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FORMULATION OF OXIDE SUSPENSIONS FOR LIQUID PROCESSING OF CERAMIC MATRIX COMPOSITES

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Introduction

• Context:
  - Oxide-Oxide ceramic matrix composites can be used as high-temperature components for gas turbine engine technologies due to their good thermomechanical properties. [1]
  - Liquid processes (Resin Transfer Molding and Liquid Resin Infusion) are more advantageous than CMC conventional ones.
  - The formulation of suspensions represents the first essential step for liquid processing. [2]
  - The suspension must be well dispersed, stable and have a low viscosity (< 1 Pa.s)

• Aim of this study:
  - Formulate a bi-component suspension (with submicron alumina and colloidal silica) suitable for the liquid processing of oxide-oxide ceramic matrix composites

Materials

• Mixture proportion: 59 vol.% of Al2O3 and 41 vol.% of SiO2, corresponding to the milli-runtime [1]

<table>
<thead>
<tr>
<th>Particle size</th>
<th>Surface area</th>
<th>Volumetric mass density</th>
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<tbody>
<tr>
<td>200±40 μm</td>
<td>129 – 155 m2/g</td>
<td>2.2 g/cm³</td>
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<table>
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<tr>
<th>Submicron Alumina</th>
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<td>AKPS0, high purity alpha-alumina powder (Sumitomo Chemical, Japan)</td>
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<thead>
<tr>
<th>Particle size</th>
<th>Surface area</th>
<th>Volumetric mass density</th>
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<tbody>
<tr>
<td>200 nm</td>
<td>10.5 m²/g</td>
<td>3.98 g/cm³</td>
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Results

Dispersion

• Measurement of the zeta potential of diluted suspensions as a function of pH – Acoustosizer IIs (Colloidal Dynamics LLC, USA)

  - The silica suspension possesses a negative zeta potential from pH = 2 to pH = 11.
  - The zeta potential of the alumina suspension is positive (ζ = 21.7 mV) at natural pH (pH = 8.3) and its value is not sufficient to avoid particles agglomeration.
  - This agglomeration can take place between particles of alumina (homo-agglomeration) but also between particles of alumina and silica (hetero-agglomeration).
  - By adding 0.26 wt % of dispersant (Darvan® C-N, Vanderbilt Minerals LLC, USA), an ammonium polymethacrylate, the isoelectric point (IEP) of the alumina is shifted from pH = 9.5 (without dispersant) to pH = 5.1.
  - The bi-component suspension is defined as the mixture of the silica suspension and the alumina suspension with 0.26 wt % of dispersant in the proportion given previously.
  - This suspension is well dispersed because the zeta potential at natural pH (pH = 9.5) is measured at ζ = -44.7 mV.

Stability

• Sedimentation tests were performed to study the stability of the suspension.
  - A 40 vol.% bi-component suspension was considered.
  - Sedimentation is observable after a time of 4 days.
  - In consequence, the bi-component suspension is stable in comparison with the process time (less than one day).

Rheology

• Measurements performed using an AR-G2 rheometer (TA Instruments, USA) equipped with a cone-plate geometry.
  - Rheological behavior of the bi-component suspension (from γ = 10² s⁻¹ to γ = 200 s⁻¹).
  - The rheological behavior of the suspension is dominated by the colloidal and hydrodynamic interactions and by the brownian motions.
  - At low shear rates, the behavior is shear thinning.
  - Whereas at high shear rates, the viscosity is constant.
  - Moreover, the solid concentration represents an essential variable regarding the rheology.

Influence of solid concentration on viscosity

• The influence of solid concentration on the suspension viscosity is well described by a Quemada model.

Wetting

• Measurement of the contact angle of a 25 vol.% bi-component suspension drop on an alumina planar substrate.
  - Surface agent: BYK-348, polyether-modified siloxane (BYK Additives & Instruments, Germany)

  - Without surface agent
  - With surface agent in the suspension
  - With surface agent on the substrate

  - The contact angle decreases by using surface agent either in the suspension and on the substrate.
  - The case for which the surface agent is deposited on the substrate surface is the most favorable.

References