

REPORT

Final Report and Recommendations for DTI Project AM4

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**Final Report and Recommendations: Project AM4,
Development of Measurement Methods for the
Characterisation of the High Stress Behaviour of Piezoelectric,
Electrostrictive and Magnetostrictive Materials**

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ABSTRACT

This report gives a summary of the work that has been carried out under project AM4 of the DTI Programme on Advanced Materials, on the development of methods for the characterisation of the high stress behaviour of piezoelectric, electrostrictive and magnetostrictive materials.

The deliverables of the project are listed together with the major achievements of the project.

The report also contains a brief review of the state-of-the-art in methods for the monitoring of the degradation of electroceramic materials.

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EXECUTIVE SUMMARY

Background

This report summarises the results of project AM4 and lists the main recommendations and conclusions.

Project AM4 has been concerned with the development of methods for the high stress characterisation of piezoelectric, electrostrictive and magnetostrictive materials. It was supported by the Department of Trade and Industry through the Materials Testing and Standards programme. The work of the project was undertaken by a multidisciplinary team managed and led by NPL, with major proportions of the work carried out by the Technology Partnership, Leeds University and Hull University.

The overall aim of the research was to provide a test and characterisation framework which will enable product developers and manufacturers easily to obtain the information they need so that they can reliably design with actuator and sensor materials operating under high stress conditions.

Achievements

- Development of understanding of state-of-the-art in measurement methods for electroceramic and magnetostrictive ceramics
- Development of simple model for the behaviour of piezoelectric materials
- Two successful industry seminars
- Development of improved strain measurement systems
- Development of modelling and measurement methods for magnetostrictive materials
- Development of methods for the measurement of electroceramic performance under cyclic electrical and mechanical stresses
- Development of methods for the measurement of magnetostrictive performance under cyclic mechanical stresses
- Discovery of improvement in performance for magnetostrictive materials when subjected to cyclic mechanical stresses
- Development of methods for the determination of the microstructure of electroceramics

Recommendations for further work

Three major recommendations for future work emerge from the results of this project.

It is clear that the degradation of electroceramics is very complex, and that project AM4 only started to develop and investigate ways of measuring the degradation of these materials under cyclic electrical and mechanical stresses. Thus further work is needed in this area to develop recommended procedures for testing under these conditions.

The work that was carried out within this project also showed that there was often a lot of variability in properties from one sample to another. This made it very difficult to relate the results of one measurement to another. It is recommended that work should be carried out to develop test methodologies which, as far as possible, allow for the integration of the individual different test methods into one complete measurement system which allows for the simultaneous measurement of as many of the properties of the electroceramic as possible. This would then enable a complete history of the performance of samples to be built up.

From the positive response to the two seminars that were held in project AM4, it is clear that there is demand from industry for further meetings of a similar type. This need will be met by a series of future meetings carried out in the new project CAM 7.

1 INTRODUCTION

This report summarises the results of project AM4 and lists the main recommendations and conclusions.

Project AM4 has been concerned with the development of methods for the high stress characterisation of piezoelectric, electrostrictive and magnetostrictive materials. It was supported by the Department of Trade and Industry through the Materials Testing and Standards programme. The work of the project was undertaken by a multidisciplinary team managed and led by NPL, with major proportions of the work carried out by the Technology Partnership, Leeds University and Hull University.

The overall aim of the research was to provide a test and characterisation framework which will enable product developers and manufacturers easily to obtain the information they need so that they can reliably design with actuator and sensor materials operating under high stress conditions.

This report summarises the results under the headings of the different project Tasks. It should be noted that the task titles and objectives have remained those defined in the original project plans, but in some cases the direction of the task was altered (with the agreement of Industrial Advisory Group (IAG) and the DTI Technical Monitor) in the light of results as the project proceeded. Where changes have occurred they will be noted in the discussion for the different Tasks.

The expertise that has been developed in project AM4 will be built on in a new project on the development of test methods for *Electroactive materials under conditions of high stress or stress rate* (CAM 7). This project is being funded by DTI under the Characterisation of Advanced Materials programme of DTI. A particular emphasis of this project is the development of methods for the characterisation of the degradation in performance under cyclic mechanical and electrical fatigue.

2 PROGRAMME TASKS

Task 1; The Extension of Constitutive Relations

Objective

The extension of constitutive relations adequately to describe operation under high stress and severe environmental conditions.

Project work:

Task 1.1, Literature review and survey with industry

A literature review was carried out by the project consortium and was amalgamated into one document by NPL. NPL and Leeds University focused on the literature concerning measurement aspects for electroceramics, Technology Partnership had a particular emphasis on the literature concerning the modelling of the behaviour of piezoelectric and

electrostrictive ceramics, and Hull University examined the literature concerning the extension of constitutive relations, and non-linear measurement techniques for magnetostrictive materials. With respect to the review of modelling of piezoelectric behaviour, the work of Chen and co-workers was found to be a useful basis on which to develop further constitutive descriptions. This description of piezoelectric behaviour was based on an explicit model of the switching dynamics of electric dipoles within the material. The review of measurement methods showed that a large number of measurement methods for piezoelectric materials already existed, but that there were many inadequacies in terms of procedures and precise test conditions. Some areas where test methods were lacking, such as mechanical fatigue, were also identified. The magnetostrictive review identified two main models for the description of magnetostrictive behaviour. These were the model by Jiles et al which is based on mean-field equilibrium thermodynamics with the additional treatment of domain wall motion, and the Preisach model which is a phenomenological empirical model. The literature review was published as an NPL report [1].

A survey with industry was carried out with the aim of determining what the major concerns of industry were in this area. The main areas where industry were asked for an input were the types of material of most interest, their conditions of use, and the measurement areas which were of most concern. The response to the request for information was excellent, with nearly all the firms contacted giving a useful response (the firms covered both materials manufacturers and a range of users). The results of the survey were discussed at an IAG meeting held at an early stage of the project, and were used to focus the direction of the remainder of the project. The survey showed clear support from industry on the need for the development of new and improved test methods. The three measurement methods that received most support were:

- Displacement-field behaviour with better displacement resolution, under conditions of mechanical loading, at different temperatures, and at different frequencies.
- The degradation in properties with time.
- Mechanical behaviour under high electrical loading.

The survey and its results were written up as an open NPL report [2].

Task 1.2, Extension of constitutive relations

The extension of the constitutive relations was carried out by the Technology Partnership. The aim was to establish a framework within which non-linear material behaviour could be characterised in a way that could be useful to device designers. The approach that was adopted was to devise a material model which had a relatively small number of empirically determined parameters which could be used to characterise the material.

The model considered the minimisation of the Gibbs free energy of the system with respect to state variables. These state variables were a measure of the effective number of

aligned dipoles which is in turn proportional to the polarisation of the piezoelectric material.

The model that was derived [3] appeared to reproduce typical hysteresis and strain behaviour quite well. However, although it was the intention to compare the predictions of the model with the results of preliminary experiments, no measurement results were available when the modelling work was completed. Results are now available, and the theoretical predictions will be compared with experiment in project CAM 7.

Task 1.3, Workshop

A workshop was held at NPL with the twin aims of discussing in an open forum the work on the extension of constitutive description of the materials under investigation in the project, and to consider how these materials should be measured. The workshop had an attendance of 35, mainly from industry. Speakers at the meeting were drawn from industry and the representatives of standards committees as well as the project consortium members. The participants were all asked to complete a response sheet about the workshop, and there was a universal opinion that the event was worthwhile, and that further workshops should be held. To fulfil this need, further events are planned to be held under project CAM 7.

Task 2; Development of Test Methods for the Measurement of Extended Constitutive Coefficients

Objective

Development of test methods for the measurement and characterisation of the extended constitutive coefficients.

Project Work

Task 2.1, Measurement of electronic properties

The practical work in this part of the project fell into two areas. The first of these was the setting up of a method for the low field measurement of dielectric constant and loss at NPL which was implemented using a HP 4192A bridge.

The majority of this task was concerned with the development of a robust technique for the measurement of the polarisation-field hysteresis loop behaviour for piezoelectric materials. The variation of polarisation with field underlies many of the useful properties of piezoelectric materials. The development of a new and improved technique was carried out by Leeds University.

Measurement of this loop behaviour has traditionally been made with the Sawyer-Tower bridge. The correct operation of this measurement technique relies on making manual adjustments for the effects of the capacitance and resistance of the device under test and

the circuit. This is prone to so much error that it is unlikely that this measurement can be made reproducibly between laboratories.

Leeds University developed a computer based data acquisition and analysis system which has the major advantage that the corrections for capacitance and resistance to obtain the essential non linear polarisation behaviour is performed automatically without the need for operator intervention. This removes the largest source of error in the measurement, and makes it convenient and simple to operate. The development of this test system has been publicised in a scientific paper [4].

Leeds University are considering the marketing of the measurement system that has resulted from this work under the trade name Dynohyst after appropriate agreements on IPR. The projected price of a test system is considerably less than the only current competitor system which is marketed in the US.

Task 2.2, Characterisation of displacement-field behaviour

The displacement that is generated by an electroceramic actuator material is the primary property of importance for actuator applications. The measurement of displacement was investigated by NPL by a number of different techniques. These were using capacitance displacement probes, an LVDT (Linear Variable Displacement Transformer), strain gauges, fibre-optic probes, and laser interferometry. The details of the measurement methods are given in the published reports and papers [5-7].

The measurement system with the best resolution, and the fastest response is the laser interferometer. The test system developed at NPL is based on a differential common path interferometer, and has a resolution and stability better than 1 nm. The system has the potential for measurement at rates up to several MHz, but was only demonstrated up to about 10 kHz. Interferometry also has the major advantage that it is inherently traceable through the wavelength of the laser used for the test system. However, laser interferometry is complex to operate and expensive to set up.

The best overall system is the fibre-optic probe which has very good resolution (but not as good as the interferometer), fast measurement rates up to 200kHz, and is reasonably cheap to purchase and set-up. There are some problems with alignment and calibration, but these can be easily overcome with proper operation.

The sample holding arrangements were found to have an important effect on the results that are obtained. This is because of the artefacts generated through mechanical resonance of the sample and sample holder. The best arrangement for sample holding was found to be a vacuum clamp.

The heat generated when high frequency high amplitude electrical fields are applied has a large effect on the results that are obtained. The temperature rise that is generated by internal heating was monitored in experiments by the use of a thermal imaging system and showed temperature rises of up to 80°C in typical experiments.

Task 2.3, Two-axis displacement measurement

Two-axis displacement measurements were made using a special test jig and fibre-optic probes so that the measurement of displacement could be carried out simultaneously in the direction parallel to the electric field and perpendicular to it. The measurements were successful, but it was found that there were significant alignment errors, particularly since the experiment was carried out on a cylindrical sample [6].

Task 2.4, Point-to-point displacement measurement

Experiments were carried out where measurements of the amplitude of displacement were made from point to point while an alternating electric field was applied to the sample. The concept behind the measurements was to investigate the feasibility of using point-to-point scanning techniques as a method of investigating defects and details in the structure of samples such as multi-layer ceramic capacitors [6].

The experiments did demonstrate the methodology successfully, but did not reveal any structure. This was because the response of the samples investigated was dominated by elastic deformation of the complete structure.

Task 2.5, Comparison with resonance methods

Work was carried out by NPL, Leeds University and Hull University on the use of resonance techniques for the determination of the properties of the electroceramics. However, apart from the further development of the Plane Wave Method by Hull University [8], this work was inconclusive and did not achieve any firm results.

Task 2.6, Measurements on magnetostrictive materials

Further development of measurement methods for magnetostrictive materials was carried out by Hull University, and was used to compare experimental results for the strain behaviour of magnetostrictive materials with theoretical models [8].

Task 3; Development of Test Methods for Fatigue and Degradation Behaviour

Objective

Development of test methods for the characterisation of fatigue and degradation behaviour including measurements in high stress and severe environmental conditions.

Project work

Task 3.1, Fatigue measurements for electroceramics

A number of different methods were investigated for the production of the high stress high frequency alternating mechanical stresses that were needed to subject electroceramic samples to mechanical cyclic loading. Possible techniques that were examined were an eccentrically driven motor system, conventional high rate hydraulic testing systems, the use of an air hammer, electromagnetic loading, and the use of piezoelectric actuators.

Practical experiments were conducted with a conventional hydraulic testing system and an electromagnetic testing system. A magnetostrictive actuator system was also purchased, for use in these experiments, but was not fully operational before the end of the project.

It was found that the piezoelectric performance of piezoelectric ceramics degraded with increasing exposure to a longer duration, and to a higher amplitude of stress loading. When care was not taken to ensure uniaxial loading of the test samples, sudden drops in performance of the ceramics were observed which were found to be related to the sudden mechanical failure of the ceramics [6,9].

Some exposure of piezoelectric samples to alternating electrical stresses was also carried out where it was found a similar drop off in functional behaviour was found.

Some work was also carried out at Leeds University on the use of a drop-weight impact testing system to investigate the response of electroceramics to repeated impact loading [10].

Task 3.2, Fatigue measurements for magnetostrictive materials

Samples of magnetostrictive material were exposed to alternating mechanical stresses at NPL and the samples' behaviour measured by Hull University. Remarkably, it was found that the magnetostrictive performance of the samples was improved by the exposure, in some cases by a factor of up to two. This result was entirely unexpected, but was found to be similar in effect to the fairly well known phenomenon of thermal annealing of magnetostrictive materials [11].

This discovery is potentially commercially important, and some discussions are underway on possible routes to build on this success.

Task 3.3, Measurements under extreme conditions

With the agreement of the Industrial Advisory Group, this task was dropped as it was felt to be too near market to be supported justifiably under project AM4.

Task 3.4, Microstructural examination

Leeds University and NPL both developed techniques for the determination of the microstructure of electroceramic materials. The investigations at Leeds University [12,13] determined that the quality of many piezoelectric samples, particularly from UK manufacturers, were of very poor quality with large processing defects and the presence of contaminants in their structure which severely degraded their performance.

Task 4; Development of Standards

Objective

Prepare draft standard for piezoelectric, electrostrictive and magnetostrictive materials incorporating the results of Tasks 1,2 and 3.

Project work:

Task 4.1, Seminar on test methods

A successful seminar was held where the main results of the project were presented by project partners. A talk was also given by a representative of the standardisation committees in the area.

About 30 delegates attended the meeting, with about half from industry. Attendance was a little poorer than foreseen due to an clash with another seminar in the ultrasonics field. However, analysis of the response sheet that was handed out to delegates showed that they thought that the event was worthwhile and that similar events should be held in future. To fulfil this need, further events are planned to be held under project CAM 7.

Task 4.2, Demonstrator

The successful transfer of the measurement technology developed during the project was demonstrated by the take up of techniques for the measurement of strain-field performance of electroceramics by two industrial firms. These were Bonas Machine Tools who used fibre-optic probes to investigate the feasibility of using electroceramic actuators for the control of their loom machinery, and GEC Marconi Materials who used fibre-optic probe technology for the determination of the performance of materials under development, and who now have installed the test system as a permanent part of their testing equipment.

Task 4.3, Interlaboratory exercise

An interlaboratory exercise was carried out with the participation of five industrial firms. However, despite the clear instructions that were circulated with the test samples, few laboratories carried out all of the required tests, and some laboratories carried out other tests that were not laid down in the testing protocol.

Nevertheless, useful information was obtained on the measurement of piezoelectric properties by resonance methods, and on the measurement of piezoelectric constants by the Berlincourt meter measurement method [14]. Analysis of these results showed that there was a systematic variation in test results from one laboratory to another. The source of this variation could not be identified within the resource constraints of the project.

Task 4.4, Reference artefacts

The feasibility of using materials with well controlled microstructures as reference artefacts to improve the reproducibility and repeatability of measurements was investigated [15]. The three materials that were examined were single crystal lithium niobate, single crystal quartz and polycrystalline hot-pressed PZT. The first two materials were chosen as they exhibit ferroelectric behaviour, but in the event it was found that the magnitude of the strain that was generated by these materials made it unmeasurable by many of the techniques that would normally be used for the measurement of electroceramics and thus unacceptable as reference materials.

The hot-pressed material showed a little more promise, but showed too much variation in the results of measurements to be an effective reference material.

Task 4.5, Draft standards

It was agreed at Industrial Advisory Group meetings and project management meetings that the results from the project were too premature to allow for the formulation of draft standards. However, many of the outputs from the project contain recommendations for good testing practice so that it was decided that the focus of this task should be the production of a final report for the project which would summarise the main results and list the main output of the project. This current document is that final report.

Task 4.6, Design guide

A reassessment of the constitutive model for piezoelectric materials was carried out by the Technology Partnership who considered that the model needed extending. This was done by considering the kinetics of how domain walls move through the switching of individual domains to arrive at a revised model which was based more reliably on appropriate consideration of the physics that underlay the behaviour of piezoelectric materials.

The revised model was written up into a design guide which was intended to be used by industry to enable them to make predictions concerning the functional behaviour of piezoelectric materials. The design guide includes brief instructions how to derive the controlling parameters for the model from experimental data, and then how to use the formulated model as the basis for predictions [16].

3 ADDITIONAL WORK

At the project review meeting with the DTI Customer in October 1995, some additional work items were agreed which were partly in preparation for the continuation of work in the area.

D1 Redefinition of contact database

The number of contacts in the contacts database in the electroceramics area was at least doubled to just under 250 contacts. Particular assistance in this work was obtained from Professor R W Whatmore at Cranfield University. The enlarged contacts database will be used for ongoing dissemination of the results of project AM4 and project CAM7.

D2 Targeted dissemination

A set of four short dissemination articles which are aimed at the technical reader that is not already fully aware of the DTI supported measurement projects have been written. The areas that have been covered are:

- microstructural assessment of piezoelectric materials
- mechanical fatigue of piezoelectric ceramics
- strain measurement for piezoelectric ceramics
- polarisation measurement for piezoelectric ceramics

The publication and mailing of these short dissemination articles will be carried out under the dissemination milestone of project CAM7.

A1 Feasibility study on degradation monitoring

The purpose of this work item was to investigate the feasibility of performing real-time evaluation of the state of a piezoelectric electroceramic whilst the piezoelectric ceramic is operating. This would be done by reviewing the state of the art in the area. There proved to be remarkably little work done, so it was not considered worthwhile to write a separate report for the work item.

Clearly the place to start with this study is to look at the degradation of the functional properties of piezoelectric materials under cyclic mechanical or electrical fatigue. The literature is now increasing in this area, with major contributions coming from Japan [A1-A3] and the US group at Penn State University [A4-A6]. In these studies the functional

performance of the electroceramics is monitored throughout the exposure to cyclic fatigue so that the degradation in performance can be recorded.

More sophisticated techniques such as acoustic emission, the observation of the extension of cracking damage, and the measurement of surface potential have been shown to be useful measures of the degradation of the material after exposure to cyclic loading [A7-A11].

Acoustic emission has been shown to be generated when domain reversal in piezoelectric ceramics occurs [A7]. More normally in the context of the monitoring of damage acoustic emission gives a measure of the accumulation of damage to the ceramic [A8-A9].

The direct observation of the growth of cracks provides clear and obvious measurements of the increase in damage to ceramic samples, and has been observed both for multilayer devices [A10] and for bulk samples with starting cracks developed from hardness indentations [A11]. However, although the technique provides useful information it is clearly can only give information about the surface of the sample, and is therefore of no use if sub-surface information is required.

Measurement of the surface potential that is developed on a piezoelectric sample is related to the capacitance of the area where the potential is measured. This is related to the damage that has been sustained by the ceramic and thus can be used for the real-time evaluation of damage [A8].

The growth of cracks in piezoelectric materials has also been the subject of theoretical studies which relate the fracture mechanics of crack growth to the stresses imposed on the ceramics both externally and also through the stresses generated electromechanically [A12-A13]

All three techniques summarised above have considerable potential for the real-time monitoring of damage and degradation in piezoelectric materials.

4 ACHIEVEMENTS AND RECOMMENDATIONS FOR FUTURE WORK

Achievements

- Development of understanding of state-of-the-art in measurement methods for electroceramic and magnetostrictive ceramics
- Development of simple model for piezoelectric materials
- Two successful industry seminars
- Development of high resolution laser interferometry strain measurement system
- Development of techniques to use fibre-optic probes for the measurement of piezoelectric strain

- Development of techniques for displacement mapping measurement
- Development of technique for 2-axis strain measurement
- Development of modelling and measurement methods for magnetostrictive materials
- Development of methods for the measurement of electroceramic performance under cyclic electrical and mechanical stresses
- Development of methods for the measurement of magnetostrictive performance under cyclic mechanical stresses
- Discovery of improvement in performance for magnetostrictive materials when subjected to cyclic mechanical stresses
- Development of methods for the determination of the microstructure of electroceramics
- Conduction of an interlaboratory exercise on measurement methods
- Investigation of the feasibility of developing reference materials for electroceramic measurements

Recommendations for further work

Three major recommendations for future work emerge from the results of this project.

It is clear that the degradation of electroceramics is very complex, and that project AM4 only started to develop and investigate ways of measuring the degradation of these materials under cyclic electrical and mechanical stresses. Thus further work is needed in this area to develop recommended procedures for testing under these conditions.

The work that was carried out within this project also showed that there was often a lot of variability in properties from one sample to another. This made it very difficult to relate the results of one measurement to another. It is recommended that work should be carried out to develop test methodologies which, as far as possible, allow for the integration of the individual different test methods into one complete measurement system which allows for the simultaneous measurement of as many of the properties of the electroceramic as possible. This would then enable a complete history of the performance of samples to be built up.

From the positive response to the two seminars that were held in project AM4, it is clear that there is demand from industry for further meetings of a similar type. This need will be met by a series of future meetings carried out in the new project CAM 7.

5 ACKNOWLEDGEMENTS

This work was carried out within the DTI Programme on Advanced Materials.

6 LIST OF DELIVERABLES

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