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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 mullite ratio

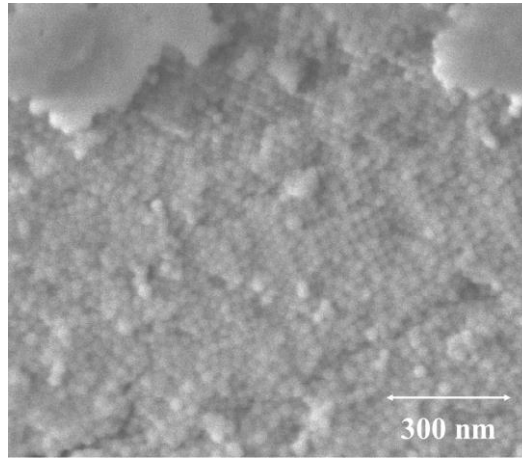
Colloidal Silica

Particle size	20.0 – 24.0 nm
Surface area	129 – 155 m ² .g ⁻¹
Volumetric mass density	2.2 g.cm ⁻³

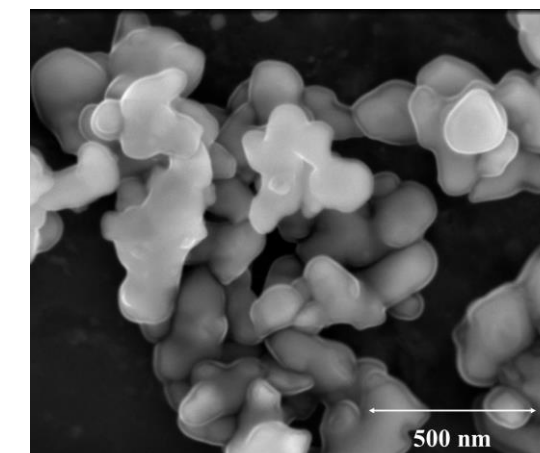
Ludox® AS-40, 40 wt.% suspension in water (Grace Davison, USA)

Technical Data

SEM Observation



Submicron Alumina



AKP50, high purity alpha-alumina powder (Sumitomo Chemical, Japan)

SEM Observation

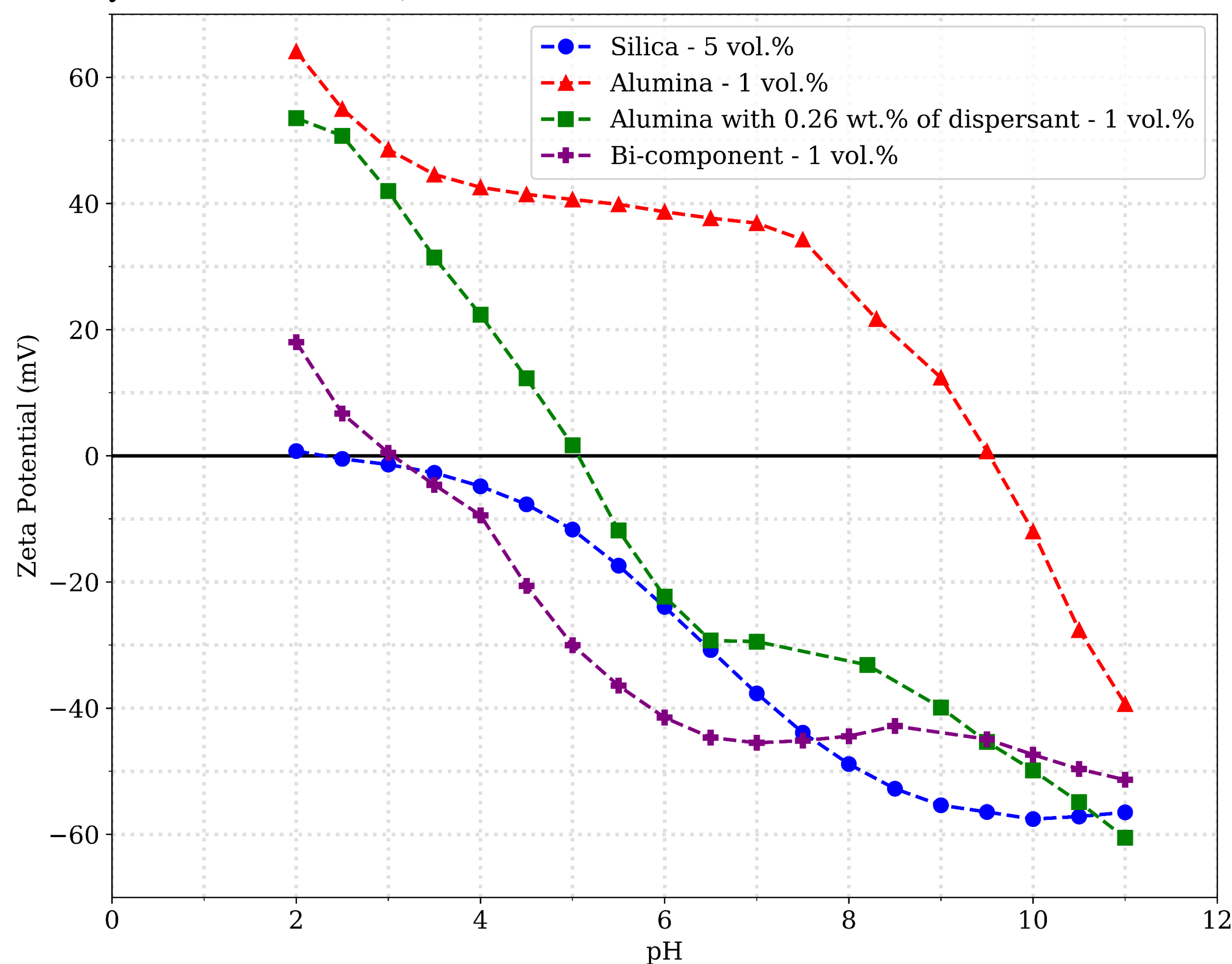
Technical Data

Particle size	200 nm
Surface area	10.3 m ² .g ⁻¹
Volumetric mass density	3.98 g.cm ⁻³

Results

Dispersion

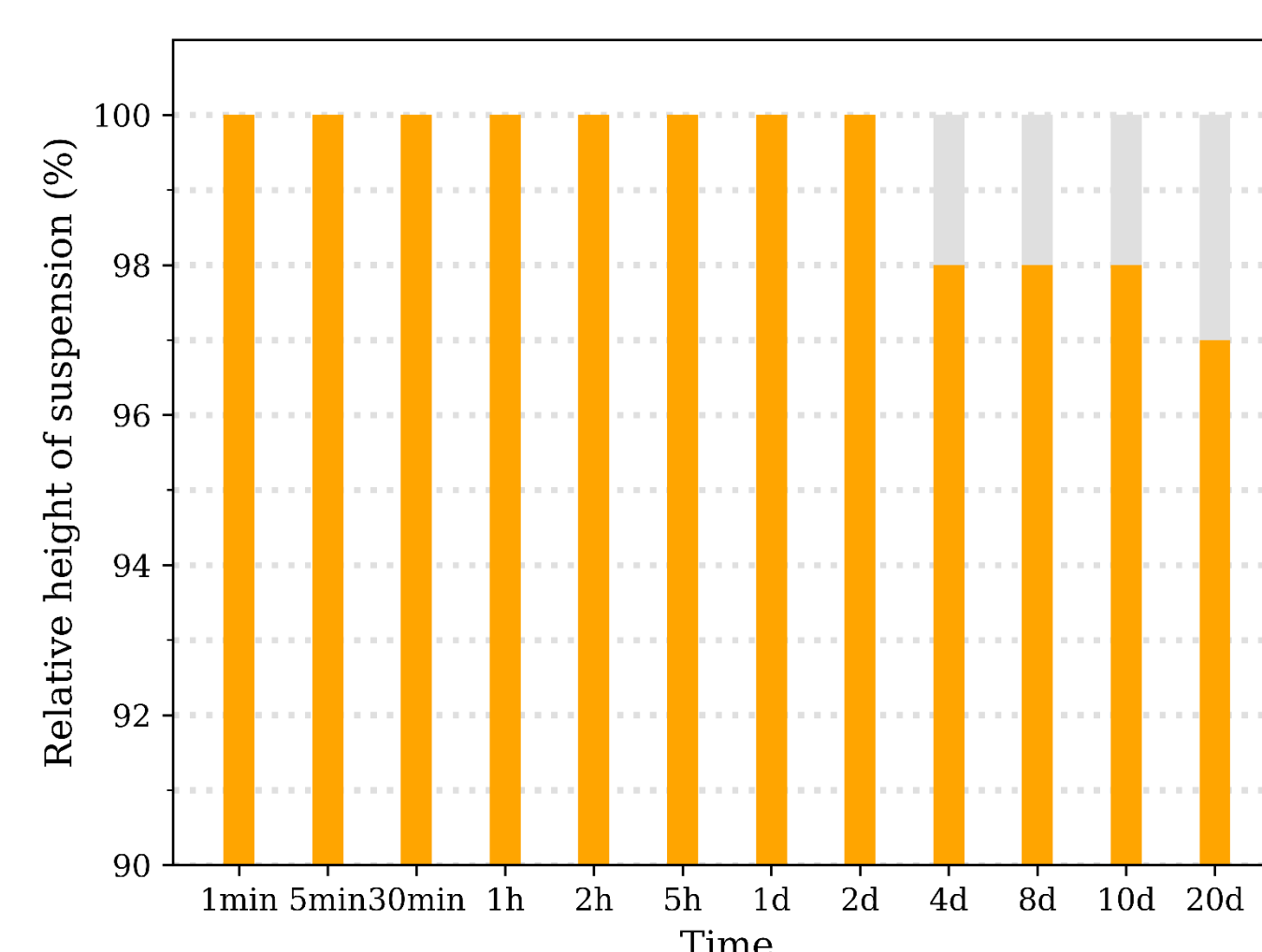
- Measurement of the zeta potential of diluted suspensions as a function of pH – Acoustosizer IIS (Colloidal Dynamics LLC, USA)



- The silica suspension possesses a negative zeta potential from pH = 2 to pH = 11.
- The zeta potential of the alumina suspension is positive ($\zeta = 21.7$ 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 $\zeta = -44.7$ 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).

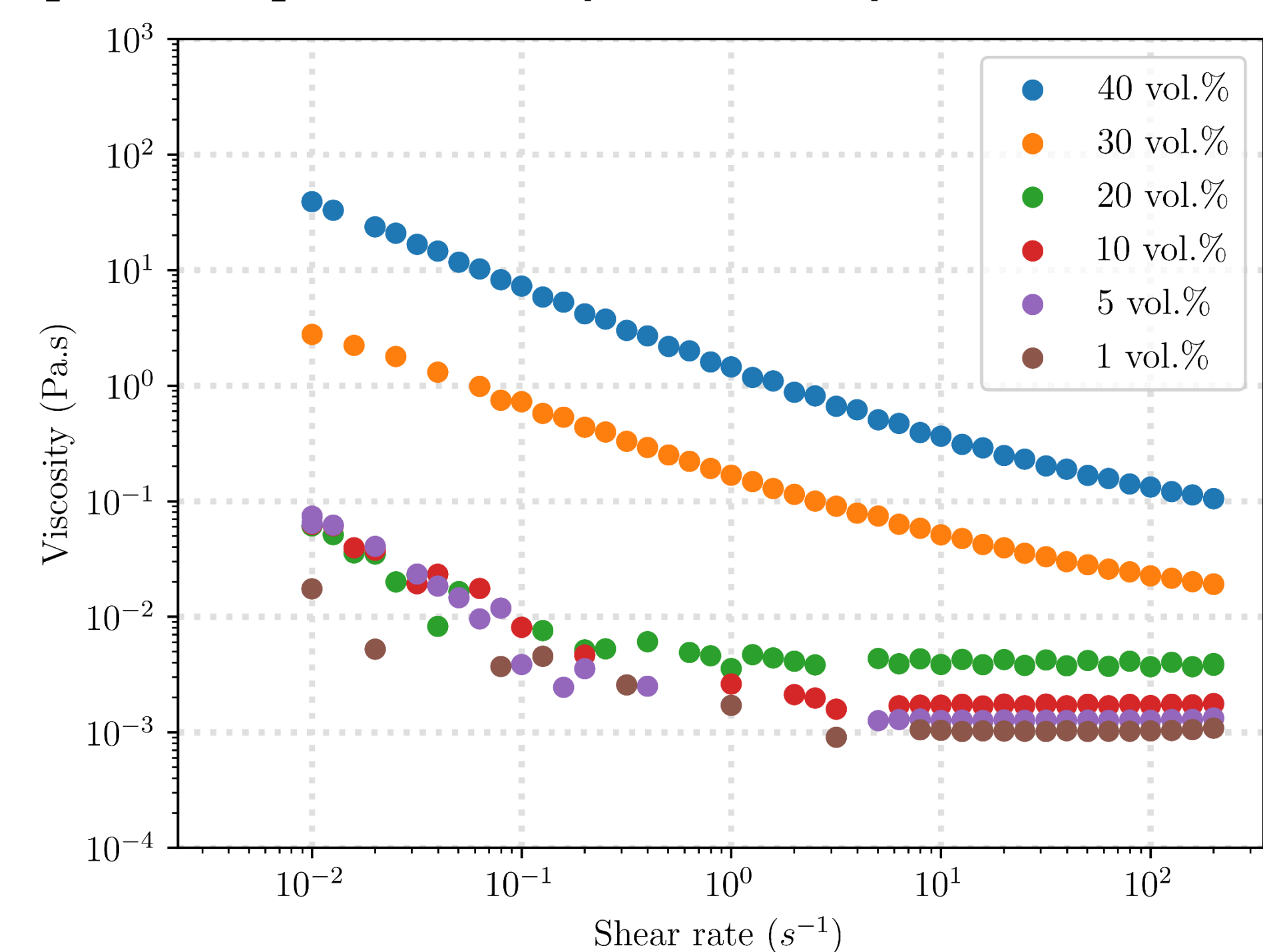


Rheology

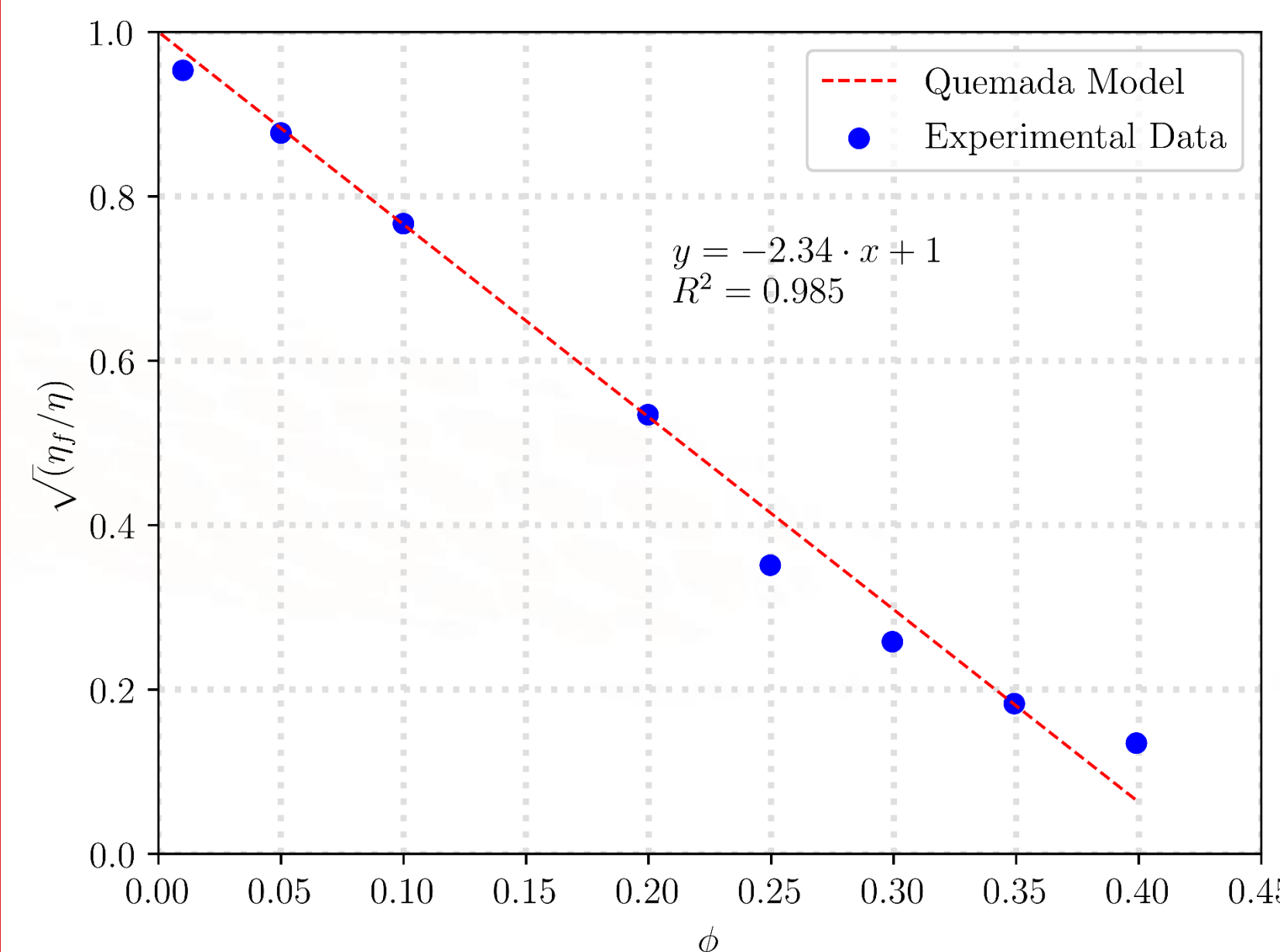
- Measurements performed using an AR-G2 rheometer (TA Instruments, USA) equipped with a cone-plate geometry.

- Rheological behavior of the bi-component suspension (from $\dot{\gamma} = 10^{-2}$ s⁻¹ to $\dot{\gamma} = 200$ 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 suspension viscosity



- Quemada model:

$$\sqrt{\frac{\eta_f}{\eta}} = 1 - \frac{\phi}{\phi_{max}}$$

η : viscosity of the suspension at the plateau (Pa.s)
 η_f : viscosity of the interstitial fluid – water (Pa.s)
 ϕ : solid concentration (vol.%)
 ϕ_{max} : maximum packing fraction (vol.%)

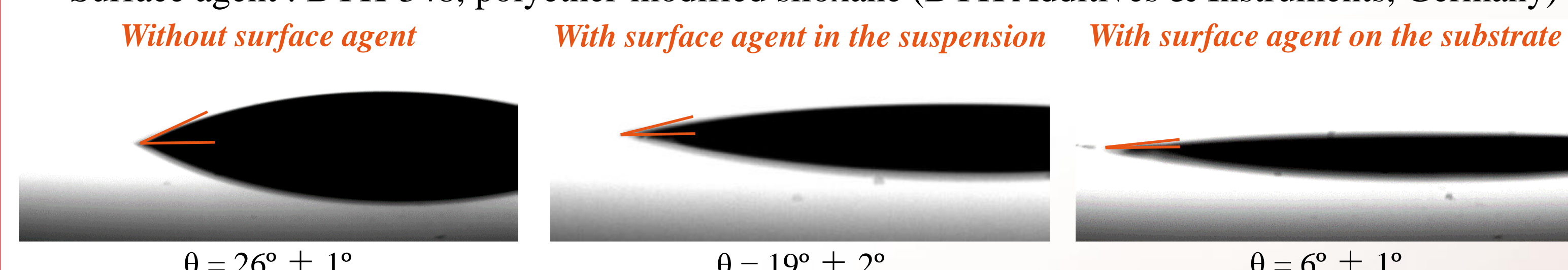
Identification of the model parameter ϕ_{max}
by linear regression

η_f	ϕ_{max}
0.001 Pa.s	42.69 vol.%

- 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)



- 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

- [1] Zok, F.W. and Levi, C.G. (2001). Mechanical properties of porous-matrix ceramic composites. *Advanced Engineering Materials*, vol. 3, n°1-2, p. 15-23.
- [2] Billotte, C., Fotsing, E.R. and Ruiz, E. (2017). Optimization of alumina slurry for oxide-oxide ceramic composites manufactured by injection molding. *Advances in Materials Science and Engineering*, vol. 2017.

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