**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

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| Title: | BOND COAT FOR SILICON BASED SUBSTRATES | | |

# SUPPLEMENTAL DECLARATION OF DR. DAVID R. CLARKE

I, David R. Clarke, declare as follows:

1. This declaration supplements the declaration entitled "Declaration of Dr. David R. Clarke" dated October 6, 2016 (hereinafter "Original Declaration"). The statements made and opinions rendered therein can be assumed to be incorporated into this supplemental declaration, except as may be explicitly noted otherwise herein.

## I. Materials Considered

2. In addition to the materials I reviewed in preparation of my Original Declaration—which were noted in paragraphs 8 through 10—I have reviewed the following list of materials in preparation of this declaration:

- Institution Decision in IPR2016-01289 entered on December 27, 2016 (Paper 7)

- M.E. Westwood *et al.*, *Oxidation protection for carbon fibre composites*, 31 Journal of Materials Science. 1389-1397 (1996). (UTC-2016)
- M. Ohring, ENGINEERING MATERIALS SCIENCE (1995). (UTC-2017)
- L.H. Van Vlack, ELEMENTS OF MATERIALS SCIENCE AND ENGINEERING (1989). (UTC-2018)
- K.N. Lee, *Key Durability Issues With Mullite-Based Environmental Barrier Coatings for Si-Based Ceramics*, 122 Transactions of the ASME. 632-636 (2000). (UTC-2020)
- N.S. Jacobson *et al.*, *Oxidation and corrosion of ceramics and ceramic matrix composites*, 5 Current Opinion in Solid State and Materials Science. 301-309 (2001). (UTC-2021)
- K.N. Lee, *Current status of environmental barrier coatings for Si-Based ceramics*, 133-134 Surface and Coatings Technology. 1-7 (2000). (UTC-2022)
- S.V. Raj, *Comparison of the Thermal Expansion Behavior of Several Intermetallic Silicide Alloys Between 293 and 1523 K*, 24 Journal of Materials Engineering and Performance, 1199-1205 (2015). (UTC-2023)
- D. Zhu *et al.*, *Thermal Conductivity of Ceramic Thermal Barrier and Environmental Barrier Coating Materials*, NASA/TM–2001-211122 (2001). (UTC-2024)

- R. Siegel and C.M. Spuckler, *Analysis of thermal radiation effects on temperatures in turbine engine thermal barrier coatings*, A245 Materials Science and Engineering, 150-159 (1998). (UTC-2025)
- M. Peters *et al.*, *Design and Properties of Thermal Barrier Coatings for Advanced Turbine Engines*, 28 Materialwissenschaft und Werkstofftechnik. 357-362 (1997). (UTC-2026)
- R.H. Doremus, *Viscosity of silica*, 92 Journal of Applied Physics, 7619-7629 (2002). (UTC-2027)

3. I have also considered all other materials cited herein.

## II. Applicable Legal Standards

4. In this supplemental declaration, I am applying the same standards and legal principles that I applied when drafting my Original Declaration, which were outlined in paragraphs 17 through 32 of that document.

## III. Design of Multilayer Coatings

5. As I noted in paragraph 34 of my Original Declaration, a coating is a thin layer of material that is formed or deposited on the surface of a substrate. Often, a coating is applied to a substrate to create a composite material that has properties that are not present in the substrate alone: "Coatings enable the attributes of two or more materials [e.g., the coating and the substrate] to be

combined to form a composite having characteristics not readily available in a monolithic material." UTC-2002, p. xii.

6. A coating including multiple layers of material is referred to as a multilayer coating. Multilayer coatings are often formed on a substrate to obtain a composite material having properties or a combination of properties or functions not available from a single coating material.

### A. *Factors in Designing a Multilayer Coating*

7. In selecting materials for a multilayer coating, one must take into account a number of factors, including the properties of the individual constituent layers as well as interactions that will occur between layers in the multilayer coating and between the coating and the substrate. Such factors include, but are not limited to, compatibility of the coefficients of thermal expansion, chemical compatibility, environmental stability, elastic moduli, and the evolution of interfaces in the coating or between the coating and the substrate. *See* GE-1011, p. 1; UTC-2002, p. 11; UTC-2021, p. 305, UTC-2022, p. 1, UTC-2002, p. 11, GE-1011, p. 1. Consideration of these factors is particularly important when designing a multilayer coating for use under harsh environmental conditions such as high temperatures, high velocity gas flows, or repeated thermal cycling to high temperatures. Absent knowledge of previously developed evidence of these factors for the actual materials being considered, a POSITA would not have

perceived a reasonable likelihood of success in combining layers into a multilayer system, capable of withstanding high temperature service in a gas turbine, without undertaking expensive and time-consuming experimentation.

8. The thermal expansion coefficient of a material is a quantitative measure of the amount by which the material will expand or contract in response to a change in temperature. *See* UTC-2017, p. 61. Selection of materials for a multilayer coating that have compatible thermal expansion coefficients is crucial for the stability of the coating. If layers in a multilayer coating have different thermal expansion coefficients, those layers will expand or contract by different amounts when the multilayer coating is heated or cooled. These differences in expansion or contraction can cause stresses to develop in the multilayer coating. In some cases, thermal stresses can result in structural failures, such as cracking in the coating or at an interface between the coating and the substrate leading to delamination of the coating from the substrate. *See* UTC-2006, p. 179; UTC-2021, p. 305; UTC-2022, p. 1.

9. Chemical compatibility of materials in a multilayer coating is an indication of whether the materials can coexist in the coating without resulting in chemical reactions between the materials. Layers of a multilayer coating should be chemically compatible, and the coating should be chemically compatible with the

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