

An electromechanical responsive filmTechnical Field

The present invention relates to an electromechanical responsive film. There is also
5 provided a method of forming the same.

Background

Electromechanical responsive materials such as piezoelectric materials can generate
mechanical strain in response to electric field or can conversely develop changes in
electric polarization when mechanical stress is applied. Such behaviour has been used
10 in a wide range of applications, including actuators, sensors, energy transducers,
acoustic filters, and biomedical detectors. The main indicator of the electromechanical
performance property of piezoelectric materials is the piezoelectric coefficient, i.e., larger
the piezoelectric coefficient, higher the performance for many electromechanical
actuators or sensors. Lead-based perovskite ferroelectric materials, such as lead
15 zirconate titanate (PZT), are widely used owing to their large piezoelectric coefficient.
However, lead-based materials are undesirable due to environmental and health
concerns.

Recently, aluminium nitride (AlN) has been more commonly used in view of its crystalline
quality and piezoelectric properties. Furthermore, AlN can be produced at lower
20 temperatures of less than 450°C compared to PZT, and is therefore more suitable for
integration with complementary metal oxide semiconductor (CMOS) technologies.
Compatibility with post-CMOS process line is highly demanded for microfabrication and
integration of piezoelectric devices with microelectronics for performance enhancement
in microdevices and cost reduction through mass production. So far, the highest
25 achieved piezoelectric coefficient of AlN is about 5.5 pm/V, which is 20 times smaller
than PZT. As a result, the application range of AlN is limited.

Further efforts have been made to enhance the piezoelectric coefficient of AlN by
partially replacing Al atoms with trivalent scandium (Sc) elements, resulting in a five-time
enhancement in the magnitude of the longitudinal piezoelectric coefficient to 31.6 pm/V.
30 However, the piezoelectric properties of AlN containing Sc are still far lower than PZT
thin films.

There is therefore a need for an improved electromechanical responsive material.

Summary of the invention

The present invention seeks to address these problems, and/or to provide an improved electromechanical responsive material.

According to a first aspect, there is provided an electromechanical responsive film
5 comprising aluminium scandium nitride, the aluminium scandium nitride having formula $Al_{1-x}Sc_xN_{1-2y/3}O_y$, wherein $0.45 \leq x \leq 0.6$ and $0 \leq y \leq 0.15$.

The electromechanical responsive film may have a suitable effective longitudinal piezoelectric coefficient ($d_{33,eff}$). According to a particular aspect, the $d_{33,eff}$ may be ≥ 50 pm/V. In particular, the $d_{33,eff}$ of the electromechanical responsive film may be 50-750
10 pm/V.

The electromechanical responsive film may be a polycrystalline film.

The aluminium scandium nitride comprised in the film may have a suitable crystalline structure. According to a particular aspect, the aluminium scandium nitride may have a mixed phase crystalline structure. In particular, the aluminium scandium nitride may
15 comprise a rock salt crystalline structure.

The electromechanical responsive film may be formed by any suitable method. For example, the electromechanical responsive film may be formed by physical vapour deposition or chemical vapour deposition. According to a particular aspect, the electromechanical responsive film may be formed by reactive sputtering.

20 The reactive sputtering may be carried out under suitable conditions. For example, the reactive sputtering may be carried out at a temperature of 25-450°C, in a reactive atmosphere comprising a mixture of nitrogen gas and inert gas and/or at a pressure of 0.75-2.0 mTorr. The mixture of nitrogen gas and inert gas may have a purity of 99-100%.

According to a second aspect, there is provided an electromechanical responsive device
25 comprising:

- a substrate; and
- an electromechanical responsive film according to the first aspect sandwiched between a first conductive layer and a second conductive layer, wherein the first conductive layer is on the surface of the substrate.

According to a particular aspect, the first conductive layer, the second conductive layer and the electromechanical responsive film may each have a suitable thickness. For example, the first conductive layer may have a thickness of 30-400 nm, the second conductive layer may have a thickness of 20-200 nm and/or the electromechanical responsive film may have a thickness of 100-2000 nm.

The first conductive layer may comprise a multi-layered structure. In particular, the multi-layered structure may comprise an adhesive layer for promoting adhesion of the first conductive layer with the surface of the substrate.

The second conductive layer may comprise any suitable material. For example, the second conductive layer may comprise a metal, a conductive oxide, a conductive nitride or a combination thereof.

Brief Description of the Drawings

In order that the invention may be fully understood and readily put into practical effect there shall now be described by way of non-limitative example only exemplary embodiments, the description being with reference to the accompanying illustrative drawings. In the drawings:

Figure 1 shows a schematic representation of a set-up for forming an electromechanical responsive film according to one embodiment;

Figure 2 shows the hysteresis in deposition rate as a function of reactive gas flow according to one embodiment;

Figure 3 shows a schematic representation of an electromechanical responsive device according to one embodiment;

Figure 4 shows a x-ray diffraction (XRD) intensity plot as a function of diffraction angle of an electromechanical responsive film according to one embodiment;

Figure 5 shows a plot of effective longitudinal piezoelectric strain coefficient ($d_{33,eff}$) as a function of frequency for an electromechanical responsive film according to one embodiment;

Figure 6 shows a 3-dimensional surface profile of surface displacement with respect to X and Y positions of an electromechanical responsive film according to one embodiment;

Figure 7 shows a XRD intensity plot as a function of diffraction angle of an electromechanical responsive film according to one embodiment;

Figure 8 shows the depth profile of elemental composition in an electromechanical responsive film according to one embodiment using X-ray photoelectron spectroscopy;

- 5 Figure 9 shows a plot of $d_{33,eff}$ as a function of frequency for an electromechanical responsive film according to one embodiment;

Figure 10 shows a 3-dimensional surface profile of surface displacement with respect to X and Y positions of an electromechanical responsive film according to one embodiment;

- 10 Figure 11 shows the depth profile of elemental composition in an electromechanical responsive film according to one embodiment using X-ray photoelectron spectroscopy;

Figure 12 shows a XRD intensity plot as a function of diffraction angle of an electromechanical responsive film according to one embodiment;

Figure 13 shows a plot of $d_{33,eff}$ as a function of frequency for an electromechanical responsive film according to one embodiment;

- 15 Figure 14 shows a 3-dimensional surface profile of surface displacement with respect to X and Y positions of an electromechanical responsive film according to one embodiment;

Figure 15 shows a XRD intensity plot as a function of diffraction angle of an electromechanical responsive film according to one embodiment;

- 20 Figure 16 shows the depth profile of elemental composition in an electromechanical responsive film according to one embodiment using X-ray photoelectron spectroscopy; and

Figure 17 shows a 3-dimensional surface profile of surface displacement with respect to X and Y positions of an electromechanical responsive film according to one embodiment.

Detailed Description

- 25 As explained above, there is a need for an improved electromechanical responsive material.

In general terms, the present invention provides an improved electromechanical responsive film. The film may have a significantly higher effective longitudinal piezoelectric coefficient ($d_{33,eff}$) as compared to prior art electromechanical responsive materials. In particular, the $d_{33,eff}$ may be 50-750 pm/V. This may enhance the electromechanical output of devices such as actuators and sensors comprising such films as their performance is determined by the effective piezoelectric coefficient.

In particular, while aluminium scandium nitride thin films are known in the art, these films have the formula $Al_{1-x}Sc_xN$, where $0 \leq x \leq 0.43$, and exhibit electromechanical response only when the films have highly oriented wurtzite crystal structures owing to the non-centrosymmetric nature of the wurtzite structure. Generally, the $d_{33,eff}$ of the aluminium scandium nitride thin films of prior art may be 5.5-31.6 pm/V. It is known that when the films have a rock salt crystal structure, i.e. when $x \geq 0.43$, there is negligible electromechanical response.

In contrast, and unexpectedly, the aluminium scandium nitride film described in the present application has a large $d_{33,eff}$ despite having scandium content beyond the scandium composition range in prior art, and having a main rock salt crystal structure which is centrosymmetric. For example, the $d_{33,eff}$ of the aluminium scandium nitride film described in the present application may be larger than 50 pm/V, particularly at an applied electric field of 45 kV/cm and below 200 Hz.

According to a first aspect, there is provided an electromechanical responsive film comprising aluminium scandium nitride, the aluminium scandium nitride having general formula $Al_{1-x}Sc_xN_{1-2y/3}O_y$, wherein $0.45 \leq x \leq 0.6$ and $0 \leq y \leq 0.15$.

For the purposes of the present application, x and y refer to the atomic percentage, expressed as a decimal. Accordingly, the aluminium scandium nitride comprised in the electromechanical responsive film may have scandium content of 45-60 atomic %.

It is known in the prior art that increasing the content of scandium in $Al_{1-x}Sc_xN$ films improves its piezoelectric properties. However, the largest achieved piezoelectric coefficient is 31.6 pm/V when Sc content is 41 atomic %. Generally, it is known that the enhancement in electromechanical response of $Al_{1-x}Sc_xN$ depends on Sc content preferably in the range of $0 \leq x \leq 0.43$, where the crystal structure of $Al_{1-x}Sc_xN$ film is wurtzite. The large electromechanical strain in response to applied electric field in $Al_{1-x}Sc_xN$ films has been known to be due to softening of crystal lattice and structural

destabilization as result of Sc substitution in AlN lattice with wurtzite phase. This is because of the difference in preferred coordination of aluminium (4-fold) and scandium (5 or 6-fold) about the nitrogen. So far, these effects have been found to be advantageous for enhancing electromechanical response of $\text{Al}_{1-x}\text{Sc}_x\text{N}$ films only up to $x \approx 0.43$. When $x > 0.43$, it is known that there is an abrupt drop in piezoelectric coefficient.

Further, the crystal structure of $\text{Al}_{1-x}\text{Sc}_x\text{N}$ films changes as the scandium content changes. In particular, $\text{Al}_{1-x}\text{Sc}_x\text{N}$ films may have a wurtzite or rock salt crystal structure – when the scandium content is 0-43 atomic %, the film may grow with a wurtzite structure, and when the scandium content is 43-100 atomic %, the film may exhibit rock salt structure. The crystal phase transition between the two structures may not be abrupt and therefore, an intermediate transition region may exist between pure wurtzite and pure rock salt phases of $\text{Al}_{1-x}\text{Sc}_x\text{N}$. The intermediate transition region may possibly have mixed phases and large number of defect densities. The film may have fine-grained crystallites. Given the oxygen content in the range of $0 \leq y \leq 0.15$, the film may contain a large number of defects such as grain boundaries and self-compensating vacancies.

In some cases, such intermediate transition region may be utilized advantageously to obtain large electric-field induced strain response in $\text{Al}_{1-x}\text{Sc}_x\text{N}$ films in comparison to conventional pure wurtzite phase $\text{Al}_{1-x}\text{Sc}_x\text{N}$ with $0 \leq x \leq 0.43$. However, with rock salt phase present when $x \geq 0.43$, an abrupt drop in the strain response is well-known.

Accordingly, it is unexpected that the electromechanical responsive film according to the first aspect has a suitable high effective longitudinal piezoelectric coefficient ($d_{33,\text{eff}}$). According to a particular aspect, the $d_{33,\text{eff}}$ of the electromechanical responsive film may be ≥ 50 pm/V, particularly ≥ 100 pm/V. In particular, the $d_{33,\text{eff}}$ of the electromechanical responsive film may be 50-750 pm/V, 75-700 pm/V, 100-650 pm/V, 150-600 pm/V, 200-500 pm/V, 250-450 pm/V, 300-400 pm/V. The $d_{33,\text{eff}}$ of the electromechanical responsive film may be measured at an applied electric field of 45-75 kV/cm and frequency ≤ 200 Hz.

The electromechanical responsive film may be a polycrystalline film. The polycrystalline film may contain a large number of crystal grains with different orientations and hence grain boundaries. Grain boundaries may be regarded as a common type of defect. Accordingly, such defects may influence the electromechanical response of the film.

The aluminium scandium nitride comprised in the film may have a suitable crystalline structure. According to a particular aspect, the aluminium scandium nitride may have a mixed phase crystalline structure. The mixed phase crystalline structure may comprise a mix of wurtzite and rock salt crystalline structures. For example, the mixed phase crystalline structure may comprise wurtzite and rock salt crystalline structures when the value of x in $\text{Al}_{1-x}\text{Sc}_x\text{N}$ is in the range of 0.4 to 0.6. In particular, the mixed phase crystalline structure may comprise a predominantly wurtzite crystalline structure when the value of x in $\text{Al}_{1-x}\text{Sc}_x\text{N}$ is 0.4-0.5. In particular, the mixed phase crystalline structure may comprise a predominantly rock salt crystalline structure, particularly when the value of x in $\text{Al}_{1-x}\text{Sc}_x\text{N}$ is in the range of 0.5-0.6. Even more in particular, when the value of x in $\text{Al}_{1-x}\text{Sc}_x\text{N}$ increases from 0.6 to 1, the crystalline structure may comprise rock salt crystalline structure.

According to another aspect, there is provided a method of forming the electromechanical responsive film described above. The electromechanical responsive film may be formed by any suitable method.

For example, the electromechanical responsive film may be formed by physical vapour deposition or chemical vapour deposition. In particular, the film may be formed by physical vapour deposition methods comprising, but not limited to, magnetron sputtering, molecular beam epitaxy, laser ablation or any other suitable method capable of depositing compound films with control over stoichiometry of the film.

The electromechanical responsive film may be formed by chemical vapour deposition methods comprising, but not limited to, metal organic chemical vapour deposition, and atomic layer deposition.

According to a particular aspect, the electromechanical responsive film may be formed by reactive sputtering, such as but not limited to, reactive magnetron sputtering. This enables the electromechanical responsive film to be formed at low temperatures. For example, the electromechanical responsive film may be formed by sputtering an $\text{Al}_{1-x}\text{Sc}_x$ alloy target or by co-sputtering Al and Sc (two separate targets). In the case of using an alloy target, the composition of the film may be determined by the target composition. In contrast, in the case of co-sputtering, the composition of the film may be determined by the relative power applied to the separate targets used in the co-sputtering.

Figure 1 provides a schematic representation of a reactive magnetron sputtering apparatus according to one embodiment. The co-sputtering system comprises a vacuum chamber 300, two sputtering guns 301 and 302, and a heater assembly 304 with a sample holder 303. The chamber 300 may be evacuated using vacuum pumping system through pump inlet at 306 and has a gas inlet 305. Internally, chamber 300 may be provided with sputtering targets 307 and 308 attached to sputtering guns 301 and 302, respectively. Externally, the sputtering guns 301 and 302 are connected to power supply. Any power supply among radio frequency (RF), direct current (DC), pulsed DC power supply or their variants or combination may be used. Choice of the power supply may depend on the target material (whether made of metal or insulator) and process design. Optionally, chamber 300 may have multiple number of sputtering guns and targets such that thin films with various elemental compositions may be deposited.

For reactive sputtering, metals may be used as targets 307 and 308, and reactive gases in addition to inert gas may be introduced to the chamber 300 through gas inlet 305. The plasma of gaseous mixture comprising reactive gas may be generated by applying a negative bias to target either through RF or DC or pulsed power supply. The sputtering guns 301 and 302 may be provided with magnets attached behind sputtering targets to confine secondary electrons near the target surface which enables sustaining the plasma at low pressure.

Choice of the materials used as targets 307 and 308 may be made based on the material needed to be deposited. For example, to deposit aluminium scandium nitride film, aluminium and scandium metals may be used as targets 307 and 308, respectively, for co-sputtering. Alternatively, a target made of alloy such as $Al_{1-x}Sc_x$ may be used to deposit the film by reactive sputtering. If the alloy target is used, the composition of the film formed may be determined by the composition of the alloy target used. With co-sputtering targets 307 and 308, films having a wide range of compositions may be obtained by adjusting the electric bias applied to the targets 307 and 308, particularly for the composition range which may be difficult to obtain with an alloy target.

Any combination of power supply among RF, DC or pulsed DC may be used to apply bias to targets 307 and 308. In some embodiments, the aluminium scandium nitride film may be deposited by co-sputtering aluminium target 307 and scandium target 308 wherein RF power is applied to aluminium target 307 and DC power is applied to

scandium target 308. To obtain the composition of interest of the film, the electric power density applied to targets 307 and 308 may vary.

According to a particular aspect, the film may be formed by applying electric power density in the range 1.97-3.73 W/cm² to scandium target 308 while keeping electric power density on aluminium target 307 at about 4.39 W/cm². A similar composition may also be obtained by keeping electrical power density constant on scandium target 308 and varying the electric power density on aluminium target 307 and/or by varying power density on both targets 307 and 308 simultaneously.

The reactive gas such as nitrogen along with inert gas such as, but not limited to, argon may be introduced into vacuum chamber 300 through gas inlet 305 during the deposition process. The plasma containing reactive gas may react with sputter target in a similar way as it does with the film being deposited. Figure 2 illustrates the influence of reactive gas on deposition rate, which also depicts the hysteresis in the deposition process as a function of reactive gas flow. Depending on proportion of nitrogen in gaseous mixture, the sputtering process may take place either in metallic mode or in compound mode (also referred as poison or reactive mode).

When nitrogen concentration is low, such that the target surface remains metallic or partially covered by a nitride layer (due to reaction with nitrogen), this is referred to as metallic mode. When nitrogen content is sufficiently high to react and the target surface is completely covered with a nitride layer, this is referred to as compound mode.

Growth rate in metallic mode is higher as compared to compound mode. Generally, compound mode is preferred for obtaining highly oriented film deposition. In several embodiments, the film may be deposited with nitrogen concentration within the range of 43% to 100% where the sum of N₂ and Ar is 100%. The different natures of compounds formed on the target surface, for instance, the insulating nature of compound aluminium nitride on the aluminium target and the conductive nature of scandium nitride in the scandium target may alter the sputter rate and hence the deposition rate. Such an effect may also provide a film with varied composition.

According to another particular aspect, in preparing the film, the vacuum chamber 300 may be pumped down to a base pressure lower than 5×10^{-8} torr and high purity gas sources, such as 99-100% purity, more particularly about 99.999% purity argon and nitrogen may be used. The purity of the targets may be about 99-100%, more particularly

about 99.99%. For example, the purity of the aluminium target may be about 99-100%, more particularly 99.9%, and that of scandium may be about 99-100%, more particularly 99.9%. However, both aluminium and scandium may exhibit a large affinity for oxygen, and may act as a source of oxygen impurity in the film. The oxygen content in the film
5 may be controlled by growth conditions such as electric power density applied to the target and growth temperature.

Further, electrically powered heater assembly 304 may be used to variably adjust the temperature of a substrate placed on sample holder 303. The substrate may be any suitable substrate on which the film is to be deposited or formed. The film may be formed
10 at a suitable temperature. For example, the temperature may be in the range of room temperature to 450°C, 25-450°C. The pressure at which the film is formed may be any suitable pressure. For example, the total pressure during the deposition process may be about 0.75-2 mTorr, preferably, 1-1.75 mTorr, and the distance between targets 307 and 308 and substrate 202 may be 15-16 cm.

15 According to a second aspect, there is provided an electromechanical responsive device comprising:

- a substrate; and
- an electromechanical responsive film according to the first aspect sandwiched between a first conductive layer and a second conductive layer, wherein the
20 first conductive layer is on the surface of the substrate.

In particular, the device may have a stacked arrangement comprising several layers to form the device. Figure 3 provides a schematic representation of a cross-sectional view of the device according to the second aspect. Accordingly, the device may comprise a
25 substrate 202, a first conductive layer 203 on a substrate surface, the electromechanical responsive film 201 according to the first aspect provided on the first conductive layer 203 and a second conductive layer 204 on the electromechanical responsive film 201.

The substrate, which supports the whole stacked arrangement of the device, may be formed of any suitable material. For example, the substrate may be formed of a bulk
30 material made of any one of single crystalline, polycrystalline or an amorphous material. According to a particular aspect, the substrate may be a single crystalline substrate and formed of materials such as, but not limited to, sapphire (Al_2O_3), silicon (Si), silicon

carbide (SiC), diamond, aluminium nitride (AlN), or a combination thereof. Examples of polycrystalline substrates may include, but not limited to, polysilicon, ceramics such as aluminium oxide, aluminium nitride, silicon carbide, metals or metal alloys, or combinations thereof. Amorphous substrates may include, but are not limited to, glass,
5 metallic glass, or any suitable organic material. The substrate choice may depend on the intended application of the electromechanical responsive film. For example, if the intended application is as a microelectromechanical device application or integration with CMOS devices, the substrate may be silicon or post-CMOS device as substrate material.

The first conductive layer may be formed over a surface of the substrate. The first
10 conductive layer may function as the bottom electrode. For example, the first conductive layer may be used to promote highly oriented growth or even epitaxial growth. Hence, the first conductive layer may be epitaxial layer or polycrystalline layer or highly textured layer. It may also be desirable that the first conductive layer is a stable material in subsequent deposition or fabrication processes.

15 The first conductive layer may be formed from any suitable material. For example, the first conductive layer may comprise metals such as, but not limited to, platinum (Pt), tungsten (W), molybdenum (Mo), tantalum (Ta), aluminium (Al), ruthenium (Ru), hafnium (Hf), or alloys thereof. The first conductive layer may comprise an alloy made of multiple metals such as, but not limited to, AlCu, PdAg. The first conductive layer may be an
20 epitaxial layer of conductive compound materials such as, but not limited to, titanium nitride (TiN), hafnium nitride (HfN), tantalum nitride (Ta₃N₅), zirconium nitride (ZrN), zirconium diboride (ZrB₂).

The first conductive layer may be a single layer or a multi-layered structure comprising an adhesion layer for promoting adhesion of the first conductive layer with the surface of
25 the substrate. For example, the first conductive layer may comprise, but is not limited to, Ti/Pt, Ti/TiN/AlCu, such that the Ti layer may be an adhesive layer. The adhesive layer may have a suitable thickness. In particular, the adhesive layer may have a thickness of 1-20 nm on the substrate side acting as an adhesive, that is between the first conductive layer and substrate. In particular, the thickness may be 2-18 nm, 5-15 nm, 10-12 nm.
30 Even more in particular, the thickness may be about 2 nm.

The first conductive layer may have a suitable thickness. For example, the first conductive layer may have a thickness of 30-400 nm. In particular, the thickness may be

50-350 nm, 100-300 nm, 150-250 nm, 200-220 nm. Even more in particular, the thickness may be 30-200 nm.

The second conductive layer may be formed on top of the electromechanical responsive film. In particular, the second conductive layer may serve as a top electrode in the
5 electromechanical device comprising the electromechanical responsive film.

The second conductive layer may be formed of any suitable material. For example, the second conductive layer may be formed from any metal such as, but not limited to, Pt, Au, Ag, Al, Cu, Mo, or alloys thereof. The second conductive layer may be formed from conductive oxides such as, but not limited to, indium tin oxide, ruthenium oxide, fluorine-
10 doped tin oxide, or from conductive nitrides such as, but not limited to, titanium nitride, tantalum nitride, hafnium nitride, zirconium nitride, or combinations thereof.

The second conductive layer may have a suitable thickness. For example, the second conductive layer may have a thickness of 20-200 nm. In particular, the thickness may be 25-175 nm, 50-150 nm, 75-125 nm, 100-120 nm. Even more in particular, the thickness
15 may be 20-100 nm.

The electromechanical responsive film comprised in the device may have a suitable thickness. For example, the thickness of the film may be 100-2000 nm. In particular, the thickness may be 150-1500 nm, 200-1000 nm, 300-900 nm, 400-800 nm, 500-700 nm, 600-650 nm. Even more in particular, the thickness may be 200-1000 nm.

20 The advantage of the electromechanical responsive film is that the film has a large strain response and thus, a higher longitudinal piezoelectric coefficient as compared to conventional piezoelectric materials such as PZT, AlN and $\text{Al}_{1-x}\text{Sc}_x\text{N}$ (where $0 \leq x \leq 0.43$). Further, since the film is lead-free and can be processed at relatively low temperature, it is environmentally friendly and also fully compatible with CMOS technology. Fabricating
25 the film may also be achieved at low cost and in a scalable manner. Accordingly, the film may be incorporated into devices such as, but not limited to, miniaturised electromechanical actuators, microelectromechanical systems (MEMS) based on-chip actuators, low frequency electromechanical sensors, and low frequency transducers for non-destructive testing, condition monitoring or structural health monitoring.

Having now generally described the invention, the same will be more readily understood through reference to the following example which is provided by way of illustration, and is not intended to be limiting.

Examples

5 *Example 1*

A device with a stacked arrangement as shown in Figure 3 was fabricated, wherein the electromechanical responsive film 201 had a composition of $\text{Al}_{0.47}\text{Sc}_{0.53}\text{N}_{0.95}\text{O}_{0.08}$, the first conductive layer 203 was formed of (111) oriented TiN, and the substrate 202 was c-sapphire (or c plane of Al_2O_3).

- 10 A magnetron sputtering apparatus consisting of a 3-inch-diameter Al and Sc target, a substrate holder and a heater assembly were placed at a distance approximately 15 cm away from the targets. An RF forward electric power density of 4.39 W/cm^2 was applied to the Al target and a DC electric power density of 3.73 W/cm^2 was applied to the Sc target. The film 201 was deposited at a substrate temperature of 400°C with 100%
15 nitrogen concentration in a reactive atmosphere and growth pressure of 1 mTorr. The film 201 was deposited at a growth rate of 2.8 nm/min .

Figure 4 is the plot of XRD intensity (in arbitrary units) as a function of diffraction angle 2θ (in degrees) showing diffraction pattern of the film having formula $\text{Al}_{0.47}\text{Sc}_{0.53}\text{N}_{0.95}\text{O}_{0.08}$ on (111) oriented TiN layer. The diffraction pattern revealed that the film predominantly
20 existed in rock salt phase. The chemical composition of the film was determined using X-ray photoelectron spectroscopy (XPS). The effective piezoelectric coefficient ($d_{33,\text{eff}}$) of the film was determined using PolyTech GmbH (Germany) scanning laser vibrometer. For this measurement, an Au top electrode (or second electrode 204, as shown in Figure 3) with a diameter of about $200 \mu\text{m}$ was deposited on $\text{Al}_{0.47}\text{Sc}_{0.53}\text{N}_{0.95}\text{O}_{0.08}$.

- 25 Figure 5 shows the $d_{33,\text{eff}}$ as a function of frequency when an alternating electric field was applied. At 180 Hz, the film exhibited $d_{33,\text{eff}}$ of 744.5 pm/V . The corresponding 3-dimensional surface displacement profile of the film under a unipolar alternating current electric field of about 47.6 kV/cm (corresponding applied voltage of 2 V) at 180 Hz is shown in Figure 6.

30 *Example 2*

A device with similar configuration as described in Figure 3 was formed, where the electromechanical responsive film 201 was the same as that described in Example 1. The first conductive layer 203 was (111) oriented platinum (Pt) and the substrate 202 was single crystalline silicon (Si). An amorphous SiO₂ and ultra-thin titanium of about 2 nm thickness was provided between the Si and Pt to enhance the adhesion of Pt with Si substrate. All other deposition parameters were the same as described in Example 1.

Figure 7 provides the plot of XRD intensity (in arbitrary units) as a function of diffraction angle 2θ (in degrees) showing the XRD pattern of the electromechanical responsive film deposited on (111) oriented Pt. The XRD pattern indicates that the film exhibited predominantly rock salt phase.

Figure 8 shows the depth profile of chemical composition of the film determined using XPS. According to Figure 8, the elemental composition particularly of aluminium and scandium are uniform across the thickness of the film, while oxygen content varied from 8.88 to 6.87 atomic %.

Figure 9 shows the $d_{33,eff}$ as a function of frequency under an alternating unipolar electric field of about 47.6 kV/cm. At 180 Hz, the film exhibited $d_{33,eff}$ of 103.8 pm/V. The corresponding 3-dimensional surface displacement profile of the film under a unipolar alternating current electric field at 180 Hz is shown in Figure 10.

Example 3

A device with similar configuration as described in Figure 3 was formed, in which the electromechanical responsive film had a composition of Al_{0.51}Sc_{0.49}N_{0.93}O_{0.11}. The first conductive layer 203 was (111) oriented Pt and substrate 202 was Si. An amorphous SiO₂ and ultra-thin titanium of about 2 nm thick was provided in between Si and Pt to enhance the adhesion of Pt with Si substrate. For this example, N₂ concentration was reduced to 57% and growth pressure was maintained at 1.75 mTorr. The remaining growth conditions were maintained the same as Examples 1 and 2. The deposition rate of the film was about 2.91 nm/min.

Chemical composition of the film was determined using XPS. Figure 11 shows the compositional depth profile of the electromechanical responsive film of Example 3, which indicates that the composition of Al and Sc was uniform across the thickness of the film

but oxygen concentration reduced slightly with the depth of the film. The surface oxygen content was around 10.6%.

Figure 12 is the plot of XRD intensity (in arbitrary units) as a function of diffraction angle 2θ (in degrees) showing XRD pattern of the electromechanical responsive film deposited on (111) oriented Pt. The XRD pattern indicates that the film exhibited predominantly rock salt phase.

Figure 13 shows the $d_{33,eff}$ as a function of frequency under an alternating unipolar electric field of about 72 kV/cm. At 110 Hz, the film exhibited $d_{33,eff}$ of 109.8 pm/V. The corresponding 3-dimensional surface displacement profile of the film under a unipolar alternating current electric field of about 72 kV/cm at 110 Hz is shown in Figure 14.

Example 4

A conventional aluminium scandium nitride film was formed, which exhibited intrinsic piezoelectric property. A stacked structure such as that as shown in Figure 3 was formed, where a first conductive layer 203 was (111) TiN and substrate 202 was c-sapphire. And the film 201 was the conventional aluminium scandium nitride film.

Figure 15 shows the plot of XRD intensity (in arbitrary units) as a function of diffraction angle 2θ (in degrees) showing XRD pattern of the film having formula $Al_{0.65}Sc_{0.35}N$. The XRD pattern was typical of a highly c axis (or (002)) oriented wurtzite phase aluminium scandium nitride film. The chemical composition of the film was determined using XPS.

Figure 16 shows the compositional depth profile of the film. As shown in the figure, the oxygen content in the film was less than 2.5 %, which was significantly lower than those obtained in the previous examples. Hence, the $Al_{0.65}Sc_{0.35}N$ film was expected to show intrinsic piezoelectric properties, such as significantly less voltage and frequency-dependent piezoelectric coefficient. The $d_{33,eff}$ of the film was about 8.7 pm/V at 180 Hz. The corresponding 3-dimensional surface displacement profile of the film under an alternating current electric field of 50 kV/cm at 180 Hz is shown in Figure 17.

Thus, it can be seen that in contrast to the conventional wurtzite phase aluminium scandium nitride thin film as described in Example 4, the aluminium scandium nitride films with substantial rock salt phase as described in Examples 1 to 3 exhibited significant enhancement in effective piezoelectric coefficient. Accordingly, such high effective

piezoelectric coefficient can offer large electromechanical outputs when incorporated in devices such as, but not limited to, MEMS-based actuators and sensors.

Whilst the foregoing description has described exemplary embodiments, it will be
5 understood by those skilled in the technology concerned that many variations may be made without departing from the present invention.

Claims

1. An electromechanical responsive film comprising aluminium scandium nitride, the aluminium scandium nitride having formula $\text{Al}_{1-x}\text{Sc}_x\text{N}_{1-2y/3}\text{O}_y$, wherein $0.45 \leq x \leq 0.6$ and $0 \leq y \leq 0.15$.
- 5 2. The electromechanical responsive film according to claim 1, wherein the electromechanical responsive film has an effective longitudinal piezoelectric coefficient ($d_{33,\text{eff}}$) of ≥ 50 pm/V.
- 10 3. The electromechanical responsive film according to claim 2, wherein the electromechanical responsive film has a $d_{33,\text{eff}}$ of 50-750 pm/V.
4. The electromechanical responsive film according to any preceding claim, wherein the aluminium scandium nitride has a mixed phase crystalline structure.
- 15 5. The electromechanical responsive film according to any preceding claim, wherein the aluminium scandium nitride comprises a rock salt crystal structure.
6. The electromechanical responsive film according to any preceding claim, wherein
20 the electromechanical responsive film is a polycrystalline film.
7. The electromechanical responsive film according to any preceding claim, wherein the electromechanical responsive film is formed by physical vapour deposition or chemical vapour deposition.
- 25 8. The electromechanical responsive film according to claim 7, wherein the electromechanical responsive film is formed by reactive sputtering.
9. The electromechanical responsive film according to claim 8, wherein the reactive
30 sputtering is carried out at a temperature of 25-450°C.
10. The electromechanical responsive film according to claim 8 or 9, wherein the reactive sputtering is carried out in a reactive atmosphere comprising a mixture of nitrogen gas and inert gas.

11. The electromechanical responsive film according to claim 10, wherein the mixture of nitrogen gas and inert gas has a purity of 99-100%.
- 5 12. The electromechanical responsive film according to any of claims 8 to 11, wherein the reactive sputtering is carried out at a pressure of 0.75-2.0 mTorr.
13. An electromechanical responsive device comprising:
- a substrate; and
 - 10 - an electromechanical responsive film according to any preceding claim sandwiched between a first conductive layer and a second conductive layer, wherein the first conductive layer is on a surface of the substrate.
14. The electromechanical responsive device according to claim 13, wherein the first
15 conductive layer has a thickness of 30-400 nm.
15. The electromechanical responsive device according to claim 13 or 14, wherein the first conductive layer comprises a multi-layered structure.
- 20 16. The electromechanical responsive device according to claim 15, wherein the multi-layered structure comprises an adhesive layer for promoting adhesion of the first conductive layer with the surface of the substrate.
17. The electromechanical responsive device according to any of claims 13 to 16,
25 wherein the second conductive layer comprises a metal, a conductive oxide, a conductive nitride or a combination thereof.
18. The electromechanical responsive device according to any of claims 13 to 17,
wherein the second conductive layer has a thickness of 20-200 nm.
- 30 19. The electromechanical responsive device according to any of claims 13 to 18, wherein the electromechanical responsive film has a thickness of 100-2000 nm.