Formulation of oxide suspensions for liquid processing of ceramic matrix composites

Florian Boutenel, Anne Aimable, Gilles Dusserre, Thierry Cutard, Thierry Chartier

To cite this version:

Florian Boutenel, Anne Aimable, Gilles Dusserre, Thierry Cutard, Thierry Chartier. Formulation of oxide suspensions for liquid processing of ceramic matrix composites. HT-CMC 10 - 10th International Conference on High Temperature Ceramic Matrix Composites, Sep 2019, Bordeaux, France. 2019. hal-02300057

HAL Id: hal-02300057
https://imt-mines-albi.hal.science/hal-02300057
Submitted on 29 Sep 2019

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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 melt ratio of Al2O3/SiO2 = 4.08 in weight.

**Surface agent in the suspension:**
- Particle size: 200 nm
- Surface area: 10.5 m²/g
- Volumetric mass density: 3.98 g/cm³

**Formulate a suspension of Alumina**
- AKP50, high purity alpha-alumina powder (Sumitomo Chemical, Japan)

**Formulate a suspension of Silica**
- Ludox® AS-40, 40 wt% suspension in water (Grace Davison, USA)

**Technological data:**
- Technical data provided by the manufacturer.

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 = -22.1 mV) at natural pH (pH = 8.3) and its value is not sufficient to avoid particle 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-11N, 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 η = 10^4 s^-1 to η = 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 content represents an essential variable regarding the rheology.

**Influence of solid concentration on suspension 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.

Conclusion

- A suspension of submicron alumina and colloidal silica has been formulated. It is well dispersed (no agglomeration of particles), stable (in comparison with the process time) and has a low viscosity (< 1 Pa.s). Moreover, the influence of solid concentration on the rheological behavior of the suspension is well described by a Quemada model. Furthermore, the wetting of the suspension can be promoted by using a surface agent.
- This bi-component suspension is thus suitable for the liquid processing of oxide-oxide ceramic matrix composites.

References


Acknowledgements

This research received a grant from the Occitanie region and fundings from Institut Carnot M.I.N.E.S (ANR).