Karlsruhe Institute of Technology

TECHNISCHE UNIVERSITÄT DARMSTADT

IMP

IAM Institute for Applied Materials

Fabrication and potential of tunable $Ba_{0.6}Sr_{0.4}TiO_3$–$Mg_3B_2O_6$ composites for microwave applications

Institute for Applied Materials (IAM-KWT) – Ceramic Materials and Technologies

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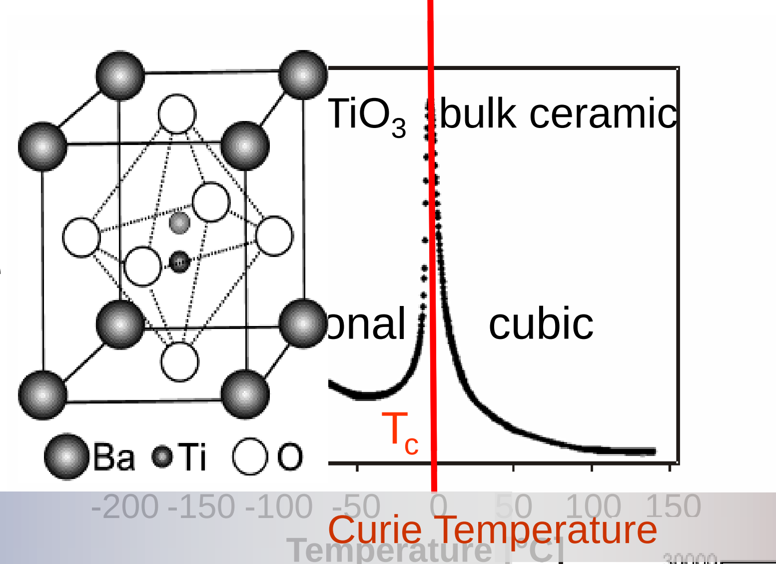
90th DKG Annual Conference & Symposium on High-Performance Ceramics 2015, Bayreuth, Germany

System $Ba_{1-x}Sr_xTiO_3$ (BST)

Ferroelectric Phase | Paraelectric Phase

Tetragonal Crystal Structure

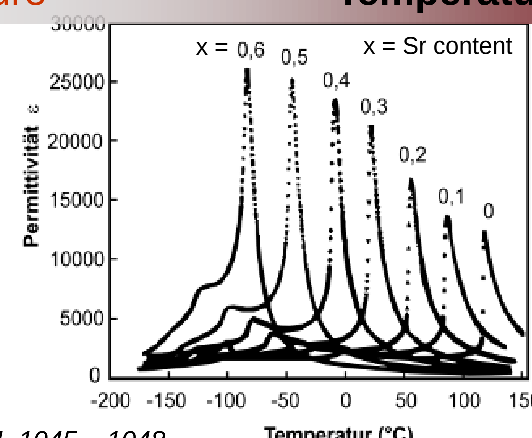


Cubic Crystal Structure

Temperatur

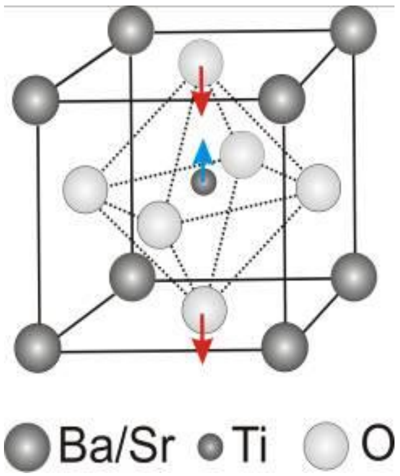
<u>For microwave applications:</u>

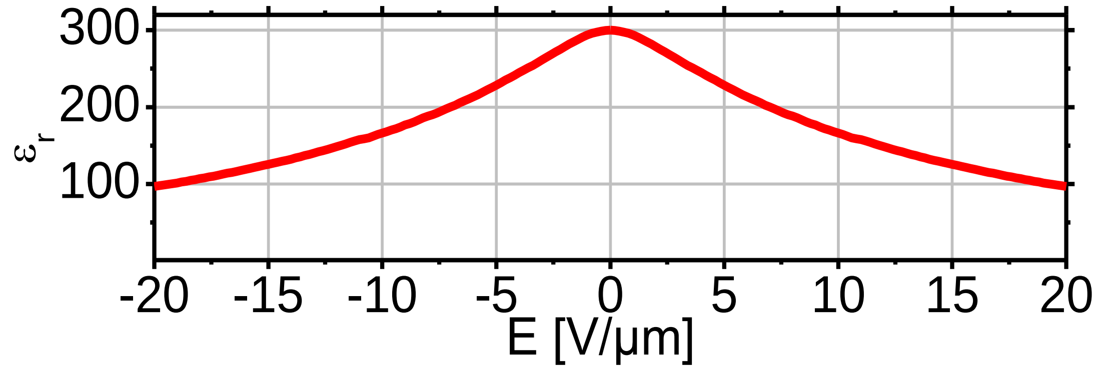
- usage of paraelectric phase
- application at RT→ $Ba_{0.6}Sr_{0.4}TiO_3$



Jeon, J., *J. Eur. Ceram. Soc., 2004, 24, 1045 – 1048*

$Ba_{0.6}Sr_{0.4}TiO_3$ as tuneable dielectric material





BST shows a non-linear dependancy of the permittivity on a static E-field

Displacement of Ti^{4+}-ion through an external electric field

- *no power consumption*
- *high linearity*
- *fast tuning speed*

Dielectric tunability
$\tau_{\varepsilon}(E) = \frac{\varepsilon_r(E=0) - \varepsilon_r(E)}{\varepsilon_r(E=0)}$

Microwave components based on BST thick-films

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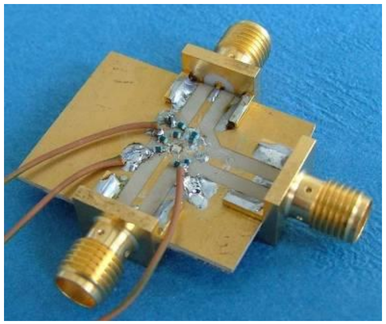
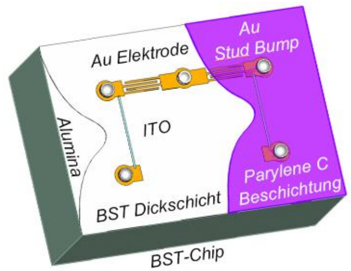
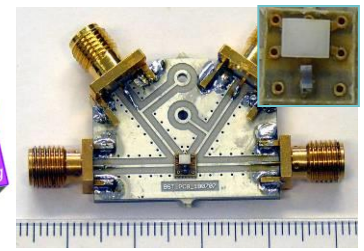
Tunable filter



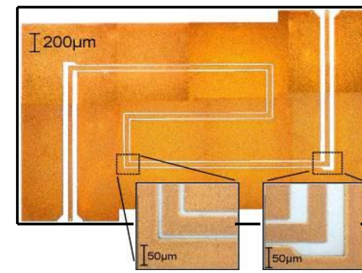
Tunable matching network



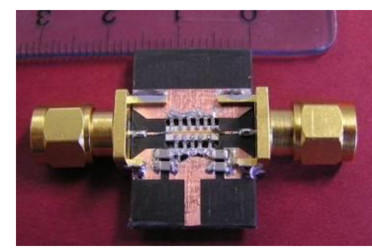


Phase shifter

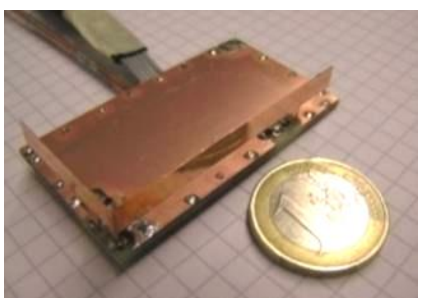
CPW based phase shifter



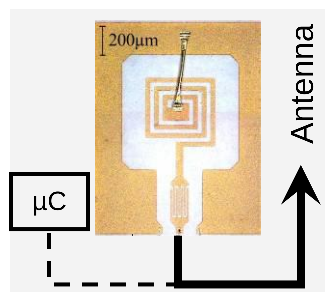
Left-handed phase shifter



Multiband antenna



RF-ID modulator



Microwave Engineering, Technical University Darmstadt

http://www.mwe.tu-darmstadt.de/de/fachgebiete/mikrowellentechnik/forschung/ferroelectrics/ferroelectrics.html

Microwave components based on BST thick-films

Karlsruhe Institute of Technology

Tunable filter

Tunable matching network

Phase shifter

CPW based phase shifter

Restrictions:

1.) High permittivity (ε_r = 200-300, porous BST thick-films)

2.) High dielectric loss for frequencies over 20 GHz (tan δ > 0.1)

Left-handed phase shifter

→ Development of composite BST with high-Q-dielectric

Perspective application: Tunable substrate with low ε_r and tan δ

Microwave Engineering, Technical University Darmstadt

http://www.mwe.tu-darmstadt.de/de/fachgebiete/mikrowellentechnik/forschung/ferroelectrics/ferroelectrics.html

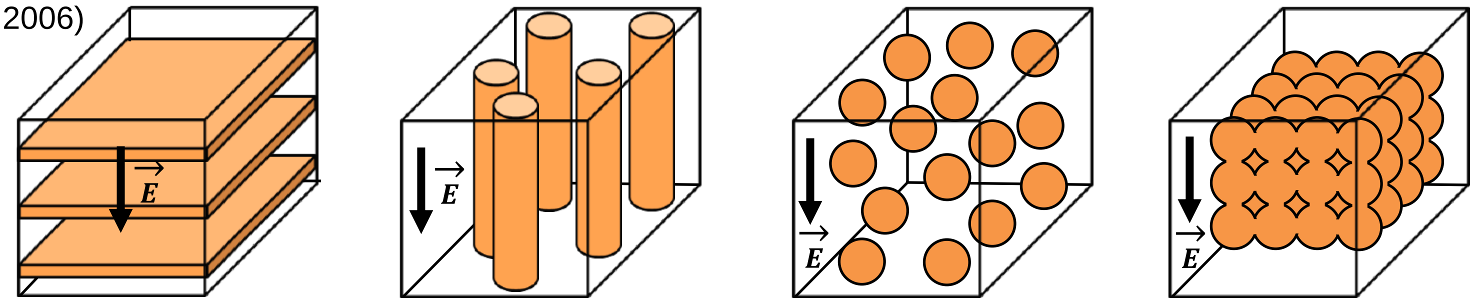
Composite models

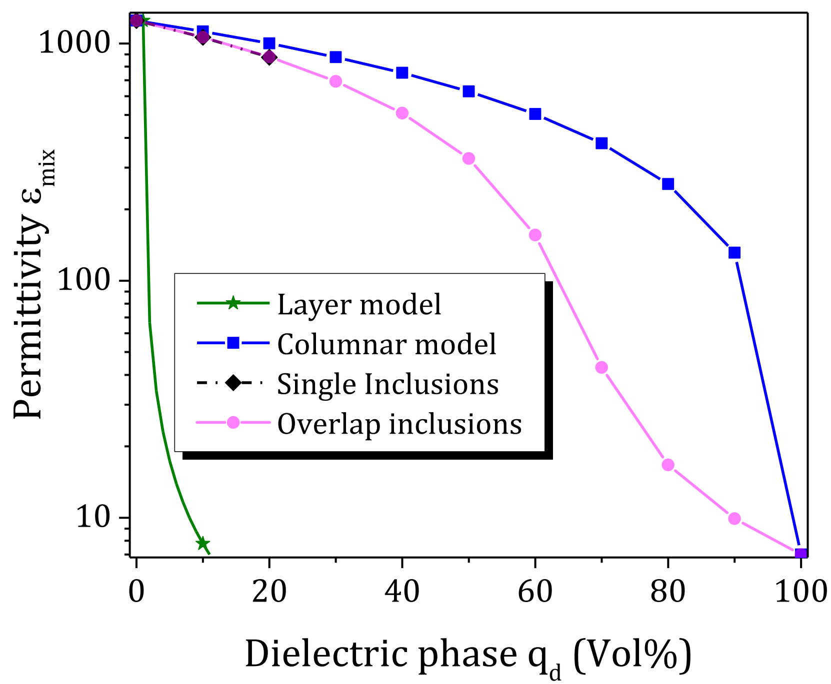
Layer model | Columnar model | Single Inclusions | „Overlap“ Inclusions

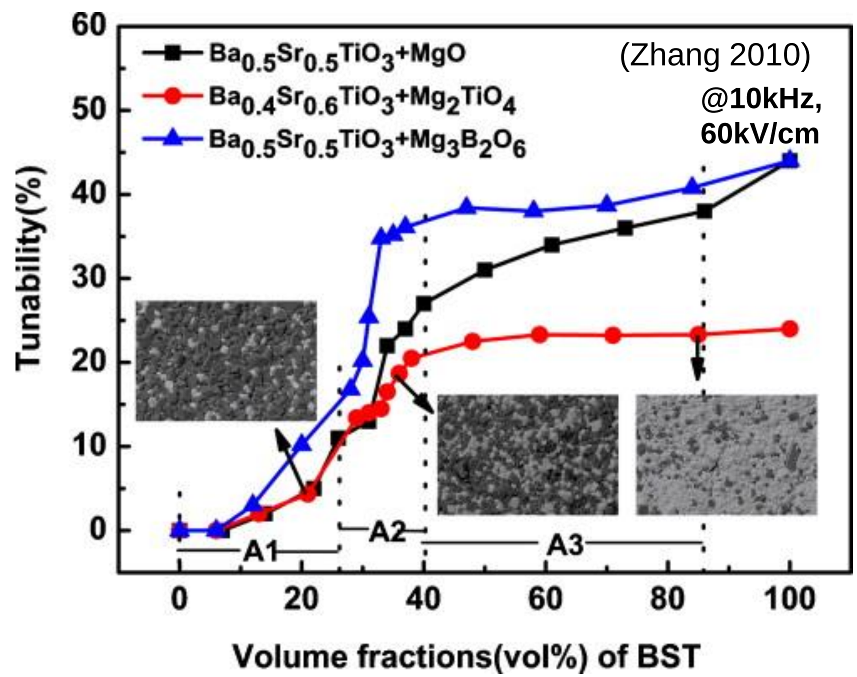
(based on Sherman, 2006)

Dielectric

Ferroelectric



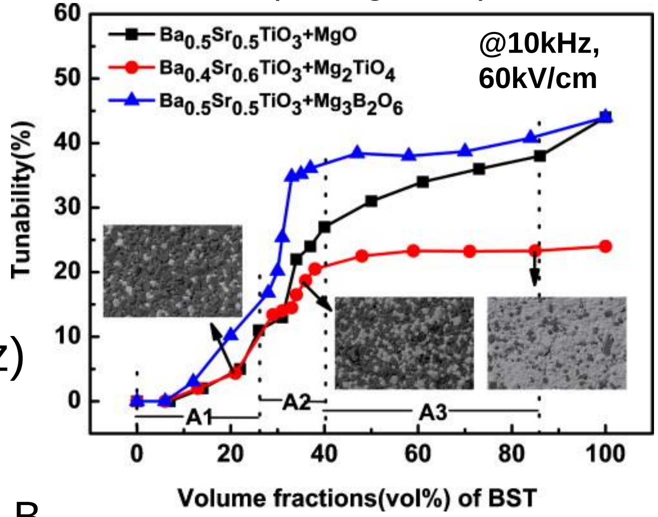




Ferroelectric – dielectric composites

Goal: Shifting of percolation threshold to lower BST content through tuning of microstructure

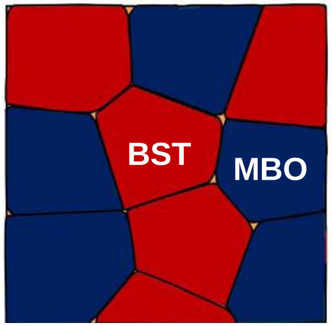
(Zhang 2010)



Composite system: $Ba_{0.6}Sr_{0.4}TiO_3$-$Mg_3B_2O_6$ (MBO)
(MBO: ε_r= 7.2, Q_f = 320 900 GHz)

Approaches:

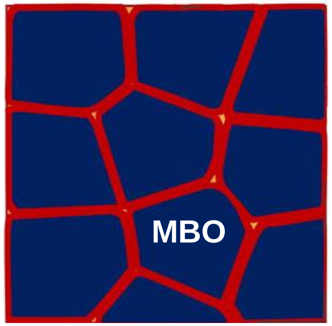
Route A



Granular matrix composite

→ Mixing of BST and MBO

Route B



Coating composite

→ Infiltration and coating technique

Granular matrix composites – Fabrication

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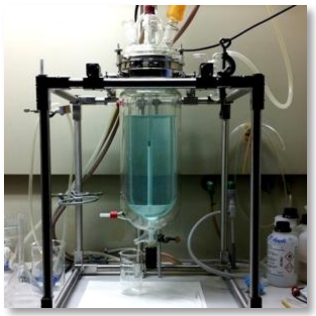
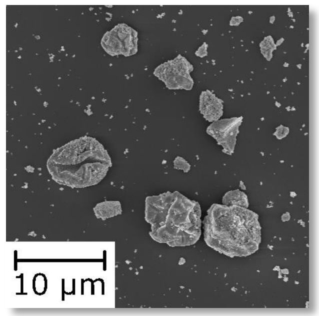
BST processing



Sol-Gel Synthesis

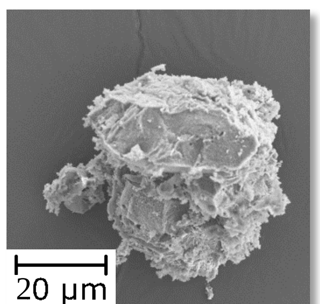


Spray drying + calcination

Milling

Mixed oxide route (H_3BO_3-MgO)

Drying + calcination



Milling

MBO processing

Mixing & Homogenisation (low power)

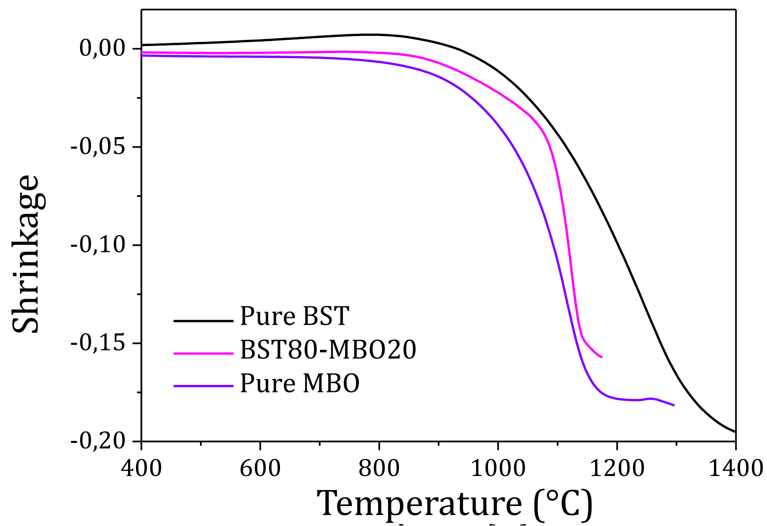
BST

MBO

$5 \cdot d_{BST} = d_{MBO}$

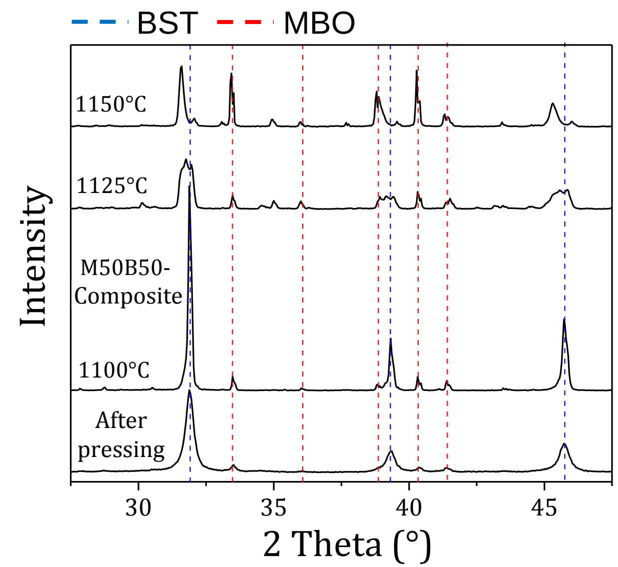
Granular matrix composites – Sintering

Dilatometry



→ Fully Densification 1100–1200°C

XRD



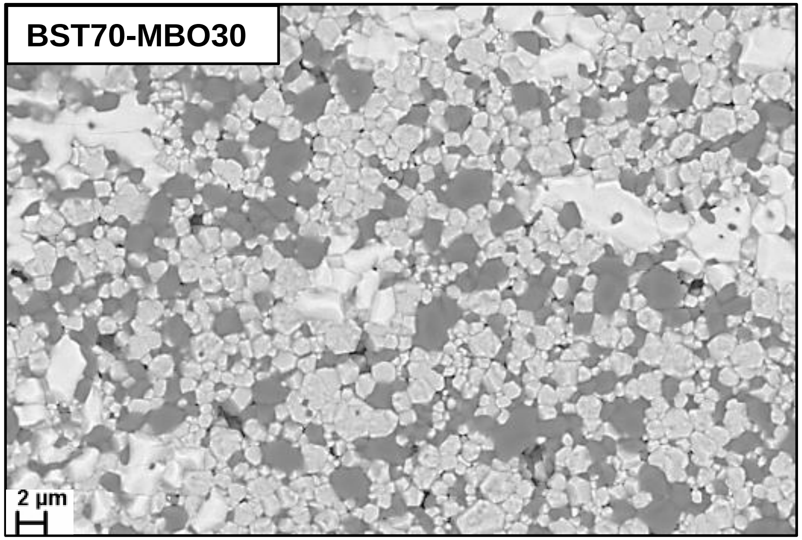
→ Side reactions above 1100°C

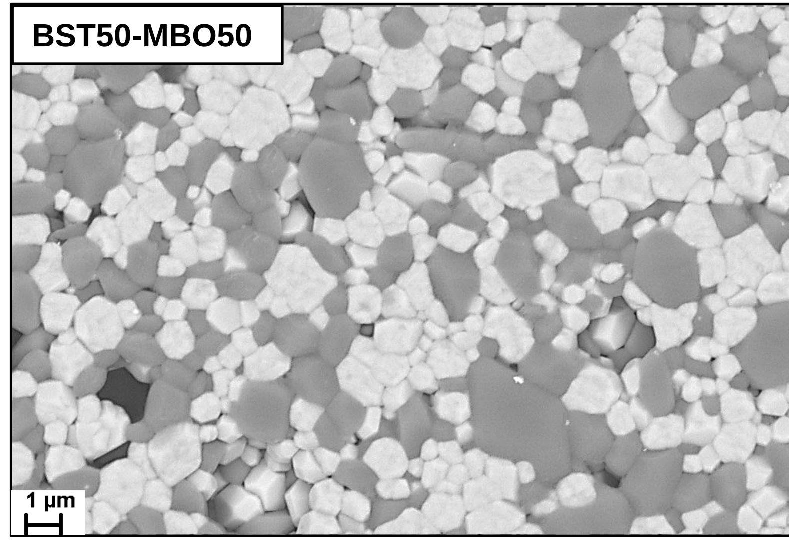
→ Trade-off sintering programme 1100°C/2h

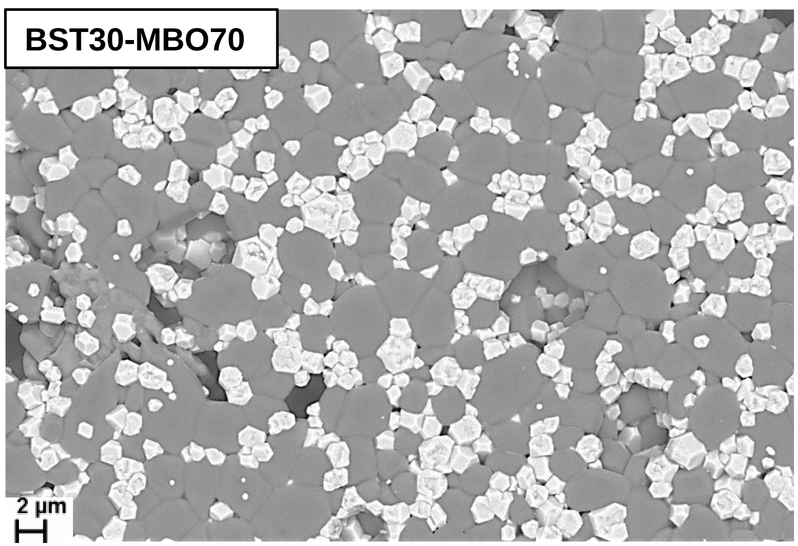
Density (based on Archimedes method)

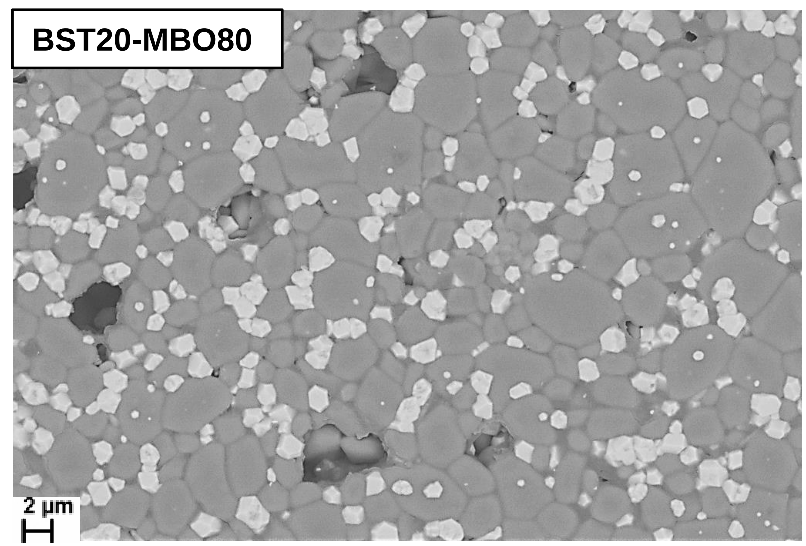
Composite BST:MBO	90:10	80:20	70:30	40:60	50:50	40:60	30:70	20:80	10:90
Relative density (%)	93,0	92,4	91,9	91,8	92,8	90,9	89,0	92,3	95,6
Open porosity (%)	3,7	3,4	5,6	4,6	3,5	3,8	4,4	0,5	0,7

Microstructure after sintering (1100°C/2h)

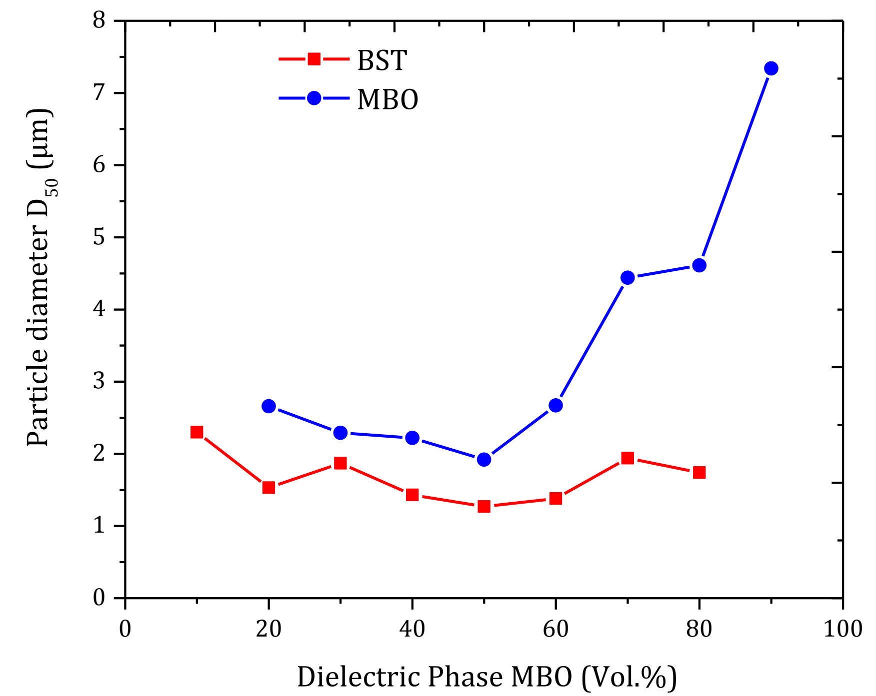








Microstructure after sintering (1100°C/2h)

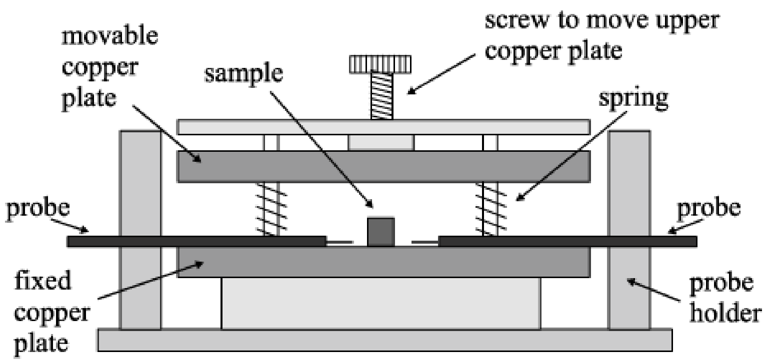


→ Higher sintering activity of MBO

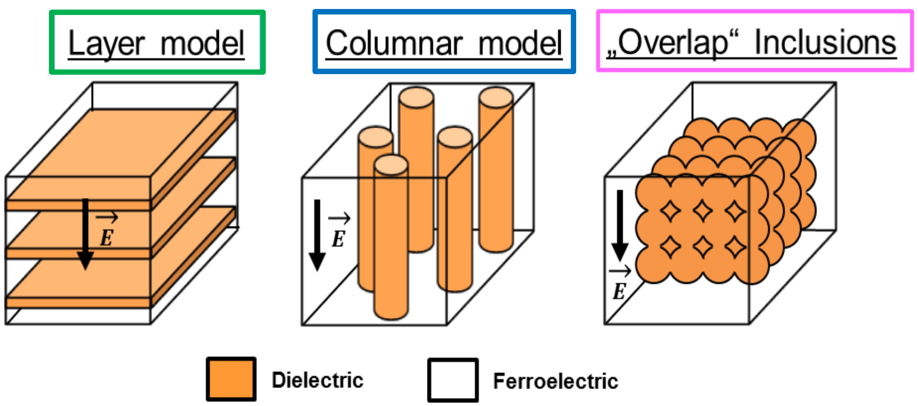
→ Constant BST grain size distribution

Dielectric Properties – permittivity

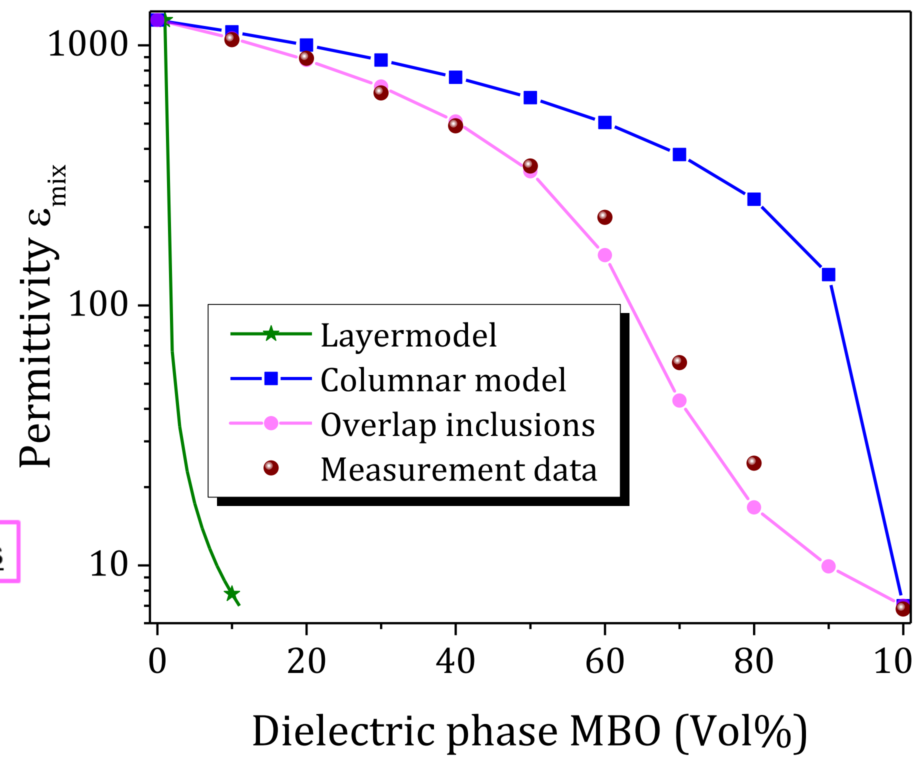
Measurement setup
(Hakki-Coleman)



(Dube et al. 1997)



Permittivity



→ Good compatibility

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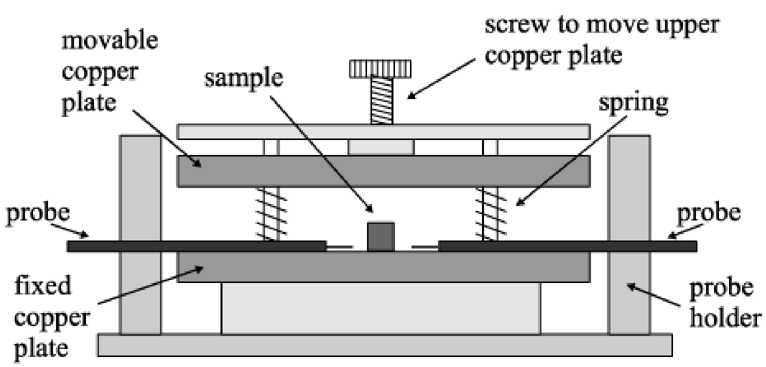
Dielectric Properties – dielectric loss

Measurement setup

(Hakki-Coleman)

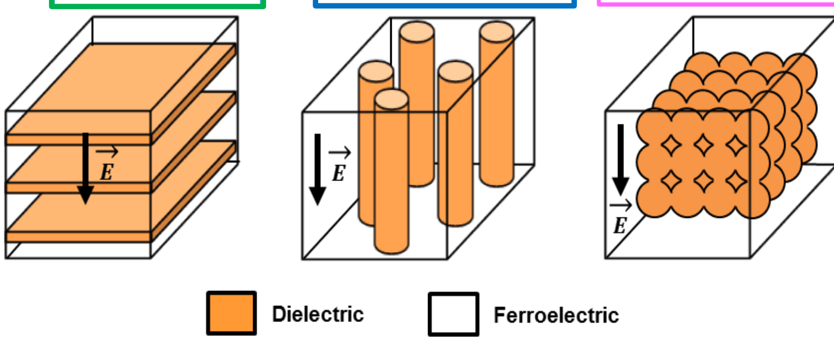


(Dube et al. 1997)

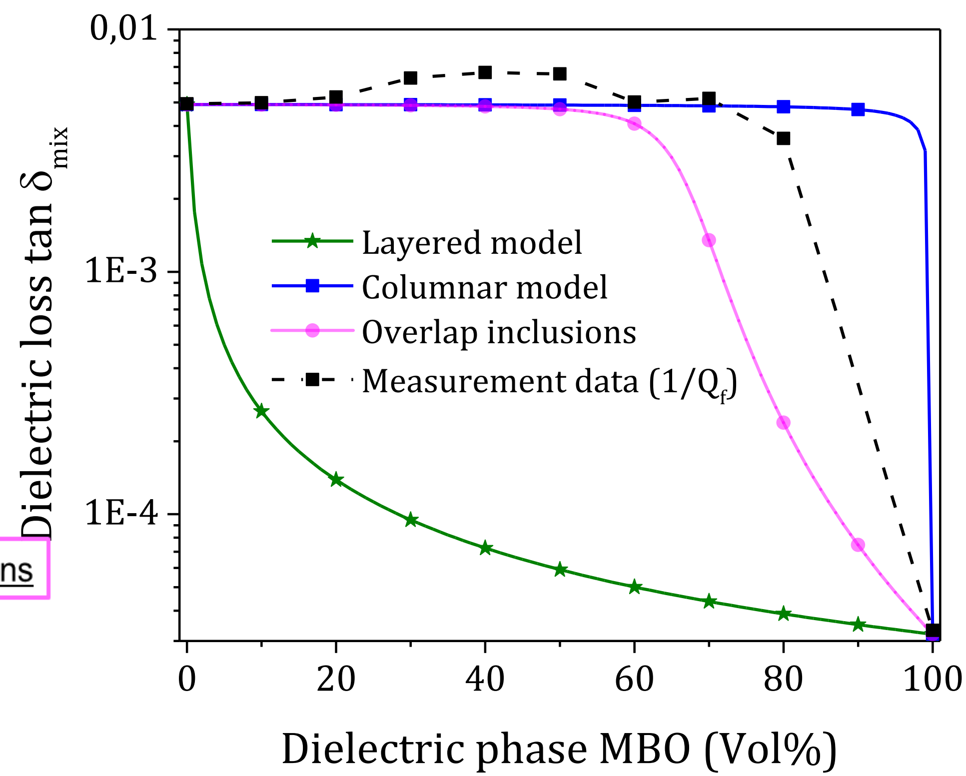
Layer model

Columnar model

„Overlap“ Inclusions



Dielectric loss



→ Extrinsic loss due to composite effects

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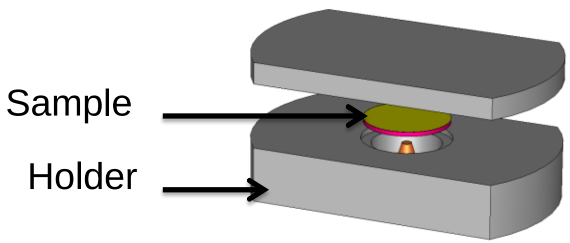
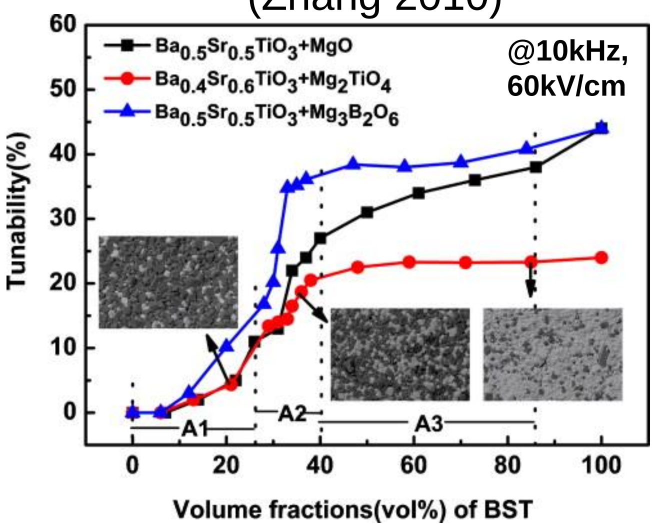
Dielectric Properties – tunability

Measurement setup

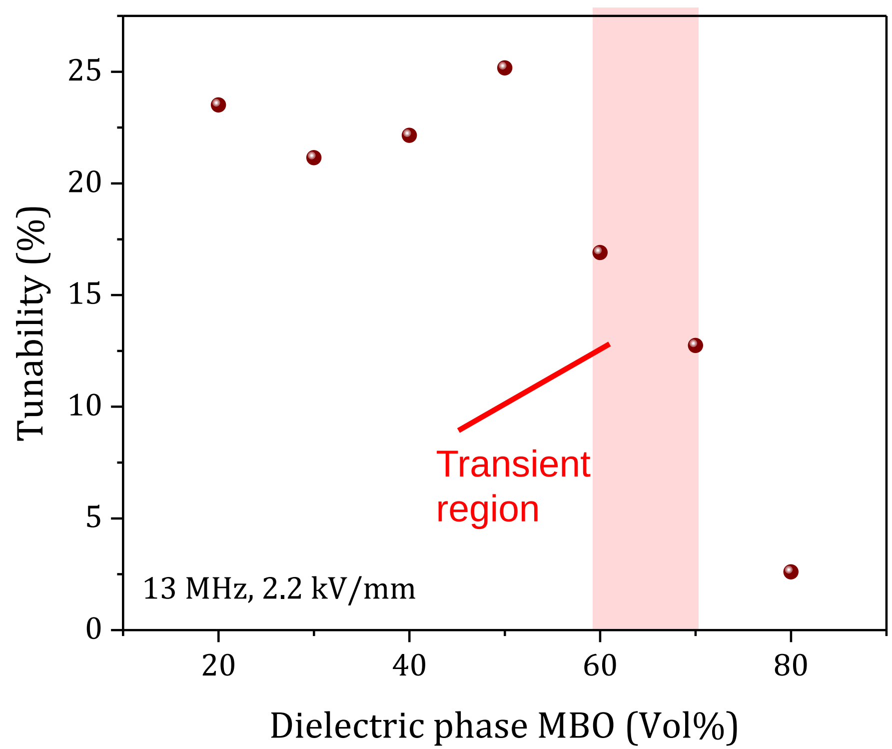
(Sample height 500 µm)



(Zhang 2010)

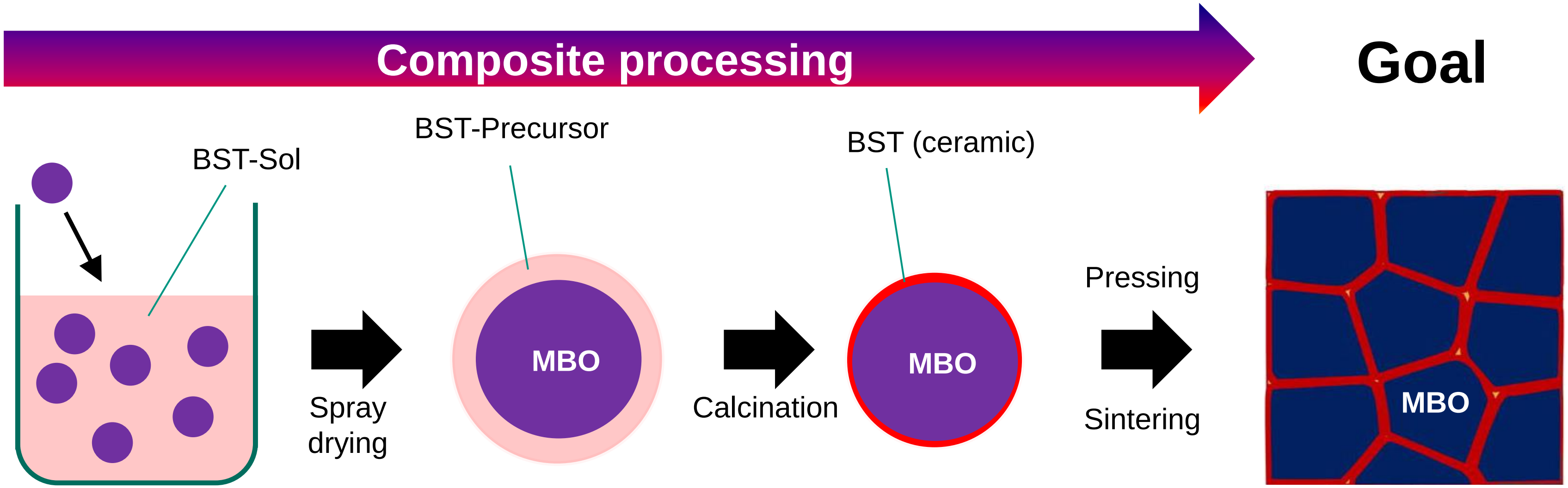


Tunability



→ No shift of percolation threshold

Outlook – Coating composites (Core-Shell)

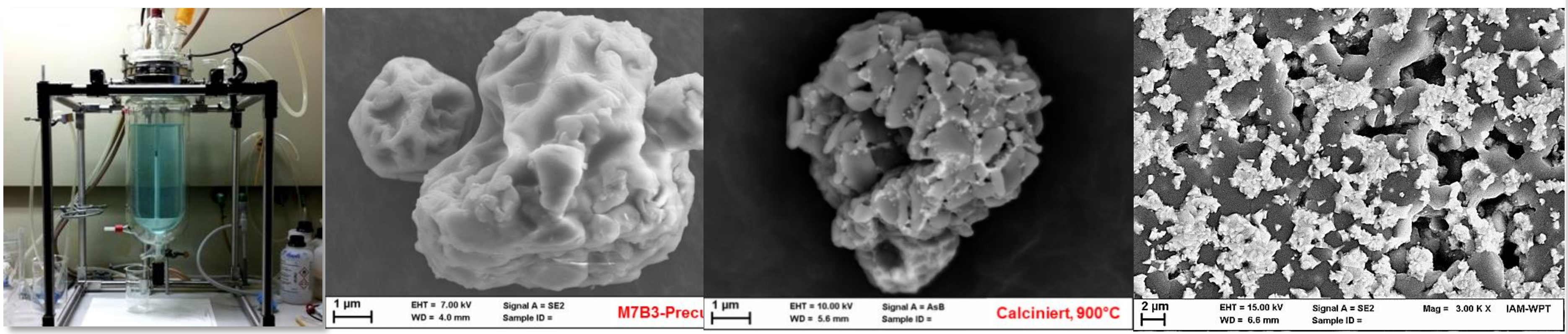


Dispersing of MBO in BST sol

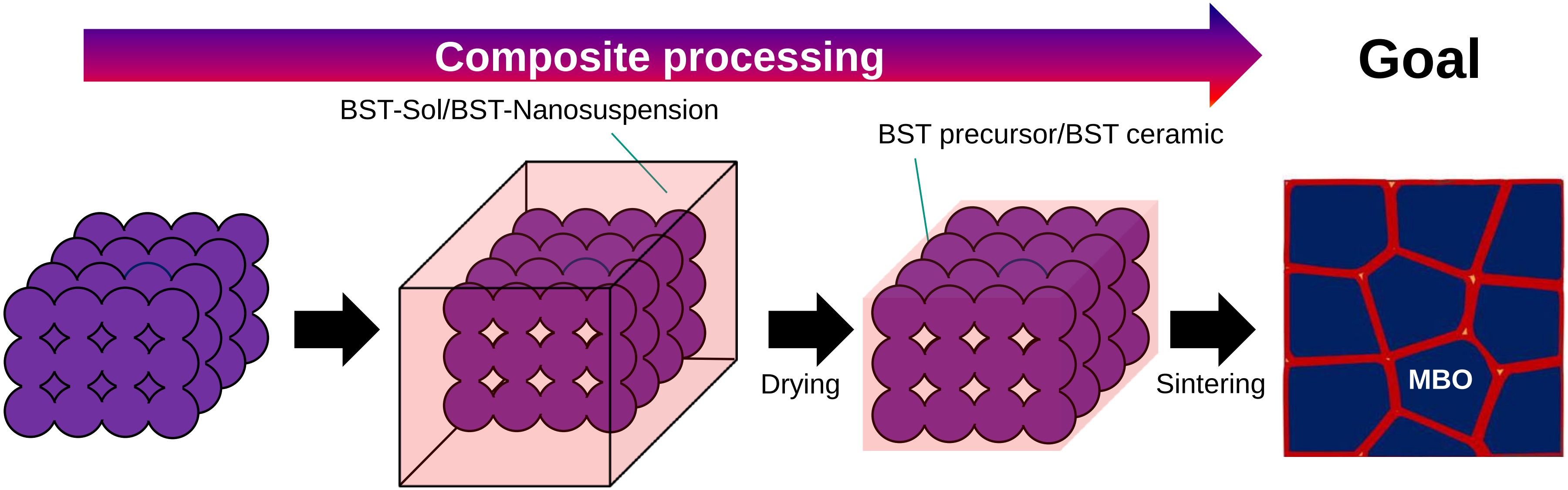
Precursor-Coated MBO

BST-Coated MBO

Sintered Composite (BST30-MBO70)



Outlook – Coating composites (Infiltration)

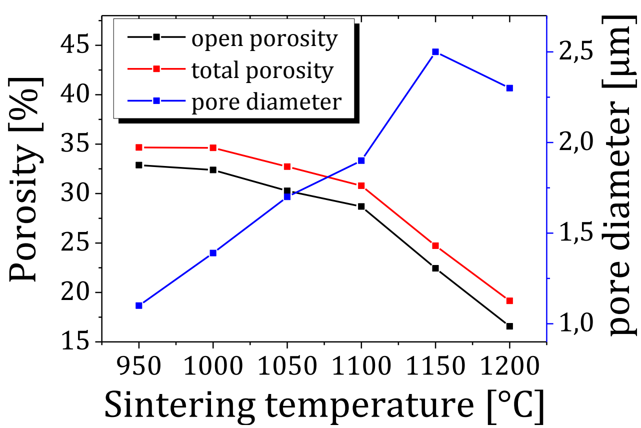


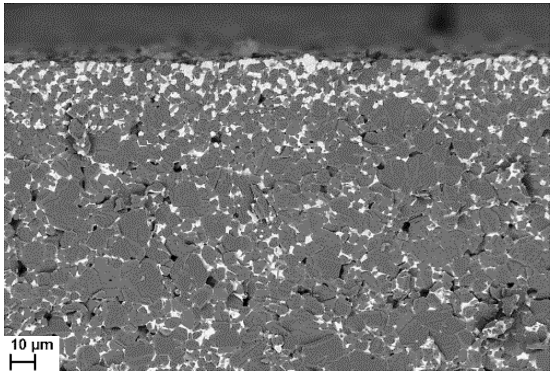
Fabrication of porous MBO infiltrant

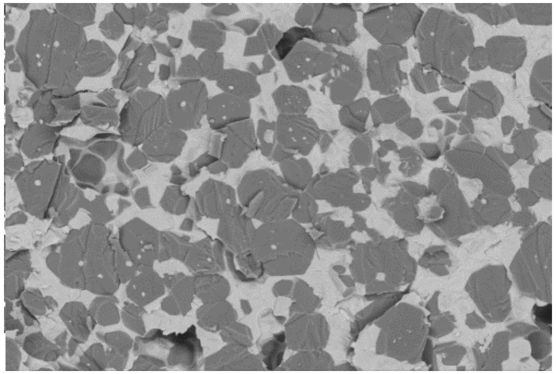
Infiltration of BST sol/ BST nanosuspension

Infiltrated MBO

Sintered composite







Multiple infiltration cycles

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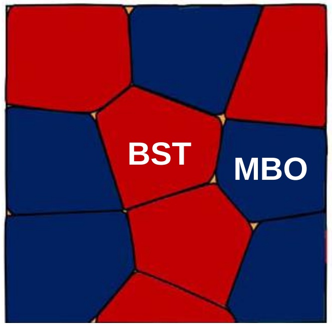
Summary – granular matrix composites

- **Composite system BST-$Mg_3B_2O_6$**

  - potential system for tunable substrate with low ε_r and tan δ

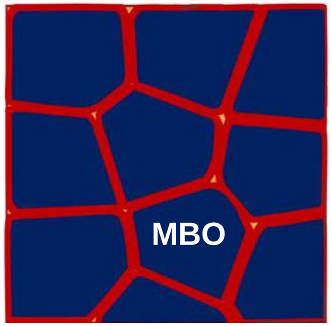
- **Granular matrix composites**

  - stepwise processing and milling of BST and MBO
  - homogeneous microstructure
  - higher sintering activity of MBO particles



- **Dielectric properties**

  - Permittivity in accordance to spherical inclusion modell
  - Dielectric loss dominated by extrinsic loss
  - Tunability reduced with collapse of BST network



- **Outlook**

  -Fabrication of coating composites

Thank you for your attention!

The authors thank the German Research Association (DFG) in Bonn for its generous financial support of this research project,