

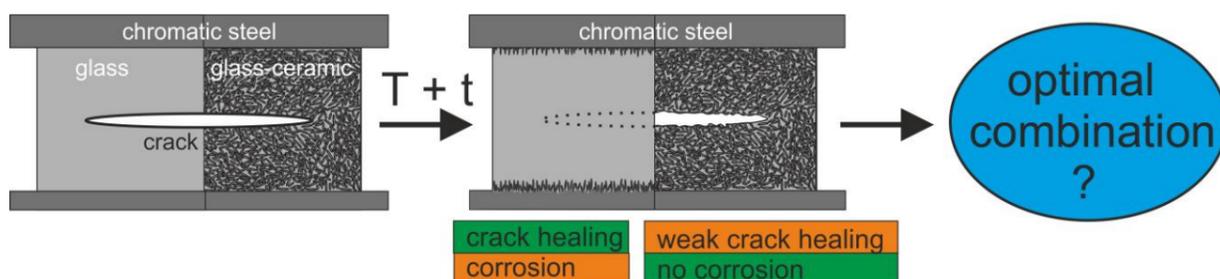
Crack healing in glass matrix composites

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Aim

Understanding of crack healing in glass and glass ceramic materials is fundamental for many applications, such as the cyclability of seals in solid oxide fuel cells (SOFC). Mostly two sealing concepts are established: non-crystallizing glass, allowing easy crack healing, and long term durable and diffusion limiting glass-ceramic material. Our study tries to find the optimum microstructure for crack healing.



Approach

Mimicking glass ceramic microstructure by glass matrix composites of non-crystallizing soda lime silicate glass (NCS) and zirconia -> stable microstructure during crack healing.

Results

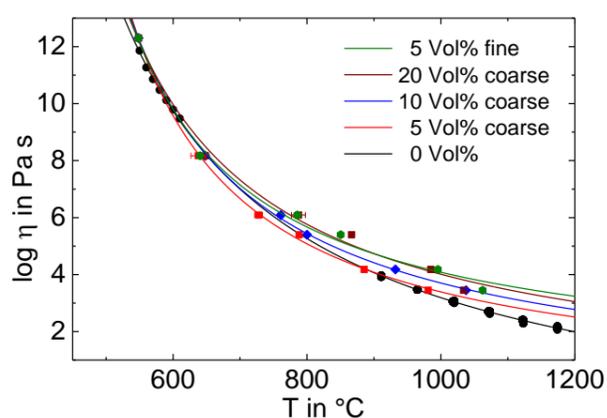


Figure 1
Viscosity of GMCs with varied ZrO_2 filler content obtained from heating microscopy [1]

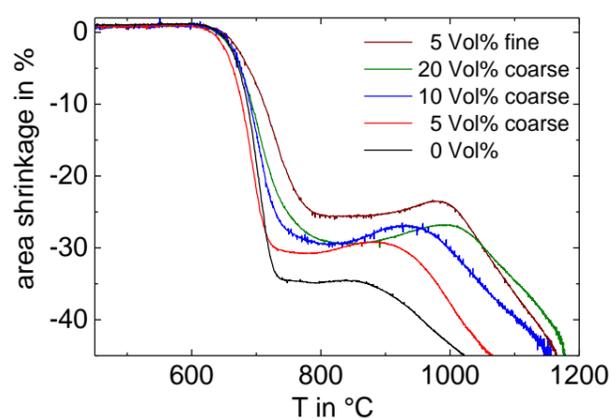


Figure 2
HM relative area shrinkage for varied ZrO_2 content vs temperature during heating at 10K/min

Experimental

Glass powder

- Soda lime silicate glass (NCS)
- Crushing in steel mortar
- Sieving to 56–250 μ m
- Milling 5min (ZrO_2 jars and balls) in water
- Drying 30 min at 120°C

Glass matrix composites (GMC)

- Zirconia particles:
 - fine: $D_{50} = 0.78\mu$ m, $D_{90} = 2.54\mu$ m
 - coarse: $D_{50} = 11.3\mu$ m, $D_{90} = 39.5\mu$ m
- Dry mix-milling in a planetary ball mill (1min, 3400rpm, ZrO_2)

Viscosity

- Heating microscopy [1]

Sintering

- Heating microscopy (10K/min)

Crack initiation

- Vickers indenter (19,62N, 10s)

Crack healing

- Interrupted isothermal annealing steps (heating microscope)

Results

Zirconia content increases viscosity (Fig.1) and delays sintering (Fig.2). 5 Vol% of fine particles have a stronger effect than 20 Vol% of coarse particles although the effective viscosity seems to be comparable (Fig.1).

Crack healing in NCS glass is delayed by crack widening due to global viscous flow minimizing surface energy.

Increasing filler content decreases the ability of global viscous flow and prevents widening (e.g. Fig.3 lower left micrograph) which results in a faster crack healing.

Using fine particles, global viscous flow is nearly completely stopped and cracks heal only in encapsulated glassy areas.

Conclusion

Encapsulated glassy regions in a rigid matrix allow local crack healing and prevent global viscous flow, which accelerates corrosion phenomena. These demands are perfectly combined in a gyroid-like or foamy microstructure.

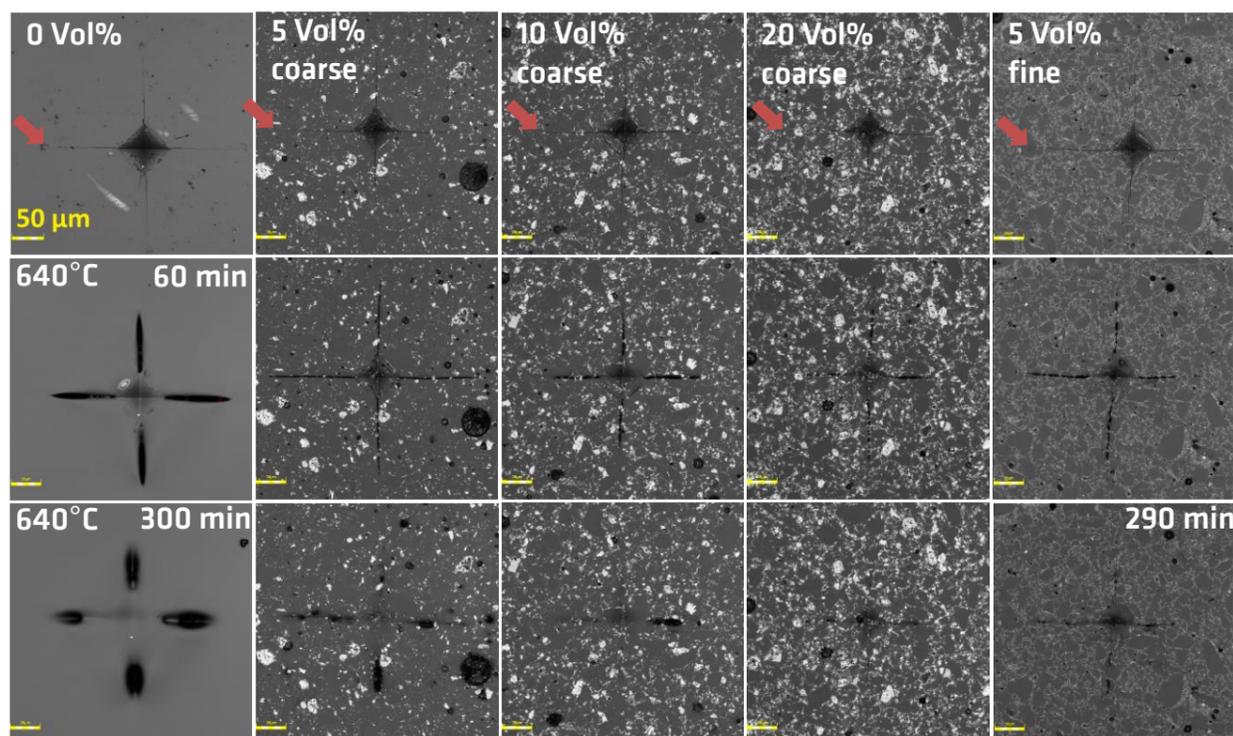


Figure 3
Laser scanning micrographs of Vickers indentations in NCS/ ZrO_2 composites with different filler content during isothermal crack healing at 640°C for 60 min and ca. 300 min

References:

- [1] M.J. Pascual, A. Duran, M.O. Prado; A new method for determining fixed viscosity points of glasses, Phys. Chem. Glasses **46** (2005), 512-520