Veröffentlichung

Autoren

Wierach, Peter;Behr, Christian;Sinapius, Michael

Titel

DIFFUSION-RESISTANT COATING FOR HARSH ENVIRONMENT APPLICATION OF PIEZOELECTRIC ACTUATORS

Publisher o. Konferenz

SMASIS, San Antonio, 2018

Jahr

2018

Internet-Link (Doi-Nr.)

Proceedings of the ASME 2018 Conference on Smart Materials, Adaptive Structures and Intelligent Systems
SMASIS2018
September 10-12, 2018, San Antonio, Texas, USA

SMASIS2018-8234

DIFFUSION-RESISTANT COATING FOR HARSH ENVIRONMENT APPLICATION OF PIEZOELECTRIC ACTUATORS

Christian Behr
DLR
German Aerospace Center
Braunschweig, Germany
c.behr@dlr.de

Peter Wierach
DLR
German Aerospace Center
Braunschweig, Germany
peter.wierach@dlr.de

Michael Sinapius
Technische Universität
Braunschweig
Braunschweig, Germany
m.sinapius@tu-braunschweig.de

1 ABSTRACT

This paper presents the development process of an electrically insulating and liquid-impermeable coating for piezoelectric actuators. Against the background of flow investigations of an adaptive airfoil in a water tunnel the adaptive lip including PZT-ceramics for the active lip deformation must be insulated and sealed up against the ingress of moisture. Due to high electric field strength of 2 kV/mm between electrodes of multilayer actuators any ingress of moisture would lead to a reduction of the dielectric strength and may cause a short circuit. In order to prevent failure of the adaptive lip the electrical connections of the actuators have to be insulated by a waterproof coating. A service life of at least 10^7 load cycles at a frequency of 100 Hz is required for the actuators. Therefore the coating should be as ductile as possible otherwise it could crack and water could diffuse into the actuators. That is why the yield strength of the coating has to be higher than of the actuators, which is 0.3 %. For the investigation of the waterproofness several samples are coated with different materials in various processes. First the actuators are moulded in epoxy resin and then a diffusion-resistant PVF-foil is applied. After a screening of different materials, an additional coating with a two-component tar-epoxy resin in combination with a gold coating applied by a PVD process seems to be the most suitable process. Another promising waterproof coating is the atomic layer deposition (ALD). It is a slightly changed chemical vapor deposition (CVD) and referring to the studies of Abdulagatov et al. an ALD of aluminum oxide (Al_2O_3) and titanium dioxide (TiO_2) can slow down the corrosion of static copper specimens in water for ~80 days [1]. Through a redrying procedure during test intermissions an increased underwater service life of the piezoelectric actuators is achieved.

2 INTRODUCTION

As part of the collaborative research center (CRC) 880 “Fundamentals of High Lift for Future Commercial Aircraft” the Technische Universität Braunschweig and the German Aerospace Center (DLR) develop an adaptive airfoil for novel high-lift devices.

One part of this system is a very compact oscillating piezo actuated lip (operating frequency up to 30 Hz) of a blowing slot ahead of a Coanda flap. To avoid flow separation on the Coanda flap, a closed loop controlled system adjust dynamically the Coanda jet to the current state of flight. The flow condition is measured using micro fabricated pressure and hot film sensors which are integrated into the airfoil. Using an additional pressurized mass flow $\dot{m}$, the velocity of the Conada jet is controlled by an adaptive lip that varies the height h of the blowing slot (Fig. 1). For flow investigations at high Reynolds number a water tunnel model of an adaptive lip is developed and manufactured. In this content an appropriate waterproof electrical insulation is evaluated by exposure and environmental tests [2]. This research investigates coatings that should be electrically insulating and water impermeable. Various coatings have been tested including tar-epoxy resin, gold sputtering, and atomic layer deposited Al_2O_3. The evaluations are performed on samples in terms of underwater service life and load cycles. The comparison of the results is presented to show the difference in coating performance. This preliminary study provides insights in coating research.

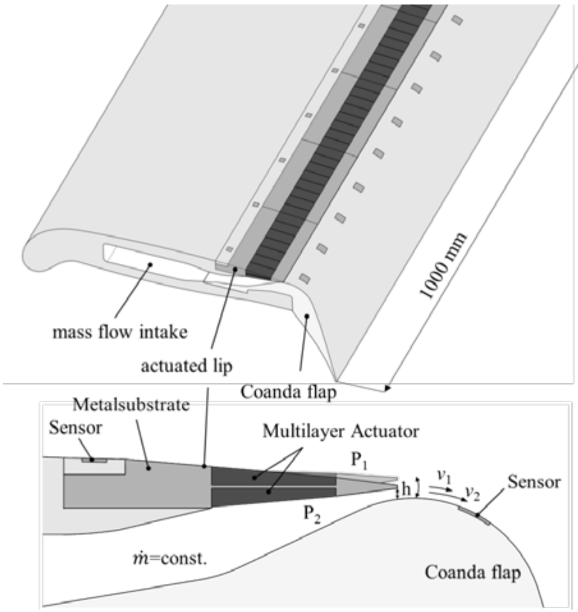


Fig. 1: Concept of the actuated slot-lip for water tunnel tests.

3 REQUIREMENTS OF THE WATERPROOF COATING

In the following the main requirements regarding the coating to fulfill the scientific and experimental goals are explained. Using water as testing environment brings several advantages. On the one hand a water tunnel allows to achieve a higher Reynolds number and a 10 times smaller flow velocity than a wind tunnel. On the other hand the actuators need to be completely sealed up against the ingress of moisture. Especially the electric contacts have to be waterproof because the actuators are operating at a voltage of 120 V. If any moisture diffuses through the coating, it may cause a short circuit, or in the worst case it could lead to a permanent damage of the actuators. Currently, it is planned to examine the high lift device for 20 days in a row in the water tunnel. During this time the actuators have to complete at least 10^7 loading cycles at a frequency of 100 Hz. Therefore the coating needs to be as ductile as posible, otherwise it can crack and water can diffuse into the piezoelectric ceramic. That is why the yield strength of the coating has to be higher than the actuator’s, which is 0.3 %. After that testing period the airfoil gets removed from the water tunnel to dry out any intruded moisture [3].

4 COATING MATERIALS AND COATING PROCESS

For preliminary tests fleece samples and actuators are coated with different materials in various processes to examine their waterproofness. The first coating, which is tested, is a two-component tar-epoxy resin (EP817 FlexShield by EPIFORM). It is used in the shipbuilding as a primer to protect hulls against corrosion of sea water. After mixing the two components (base and hardener) in the defined ratio, the primer can be applied with a brush on the specimens. When the varnishing is finished, the samples have to cure for at least five days. Now they can be examined to determine the layer thickness. Therefore one fleece sample gets sawed up. Afterwards the cutting area is polished and investigated under a microscope. It can be seen that the two-component tar-epoxy resin enclosed the sample completely, but because of the application is brushed by hand, the layer thickness is not constant. The average thickness is about 130 µm. Occasionally, there are places with a coating thickness up to 200 µm. Fig. 2 shows the magnified cutting surface of the fleece sample with the two-component tar-epoxy resin coat.

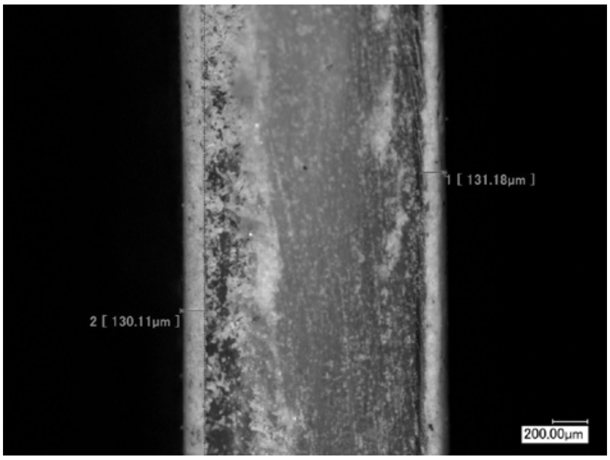


Fig. 2: Microsection of a 2 component tar-epoxy resin coated fleece sample to determine the layer thickness.

The second coating process, which should protect the actuators and the fleece samples against the ingress of water, is the sputter deposition of gold. The method is exactly known as physical vapor deposition (PVD). During the process, atoms are extracted from a solid gold body (target). This happens by a bombardment with a high energy noble gas ions. After the gold atoms are dissolved out of the target, they change into the gas phase and a thin film deposited on the specimens. Thus, very thin metallic coats can be realized. Fig. 3 shows a magnified cutting surface of a gold sputtered sample. By looking at the cross-section, a thin gold layer of about 1 µm can be detected on the left side of the fleece simple cominig from the right range.

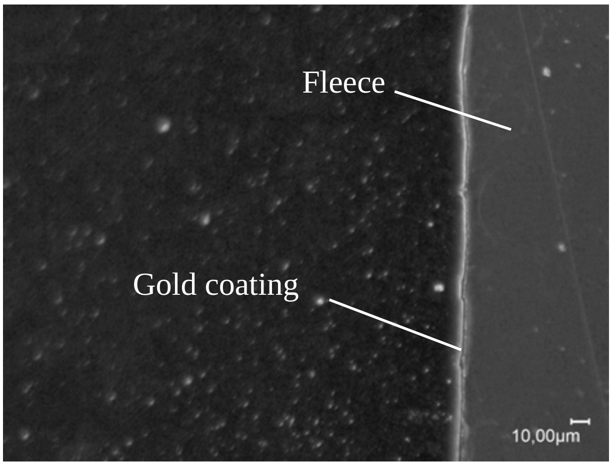


Fig. 3: Microsection of a gold sputtered fleece sample.

A disadvantage of metallic coatings is there electrical conductivity. That is why the contacts of the actuators have to be insulated by a separate layer before the samples are coated with gold. Therefore, a two-component glue (Loctite Hysol 9466 A&B) is used.

Another promising waterproof coating process is the atomic layer deposition (ALD). It is a slightly changed chemical vapor deposition (CVD). Referring to the studies of A. I. Abdulagatov et al. an ALD of aluminum oxide (Al_2O_3) and titanium dioxide (TiO_2) can slow down the corrosion of static copper specimens in water for ~80 days [1]. This aspect makes the ALD an interesting process regarding the actuators' coating for the tests in the water tunnel. In this study the actuators are coated with Al_2O_3. Therefore two reactants are needed: trimethylaluminum (C_3H_9Al or TMA) and water (H_2O). During the process these two substances react to Al_2O_3 in a process cycle. This cycle can be divided in four characteristic steps:

In the first step, the precursor (TMA) is being pumped in to the reaction chamber, in which the sample is placed. This step takes exactly 1 second. Afterwards, the chamber is being purged for 65 seconds with a non-reacting gas (in this case nitrogen N_2) to evacuate it. When all of the not reacted TMA is being flushed out, the third step can begin. During this step, which lasts 1.25 seconds, the second reactant (water) is initiated. The reaction between these two components starts and a single atom layer of Al_2O_3 is being deposited onto the specimen. In the final fourth step the chamber gets purged again to remove surplus material. During one cycle only one atom layer (1 Å) is deposited on the surface of the specimen. Fig. 4 illustrates the schematically process of an ALD cycle.

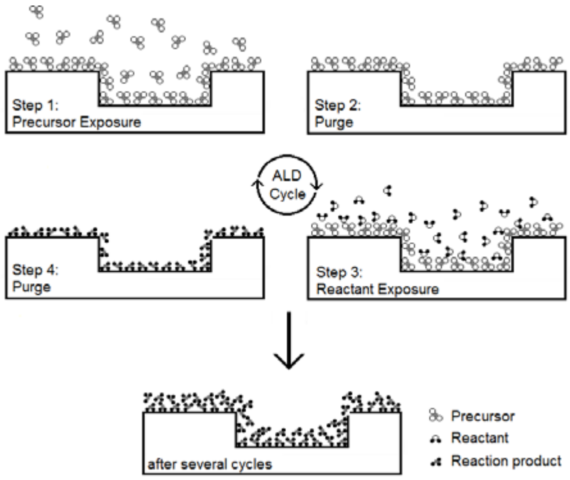


Fig. 4: Schematic presentation of an ALD cycle.

To achieve a functional coating, 2500 of these cycles are necessary. Subsequent measurements of the reflection index show that the coating has a thickness of 243 nm. An optical examination of the coated piezo actuator shows a light violet discoloration. Fig. 5 shows the Al_2O_3 coated actuator.

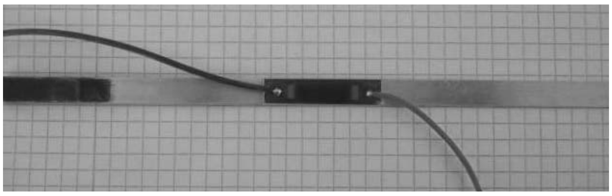


Fig. 5: Piezoelectric actuator with Al_2O_3 coating.

5 ENVIRONMENTAL TESTS

For the investigation of the water resistance of the different coatings two experiments are performed. In the first one, the coated fleece samples are tested for their water absorption. Therefore, the weight of every sample is determined with a precision scale. The scale has a measuring accuracy of 0.1 milligram. Afterwards the samples are exposed in a water basin. At regular intervals, the weight is measured again. So it can be noticed how much moisture ingress into the samples. To see how the water absorption develops under different temperatures, these tests are performed at 50 °C and at 23 °C.

The other experiment examines the functionality of the coated piezoelectric actuators (P-878 DuraAct Power by PI Ceramic) underwater. Therefor the actuators are placed in a water basin and operated with a triangular signal. The voltage amplitude is 0 – 120 V at a frequency of 30 Hz. With these settings, the actuators are tested until failure. Thus, the different coatings can be compared regarding their ability to protect the actuator from ingress of water.

During the subsequent operation in the high lift device, the piezoelectric actuators will have to deform the slot lip. Therefore they need to work against the slot lip's stiffness. To simulate this load during the underwater experiments, the actuators are applied on several aluminum beams. So they have to work against the beam's stiffness. Apart from that aspect, the implementation of the experiment stays the same. As in the previous test, the applied actuators are operated with a triangular signal of 30 Hz and 0 – 120 V until failure.

5.1 EVALUATION OF THE COATED FLEECE SAMPLES

After 28 days the measurement of the fleece samples' water absorption is stopped because the weight gain approximates to zero. So it can be assumed, that the samples have reached the saturation state. Fig. 6 and Fig. 7 illustrate the saturation curves of the samples by a water temperature of 50 °C and 23 °C. These curves show the percentage weight gain over time.

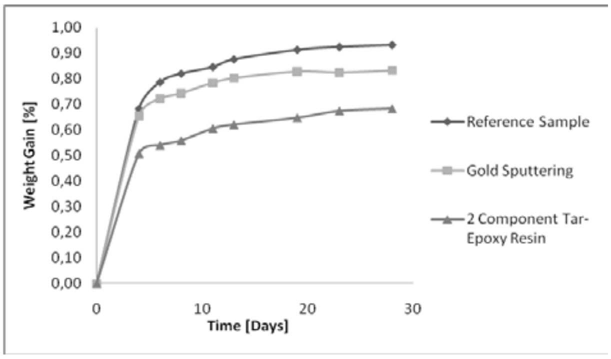


Fig. 6: Saturation curve of the coated fleece samples in a water basin (T = 50 °C).

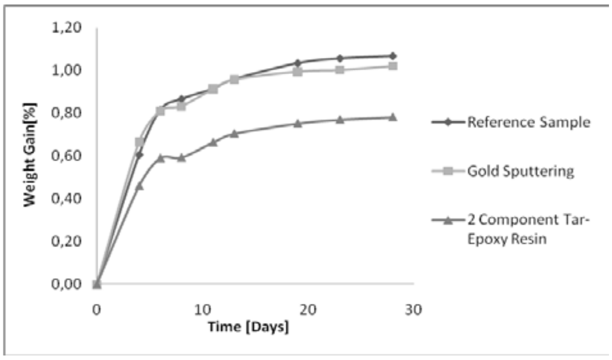


Fig. 7: Saturation curve of the coated fleece samples in a water basin (T = 23 °C).

In both diagrams can be seen, that the two-component tar-epoxy resin provides the best protection against water. This might be because the tar-epoxy has a much thicker coating than the sputtering. As expected, the reference sample absorbs the most water. In both examinations the weight gain of the gold sputtered sample is in between these two. Another noticeable aspect by looking at these figures is that the water absorption of the gold sputtered sample does not differ much from the reference sample. The reason for that could be that the sputtered layer of 1 µm is too thin, so water can diffuse through defects in the gold coating. For thicker metal layers an electroplating process is thinkable. It can also been seen that the samples gain more weight in the basin of 23 °C warm water. That might be an advantage, because the subsequent tests in the water tunnel will be performed at a temperature of about 40 °C to increase the Reynolds number.

5.2 EVALUATION OF THE COATED ACTUATORS

The results of the dynamic underwater actuator test are shown in Fig. 8. The hatched bars represent the lifespan of the electrical operated actuators, while the filled bars show the functionality (in days) of the preloaded actuators.

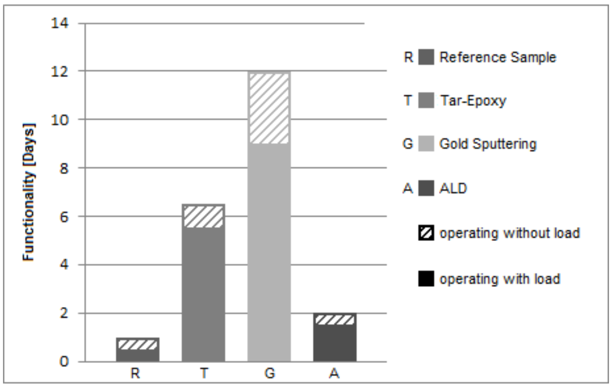


Fig. 8: Underwater service life of actuators with and without mechanical load.

As expected, the service life of all actuators, which operate under additional load, is lower than the life span of the actuators only driven electrically. In both cases, it can be seen, that the sputtered actuators remain the longest operation time underwater, followed by the actuators with the two-component tar-epoxy resin. The ALD with Al_2O_3 achieves just slightly better results than the reference samples. In consequence of the extremely thin and brittle Al_2O_3 layer the coating cracks under dynamic stress, so it cannot protect the actuator from the ingress of moisture any longer. Thus it appears that ceramic coatings are inappropriate for highly stressed surfaces of morphing structures because of their brittleness.

In Tab. 1, the completed load cycles of the loaded actuators are listed.

Reference	Tar-Epoxy	Sputtering	ALD
972000	13608000	22788000	4104000

Tab. 1: Underwater load cycles of loaded actuators.

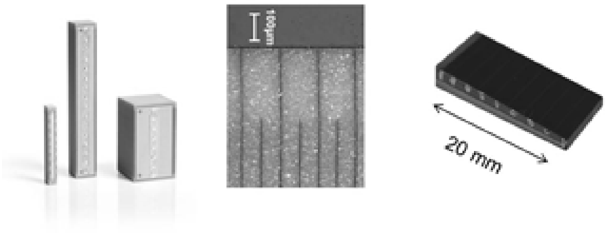
None of the tested coatings have fulfilled the requirement to remain underwater functionality for more than 3 weeks. In Tab. 1, it can be seen, that the reference and the ALD coated actuators even did not reach the required numbers of 10^7 load cycles. However, the actuator with the tar-epoxy and especially the gold sputtered actuator have surpassed this request.

6 LAYOUT OF THE DIFFUSION-RESISTANT COATING FOR THE SLOT-LIP

With the gained experience several slot-lip segments are constructed with different coatings for environmental tests. The design of the actuated slot-lip is based on a finite element analysis (FEA) which is specified in [2]. The adaptive slot-lip is built as a trimorphic bending actuator. The advantage of this configuration to a monomorphic configuration is the ability to actuate in both directions. The slot-lip itself is composed of several span-wise segments for better controllability and adjustability. This design also allows a separate control of each single segment for phase-shifted operating in span-wise direction. Each segment consist of a stainless steel substrate, building the backbone of the slot-lip structure, and two trapezoidal cut piezoelectric multilayer actuators (SCMAP-NCE51 by Noliac), applied on each side of the metal substrate.

The substrate for the slot-lip is manufactured from stainless steel (1.4301). The cutouts for the actuators in the lip are achieved by wire-cut EDM. The mounting of the actuators is realized by an established bonding method with a two-component epoxy under vacuum. The electrical contacts are made at the lateral electrodes of the actuators using PTFE insulated copper wires (Ø 0.5 mm). The wires lead to solder terminals which are applied to the passive structure for strain relief during operation.

The linear multilayer actuators are constructed of 30 ceramic layers (each 64 µm thick including electrodes) which are co-fired to a monolithic ceramic with a height of 2 mm. One stacked multilayer actuator of the adaptive lip has a total length of 20 mm and is made of 10 linear plate actuators glued together. The free displacement of a stacked actuator is about 30 µm by an operating voltage of 200 V. The piezo ceramics must be molded to the shape of the lip before the actuators are bonded to the structure. After the manufacturing the ceramics are machined with a wafer saw and sanded to bring them into the final shape. (Fig. 9).



Multilayer microsection with electrodes, right: customized actuator.

Due to the gained findings the actual slot-lip design is based on several diffusion barriers shown in Fig. 10. The slot-lip with the applied actuators is moulded in epoxy resin for electrical insulation and to achieve a smooth surface for flow investigations. This is the reference configuration, each additional coating is based on this basis. Finally all configurations are gold sputtered to have a completely closed outer coating. Different coating configurations with several diffusion barriers are built up and tested until failure in a water basin. The actuators are operating with a sinus signal in a voltage range from 0 – 150 V at a frequency of 10 Hz.

First samples of the actuators are applied with the active material lying outside. This setup leads to early short circuits between single electrodes due to the ingress of water into the piezoelectric ceramic. Due to this awareness, for further tests the actuators are applied with the active side to the substrate so the passive ceramic on the outside acts as an additional diffusion barrier.

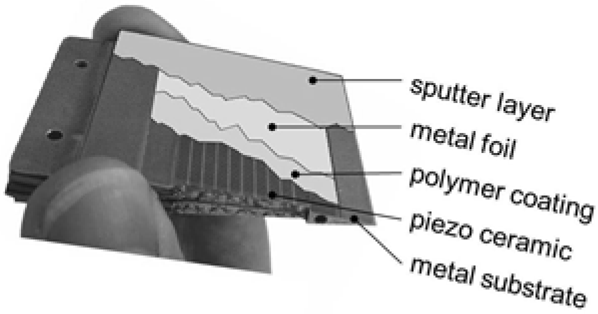


Fig. 10: Slot-lip design layout.

6.1 Evaluation of the coated slot-lip

The results of the underwater test of the actuated slot-lip are shown in Fig. 11. The hatched bars represent the lifespan of the slot-lip with inlying ceramics, while the filled bars show the functionality (in days) of the actuators with external active ceramics.

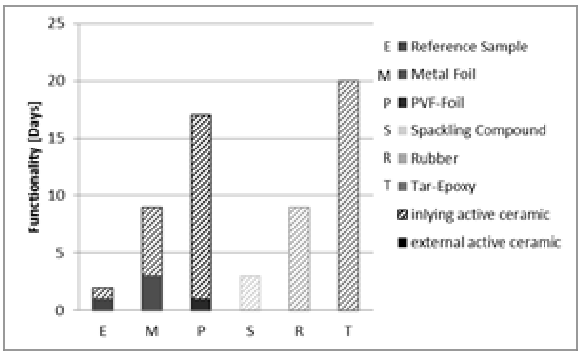


Fig. 10: Underwater service life of the slot-lip.

In the first configuration, a 5 µm thick stainless steel foil (1.4310) is applied with an epoxy film adhesive (Redux 312 by Hexcel) on the moulded actuators. To ensure that the adhesive layer has a nearly constant thickness of 100 µm an epoxy film adhesive is used. By this setup it is observed that any ingress of moisture reduces the dielectric strength which leads to breakdowns between electrodes of the actuator and the metal foil.

In the second configuration the metal foil is replaced by a 25 µm thick PVF-foil (Tedlar® by DuPont) for a better electrical insulation. Here the failure behavior is different. Over time the PVF-foil peels off and water can easily ingress at the lateral surface between single diffusion barriers up to the electrodes of the actuator which results in short circuits and inducts a complete failure.

Furthermore, one lip segment is filled with a spackling compound (EP 8012 by Lackwerke Peters) and another one is dip coated with liquid rubber (Plasti Dip Flüssiggummi by Plasti Dip). These two configurations show the same failure behavior. The spackling compound is too coarsely porous and the rubber is too hygroscopic so that both coatings don’t provide a sufficient diffusion barrier.

The last lip segment is vanished with the same two-component tar-epoxy resin (EP817 FlexShield by EPIFORM) used in previous tests. A low absorption of water and a fine pored structure is the feature of the tar-epoxy.

In Tab. 2, the completed load cycles of the actuated slot-lips are listed.

Ref.	Metal Foil	PVF-Foil	Spackling	Rubber	Tar-Epoxy
$1{,}7 \mathrm{x} 10^{0}$	$7{,}8 \mathrm{x} 10^{6}$	$1{,}5 \mathrm{x} 10^{7}$	$2{,}6 \mathrm{x} 10^{6}$	$7{,}8 \mathrm{x} 10^{6}$	$1{,}7 \mathrm{x} 10^{7}$

Tab. 2: Underwater load cycles of slot-lips with inlying active ceramic.

It can be seen, that the slot lip with the PVF-foil achieved the demanded load cycles but not the minimum lifespan. Most other coatings reach failure already before. Only the tar-epoxy coating meets all requirements. This configuration achieved a service-life of 20 days and over 10^7 load cycles.

7 CONCLUSIONS

One of the main tasks during this research project is the investigation of suitable coating methods and materials to insulate the electrical connections and shield the piezoelectric actuators against the ingress of moisture.

Only one of the tested slot-lip configurations meets the requirements of all performed environmental tests. It has been found out that a two-component tar-epoxy resin in combination with a gold sputtering is a suitable diffusion resistant coating in this particular case of application. This slot-lip configuration achieved the demanded minimum service-life of 20 days and surpassed the required number of load cycles of over 10^7. For extensive flow investigations the conditions of the upcoming water tunnel tests have to be adapted to the maximum service life of the actuators. It is proposed to have measurements for 5 days (Monday till Friday) in the water tunnel and then to blow-dry the actuators for 2 days over the weekend. Through this redrying procedure it is anticipated to achieve an actuator service life of several months.

ACKNOWLEDGMENTS

This work was founded by the German Research Foundation (DFG) in the framework of the collaborative research center (CRC) 880 “Fundamentals of High Lift for Future Commercial Aircraft”.

REFERENCES

[1] A. I. Abdulagatov, et al., Al_2O_3 and TiO_2 Atomic Layer Deposition on Copper for Water Corrosion Resistance. *ACS Applied Materials & Interfaces*, 2011, 3 (12), pp. 4593-4601.

[2] M. E. Zander, C. Behr, M. Sinapius, P. Wierach, *A Piezo-Actuated Slot Lip for avtive High Lift*. SFB 880 – Fundamentals of high-lift for future commercial aircraft,
CFF – Forschungsbericht 2013-03,
TU Braunschweig – Campus Forschungsflughafen, Germany, 2013, pp. 96-109.

[3] C. Behr, M. Schwerter, P. Wierach, M. Leester-Schädel, A. Dietzel, S. Büttgenbach, M. Sinapius, *A Piezo-Actuated Slot Lip for avtive High Lift*. SFB 880 – Fundamentals of high-lift for future commercial aircraft, Niedersächisches Forschungszentrum Luftfahrt – Forschungsbericht 2015-04,
TU Braunschweig, Germany, 2015, pp. 85-95.

[4] C. Behr, M. Schwerter, J. Petersen, P. Wierach, M. Leester-Schädel, A. Dietzel, S. Büttgenbach, M. Sinapius, *A Piezo-Actuated Slot Lip for avtive High Lift*. SFB 880 – Fundamentals of high-lift for future commercial aircraft, Niedersächisches Forschungszentrum Luftfahrt – Forschungsbericht 2017-14,
TU Braunschweig, Germany, 2017, pp. 79-91.