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Wafer-level fabrication process for fully encapsulated micro-supercapacitors with high specific energy

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Patrice Simon · Pierre-Louis Taberna ·
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Abstract In this paper a wafer-level process is proposed to fully integrate carbon-based micro-supercapacitor onto silicon substrate. This process relies on the deposition of a paste containing carbon, PVDF and acetone into cavities etched in silicon. After electrolyte deposition in a controlled atmosphere, a wafer-level encapsulation is realized. Cyclic voltammetry performed on non-encapsulated micro-components showed specific energy of 257 mJ cm^{-2} for $336 \text{ }\mu\text{m}$ deep cavities. The specific encapsulation process developed was tested separately and proved to be efficient in terms of resistance to organic electrolytes and mechanical strength.

1 Introduction

The development of low-power integrated circuits (such as sensors, microprocessors or wireless communication chips) over the last decade has made embedded systems increasingly popular. These systems are meant to operate in controlled/uncontrolled environments, gathering, processing, storing and communicating information without connection to the power grid. Dedicated technologies to supply power for these systems were developed such as thin film Li-ion batteries in the late 1990s (Jones and Akridge 1996).

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Despite recent major enhancements in performance through the use of enhanced materials (Hassoun et al. 2009; Sun et al. 2009), micro-batteries do not fulfill the requirements imposed by autonomous systems, i.e. long life time, capability to charge and discharge at high rate and resistance to severe environment (low temperatures in particular). Supercapacitors, on the other hand, show very interesting characteristics when it comes to these applications. Two types of supercapacitors are known: electrochemical double layer capacitors (EDLCs) and pseudo-capacitors. In their principle, EDLCs store energy using accumulation of ions at the interface between a highly porous electrode and an electrolyte with no chemical modification of the electrode involved during the charge/discharge process. As a result, they can sustain millions of cycles, can provide fast charge/discharge rates and subsequently high specific power, while keeping a reasonable specific energy (Vullers et al. 2009). Pseudo-capacitors use fast and reversible surface redox reactions for charge storage and are based usually on metal oxides (RuO_2 , MnO_2) or conductive polymers. Their cycle life and potential window are however limited. It is believed then that EDLC miniaturized to a microscopic scale could satisfy in this sense a variety of micro-power demands and complement or replace micro-batteries in electrical energy storage and harvesting applications where high power delivery or uptake pulses are required in very short times. In harvesting applications indeed, a micro-device extracts energy from various ambient environment sources: thermal gradients (Bailly et al. 2008), mechanical vibrations (Beeby et al. 2006; Durou et al. 2008), solar radiations (Corke et al. 2007) and delivers it to a sensor. The storage stage requires consequently fast charging and discharging characteristics.

Various technologies were considered for miniaturization and integration of supercapacitors on planar substrates going from “printed electronics” (Kaempgen et al. 2009;

Pushparaj et al. 2007; Hu et al. 2010) to microfabrication technologies involving either sputtering or electrodeposition of metal oxides (Yoon et al. 2001; Kim et al. 2003; Liu et al. 2010), synthesis of conductive polymers (Sung et al. 2003, 2006; Sun and Chen 2009) or printing of activated carbon (In et al. 2006; Jiang et al. 2009; Pech et al. 2010a, b). As it will be detailed hereafter, the main difficulty lies in the integration of the active material and the electrolyte (liquid, gel or solid) within a microfabrication process, while reaching highest possible specific energy. Up to now, the best specific energies reported concerned pseudocapacitors. Kim et al. (2003) produced a stack of RuO₂ electrodes with LiPON electrolyte showing a specific energy of 400 mJ cm⁻², and Sun et al. (2010) reached 28 mJ cm⁻² with interdigitated polypyrrole electrodes. Very recently, Shen et al. (2011) have demonstrated a micro-EDLC containing activated carbon with a specific capacitance of 90.7 mF cm⁻² corresponding to a specific energy of 45 mJ cm⁻² with a 1 M NaNO₃ aqueous electrolyte.

All the energy performance found in the literature were extracted from non-encapsulated micro-devices, they were indeed either tested at open air which limited the voltage to 1 V (In et al. 2006; Shen et al. 2011) and thus the energy, or tested in controlled atmosphere via a glove-box environment (Pech et al. 2010a, b). To enable the technology, a fully operating micro-device implementable on-chip and able to operate over a potential window larger than 1 V must be proposed. Carbon-based EDLCs generally operate with organic liquid electrolyte (potential window up to 2.5 V). The challenge for micro-EDLCs realization is thus to provide a wafer-level sealing with the following characteristics:

1. Hermeticity to water
2. Low temperature process, typically in the range of 100–150°C depending on the solvent used to prevent evaporation
3. Aggressive electrolyte withstanding, acetonitrile in particular
4. Electrical feedthroughs to connect the micro-device to electrical circuits

As reviewed by Esashi (2008), wafer level packaging techniques can be performed using interfacial bonding or deposition sealing. Since micro-EDLCs involve a liquid electrolyte that has to be deposited under controlled Ar atmosphere, interfacial bonding techniques are preferred. And among interfacial bonding techniques, anodic bonding involving high temperature [lowest reported temperature is 250°C in (Chen et al. 2007)] should be avoided. Among intermediate melting materials, polymers are potentially suitable (Jourdain et al. 2005; Kim et al. 2008; Zine-El-Abidine and Okoniewski 2009). Adhesive bonding relies on viscous polymers that are deposited onto the

surface of one of the wafers and that fit the space in between the lid wafer and the substrate wafer. The polymers are then hardened using UV (photo-curable) or temperature (thermo-curable) (Niklaus et al. 2006). This technique allows the use of lateral electrical feedthroughs. Hermeticity is then the main issue since gas permeates polymers. Most of the recent work in the literature presents hermeticity tests, using He leakage (according to the MIL-STD-883 specification) or calcium degradation (Marquardt et al. 2010).

In this paper, we present a complete process to fabricate micro-EDLCs using deposition of carbon-based material and wafer-level encapsulation of liquid organic electrolyte. Deposition provides several hundred microns thick electrodes, giving rise to the highest specific energy reported for micro-EDLCs. This technology is coupled to a simple wafer-level encapsulation technology based on a photo-sensitive and thermocurable resist. The overall process is performed at low temperature (150°C), on surfaces presenting a topology (for lateral electrical feedthroughs) and that proved to be water-tight, ideal thus for the realization of fully packaged micro-EDLCs.

2 Fabrication

Figure 1 shows the steps of the micro-EDLCs fabrication process. The objective is to achieve the highest specific energy by creating electrodes as thick as possible. Deep cavities were thus etched in a silicon substrate with a potassium hydroxide (KOH) solution at 90°C and with Si₃N₄ as a hard mask (Fig. 1a). Etching depth depends on the desired electrode resolution (electrode width and spacing). KOH etching implies indeed a compromise between maximum depth and electrodes width, as described by the expression: $depth_{max} = width/(\sqrt{2})$.

In the reported experiments, each electrode is constituted of four fingers, 580 μm wide and separated by 50 μm. The cavities were etched to a depth of 336 μm. After this step and the removal of the mask, a 80 nm thick Si₃N₄ layer was deposited using low pressure chemical vapour deposition (LPCVD) to provide electrical insulation between the component and the silicon substrate (Fig. 1b). Given the anisotropic nature of the KOH etching of silicon, the cavities present inclined walls. Current collectors were then realized using evaporated Ti/Au (100/800 nm) and patterned in the shape of interdigitated fingers with KI/I₂ wet etching (Fig. 1c).

The next step of the fabrication process is the deposition of the active material (activated carbon with organic binders). Large cavities with large spacing are easy to fill without leaving active material in between electrodes (that would produce a short circuit), but large spacing directly

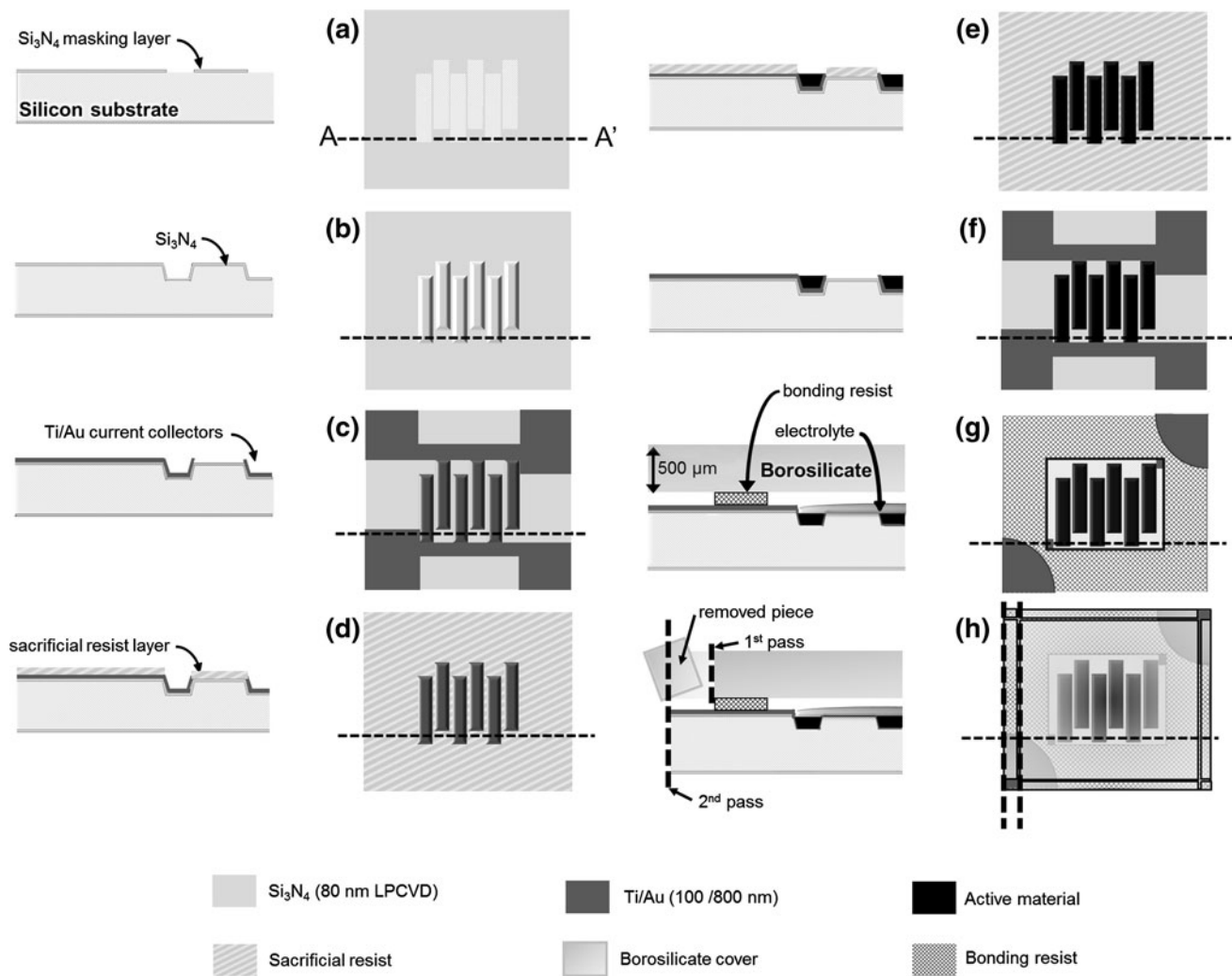


Fig. 1 Fabrication process of the micro-EDLC. **a** Preparation of hard mask (Si_3N_4) for wet etching, **b** KOH etching of cavities in silicon and deposition of insulation layer (Si_3N_4), **c** deposition and patterning of current collectors (Ti/Au), **d** deposition of sacrificial layer,

e deposition of active material, **f** dissolution of sacrificial layer, **g** alignment and bonding to the silicon substrate of borosilicate wafer on which UV curable resist is deposited, **h** two-pass dicing to reveal electrical contact

decreases specific power. There is a strong correlation indeed between electrode configuration (width and spacing) and equivalent series resistance (ESR) of the resulting micro-supercapacitor (Pech et al. 2010a, b). Larger spacing implies higher ESR (because of electrolyte resistance), which translates then into lower maximum power achievable by the device.

To keep the spacing between the fingers as small as possible, a process with a sacrificial layer was developed. Before filling the cavities with the active material, a photoresist sacrificial layer was coated and patterned (Fig. 1d) to cover the regions where no active material is required. A paste containing the active material (Kuraray Chemical YP-50 activated carbon mixed with 20% of PVDF-HFP into acetone) was spread over the wafer (to fill the cavities) using a soft polybutadiene blade (Fig. 1e). The wafer was

then cured at 150°C for 2 h to evaporate the acetone and to give the electrodes a sufficient mechanical resistance. Finally, the sacrificial layer was removed by immersing the wafer in a basic solution (AZ-400K developer) and the wafer was dried at 200°C for 24 h to evaporate all traces of water (Fig. 1f).

The following steps concern the electrolyte encapsulation. A negative photosensitive resist, Fujifilm[®] SC resist 180 was used to provide water-tight sealing that will not be damaged by the organic electrolyte such as acetonitrile or propylene carbonate. This resist relies on a polymer (cyclized polyisoprene), and a photosensitive component (bisazide) in non-polar organic solvents, such as xylene. After having spin-coated the resist on the borosilicate wafer (or lid), a low temperature cure (85°C) evaporates the solvent. UV exposition makes aromatic azides to

decompose into highly reactive nitrenes, which then react into the polyisoprene polymer matrix to produce cross-linking. Exposed areas are no longer solvable into the developer. A process providing a 13 μm thick resist film was developed to make the resist film able to fit the space between the lid and the silicon wafer, even in the presence of 900 nm thick current collectors. The relatively high thickness proved to be also useful for step h) of the process in Fig. 1 as it provides more margin for the two-pass dicing.

To prevent water from contaminating the electrolyte, the electrolyte deposition and the bonding were done in a controlled Ar atmosphere glove-box (less than 1 ppm of H_2O and O_2). For convenience, the electrolyte was deposited manually with a syringe on the active material, but this step could be automated. Then, the silicon wafer and borosilicate lid were assembled, pressed in a custom-made portable wafer-bonder (made of machined stainless steel) that provides 2 bar of pressure and heated at 150°C for 120 min (Fig. 1g).

A two-pass dicing technique with Disco DAD 321 automatic dicer was used to access the electrical contact zones. The borosilicate lid wafer (500 μm thick) was cut first by a 200 μm wide diamond saw. Another pass (full depth) was then done 400 μm away from the first pass to cut the stacked silicon wafer, resist layer and glass wafer. To ensure sufficient margin during the first cut, the cut depth was set at 20 μm less than the glass lid thickness. Once those two steps were performed, the small borosilicate fragments covering the electrical contact zones are easily removed as there is no resist to bond them with the silicon wafer (Fig. 1h). Ti/Au lateral feedthroughs are then freely available for connections.

3 Results and discussion

3.1 Electrochemical characterization of non-encapsulated micro-EDLCs

Figure 2a shows the realized prototypes at the end of step e and Fig. 2b, the same prototype at the end of step f. With this technique, a 64% filling of the 336 μm deep cavities was achieved.

For the electrochemical characterization of the micro-EDLCs performance (without encapsulation), the processed wafers were diced and the resulting dies were bonded to TO8 packages using gold wire wedge bonding technique. To ensure water is removed from activated carbon pores, they withstood a 24 h long 200°C vacuum cure. 1 M Et_4NBF_4 into propylene carbonate was used as electrolyte and deposited under controlled water-free Ar atmosphere. Electrochemical characterization was carried

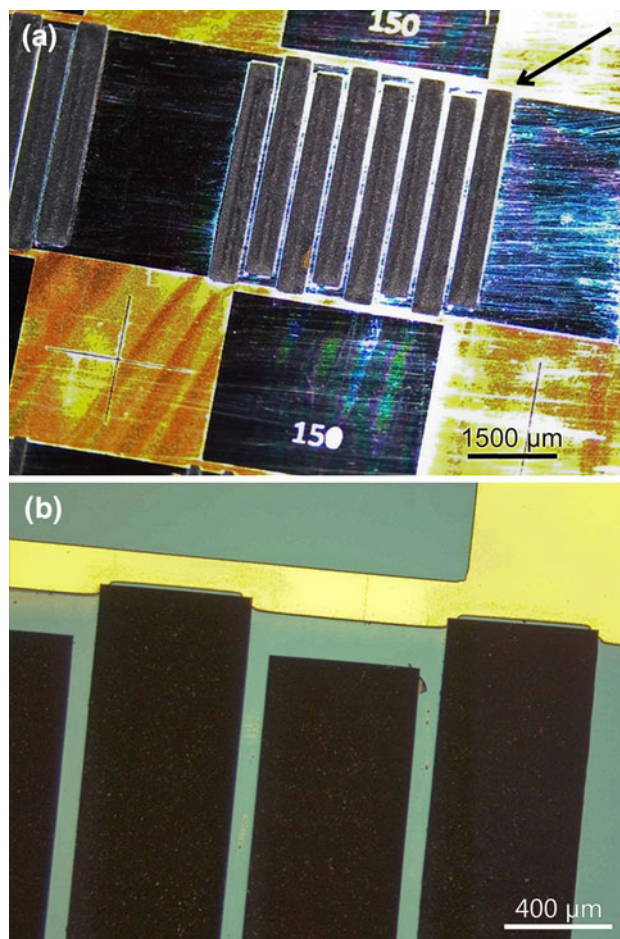


Fig. 2 a Optical microscope image of a micro-EDLC after active material deposition and before removal of the sacrificial layer, b optical microscope close-up of the marked zone on a after removal of the sacrificial layer

out by performing cyclic voltammetry with a Bio-logic VMP-3 potentiostat. Specific cell capacitance was calculated using the expression $C = Q/(\Delta E \cdot S)$, where Q is the charge (expressed in C), ΔE the potential range (in V) and S the total surface of the component (in cm^2) which is here 0.227 cm^2 .

On the measurements shown in Fig. 3, the fabricated micro-EDLC exhibits a 81.0 mF cm^{-2} specific capacitance at 5 mV s^{-1} scan rate. By estimating the volume of active material deposited in the cavities to be 2.7 mm^3 , we find a corresponding volumetric capacitance of 6.8 F cm^{-3} , i.e. a 27.2 F cm^{-3} electrode capacitance. This value is coherent with usually reported values given the binder part used in this work: 15 wt% PVDF. All these voltammograms were performed on a 2.5 V potential range, so the maximum recorded specific energy is 257 mJ cm^{-2} , which is the highest value reported for carbon-based micro-supercapacitors.

The variation of the specific capacitance with the scan rate is detailed in Fig. 4, it can be observed that the specific

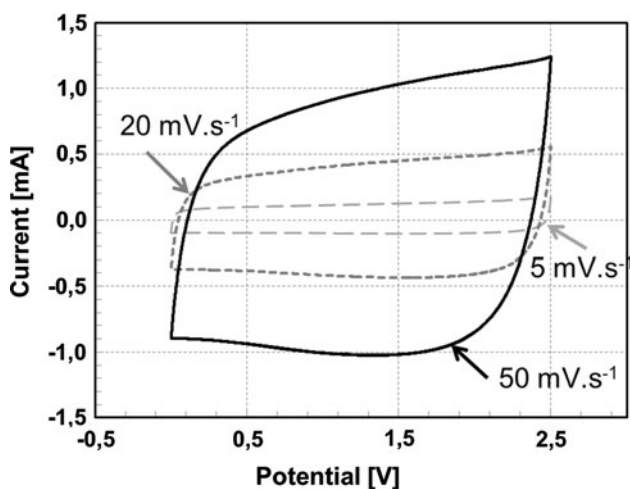


Fig. 3 Voltammograms recordings of a non-encapsulated micro-EDLC tested with 1 M Et_4NBF_4 + propylene carbonate in glove-box environment

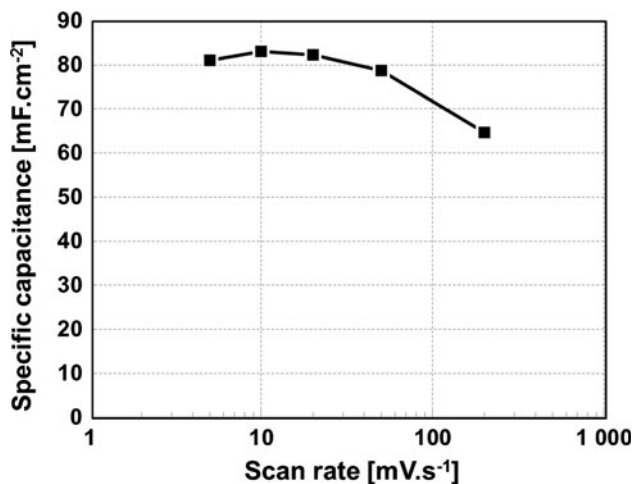


Fig. 4 Specific capacitance versus scan rate for the micro-EDLC tested in Fig. 3

capacitance is stable ($81.0 \pm 2 \text{ mF cm}^{-2}$) from 5 to 50 mV s^{-1} , then it decreases as the scan rate increases, due to the high equivalent series resistance of the device. It was estimated from the CV curves to be about 200Ω ($45.4 \Omega \text{ cm}^2$) leading to a specific power of 34.4 mW cm^{-2} . Contributions to series resistance include resistance from the electrolyte, resistance from current collectors and resistance from electrode material (activated carbon + binder). In the particular case of fabricated micro-supercapacitors, the resistive contribution comes mainly from the current collectors that were thinned down on the edge of the cavities (see Fig. 2b) and from the electrode material containing a large amount of polymer binder. To improve the conductivity of the electrode material, a conductive agent such as acetylene black could be added.

3.2 Chemical resistance and mechanical tests for encapsulation process

We achieved complete fabrication of sealed electrolyte (1 M Et_4NBF_4 into propylene carbonate) into buried cavities, as shown in Fig. 5. Figure 5b shows a close-up view of the encapsulated electrolyte. The micro-component total surface (with connection pads) is 0.65 cm^2 , and the total thickness is $1130 \mu\text{m}$.

Several tests were conducted to validate the encapsulation process. To test the resistance of Fujifilm[®] SC resist 180 to the electrolyte, the sealed and diced component was immersed into propylene carbonate (PC) for 19 days. Microscope images of a region of the component after 3 and 19 days are shown in Fig. 6a and b. There is no evidence of visual damage of the resist, and no evolution of the voids can be seen. Resistance to acetonitrile (ACN) was also tested by immersing sealed and diced components into the electrolyte and monitoring it, bonded components show good overall resistance, yet slight void evolution as shown in Fig. 6c, d.

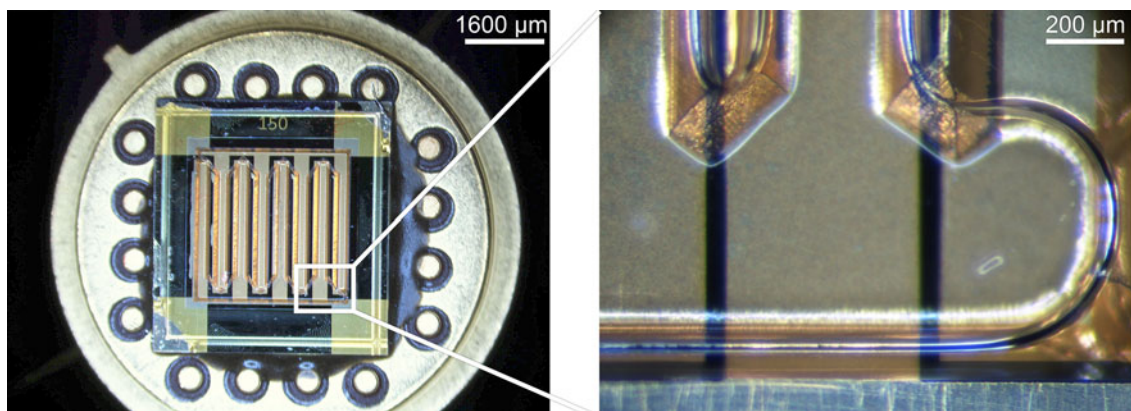


Fig. 5 a Optical microscope image of encapsulated micro-EDLC containing propylene carbonate. The component is $8.4 \times 7.8 \text{ mm}$. $400 \mu\text{m}^2$ wide electrical contact pads are available on the lower left and higher right corner. b Close-up view showing the encapsulated electrolyte

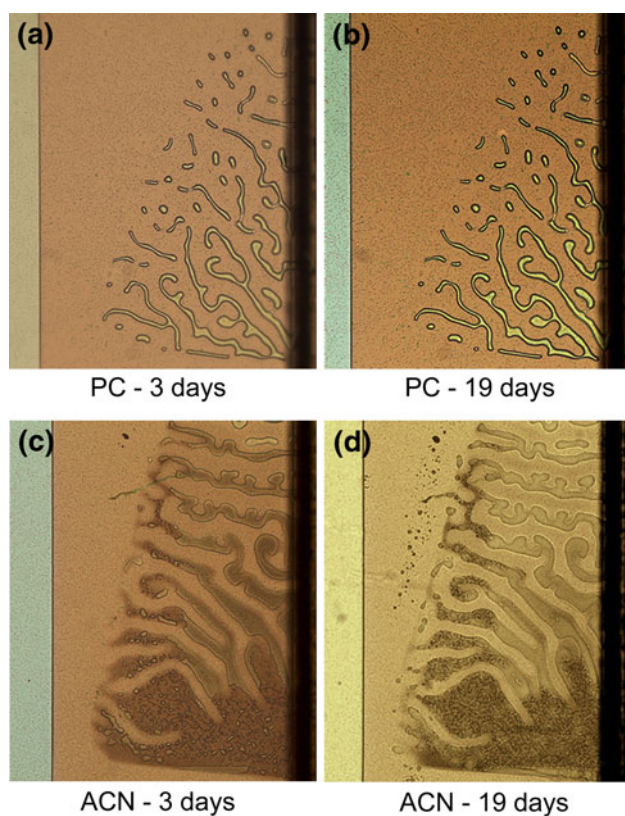


Fig. 6 Optical microscope images of regions exposed to propylene carbonate (PC) and acetonitrile (ACN) of the polyisoprene resist on a sealed micro-component

The shear resistance was tested with and without encapsulated propylene carbonate to see if it modifies the mechanical properties of the resist. The test apparatus (DAGE Series 4000T) was made of a vise (holding the component Si wafer) and a tip that pushes the borosilicate lid until the bonding breaks. Encapsulated components have 500 μm sealing rings of SC-180 resist, which represents an approximate surface of 22.95 mm^2 . Measured shear force was 27.9 kgf (without electrolyte) and 27.4 kgf (with electrolyte), which corresponds to 11.9 and 11.7 MPa shear strength. This exceeds the MIL-STD-883 norm, which specifies that for areas larger than 4 mm^2 , the shear force must be larger than 2.5 kgf and it is in the range of what was found in the literature for polymer sealing (Jourdain et al. 2005; Marquardt et al. 2010).

4 Conclusion

We proposed a complete process to integrate carbon-based micro-electrochemical double layer capacitor on silicon substrate, depositing the active material into KOH-etched silicon cavities. Electrochemical characterisation of the realized micro-devices showed that a specific energy of

257 mJ cm^{-2} on a 2.5 V potential range could be achieved, which is 5.7 times more than the best previously reported performance for carbon-based micro-EDLC by Shen et al. (2011). In order to provide a fully operational component, a dedicated low temperature wafer-level encapsulation technology was developed. The technology proved to be efficient in terms of resistance to typical organic electrolytes used in EDLCs (acetonitrile and propylene carbonate) and in terms of mechanical strength. Future work will focus on the improvement of particular steps of this process. For improving power performance of the micro-device, it will be necessary to increase the current collectors thickness and uniformity in 3D structures so as to reduce series resistance. New composition of electrode material (with conductive agent, different binder) will also be investigated.

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