NASA Technical Memorandum 101948

The Emittance of Space Radiator Materials Measured at Elevated Temperatures

Michael J. Mirtich
Lewis Research Center
Cleveland, Ohio

Frank DiFilippo
Case Western Reserve University
Cleveland, Ohio

and

Jennifer Barry and Michael Kussmaul
Cleveland State University
Cleveland, Ohio

Presented at the
15th International Conference on Metallurgical Coatings
sponsored by the American Vacuum Society
San Diego, California, April 11-15, 1988

NASA

THE EMITTANCE OF SPACE RADIATOR MATERIALS MEASURED AT ELEVATED TEMPERATURES

Michael J. Mirtich
National Aeronautics and Space Administration
Lewis Research Center
Cleveland, Ohio 44135

Frank DiFilippo
Case Western Reserve University
Cleveland, Ohio 44106

and

Jennifer Barry and Michael Kussmaul
Cleveland State University
Cleveland, Ohio 44115

E-4641

SUMMARY

The spectral emittances of textured space radiator materials between 1.7 and 14.7 μm have been evaluated at room temperature and elevated temperature (630 °C) in air. Heating in air caused a permanent increase in spectral emittance for all materials tested: HCl/ion beam textured 304 stainless steel, untextured Ti (6 percent Al, 4 percent V), and sandblasted Ti (6 percent Al, 4 percent V). Changes in the surface chemistry and/or surface morphology of these materials were also observed. Elevated temperature spectral emittance was measured in an argon atmosphere and compared to the measurements in air. Similarity between the room temperature and elevated temperature spectral emittance measurements was also investigated, and limited agreement was found.

INTRODUCTION

The development of nuclear reactor power systems for space applications requires that the system be able to fit in the Shuttle bay. To assist in meeting this requirement, the area of the system's radiator can be minimized by increasing the thermal emittance of the radiator surface. For the SP-100 nuclear space power system, the desired thermal emittance is greater than or equal to 0.85 at 700 to 900 K.

Various techniques, such as sandblasting, discharge chamber texturing, and carbon arc electrical discharge, have been used on candidate radiator materials to achieve high emittance values (refs. 1 and 2). The thermal emittance at elevated temperatures for these materials was determined by normalizing the room temperature spectral emittance to a 700 to 900 K blackbody. This method of calculating the thermal emittance assumed that the spectral emittance at elevated temperatures was the same as that at room temperature. However, this may not be a valid assumption, especially if the surface chemistry, composition, or texture of the materials changes upon heating.

For this reason, the effects of elevated temperatures on the emittances of two materials, Ti (6 percent Al, 4 percent V) and 304 stainless steel, were investigated at elevated temperatures and in atmospheres of variable composition. Spectral emittances were measured at both room temperature and elevated

temperatures in order to detect a change in emittance, and surface analysis was performed to provide explanations for the emittance changes.

APPARATUS AND PROCEDURE

Surface Texturing

Titanium (6,4) samples were textured in a S.S. White sandblaster with 60 μm SiC as the impinging particle at an operating pressure of 10 psi. The samples were positioned 6 to 7 cm from the nozzle with the particles directed normal to the surface. The samples were sandblasted until there was no visible change in appearance.

Stainless steel (304) samples were etched in hot concentrated HCl for 12 min, rinsed in distilled water, and blown dry with nitrogen gas. Afterwards, the samples were placed in an argon ion discharge chamber operating at 1500 V and ~700 °C for 2 hr. The discharge chamber texturing system is described more completely in reference 1.

Spectral Emittance Measurements

Reflectance measurements were made at room temperature from 1.7 to 14.7 μm using the hohlraum reflectometer attachment of a Perkin-Elmer model 13 spectrophotometer (fig. 1). Total reflectance was measured as the intensity ratio of the radiation reflected by the water-cooled sample to the radiation emitted by the wall of the heated cavity (>0.99 blackbody), which was kept at 630 °C. The measured spectral reflectance (r_λ) was then converted to spectral emittance (ε_λ) by:

$$\varepsilon_\lambda = 1 - r_\lambda \tag{1}$$

which follows directly from Kirchoff's law for an opaque surface.

Spectral emittance measurements were also made at elevated temperatures over the same wavelength range using the emissometer attachment of the spectrophotometer (fig. 2). The samples were heated to 630 °C in the emissometer with the normally emitted radiation reflected into the spectrophotometer. Calculating the intensity ratio of the radiation emitted by the sample to the radiation emitted by the cavity wall at the same temperature yielded the spectral emittance in air at 630 °C.

A special hood was designed to allow emittance measurements in atmospheres other than air (fig. 3). The hood was a metal dome 15 cm in diameter and 7.5 cm in height which covered the sample and had a transmitting ZnSe window in the optical path. The gaseous environment was created by running a slow purge of gas through the dome, entering through an inlet at the top and exiting through a side outlet. The purge was maintained while obtaining the spectral emittance and after the run while the sample was cooling.

The spectral emittances of untextured and sandblasted Ti (6,4) samples were measured at room temperature in the hohlraum reflectometer before and after heating at 630 °C for 4 hr. The spectral emittance of the sandblasted Ti (6,4) was also measured at 630 °C in the emissometer after 0 to 10,

20 to 30, 40 to 50, and 60 to 70 min of heating. The 10-min time spans corresponded to the time necessary to complete the measurements. The spectral emittance of another sandblasted Ti (6,4) sample was then determined at 630 °C in an argon atmosphere using the metal dome attachment for the emissometer after heating for 1 hr.

Similarly, the spectral emittance of the HCl etched and then ion beam textured stainless steel type 304 sample was measured at room temperature before and after heating at 630 °C for 4 hr. After 1 hr of heating, the spectral emittance was measured at 630 °C in the emissometer.

Surface Analysis

Surface analysis was performed before and after heating in the emissometer in order to determine the causes for changes in spectral emittance. The chemical compositions of the sample surfaces were determined by ESCA analysis. A Scanning Electron Microscope was used to observe the surface morphology of all the samples before and after heating.

RESULTS AND DISCUSSION

HCl-Discharge Chamber Textured Stainless Steel 304 Sample

The spectral emittance data in air for textured 304 stainless steel are presented in figure 4 for three different heating durations. The textured stainless steel emittance was first measured in the hohlraum reflectometer with the sample at room temperature. Its emittance was then measured in the emissometer after it was at 630 °C for 1 hr. Finally, after the sample was heated to 630 °C for 4 hr in air, its emittance was remeasured in the hohlraum reflectometer at room temperature. The resulting three spectral emittance curves were close together (they were the same between 0.75 and 0.88 μm) between 2 and 7 μm, but not for the longer wavelengths. The original room temperature emittance at 13 μm was 0.36. The elevated temperature emittance at 13 μm after 1 hr heating increased to 0.55, but the room temperature emittance after 4 hr heating in air increased to a peak value of 0.80 at 13 μm.

The SEM photographs of the sample before and after heating are shown in figure 5. Before heating, the surface consisted of small spires (about 0.2 μm wide) spaced less than 1 μm apart. After heating, the spires on the surface were much larger (about 1 μm wide) and were spaced farther apart (about 2 to 3 μm). During heating, therefore, the small spires coalesced to form a surface with a rougher texture. The increases in the long wavelength emittances could be explained by this change in the surface morphology. The original sample had a low emittance for the longer wavelengths because the dimensions of the surface texture were much smaller than these wavelengths. The emittance for the longer wavelengths increased after heating because the size and spacing of the spires increased.

The spectral emittance taken with the sample in the emissometer at elevated temperatures fell between the two hohlraum reflectometer curves. The emittance remained high for the shorter wavelengths but was found to decrease at wavelengths longer than 10 μm. The data suggests that while the emissometer measurement was being made, the sample morphology was in an intermediate state

in comparison to the room temperature measurements. The sample had been heated for only 1 hr instead of 4 hr, and it is likely that the surface texture had not reached equilibrium at the time of the measurement.

The ESCA results for textured stainless steel are listed in table I. The results indicate that the chemistry of the surface did not change much during exposure to air while at 630 °C.

Untextured Ti (6,4) Sample

In figure 6, the spectral emittance data for a prestine (untextured) Ti (6,4) sample is illustrated. For this material a substantial and permanent increase in room temperature emittance resulted upon heating in air to 630 °C for 1 hr. SEM photographs (fig. 7) revealed that the surface texture became rough as a result of heating. ESCA analysis (table I) shows that the surface was heavily oxidized; the Ti:O ratio had decreased from 0.18 before heating to 0.04 after heating. The presence of oxygen while heating was therefore responsible for the increase in emittance by changing the surface chemistry and morphology.

Sandblasted Ti (6,4) Sample

Figure 8 shows the spectral emittances of sandblasted Ti (6,4) measured at room temperature before and after heating to 630 °C for 1 hr at elevated temperature. Comparison of the initial room temperature spectral emittance of sandblasted Ti (6,4) with that of pristine Ti (6,4) (fig. 6) shows that sandblasting significantly increased the emittance. As with the other samples tested, the room temperature emittance of sandblasted Ti (6,4) increased upon heating in air. The spectral emittance at elevated temperature was slightly below the final room temperature emittance, particularly at the shorter wavelengths.

Figure 8 also shows the elevated temperature spectral emittance of sandblasted Ti (6,4) measured after 1 hr at 630 °C in the argon atmosphere. The measurements were made through the ZnSe window, and hence the spectral emittance must be corrected for the absorption by the window:

$$\varepsilon(\lambda) = \frac{\varepsilon'(\lambda)}{t(\lambda)} \qquad (2)$$

where $\varepsilon'(\lambda)$ was the measured spectral emittance and $t(\lambda)$ was the spectral transmittance of the window. The figure shows that the emittance was low at the shorter wavelengths and high at the longer wavelengths. The short wavelength results appear inconsistent with the initial room temperature measurement. It seems unlikely that there would be such a large difference between the emittance at room temperature and the emittance at 630 °C in an inert gas atmosphere. A possible explanation is that the sample had cooled while the short wavelength emittance was measured, yielding a lower result. Unfortunately, another measurement could not be made to check the results because the emissometer heater failed. The long wavelength emittance, on the other hand, was consistent with the other curves, falling between the original and final room temperature measurements.

The results for the sandblasted Ti (6,4) samples can also be explained by surface analysis. Although the SEM photographs (fig. 9) did not show a change in the surface morphology, the ESCA results (table III) clearly revealed a change in the surface chemistry. As a result of sandblasting with SiC, the sample surfaces were primarily silicon-based compounds instead of titanium-based. The surface of the sample heated in air had the largest amount of SiO_2 relative to SiC, followed by the sample heated in argon, and then by the room temperature sample. For wavelengths between 8 and 15 μm, the emittances of the samples followed the same order. One can conclude that the permanent increase in emittance resulted from the conversion of SiC to SiO_2 at elevated temperatures. Figure 10 shows the elevated temperature spectral emittance measured in air over time, and the reaction appeared to be complete after 1 hr of heating in air.

CONCLUDING REMARKS

Permanent increases in the spectral emittance of the candidate space radiator materials was found to occur as a result of heating the sample of 630 °C in air for 1 hr. The spectral emittance of HCl/ion beam textured stainless steel was unchanged between 2 to 7 μm but increased between 7 to 15 μm. The increase was caused by a rougher surface texture with no apparent change in the surface chemistry. The spectral emittance of untextured Ti (6,4) increased between 2 to 15 μm as a result of heating in air. The surface became heavily oxidized, and also appeared to have a rougher surface texture. The spectral emittance of sandblasted Ti (6,4) also increased between 2 to 15 μm. Again, oxidation was found to be the cause of the increased emittance, even though the surface morphology was not observed to change.

One of the goals of the experiment was to investigate the relationship between room temperature and elevated temperature spectral emittance. After a material was heated to 630 °C in air for 1 hr, the final room temperature spectral emittance was close to the elevated temperature spectral emittance. The two measurements differed by no more than 0.1 emittance units for sandblasted Ti (6,4) between 2 to 15 μm (see fig. 8) and also for textured stainless steel between 2 to 10 μm (see fig. 4). These measurements, however, were performed in air, and heating to 630 °C in air caused a permanent change in the emittance. The purpose for heating the sandblasted Ti (6,4) sample in the inert gas argon was to eliminate the effects of the air and to obtain a true elevated temperature emittance. A large difference between elevated and room temperature emittance for the short wavelengths was found, but this result could not be repeated because the emissometer heater failed.

In the future, it is planned to replace the emissometer attachment with a bell jar system. The new system will allow elevated temperature spectral emittance measurements to be made in vacuum so that there will not be any changes in the sample surface caused by the surrounding atmosphere. Also, various gases could be bled into the chamber so that spectral emittance measurements could be made in any atmosphere. Currently, total hemispherical emittance measurements are being made in a calorimetric vacuum emissometer (CVE). The CVE results could then be compared with the total emittance calculated from the spectral emittance results made in vacuum.

REFERENCES

1. Mirtich, M.J. and Kussmaul, M.T. "Enhanced Thermal Emittance of Space Radiators by Ion-Discharge Chamber Texturing," NASA TM-100137, 1987.

2. Banks, B.A. et al., "Arc-Textured Metal Surfaces for High Thermal Emittance Space Radiators," NASA TM-100894, 1988.

TABLE I. - ESCA AND MORPHOLOGY FOR SAMPLES TESTED

Sample	Surface treatment	Temperature/ atmosphere	ESCA result	Morphology
Ti(1)	None	Room temperature	Ti/O = 0.18	Smooth
Ti(2)	None	630 °C/air	Ti/O ◻ 0.04	Changed
Ti85C	Sandblasted	Room temperature	Si, SiC, some SiO_2: SiC/SiO_2 = 2.7	Unchanged
Ti25A	Sandblasted	630 °C/air	Mostly SiO_2, some SiC, Si: SiC/SiO_2 = 1.1	Unchanged
Ti56AR	Sandblasted	630 °C/air	Si, SiC, moderate SiO_2: SiC/SiO_2 = 1.9	Unchanged
S.S. 304	HCl discharge chamber texture	Room temperature	C, Fe, Cr, 4 percent Ta: all in oxidized states	Spires 1 μm
S.S. 304	HCl discharge chamber texture	630 °C/air	C, Fe, Cr, 4 percent Ta: all in oxidized states	Spires 2 to 3 μm

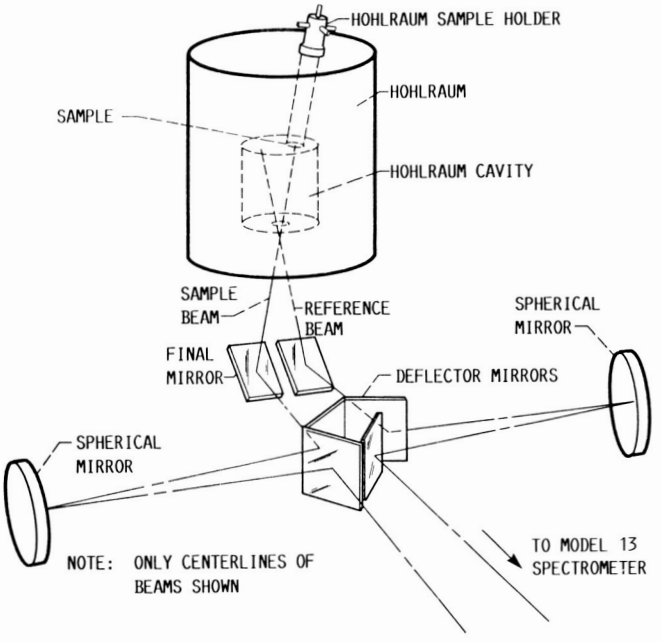


FIGURE 1. - REFLECTIVITY MEASUREMENT OPTICAL PATH TO SPECTROMETER.

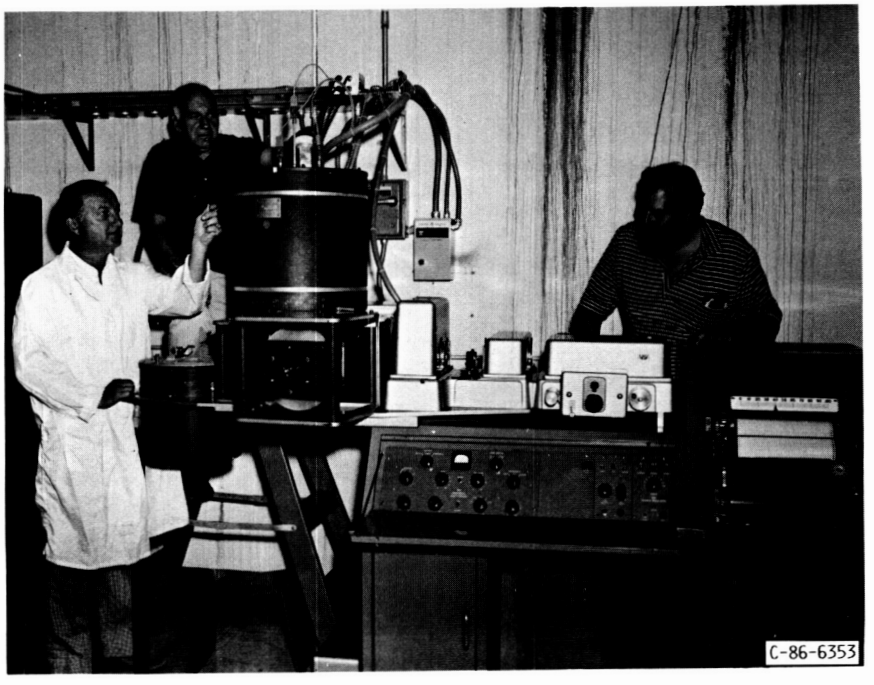


FIGURE 2. - HOHLRAUM REFLECTOMETER-EMISSOMETER.

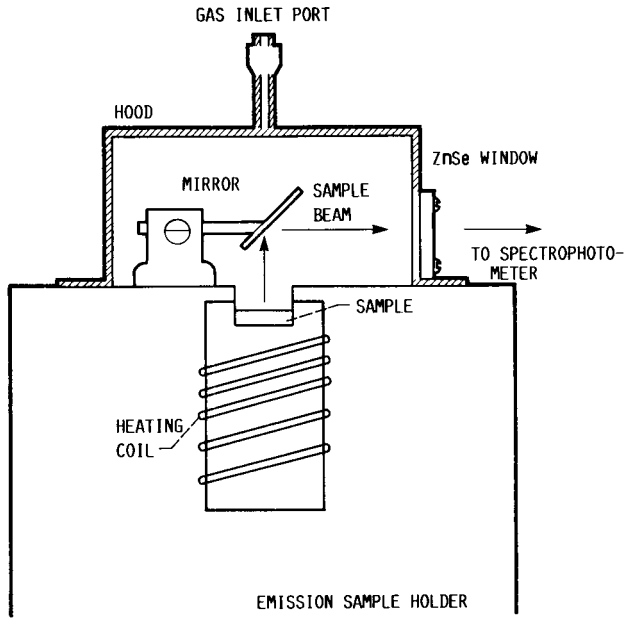


FIGURE 3. - SCHEMATIC OF HOOD AND HEATED SAMPLE IN EMISSOMETER.

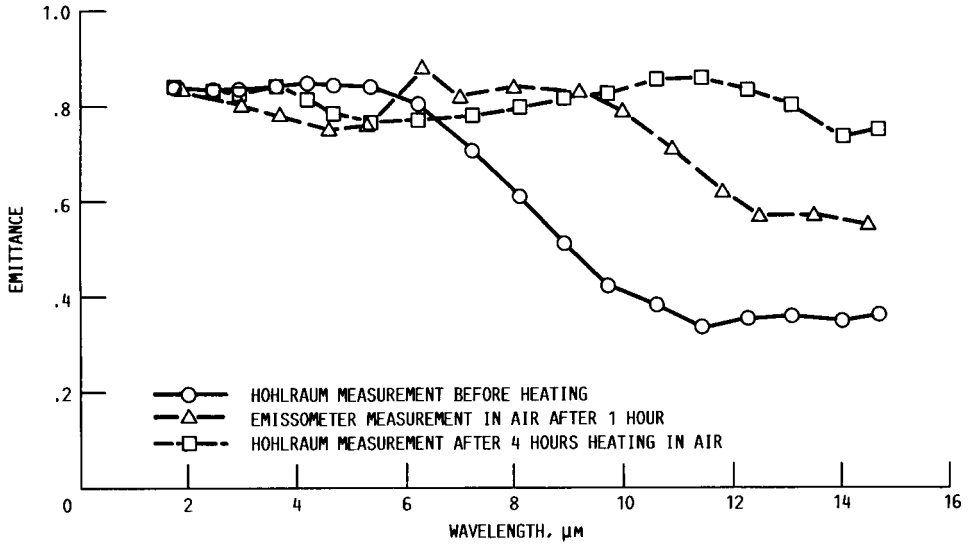
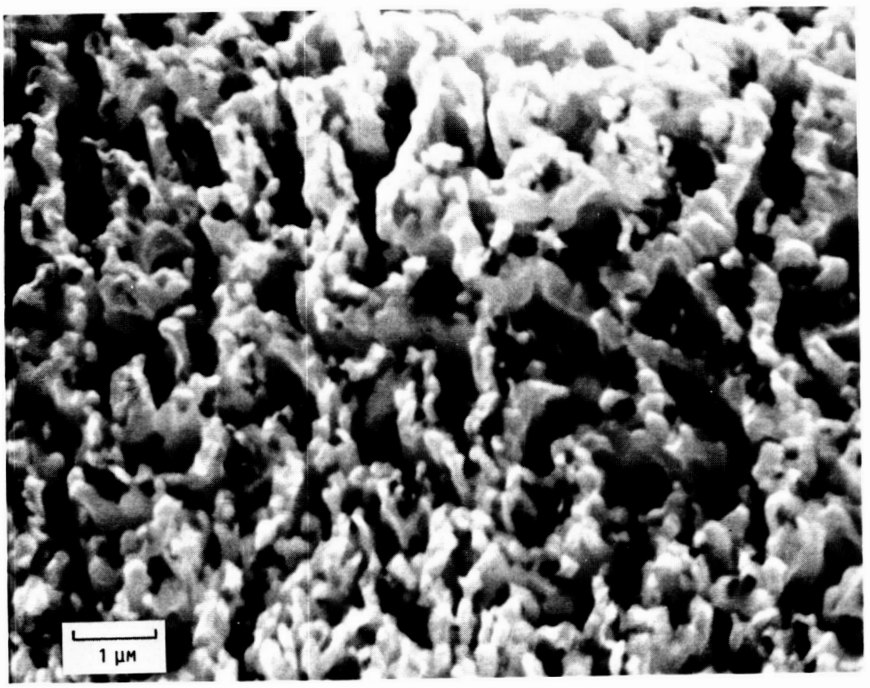
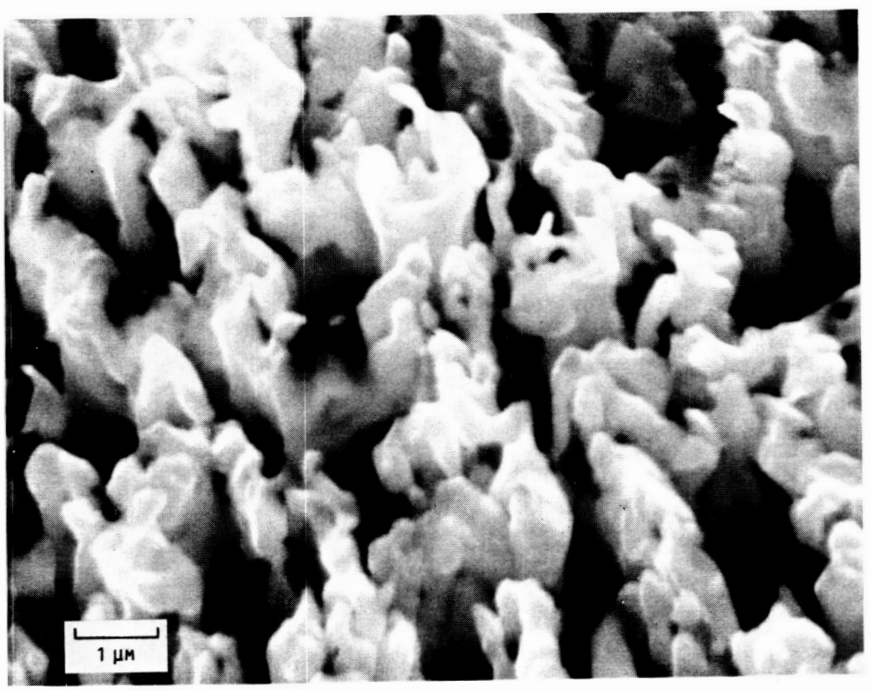


FIGURE 4. - SPECTRAL EMITTANCE OF TEXTURED STAINLESS STEEL.



(A) BEFORE HEATING.



(B) AFTER HEATING IN AIR FOR 4 HOURS.

FIGURE 5. - SEM MICROGRAPHS OF TEXTURED STAINLESS STEEL BEFORE AND AFTER HEATING.

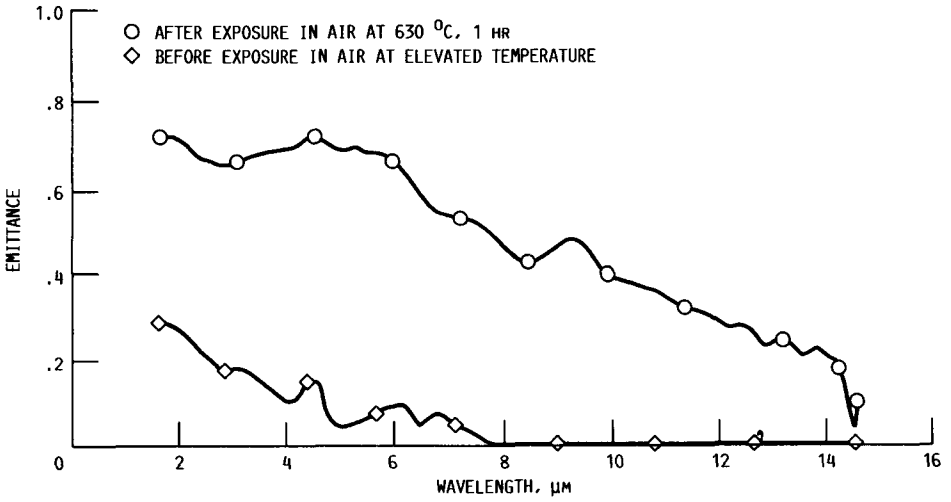
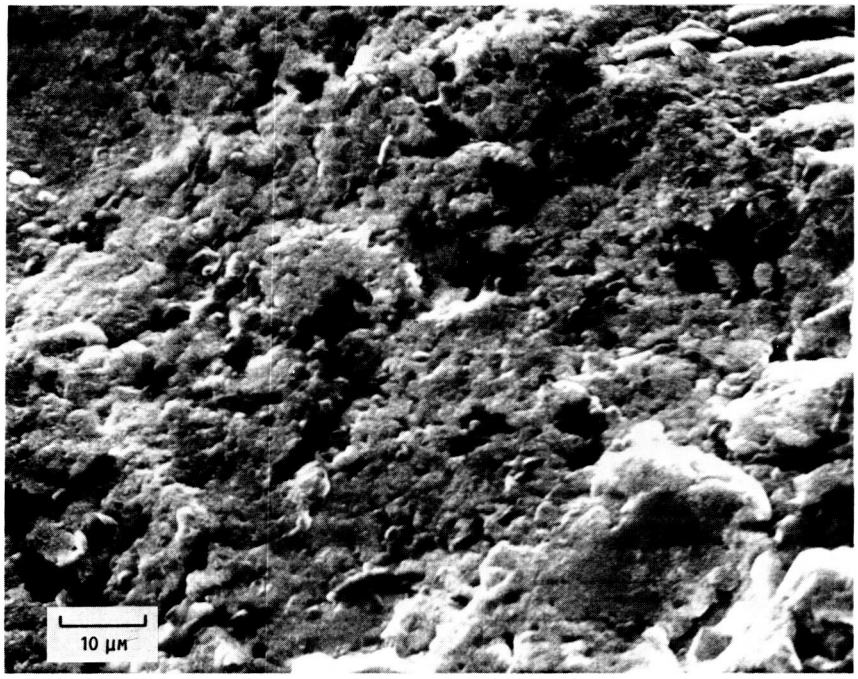
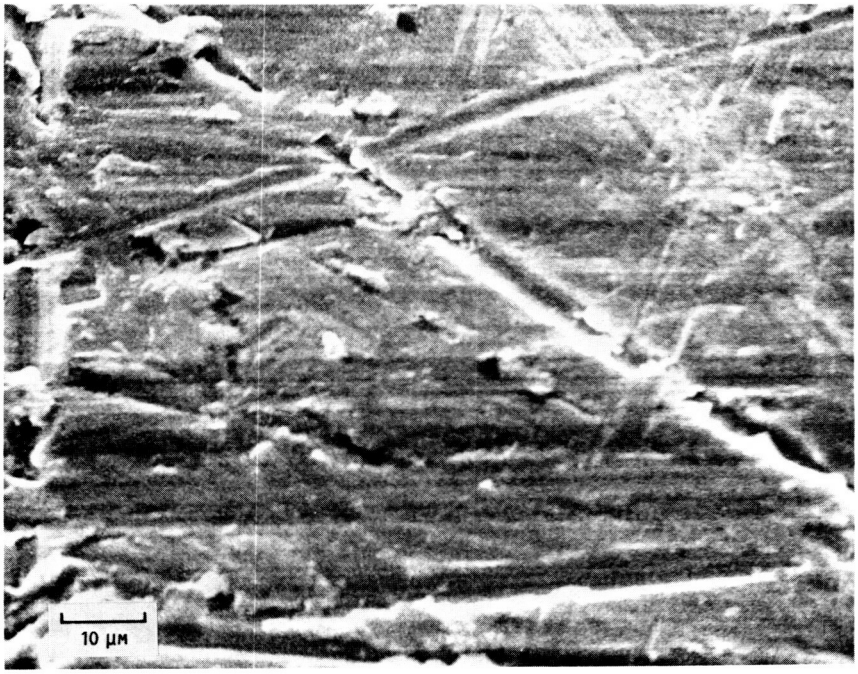


FIGURE 6. - EMITTANCE OF PRISTINE Ti(6,4) MEASURED IN THE HOHLRAUM REFLECTOMETER.



(A) TITANIUM AFTER EXPOSURE IN AIR AT ELEVATED TEMPERATURE, 630 ^{0}C.



(B) UNTREATED TITANIUM (6,4).

FIGURE 7. - SEM PHOTOGRAPHS OF PRISTINE TITANIUM BEFORE AND AFTER HEATING IN AIR AT 630 ^{0}C.

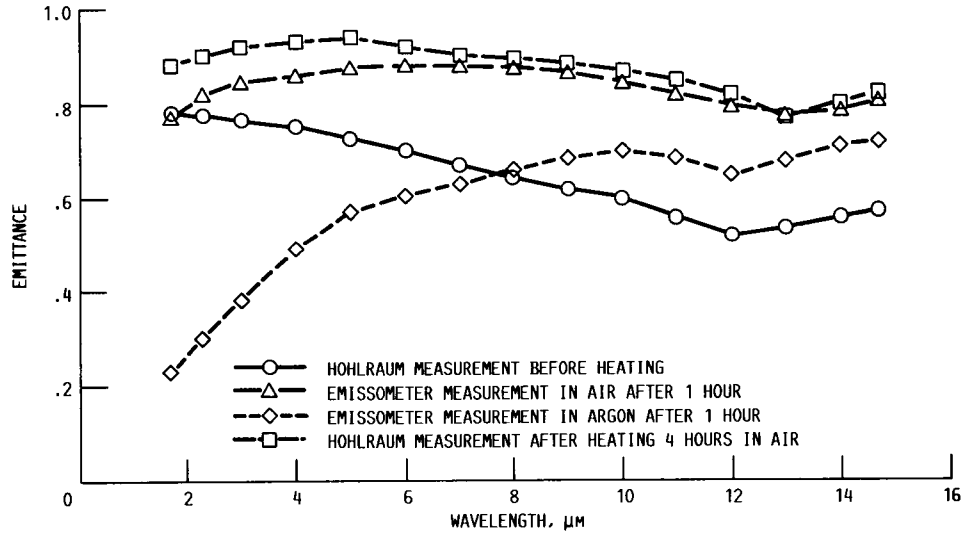
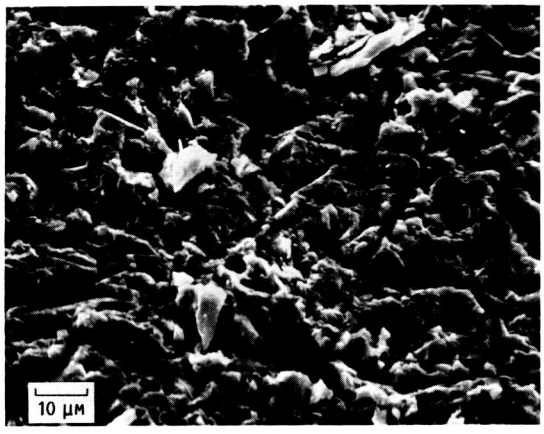
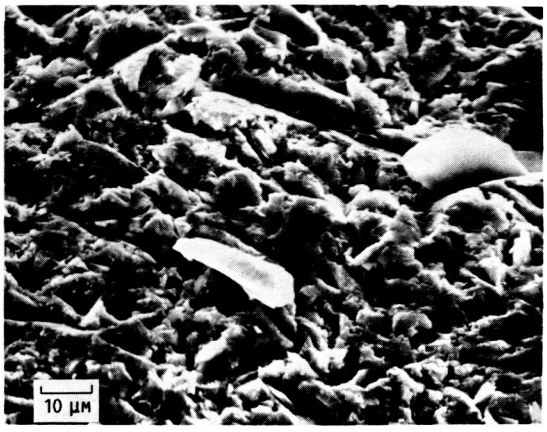


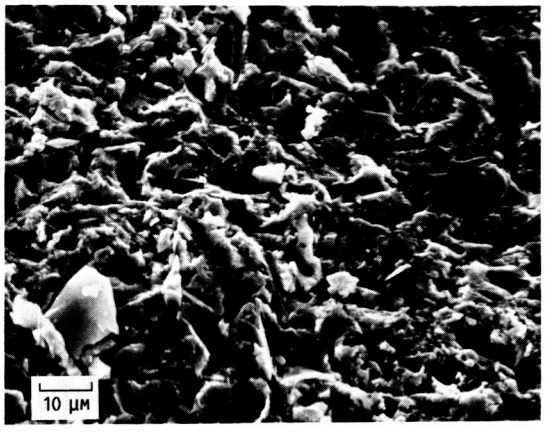
FIGURE 8. - SPECTRAL EMITTANCE OF SANDBLASTED Ti (6,4).



(A) Ti 85 ^{o}C (ROOM TEMPERATURE).



(B) Ti 25A (630 ^{o}C IN AIR).



(C) Ti 56AR (630 ^{o}C IN ARGON).

FIGURE 9. - SEM PHOTOGRAPHS FOR SANDBLASTED Ti(6,4).

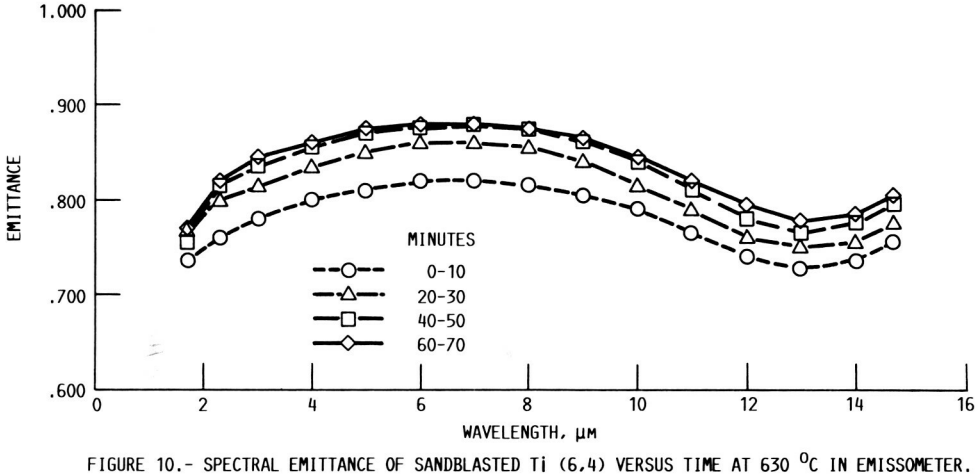


FIGURE 10.- SPECTRAL EMITTANCE OF SANDBLASTED Ti (6,4) VERSUS TIME AT 630 ^{o}C IN EMISSOMETER.

NASA
National Aeronautics and Space Administration

Report Documentation Page

1. Report No.	2. Government Accession No.	3. Recipient's Catalog No.
NASA TM-101948		

4. Title and Subtitle

The Emittance of Space Radiator Materials Measured at Elevated Temperatures

5. Report Date

6. Performing Organization Code

7. Author(s)

Michael J. Mirtich, Frank DiFilippo, Jennifer Barry, and Michael Kussmaul

8. Performing Organization Report No.

E-4641

10. Work Unit No.

586-01-11

9. Performing Organization Name and Address

National Aeronautics and Space Administration
Lewis Research Center
Cleveland, Ohio 44135-3191

11. Contract or Grant No.

13. Type of Report and Period Covered

Technical Memorandum

12. Sponsoring Agency Name and Address

National Aeronautics and Space Administration
Washington, D.C. 20546-0001

14. Sponsoring Agency Code

15. Supplementary Notes

Presented at the 15th International Conference on Metallurgical Coatings sponsored by the American Vacuum Society, San Diego, California, April 11-15, 1988. Michael J. Mirtich, NASA Lewis Research Center; Frank DiFilippo, Case Western Reserve University, Cleveland, Ohio 44106; Jennifer Barry and Michael Kussmaul, Cleveland State University, Cleveland, Ohio 44115.

16. Abstract

The spectral emittances of textured space radiator materials between 1.7 and 14.7 μm have been evaluated at room temperature and elevated temperature (630 °C) in air. Heating in air caused a permanent increase in spectral emittance for all materials tested: HCl/ion beam textured 304 stainless steel, untextured Ti (6 percent Al, 4 percent V), and sandblasted Ti (6 percent Al, 4 percent V). Changes in the surface chemistry and/or surface morphology of these materials were also observed. Elevated temperature spectral emittance was measured in an argon atmosphere and compared to the measurements in air. Similarity between the room temperature and elevated temperature spectral emittance measurements was also investigated, and limited agreement was found.

17. Key Words (Suggested by Author(s))	18. Distribution Statement
Radiators Emittance of materials	Unclassified – Unlimited Subject Category 20

19. Security Classif. (of this report)	20. Security Classif. (of this page)	21. No of pages	22. Price*
Unclassified	Unclassified	14	A03

*For sale by the National Technical Information Service, Springfield, Virginia 22161