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Dielectric, Piezoelectric,  
Ferroelectric and  
Multiferroic Materials

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## PIEZOELECTRIC COMPOSITE MATERIALS AND STRUCTURES

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### ABSTRACT

The present paper considers the correlations between advanced piezoceramic technologies and the development of smart devices, microsystems and structures. Bob Newnham proposed the idea of smart materials in the 70 ties. Since that we find research programs focused on systems with embedded actuators, sensors and controller units. Active structures proved beneficial, which for example arises from increased safety, reduced energy consumption, extended life cycle and unique performance. But as a non-series product they failed often by high production costs. In other words, extension of market share to active structures by commercial applications requires technology chains and designs, which are compatible with mass production. An effective approach to overcome this obstacle might be the use of microsystems / MEMS technology as intermediate step providing pre-integration of active functions.

Technological developments of the recent past following the here expressed strategy are summarized in the paper. We tried to bridge the gap between piezoceramic transducer and smart structure fabrication by qualification and adjustment of high efficient production methods of piezoceramic transducers (extrusion spinning, molding and screen printing), electronic circuits (electronics packaging and electronics production) and structural components (metal die casting). Examples will be given related to integrated thick films in silicon technology, and to ceramic multilayer, polymer and metal matrix architectures.

### INTRODUCTION

The present paper considers piezoelectric ceramics as key functional material in composites and structures. Most of piezoelectrics presently exploited commercially are solid solutions based on lead zirconate titanate (PZT) ceramics. Still, compositional developments within the PZT family are performed to meet custom requirements perfectly<sup>1</sup>. Lead free piezoelectrics, such as the sodium potassium niobate solid solution<sup>2,3,4</sup> and bismuth sodium titanate solid solutions<sup>5,6,7</sup> became the topic of much research at the end of the 1990s, which is due to increased environmental awareness<sup>8</sup>.

As part of these investigations textured microstructures were studied, which may be interpreted as ceramic composite approach in the microstructure scale. New fabrication processes of piezoelectric ceramics are desirable through which the texture in the ceramic can be well controlled to give preferred grain orientations. A controlled grain structure during the ceramic sintering process gives the ceramics an anisotropic pseudo-single crystal behaviour<sup>1</sup> and may improve key parameters, considerably.

The development of smart structures, as considered in the present paper, involves the integration of piezoceramics and further dissimilar materials performing separate functions into one device. The focus of the present paper is given to commercial PZT ceramics, but the ideas may be extended to new piezoceramics as well. Essentially, the challenge lies in the availability of complete and adjusted manufacturing chains and in the maintenance of the properties of constituent materials during packaging into an individual unit.

Bob Newnham proposed the idea of smart materials in the 70<sup>ties</sup>. Since that we find research programs focused on systems with embedded actuators, sensors and controller units. Active structures proved beneficial, which for example arises from increased safety, reduced energy consumption, extended life cycle and unique performance. But as a non-series product they failed often by high production costs. In other words, extension of market share of active structures to commercial applications requires technology chains and designs, which are compatible with mass production, like typical found in care manufacturing.

An effective approach to bridge the gap between piezoceramic transducer and smart structure fabrication might be the use of microsystems / MEMS technology. It is well established and industrialized and well suited to make use of the potential of active materials in custom devices. It guaranties for pre-integration of sensing, actuation and control. Active structures may then be designed by integration of single or cross-linked piezo-electronic modules. As consequence, integration of these microsystems under the technological conditions of smart structure fabrication, especially in terms of process temperature, mechanical impacts and cycle time, is required.

The present paper summarizes the potential of advanced, microsystems compatible piezo technologies for active structures and systems. Examples will be given related to the integration in silicon wafer, ceramic multilayer, polymer and metal matrix architectures.

#### KEY CERAMIC TECHNOLOGIES

This chapter considers extrusion spinning, soft molding and tape casting as efficient green forming technologies of single element transducers. High performance piezoceramic units can be obtained and further processed to piezocomposite materials. Screen printing is well suited for the preparation of flextensional transducers that amplifies and changes the direction of generated displacement.

#### Single element transducer fabrication

##### *PZT fibers by spinning*

An essential motivation for the development of piezoceramic fibers arose from the concept of smart fiber-reinforced structural materials with integrated fibers for sensing and actuation<sup>9</sup>. Powder suspension based piezoceramic fibers have been developed to make high performance, single fibers in the thickness range of 100–1000 $\mu$ m available. Suspension extrusion<sup>10</sup> and suspension spinning<sup>11,12,13</sup> were used as basic process routes allowing for different fiber cross sections, like cylinder shaped fibers, hollow fibers and rectangular shaped fibers. See Fig. 1.

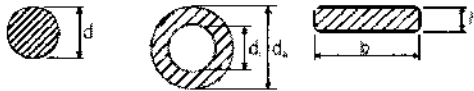


Figure 1. Cross sections of PZT fibers. Data are given in Table 1 and the text.

Suspension spinning is considered as well suited for mass production because basically textile fabrication methods are used. Two process variants<sup>11, 13</sup> have been commercialized.

In 1999, Smart Material Corp. (Florida) in co-operation with smartfiber AG, Germany, established the production of PZT fibers by the ALCERU<sup>®</sup> process<sup>14</sup>. They are made from Type

II and Type VI (U.S. Navy designation standards) piezoceramics and are offered in the diameter range between 105  $\mu\text{m}$  and 1,000  $\mu\text{m}$ . See Table 1. Smart Material Corp. launched their 1–3 piezoelectric fiber composites on the market, allowing for fiber composite engineering and production. A variety of custom products have become available since that.

Table 1: Commercial PZT fibers and tubes offered by Smart Material

Fiber diameters	d : 105 $\mu\text{m}$ ... 1000 $\mu\text{m}$
Tube outer diameters	$d_o$ : 400 $\mu\text{m}$ and 1000 $\mu\text{m}$
Tube wall thickness	$d_w - d_i$ > 100 $\mu\text{m}$
Fiber and tube length	< 200 mm
Piezoceramic materials	Type: PZT 4, PZT 5A , PZT 5H

A third process, the polysulfon spinning technology, has recently been developed by Fraunhofer IKTS and TU Dresden for PZT fiber fabrication with the benefit of room temperature processing and the use of environmental harmless solvent NMP (N-methylpyrrolidon) in small quantities<sup>15</sup>. The process chart of the polysulphone process is sketched in Fig. 2.

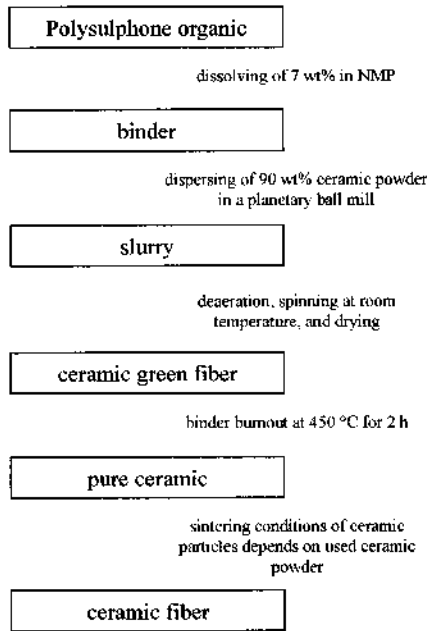


Figure 2. Workflow for the production of ceramic fibers by the polysulphone process<sup>15</sup>.

The reduction of porosity and vacuoles turned out to be the main issue. As result of systematically process development sintered fibers with low porosity of < 5 % can be fabricated. The sintered fibers are straight with homogeneous consistency, showing a line fraction of parallel aligned fibers up to 97 %. See Fig. 3. This offers a high attainable volume concentration if processed to composites. Access to all technological steps allows now for processing of fiber with custom key functional data.

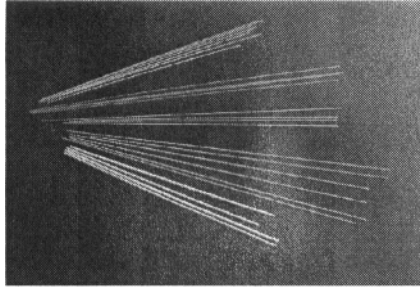


Fig. 3 shows ceramic fibers prepared by the polysulphone process<sup>15</sup>. The Diameter  $d$  can be varied between 100  $\mu\text{m}$  ... 1000  $\mu\text{m}$ .

#### *PZT plates by tape casting*

Basically, PZT plates are prepared by tape casting, drying and sintering under controlled process conditions. As result, single rectangular shaped PZT wafers are obtained. Different contours can be processed by Laser cutting (Fig 4). Dicing is successfully used to process rectangular shaped fibers, already parallel arranged on carrier foils. This is the key ceramic process in MFC fabrication.

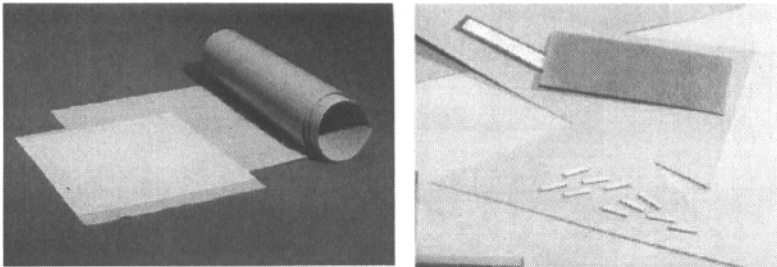


Fig. 4: PZT units by tape casting. Left: green tapes, right "tailored" units.

#### *PZT array by soft molding*

Arrays of distributed pillars can be manufactured using a patented technology that was invented at the Fraunhofer IKTS, Germany<sup>16,17</sup>. This process consists of copying a soft mold from a positive form of the final structure, filling the mold with a slip loaded with the calcined piezoceramic powder and subsequent firing of the element in PbO-controlled atmosphere.



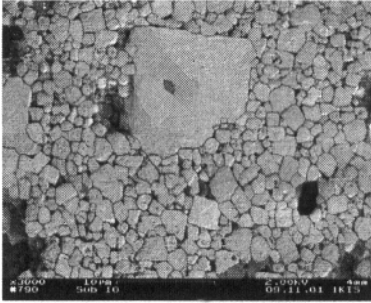


Figure 6. SEM micrograph of a polished and etched cross section of a PZT thick film

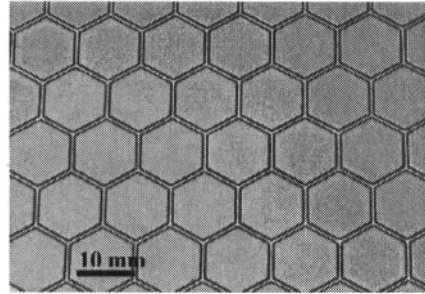


Figure 7. Honeycomb structured array of PZT thick films on metalized Si-wafer, prepared by screen printing technology.

Table 2: Properties of PZT thick films (active area  $a = 400 \text{ mm}^2$ , thickness  $100 \mu\text{m}$ ) on various substrate materials using Au electrodes

Property	PZT	PZT	PZT
	on $\text{Al}_2\text{O}_3$ (99.7 %)	on LTCC (DP 951)	on Si-wafer
Dielectric constant $\epsilon_{33}^T/\epsilon_0$ at 1 kHz	1900	1500	1600
Dielectric loss $\tan \delta$ at 1 kHz	0.038	0.033	0.055
Piezoelectric coefficient $d_{33}$ [pC/N]	210	180	140
Remnant polarization $P_r$ at 50 Hz [ $\mu\text{C}/\text{cm}^2$ ]	16	10	9
Coercive field $E_c$ at 50 Hz [kV/cm]	15	13	12
Internal resistance $R_{is}$ at 30 kV/cm [ $\Omega\text{cm}$ ]	$2 \times 10^{11}$	$2 \times 10^{11}$	$2 \times 10^{11}$

*Piezo fiber composites - Arrange and fill process*

Piezoelectric 1–3 fiber composites using the Fraunhofer methodology are prepared by epoxy infiltration of fiber bundles or fixed fiber patterns and curing and dicing of the composite. The obtained 1–3 fiber composite materials can be machined into virtually any shape or size transducer element. This “Arrange & Fill” process enables a quick, cost-efficient method of producing large quantities of composites. The piezoceramic fibers can be aligned either with or without spacer in a form and filled with polymer. Thus, regular or nonregular spacing patterns are obtainable. Especially know-how is required concerning the piezoceramic fiber fabrication, the fiber arrangement, epoxy selection, and the curing step. Originally developed at Fraunhofer IKTS, the process has been commercialized and up scaled by Smart Material Corp.<sup>14</sup>.

Obtained piezocomposite blocks (Fig. 8) can be machined into virtually any shape or size transducer element (example, see Fig. 8). Through the use of custom molds and precision machining, concave surfaces can be produced to create a broadband sensor. The presence of polymer creates a “cushion-effect” that protects the fibers and enables the structure to resist chipping and cracking during the machining process. In guided wave inspections, where sheets or tubes type surfaces require inspection without extensive scanning, a broadband transducer is required. The piezoelectric fiber composite not only provides this desirable characteristic, but also allows for high-pulse amplitude (low noise), conformability, and an excellent acoustic impedance match to the test piece.

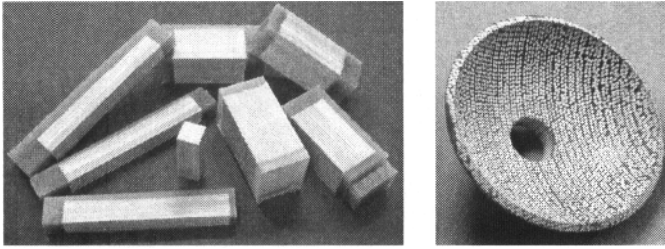


Figure 8. Left: piezo fiber composite block material, right: machined piezo fiber composite for transducer application

#### *Molded composites*

Fine scaled composites can be prepared starting with molded and sintered ceramic arrays made by the soft mold process. Once the piezoceramic pillars have been formed, the remaining spaces are filled with a polymer matrix material. Next the base is removed by grinding. Metal electrodes are then bonded to the ends of the fibers and first used to polarize the piezoceramic at an elevated temperature and then to apply an electric field or collect developed charges from the material. At this point, the active elements are ready to be used as piezoelectric transducer element. Experiments have shown that high-performance composites can be prepared<sup>17</sup>.

#### MODULES BY MICROSYSTEMS TECHNOLOGY

Exemplary developments of microsystems as pre-integration platform in smart structure technology are described in the present paragraph. There are two promising approaches seen allowing for the integration of piezoceramic units and electronic circuits, flexible circuits boards and active ceramic multilayer devices.

#### Flexible circuit boards

PZT plates, preferable in the thickness range between 100  $\mu\text{m}$  to 250  $\mu\text{m}$  in thickness, are processed to sensor and actuator modules by packaging using flexible circuit board processing. A plenty of designs are known<sup>20,21,22,14</sup>.

A modular concept for pre-encapsulated actuators has been developed and transferred to production by German Aerospace Center DLR Braunschweig<sup>22</sup>. The multifunctional elements can be designed to meet the requirements of industrial applications. This involves for example driving voltages, size and shape of the elements and the piezoceramic material itself. Recently, modules for raised operation temperatures up to 180°C have been developed. See Fig. 9. The selected materials allow for optimum actuator performance at 180 °C, see Fig. 10.

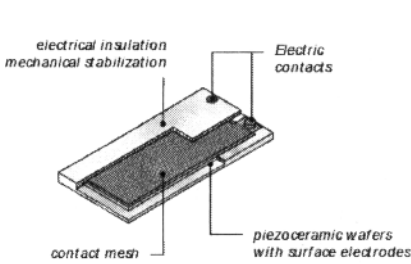


Figure 9: Sketch of the  $d_{31}$  module consisting of a monolithic layer of PZT which is embedded into epoxy, as originally developed by the German Aerospace Center<sup>22</sup>.

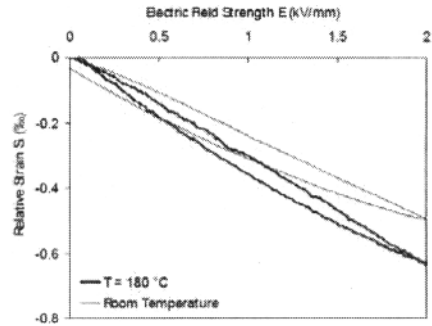


Figure 10: Measured strain characteristic of the special designed high temperature module at room temperature and 180°C

The sensor/actuator-modules modules, sketched in Fig. 9, are commercialized and are offered by PI Ceramic, Germany under the brand name DuraAct<sup>®</sup>. The transducers, typically, have an overall geometry of 40 x 20 x 0.5 mm<sup>3</sup>, the PZT plate of 30 x 15 x 0.2 mm<sup>3</sup>. Since the ceramic layer is embedded into a polymer structure, these modules offer a relative high damage tolerance; they even can be applied on curved structures.

The Macro-Fiber Composite, or MFC<sup>21,14</sup> is made up of rectangular uniaxially aligned ceramic fibers sandwiched between layers of adhesive and electroded polyimide films. These fibers are prepared by dicing of PZT plates using a wafer saw. Developed at NASA Langley Research Center during the late 90's, the MFC are manufactured by Smart Material Corp. in a full-scale production, today. A variety of design forms are offered<sup>14</sup>. The migration from research projects to high volume, cost effective commercial applications has generated additional need for new MFC designs, electronics on microcontroller and chip level, and system design tools, as well.

By applying voltage to the MFC, the ceramic fibers change shape to expand or contract and turn the resulting force into a bending or twisting action on the material. Likewise, voltage is generated in proportion to the force applied to the MFC material. Numerous research projects have proven the concept of using the MFC in vibration and noise control applications, as well as for health monitoring, morphing of structures and energy harvesting.

#### Active ceramic multilayer

IKTS is now developing a piezoelectric modules made completely of ceramic<sup>23</sup>. The new packaging technology uses the lamination of sintered PZT ceramic plates with green low temperature co-fire ceramic layers (LTCC, Heraeus HeraLock<sup>®</sup> Tape-HL2000) and post-sintering to obtain the sensor/actuator module. This design completely integrates the PZT in the substrate material.

This approach combines LTCC microsystems technology and piezo technology and allows for a tremendous improve of functional integration, e.g. sensing, actuation, buried electronic circuits, and strain- stress transformation. The challenge exists in avoiding tension cracks at shrinking of

LTCC layers on the already sintered piezoceramic during the firing process. The advantages of a module with fully integrated PZT ceramic unit are the mechanical stabilisation of the piezoceramic, the electrical insulation and the shielding of external environmental influences. Special designed modules were integrated into Al-components by die casting. The piezoelectric modules survived this manufacturing step with full functionality, confirming the idea of adaptive metal structures in automotive and machine building industry.

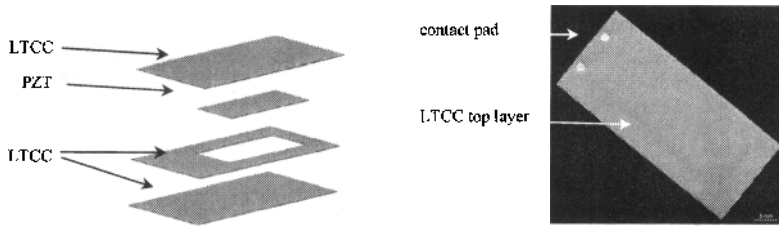


Figure 11: LTCC packaged piezo module<sup>23</sup>. Left: exploded view, right: prepared module

After preparation, the ceramic modules were introduced in the manufacturing chain of Aluminium die casting.

#### ADAPTIVE STRUCTURES

At present, a big number of research organizations (NASA, Fraunhofer, German Aerospace Center DLR, Universities) and customers (estimation a few hundred) are dealing with the use of active packaged devices for sensing, actuating, energy harvesting, health monitoring and structural control. The distribution of MFC by market segment was reported to amount to 20% defense (USA, Europe), 20% research labs, universities, 20% automotive, 15% machining equipment, 15% aerospace and 10% sporting goods, white ware, and buildings<sup>24</sup>. Examples of new approaches based on microsystems technologies are given in the next paragraph.

#### Active optical devices

The use of deformable mirrors to compensate the aberration of astronomical images caused by the turbulence of the earth atmosphere has led to outstanding successes of ground-based astronomy. In collaboration with the Active Structures Laboratory of Brussels University a demonstrator for a bimorph mirror was developed and manufactured as shown in Fig. 12<sup>25</sup>. It consists of a silicon wafer with a diameter of 150 mm and 0.75 mm thickness as well as 91 honeycomb thick film piezoelectric actuators with 80  $\mu\text{m}$  thickness, which have been screen printed at the backside of the silicon wafer. Each actuator can be driven individually to control the shape of the bimorph mirror and thus optical aberrations.

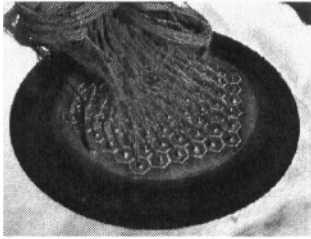


Figure 12. PZT thick film actuators with wiring for controlled driving and image correction

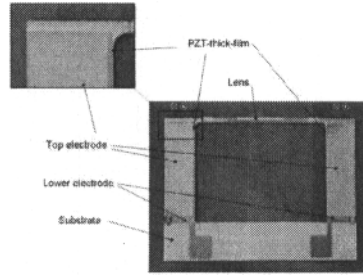


Figure 13. Active optical system for adjusting a FAC lens in with two degrees of freedom.

The combination of cantilever structures based on PZT thick films with solid hinges and platforms for optical devices enables for active systems with two or more degrees of freedom in deformation. In collaboration with the Fraunhofer IOF Jena, we developed a movable platform carrying the FAC (fast axis collimator) lens<sup>26, 27</sup>. See Fig. 13. Simultaneous driving of the two cantilevers with  $E = 2 \text{ kV/mm}$  resulted in an up and down movement of the platform. Static measurements of the deflection by laser triangulation method resulted in a deflection of  $\Delta l = 6 \text{ }\mu\text{m}$ . Tilting and twisting movements were possible by driving only one cantilevers.

Further investigations will build up on these experiments to open up new fields of applications for adaptive optical systems.

#### Active light metal components

Lightweight construction is a trend in car industry to save weight and hence to reduce fuel consumption. The use of light metals like aluminium or magnesium is one option. As general in light – weight structures, noise and vibrations becomes a problem. In 2004 we introduced the idea of direct integration of metalized and piezoelectric elements with insulating coating in a metal matrix by casting using a metallurgical fusing technique<sup>28</sup>. Bräutigam et al<sup>29</sup> investigated high pressure die casting for the preparation of active light metal structures. This technology is well established for mass production of light metal parts.

Due to the dynamic die filling and the high melt temperatures die casting is both mechanically and thermally a very challenging process for the integration of sensitive sensor/actuator-modules. For example, the velocity of the liquid metal is locally far above 100 m/s. Die filling usually takes place in less than 50 ms. As soon as the die is filled, a dwell pressure of several 100 bar is applied to compensate shrinkage and to minimize porosity. Aluminium alloys have a casting temperature within the range of 600°C to 750°C. The typical die temperature is between 150°C and 250°C.

Nevertheless, the piezoelectric LTCC-PZT modules as introduced in the preceding paragraph survived this manufacturing step without deterioration and fortify the concept of adaptive metal structures in automotive and machine building industry. Figs. 14 and 15 give an impression of the integrated ceramic module in an Aluminium plate. Die casting was done at University of Erlangen.

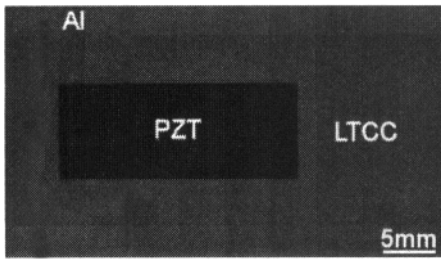


Figure 14: X-ray image (UA = 130 kV, I = 100  $\mu$ A) of an aluminium die casting block with integrated LTCC piezo module<sup>23</sup>

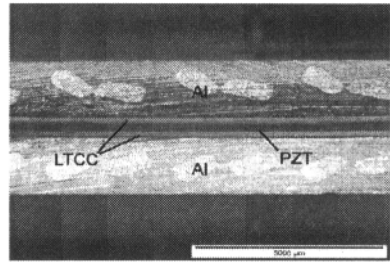


Figure 15: cross-section of an integrated LTCC piezo-module with PZT-unit<sup>23</sup>

#### Development of self-powered sensor nodes

Safety of materials and structures, especially in aircraft engineering, is currently guaranteed by redundant dimensioning, periodic inspections and preventive replacement of safety relevant assemblies. Recent success in sensor - and micro electronics technology opens up the perspective of in flight inspection. Key elements are self-organizing, wireless sensor nodes with implementation of energy harvesting capability. In 2009 a mayor industrial project started in Germany, aiming at energy efficient solutions in the three focus areas computing, broadband wireless, and sensor networks<sup>30</sup>. One lead project deals with the development of wireless, self-powered sensor nodes using guided acoustic waves for health monitoring and life time estimation of light-weight structures. A great impact on piezo and smart structure technology can be expected in the near future from that funding.

#### CONCLUSIONS

Smart structure technology is going to be used for advanced products in a multitude of industrial branches. We take microsystems technologies as most promising approach for pre-integration of sensor, actuator, generator and control functions into one module. This guarantees to meet the capability of mass production at competitive cost limits. The material and design basis of these modules must be adjusted to the type of load carrying structure and the conditions of its serial production technology. As shown by example, ceramic, Si and polymer based micro system approaches are attractive starting points. The combination of multifunctional piezoceramics with electronic circuits and control and communication software in one device give tremendous potential for structures of high functionality. Let us take the continuous built-in structural health monitoring approach, rather than a traditional NDT-type maintenance approach, by self-powered sensor nodes, as example to see the possible progress of the near future.

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