Flexural strength of methacrylate resins with experimental filler surface treatment.

M. ROSENTRITT*[1], A. KOKOTT[2], B. HOFFMANN[2], O. SHAFRANSKA[2], M. BEHR[1], G. ZIEGLER[2], and G. HANDEL[1]
([1] University of Regensburg, Germany, [2] Friedrich-Baur-Research Institute for Biomaterials, Bayreuth, Germany) **# 0343**

Introduction:

The surface of inorganic fillers is silanized to guarantee a chemical adhesion between filler and resin matrix. The aim of this investigation was to compare mechanical properties of resins with alternative surface activating methods.

Materials and methods:

Materials:
SiO_2 fillers (Aerosil200, Degussa, G) were chemically modified with:
- sterylethyltrimethoxysilane (SES),
- p-aminostyrene functionalized copolymer of styrene and maleic anhydride (AS1),
- styrene/maleic anhydride copolymer (ST-MA), photo-immobilized dibenzoylperoxide (BPO),
- (O-(methacryloxyethyl)-N-(triethoxysilylpropyl)urethane (MSU),
- p-aminosytrene functionalized copolymer of methyl methacrylate and maleic anhydride (MA-AS) and
- (3-methacryloxypropyl)trimethoxysilane (MPTS) as a control.

Blends were mixed of UDMA/Decandioldimethacrylate (ratio: 70:30) resin with 30weigth% surface-treated filler. Camphorquinone/amine was added for activation. Specimens (2mmx2mmx22mm) were light/heat cured (107°C, 25 min, Targis Power, Ivoclar-Vivadent, FL).

Flexural Strength:
Using a three-point-bending design, flexural strength (FS) was investigated with a UTM Zwick 1446 (v=1mm/min).

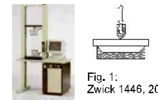

Fig. 1:
Zwick 1446, 20mm support

Storage Modulus/ Glass-Transition:
Temperature dependent storage modulus (E') and glass-transition (Tg) were determined with a dynamic mechanical analyser (DMA, 1Hz, Netzsch, G) in an temperature range between 0°C and 80°C.

$$|E^*| = \frac{F_a}{L_a} g \qquad E' = |E^*| \cos \delta \qquad E'' = |E^*| \sin \delta \qquad \tan\delta = \frac{E''}{E'}$$

Fig. 2: Formulas to calculate the complex modulus E*, the storage modulus E´, the loss modulus E´´ and the loss factor tan d.
20 mm

Fig. 3:
Netzsch DMA 242

Fig. 4:
three-point bending sample holder 16mm

Statistical analysis was performed using One-way ANOVA (p=0.05).

Results:

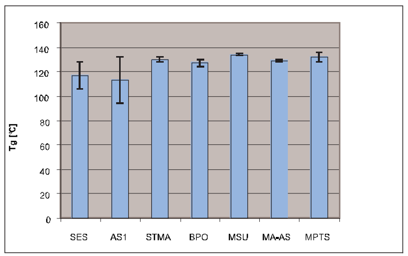

Fig. 5:
Glass-Transition Tg (°C)

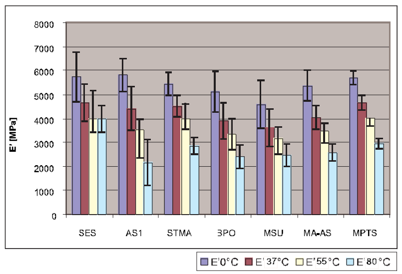

Fig. 6:
Storage Modulus

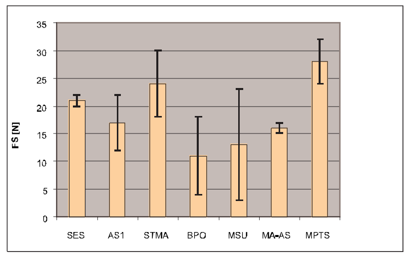

Fig. 7:
Flexural Strength

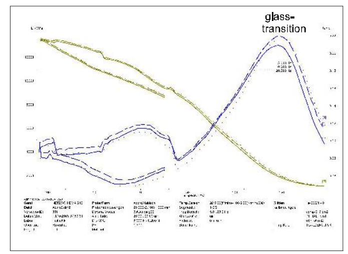

Fig. 8: DMA graph example
E' and tan δ between -100°C and 200°C

The resins showed no significant different glass-transitions. The highest flexural strength was found for the MPTS, SES and ST-AM. Only MSU had a storage modulus E' at 0°C lower than 5000 MPa. All materials showed a strong decrease of the modulus in the tested temperature range between 0°C and 80°C. Only SES had a storage modulus at 80°C of about 4000 MPa.

Conclusion:

Experimental filler surface activation showed comparable or higher loss-modul but lower fracture strength than conventional silane treatment.