



# Microwave sintering of $\text{Ba}_{0,6}\text{Sr}_{0,4}\text{TiO}_3$ Thick-Films

F. Paul, W. Menesklou, J. R. Binder, G. Link, X. Zhou, J. Haußelt

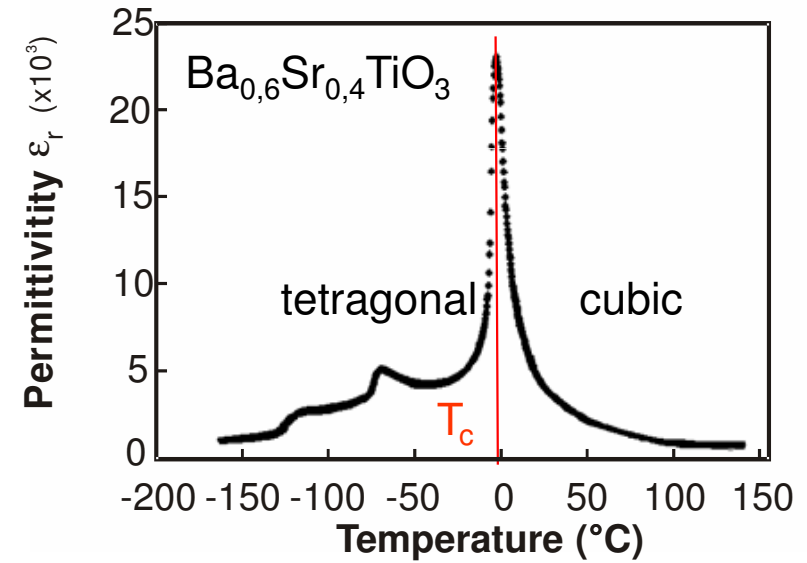
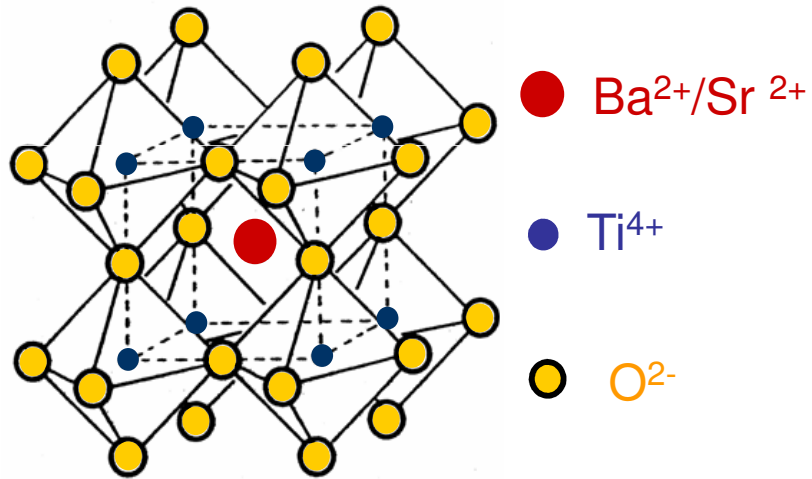
Laboratory for Materials Processing  
Institute of Microsystems Engineering - IMTEK  
University of Freiburg, Germany

UNI  
FREIBURG

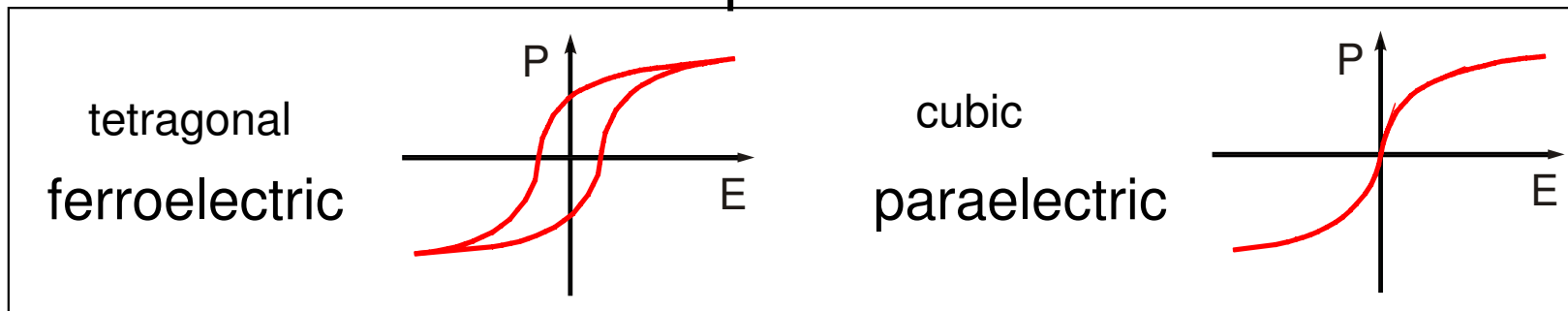


ISIF 2012, 21st June 2012, Hong Kong

# Introduction



## P-E-Loops

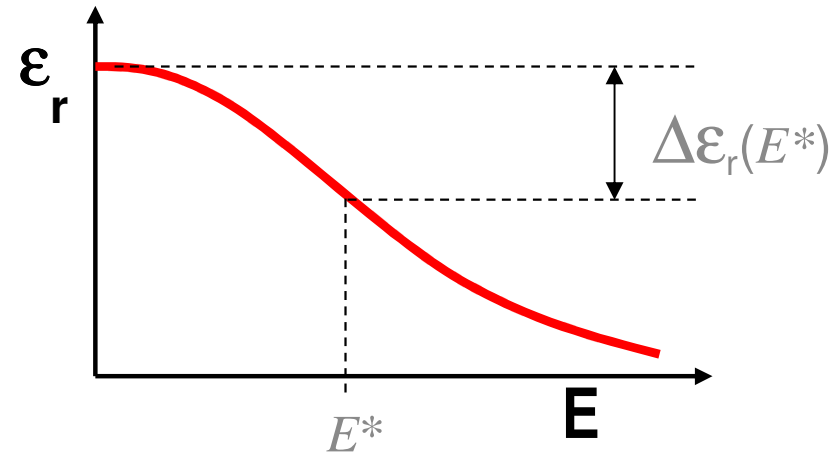
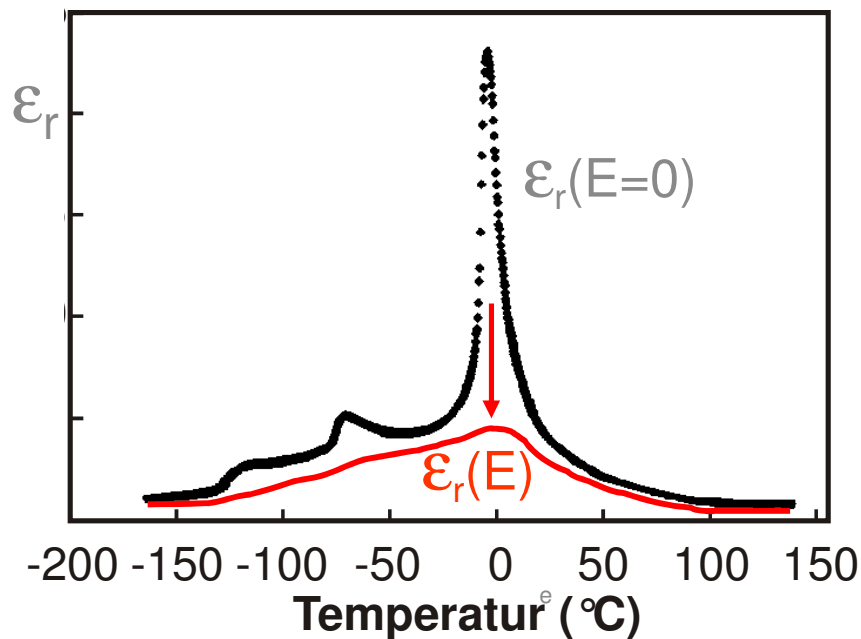


# Introduction

## Decrease of permittivity

tunability : decrease of permittivity through external el. field

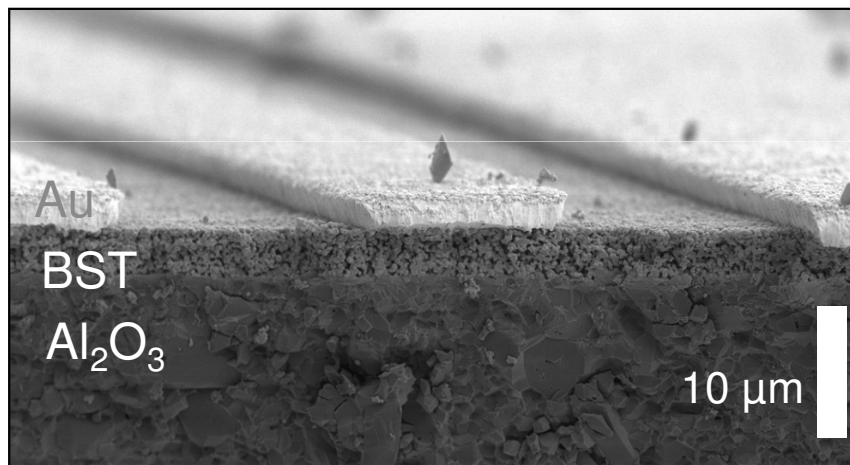
$$\text{tunability } \tau(E^*) = \frac{\Delta\epsilon_r(E^*)}{\epsilon_r(E=0)}$$



# Motivation

## functionality

- permittivity of  $\text{Ba}_{0.6}\text{Sr}_{0.4}\text{TiO}_3$  shows nonlinear tunability under static E-field

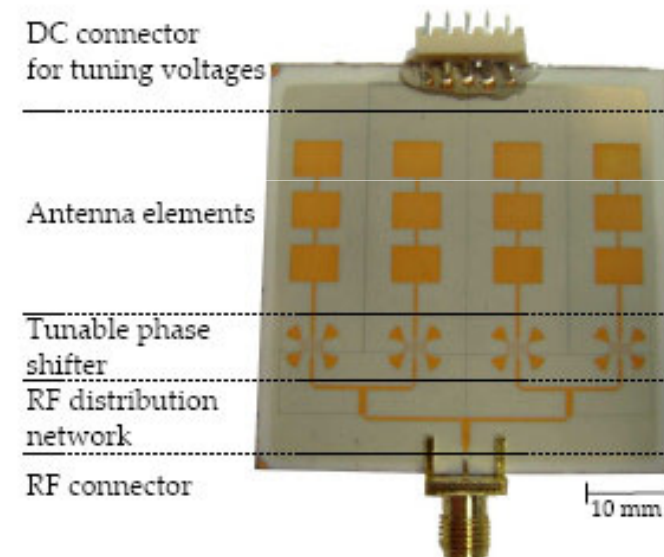


cross-section of coplanar waveguide

## application

tunable RF-components

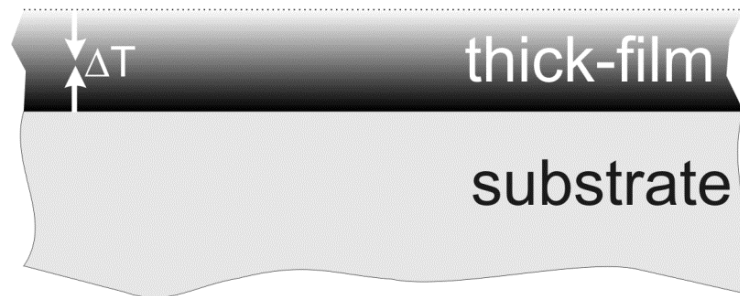
- reconfigurable circuits
- phase shifters (delay lines)
- electronically steerable oscillators & filters



passive phase array antenna

# Microwave sintering (MWS)

conventional



- heating from „outside“ in conventional sintering

- inverse heating profile in MWS

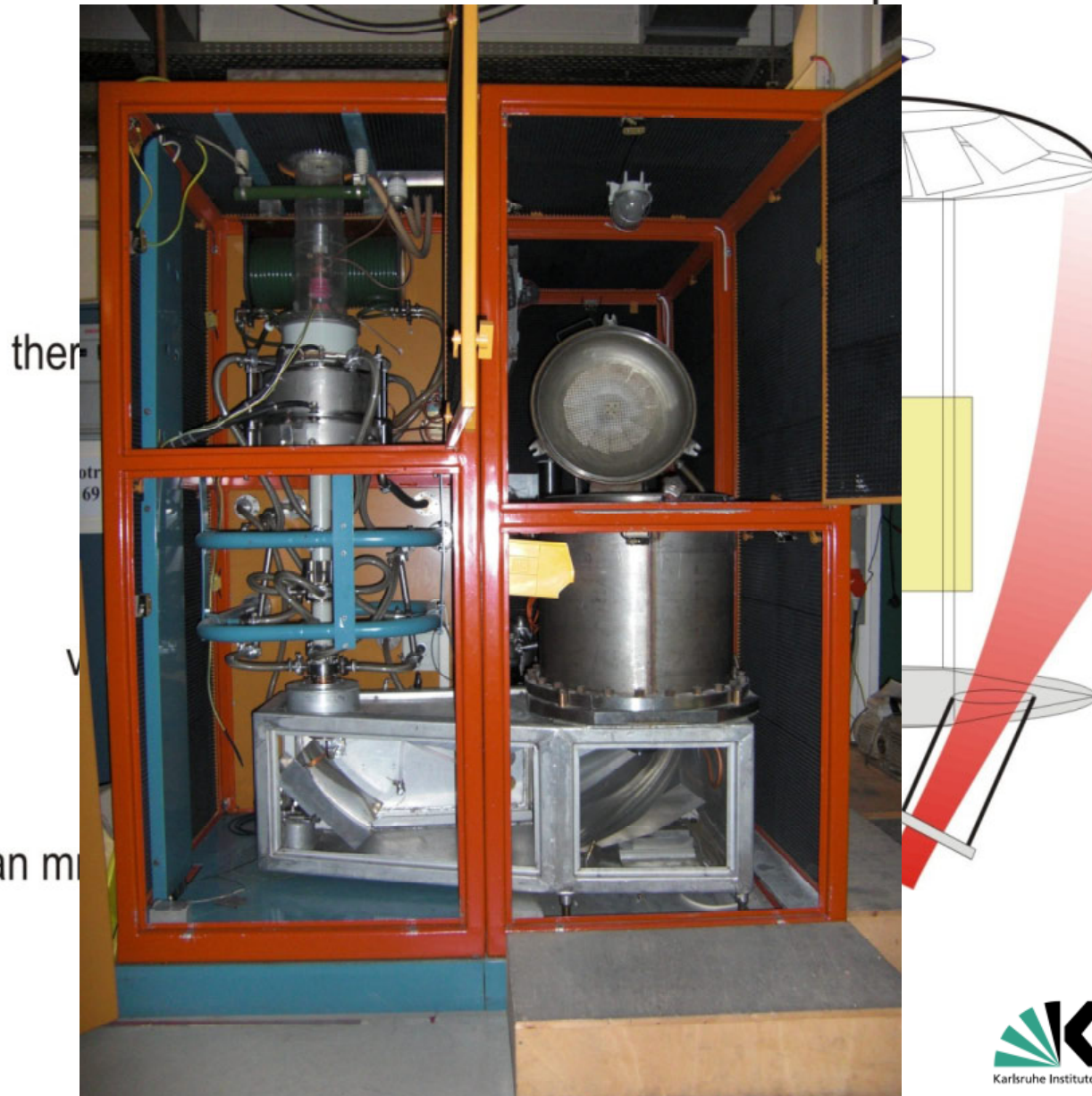
microwave



- regions with high dielectric loss heat up stronger

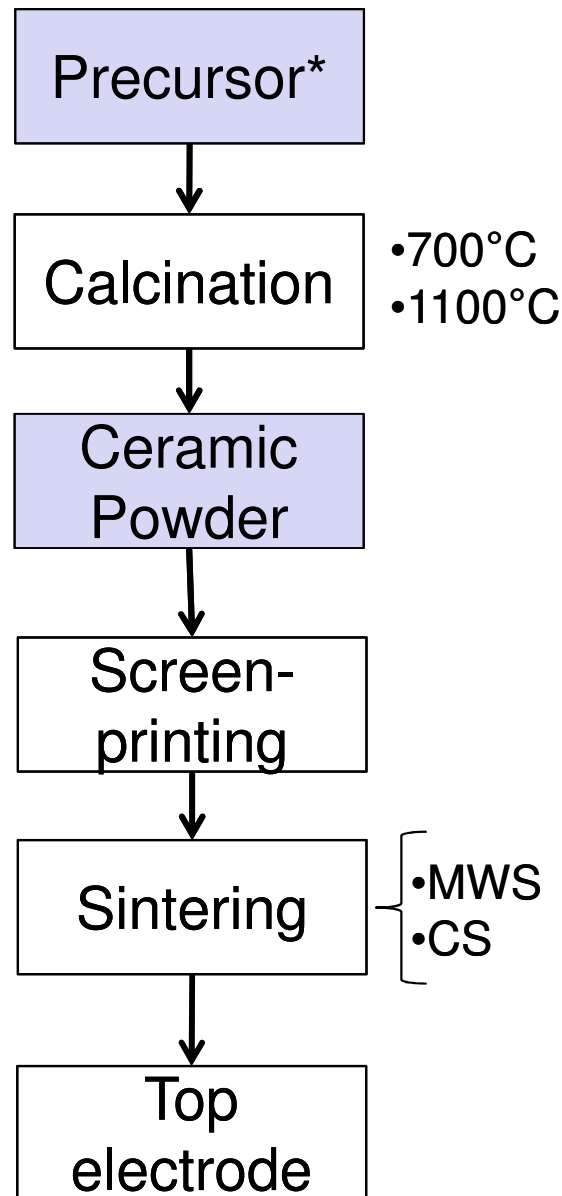
- short processing time and high heating rates

# Microwave sintering (MWS)



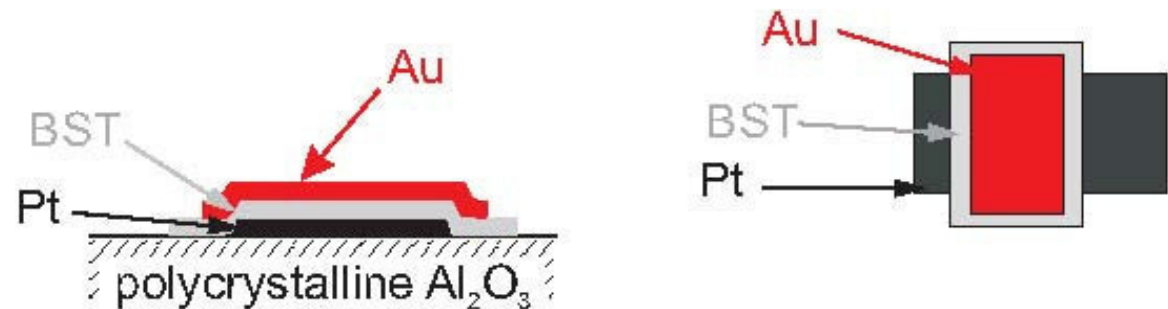
Gaussian m

# Specimen fabrication



thermal treatments in air

Name of specimen	Calcination temperature (CT)	conventional sintering (CS)	microwave sintering (MWS)
heating	5 K/min	5 K/min	20 K/min
cooling	max. 10 K/min	5 K/min	5 K/min
BST-CT700-CS	700°C, 1h	1200°C, 1h	-
BST-CT1100-CS	1100°C, 1h	1200°C, 1h	-
BST-CT700-MWS	700°C, 1h	-	1200°C, 20 min

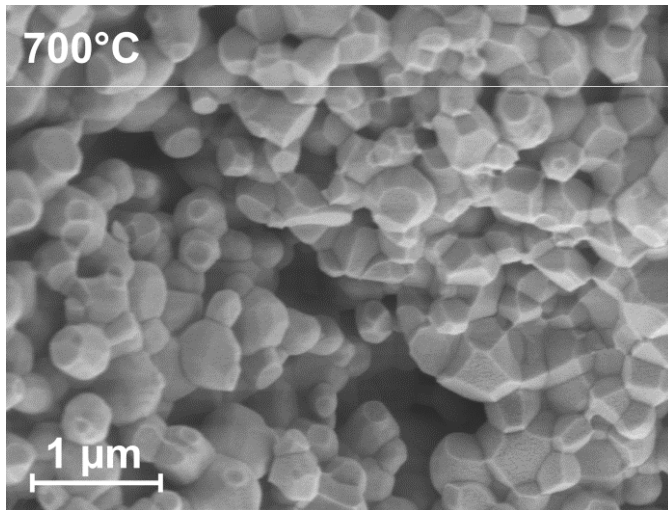


MWS: Microwave sintering  
 CS: conventional sintering

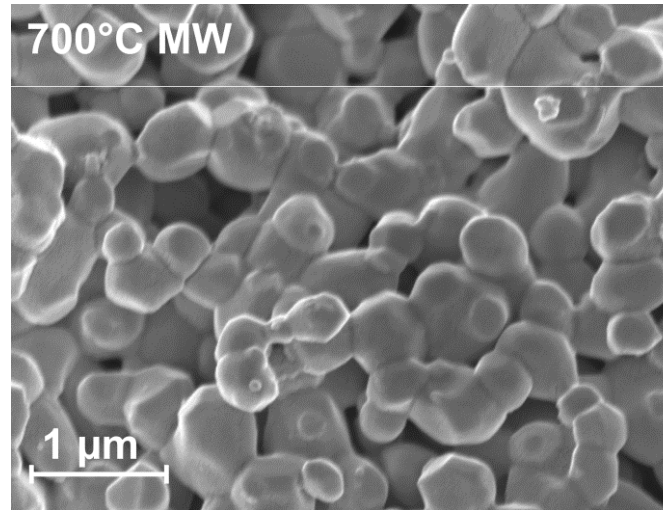
# Microstructure

## SEM cross-sections

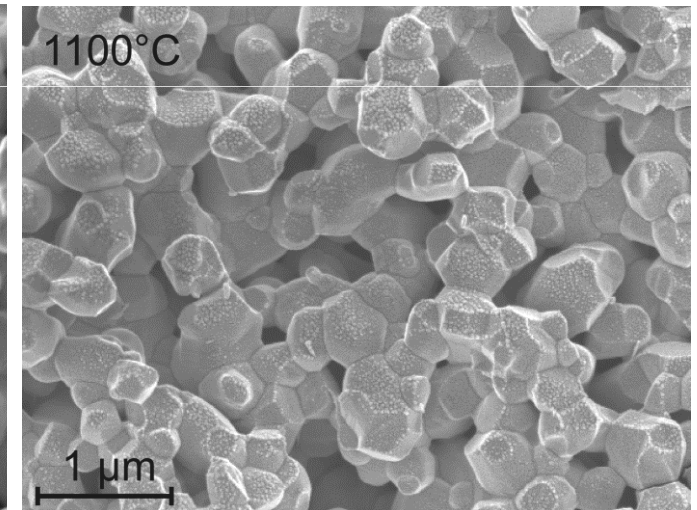
BST-CT700-CS



BST-CT700-MWS



BST-CT1100-CS



- calcined 700°C, 1h
- sintered conventionally 1200°C, 1h

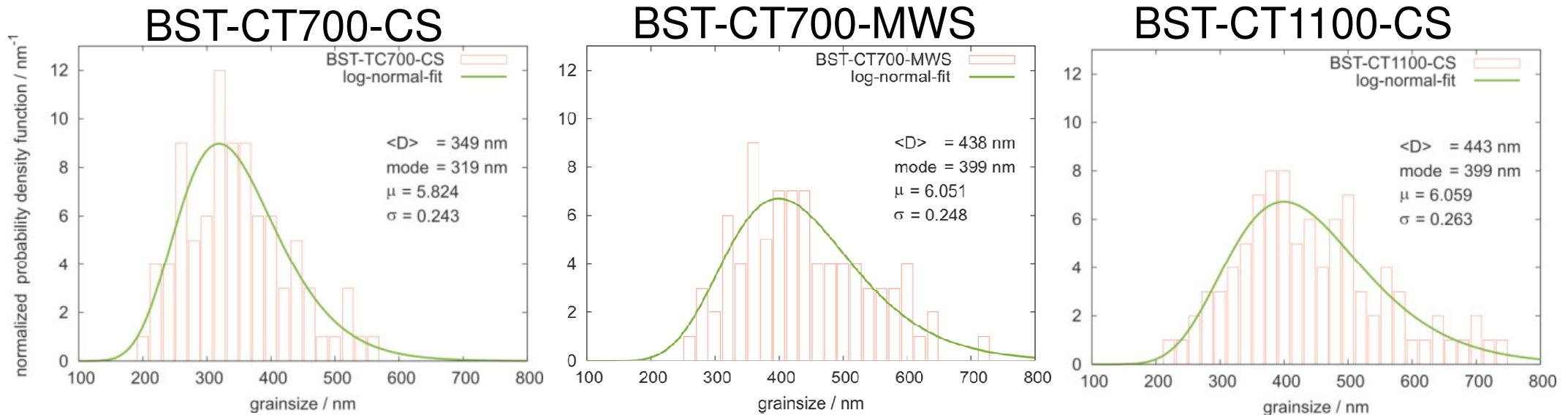
- Calcined 700°C
- sintered by microwave (30 GHz) 1200°C, 20Min.

- calcined 1100°C, 1h
- sintered conventionally 1200°C, 1h



# Microstructure

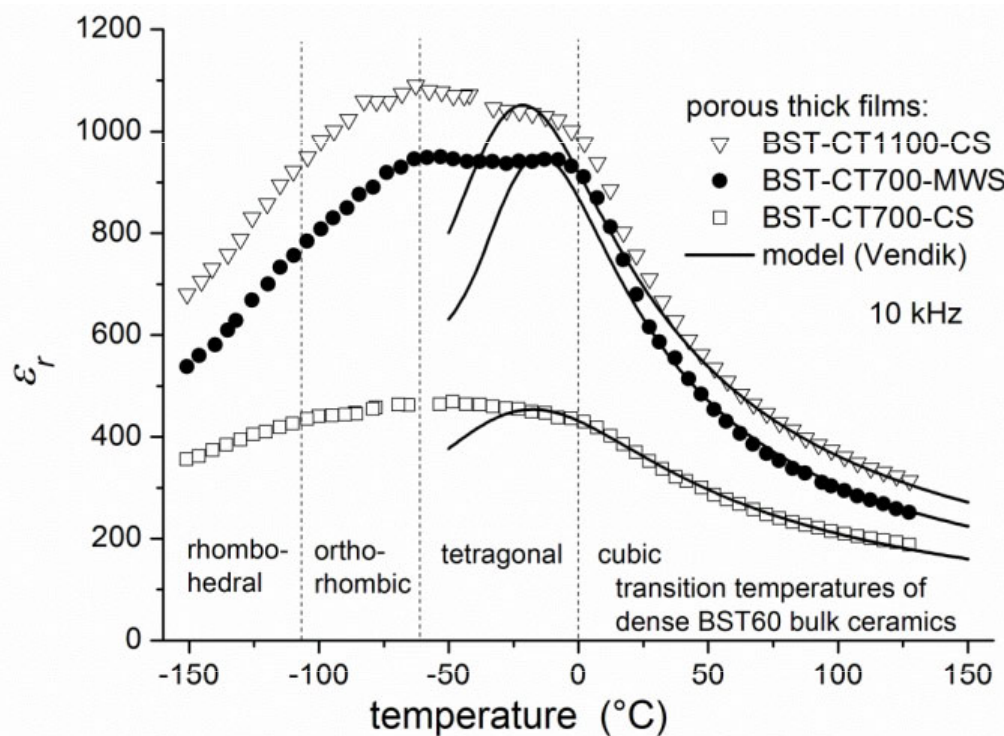
## grain sizes and porosities



name of film / sample	mean grain size $\langle D \rangle$ (nm)	$\sigma_D$ (nm)	mode (nm)	porosity (%)	thickness ( $\mu\text{m}$ )
BST-CT700-CS	349	85	319	41	10,5
BST-CT700-MWS	438	109	399	34	15,5
BST-CT1100-CS	443	116	399	33	19

# Dielectric properties

## Permittivity



- Low  $\epsilon_r$  compared to bulk ceramics
- Decreased temperature dependence
- Broad phase transition peaks
- High calcination Temp.: increases  $\epsilon_r$
- MWS.: increases  $\epsilon_r$
- $T_c$  is shifted to lower temperatures

modelling of the dielectric data after O. G. Vendik, S. P. Zubko, Journal of Applied Physics, 88(2000) 5343-5350

# Dielectric properties

## fitted parameters (Vendik model)

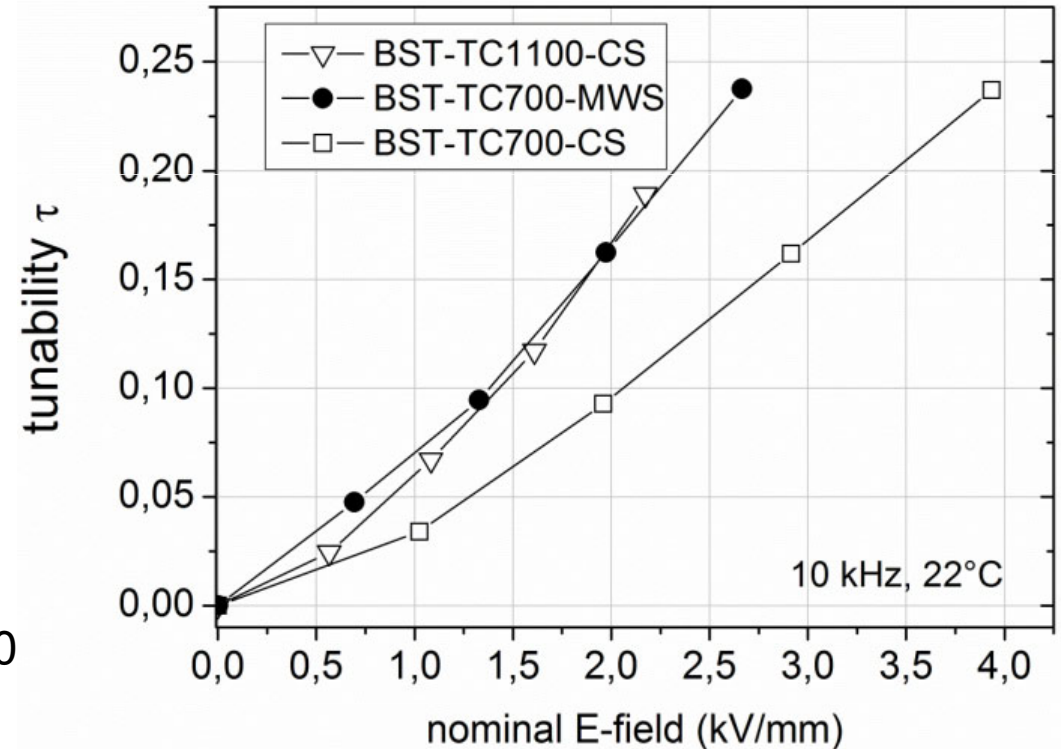
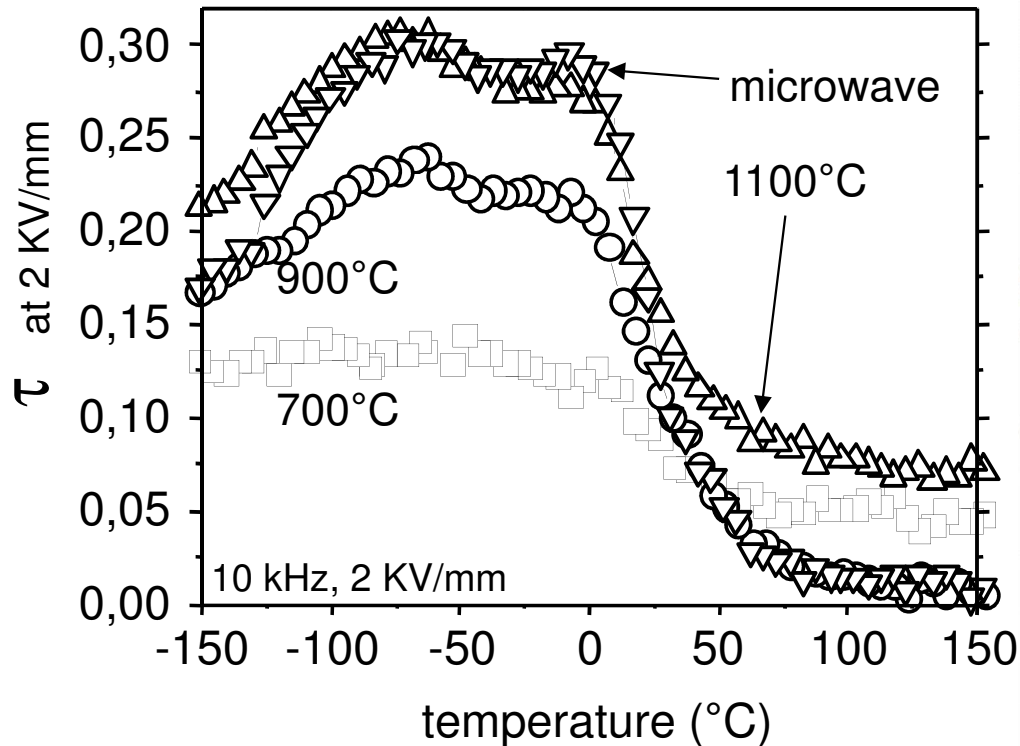


	$T_C / K$ ( $\pm 3$ )	$\xi_S$ ( $\pm 0.005$ )	mean grain size / nm	porosity / %
BST-CT700-CS	223	0.13	349	41
BST-CT700-MWS	240	0.055	438	34
BST-CT110-CS	230	0.073	443	33
BST60 ceramic	270	0.01*	-	-

- higher crystal quality after CT= 1100°C and MWS
- more bulky behavior after CT= 1100°C and MWS ( $T_C$ ,  $\xi_S$ )
- \*BST60 bulk-ceramics exhibit much lower values of  $\xi_S$  as suggested by Vendik ( $\xi_S > 0.1$ )

# Dielectric properties

## tunability



- high tunability due to high calcination temperature and microwave sintering
- increased tunability, compared to bulk ceramics with similar grain size

# Conclusion

---

- thermal treatment plays a crucial role in processing of BST ceramics and thick films
- Dielectric properties are heavily dependent on microstructure and crystal quality
- porous BST thick films show decreased
  - permittivity
  - $T_c$
  - temperature dependence
  - crystal quality as bulk ceramics
- MWS is a high impact sintering method, comparable to long lasting conventional heating and sintering

# Thank you for your attention

---



Thank you for collaboration and help:

- Dr. Menesklou, Dr. Binder, Dr. Link, Dr. Zhou, Karlsruhe Institute of Technology



- Prof. R. Jakoby, Dr. H. Maune, Dr. Giere, Microwave Engineering, University of Darmstadt



These results will be published in the Proceedings of ISIF 2012