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# Thermal Optical Properties of Plasma-Sprayed Mullite Coatings for Space Launch Vehicles

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**Thermal optical properties, for example, solar absorptance $\alpha$ and thermal emittance $\varepsilon$ of thin plasma sprayed mullite coatings, were determined to assess the material's suitability for space application. The mullite coatings were developed in an attempt to find a new material for thermal protection systems as well as thermal barrier coatings for atmospheric reentry vehicles. All mullite coatings show the properties of a solar reflector and hence suggest a potential application as thermal control coating to protect space-bound structures from the effect of heating by solar radiation in the low Earth orbit. Preliminary space stability tests including atomic-oxygen resistance tests confirmed that the coatings show only negligible variations both in their mechanical and thermal optical properties under the conditions selected.**

## I. Introduction

SPACE structures and spacecrafts are being exposed to a multitude of deleterious environmental effects quite different from the environment prevailing at the Earth's surface. In particular, owing to the absence in outer space of a protective atmosphere, the spacecraft will be exposed to the full strength of the solar UV radiation as well as to other external sources of energy, such as Earth-emitted infrared (IR) and albedo (planetary reflection).[1,2] Thus, the thermal control of a spacecraft becomes very crucial. However, this thermal control is a basic requirement for the integrity of all components and subsystems of the spacecraft. Hence to maintain nominal temperatures of all different components onboard, the spacecraft must be equipped with a suitable thermal control system. One important part of this thermal control system is the external surface of the space structure that is prominently characterized by its thermal optical properties. The thermal optical properties, for example, thermal (IR) emittance $\varepsilon$ and solar absorptance $\alpha$ indicate how the material's surface handles the thermal energy supplied by different sources.[3] There are four basic categories of thermal control surfaces: solar reflectors, solar absorbers, flat reflectors, and flat absorbers (Fig. 1) that are defined as follows[3,4]:

1) Solar reflector is a surface reflecting the incident solar energy while absorbing and emitting IR energy (low $\alpha$, high $\varepsilon$, very low $\alpha/\varepsilon$ ratio $< 1$).

2) Solar absorber is a surface absorbing solar energy while emitting only a small percentage of the IR energy (high $\alpha$, low $\varepsilon$, very high $\alpha/\varepsilon$ ratio $> 1$).

3) Flat reflector is a surface reflecting the energy incident upon it throughout the spectral range from UV to far IR regions (low $\alpha$, low $\varepsilon$, and $\alpha/\varepsilon$ ratio $= 1$).

4) Flat absorber is a surface absorbing the energy incident upon it throughout the spectral range from UV to far IR regions (high $\alpha$, high $\varepsilon$, $\alpha/\varepsilon$ ratio $= 1$).

This study is part of the research project "Advanced Ceramic Coatings for Space Construction Materials," jointly sponsored by the German Federal Ministry of Education, Research, Science and Technology (BMBF), and the National Research Council of Canada under the auspices of the Bilateral German–Canadian Agreement on Cooperation in Science and Technology (WTZ; project CAN 02/010). Thin (10–20 $\mu$m) mullite ($2Al_2O_3 \cdot SiO_2$) coatings have been developed in an attempt to find a new material for thermal protection systems (TPS) as well as thermal barrier coatings (TBC) for application in space.[5] The mullite coatings were deposited onto aluminum substrates by atmospheric plasma spraying (APS).[6]

There is previous work describing the application by plasma-spray technique of mullite coatings to various substrates, preferentially silicon carbide to control oxidation at high temperatures.[7–12] In this presentation we report on the deposition of mullite on aluminium substrates to establish whether very thin thermally sprayed mullite coatings would be suitable to provide sufficient thermal barrier functions to space-bound construction materials in an Earth orbit as well as to protect the surface of atmospheric reentry vehicles from thermal damage and ablation during the reentry phase.[13–15] Because the thermal optical properties are basic parameters for characterizing the selective nature of various coatings, they are of primary interest for the development of new external or internal coatings. In the present work, the thermal optical properties of various mullite coatings were measured to compare the values with those of aluminium.

## II. Experimental Methods

The starting material used for the present investigation was a fused 2:1 mullite powder ($2Al_2O_3 \cdot SiO_2$; Treibacher Schleifmittel Zschornewitz GmbH, Germany) supplied with two different grain size distributions (fine particles, <89 $\mu$m; average grain size 23.4 $\mu$m and coarse particles, <136 $\mu$m; average grain size 50.6 $\mu$m). The powder consisted of 87 mass% mullite, 3 mass% corundum, and 10 mass% alumina-silica glass. The mullite powder was deposited by APS onto aluminum substrates ($50 \times 20 \times 2$ mm) to obtain thin ceramic coatings. Various plasma-spray parameters were applied to produce eight different sample types for each powder grain size. In addition, two samples were precoated with an 88Al/12Si powder (<45 $\mu$m; Sulzer Metco) supposed to

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**Table 1 Plasma-spray parameters**

| Spray parameter[a] | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | F | 88Al/12Si |
|---|---|---|---|---|---|---|---|---|---|---|
| Ar, slpm | 45 | 45 | 45 | 45 | 45 | 40 | 45 | 45 | 45 | 30 |
| $H_2$, slpm | 5 | 5 | 5 | 5 | 5 | 10 | 5 | 5 | 5 | 1.5 |
| PG, slpm | 3 | 3 | 3 | 3 | 3 | 3 | 3 | 3 | 3 | 3 |
| $T_R$, % | 20 | 30 | 40 | 20 | 20 | 20 | 20 | 20 | 20 | 8 |
| $R$, % | 40 | 40 | 40 | 40 | 40 | 40 | 40 | 40 | 40 | —— |
| $l_s$, mm | 100 | 100 | 100 | 120 | 80 | 100 | 100 | 100 | 100 | 120 |
| $U$, V | 55 | 55 | 55 | 53 | 54 | 59 | 55 | 55 | 55 | —— |
| $I$, A | 400 | 400 | 400 | 400 | 400 | 500 | 400 | 400 | 400 | —— |
| $P$, kW | 22 | 22 | 22 | 21.2 | 21.5 | 29.5 | 22 | 22 | 22 | 9.5 |
| Number of traverses | 1 | 1 | 1 | 1 | 1 | 1 | 2 | 1 | 1 | 1 |
| $v$, m/min | 6 | 6 | 6 | 6 | 6 | 6 | 6 | 4 | 6 | 6 |

[a]slpm, Standard liters per minute; PG, powder carrier gas; $T_R$, rotation of powder feeder plate; $R$, rotation of powder feeder stirrer; $l_s$, spraying distance; $U$, voltage; $I$, current; $P$, plasma power; and $v$, traverse speed.

**Table 2 Values of thermal optical properties ($\alpha$ obtained from one measurement, $\varepsilon$ average of three measurements)**

| Sample (coarse) | $\alpha$ | $\varepsilon$ | $\alpha/\varepsilon$ | Sample (fine) | $\alpha$ | $\varepsilon$ | $\alpha/\varepsilon$ |
|---|---|---|---|---|---|---|---|
| Al uncoated | 0.232 | 0.042 | 5.50 | Al grit blasted | 0.497 | 0.250 | 1.99 |
| 1 | 0.522 | 0.827 | 0.63 | 1a | 0.532 | 0.784 | 0.68 |
| 2 | 0.519 | 0.823 | 0.63 | 2a | 0.492 | 0.796 | 0.62 |
| 3 | 0.444 | 0.835 | 0.53 | 3a | 0.463 | 0.652 | 0.71 |
| 4 | 0.500 | 0.789 | 0.63 | 4a | 0.505 | 0.636 | 0.79 |
| 5 | 0.549 | 0.825 | 0.67 | 5a | 0.495 | 0.755 | 0.66 |
| 6 | 0.480 | 0.843 | 0.57 | 6a | 0.469 | 0.691 | 0.68 |
| 7 | 0.449 | 0.843 | 0.53 | 7a | 0.476 | 0.767 | 0.62 |
| 8 | 0.484 | 0.822 | 0.59 | 8a | 0.480 | 0.796 | 0.60 |
| F | 0.615 | 0.833 | 0.74 | Fa | 0.616 | 0.806 | 0.76 |

act as a bond coat and subsequently covered with mullite powder (see Table 1, sample F). For deposition, a PT-M1000 system (Plasmatechnik Wohlen, Switzerland) was used in conjunction with an F4 Plasmatron [Sulzer Metco (Deutschland) GmbH]. Before deposition the aluminum substrates were grit blasted with corundum ($Al_2O_3$) to roughen the surface for better coating adhesion.

The arithmetic mean surface roughness $R_a$ of the plasma-sprayed coatings was measured with a diamond-stylus tester (Surftest 301; Mitutoyo Corp., Japan) and evaluated according to DIN 4772 designation.[16]

All coatings as well as some uncoated substrates were subjected to measurements of the thermal optical parameters, that is, solar absorptance $\alpha$ and thermal emittance $\varepsilon$. The solar absorptance was indirectly determined by the measurement of the reflectance of a surface in different ranges of the spectrum (UV, visible, near IR) by an integrating sphere[17] using a Beckman DK-2A Ratio Recording Spectroreflectometer. The final calculation of the solar absorptance $\alpha$ was based on the ASTM E 903-96 designation[18] using standard data from ASTM E 490-00a (Ref. 19).

The thermal emittance of the coatings was measured using an Infrared Reflectometer Model DB 100 (Gier-Dunkel Instruments). This method measures radiant energy reflected from the specimen.[20] Finally, the thermal emittance was determined by subtracting the measured reflectance from unity.

To provide some preliminary evidence of the space stability of the 2:1-mullite coatings, selected samples were exposed to a cold plasma environment provided by an inductively coupled radio-frequency plasma generator operated at 13.56 MHz (Ref. 21). The effective atomic-oxygen (AO) fluxes and fluences, respectively, were calculated according to ASTM E 2089 designation[22] using a Kapton™ control surface with an assumed erosion yield of $3 \times 10^{-24}$ cm$^3$/atom oxygen.[23]

## III. Results and Discussion

The solar absorptance was measured only once per sample. However, one sample was measured five times to assess the precision of the measurement. The resulting standard deviation was only 0.004.

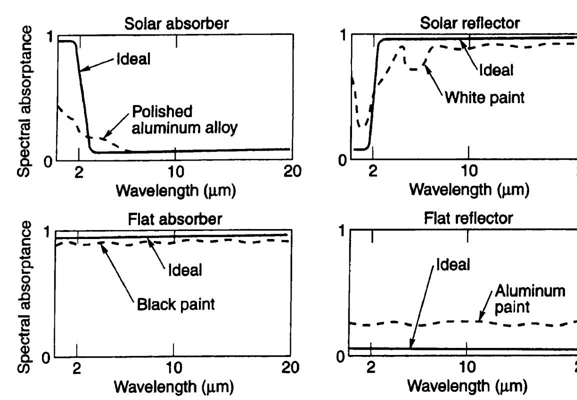


**Fig. 1 Ideal representation of the four basic passive-control surfaces.[3]**

Thus, all other measurements are expected to be within slight variations as well. To determine the thermal emittance, three measurements for each sample were taken, and the mean value was calculated. In addition, the standard deviations that range for all samples from 0.001 to 0.020 were calculated. The mean values of the solar absorptance $\alpha$, the thermal emittance $\varepsilon$, and their ratios $\alpha/\varepsilon$ are given in Table 2.

From Table 2 it is evident that uncoated aluminum represents a typical solar absorber, which is in accord with values from the literature.[3] Consequently, uncoated aluminum absorbs more thermal energy from solar radiation than it can emit back into space. The grit-blasted aluminum substrate represents also a solar absorber even though the values for $\alpha$ and $\varepsilon$ are both higher than the values of the uncoated smooth substrate. Furthermore, it can be observed that all mullite coatings have a very high thermal emittance and a moderate solar absorptance, which results in a $\alpha/\varepsilon$ ratio less than 1. Thus, the mullite coatings change the original properties of the substrate into that of a solar reflector (Fig. 1). In contrast to the solar absorber, the latter reflects solar energy while absorbing and reemitting the thermal energy back into space.

**Table 3 Values of the average surface roughness $R_a$**

| Sample (coarse powder) | $R_a$, $\mu$m | Sample (fine powder) | $R_a$, $\mu$m |
|---|---|---|---|
| Al | $4.39 \pm 0.22$ | Al | $4.39 \pm 0.22$ |
| 1 | $7.35 \pm 0.42$ | 1a | $5.59 \pm 0.07$ |
| 2 | $7.05 \pm 0.38$ | 2a | $5.97 \pm 0.68$ |
| 3 | $7.35 \pm 0.42$ | 3a | $5.60 \pm 0.46$ |
| 4 | $6.45 \pm 0.43$ | 4a | $5.87 \pm 0.15$ |
| 5 | $6.87 \pm 0.57$ | 5a | $6.28 \pm 0.13$ |
| 6 | $8.10 \pm 0.60$ | 6a | $5.98 \pm 0.29$ |
| 7 | $7.16 \pm 0.30$ | 7a | $6.03 \pm 0.51$ |
| 8 | $6.54 \pm 0.59$ | 8a | $6.16 \pm 0.19$ |
| F | $7.70 \pm 0.70$ | Fa | $6.56 \pm 0.37$ |

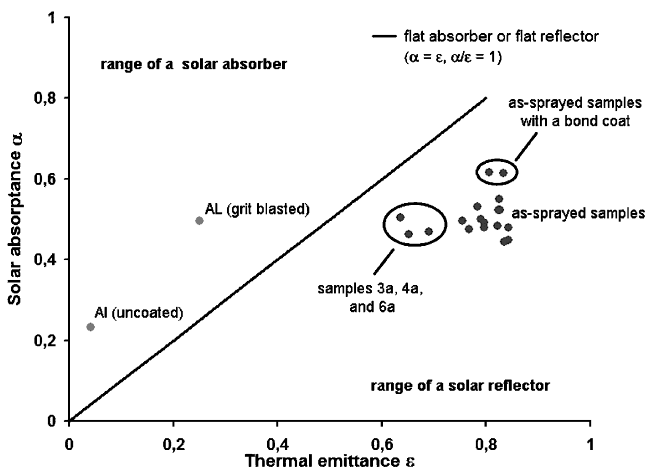


**Fig. 2 Correlation between solar absorptance $\alpha$ and thermal emittance $\varepsilon$. The line $\alpha = \varepsilon$ illustrates a flat absorber and flat reflector, respectively.[3]**

Figure 2 shows a graphical representation of the measured values for $\alpha$ and $\varepsilon$ of all samples classified according to the basic categories. The line dividing the diagram separates the range of a solar absorber from the range of a solar reflector. From this figure, it is obvious that the optical properties of aluminum clearly differ from those of the mullite coatings. The measured optical properties of aluminum are similar to those of a solar absorber, and the optical properties of the mullite coating are similar to those of a solar reflector.

The variation of the solar absorptance of all as-sprayed samples is negligible. Only the samples with an intermediate 88Al/12Si layer ($F$, $F_a$) hold slightly higher $\alpha$ values. The thermal emittance shows also nearly uniform values for most as-sprayed coatings with the exception of the samples 3a, 4a, and 6a that exhibit somewhat lower values suggesting an influence of the aluminum substrate. Although the substrates of samples 3a, 4a, and 6a were completely covered by the coating, there were some areas where the coating flaked off during the cooling process or where the coating might be very thin. Hence, the aluminum substrate shone through the coating shifting the thermal emittance toward lower values and affecting also the $\alpha/\varepsilon$ ratio.

Futhermore, the optical parameters, in particular the thermal emittance, are a function of the surface roughness. This is evident from the direct comparison between the values of an untreated aluminum substrate and a grit-blasted aluminum substrate with an average roughness of 4.39 $\mu$m. The grit-blasted aluminum substrate shows a higher solar absorptance and a higher thermal emittance than the uncoated smooth aluminum substrate (Table 2). It is obvious that with increasing surface roughness the thermal emittance increases (Fig. 3), whereas the ratio of $\alpha/\varepsilon$ decreases. Table 3 shows the values of the average roughness $R_a$, ranging from 5.6 to 8.1 $\mu$m. The samples prepared with the fine powder show a slightly lower average surface roughness ($6.00 \pm 0.09$ $\mu$m) as well as a slightly lower thermal emittance $\varepsilon$ than the samples sprayed with coarse powder ($7.17 \pm 0.28$ $\mu$m).

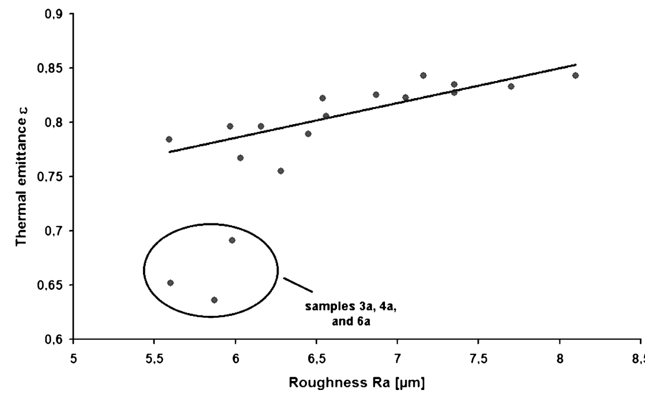


**Fig. 3 Thermal emittance $\varepsilon$ as function of the average surface roughness $R_a$.**

Figure 3 shows the thermal emittance $\varepsilon$ as function of the average surface roughness $R_a$ that follows the linear equation $\varepsilon = 0.03(2) \cdot R_a + 0.59(1)$ with a correlation coefficient $r = 0.82$. The three samples 3a, 4a, and 6a with significantly lower $\varepsilon$ values vary from this trend (lower ellipse).

Comparable values of the thermal emittance of around 0.80 were found for $86Al_2O_313ZrO_21Y_2O_3$ thermal control coatings developed by Lockheed Martin Vought Systems Corp.[24] and applied to Al 6061 and C/C composite substrates. However, in this case the solar absorptance was much lower with 0.14–0.19 at beginning of life and increased to 0.30–0.31 at end of life after 1000 ESH (equivalent sun hours). Lifetime was defined by a space stability AO resistance test with an accelerated AO flux of $3 \times 10^{15}$ atoms/cm$^2$s (total fluence $10^{21}$ atoms/cm$^2$). These coatings were also designed to be typical solar reflector coatings ($\alpha/\varepsilon = 0.21$–$0.33$) for high thermal conductivity substrates, in particular carbon/carbon honeycomb radiator panels.

In the present study selected mullite coatings on aluminum substrates were exposed to a cold plasma for 6 h to simulate outer-space conditions. The effective AO flux was determined from the specific mass loss (mass loss rate per exposed unit surface area) of Kapton™ and further used to calculate the ETS (equivalent time in space) according to Tennyson.[25] The ETS varied between 4000 ESH (equivalent sun hours) at an effective accelerated solar maximum AO flux of $2 \times 10^{13}$ atoms/cm$^2$s at 8-km/s spacecraft speed at an altitude of 800 km [upper low Earth orbit (LEO)] and 22 ESH at a corresponding AO flux of $5.5 \times 10^{15}$ atoms/cm$^2$s at an altitude of 200 km (lower LEO). Measurements of the solar absorptance $\alpha$ and the thermal emittance $\varepsilon$ before and after treatment with AO did not show differences other than those attributable to statistical error.[5] Hence it can be safely assumed that the coatings appear to be environmentally stable under the conditions indicated. However, during prolonged exposure of the coatings to AO erosion might occur that will wear away the rather thin coating to a point when the thermal properties will noticeably degrade. In this case the integrity of the thermal control coating will be compromised. To ensure spacecraft designers that coating degradation time is beyond the lifetime of the mission, much longer lasting AO stability tests are required, for example, space-bound tests in a LEO.

## IV. Summary

Solar absorptance $\alpha$ and thermal emittance $\varepsilon$ of thin mullite coatings were measured to evaluate their suitability for space applications. Thin coatings were prepared by APS of a fine fused 2:1 mullite powder with two different powder grain sizes onto aluminum substrates. Some substrates were precoated with an 88Al/12Si powder as a bond coat and subsequently covered with a mullite coating. All coatings measured show thermal optical properties in the range of a solar reflector and hence change the original thermal optical properties of the aluminum substrate from that of a solar absorber to that of a solar reflector such as second-surface mirrors, white paints, and silver- or aluminum-backed poly(tetrafluoroethylene). In contrast to metallic finishes such as aluminum, the solar reflectors are generally used to minimize absorbed solar energy and at the same time emit energy akin to blackbody radiation.

Furthermore, it is evident that the optical properties, in particular the thermal emittance $\varepsilon$, will be influenced by the surface roughness of the coatings as well as of the substrate. The thermal emittance increases linearly with increasing surface roughness, whereas the ratio of $\alpha/\varepsilon$ decreases. Thus, the thermal optical properties are slightly dependent on the powder grain size of the mullite powder used.

Owing to the property of a solar reflector, the mullite coatings are thought to be suitable for applications on large exterior surfaces of space structures in a LEO. Hence, in addition to the originally anticipated application as TBC as well as TPS for reentry space vehicles, mullite coatings could also potentially be applied as thermal control coating of solar reflector type to protect space-bound structures from the effect of heating by solar radiation in a LEO as a result of their very low thermal conductivity around 0.1–0.2 W/mK (Ref. 15).

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