Methods to Determine the Stability of Polymer Encapsulations

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Abstract

Biomedical Microsystems that are designated to be implanted in the human body as neural prostheses have to fulfill strict requirements regarding their biostability. Applications like retina implants that have to be flexible and transparent, at least if parts are integrated into an artificial intraocular lens. In these cases, metal or ceramic housings to protect electronic circuitry cannot be used. Therefore, polymer layers were proposed as long-term stable encapsulation. However, no microimplant without hermetic packaging (glass, metal, ceramics) has been transferred into clinical practice yet. Potential candidates for flexible polymer-based substrates, insulation, and encapsulation layers for microimplants are polyimide, parylene C and silicone rubbers. The development of test structures for electrical measurements to investigate the insulation resistance of the encapsulation itself and the impedance of the encapsulation-substrate compound will be evaluated under accelerated testing paradigms to estimate the mean time to failure. Optical measurements (Fourier transformed infrared spectroscopy-FTIR) accompany electrical measurements under in vitro conditions to correlate structural material changes to variations in insulation resistance.

1. INTRODUCTION

Biomedical Microsystems that are designated to be implanted in the human body as neural prostheses have to fulfil strict requirements regarding their material and system properties. They have to be small, non-toxic, and biostable. Flexibility and low weight help to improve structural biocompatibility. From technological aspects, the fabrication and assembly should be compatible to CMOS electronic components. The implants must be fabricated according to international standards, e.g. the active medical device directive (AMDD) to get approved by FDA in the USA or to get a CE mark in Europe. State of the art implants use hermetic packages to protect electronic circuitries over the lifetime of an implant, i.e. more than 20 years. Therefore, the housings were made out of metal, ceramics, and glass. Only cables and electrodes were encapsulated in polymer-based materials – mainly silicone rubber – as insulation and contact material to the body. However, new applications of biomedical microimplants like retinal vision prostheses recommend flexible housing and encapsulation concepts. Sometimes, these encapsulations have to be transparent, e.g. if telemetry circuitry has been integrated into an artificial intraocular lens. The challenge of polymer encapsulations mainly lies in their biostability. Degradation in polymers includes hydrolytic, oxidative and enzymatic mechanisms that deteriorate the chemical structure. Finally, the mechanical strength and electrical insulation resistance decrease and a device failure occurs. Some polymers already proved their applicability in electrical active implants. Silicone rubber has been established as material for cable insulation, epoxies were partly used for encapsulation of electronic components and parylene C is deposited as insulation coating on electronics in implants. The only implant which is solely encapsulated in silicone rubber is the Brindley-Finetech stimulator for urinary bladder management. However, no microimplant with a complete polymer encapsulation has been transferred into clinical practice yet.

This paper describes fundamental methods that might be used for non-destructive characterization of polymer encapsulation layers during in vitro and probably also in vivo studies.

2. METHODS

2.1. Electrical measurement methods

Polymer-based encapsulations have been mainly used for wires in the past. Insulated wires were soaked the in physiologic solutions under a voltage bias (Figure 1) and leakage cur-

rents were measured [1]. Insulation layers on integrated circuits (ICs) were monitored in a similar way [2].

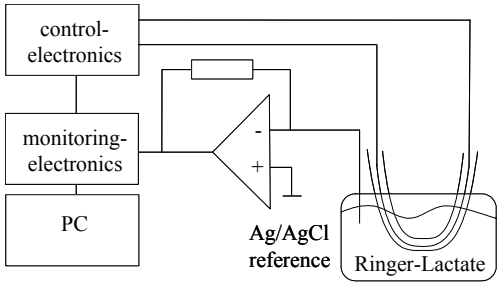

Figure 1: Setup for the in vitro characterization of the insulation resistance of wires.

The main parameters to be monitored are the volume resistance of insulation and encapsulation materials, their surface resistance, the dielectric loss angle of the materials during soaking tests as indicator for water absorption, and the permittivity (table 1). The chosen parameters during the tests should be well below the dielectric strength of the materials to prevent electric damage that is not related with degradation processes.

Table 1: Electrical properties of insulation materials.

parameter	***unit***	***value***
volume resistance (DC)	Ωcm	$10^{11}…10^{18}$
surface resistance (DC)	Ω	$10^{11}…10^{18}$
loss factor (tan δ)	1	0,1…100 ‰
permittivity	ε_r	2…10
dielectric strength	kV/cm	200…700

We suggest an electrical model (figure 2) for polymer-based microimplants or encapsulations to design and develop test structures that might be integrated into ICs of substrates to distinguish between different failure mechanisms.

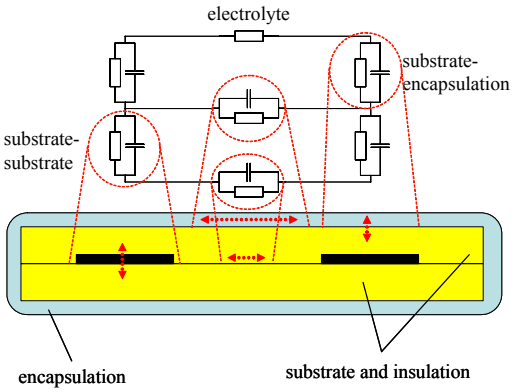

Figure 2: Electrical model of the substrate-encapsulation-layer compound.

2.2. Optical measurements

Fourier transformed infrared spectroscopy (FTIR) is the method of choice to investigate the chemical structure of polymer-based encapsulation materials and semiconductor insulation layers. Complete microimplants can be investigated in a non-destructive manner using an attenuated total reflection (ATR) unit. Measurements take place as well in the fingerprint region (1500-400 cm^{-1}) as in the functional group region (4000-1500 cm^{-1}).

Table 2: Characteristic regions in FTIR spectra for polymer encapsulation materials.

Peaks observed in the functional group region (4000-1500 cm^{-1})		
Material	peak / wave no (cm^{-1})	explanation
Parylene C	3018	C-H stretch due to aromaticity or unsaturation
	2926	C-H stretch vibration
	2860	C-H stretch vibration
	1608	complex deformation of aromatic ring
	1555	C-H overtones of aromatic ring
Polyimide	1774	imide ring
	1770	symmetric C=O stretch vibration
	1720	symmetric C=O stretch vibration
	1690-1560	area under curve ~ amount of solvent (NMP)
	1514	benzoid ring (reference)
Peaks observed in the fingerprint region (1500-400 cm^{-1})		
Parylene C	1494	C=C-C aromatic ring stretching
	1451	C-H bending vibrations
	1209	planar C-H vibrations from aromatic ring
	1050	halide substitution of ring
	825	para substitution of halides on benzoid ring
Polyimide	1350	C-N stretch vibration (pyromellitic imide system)

Polyimide as potential candidate for substrates and parylene C as encapsulation material have distinct absorption peaks in FTIR that correlate with degradation mechanism (table 2). Sheets with 10 to 20 micron thickness have been investigated to prove theoretical values and look

for changes when process parameters were changed during polyimide processing.

3. RESULTS

3.1. Electrical measurement methods

Considerations on the electrical model of (combination) layers for encapsulation and literature, e.g. [1, 2], lead to multiple test structures: insulated strip lines for volume (1 line) and surface (2 lines) resistance, four-electrode arrangements for complex insulation layer impedance, large dots to investigate larger area volume resistance, and inter-digital electrodes to measure the capacitance of thin and thick encapsulation layers (figure 3) will be realized to distinguish between different failure mechanisms.

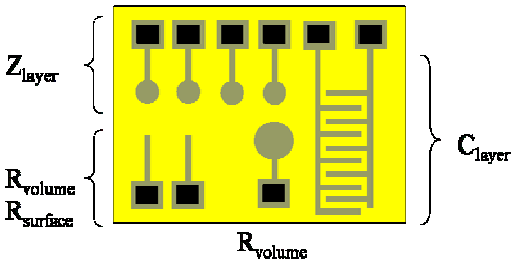

Figure 3: Test structures to investigate insulation and encapsulation layer properties.

3.2. Optical measurements

FTIR-ATR spectra from parylene C (figure 4) and polyimide showed the characteristic absorption peaks (table 2).

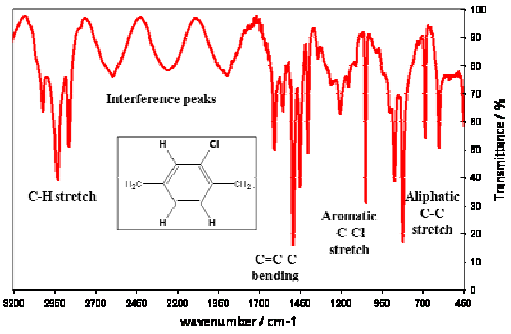

Figure 4: FTIR spectrum of a parylene C layer.

Polyimide spectra clearly differed in the regions that were related to the solvent (NMP) and the imidization peaks (table 2) depending on the process duration and temperature. The relation of the areas under the curve at the imide ring (1774 cm^{-1}) and the benzoid ring (1514 cm^{-1}) between a probe under test and the polyimide from a reference process clearly indicated the degree of imidization [3] that we used in the process development towards low temperature curing regimes.

4. DISCUSSION AND CONCLUSIONS

Test methods are only a first step to transfer microimplants into clinical practice. While silicone rubber encapsulated microimplants only work for a limited time with a wide variety of mean time to failure [4], stimulators based on polyimide substrates with a parylene encapsulation and additional silicone rubber coating lasted for more than 12 months [5] in the eye.

In principle, measurement methods are available to investigate the behaviour of encapsulation and insulation layers. Electrical methods only indicate material changes while optical methods help to identify the structural material changes. However, there is a lack of standardized test structures and measurement methods to arrive at comparable conclusions in different research groups or products. Material properties are process depending and fundamental electrical and mechanical parameters change from the macro to the micro scale. So long, only few publications have been published in this field and a lot of work has to be done until all-polymer microimplants will reach high numbers in clinical applications.

References

[1] Edell, DJ, Insulating Biomaterials. In: Horch KW, Dhillon GS (eds.). Neuroprosthetics. River Edge, London: World Scientific Publishing, 517-579, 2004.

[2] Kazemi M, Basham E, Sivaprakasam M, et al. A Test Microchip for Evaluation of Hermetic Packaging Technology for Biomedical Prosthetic Implants, Proc. 26th Ann. Int. Conf. IEEE EMBS, 4093-4095, 2004.

[3] Yang CC, Hsieh KH, Chen WC, A new interpretation of the kinetic model for the imidization reaction of PMDA-ODA and BPDA-PDA poly(amic acid)s. In: Mittal KL. VSP: Polyimides and Other High Temperature Polymers, 2: 37-45, 2003.

[4] Laube T, Brockmann C, Buss R, et al. Optical Energy Transfer for Intraocular Microsystems Studied in Rabbits, Graefe's Arch Clin Exp Ophthalmol, 242: 661-667, 2004.

[5] Stieglitz T, Haberer W, Kammer S, Schanze T. A Simple Model to Investigate the Stability of Flexible Micromachined Retina Stimulators, Proc of the 8th Ann. Int. Conf. of the IFESS, 69-73, 2003.