RHEOLASER MASTER

Screening of sol-gel mechanical properties
Ceramic inks for 3D-printing

INTRODUCTION

Stereolithography is a powerful 3D printing technique to fabricate prototypes quickly and precisely. Composite materials of ceramics and photocurable polymers allow the preparation of objects with high mechanical properties. The inks used for this purpose must fulfill several conditions:

- Shear thinning behavior must get through the printing nozzle
- Fast recovery to stay in place after application
- Fast sol-gel reaction and curing to get strong materials

This application note shows, how Rheolaser Master is used as a screening tool to correlate the viscoelastic properties during gelation with the final mechanical properties after total curing, which accelerate the development of new ceramic inks.

Ceramic inks | Sol-gel | 3D printing

HOW IT WORKS

Rheolaser Master is based on Diffusing Wave Spectroscopy (DWS), a multiple light scattering technique. Light is backscattered by scatterers in the sample. The microstructure motion inside the sample (droplets, crystallites, etc.), creates an interference pattern (Speckle Image). Variation of this image in time is directly related to the mobility of the scatterers. (Figure 1). The faster the Speckle Image changes in time, the higher the mobility of the microstructure.

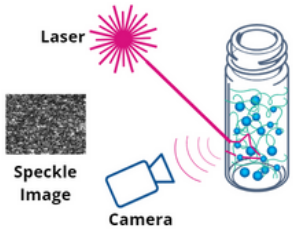

Figure 1. Schematical representation of the measurement set-up.

By mathematical treatment, Mean Square Displacement (MSD) curves are obtained (Figure 2), which contain the viscoelastic information. Short straight lines (blue curves) indicate the liquid behavior of the sample, whereas curves with a plateau, the so-called elastic plateau (red curves), indicate gel-like or solid-like behavior.

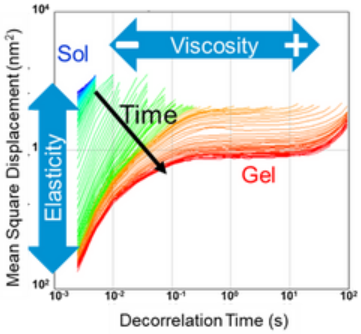

Figure 2. Typical evolution of MSD curves showing a gelation with liquid curves (bleu) adn viscoelastic curves (red).

SAMPLE AND PROTOCOLE

Ceramic inks were composed of aluminum oxide particles, embedded in a photocurable polymer. Particles were linked with 6 different silane coupling agents (SCA 1 to SCA 6) to improve mechanical properties. For more detailed information, see Song et al **(2018; doi:10.3390/nano8020093)**. Rheolaser Master® measurements studied the curing process of the sol gels, whereas nano-indentation measurements were used to study the mechanical properties after complete curing.

RESULTS AND DISCUSSION

Curing was studied with Rheolaser Master for 6 different Silane Coupling Agents (SCA). Figure 3 shows the MSD curves after 12 hours for each SCA. SCA3 and SCA6 do not show a plateau, indicating any gel formation. The other SCA shows a plateau. The lower the plateau, the higher the elasticity of the gel.

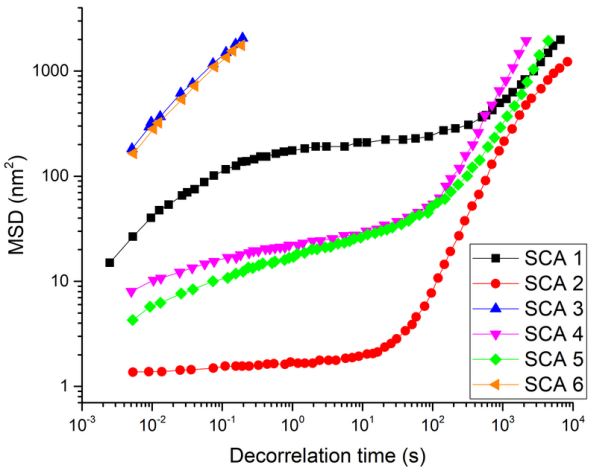

Figure 3. MSD curves after 12h of gelation.

FORMULACTION Scientific instruments

Figure 4 shows the Nanoindentation measurements after complete curing. The shorter the depth and the higher the loading, the higher the mechanical properties of the samples. These measurements were conducted after 72 hours of drying.

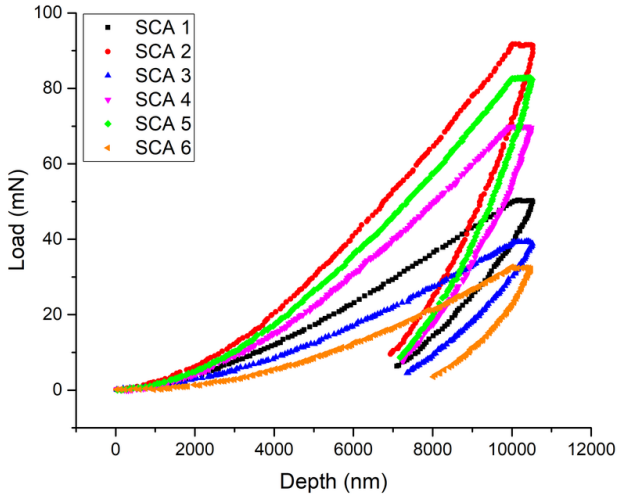

Figure 4. Mechanical tests using nanoindentation of 6 different ceramic inks after 72 hours.

SCA 2 and SCA 5 have the highest mechanical properties, which corresponds to the high elasticity measured during gelation with the Rheolaser Master.
For better comparison, the values of the Elasticity Index (reverse of MSD value at a decorrelation time of 0.1 s) and the Nanoindentation Elastic Modulus for the 6 samples were plotted in Figure 5.

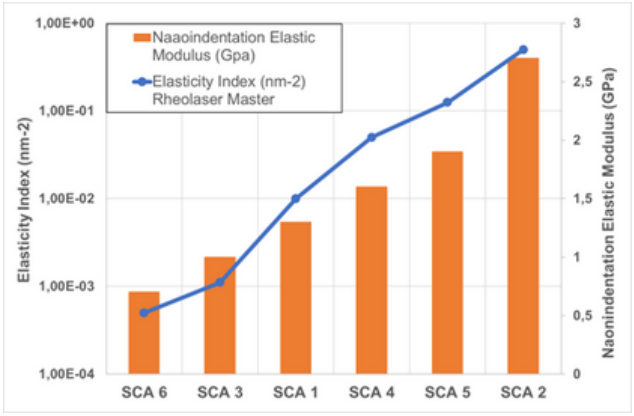

Figure 5. Comparison of measurements during gelation with Rheolaser Master and after 72 hours of drying with nanoindentation.

There is a good correlation between the measurements done during the sol-gel step using Rheolaser Master and the measurements were done after complete curing with nanoindentation. The higher the elasticity Index after 12 hours, the higher the mechanical properties after complete drying. Rheolaser Master helps therefore by screening to find the right coupling agent for the application and reduces the development time.

CONCLUSION

Rheolaser Master is a powerful tool for the analysis of sol-gel processes. The 6 measurement positions allow a fast screening of different formulations, which accelerates the optimization of the development of new sol-gel materials. A very good correlation between viscoelastic properties during gelation with the mechanical properties after complete curing was found.