The invention disclosed in this document resulted from research in aeronautical and space activities performed under programs of the National Aeronautics and Space Administration. The invention is owned by NASA and is, therefore, available for licensing in accordance with the NASA Patent Licensing Regulation (14 Code of Federal Regulations 1245.2).

To encourage commercial utilization of NASA-owned inventions, it is NASA policy to grant licenses to commercial concerns. Although NASA encourages nonexclusive licensing to promote competition and achieve the widest possible utilization, NASA will consider the granting of a limited exclusive license, pursuant to the NASA Patent Licensing Regulations, when such a license will provide the necessary incentive to the licensee to achieve early practical application of the invention.

Address inquiries and all applications for license for this invention to NASA Patent Counsel, Lewis Research Center, Mail Stop LE-LAW, 21000 Brookpark Road, Cleveland, OH 44135. Approved NASA forms for application for nonexclusive or exclusive license are available from the above address.

Serial No.: 699,130
Filed Date: 5/13/91

LeRC
CERAMIC COATINGS ON SMOOTH SURFACES

A metallic coating is plasma sprayed onto a smooth surface of a metal alloy substrate, or on a bond coating. An initial thin ceramic layer is low pressure plasma sprayed onto the smooth surface of the substrate or bond coating. Another ceramic layer is atmospheric plasma sprayed onto the initial ceramic layer.
CERAMIC COATINGS ON SMOOTH SURFACES

Origin of the Invention
The invention described herein was made by employees of the United States Government and may be manufactured and used by and for the Government for governmental purposes without the payment of any royalties thereon or therefor.

Technical Field
This invention is concerned with applying durable coatings onto smooth surfaces. The invention is particularly concerned with plasma sprayed ceramic coatings, such as zirconia-yttria based thermal barrier coatings.

Components operating in the hot gas paths of advanced turbine engines normally must be coated. The coating is conventionally a metallic "overlay" which may be applied by any of a variety of processes including pack aluminizing, electron beam-physical vapor deposition (EB-PVD), and low pressure plasma spraying. Engine designers frequently specify thermal barrier coating systems instead of overlay coatings. These coating systems normally comprise an insulating ceramic material applied over an oxidation resistant metallic bond coat. The ceramic material is usually applied by atmospheric pressure plasma spraying (APPS) or by EB-PVD. Plasma sprayed ceramic coatings require rough metal/ceramic interfaces. The APPS ceramics are generally applied over a rough low pressure plasma sprayed bond coat which typically has a roughness of 300 micro-inch or greater. APPS bond coats may be utilized for less demanding applications. The EB-PVD ceramics are applied over smooth pack aluminide or EB-PVD bond coats. Alternately, the smooth bond coats may be obtained by grinding low pressure plasma sprayed coatings to a roughness of about 100 micro-inch.

While a variety of factors may influence bond coat selection, one practical approach is to select the original overlay as the bond coat layer for the thermal barrier system. This enables the proven technology of the overlay to be retained while adding the insulation of the ceramic material. This approach may favor the selection of EB-PVD ceramics inasmuch as the EB-PVD ceramics can be applied to any type of
bond coat as long as the bond coat is either smooth or can be ground to be smooth as pointed out above. The disadvantage of the EB-PVD ceramic coatings is that they do not insulate as well as plasma sprayed ceramic coatings.

Prior Art

Traditional APPS ceramic coatings are only adherent and durable if applied over a rough-plasma sprayed metallic bond coat. As a result, traditional APPS ceramic coatings cannot be used if a designer wishes to retain a pack aluminide or a EB-PVD overlay coating.

It is, therefore, an object of the present invention to provide a plasma sprayed ceramic coating on a smooth surface thereby removing the traditional requirement for a rough metal-ceramic interface.

A further object of the invention is to improve coating durability by eliminating the stress concentration at roughness asperities.

Another object of the invention is to provide a process for preparing plasma sprayed ceramic coatings which are well bonded to smooth bond coats or overlays.

Background Art

U.S. patent Nos. 4,382,976, 4,576,874 and 4,861,618 disclose processes of plasma spraying MCrAlY and ceramics onto base materials. These coatings improve the thermal or corrosive properties of the end product.

U.S. patent No. 4,623,555 is directed to the general concept of plasma spraying various layers on top of one another onto a base material. U.S. patent No. 4,666,733 is concerned with the broad concept of low pressure plasma spraying (LPPS) for depositing MCrAlY/Cr₃C₂ compositions on a metallic surface.

Disclosure of the Invention

The process of the present invention comprises applying an initial thin layer of low pressure plasma sprayed ceramic to a smooth bond coat followed by a layer of conventional, low thermal conductivity atmospheric pressure plasma sprayed ceramic for good insulation. The ceramic coating is preferably a zirconia-yttria ceramic. It is contemplated that the yttria component may be replaced by another oxide such as
ytterbium, ceria, magnesia or calcia. Also the zirconia component may be replaced by hafnia. Other oxides such as alumina, titania, chromia, calcium silicate or combinations of these materials may replace the zirconia-or yttria-based oxides.

The smooth bond coat may be of the MCrAlX family of alloys where M is Ni, Co, Fe or combinations thereof and X may be an active element such as yttrium or ytterbium. Other bond coats such as NiAl, NiCr, pack aluminide coatings or no bond coat, such as with direct application of the ceramic to a metallic substrate, may also be employed.

**Brief Description of the Drawing**

The foregoing, as well as other objects, features, and advantages of the invention will become more apparent from the following detailed description when taken in conjunction with the drawings in which;

- FIG. 1 is a graph illustrating increases in the life of smooth interface thermal barrier coatings produced in accordance with the present invention, and
- FIG. 2 is a bar graph showing the test lives for specimens treated according to the schedules shown in TABLE 1.

**Best Mode for Carrying Out the Invention**

Specimens of 0.5 inch thick solid nickel-based superalloy substrates commercially available as Waspaloy were provided with a substantially smooth surface having a roughness less than about 120 microinch Ra. A NiCrAlYb bond coat was low pressure plasma sprayed onto this smooth surface to a thickness of approximately 6 mils. Metallographic analysis of these specimens revealed that the coatings were less than optimum quality because of excessive porosity.

Conventional thermal barrier coating systems were prepared by atmospheric pressure plasma spraying 10 mils of ZrO$_2$-8% Y$_2$O$_3$ onto four of the previously low pressure plasma sprayed specimens. When cycled between room temperature and 1150°C in a burner rig, the test lives of the four specimens were 306, 331, 397, and 433 cycles, respectively with a mean value of 367 cycles. While this is about one-third of the anticipated life, the specimens were judged to be of sufficient quality for use for exploratory smooth bond coat development.
Nine of the bond coated specimens were centerless ground to an average roughness of about 110 micro-inch. The remaining bond coat thickness was now only 3 mils. Eight of these were low pressure plasma spray coated with between 0.9 and 1.9 mil of ZrO$_2$-8% Y$_2$O$_3$ followed by about 8 mil of conventional atmospheric pressure plasma sprayed ZrO$_2$-8% Y$_2$O$_3$. The last of the ground specimens was coated with 10 mil of the conventional APPS ZrO$_2$-8% Y$_2$O$_3$ ceramic only.

The arc power levels employed for the low pressure plasma spray were 54, 63, 72, or 81 KW at two levels of preheating resulting from four or 10 preheat passes. The reverse transfer arc feature of the low pressure plasma spray unit was used in each case. The roughness of the low pressure plasma sprayed ceramic, measured on the specimen prepared at 54 KW after 10 preheat passes, was 230 micro-inch.

The test lives defined by the number of cycles to spalling of the ceramic specimens were 110, 58, 47, and 43 cycles in order of increasing arc power for the case of 10 preheat cycles. These are shown by the line 12 in FIG. 1. The thickness of the low pressure plasma sprayed ceramic layer was 1.7, 1.3, 1.9, and 1.7 mil, respectively for the four data points shown in FIG. 1. For the case of two preheat cycles three of the specimens failed in the range of 60-106 cycles. However, the 72 KW specimen failed in only four cycles.

These tests illustrated that the life of a low pressure plasma sprayed-APPS ceramic coating on a smooth bond coat of 110 micro-inch was up to 10 times greater than the life of a conventional APPS ceramic on a smooth bond coat. The life of the present invention was over 30% of the life of a conventional rough interface thermal barrier coating system.

Greater lives were achieved with another set of Waspaloy specimens which were processed according to the schedule given in table I. All of these were first coated with about 8 mils of NiCrAlYb bond coat and centerless ground to a surface roughness of about 18 microinch and a thickness of about 5 mils. The first ten of these were processed together as a batch with the first eight of these comprising a standard $2^{4-1}$ fractional factorial design plan. The four variables investigated in this design were bond coat surface roughness, bond coat pre-oxida-
tion, specimen preheat temperature (i.e. the temperature of the specimen immediately before application of the APPS ceramic layer), and post-coating heat treatment.

Roughness was achieved by grit blasting four of the specimens to a roughness of about 46 microinch and another four to about 72 microinch. The pre-oxidation consisted of heating the bond coated specimen in air for 70 minutes at 1150°C (vs. no oxidation treatment), specimen preheat temperatures were 150°C or 370°C, and the post-coating heat treatment consisted of 4 hours at 1080°C in a vacuum furnace (vs. no heat treatment).

Each of the first eight specimens had a 0.60 to 0.85 mil layer of low pressure plasma sprayed zirconia-yttria (sprayed at 54 Kw) and a layer of air plasma sprayed ceramic to bring the total ceramic layer thickness to about 10 mils. Specimens nine and ten were deposited on the as-ground (18 microinch) bond coats using the higher level of both preheat and heat treatment. One of these was preoxidized and the other was not. As shown in FIG. 2 the 1050°C cyclic burner rig test lives of these 10 specimens varied from 287 to 1086 cycles compared to a standard thermal barrier coating which survived 1376 cycles. One of the longest lived specimens was an as-ground specimen which survived 1009 cycles. Statistical analysis of this data indicated that longer test lives were associated with the 150°C APPS preheat and two unintentional covariates -- namely, the thickness of the low pressure plasma sprayed ceramic layer and the presence of the gray oxide that is characteristic of alumina formation.

Specimens 11 through 16 were then processed to further test the effect of pre-oxidation and low pressure plasma sprayed ceramic layer thickness. As shown in the table, all of the specimens were deposited on the as-ground bond coats and their lives, in the same rig as above but with modified cooling air, varied from 231 to 430 cycles. This compared well with the 664 cycles observed for a standard thermal barrier coating. Statistical analysis supported the observation that
<table>
<thead>
<tr>
<th>Treatment Number</th>
<th>Test Life (Cycles)</th>
<th>Ra (Microinch)</th>
<th>Pre-Oxidized</th>
<th>Post Heat Treatment</th>
<th>APPS CERAMIC Temp. (C)</th>
<th>Oxide Color</th>
<th>LPPS CERAMIC THICKNESS</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1806</td>
<td>72</td>
<td>No</td>
<td>Yes</td>
<td>150</td>
<td>Gray</td>
<td>0.85 mils</td>
</tr>
<tr>
<td>2</td>
<td>487</td>
<td>46</td>
<td>No</td>
<td>No</td>
<td>150</td>
<td>Green</td>
<td>0.72</td>
</tr>
<tr>
<td>3</td>
<td>713</td>
<td>72</td>
<td>Yes</td>
<td>No</td>
<td>150</td>
<td>Gray</td>
<td>0.68</td>
</tr>
<tr>
<td>4</td>
<td>714</td>
<td>72</td>
<td>Yes</td>
<td>Yes</td>
<td>370</td>
<td>Gray</td>
<td>0.78</td>
</tr>
<tr>
<td>5</td>
<td>450</td>
<td>72</td>
<td>No</td>
<td>No</td>
<td>370</td>
<td>Green</td>
<td>0.78</td>
</tr>
<tr>
<td>6</td>
<td>374</td>
<td>46</td>
<td>No</td>
<td>Yes</td>
<td>370</td>
<td>Green</td>
<td>0.78</td>
</tr>
<tr>
<td>7</td>
<td>563</td>
<td>46</td>
<td>Yes</td>
<td>No</td>
<td>370</td>
<td>Green</td>
<td>0.80</td>
</tr>
<tr>
<td>8</td>
<td>698</td>
<td>46</td>
<td>Yes</td>
<td>Yes</td>
<td>150</td>
<td>Green</td>
<td>0.75</td>
</tr>
<tr>
<td>9</td>
<td>287</td>
<td>18</td>
<td>No</td>
<td>Yes</td>
<td>370</td>
<td>Gray</td>
<td>0.87</td>
</tr>
<tr>
<td>10</td>
<td>1009</td>
<td>18</td>
<td>Yes</td>
<td>Yes</td>
<td>370</td>
<td>Green</td>
<td>0.72</td>
</tr>
<tr>
<td>11</td>
<td>243</td>
<td>18</td>
<td>Yes</td>
<td>No</td>
<td>150</td>
<td>Gray</td>
<td>0.60</td>
</tr>
<tr>
<td>12</td>
<td>388</td>
<td>18</td>
<td>Yes</td>
<td>No</td>
<td>150</td>
<td>Gray</td>
<td>0.80</td>
</tr>
<tr>
<td>13</td>
<td>285</td>
<td>18</td>
<td>Yes</td>
<td>No</td>
<td>150</td>
<td>Gray</td>
<td>0.91</td>
</tr>
<tr>
<td>14</td>
<td>293</td>
<td>18</td>
<td>No</td>
<td>No</td>
<td>150</td>
<td>Gray</td>
<td>0.75</td>
</tr>
<tr>
<td>15</td>
<td>430</td>
<td>18</td>
<td>No</td>
<td>No</td>
<td>150</td>
<td>Green</td>
<td>1.04</td>
</tr>
<tr>
<td>16</td>
<td>231</td>
<td>18</td>
<td>No</td>
<td>No</td>
<td>150</td>
<td>Green</td>
<td>1.10</td>
</tr>
</tbody>
</table>
pre-oxidation had no effect on life and that there was a general upward trend in life with increasing low pressure plasma sprayed layer thickness for the 5 specimens exhibiting the gray oxide although the effect was less than expected and may indicate a diminishing effect after about 0.9 mil. The analysis also indicated that the life of the sixth specimen was diminished by the presence of the green oxide determined by x-ray analysis to consist primarily of nickel oxide. This was also in agreement with the previous result.

While a preferred embodiment of the invention is shown and described it will be appreciated that various procedural modifications can be made without departing from the spirit of the invention or the scope of the subjoined claims. By way of example, it is contemplated that a smooth uncoated surface of an oxidation resistant heat resistant alloy may be protected in a hot environment by a durable coating applied in accordance with the present invention. The surface is substantially smooth having a roughness less than about 120 microinch Ra. The first layer of ceramic material is low pressure plasma sprayed onto this smooth surface. This first layer is then covered by a second layer of ceramic material.
ABSTRACT OF THE DISCLOSURE

CERAMIC COATINGS ON SMOOTH SURFACES

A metallic coating is plasma sprayed onto a smooth surface of a metal alloy substrate, or on a bond coating. An initial thin ceramic layer is low pressure plasma sprayed onto the smooth surface of the substrate or bond coating. Another ceramic layer is atmospheric plasma sprayed onto the initial ceramic layer.
Fig. 1
Fig. 2

STANDARD

TREATMENT

BURNER RIG LIFE (CYCLES)