

Influence of Lead Oxide Stoichiometry on the Microstructure and Characteristics of PZT Ceramics and Multilayer Actuators



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Project T2

Motivation, Methods and Objectives

Volatility of lead oxide creates inhomogeneous sintering atmosphere in industrial sized furnaces, therefore effects of variation in PbO content are relevant for quality of PZT multilayer actuators. Two lines of investigations on effects resulting from PbO content variation were followed:

- preparation of soft doped PZT ceramics with batch PbO content varying from 2 mol% deficit to 2 mol% PbO excess and
- fabrication of PZT multilayer actuators under different sintering setups providing different PbO vapor pressure and mass losses during sintering.

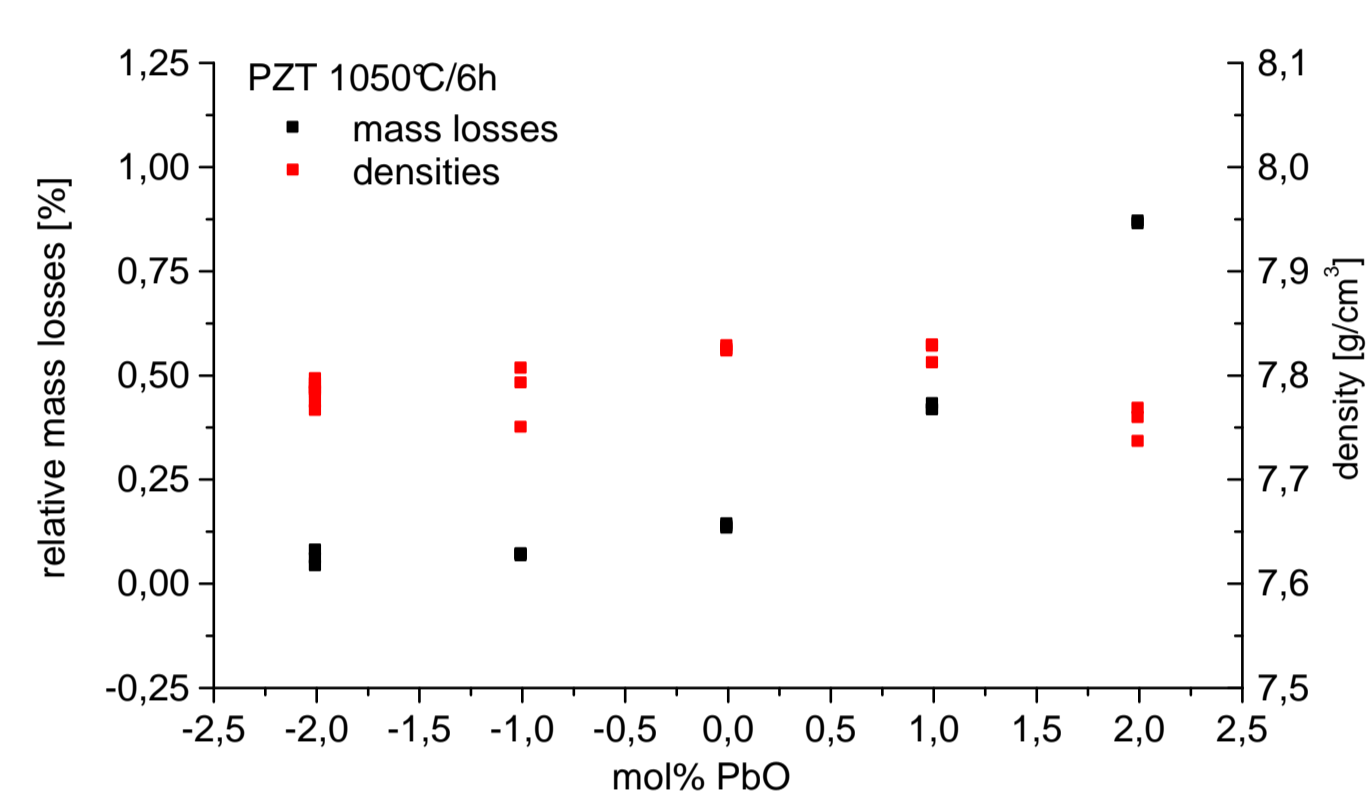
Both ceramics and actuators were characterized by SEM, XRD and measurements of dielectric low field and high field strain. Objectives of the work are (i) to point out the effects of PbO content variation on properties of the PZT ceramics and devices, (ii) to gain first insight in the mechanism leading to changes with variation in PbO content and (iii) to establish correlations between sintering weight loss and important parameters for performance of PZT.

Ceramics prepared with varying PbO content

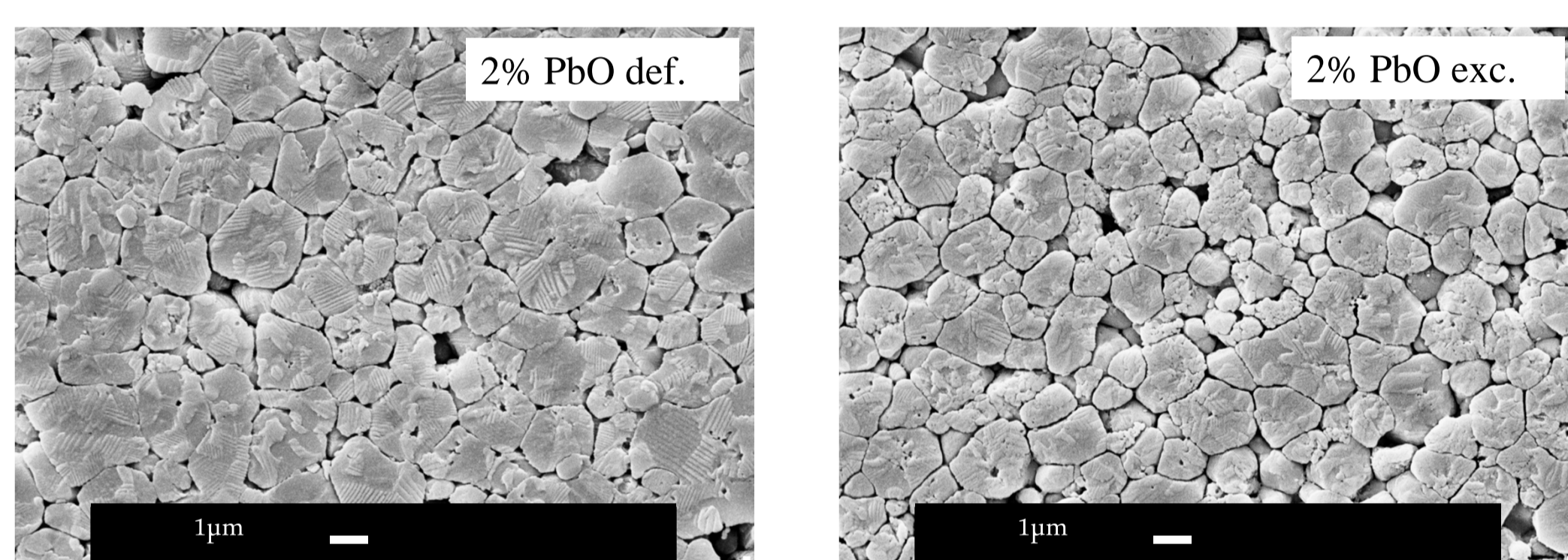
PbO-content in batch composition varying from 2mol% deficit to 2mol% excess

PZT + PbO

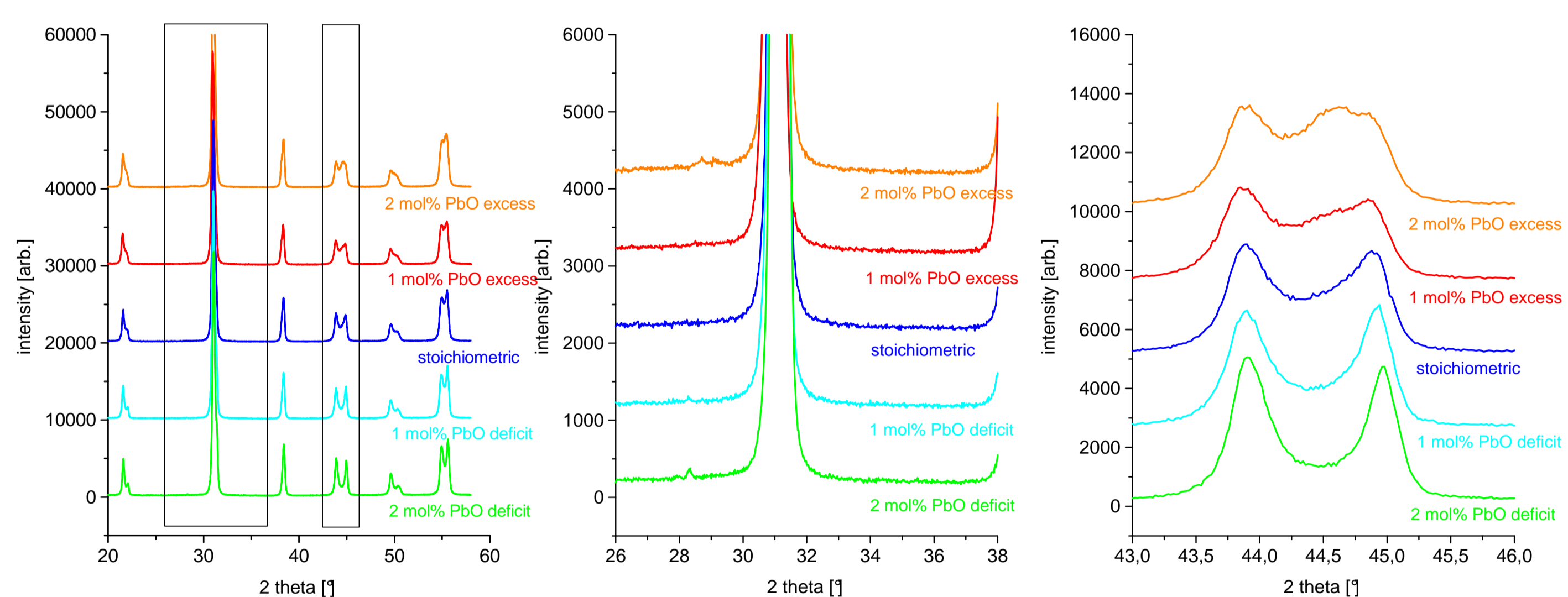
batch compositions
2 mol% PbO deficit
1 mol% PbO deficit
Stoichiometric
1 mol% PbO excess
2 mol% PbO excess



Microstructure and Structural analysis

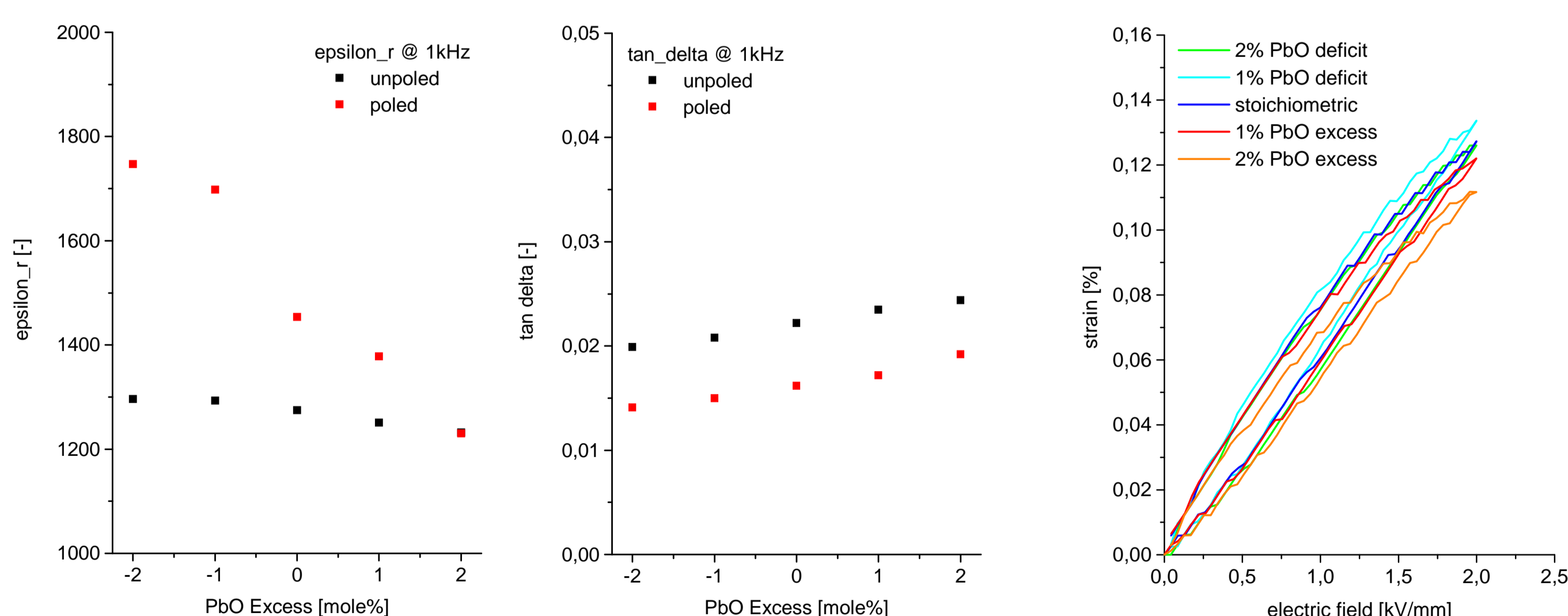


Similar grain size for PZT ceramics with PbO excess and deficit



In ceramics with PbO deficit occurs the formation ZrO_2 secondary phase
PbO peaks in X-ray diffraction patterns can be detected for PZT > 2mol% PbO excess
Shift of PZT structure to more tetragonal structure with decrease in PbO content

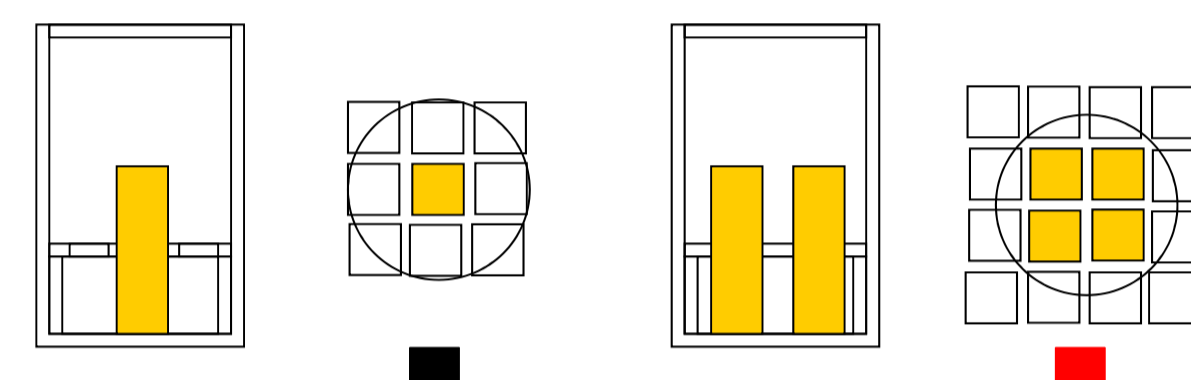
Dielectric properties and high-field strain



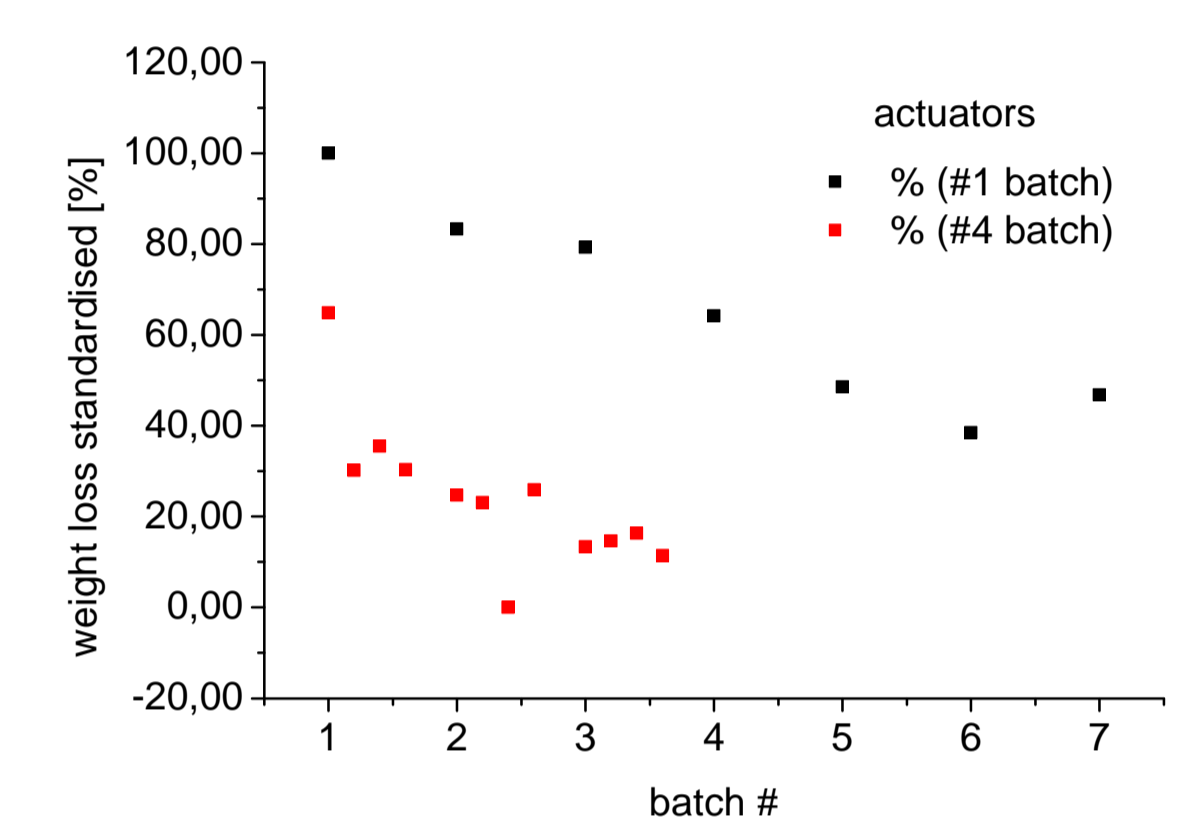
Permittivity decreases slightly in unpoled, but strongly in poled state in PZT with PbO excess.
Dielectric losses ($\tan \delta$) increase with PbO content.
Electric field induced strain decreases for PZT with PbO excess.

Actuators fabricated using different sintering setups

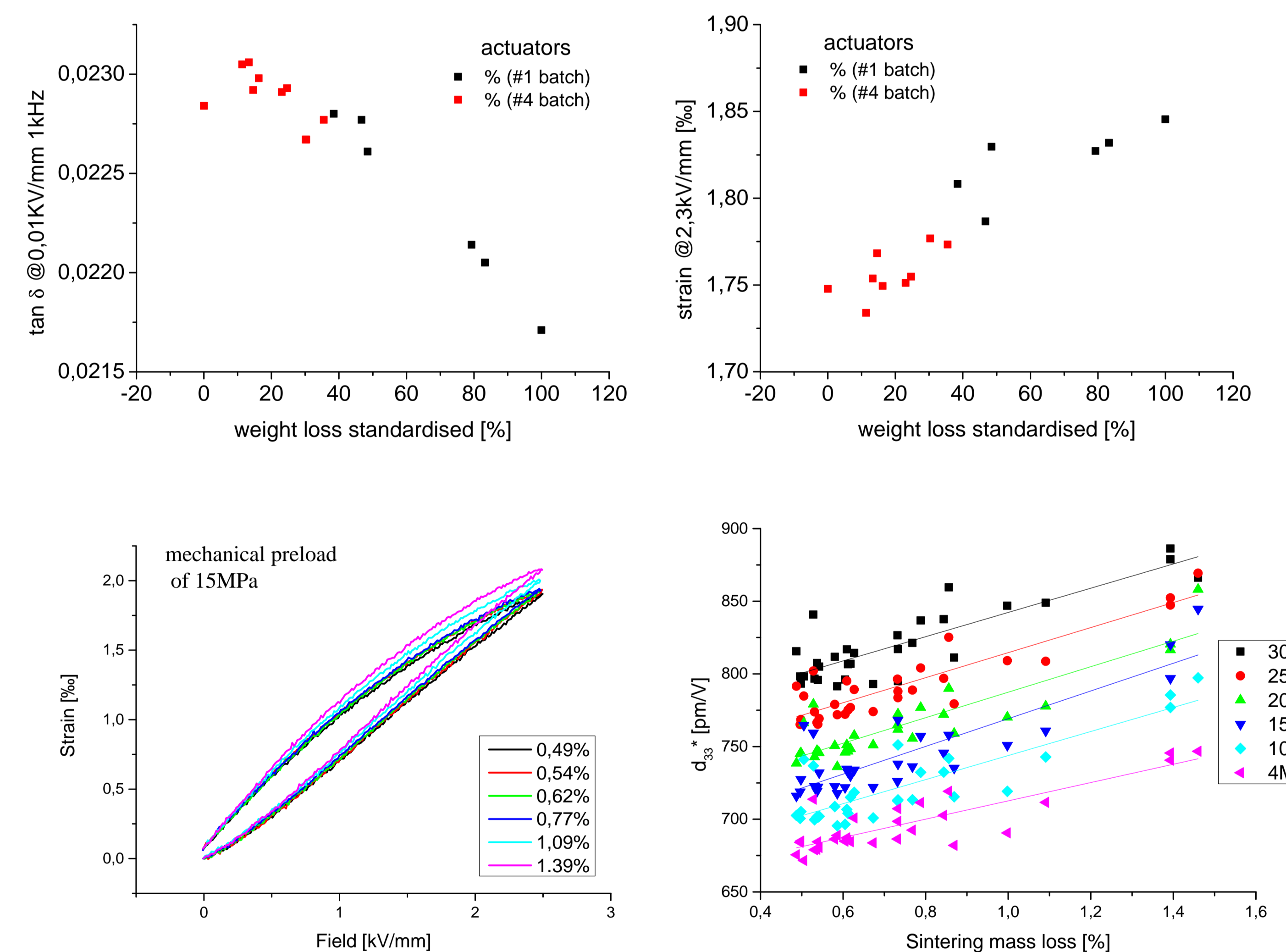
Laboratory Sintering Setups: commercial soft PZT, 1/4 actuators encapsulated, standing atmosphere, progressive PbO contamination



Higher loading of crucibles (4 vs. 1 actuators)
results in lower sintering mass losses



Dielectric properties and high-field strain



Linear negative correlation between weight loss and dielectric loss $\tan \delta$
Linear positive correlation between weight loss and high field strain
Strong influence of PbO-content on electric properties of actuators
Strong influence of mechanical preload on high field strain

Conclusions

Lead oxide content markedly influences electrical properties of PZT. The influence is the same for bulk ceramic specimens and for multilayer actuators.

Most significant correlations are an increase in high field strain and a decrease in dielectric losses with lower PbO content

In ceramics, there are only slight effects from grain size, but very pronounced effects from changes in structure.

In actuators, a mechanical preload of up to 30MPa strongly enhances the high field strain independent of PbO content.

Publications last funding period

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- "De-aging of Fe-doped lead-zirconate-titanate ceramics by electric field cycling: 180 degrees- vs. non-180 degrees domain wall processes", J. Glaum, Y.A. Genenko, H. Kungl, L.A. Schmitt & T. Granzow, J. Appl. Phys. 112, 2012
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