Rheology coupled with infrared spectroscopy in monitoring a hydrosilylation reaction of a hyperbranched polymer.

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In the area of non-oxide ceramic, the polymer derived ceramic route was a major breakthrough to obtain silicon based ceramics with excellent thermo-mechanical properties such as high strength, hardness, low density and high temperature materials with self-healing properties [1, 2]. In the interest of obtaining ceramics that can be shaped easily, we have characterized the process of synthesis and crosslinking from a monomer to a preceramic polymer using a rheometer together with a Fourier Transform Infrared spectroscopy [3]. This will help in monitoring and controlling the chemical and physical properties of the precursor to finally lead to the desired SiC.

This presentation will emphasize the coupling of a rheometer with an infrared spectrometer. A hydrosilylation reaction was used to successfully synthesise a hyperbranched polymer [4, 5]. The polymerization reaction was monitored in situ by combining the rheological and infrared measurements [6]. A real time control of the construction of the macromolecular structures under defined atmosphere and temperature was performed. The results indicate a gel-like behavior for alkene conversions higher than 0.55. The hydrosilylation reaction was able to determine the overall second–order kinetics. Porous material β-SiC and free carbon were obtained after undergoing a pyrolysis at 1400 °C.

To complete the coupling system, a portable Raman spectrometer is added. Indeed, it is a well-known powerful tool to characterise materials and it leads to higher depth penetration in analysed materials [7-9]. Moreover, the FTIR and Raman complement each other, improving the analysis of data obtained during simultaneous monitoring. Therefore, a new system consisting of a rheometer, FTIR and Raman spectroscopies will enable us to monitor in-situ the polymerization reaction in real time at controlled atmosphere and temperature.

[1] Bougoin, M.; Thevenot, F.; Dubois, J.; Fantozzi, G. *Journal of the Less-Common Metals*, 132 (1987) 209.
[2] Yajima, S.; Okamura, K.; Hayashi, J; Omori, M. *Journal of the American Ceramics Society*, 59 (1976) 324.
[3] Darsy, G.; Bouzat, F.; Muñoz-Hoyos, M.; Lucas, R.; Foucaud, S.; Coelho, C.; Babonneau, F.; Maître, A. *Polymer,* 79 (2015) 283.
[4] P. Ji, X. Pei, Y. Miao, L. He, Q. Huang, *Advance. Applied. Ceramics,* 116 (2017) 445.
[5] C. Lach, P. Müller, H. Frey, R. Mülhaupt, *Macromolecular Rapid Communication*, 18 (1997) 253.
[6] Piriou, C.; Viers, L.; Lucas, R.; Bouzat, F.; Laadoua, H.; Champavier, Y.; Foucaud, S.; Coelho, C.; Babonneau, F, *Applied Organometallic Chemistry,* 32(9): e4443 (2018) 2.
[7] Kudelski, A. *Talanta,* 76 (2008) 1.
[8] Simsek, G.; Colomban, P.; Milande, V, *Journal of Raman Spectroscopy,* 41 (2010) 529.
[9] Quang, L.X.; Lim, C.; Seong, G.H.; Choo, J.; Do, K.J.; Yoo, S.Y. *Royal society of chemistry, Lab Chip* 8 (2008) 2214.