function of temperature are presented in Figures 12 and 13, and basic data are presented in Figures 14 through 21.

TABLE I
Corrected (Extrapolated) Emittance Values (D67-14509)

Specimen Number	Test Temperature (°R)	Test Vacuum	Emittance *	Test Furnace	Oxidation Profile
27	1500	4×10^{-4}	0.28	B	Preorbital Abort
28	1500	4×10^{-4}	0.28	B	Preorbital Abort
29	1500	4×10^{-4}	0.27	B	Preorbital Abort
48	1500	4×10^{-4}	0.17	C	Preorbital Abort
43	1500	2×10^{-4}	0.09	C	Orbital Decay
44	1500	2×10^{-4}	0.09	C	Orbital Decay
45	1500	2×10^{-4}	0.10	C	Orbital Decay
46	1500	2×10^{-4}	0.10	C	Orbital Decay
25	2100	4×10^{-4}	0.53	B	Preorbital Abort
26	2100	4×10^{-4}	0.48	A	Preorbital Abort
49	2100	4×10^{-4}	0.58	C	Preorbital Abort
50	2100	4×10^{-4}	0.59	C	Preorbital Abort
39	2100	9×10^{-4}	0.35	A	Orbital Decay
40	2100	6×10^{-4}	0.41	A	Orbital Decay
41	2100	6×10^{-4}	0.36	A	Orbital Decay
42	2100	6×10^{-4}	0.34	A	Orbital Decay
20	2500	6×10^{-4}	0.62	A	Preorbital Abort
51	2500	4×10^{-4}	0.72	C	Preorbital Abort
52	2500	4×10^{-4}	0.71	C	Preorbital Abort
53	2500	4×10^{-4}	0.69	C	Preorbital Abort
35	2500	4×10^{-4}	0.70	A	Orbital Decay
36	2500	9×10^{-4}	0.82	C	Orbital Decay
37	2500	7×10^{-4}	0.80	C	Orbital Decay
38	2500	4×10^{-4}	0.80	C	Orbital Decay
3	2800	4×10^{-4}	0.90	D	Preorbital Abort
4	2800	4×10^{-4}	0.90	D	Preorbital Abort
17	2800	4×10^{-4}	0.90	C	Preorbital Abort
18	2800	4×10^{-4}	0.86	D	Preorbital Abort
31	2800	4×10^{-4}	0.89	D	Orbital Decay
32	2800	4×10^{-4}	0.89	D	Orbital Decay
33	2800	4×10^{-4}	0.89	D	Orbital Decay
34	2800	4×10^{-4}	0.89	D	Orbital Decay

* All emittance values ± 0.01

Figure 12. Preorbital abort emittance values (D67-14510)

Figure 13. Orbital decay emittance values (D67-14511)

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Figure 14. Total normal emittance values
(specimens 27, 28, 29, 48)
(D67-14512)

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Figure 15. Total normal emittance values
(specimens 43, 44, 45, 46)
(D67-14513)

Figure 16. Total normal emittance values (specimens 25, 26, 49, 50) (D67-14514)

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Figure 17. Total normal emittance values
(specimens 39, 40, 41, 42)
(D67-14515)

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Figure 18. Total normal emittance values
 (specimens 20, 51, 52, 43)
 (D67-14516)

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Figure 19. Total normal emittance values
(specimens 35, 36, 37, 38)
(D67-14517)

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Figure 20. Total normal emittance values
(specimens 3, 4, 17, 18)
(D67-14518)

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Figure 21. Total normal emittance values
(specimens 31, 32, 33, 34)
(D67-14519)

CHAPTER III -- MEASUREMENT OF TOTAL HEMISPHERICAL EMITTANCE OF HAYNES-25

Total Hemispherical Emittance

The total hemispherical emissivity of oxidized Haynes-25 was measured using the "filament in vacuum" technique. For these measurements, a strip of Haynes-25 sheet stock 12-inches long, 0.25-inch wide, and 0.017-inch thick was oxidized as shown in Figures 22 and 23. This specimen was instrumented with four platinum - platinum 10 percent rhodium thermocouples and mounted as a filament in the test chamber (Figure 23). The test chamber was then reduced to a vacuum of 1×10^{-4} Torr.

Emissivity measurements were then determined using the following equation:

$$\epsilon = \frac{\text{POWER}}{(\sigma T_1^4 - \sigma T_2^4)A}$$

where

Power = voltage measured between center two thermocouples times the current;

T_1 = average temperature between thermocouples;

T_2 = temperature of wall sitting in liquid nitrogen bath, assumed to be LN_2 temperature;

σ = Stefan-Boltzmann constant; and

A = surface area of specimen between voltage taps.

The total hemispherical emissivity values show a decrease in emittance at approximately 2,000°F (Figure 24). This trend is also shown in Reference 2. The total hemispherical values shown in Figure 24 are lower than the expected values for stably oxidized Haynes as presented by Wade in Reference 1. The specimen was rerun and checked at 600, 1000, and 2000°F and duplicated the values shown in Figure 24. Posttest examination of the specimen showed that some of the oxide was lost.

Figure 22. Total hemispherical oxidation technique
(D67-14520)

Figure 23. Total hemispherical emittance measuring system (D67-14521)

Figure 24. Total hemispherical emissivity measurements
(D67-15560)

In measuring total hemispherical emittance using the "filament in vacuum" technique, it was impossible to maintain the oxide coatings for the length of time required (6-8 hours) to perform measurements up to 2000°F.

The total hemispherical emissivity of stably oxidized Haynes-25, as determined by W. R. Wade,¹ was established by taking measurements of the radiant flux at angles of incidence from 0 to 60 degrees over the temperature range from 600 to 2000°F. These measurements exhibited good correlation with the Lambert cosine law for diffuse emission. Therefore, as stated by Wade, total normal emissivity values are very close to the values of total hemispherical emissivity for oxidized surfaces of Haynes-25 with diffuse emission.

CHAPTER IV -- CONCLUSIONS

Total normal emissivities of oxidized Haynes-25 specimens were determined in accordance with a simulated reentry environment of a typical SNAP isotopic generator. The Haynes-25 surface which develops the oxide during reentry has emittance values ranging from 0.1 to 0.9 up to a temperature of 2800°R. Early in the reentry trajectory the Haynes-25 surface is unoxidized and exhibits a low emissivity value. Once the oxide begins to form there is sufficient oxygen present to maintain this oxide buildup. A Haynes-25 surface with a fully developed oxide coating has emissivity values of up to 0.9.

Based on the data obtained and the correlation by Wade,¹ it is concluded that the total normal emissivity approximately equals the total hemispherical emissivity for similarly oxidized diffuse surfaces of Haynes-25.

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