

Investigation on the structure of silica/polyurethane nanocomposites

Katrin Rübner¹, Harald Goering¹, Peter Klobes¹, Hartmut Tschritter²

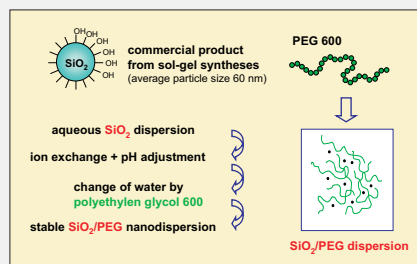
¹ BAM Federal Institute for Materials Research and Testing, Berlin, Germany

² Chemiewerk Bad Köstritz GmbH, Bad Köstritz, Germany

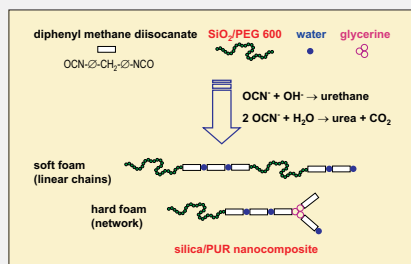
Introduction

Inorganic-organic nanocomposites are synthesised from polyethylene glycol with an average molar mass of 600 g/mol containing colloidal silica (silicic acid) and diphenyl methane diisocyanate as compact and foamed materials according to the European patent EP1414880 [1]. Beside the macroscopic properties, the pore structure and the structure of the polymer matrix of the silica/polyurethane nanocomposites with varying silica contents were studied.

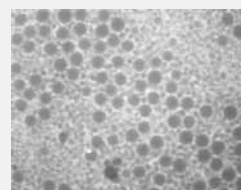
Synthesis



Dispersion of silica nanoparticles (SiO₂) in polyethylene glycol (PEG)



Syntheses of polyurethane (PUR) by polyaddition including formation of foaming agent

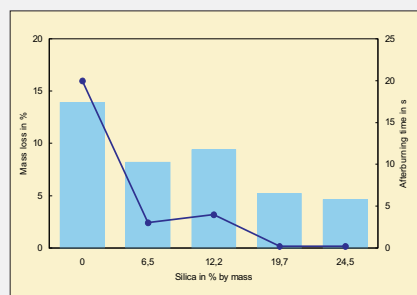


TEM transmission micrograph of the system silica/polyethylene glycol containing solid particles of about 30% by mass



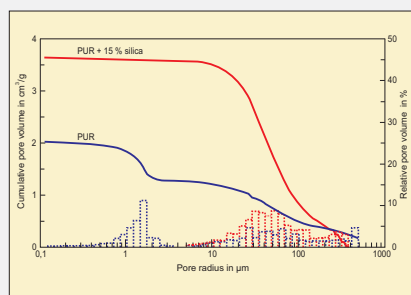
SEM micrograph of nano-composite foam containing 17% silica, detection of SiO₂ particles by EDX

Properties



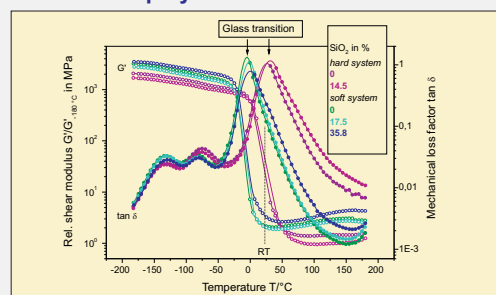
Flammability of hard nanocomposite foams

Pore structure



Pore size distribution of hard nanocomposite foams from mercury intrusion measurements

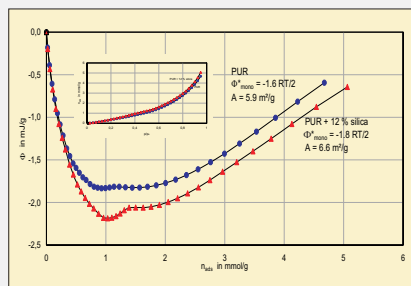
Structure of polymer matrix



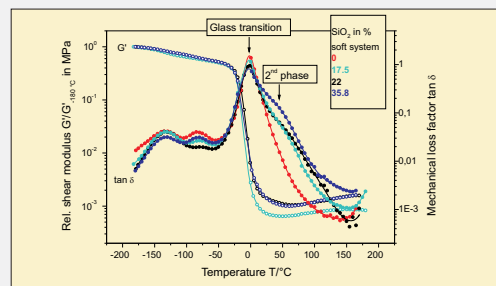
Dynamic mechanical analysis of hard and soft compact nanocomposites (Torsional pendulum 1 Hz)

Silica content in % by mass	Bulk density in g/cm ³	Density in g/cm ³	Total porosity in %	Compressive strength in MPa	Flexural strength in MPa
0	0.16	1.37	88.4	1.30	1.80
6.5	0.17	1.56	89.1	1.75	2.25
12.5	0.14	1.52	90.8	1.65	1.43
19.7	0.16	1.50	89.3	1.63	1.60
24.5	0.17	1.54	89.0	1.44	1.68
0	0.20	1.28	84.4	1.35	1.81
12.2	0.21	1.45	85.5	3.01	3.64
24.5	0.22	1.45	84.8	1.55	1.82

Density, porosity and strength of hard nanocomposite foams



ESW diagrams of hard nanocomposite foams according to the theory of excess surface work by Adolphs [2] calculated from water vapour isotherms at 298 K



Dynamic mechanical analysis of soft nanocomposite foams (Torsional pendulum 1 Hz)

Conclusions

- The PUR system is able to incorporate silica up to 35% by mass.
- An inorganic-organic SiO₂/PUR nanocomposite is formed.
- Hydroxyl groups on the surface of the silica nanoparticles and adsorbed water molecules take part in the synthesis of polyurethane (PUR).
- The silica particles take part in the development of the polymer network and influence the foaming process of PUR.
- Beside a relatively homogeneous distribution of SiO₂, there are silica agglomerates on lamellas, strips and nodes of the polymer.
- The pore structure of PUR is changed by a silica layer formed on the polymer lamellas, which reduces the formation of small pores.
- The SiO₂ nanoparticles influence only marginally the molecular dynamics as indicated by nearly unchanged glass transition. There is an additional relaxation region above glass temperature.
- The polymer is little reinforced by silica particles as shown by small changing of plateau modulus. Silica additions stabilise the polymer network indicated by better entropy elasticity.
- Silica slightly reduces the hydrophobic characteristics of PUR.
- The compressive strength and flexural strength as well as the flammability of the SiO₂/PUR nanocomposites are improved.

References

- [1] H. Tschritter, G. Knöfle, H. Oehlert, M. Ottow and J. Rübner, European Patent EP1414880, 6.5.2004
- [2] J. Adolphs, Appl. Surface Sci. 253 (2007) 5645-5649