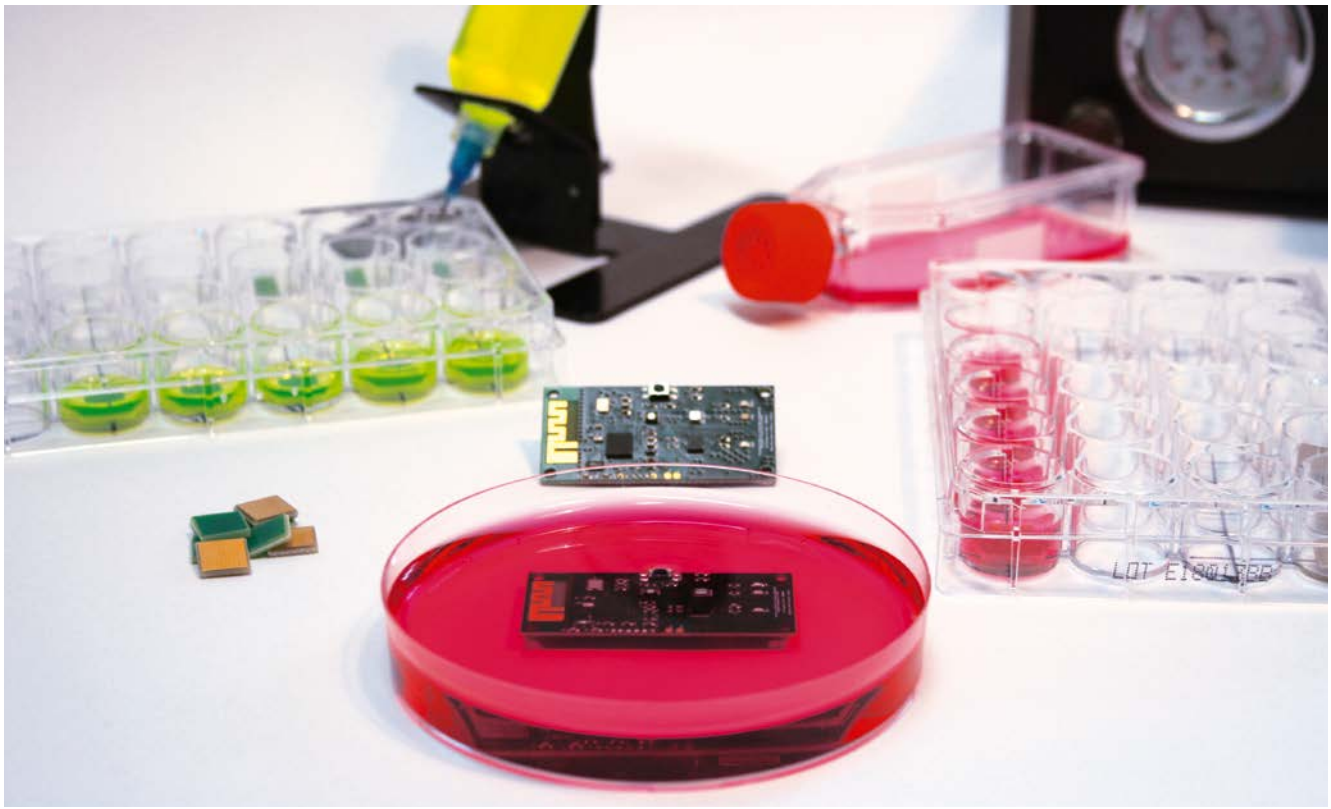
Protective Coat for Electronics and Body

Barrier Effect of Polymer Coatings for Encapsulating Smart Biomedical Devices

Cordless, mobile, networked devices promise comprehensive in vitro diagnostics. However, for applications in biological surroundings, the toxic electronic components must be carefully encapsulated. Therefore, cost-effective coating materials were examined regarding their interaction with biological systems.



The newly developed sensor platform with different sensors and cordless communication (center). In order to identify suitable coating materials, biological tests must be conducted with the material itself (right), and the barrier effect on electronic components (left) must be checked (© TUM)

In medical technology, an Internet of Medical Things is used, in which medical products are networked by means of new communication structures and systems. The integration of cost-effective electronic components in medical mass-products for the sensory detection of parameters will be one important feature of such products. This trend will become a necessity, as the retraceability of medical products (Unique Device Identification, UDI) will be compulsory in future [1–3]. Due to the wide range of possible modifications, manufacturing cost pressure, and particularly the biocompatibility required in medical technology, plastic is a key material for future Smart Medical Devices.

For the in vitro field, this technical advance prepared the way to Laboratory 4.0. Hereby, Laboratory 4.0 will be determined decisively by three trends: digitalization, automation, and miniaturization [4, 5]. Electronic in vitro devices belong to the group of Smart Biomedical Devices, which are cordless, mobile, networked, fitted with different sensors, and are used in biological surroundings [6]. Particularly for in vitro devices, which come into contact with sensitive biological systems, maximum biocompatibility is required. Contrary to the human body, in vitro systems have no possibilities for eliminating toxic substances from their surroundings. In spite of ever-smaller systems, adequate bioprotection is necessary by means of corresponding encapsulation of the toxic electronic components. The electronic systems must be protected from physical and chemical damage, and also the bio-

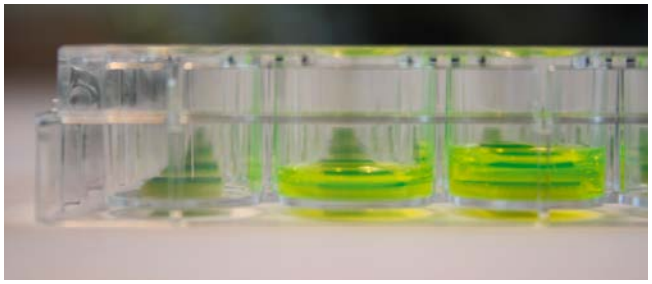


Fig. 1. Side view of a prepared multi-well plate for checking bioprotection: uncoated electronic samples with cytotoxically tested solder mask, minimum and maximum layer thickness of a polymer lacquer acc. to manufacturer specifications (left to right) (© TUM)

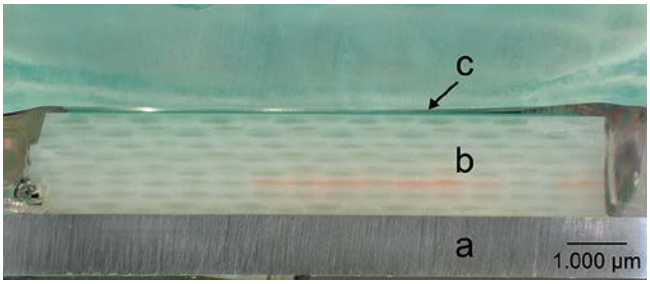


Fig. 2. Example of an intact barrier using Evonik's Silikopon ED. Microsection through the floor of a well made of polystyrene (a), FR-4 sample (height h = 1570 µm) with solder mask (h = 35 µm) (b), transparent lacquer layer of Silikopon ED (h = 40 µm at the thinnest part) (c) (© TUM)

logical surroundings must be protected from the emission of potentially toxic constituents from the electronics [6]. Coating materials – these mainly include lacquers, pottings, adhesives, and gels – that provide a very thin-walled and form-fitting layer have proved to be most suitable for protecting electronics under adverse environmental conditions [7–11].

Requirements for Selection

Three aspects are essential to enable coating materials to be used for Smart Biomedical Devices: interface surface bonding, material analysis, and biocompatibility testing. Hereby, the best possible interface surface bond between electronic components and coating materials is one of the most important challenges [12]. Apart from interface surface effects, chemical and physical aging occurs within a coating material (bulk) [13] due to internal and external aging causes [14]. Contrary to aging, corrosion not only involves the material, but also the environment in which the coating is used [15]. Corrosion with or in the surroundings can damage the coating. Therefore, impairments can occur not only in the material, but also in the surrounding medium [15]. Corrosion resistance is a central suitability criterion for a coating material used as protective barrier for electronic systems. If a coating material is biocompatible and corrosion resistance, it can be examined more closely regarding its barrier effect and – in biological surroundings – its bio-protective properties. Consequently, bioprotection describes the barrier effect results in biological applications.

In terms of processing, the challenge involves the application of thin-walled, void-free, and form-fitting coating layers. Miniaturization of electronic components requires less-bulky encapsulations with simultaneous necessary media sealability. In order to meet these demands, a wide selection of coating materials is found in the electronics processing industry.

The aim of the study was die scientific investigation of cost-effective coating materials with different base polymers regarding their suitability for protecting electronics in contact with biological fluids. Material selection was based on conventional thin-film and thick-film lac- »

Parylene coatings
Plasma Parylene Systems: Parylene C
Plasma Parylene Systems: Parylene F
Lacquers
Elantas: Bectron PL 4122-45T
Elantas: Bectron PT 4842
Elantas: Bectron SC 75V1-16
Electrolube: DCA SCC3
Electrolube: Tropicalised Varnish RS 199-1496
Evonik: Silikoftal ED (+ Dynasylan Ameo, + Albidur 1223, + Polycat DBU Sn)
Evonik: Silikophen AC 1000 + TEGO Kat 1
Evonik: Silikopon EF (+ Dynasylan Ameo, + Albidur 1223, + Polycat DBU Sn)
Evonik: Silikotop E 901 + Vestanat HAT 2500 LV / + Covestro Desmodur N3600
Lackwerke Peters: Elpeguard SL 1305 AQ-ECO
Lackwerke Peters: Elpeguard SL 1307 FLZ/2
Lackwerke Peters: Elpeguard SL 1308 FLZ
Lackwerke Peters: Twin-Cure DSL 1600 E-FLZ 75
Pro3dure medical: Generative Resin GR-20
Struers: Epofix E1232 Resin
Lackwerke Peters: Wepuran VT 3402 KK-NV-HE
Struers: Clarofast
Wacker: Silpuran 2430 A/B
Pottings
Bühnen: C 40460
Elantas: Bectron MR 3404
Elantas: Bectron PB 3251
Elantas: Bectron SK 75V1-35
Elantas: Bectron SK 75V2-65
Elantas: Bectron SK 76V2-50
Elantas: EP 5610
Elantas: EP 5611
Electrolube: Epoxy Resin ER2223
Electrolube: PE7501
Iso-Elektra: ISO-PUR K750
Iso-Elektra: ISO-PUR K762
Momentive: ECC 3050S
Momentive: ECC 3051S
Panadur: Clear
Lackwerke Peters: Elpecast Wepox VU 4085/51SB
Lackwerke Peters: Wepesil VT 3602 KK
Adhesives
Delo: Duopox AD840
Delo: Katiobond GE680
Delo: Monopox HT2860
Delo: Monopox HT760
Elantas: Bectron SA 70P1-34
Elantas: Bectron SA 70P9-60
Elantas: Bectron SA 70V1-36
Elantas: Bectron SA 75L7-70
Henkel: Loctite AA 3321
Henkel: Technomelt AS 5374
John P. Kummer: EPO TEK 302-3M
John P. Kummer: EPO TEK 353 ND
Panacol: Vitralit 1655
Polytec: EP 630
Ruderer Klebetechnik: technicoll 9302
Ruderer Klebetechnik: technicoll 9310
Gels
Elantas: Bectron SG 75V1-75
Elantas: Bectron SG 75L2-30
Elantas: Bectron SG 75V1-15

Table 1. Examined coatings (manufacturer and product respectively). Materials distinguished by resistance to steam sterilization, negative cytotoxicity, and good processability, are represented in blue. Subsequently, these were subjected to the barrier effect test. Materials that passed the barrier test with CCK-8 Assay, are represeted in green. Layer thicknesses rising downwards (© TUM)

quers, pottings, gels, and adhesives. Primarily, the material was to provide protection for electronics, and was not developed for medical use. Therefore, a pre-study was used to clarify the basic question of biocompatibility. This was accompanied by an investigation of the resistance during the sterilization procedure. Subsequently, a procedure for testing the barrier effect on standard electronic substrates was developed and implemented.

Material and Methods

In total, 56 coating materials from different manufacturers were investigated (**Info box**). In two independent studies (m = 2, random sample size per material n = 3, random sample size within one sample i = 3), 36 materials proved to be non-cytotoxic and resistance to steam sterilization in a cytotoxicity test acc. to DIN EN ISO 10993-5 and -12 as well as a steam sterilization resistance of up to 50 cycles. In addition to this, results for simple processing by means of time/pressure-controlled dispensers and an optimum coating conditions of multi-component materials were included. On this basis, the study investigated the barrier effect on electronic substrates of 25 coating materials.

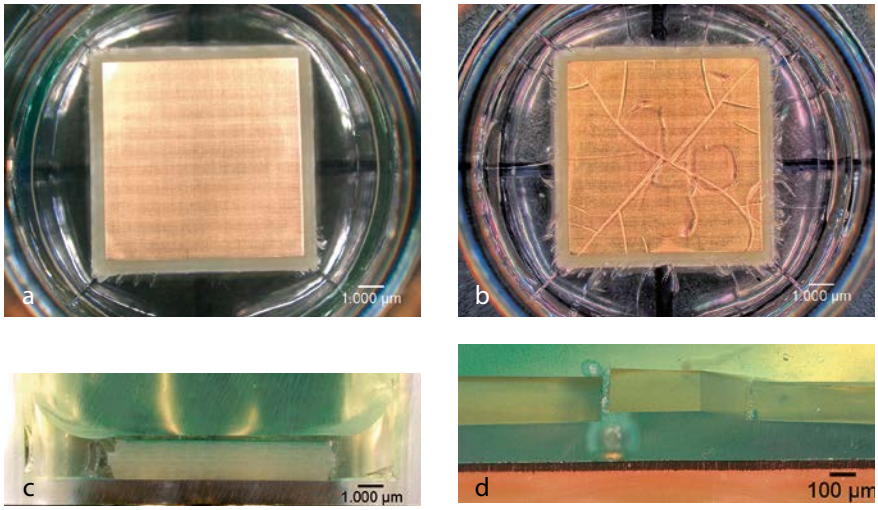


Fig. 3. Example of a failed barrier using Evonik's Silikophen AC 1000. a) Coated ENIG sample, dry layer thickness 40 µm without fluid medium contact. b) Material damage after a 7-day medium contact with cell culture medium DMEM. c) Microsection of the well with sample and lacquer coating. d) Microscopically documented material damage in the lacquer coating and loss of adhesion to the ENIG surface on the FR-4 substrate (© TUM)

The barrier test was conducted in a multi-well plate in contact with Dulbecco's Modified Eagle Medium (DMEM) with 5 % fetal bovine serum (FBS), 1 % antibiotic, and 1 % fungicide in a 7-day eluate test. Standard FR-4 substrate, basic material H140A DS (Zhejiang Huazheng New Material Co. Ltd. in Hangzhou, China) was used as electronic substrate, together with the cytotoxically-tested solder mask Probimer 77 9002/9021 (Huntsman Advanced Materials LLC in Salt Lake City, UT/USA) and the final surface coating of electroless nickel immersion gold (ENIG), produced by Beta Layout GmbH (Aarbergen, Germany). The biocompatibility test was carried out using CCK-8 Assay with fibroblasts of the cell line Hs 27 acc. to DIN EN ISO 10993 for cytotoxicity. The sample sizes were n = 5 for solder mask and ENIG coating material composite respectively, of which i = 3 eluate samples each were drawn and placed in cell contact for three days. Decisive factor hereby was the material layer thickness applied on the electronics (**Fig. 1**). The dry layer thickness was applied according to the respective manufacturer specifications, and was determined in a layer thickness study (wet to dry layer thickness).

Apart from the quantitative photometric evaluation on the biological side, the specimens were subjected to a differentiated examination of the electronics/coating interface surface as well as the effects within the material by means of a microsection analysis. Under the microscope, local changes such as coating adhesion loss from the electronics, damage in the form of cracks, embedded medium in the polymer matrix, as well as aging and corrosion effects were revealed.

Results and Different Effects

Outstanding results were exhibited by the coating materials highlighted green in **Table 1**. With them, no toxic constituents of the solder mask or the ENIG penetrated the coating material during the 7-day eluate contact. In correlation with the CCK-8 Assay, an intact coating (**Fig. 2**) or failed barrier (**Fig. 3**) could be validated visually. Irregular layer thicknesses occurred through meniscus formation along the sample edges. However, very thinly coated samples showed a tendency for thinning along the sample edges due to sub-optimal wettability of the substrate and internal forces of the coating material. However, complete encapsulation was guaranteed. Therefore focus was placed on the minimum applied layer thicknesses at the sample's center in the case of meniscus formation, and on the sample's edges in the case of higher cohesive and adhesive forces of the coating material. With Parylene coating, the minimum layer thickness was 16 µm, and at least 30 µm for the lacquers (average 60 to 80 µm), at least 40 µm with pottings (average 150 µm), and at least 60 µm with adhesives and gels

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(average 300 µm). The positively tested bioprotective barrier effects of the coating materials leads to a range of selection possibilities regarding the different base polymers, layer thicknesses, viscosities, number of components, curing mechanisms and times, as well as individual application-dependent processing aspects.

Summary

The study results show that the examined Parylene coatings, lacquers, pottings, adhesives, and gels can be used as a bioprotective, thin-layer barrier for protecting electronics. They are therefore suitable for Smart Biomedical Devices. At the Institute of Medical and Polymer Engineering of the Technical University of Munich (TUM), Germany, the acquired findings for protecting IoT electronics are used in the form of a cordlessly communicating "sensor fish" as an in vitro device for monitoring the conditions in a Petri dish in direct contact with cell culture media (**Title figure**). Apart from the bioprotective barrier effect of the encapsulation materials used, the challenge lies in the procedural implementation. Hereby, material combinations that ensure a media-tight and climate-proof module are mostly used. Apart from sealed packages and sensors (e.g. microcontrollers, memories, temperature, gyroscope, etc.), transparent sensors (e.g. UV) as well as open sensors in particular (e.g. humidity, pressure, biosensors) require locally differentiated integration strategies that will continue to be researched in ongoing and future studies. ■

Coating materials from the following manufacturers were investigated:

- Bühnen GmbH & Co. KG, Bremen, Germany
- Delo Industrie Klebstoffe GmbH & Co. KGaA, Windach, Germany
- Elantas GmbH, Wesel, Germany
- Electrolube, H K Wentworth Ltd., Leicestershire, UK
- Evonik Industries AG, Essen, Germany
- Henkel AG & Co. KGaA, Düsseldorf, Germany
- Iso-Elektra Elektrochemische Fabrik GmbH, Elze, Germany
- John P. Kummer GmbH, Augsburg, Germany
- Lackwerke Peters GmbH & Co. KG, Kempen, Germany
- Momentive Performance Materials Inc., Waterford, NY/USA
- Panacol-Elosol GmbH, Steinbach, Germany
- Panadur GmbH, Halberstadt, Germany
- Plasma Parylene Systems GmbH, Rosenheim, Germany
- Polytec PT GmbH Polymere Technologien, Karlsbad, Germany
- pro3dure medical GmbH, Iserlohn, Germany
- Ruderer Klebetechnik GmbH, department technicoll, Zorneding, Germany
- Struers GmbH, Willich, Germany
- Wacker Chemie AG, Munich, Germany