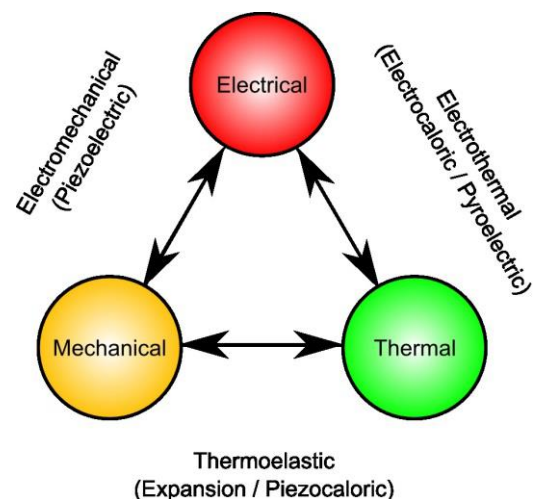


Final Publishable JRP Summary for METCO Metrology of electrothermal coupling for new functional materials technology

Overview

This project developed the metrological infrastructure and facilities within Europe for the traceable metrology of coupling between thermal, mechanical and electrical properties in new materials technologies at high temperatures (up to 1000 °C) and high electric fields (up to 5kV/mm).

New technologies are being developed that will enable reductions in energy consumption and improvements in efficiency and reliability in automotive, energy, process, electronics and medical industries through the use of new functional materials which operate at temperatures up to 1000 °C. These applications exploit the actuation, sensing and cooling functionality of ferroelectric materials which result from strong coupling between electrical, thermal and mechanical properties. Degradation of materials properties at high temperature means that before the project these applications are currently only possible below 200 °C.



Before the project, the new materials for high temperature operation were not supported by a metrological framework for traceable measurement of the coupling at high temperatures. Understanding these materials and being able to measure their properties will encourage further research and industrial applications.

Need for the project

Reliable, accurate and traceable measurement of electro-thermo-mechanical coupling at high temperatures is essential to provide the data required for the development of new materials technology, effective design of new devices, reliable characterisation and reproducible testing; and therefore ensure quality in manufacture and reliability in service. It requires a robust measurement infrastructure that is traceable to national and international standards for SI units, and measurements under industrially relevant harsh environments. This metrology infrastructure does not currently exist, but is required by companies from the aerospace, automotive, power, oil & gas, instrumentation and medical sectors, many of whom participated in the project.

Piezoelectric materials are widely used in sensors, actuators and transducers, as applying a voltage results in mechanical displacement and vice-versa. Currently their use is limited to near ambient temperatures where their properties are well understood. Reliable, accurate and traceable measurement of electro-mechanical coupling at high temperatures is complex and large systematic errors are common. Electrocaloric materials show a temperature change under an applied electric field, making them of great interest for use as on-chip cooling of electronics and efficient cooling that does not require refrigerant gases. The project developed the underpinning metrology to facilitate further research into these materials.

Report Status: PU Public

Scientific and technical objectives

1. To develop facilities for the traceable measurement of electromechanical and thermoelastic coupling at high temperatures (up to 1000 °C) and under high electric fields (up to 5kV/mm) using new high temperature interferometry and new high temperature resonance methods.
2. To develop novel facilities for the traceable measurement of electrothermal coupling and electrocaloric figure of merit in bulk and thin films under high electric fields at temperatures up to 250 °C.
3. For the first time, to extend high accuracy traceable full-field interferometry of electromechanical and thermoelastic coupling to high temperature applications up to 200 °C.
4. To develop traceable measurement of temperature and thermophysical properties up to 1000 °C.
5. To develop new models of the metrological linkages between thermo-electro-mechanical coupling in materials and the measurement protocols developed in the previous objectives and performance in devices.
6. To provide state of the art new materials technologies and apply the metrology developed in this project to the development of new high temperature materials.

Objective 2 and part of 4 were concentrated on electrocaloric materials, and the others were primarily on piezoelectric materials.

Results

1. To develop facilities for the traceable measurement of electromechanical and thermoelastic coupling and under high electric fields up to 1000 °C using new high temperature interferometry and new high temperature resonance methods.

The project developed four separate instruments capable of measuring the indirect thermoelastic and electromechanical coupling to 1000 °C, and suitable for eventual deployment in industry. Three of them worked on interferometry techniques measuring the absolute length change of the sample under test, and the final system was based on an established electromechanical resonance measurement technique.

The systems could operate at different measurement frequencies, allowing greater insight into the materials, as well as enabling a comparison between the systems. They were also compared with the metrological instrumentation developed in objective 3.

Two of the systems were upgraded versions of commercial instrumentation, meaning that end-users are now able to make their own measurements at high temperatures with greater confidence.

Measurements above 500 °C were hampered by the lack of suitable electrically conductive vacuum depositable coatings and availability of suitable high temperature ceramics.

2. To develop novel facilities for the traceable measurement of electrothermal coupling and electrocaloric figure of merit in bulk and thin films under high electric fields at temperatures up to 250 °C.

The project developed methods for measuring the electrocaloric effect in both thin films and bulk (thick film) materials in air and in a vacuum that could be used in industry and academia. The devices have been specially designed to minimise the heat transfer. During the design phase, it became apparent that the only method of measuring temperature that was suitable was an infrared camera. The project was successful in characterising bulk electrocaloric materials. The instrument was capable of making measurements in air or vacuum, with a high degree of spatial resolution and sensitivity. The project did not manage to measure a cooling effect in any electrocaloric thin films, and certainly not to the levels predicted by others working in the field.

Several Figures of Merit (FoM) have been discussed and a joint paper will be published. An agreed FoM would be a single reference number calculated from a number of material properties that would allow quick and easy comparison of materials for their suitability in electrocaloric cooling applications.

3. For the first time, to extend high accuracy traceable full-field interferometry of electromechanical and thermoelastic coupling to high temperature applications up to 200 °C.

A metrology grade high precision full field interferometric instrument was upgraded to enable it to be used up to 200 °C, and with an applied voltage across the specimen. The mechanical deformation of the piezo-ceramic samples were measured with very low uncertainties, which helped with comparison with the results being obtained from the other instruments and techniques. It also enabled changes in the material processing to be developed to reduce sample flexure, which was affecting the measurements.

4. To develop traceable measurement of temperature and thermophysical properties up to 1000 °C.

Measurement of thermophysical properties of thermoelastic or thermoelectric materials in a traceable manner requires measuring specific heat and thermal diffusivity in similar conditions to those the materials will be employed in. Several National Metrology Institutes upgraded their test facilities to enable them to apply a high voltage to the test samples. The measured results provided essential data to the modelling, as well as for making non-contact temperature measurements in the high temperature interferometer system.

5. To develop new models of the metrological linkages between thermo-electro-mechanical coupling in materials and the measurement protocols developed in the previous objectives and performance in devices.

State of the art models of heat flow in thin film and bulk devices, and dynamic models of temperature changes over small time scales and length scales in both piezoelectric and electrocaloric materials have been developed and validated against experimental results. The models underpinned the development of new devices and the experimental uncertainties. Modelling the interactions on a nano-scale in relation to Scanning Probe Microscopy (SPM) interactions will give a significant insight into the workings of the materials because it has been validated against actual experimentation.

6. To provide state of the art new materials technologies and apply the metrology developed in this project to the development of new high temperature materials.

After an initial industrial consultation, the project evaluated a large number of different compositions of materials. The project successfully developed a reference material that would work up to at least 380 °C. This will allow traceable evaluation of the uncertainties of the instrumentation, and it is suited to wider commercial application in industrial applications.

Actual and potential impact

Dissemination of results

To raise awareness of the project's results, five articles have been published in technical magazines such as 'Materials World' and 'Electronics Weekly' on both high temperature piezoelectric and electrocaloric materials and the potential of these materials. Five peer reviewed papers were published as well as twenty two posters and presentations given at scientific conferences.

The industrial and academic community were kept up to date with the progress through a series of three newsletters and eleven webinars organised by the project team on different aspects of the project, as well as two workshops to transfer the knowledge generated by the project.

Early impact

The project has led to commercial opportunities for the companies involved:

- The two systems developed by aixACCT Systems and validated during the project for high temperature measurement of piezoelectric materials are now commercially available. They are suitable for deployment in industry and academia, which means for the first time that there is traceable equipment readily available to evaluate high temperature sensors for the majority of the current anticipated requirements.

- State of the art piezoelectric materials have been developed for use in the project up to to 380 °C by Leeds University, which are suitable as reference grade materials. IONIX, a spin-off company of the University, is now engaged in the commercialisation of the reference material developed in this project for wider deployment into industrial applications.
- The combination of Ionix work with aixACCT instrumentation means that there are no longer significant barriers to the commercial exploitation of high temperature piezoelectric materials.

Longer term impact

The accurate and traceable metrology framework developed in the project supports development and manufacture of piezoelectric and electrocaloric materials and has already increased our understanding of materials in non-equilibrium states. These materials will form the basis of novel sensors, actuators and transducers for use at higher temperatures. The ability to monitor industrial processes and carry out sensing at higher temperatures than is currently possible, will give efficiency improvements in a wide range of industrial applications. This project also supports the development of electrocaloric cooling which has the potential to reduce the use of propellant gases and benefit industries such as electronics, automotive, healthcare and oil-gas, which rely on the development of innovative cooling technologies that can easily be integrated.

List of publications

Please refer to the publications list in the Final Publishable JRP Report.

JRP start date and duration:	01 June 2012, 36 months
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JRP-Partner 2 CMI, Czech Republic	JRP-Partner 5 PTB, Germany
JRP-Partner 3 LNE, France	JRP-Partner 6 aixACCT, Germany
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