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Development of a novel coating process, devices, and their technology transfer with the discovery of Room Temperature Impact Consolidation phenomenon

常温衝撃固化現象の発見とプロセス、デバイス応用、そして産学連携

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1. Introduction

Technological innovations in manufacturing processes are critical for devices that require high-quality thick piezoelectric ceramic films. High-quality ceramics generally require sintering temperatures higher than 1000°C, which makes it difficult for integration into monolithic structures containing low melting temperature components or substrates such as metal, glass, or plastic. This incompatibility is a major obstacle for emerging electroceramic and optical components fabrication. In this regard, development of innovative new manufacturing processes is of critical importance in the near future for producing high performance functional devices that utilize thin/thick films and at the same time reduce the device fabrication cost. **Fig.1** highlights the application spectrum of piezoelectric films with desired thickness. Piezoelectric films with a thickness range of 1–100 µm have been used in devices such as microfluidics, micropumps, accelerometers, acoustic sensors, infrared detectors, and energy harvesters. For these applications, dense crack-free piezoelectric films are required. However, synthesis of films fulfilling these requirements is challenging, since these dense thick films are more suscep tible to cracks due to thermal stresses caused by differences between the thermal expansion coefficients of the film and substrate.As **Fig. 1** demonstrates, the film thickness is a major factor in tuning a device for a target application. If a film is too thin, it may generate too small of a displacement/force; on the other hand, a film that is too thick may require too high of a driving voltage. The window of utility for piezoelectric or electrostrictive materials that achieve the needed displacement/force and high-speed response are found to be a thickness that exceeds 1 µm, but this is thinner than bulk ceramics. However, thick films prepared by conventional methods usually exhibit cracks and may easily be peeled from the substrates, have difficulty in producing stoichiometrically complex materials, and require time-consuming and costly fabrication processing, which is prohibitive for industrial mass-production.

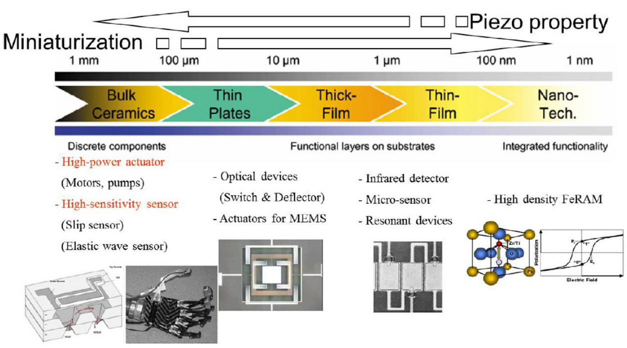

Fig.1 Application areas of piezoelectric materials with varying thickness.

2. Aerosol deposition method

The aerosol deposition (AD) process[1),2)] is a unique approach for depositing thick piezoelectric ceramic films to overcome these technological hurdles. This process is based on shock-loading solidification due to the impact of ceramic fine powder. In this process, (sub)-micron ceramic particles fluidized by gas flow are accelerated in a low-vacuum environment at velocities of up to 100–300 m/s to be impacted on the desired substrates. The impact results in a thick, dense, uniform, and homogeneous polycrystalline ceramic

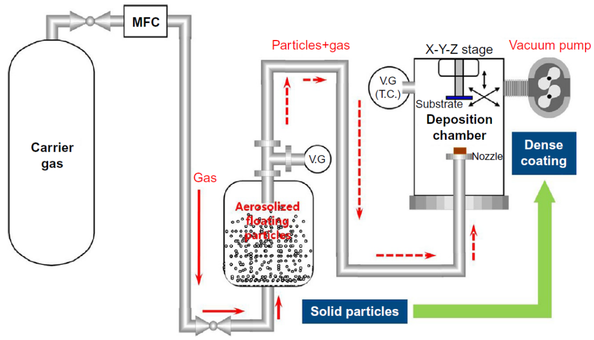

Fig.2 Schematic of AD system.

film formed at room temperature without the need for any additional heating to solidify the resulting film, shown in **Fig.2**. During the impact with the substrates, part of the particle's kinetic energy is converted into bonding energy causing an increase in surface activation at the point of impact, which promotes particle-substrate and particle-particle bonding. We found this interesting consolidation phenomenon of ceramic over 20 years ago and named "Room Temperature Impact Consolidation (R.T.I.C)". It is expected to reduce energy, cost, difficulty to fabricate the thin or thick film coating with complicate material compositions and the number of processes with lift-off for micro patterning during fabricating electronic devices and others, as well as to improve their performances substantially. AD process maybe one of the good candidate of the strategic advanced coating for a future innovative technology. This technology was transferred to TOTO Co. Ltd., and succeeded in commercialization from 2010 as Y_2O_3 ceramic coating for low plasma dust generation components of semiconductor manufacturing equipment. .

3. Fabrication of piezoelectric thick film

With traditional deposition processes, such as sol-gel, sputtering, pulsed laser, or chemical vapor deposition and etc., films thinner than 1 μm can be easily fabricated, but it is difficult to make films thicker than 10 μm due to the low deposition rate (about 1 μm/h) and high interfacial stress. Screen-printing is a well-known process capable of generating thick films at a very high production rate. However, this process suffers some shortcomings such as applicable substrate materials, low sintered density, high sintering temperature (typically over 800°C), weak adhesion strength between the film and substrate, and a quite porous microstructure with large pores. A process capable of forming dense films with a thickness in the range of 1–100 μm is urgently required to satisfy the needs of various applications.

The AD process can overcome these limitations. In ferroelectric and piezoelectric thick films, such high insulating and electrical breakdown characteristics are desired for high performance and low loss devices. Typically PZT films deposited by AD at room temperature exhibit polarization switching at a high external field over 1 MV/cm. The remanent polarization (Pr) and coercive field (Ec) were 13 $\mu C/cm^2$ and 300 kV/cm, respectively. These results indicate that as-deposited AD-PZT films have very high electrical resistance and breakdown voltage due to the high density film structure. These films also have spontaneous polarizations in spite of the structural defects introduced by the reduction of the crystallite size during AD process. It should be noted that these properties are unacceptable for practical applications, but they can be improved to acceptable standards by post-annealing in air at temperatures ranging from 500 to 700°C. As a result of this, post-deposition treatment grain growth of fine crystals and defect recovery in the AD films were observed, which dramatically improved the ferroelectric properties. However, to fabricate high-quality thick films by AD, stress control in the film is critical. By using the powders with organic species the residual stress was dramatically reduced from 250 MPa compressive stress to 46 MPa tensile stress. The stress relaxation in the PZT film was attributed to the evaporation of organics during the annealing process. This innovative fabrication process for stress control can maximize actuating/sensing/transducing properties of piezoelectric thick films, as well as related devices.

4. Application to Optical scanner

High performance micro optical scanner with wide scanning angle, high speed scanning and large mirror size is strongly demanded for a micro projector for head mount display and a laser sensor such as laser LIDAR (laser imaging detection and ranging for automatic driving vehicle), which are expected huge market as piezoelectric actuator in the world. We demonstrate the lamb wave resonance type optical scanning devices[3)] actuated by piezoelectric AD film and report their durability. The optical scanner with high scanning angle of over 90 degree were fabricated. A high optical scanning angle (over 60 ˚) with high resonance frequency (over 30kHz) for micro projector, and a high optical scanning angle (over 100 ˚) with large mirror size (20mm square) and with very low resonance frequency (blow 100Hz) for laser LIDAR were obtained in ambient air without vacuum packaging. The resonance frequency and scanning angle were not change during life test for over 50,000 hours.

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