

# EUROMAT 2011 **FEMS**



Comedie SCLM Communication

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Montpellier, France

## SF2M

Société Française  
de Métallurgie et de Matériaux



Associazione Italiana  
di Metallurgia

### Main Topics

Functional Materials: Properties & Applications  
Magnetic Materials; Materials for Nanostructures; Functional Polymeric Materials;  
MEMS, NEMS and Devices

Structural Materials: Properties & Applications  
Advanced Metals; Advanced Ceramics; Hybrid and Composite Materials;  
Advanced Concepts in Structural Materials

Materials Processing  
Solidification and Solid State Transformations; Joining; Powder Routes: from Synthesis  
to Materials; Coatings and Surface Engineering; New concepts in Materials Processing

Characterisation and Modeling  
Microstructural Characterisation Techniques; Mechanical Characterisation  
Techniques; Materials Modeling on all Length Scales

Energy and related Applications  
Energy Production, Transportation and Management; Materials for Energy  
in a Sustainable Society; Materials for Transportation

Health Care Applications

Materials for Health Care Applications

Education

Topic Area	C Processing	
Topic	C3 - Powder routes : from synthesis to materials	C31-O-3-2
Symposium	C31 - Powder Synthesis and Processing	0875
Session	Ceramic powders synthesis I	

**Self-organization in PT and PZT powders produced by hydrothermal route: highlights for the comprehension of mesocrystals formation to oriented attachment growth**

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**Abstract**

The technological importance of piezoelectrics, such as lead titanate (PT) and lead zirconate / titanate (PZT) motivate the development of different techniques for the synthesis of these materials. However, an accurate control of growth steps in these structures is necessary if one desires particles with nanometric, near monodisperse sizes. To this goal, we developed a simple synthesis based in the decomposition of a peroxo-titanate or a mixture of peroxo-titanate and peroxo-zirconate by the pH dislocation imposed by the addition of an excess of lead carbonate. This mixture produced amorphous powders, which were crystallized under hydrothermal conditions (120 - 200°C during 2 - 12 hours under pH around 14 in KOH medium). All the particles produced showed a regular cubic-shape revealed by scanning electron microscopy, with sizes around 500 nm to 1 micron and were confirmed as the desired phases (PT or PZT) by Raman spectroscopy and X Ray diffraction. However, an accurate analysis by high resolution transmission electron microscopy revealed that in all the cases the cubic shapes observed were related not to single crystals, but are formed by a ordered structure of very small, semi-spherical particles, around 10 - 50 nm. Those particles appeared as oriented in a mesocrystal, self-organized, but instable: a series of HRTEM images were taken in same regions after and before concentrating the electron beam under the sample. This test revealed that the polarization imposed by the electron beam was enough to separate the nanoparticles, destroying the mesocrystal and confirming that the particles were only oriented but not attached in a single crystal, despite in same crystallographic orientation. However, a series of nanoparticles were observed as coalesced by oriented attachment mechanism, which suggests that the mesocrystal formation was a previous step for the oriented attachment in those materials. It was supposed that the polarization feature of PT and PZT may act as a guide to the oriented organization of the cubic mesostructure, by minimizing the repulsion between the nanoparticles in same crystallographic directions. By this structure, the oriented attachment may occur after a heating step or under hydrothermal conditions, in longer times. Those results are important to understand the formation of larger crystal from smaller ones by oriented attachment mechanism, since it reveals an intermediate step, which is the mesocrystal formation.