Stretching the limits of stretchable electronics:
Towards high-density, highly stretchable electronics

Salman Shafqat
Stretching the limits of stretchable electronics: Towards high-density, highly stretchable electronics

Salman Shafqat

A catalogue record is available from the Eindhoven University of Technology Library.
ISBN: 978-90-386-4888-0

Cover design: Anastasija Mass
Printed by: ProefschriftMaken || www.proefschriftmaken.nl

This research was carried out under the project project number 12966 in the framework of the research program of the Netherlands Organization for Scientific Research (NWO).

© Copyright, 2019, Salman Shafqat. All rights reserved.
Stretching the limits of stretchable electronics: 
Towards high-density, highly stretchable electronics

PROEFSCHRIFT

ter verkrijging van de graad van doctor aan de Technische Universiteit Eindhoven, op gezag van de rector magnificus prof.dr.ir. F.P.T. Baaijens, voor een commissie aangewezen door het College voor Promoties, in het openbaar te verdedigen op donderdag 7 november 2019 om 16:00 uur

door

Salman Shafqat

geboren te Bhaun, Pakistan
Dit proefschrift is goedgekeurd door de promotoren en de samenstelling van de promotiecommissie is als volgt:

voorzitter: prof.dr. L.P.H. de Goey
1e promotor: prof.dr.ir. M.G.D. Geers
2e promotor: dr.ir. J.P.M. Hoefnagels
leden: prof.dr.ir. J. Vanfleteren (Ghent University)
       prof.dr. T. Pardoen (Université Catholique de Louvain)
       prof.dr.ir. J.M.J. den Toonder
       prof.dr.ir. P.R. Onck (Rijksuniversiteit Groningen)
       prof.dr.ir. R. Dekker (Technische Universiteit Delft, Philips Research)

Het onderzoek of ontwerp dat in dit proefschrift wordt beschreven is uitgevoerd in overeenstemming met de TU/e Gedragscode Wetenschapsbeoefening.
Summary

The advent of stretchable electronics (SE) has resulted in an expanding group of novel and exciting applications, e.g., smart health monitoring patches, 3D formed electronics, and balloon catheters with integrated multifunctional instrumentation for minimally invasive surgery, among others. Such SE devices are typically based on a hybrid approach with stiff application-specific integrated circuit (ASIC) islands distributed on an elastomer substrate and electrically connected by stretchable interconnects. For most SE applications, especially stretchable detectors, a boost in the currently low ASIC island density is desired to maximize device performance. This entails miniaturizing the interconnect footprint by an order of magnitude compared to typical current solutions while reaching ultra-stretchability beyond 1000%, whereas most of the existing interconnect geometries stay below 100%. This PhD thesis therefore aims at bridging this gap by introducing a new type of miniaturized ultra-stretchable interconnect with a reliable performance.

A new freestanding ultra-stretchable interconnect design was conceived (Chapter 2). The freestanding nature of the interconnect, as opposed to substrate embedded interconnects, allows to exploit new kinematic degrees of freedom such as torsion and out-of-plane bending to achieve ultra-stretchability. The mechanics-based design was built on insights from Finite Element (FE) simulations. The thin-film interconnect structure was designed to be conveniently processed with standard microfabrication recipes to allow miniaturization to the micrometer scale while providing straightforward integration with ASIC processing. Samples were produced by project collaborators based on their novel Flex-2-Rigid (F2R) microfabrication approach. Subsequently, many in-situ, scanning electron and optical microscopy experiments were performed to characterize the interconnects.

FE simulations of a flat to hemispherical configuration inflatable ASIC array on a catheter tip were performed, which illustrated that the interconnects experience significant in-plane and out-of-plane shearing, whereas the literature primarily focused on uniaxial stretching. Detailed FE simulations were performed on the proposed interconnect design along with two other existing interconnect geometries, stretching them along all three axes till the onset of plasticity to construct limiting envelopes (Chapter 3). The results demonstrate that the response of the developed interconnect is ‘omnidirectional’, revealing typical advantages and disadvantages of the choice of interconnect geometries with respect to multiaxial loading. Finally, multiaxial experiments were performed on the novel SE interconnect to demonstrate that they can indeed stretch omnidirectionally along all three Cartesian axes.

A number of novel experimental methodologies were developed for characterizing the material properties and the electro-mechanical performance of the interconnect. A key aspect of the micromechanical characterization of such (sub-)micron sizes structures, is (size-dependent) plasticity. To quantify and study these effects, the approach adopted
here was to apply Digital Height Correlation (DHC), previously developed in the Mechanics of Materials group, with an Integrated-DIC (IDIC) algorithm, developed in the present project by a fellow PhD student. Digital image correlation requires application of a random speckle pattern on the micron-sized freestanding sample, for which available techniques in literature fall short. Therefore a new technique, involving dry near-room temperature deposition of microspheres, was developed and applied to the samples (Chapter 4).

For the quasi-static and cyclic electro-mechanical testing of the miniature freestanding interconnects, a methodology consisting in a miniature multi-axial micro-tensile stage with integrated electrical probing was developed (Chapter 5). The SE samples were separately microfabricated on a test-chip which ensured the integrity of the structures during processing and handling.

In order to further optimize of the choice of materials for the freestanding interconnects, the (size-dependent) performance of different thin-film materials needed to be evaluated. To this end, the bulge test method was adopted. Here, the bulge test method was extended to enable the characterization of thin-films with (processing-induced) compressive residual stresses (Chapter 6). This extension enables using the bulge test method to characterize both classes of thin-film samples, i.e., with tensile as well as compressive residual stresses.

Using these methodologies the samples were tested and a high elastic stretchability beyond 2000% and an ultimate stretch (involving plasticity) beyond 3000% was reached, well beyond the goal of 1000% elastic stretch. Furthermore, the electrical resistance of these interconnects proved to be stable under deformation with an increase of <0.3% till interconnect failure. During cyclic loading the interconnects sustained >10 million cycles at 1000% stretch and <1% resistance change.
# Contents

Summary iii

1 Introduction 1
   1.1 Background ......................................................... 1
   1.2 Objective ............................................................. 5
   1.3 Strategy of WP1 ..................................................... 6
   1.4 Thesis Layout ......................................................... 8

I Design and performance of a highly stretchable interconnect for high density stretchable electronics 9

2 Ultra-stretchable interconnects for high density stretchable electronics 11
   2.1 Introduction ........................................................... 11
   2.2 Interconnect Design and Mechanics ................................. 13
   2.3 Results ................................................................. 17
   2.4 Conclusions and discussion ......................................... 20

3 Omnidirectional stretchability of freestanding interconnects for stretchable electronics 25
   3.1 Introduction ........................................................... 26
   3.2 Multiaxial elastic stretchability of typical freestanding interconnect geometries ......................... 28
   3.3 Multiaxial experimental testing of the rope interconnect 34
   3.4 Discussion and conclusions ......................................... 34

II Experimental characterization techniques 45

4 Cool, dry, straightforward, sub-micron nebulization, DIC patterning of delicate, heterogeneous, non-planar specimens 47
   4.1 Introduction ........................................................... 47
   4.2 Setup Design ........................................................... 50
   4.3 Methodology ........................................................... 52
   4.4 Experiments: Pattern test cases .................................... 57
   4.5 Concluding remarks .................................................. 66

5 Multi-axial electro-mechanical testing methodology for highly stretchable freestanding micron-sized structures 71
   5.1 Introduction ........................................................... 71
   5.2 Setup design and experimental methodology ..................... 72
Introduction

1.1 Background

1.1.1 The need for making electronics conformal

The advent of microelectronics brought forth a revolution which is responsible for the exponential advancements in almost all areas of technology in the last few decades. However, a physical limitation of conventional microelectronics is that it is planar, rigid and brittle. This intrinsic limitation originates from its constituent materials such as silicon. This limitation hinders the full utilization of microelectronics when it has to be closely integrated on the application surface, most of which are non-planar and can even be dynamic in nature. A prime example of is the human skin. Close integration of microelectronic sensors and stimulatory actuators can greatly assist medical diagnosis as well as treatment. An illustrative example of this challenge is shown in Figure 1.1 showing a paediatric patient being fitted with numerous sensors for the diagnosis of sleep apnea, a debilitating respiratory obstruction disorder that occurs during sleep. The polysomnography test, used to diagnose the condition, involves monitoring of a number of body functions including brain waves with EEG electrodes, blood oxygen content, pulse rate, limb movement, snoring sounds, air flow etc., while the patient sleeps [1]. The myriad of non-standalone and stiff sensors which need to be attached to the patient’s body makes the test uncomfortable to undergo, while a reliable diagnosis requires the patient to be able to sleep normally.

1.1.2 The emergence of stretchable electronics

To overcome such limitations of conventional microelectronics, the field of ‘soft-electronics’ has emerged. It initiated with the development of flexible electronics, which are devices that can undergo bending and thus conform to cylindrical surfaces [2–8]. With further advancements, devices have been developed that can undergo more complex deformations, especially stretching which are termed as stretchable electronics (SE). Due to the enhanced compliance of SE devices, they can take up more complex shapes with non-zero Gaussian curvatures, e.g., a spherical surface [2–8]. The development of soft-electronics has enabled numerous novel applications. One such application, which is currently also one of the most popular commercial applications of SE, is the smart patch which can be applied on the skin with stand-alone wireless functionality for sensing a number of body parameters [2] (see Figure 1.2a).

SE allows (micro-) electronics, which are primarily processed in a planar configuration as for integrated circuit (IC) microfabrication or printed circuit board (PCB) fabrication,
to be morphed into required non-planar shapes. This can enhance the functionality of certain devices, e.g., the dynamically tunable electronic eye detector (see Figure 1.2b) which mimics the physiology of the human eye and thus requires minimal additional components such as movable lenses to focus or zoom, and thus promises a more compact design over conventional detectors [9]. Soft-electronics can also be used to simplify (micro-)electronics fabrication procedures of geometrically complex electronics devices and thus reduce production costs. An example of this is shown in Figure 1.2c, which shows a contact lens embedded with glucose sensing circuitry [10]. Such applications, termed as 2.5/3D electronics, are processed in a planar configuration and subsequently moulded into their final shape. They provide the flexibility of integrating the electronics with associated moulded (polymer) components already in the moulding stage thus circumventing the requirement for complex (and typically manually performed) pick and place assembly and wiring of the electronics [11].

1.1.3 Overview of current stretchable electronics approaches

A number of approaches to enable SE have been developed, each with its own sets of advantages and disadvantages. The diverse approaches can broadly be classified into three general categories.

1.1.3.1 Intrinsically stretchable and conductive materials

The first involves utilizing materials that are intrinsically soft and stretchable while also being conductive. These include liquid metals and conductive polymers such as PEDOT:PPS [3, 4, 8, 13]. These materials offer moderate sheet resistance and high stretchability, however, achieving micron-sized features remains to be challenging, as
Chapter 1.

Figure 1.2: Applications of stretchable electronics: (a) an epidermal electronics ‘smart patch’ that can be applied on the skin like a tattoo and can monitor a number of body parameters with stand-alone wireless functionality, (from [12] reprinted with permission from AAAS); (b) a dynamically tunable detector array which mimics the human eye and offers an imaging system which can zoom and focus without the requirement of bulky lenses [9]; (c) a smart lens for continuously monitoring blood glucose levels (© [2012] IEEE) [10].

they are typically processed in fluidic configuration (for liquid metals) or nozzle printing based processing [3, 4]. Such materials are typically considered good candidate for large-area, low density SE applications.

1.1.3.2 Intrinsically stretchable insulating materials made electrically conductive

The second category involves imparting conductivity to intrinsically stretchable material but electrically insulating materials, e.g., by addition of metal particles or carbon nanotubes, etc. Such materials offer moderate sheet resistance and moderate stretchability [3, 4, 14, 15]. The conductive content in the composite structure is directly proportional to the electrical sheet resistance, while inversely proportional to the stretchability. Hence, a compromise between the two desired functional properties need to be made. Similar to the first category, micron-sized feature sizes can be difficult to achieve, however the roll-to-roll processability makes them a good candidate for large-area electronics as well.

1.1.3.3 Conventional electrically conductive stiff materials

The last category involves utilizing, conventional electrically conductive materials, typically metals, in SE devices. Metals offer low sheet resistance and micron-sized features can be easily achieved with current processing methods such as PCB technology or microfabrication [2–8, 16, 17]. Furthermore, within the elastic deformation regime, metals offer stable electrical performance. However, intrinsically metals fail (plastically deform) at relatively low strains, therefore, they are utilized in the form of structured geometries, which can globally stretch to high values while the local strains remain low. A prime example of that is the planar equivalent of the helical spring, in the form of the serpentine
geometry [2–8, 18] The stretchability in this category is dependent on the geometry of the structure, e.g., the conventional serpentine structure can stretch by approximately 30-100% global strain. The focus of this thesis is on metal interconnects, as they fulfill the requirement of miniaturization and processability discussed in the following section.

1.1.4 Need for highly stretchable, high-density, CMOS compatible stretchable electronics

1.1.4.1 Hybrid nature of SE devices

Utilizing these stretchable conductors, SE devices are typically built using a hybrid approach, in which conventionally microfabricated Si based rigid functional microelectronics islands lay on a stretchable elastomeric substrate and are electrically interconnected by stretchable conductors described earlier (see Figure 1.2) While efforts are being made to make the functional electronics islands stretchable as well [3], even for limited electronics functionality especially at small scales, microfabricated rigid chips remain the most viable option. Due to the rigid nature of the functional islands, only the interconnects can contribute to the device stretchability. In conjunction with the requirement of the global device stretchability, the required interconnect stretchability is governed by the fill-factor (areal density) of the functional islands. The fill-factor is defined as the ratio of area taken up by the functional islands over the device surface area. This is illustrated with the help of an application example of a stretchable detector array that can be inflated for a variable field-of-view functionality (see Figure 1.3).

1.1.4.2 High density SE devices require very high stretchability

The array inflated from a flat to a hemispherical configuration entails a detector global strain of 57%. For an initial fill factor of 0.5, the interconnects are required to stretch by ~200% (as discussed in chapter 2). However, for any detector array it is important to maximize the fill factor. For instance, for typical CCD detectors, a fill factor of ~0.9 is common [19]. For a fill factor of 0.9, the required interconnect stretchability jumps to 1100% (as discussed in chapter 2). Furthermore, in the case of a stretchable detector, an initially high fill factor is even more important since the fill factor significantly reduces in the stretched configuration. For instance, 0.5 and 0.9 fill factor reduce to 0.25 and 0.45, respectively for a circular array stretching to a hemisphere. Therefore, for such applications, very high interconnect stretchability is of key importance. However, in literature, stretchability of even 300% is considered to be very high [8]. Consequently, the fill factors of current SE devices are severely limited and in order to fully utilize the potential of SE applications, especially SE detector applications as shown in Figure 1.3, the interconnect stretchability needs to be significantly boosted.

1.1.4.3 Potential of CMOS compatible SE devices

For the commercial adoption of SE, manufacturability is an important criterion. Ideally, already proven and industrially standardized manufacturing processes should be utilized after any necessary modifications. Additionally, if such a process is fully integrated, i.e., if the functional island fabrication, elastomer substrate application and stretchable interconnect fabrication can be performed in the same process flow, it can
greatly improve the manufacturability. In many SE demonstrators shown in literature complicated multi-step processing approaches are used, typically involving transfer printing of the interconnects on to a (pre-stretched) elastomer substrate with the functional islands being assembled by pick-and-place, which makes it challenging to develop it into an industrially viable manufacturing process. Another important factor for many SE applications, such as the inflatable detector is that a manufacturing approach (such as CMOS-processing) that allows miniaturization of the functional islands enables improved device functionality and room for future developments. At the start of the thesis work, to the best of our knowledge, this particular area of fully integrated microfabricated SE devices had not been explored in literature, with the major focus being on large-area SE devices.

1.2 Objective

To enable future high-density, highly stretchable miniaturized stretchable electronics devices, such as the application illustrated in Figure 1.3, the NWO Vidi project titled “Stretching the limits of IC Stretchability”, granted to principle investigator J. Hoefnagels, set the objective to tackle the following key challenges:

- Developing highly stretchable and highly reliable interconnects with significantly increased elastic stretchability of at least 1000% and ideally a factor of two higher to account for a safety margin.
- Utilizing (and adapting) an IC microfabrication-based (industrially standardized) approach for the fabrication of highly stretchable interconnects and SE device such that it ensures:
  - Mass productions of SE devices, and
  - Miniaturization of the interconnects to enable micrometer sized interconnect footprints as well as miniaturization of the active electronics which enables increased performance.
To this end, the global project was subdivided into the following Work Packages (WP) with corresponding tasks:

(WP1) **PhD 1** *(this thesis work)*, involving designing highly stretchable interconnects and performing micromechanical characterization and testing.

(WP2) External collaboration with the group of Prof. R. Dekker, with Ph.D. students A. Savov and S. Joshi, at Philips Research for the microfabrication of test chips with highly interconnects.

(WP3) **PhD 2** *(S. Kleinendorst)*, focused on developing (integrated DIC based) inverse techniques to enable characterization of the as-processed highly stretchable samples.

(WP4) **Postdoc** *(L. Bergers)*, responsible for integrating the highly stretchable interconnects in the design and process flow of a demonstrator application.

### 1.3 Strategy of WP1

To enable the highly stretchable, high-density, CMOS compatible SE, the approach for the design, fabrication and reliability testing of the highly stretchable interconnects is summarized below.

#### 1.3.1 Highly stretchable interconnects by means of a freestanding design

Typically, metal-based interconnects such as the serpentine structures are embedded or bonded on the elastomer substrate surface to provide protection against external contact-based damage. However, such a configuration intrinsically suffers from the challenge of incompatible deformation between the soft and highly stretchable substrate and the locally stiff metal interconnect. This results in high stresses developing at the interconnect-elastomer interface leading to interface debonding at moderate levels of stretchability (typically $<100\%$) [20, 21]. Furthermore, typically interconnects are of a planar nature, since it is challenging to deposit and etch large thicknesses. Such planar structures are not ideal for in-plane opening to which they are constrained and develop high stresses at relatively low global strains leading to failure, as discussed in Ref ([3]).

Freeing the interconnect from the substrate allows the interconnect to have more degrees of freedom with the potential to reach high stretchabilities. While a publication reporting a freestanding interconnect was published just before the start of the project, a maximum stretchabilities up to 300% could be attained [22]. Therefore, in order to reach the very high levels of stretchabilities desired here a new freestanding interconnect design was needed. To this end, a mechanics-based approach was followed to understand the deformation modes that can lead to high level of stretchability and come up with a design in which these modes could be induced.
1.3.2 Integration of the freestanding SE interconnect design with Polymer-last based microfabrication

To enable the microfabrication of the freestanding interconnects and the SE demonstrator, the so-called “Polymer-last” approach developed by project collaborators at Philips Research for microfabrication of flexible electronics is utilized [23]. The technology hinges on introducing the polymers (e.g., PDMS for the substrate) as the last step after the device processing has been performed, since the polymer materials are challenging in terms of integration in device microfabrication flow. A demonstration of the technology was shown by Mimoun et. al. [23] where an ultrasound detector was fabricated as a monolith on a single wafer with a number of rigid device islands interconnect by flexible metallic interconnects sandwiched between layers of polyimide. Subsequently the flexible device could be wrapped around a minimally invasive catheter tip. The same technology was chosen for the processing of the freestanding interconnects and the demonstrator device fabrication (WP4). The microfabrication-based approach warrants the miniaturization of the interconnects and the device islands as well as an integrated process flow for the complete SE device. Furthermore, it gives the possibility of a CMOS compatible fabrication-process useful for mass production.

1.3.3 Reliability testing and characterization

In order to ensure the lifetime of the highly stretchable electronics, it is important to perform reliability testing by characterizing the elastic stretchability of the interconnects not only in the idealized uniaxial tension cases but also in more complex multiaxial deformations mimicking realistic interconnect loading during device operation. Furthermore, it is important to characterize the cyclic fatigue life of the interconnects and their electrical characteristics as a function of the applied deformation.

To this end, one of the project goals was to develop a tensile tester capable of applying high-resolution multiaxial displacements in conjunction with high resolution microscopy techniques such as scanning electron microscopy (SEM), optical profilometry and high-resolution optical microscopy, etc. Furthermore, due to the freestanding and miniaturized nature of the interconnect, 100s of microns of displacements due to vibrations can induce 1000s of percent of global interconnect strain leading to interconnect failure. Therefore, specimen handling during processing all the way until testing is challenging. Hence, the testing methodology should be integrated with a sample platform (silicon test chip) using which the interconnects can be delicately handled while ensuring sample integrity.

Due to the small-scale nature of the samples, especially due to the sub-micron sample thickness, common for microfabricated samples, bulk material properties cannot be assumed due to the likely presence of mechanical size-effects [24, 25]. The material properties and especially the yield strength of the sample is important to be characterized for reliability predictions and further interconnect geometry optimization. To this end specialized experiments were planned to be performed such that they could be utilized in conjunction with WP3 for on-sample characterization using integrated digital image correlation (IDIC). Furthermore, to enable the application of digital image correlation (DIC) on the highly stretchable sample a new DIC patterning technique needed
to be developed to allow patterning of the highly delicate sample as current patterning techniques are inadequate.

1.4 Thesis Layout

The thesis is divided into two parts. In Part I (chapter 2 and 3) the design and performance of the highly stretchable interconnects developed in this project is discussed. Part II (chapters 4, 5 and 6) describes the experimental testing techniques and tools developed for the characterization of the interconnect structure and its constituent materials. In chapter 2, the design of a highly stretchable freestanding interconnect termed as the Rotation Out-of-plane Elongation (ROPE) interconnect is discussed. Experimental characterization test of the interconnect with elastic and plastic stretchability, electrical resistance change with stretching and fatigue life are presented. Chapter 3 discusses the multiaxial stretchability of the ROPE interconnect and the results are compared with two other freestanding interconnect geometries as well as the multiaxial stretchability requirements of an inflatable detector application using FEM modelling. Experimental results of the multiaxial loading of the microfabricated ROPE sample are compared against FEM simulations. In Chapter 4 a new patterning technique to enable patterning of highly delicate small-scale samples such as the ROPE interconnect is presented. A speckle pattern is necessary for application of DIC and thus IDIC, which is utilized here for the characterization of the ROPE interconnect. Results from patterning of the ROPE interconnect as well as a number of other challenging delicate samples are presented. Chapter 5 discusses the experimental testing methodology developed to test the ROPE interconnects for various characteristics. The methodology consisting of a test-chip design combined with the design of the multiaxial loading tensile stage to perform accurate electro-mechanical as well as fatigue characterization is presented. In Chapter 6 a bulge test-based test methodology to characterize the material properties of thin-film samples under compressive residual stress is discussed. The Bulge test method is a convenient technique for characterizing freestanding thin-films and the extension to compressive residual stress samples allows, to test different materials to optimize the material used for the stretchable interconnects. Finally, in chapter 7 key conclusions from this work are presented along with recommendations for future work.
Part I

Design and performance of a highly stretchable interconnect for high density stretchable electronics
Ultra-stretchable interconnects for high density stretchable electronics

Reproduced from:
Micromachines 8, no. 9 (2017): 277.

Abstract

The exciting field of stretchable electronics (SE) promises numerous novel applications, particularly in-body and medical diagnostics devices. However, future advanced SE miniature devices will require high-density, extremely stretchable interconnects with micron-scale footprints, which calls for proven standardized (complementary metal-oxide semiconductor (CMOS)-type) process recipes using bulk integrated circuit (IC) microfabrication tools and fine-pitch photolithography patterning. Here, we address this combined challenge of microfabrication with extreme stretchability for high-density SE devices by introducing CMOS-enabled, free-standing, miniaturized interconnect structures that fully exploit their 3D kinematic freedom through an interplay of buckling, torsion, and bending to maximize stretchability. Integration with standard CMOS-type batch processing is assured by utilizing the Flex-to-Rigid (F2R) post-processing technology to make the back-end-of-line interconnect structures free-standing, thus enabling the routine microfabrication of highly-stretchable interconnects. The performance and reproducibility of these free-standing structures is promising: an elastic stretch beyond 2000% and ultimate (plastic) stretch beyond 3000%, with <0.3% resistance change, and >10 million cycles at 1000% stretch with <1% resistance change. This generic technology provides a new route to exciting highly-stretchable miniature devices.

2.1 Introduction

The emerging field of stretchable electronics, especially with its recent advances in high stretchability, has opened a new arena of novel and exciting applications, particularly for medical devices [2, 6, 26]. Two main approaches for realizing stretchable electronics (SE) exist: intrinsically stretchable (organic) materials [27–29], and (inorganic) conductor materials made stretchable through inventive mechanisms that convert small strains into a larger global stretch [5–7, 30]. In order to realize advanced high-density stretchable electronic miniature devices on a commercial scale, e.g., an inflatable catheter-tip
ultrasound camera with variable-zoom functionality for minimally-invasive surgery (Figure 2.1a), challenges regarding (i) miniaturization; (ii) manufacturability; and (iii) high interconnect stretchability need to be addressed. High-density circuit integration of application-specific integrated circuit (ASIC) devices requires high-density stretchable wiring with width and thickness dimensions in the range of 1 \( \mu m \), and an overall footprint in the order of tens of micrometers. The required feature sizes are one to three orders of magnitude smaller than the (sub)millimeter-sized serpentines and arches commonly proposed in the literature [6]. This constitutes a real challenge for mesoscopic fabrication procedures such as screen printing and transfer printing, which are frequently used for fabricating large-scale stretchable electronics [6, 20, 22, 31], while advancements in this area are being made [32]. Conversely, the submicron-sized features are naturally achieved when using the standard fine-pitch photolithography-based IC techniques that are routinely used for the ASIC fabrication. This also warrants the commercial mass production of highly stretchable devices, as long as the process flow utilizes available bulk IC microfabrication equipment and proven CMOS-type process flows. For most (stretchable) electronics applications, having high areal coverage is important, especially for detectors [33]. This calls for a smaller interconnect footprint area and a higher interconnect stretchability. For instance, the inflation of a typical, planar 2D charge-coupled device (CCD) detector array with a fill factor of 90% [19] (i.e., 90% of the surface area covered with rigid, square detector islands) to a hemisphere entails a global strain of 57%, but requires the interconnects between the islands to stretch beyond 1100% (using relation from [34], see Supplementary Materials for details, also illustrated in Figure 2.1b). For a fill factor of only 50%, still, a stretchability of \( \sim 200\% \) is needed. Note that ‘stretch’ is defined as the global linear strain of the stretchable part of the interconnect (i.e., change in footprint width over initial footprint width (illustrated in Figure 2.2)). In addition, reliable device operation requires the interconnect structures to accurately recover their original shape upon unloading, which means that the interconnect material should remain below its engineering yield limit. In other words, the key parameter to ensure mechanical reliability is elastic stretchability (defined here as fully recoverable deformation without any visible interconnect shape change upon unloading [35]) and not the ultimate (plastic) stretchability (i.e., stretchability at interconnect fracture in the plastic regime) that is often reported in the literature. Moreover, a large safety factor (on the order of two) is required for cyclic operation below the fatigue limit, as well as for medical applications. Therefore, to unlock the full potential of future high-density SE devices, interconnects that can stretch well beyond 1000% should be combined with a microfabrication solution that warrants the miniaturization and manufacturability of these interconnects and their stretchable devices.

Most of the current stretchability solutions stay (well) below 100% elastic stretch [6]. Many proposed solutions hinge on some sort of serpentine-shaped metal interconnect pattern adhered to a rubber substrate [6], for which the stretching behavior is confined to in-plane deformation. This deformation triggers early plasticity at the interconnect corners, and eventually results in interface delamination, which causes local interconnect rupture [20, 31]. In a recent thorough optimization study, the elastic stretchability of substrate-adhered interconnects has been pushed up to 350% [36]. A further huge increase in stretchability calls for a different approach to circumvent such issues as interface delamination and a substrate-induced confinement of deformation modes. Recently,
in two milestone studies [22, 36], free-standing interconnects have been utilized to boost stretchability. For both approaches, however, integration with standard IC microfabrication has not yet been explored and would be far from trivial, as the processing starts from a polymethyl methacrylate/polyimide (PMMA/PI) layer [22] and a Kapton film [36] as substrate, respectively, while the amount of elastic stretchability is, ∼200% and unreported, respectively (as Su et al. discussed in [37]). To achieve advanced high-density SE devices, it would be highly advantageous if the interconnect processing would be fully integrated with (CMOS-type) microfabrication.

Here, the Flex-to-Rigid (F2R) microfabrication platform is adopted and extended to enable CMOS-type processing of highly stretchable (>1000%) interconnects with micron-scale interconnect footprints. F2R is a generic technology that makes CMOS devices flexible by etching the silicon ASICs into separate islands connected by flexible interconnects (formed from the original CMOS-produced interconnects of the ASICs) [23, 38]. The strength of the F2R technology has been demonstrated by a flexible (not yet stretchable) ultrasound camera mounted on top of a catheter tip, which was microfabricated in a planar configuration (Figure 2.1d) and subsequently wrapped around a narrow tip (Figure 2.1c). Subsequently, this technology was extended using meander-type interconnects, which allow for limited stretchable CMOS-devices [39]. It is important to note that the F2R approach is based on a true post-processing procedure that works on any (pre-processed) CMOS-processed IC wafer, i.e., all ‘exotic’ processing steps and materials are introduced at the end without affecting the ASIC functionality. By extending the F2R technology to process highly stretchable interconnects, the technological requirements on miniaturization and manufacturability of stretchable interconnects for high-density SE devices are automatically fulfilled. Therefore, the combined challenge consists of (i) an extension of F2R technology to enable free-standing interconnects; (ii) a dedicated stretchable interconnect design that takes into account that the back-end-of-line metallization with a typical thickness of ∼0.5µm is processed into the highly free-standing interconnects; and (iii) a full exploitation of the broad 3D kinematic freedom through an interplay of buckling, torsion, and bending to achieve a reversible stretchability well beyond 1000%.

2.2 Interconnect Design and Mechanics

The conceptual design of the free-standing interconnect consists of slender beam elements (see Figure 2.2 (left)), i.e., the beam thickness is small compared to the beam width and length. This is typical for CMOS-processed (planar) structures. For such beam elements, the maximum elastic tip deflection can be achieved in bending, and more specifically by loading the beam along its thickness direction rather than the (considerably stiffer) width direction. Therefore, the bending beams should rotate in order to align this direction in the global stretch direction, as visualized in Figure 2.2 (right). To this end, at both island connection points, a slender torsion beam is inserted that rotates the bending beams sideways. Since the structure is initially planar, it will first deform by in-plane opening, which is a deformation mode that quickly exceeds the elastic limit and should be avoided. Therefore, a buckling instability is intentionally triggered, by ensuring that the beam elements are long and thin, to induce a transition from in-plane opening to
Figure 2.1: Concept illustration of an inflatable catheter-tip ultrasound detector with highly stretchable interconnects (a,b), versus the current state-of-the-art technology in miniature flexible devices: the capacitive micromachined ultrasound transducer (CMUT) array on a catheter tip produced using the F2R technology (c,d). © (2013) IEEE. Reprinted, with permission, from [23]. Proposed stretchable detector head, adding a variable-zoom functionality with a large magnification factor by inflating and deflating the stretchable membrane. A flat (deflated) configuration illustrates the rigid silicon islands with a fill factor of 0.9 on a rubber substrate (a), and a finite element (FE) simulation of (half of) the cross-section, which demonstrates the requirement of the interconnect to stretch >1200% upon inflation of the detector to a hemisphere with detector islands covering 90% of the membrane area (b); (c) The fully-assembled CMUT detector with the detector islands bent around the catheter tip; (d) The device was microfabricated on a flat standard wafer and suspended by removable polyimide tabs using F2R post-processing technology.

out-of-plane rotation, well before the onset of plasticity due to in-plane opening. Finally, by connecting the slender bending and torsion beams alternatively at opposite ends, a continuous interconnect structure is formed, as shown in Figure 2.2 (right). It will be shown that this mechanics-based design of an ultra-compliant, free-standing interconnect activates bending, torsion, and their interplay, and enables it to reach beyond 2000% reversible stretchability.

To ensure that the metal is strained only in the elastic regime, the von Mises yield criterion is used, i.e., the von Mises equivalent stress, $\sigma_{vm,max}$ should remain below the yield stress, $\sigma_y$. The bending beams (with a rectangular cross section) are being deflected as guided cantilever beams. For an individual guided cantilever beam, the equivalent von Mises stress is equal to the maximum normal $\sigma_{vm,max}$ along the beam length, which is maximum at the top and bottom surface at the beam’s ends (e.g., point b in Figure 3), i.e.,
Chapter 2.

Figure 2.2: Concept illustration of the basic working principle of the free-standing interconnect design. (Left) Basic components of the design, with two torsion beams at the corners and multiple bending beams in the middle; (right) Basic deformation modes in the stretched state with the corner beams elastically rotated by 90°, which allows the inner beams to elastically bend in the direction of their thickness. δ and θ represent the tip deflection of the bending beam and the rotation angle of the torsion beam, respectively.

\[ \sigma_{vm,max} = \sigma_{max} \leq \sigma_y, \]  
(2.1)

The maximum tip deflection \( \delta_{max} \) (δ illustrated in Figure 2.2) is related to the maximum stress in the beam, i.e., \( \sigma_{max} \) using eq. 2.2. At the limit where the complete beam is still in the elastic regime (\( \sigma_{max} = \sigma_y \)), \( \delta_{max} \) is proportional to the length squared and inversely proportional to the thickness.

\[ \delta_{max} = \frac{(\sigma_{max})l^2}{3Et} = \frac{\sigma_y l^2}{3Et} \]  
(2.2)

For an individual torsion beam with a rectangular cross-section, for which plane stress can be assumed in both the thickness and width direction, the maximum equivalent stress equals \( \sqrt{3} \) times the maximum shear stress at the beam’s surface i.e.,

\[ \sigma_{vm,max} = \sqrt{3} \tau_{max} \leq \sigma_y \]  
(2.3)

The maximum angle of twist \( \theta_{max} \) (θ illustrated in Figure 2.2) is related to the maximum shear stress \( \tau_{max} \) in the beam using eq. 2.4 (see Supplementary Materials for details), which shows that at a certain yield stress, \( \theta_{max} \) is directly proportional to length \( l \) and inversely proportional to thickness \( t \).

\[ \theta_{max} = \frac{(\tau_{max})l}{ctG} = \frac{\sigma_y l}{\sqrt{3}ctG} \]  
(2.4)

where \( c \) is a scalar function of the aspect ratio of the beam width \( b \) and thickness \( t \), which is approximately equal to one for \( b/t > 4 \), while \( b/t > 6.5 \) for the current structures due to processing constraints.

The total stretchability of the interconnect, \( \varepsilon_{global} \) i.e., (\( \frac{\text{stretched interconnect footprint width}}{\text{initial interconnect footprint width}} - 1 \)) can be approximated as:
\[ \varepsilon_{\text{global}} \approx \frac{(n-2)\delta_{\text{max},c} + 2\delta_{\text{max},t}}{nb + (n-1)g} - 1 \] (2.5)

where, \( n \) is the total number of beams, while \( b \) is the beam width and \( g \) is the gap between consecutive beams (see Figure 2.2 (left)). \( \delta_{\text{max},c} \) represents the maximum deflection provided by each of the guided cantilever beams, while \( \delta_{\text{max},t} \) denotes the deflection of each of the torsion beams in the stretch directions. Unlike the guided cantilever beams, a straightforward analytical equation for \( \delta_{\text{max},t} \) is far from trivial, since the torsion beam stiffness is highly non-linear due to its effective area moment of inertia (\( I \)) varying with increasing angle of twist \( \theta_{\text{max}} \). Moreover, the contribution of the torsion beam deflection is only significant for large beam lengths and high global displacements. For example, finite element (FE) simulations show that for Figure 2.4g, with a relatively high beam length of 100 \( \mu \text{m} \), the contribution of the torsion beam is \( \sim 23\% \) of \( \varepsilon_{\text{global}} \) at \( \varepsilon_{\text{global}} = 2040\% \), while at \( \varepsilon_{\text{global}} = 1625\% \), this contribution reduces to \( \sim 13\% \) of \( \varepsilon_{\text{global}} \). Furthermore, for typical dimensions, such as those considered here, \( \sigma_y \) is reached first in the guided cantilever beams. Thus, it does not dictate the interconnect elastic limit. Therefore, \( \delta_{\text{max},t} \) is neglected, resulting in:

\[ \varepsilon_{\text{global}} \approx \frac{(n-2)\sigma_y l^2}{nb + (n-1)g 3Et} - 1 \] (2.6)

It should be noted that eq. 2.6 is an approximation, and does not take into account the stress concentrations in the inner corners. Thus, it is not used here to exactly estimate the maximum elastic stretch, for which FE simulations are employed instead. Nonetheless, it provides direct relationships (proportionalities) between maximum stretchability (for a given value of \( \sigma_y \)) and geometric parameters i.e., beam length \( l \), width \( b \), thickness, \( t \), number of beams \( n \), etc., which is invaluable for the design process.

It can be seen from eq. 2.2, 2.4, and 2.6 that in order to achieve higher stretchability, i.e., to postpone the onset of plastic yielding, the thickness of the members (both torsion and bending) should be as small as possible, while the length should be maximized. The width of the beams does not directly influence the maximum stress, and can be chosen to accommodate other design requirements such as interconnect electrical resistance. However, wider members result in a larger initial footprint area, and thus a reduced global stretchability. The gap between adjacent members should be minimal to reduce the initial footprint area, and leave as much space as possible for the ASIC islands.

To reduce stress concentrations, the radii of the inside corners of the beam connection points should be at their maximum without increasing the footprint width, i.e., half the gap size. An additional reason to choose the dimensions as small as possible is to benefit from the so-called ‘mechanical size effects’, which may greatly enhance the yield strength of the aluminum and thus the global elastic stretchability, as is addressed in the following section. On the other hand, a higher length and smaller thickness result in a higher out-of-plane deflection. For example, for a footprint length of 50 \( \mu \text{m} \) (\( b = 2 \mu \text{m} \) and \( t = 0.3\mu \text{m} \)), the interconnect deflects out-of-plane by \( \pm 7.5 \mu \text{m} \), as seen in Figure 2.3, while for a length of 100 \( \mu \text{m} \) (\( b = 2 \mu \text{m} \), \( t = 0.3 \mu \text{m} \)), the maximum out-of-plane deflection is estimated at \( \pm 27 \mu \text{m} \) by FE simulation. Therefore, the depth of the trench between the two silicon islands connected by the interconnects should be at least twice that of the maximum out-of-plane deflection, which, for the
Chapter 2

Figure 2.3: Finite element method (FEM) simulation of an interconnect design, showing: (left) Initial unloaded structure; (right) Stretched interconnect structure, with an out-of-plane displacement field $U_z$ overlaid on top. Note that the two ends marked a and a’ always remain in the initial interconnect plane (i.e., $U_z = 0$), along with the three parallel gold-colored solid lines. Point b and c denote the location of $\sigma_{\text{max}}$ and $\tau_{\text{max}}$, respectively.

studied interconnects’ geometries is within the range of typically used island heights. Lastly, a key aspect of the design is that the footprint width and footprint length are uncoupled. This results in the freedom to, for example, increase the length of the beams and decrease their thickness in order to increase interconnect stretchability, while also keeping the beam width and gap, and thus the interconnect footprint width fixed, in order to achieve a high fill factor for the ASIC island at the same time. However, the processing-induced curvature, interconnect resistance, and out-of-plane interconnect deflection, which increase with increasing length and decreasing thickness, together or individually will act as the design constrains, on a case-by-case basis.

2.3 Results

Aluminum test structures for accurate micromechanical testing (see 2.4 for details) inside a scanning electron microscope were fabricated using the F2R processing scheme (see 2.4 for details). Figure 2.4a shows a 100 $\mu$m-long test structure with four inner members in the initial (load-free) state, which clearly shows an out-of-plane curvature due to processing-induced residual stresses, as is common in microfabrication. As predicted, at $\sim 10\%$ global stretch, the initial regime of in-plane deformation started to transition to out-of-plane rotation of the structure, while $\sim 45^\circ$ out-of-plane rotation occurs at 190% global stretch, see Figure 2.4c. Note that due to the (processing-induced) out-of-plane curvature, along with the small width and thickness of the beams, the alignment of the thickness direction of the bending beams with the loading direction after rotation only becomes clear upon close inspection.

To study the elastic stretchability, at each loading step, with increasing applied displacement, the sample is brought back to its unloaded configuration, after which possible permanent shape changes are analyzed in detail. When the structure is unloaded after 190% stretch, no systematic shape change was observed, which demonstrates that the
Figure 2.4: Selected experimental and numerical results. (a) The initial (load-free) state of the interconnect structure with a beam length, width, and thickness of 100, 2, and 0.3 μm, and gap of 1 μm; (b) Microtensile stage placed inside a large chamber scanning electron microscope (SEM), with a magnified view of the tensile stage; (c,d) Demonstration of reversible stretchability: in each stretching cycle, the interconnect structure is stretched to an incrementally higher global strain (left figure), while the shape after unloading (corresponding right figure) should be compared with the initial configuration; (e) Beyond 2040% global stretchability, plastic deformation sets in, which becomes clearly visible after unloading from the 2650% stretched state; (f) The ultimate (plastic) stretchability is reached beyond 3000% global strain. Exactly the same deformation behavior was observed for the other four parallel structures in the field of view (Figure S1 in the Supplementary Materials section); (g) finite element method (FEM) elastoplastic simulation at 2040% global strain (compare with (d)), for a yield stress of 700 MPa: equivalent von Mises stress overlaid on the stretched configuration (left) and subsequent unloaded configuration (right); (below) Unloaded configurations for increasing yield strength values, showing that the yield stress is at least $\sigma_y = (700 \pm 100)$ MPa; (h,i) Electrical resistance measurements (h) for a quasi-static stretch to 3000% and (i) during 10 million cycles of 1000% elastic stretch.
stretchability is fully reversible. After increased loading, the corner members and thus also the middle bending members rotate further out of plane. At \(\sim 1500\%\) stretch, the middle members have completely rotated by \(\sim 90^\circ\), and further deformation is accommodated completely by bending in the stretch direction, as indented by the design. Note that even for the case of Figure 2.4d, no permanent shape change could be detected, showing that the structure can be elastically stretched up to 2040%.

Elastic reversibility is lost at 2265% global stretch, where for the first time a minute shape change could be observed in the corners of the structure, where the highest stress occurs. The shape change only becomes clearly visible after unloading from the 2650% stretched state, as seen in Figure 2.4e. Upon further deformation, the structure stretches into an almost straight wire at 3004% of global stretch, see Figure 2.4f. All of the structures remained intact, while the four parallel structures within the field of view (Figure S1 in the Supplementary Materials) demonstrated exactly the same deformation behavior (including the minor asymmetry due to the processing-induced curvature). This demonstrates the robustness of the interconnect structures due to their ultra-flexible nature. In fact, contrary to their fragile appearance, the interconnects prove to be quite immune to rough handling of the wafer and test chip.

Elastoplastic FEM simulations were performed (see 2.4 for details) for the 100 \(\mu\)m-long interconnect structure. Figure 2.4g shows the deformed shape at 2040% global stretch, and the corresponding shape after unloading. Even though the processing-induced curvature is not included in the simulations, the deformed shape shows excellent agreement with the experiments (Figure 2.4d), which supports our conclusion that the elastic stretchability indeed exceeds 2040%. Also, the local curvatures due to the bending of the inner members and the torsion and bending of the outer members are predicted accurately, which provides further confirmation that the underlying mechanics principles exploited in the design are valid for these microfabricated free-standing structures.

The electrical resistance measurement (Figure 2.4h) shows that (i) the absolute value (32.5 \(\Omega\)) varies by 10% from the theoretical value based on the interconnect geometry, and (ii) the resistance remains constant within 0.26% up to an extreme ‘plastic’ stretch of 2860%, which proves that the electrical behavior of the interconnects is very stable. More importantly, the interconnects exhibit excellent fatigue response. As seen in Figure 2.4h, all six parallel interconnects survived 10 million cycles at a maximum cyclic stretch of 1000% without failure (see video in Supplementary Materials section), with the resistance remaining constant within 1% and no sign of plasticity. This confirms the goal of achieving a highly reproducible, high elastic stretchability that could ensure reliable device operation.

The onset of plasticity, which manifests as a permanent shape change in an interconnect (see Figure S2 in the Supplementary Materials for details) is predicted by the yield strength of the material. However, at submicrometer scales, the yield strength can significantly vary (and typically increase) from bulk material characteristics due to well-known ‘mechanical size effects’, which are commonly referred to as ‘smaller is stronger’ [24, 25, 40]. The yield strength of the interconnect material is estimated by qualitatively fitting the deformed shape of the FEM simulation onto the experiment shape by perturbing the yield strength until the simulated unloaded configuration at the elastic limit (i.e., 2040%, see (Figure 2.4h)) is similar to the experimental unloaded configuration (this procedure is similar to inverse characterization methods reported in the literature
Chapter 2.

[41–44] for MEMS and thin films). An unloaded configuration after a stretch of 2040% without any discernable shape change is achieved only when a yield strength of 700 (±100) MPa is used, which is 10 times higher than that of bulk unalloyed aluminum (∼70 MPa), as seen in Figure 2.4h. Since the maximum stress is found in a small volume of ∼0.1 µm$^3$ (near the surface at the bottom of the torsion beams and near the surface in the inner corners), the high yield strength likely results from the aforementioned ‘mechanical size effects’. The statistical size effect in polycrystalline aluminum samples with similar dimensions, even in pure tension, has been reported in the literature [45]. Furthermore, the three most prominent strengthening effects in metals are due to strain gradients [24, 46], dislocation starvation [24, 47], and constrained boundary layers [24]. Our miniature interconnect structures have a native oxide layer, and are extremely bent with highly localized stresses and high strain gradients in the inner corners and submicron-sized grains. Therefore, the individual role of each of these effects cannot be trivially assessed. A detailed study into these size effects is of high scientific interest and may be utilized to further enhance the elastic stretchability. It should be noted that the goal here was not to precisely determine the yield strength of the interconnect material, but rather to show that the yield strength can vary substantially from bulk properties (due to size effects), which can be exploited to boost interconnect stretchability.

2.4 Conclusions and discussion

To conclude, in this work, a new route is opened towards standard CMOS processing of ultra-stretchable, free-standing interconnects with a micron-sized footprint, which could enable extreme device stretchability. This could be realized by reserving areas on a CMOS-processed wafer upon which the free-standing interconnects are fabricated to connect the individual devices, followed by through wafer etching to separate the devices and release the interconnects, similar to proposals in [23, 39]. Such a process scheme keeps the thermal budget below 400 °C, which is a key requirement if CMOS devices are being post-processed. Furthermore, the starting point of the process is a Si-substrate without any specific mechanical or electrical requirements for the substrate. Hence, any CMOS wafer, e.g., a commercially available ASIC wafer, can serve as a substrate.

Further steps towards actual applications are to be taken next. High-density SE devices typically require multi-level wiring between the ASIC islands. The adopted F2R processing scheme is already capable of producing multi-level wiring, which does require the interconnects to be electrically isolated. This could be easily achieved by adding a single processing step to the F2R processing scheme, i.e., deposition of a very thin conformal coating (e.g., of parylene). Moreover, for a 2D (initially flat) detector array conforming to a curvilinear surface (Figure 2.1b), the ASIC islands connected by the interconnect show some degree of relative displacement perpendicular to the main interconnect stretch direction, as well as relative rotations (currently being studied), which need to be accommodated by the interconnect.

The discussed 100 µm-long interconnects yield an exceptional elastic stretchability of >2000%, which enables breakthrough applications such as the variable-zoom catheter-tip ultrasound detector (Figure 2.1). Moreover, the ultimate (plastic) stretch of >3000% is also highly interesting for promising one-time stretchable devices [48]. For instance,
a CCD-detector stretched into an almost full sphere that mimics a fly’s eye and can ‘see’ in all directions (such as devices reported in [49, 50]), can serve as a lightweight omni-directional camera on top of bug-like miniature drones.

Supplementary Materials

The following are available online at www.mdpi.com/2072-666X/8/9/277/s1, Figure S1 Parallel interconnects, Figure S2 Equivalent accumulated plastic strain and Video S1 Fatigue test.

Acknowledgements

This work was supported by the Vidi funding of J.P.M.H. (project number 12966) within the Netherlands Organization for Scientific Research (NWO). This work was also partially funded by project T62.3.13483 in the framework of the Research Program of the Materials innovation institute (M2i) (www.m2i.nl), and in the framework of the ECSEL JU Project InForMed, grant number 2014-2662155 (www.informed-project.eu).

Fabrication

The fabrication process starts with the growth of 1 µm and 5 µm PECVD SiO₂ on the front and backside respectively of a 150-mm double side polished 400 µm thick Si substrate. First, the oxide layer on the back side is patterned by photolithography and dry etching, forming the hard etch mask for the through-wafer etching required at a later stage. Next, a 300 nm thick layer of aluminum is sputter deposited on the front side of the wafer. A photoresist is spin coated (90 °C soft baked), exposed and developed. Using this resist mask, the aluminum is dry etched defining the desired interconnect patterns along with the re-routing layer and the bond pads. After etching, the resist mask is removed in O₂ plasma. A 5.2 µm thick layer of polyimide (PI 2611) is spin coated (soft baked on a hotplate 120 °C for 5 min) on top of the Al structures and cured in nitrogen atmosphere at 275 °C for 2 h. A layer of Al is sputtered (200 nm) on top of the cured polyimide. A layer of photoresist (HPR504) is spin coated, exposed by stepper lithography and developed. The top Al is wet etched with the resist serving as a mask. The patterned Al layer will serve as a hard etch mask for the patterning of the polyimide at the end of the process. The interconnects are next released from the Si substrate by back side Deep Reactive Ion Etching (DRIE) for 25 min with the aid of the 5 µm hard etch PECVD Oxide mask. The final release step is done when the PI is etched selectively from the interconnects from the top using the patterned Al hard etch mask.

Experiments

Proof of principle interconnect test structures suspended between two silicon islands, resembling actual applications, were fabricated using the F2R processing scheme. The free-standing interconnects were fabricated on a (16mm×5 mm) test chip, as shown in
Figure 2.5: Design and details of the fabricated test chip with highly stretchable interconnects. (a) The design of the (16 mm × 5 mm) chip (in green), which is suspended in the silicon wafer (in blue) by polyimide tabs (in orange). Notched silicon columns (i) are processed to provide rigidity to the structure during processing and handling. Sacrificial silicon islands (ii) are created and suspended by similar polyimide tabs to reduce etching time; (b) Magnified view of two parallel free-standing interconnects; in total there are six parallel interconnect structures, processed in each test chip; (c) Magnified side-view of a (200 μm wide, 100 μm thick, double V-notched) silicon bridge inside the bottom ‘column’ of the test chip; (d) Magnified top-view of a polyimide tab, which can easily be locally melted to singulate the test chip.

To maximize space, six parallel interconnect structures are processed on each test chip, which can be simultaneously mechanically tested. These test structure were tested with a commercial (Kammrath & Weiss GmbH, Dortmund, Germany) microten-sile stage designed for in-situ scanning electron microscope (SEM) (Figure 2.4b). The test chip was glued onto two clamps with a flat acrylic top surface using UV-curable glue by Loxeal®. The in-situ experiments were performed inside a FEI Quanta 600 FEG-SEM under high vacuum, in secondary electron imaging mode. The four-probe electrical resistance measurements were performed using integrated electrical probes that make contact with bonding pads connected to the interconnects on the test chip, while the deformation state of the interconnects was visualized with an optical microscope. The fatigue measurements were also performed under the optical microscope with cyclic load-ing applied at 10 Hz. After every 300 cycles, the electrical resistance of the interconnects was measured to characterize interconnect failure. Optical images of the interconnects were obtained every million cycles.
Simulations

The elastoplastic FE simulations were performed using a commercial FEA package, Marc Mentat 2014®. 20-node quadratic brick elements were used to model the geometry, with three elements through the thickness to accurately capture the bending behavior. A non-linear solution scheme was used to capture the large displacements upon stretching as well as the buckling bifurcation in the initial phase of loading. To assist the structure to buckle, a minute perturbation force of $10^{-10}$ N was applied to a corner node in the middle member of the structure and removed after the bifurcation point has been passed. Bulk elastic properties of pure aluminum were used for the material properties, i.e., a Young’s modulus of $E = 69$ GPa and a Poisson’s ratio of $\nu = 0.33$. Moreover, a standard Swift power law relationship was used to describe the hardening behavior with a strength coefficient ($K$) varying from 0.278 GPa ($\sigma_y = 70$ MPa) to 10.1 GPa ($\sigma_y = 700$ MPa) and strain-hardening exponent of $n = 0.2$, as common for aluminum.
CHAPTER 3

Omnidirectional stretchability of freestanding interconnects for stretchable electronics

Abstract

Advanced stretchable electronics applications, such as health monitoring patches, high-density curvilinear detectors etc., not only require the interconnects in these devices to accommodate large uniaxial strains but they should also be able to withstand complex multiaxial loading such as in-plane and out-of-plane shear loading for reliable and optimal device operation. However, only uniaxial stretchability of these stretchable interconnects is typically characterized and reported in the literature. Here, we compare three freestanding typical interconnect geometries on the basis of their multiaxial stretchability to evaluate their feasibility for typical stretchable electronics applications. The three geometries include: (1) the (traditional) serpentine structure, that has been studied most extensively in the literature (2) the Rotation Out-of-Plane Elongation (ROPE) interconnect, designed to exploit buckling and out-of-plane rotation to enhance stretchability [Shafqat et. al., Micromachines, 2017, 8(9), 277] and the (3) the non-buckling In-Plane Elongation (IPE) interconnect, designed to fit a maximum number of beam members within the footprint area. Multiaxial Finite Element (FE) simulations were performed by loading the interconnects in the xy-, yz- and xz- planes under a range of loading angles. The simulations show that while the serpentine interconnect is able to stretch under multiaxial loading, the magnitude of stretchability is relatively low (~40%). The IPE interconnect exhibits a very high stretch (>1200%) under (normal) uniaxial in-plane stretching, yet even moderate shear strains already result in self-contact, which typically leads to failure at the nm/µm scales considered here. The FE simulations for the ROPE interconnect show that it can stretch omnidirectionally in the xy- yz- and xz loading planes with high values of stretchability (>400%), for the geometry considered here. To validate this, multiaxial loading experiments were performed on the ROPE interconnect samples by loading the samples in uniaxial in-plane stretching, in-plane shear and out-of-plane shear loading cases. The results show that microfabricated samples, with a footprint height of 50 µm, can indeed deform omnidirectionally, with the experimental results adequately matching FE simulations.
3.1 Introduction

Stretchable electronics has recently emerged as a new technology resulting in electronic devices which can bend, twist and stretch, in contrast to the traditionally rigid and brittle (micro-) electronics [2, 5, 51]. The deformable nature of these devices allows them to be conveniently and closely integrated with non-flat and non-rigid surfaces, such as the human body, resulting in applications such as smart health monitoring body patches [52, 53]. Furthermore, the deformability of the typically initially flat-processed microelectronics components has been used to add more functionality to several applications, e.g., for a dynamically tunable electronic eye [9], a smart electronics contact lens [54], a fictionalized inflatable balloon catheter [2] and a wide angle arthropod eye camera [50], etc. Recent developments in the field have enabled highly stretchable electronics which on top of high stretchabilities allow to boost the typically low areal coverage of the detection circuitry, as discussed in Refs. [35, 55]. The configuration typically employed in these highly stretchable devices consists of rigid Application-Specific Integrated Circuit (ASIC) islands placed on top of an elastomer substrate and electrically interconnected by free-standing interconnects. The freestanding interconnects, contrary to the conventional elastomer-embedded configuration [17], are less constrained by the substrate and can subsequently result in significantly higher stretchabilities [22, 35, 36, 55, 56]. However, even in the case of highly stretchable electronics, only the uniaxial stretch behaviour is typically considered, while in real-world applications pure uniaxial deformation is rarely the case and even significant multiaxial deformation can be required. For instance, in health monitoring smart patches, which is currently one of the most popular commercial applications of stretchable electronics, it is easy to imagine that the compliant patch would be deformed not only in biaxial tension but also by shearing, twisting, and bending to comply to the complex body movements. Furthermore, even in more well-controlled applications such as a circular detector array being inflated to a hemi-sphere, such as in the dynamically tunable electronic eye [9], wide angle arthropod eye camera [50], or a variable field of view catheter tip detector (see Figure 3.1a), significant multiaxial interconnect loading may be required. As it can be seen in Figure 3.1, that the ASIC islands closer to the detector periphery show a significant misalignment, resulting in the interconnects between these islands experiencing a maximum in-plane shear strain ($\varepsilon_{xy}$) of 134.3%, a maximum rotation (about the local y-axis $\theta_y$) of -18.2°, with a maximum in-plane normal strain ($\varepsilon_{xx}$) of 345.5%. It should be noted that the ASIC islands close to the detector periphery (showing the maximum misalignment) are of special interest for the device functionality as they provide the high-angle signals, which is the key reason for transforming the flat detector array into a curvilinear shape.

Therefore, it is important to consider the complex multiaxial deformation that the stretchable electronics devices and consequently the stretchable interconnects would be subjected to and to investigate whether the interconnects would be able to sustain this multiaxial deformation. In this paper, three typical highly stretchable interconnect geometries are analysed by virtual testing (finite element simulations) and compared on the basis of their multiaxial stretchability. Finally, multiaxial loading experiments are presented for the interconnect geometry with the best performance in terms of omnidirectional deformation.
Figure 3.1: Finite element simulation of the inflation of a circular detector array, with (a) geometry of the full array in the uninflated state with a quarter of the array highlighted and modelled here, (b) inflation of the array to a hemisphere, e.g., to obtain wide-angle images. The rigid cuboid detector islands are placed on a stretchable (elastomer) substrate and electrically connected to adjacent islands by stretchable interconnects (represented by dashes lines here). Post processing (see 3.4), shows that in the hemispherical configuration, the interconnects (with an initial length of 19 \( \mu m \)) experience a maximum in-plane normal strain (\( \varepsilon_{xx} \)) of 345.5\% (no 17), a maximum in-plane shear strain (\( \varepsilon_{xy} \)) of 134.3\% (no 27), out-of-plane shear strain (\( \varepsilon_{xz} \)) of 5\% (no 21), \( \theta_z \) of -7.7\(^\circ\) (no 27), \( \theta_y \) of -18.2\(^\circ\) (no 17) and \( \theta_x \) of 14.6\(^\circ\) (no 30) with respect to the local coordinate axes. The substrate has a diameter of 1 mm, with the rigid cubic islands of size 50 \( \times \) 50 \( \times \) 50 \( \mu m^3 \) and a spacing between adjacent islands of 19 \( \mu m \).
3.2 Multiaxial elastic stretchability of typical freestanding interconnect geometries

The three freestanding interconnect geometries considered here are shown in Figure 3.2 and are compared here on the basis of their elastic multiaxial stretchability performance. All three designs have a planar geometry to comply with the requirement of manufacturability with conventional microelectronics fabrication by means of layer by layer deposition and selective removal. The first choice (see Figure 3.2a) is the serpentine interconnect which is a well-known example of a conventional interconnect structure that has been extensively studied in the stretchable electronics literature [6, 17, 57]. Due to its freestanding nature and its larger in-plane cross-sectional width compared to its out-of-plane thickness, the interconnect is designed to buckle out-of-plane upon uniaxial loading as reported in Refs. [30, 58]. In these studies it has been shown that due to buckling and subsequent complex deformation consisting of a superposition of torsion and bending, the interconnect design can stretch more compared to its elastomer-embedded equivalent configuration which is restricted to only in-plane deformation [30, 58].

The second geometry considered is the Rotation Out-of-Plane Elongation (ROPE) interconnect (see Figure 3.2b), which was demonstrated to have a uniaxial elastic stretchability of 2000%, as discussed in Ref. [55]. This interconnect also has an initially planar configuration to enable fabrication by conventional thin-film processing techniques. Upon loading the structures buckles out-of-plane, aligning the thin dimension of the slender beams with the loading direction, as illustrated in Figure 3.3. While stretching, the central beams act as bending beams, while the corner beams act as torsion beams.
Figure 3.3: Schematic of the working mechanism of the ROPE interconnect. (Left) The planar interconnect structure in its initial state. (Right) The structure buckles out-of-plane after being loaded, with the central beams bending to provide the stretch and the corner torsion beams twisting to align the thin dimension of the bending beams with the loading direction [55].

The third geometry considered is shown in Figure 3.2c and referred here as the In-Plane Elongation (IPE) interconnect. This is a non-buckling freestanding interconnect, where in the initial configuration, the slender beams have a small (in-plane) width compared to a larger out-of-plane thickness. Hence, the slender beams are already aligned with the loading axis in their initial configuration. As can already be expected, this geometry appears to be ideal for a maximum in-plane stretching due to its compact footprint or, equivalently, a maximum number of beams within the footprint area. While this seems to be an ideal geometry, the fabrication of such structures using conventional thin-film processes is very challenging as deposition of thick films is not trivial. Furthermore, subsequent well-controlled uniform etching over a large out-of-plane thickness is also very difficult, typically resulting in undercuts and a variable cross-sectional width. Nevertheless, such structures have been produced successfully and tested, resulting in large stretchabilities [36, 56, 59–61]. Therefore, this structure is included here to assess its multiaxial stretchability performance.

The performance of the interconnects is compared based on the plastic deformation in the interconnects upon stretching, since that directly relates to their life-time. In experiments, the plastic deformation in such (sub)-micron sized, high compliant structures is difficult to quantitatively characterize due to the required force resolution in the range of nano-Newtons. One way to determine the onset of plasticity is to observe the shape change in the interconnect with respect to the original configuration after being unloaded from a certain applied stretch [55]. In order to enable a comparison with experiments, the same criterion for identifying the onset of plasticity must be used in the simulations, i.e., a shape change (maximum displacement) $\sim 300$–$500$ nm after unloading to the initial configuration is used as a criteria for all the interconnects. Three loading cases are applied for each of the interconnects, namely loading in the $xy$, $xz$, and $yz$- Cartesian planes. In each plane, the interconnects were loaded along $0^\circ$, $15^\circ$, $30^\circ$, $45^\circ$, $60^\circ$, $75^\circ$ and $90^\circ$ degrees. Upon subsequent unloading to the original configuration, when the shape change corresponded to a displacement of 300-500 nm, the applied stretch (hereon referred to as global strain) was marked as a limit point in the corresponding limit curve. This applied global strain along any axis was calculated as
the applied displacement along this axis divided by the interconnect footprint width, i.e., 19 \( \mu m \). Moreover, self-contact was taken as a secondary failure criterion, i.e., as soon as the interconnect makes contact with itself, it is marked as a limiting case in the limit curves. This is due to the fact that at the (sub)-micron scales, the strong adhesive interactions at contact immediately lead to stiction of the structures and ultimately failure upon reloading [62].

Additionally, all three interconnects have the same cross-sectional area (see Figure 3.2). Both the serpentine and the ROPE interconnect have an in-plane cross-sectional width of 2 \( \mu m \) and an out-of-plane cross-sectional thickness of 0.3 \( \mu m \). These values are based on the minimum dimensions that achievable in with the microfabrication scheme used to fabricate the ROPE interconnect samples (as discussed in Ref. [55]). For the IPE interconnect, the aspect ratio of the cross-sectional area is inverted with respect to the other two interconnects, with an in-plane width of 0.3 \( \mu m \) and an out-of-plane thickness of 2 \( \mu m \). Note that these dimensions are not possible with the particular microfabrication technology available to us, therefore the IPE interconnects could not be experimentally tested. Nevertheless, these values are assumed in the Finite Element (FE) analysis for consistency of the numerical comparison. For consistency, the minimum gap between any two beams possible, i.e., 1 \( \mu m \), is used here for both the ROPE and the IPE interconnect. The same value is used for the minimum gap between the interconnect and the attachment point with the substrate at both ends. The width of the foot-print area (illustrated in Figure 3.3) is set to 19 \( \mu m \), resulting in 6 and 12 beams for the ROPE and the IPE interconnect, respectively. For the same footprint width, the serpentine interconnect can reach a maximum in-plane height of only 25.8 \( \mu m \) (compared to 50 \( \mu m \) for the ROPE and the IPE interconnects), resulting in a handicap, which is discussed in the results section.

All numerical simulations were performed using a commercial FE package (MARC Mentat). A non-linear, large displacement formulation was used to capture the non-linearities due to buckling and subsequent large deformation. 20-node, quadratic, solid brick elements were used with at least three elements through the out-of-plane thickness to capture the bending deformation properly. In order to allow the onset of buckling in the serpentine and the ROPE interconnects, a small imperfection geometrical or perturbation was needed. Here that was done by lifting up one of the clamping ends of the interconnect out-of-plane initially, by a value that is equivalent to the thickness of the interconnects, i.e., 0.3 \( \mu m \). Subsequently, after buckling, this imposed out-of-plane displacement was gradually removed, well before the elastic limit was reached. Furthermore, an adaptive time-stepping routine, with an initially small time step, was used to capture the buckling bifurcation. All interconnects were modelled with the material properties of the microfabricated ROPE interconnect sample material, i.e., pure aluminium, with a Young’s modulus of 69 GPa and a Poisson’s ratio of 0.33. A yield strength of 600 MPa was assumed here as estimated in Ref. [55] from uniaxial stretch tests of the ROPE interconnect samples, where the large increase of the yield stress with respect to bulk aluminium was attributed to mechanical size effects. An isotropic hardening model with a power-law hardening relationship (see eq. 3.1) was assumed, where \( \sigma_y \) is the yield strength, \( \varepsilon_0 \) is the strain corresponding to the initial yield strength, \( \bar{\varepsilon} \) is the equivalent plastic strain, and K and n are the strength and strain hardening coefficients,
respectively. Here a strain hardening coefficient of 0.2 was assumed, as common for alu-
minium. In order to detect the moment of contact between the different beam members,
a permanently glued type contact interaction was included for all the interconnects.

\[ \sigma_y = K(\epsilon_0 + \bar{\epsilon})^n. \] (3.1)

Figure 3.4 shows the limit curves of all three interconnect geometries in the xy-plane. Both the serpentine and the ROPE interconnects have a relatively ‘isotropic’ limit curve shape, i.e., a relatively small variation of the elastic limit with changing load angle in the xy-plane. In contrast, the IPE interconnect (shown in Figure 3.4f) can withstand higher global strains for 0° and 15° loading angles, however beyond that loading angle the maximum global strain drops to well below that of the ROPE interconnect and even below that of the serpentine interconnect. This is due to the fact that at a 30° loading angle and beyond, the IPE interconnect beams make contact with each other (shown in Figure 3.4d), which limits its application. The ROPE and the serpentine interconnect have a similar limiting curve shape, as the stretchability results from out-of-plane buckling and subsequent elongation. In case of the ROPE interconnect, for all angles, the interconnect can buckle such as to align the bending beams favourably with the loading direction, as seen in Figure 3.4e for 0° loading, Figure 3.4b for 45° loading and Figure 3.4a for 90° in the xy-plane. However, for the 0° and 15° degrees angles, the magnitude of the global strain for the ROPE interconnect is approximately 3 times less than for the IPE interconnect, since due to its initial planar configuration the ROPE interconnect has only 6 beams, of which only 4 inner beams that fully contribute to the in-plane bending. The IPE interconnect instead has 12 beams which all contribute to in-plane bending. Similar to the ROPE interconnect, the serpentine interconnect can buckle out-of-plane for each loading angle and exhibits an approximately equal global strain for all angles. However, the magnitude of the global strain is approximately 10 times smaller than for the ROPE interconnect due to its non-optimal geometrical shape.

The global strains that would be required by the interconnects bridging the detector islands in the balloon detector simulation (shown in Figure 3.1) have been plotted in the xy-plane limiting curve (open green circles in Figure 3.4). This allows to compare the performance of each of the three interconnect geometries against the requirements in a typical stretchable electronics application. For the balloon detector strain values shown in Figure 3.4, a projection of the 3D strain values is taken by ignoring the out-of-plane shear strain component \( \varepsilon_{xz} \) which is also negligible here, as shown in Table 3.1 (in 3.4). The results show that the serpentine design fall short significantly for all 30 interconnects in the balloon detector. The ROPE design, on the other hand meets the requirement of all 30 interconnects. The IPE design, while meeting the requirement of most of the interconnects by a big margin, clearly fails to provide the required global strain for 8 interconnects due to the higher loading angle required. It should be noted that these 8 interconnects (no 11, 12, 24, 26, 27, 28, 29 and 30) correspond to the islands at the periphery of the detector, which provide the essential high angle detector information.

The limit curves for the yz-plane and xz-plane loading are shown in Figure 3.5 and Figure 3.6, respectively. The serpentine and the ROPE interconnect show a similar yield curve shape, with the magnitude of the serpentine interconnect global strains being considerably smaller than the ROPE interconnect global strains. Both interconnect
Figure 3.4: (Top left) limit curves plotted for the serpentine, ROPE and IPE interconnects in the xy-plane at 7 different loading angles, along with snapshots of the interconnects at selected limit points marked by black lettering (a-f) in the limit curves. The experimental results for the ROPE interconnect at two different loading angles have also been plotted (with blue filled circles). Furthermore, the global strain values required by the interconnects of the balloon detector simulation (shown in Figure 3.1) are plotted (with green unfilled circles) on top of the limit curves. The sub-figures (a-f) show the out-of-plane displacement field $u_z$ in the deformed configuration of the particular interconnect, for: (c) the serpentine interconnect at 0° loading, (a, b, e) ROPE interconnect at (e) 0°, (b) 45° and (a) 90° loading and (d, f) the IPE interconnect at (f) 0° and 30° loading.
Figure 3.5: (Top left) limit curves plotted for the serpentine, ROPE and IPE interconnects in the yz-plane at 7 different loading angles, along with snapshots of the interconnects at selected limit points marked by black lettering in the limit curves. The experimental results for the ROPE interconnect at two different loading angles have been plotted with blue filled circles. Furthermore, the global strain values required by interconnects in the balloon detector simulation (shown in Figure 3.1) are plotted (with green unfilled circles) on top of the limit curves. The sub-figures (a-c) show the out-of-plane displacement field $u_z$ plotted on top of the deformed configuration of the particular interconnect, with: (a) ROPE interconnect at 45° loading, (d) IPE interconnect at 45° loading and insets showing parts of the interconnect making self-contact and (c) serpentine interconnect at 45° loading.
exhibit an elliptical shape, with the major ellipse axis aligned along the 90° loading angle, because both interconnects have their thin cross-sectional direction optimally aligned for loading along the z-axis. In contrast, for yz-plane loading at all loading angles, the IPE interconnect results in self-contact at very low global strain values (as shown in Figure 3.5b) making it infeasible for loading in the yz-plane. Loading of the IPE interconnect in the xz-plane results in self-contact above 60° loading angle.

3.3 Multiaxial experimental testing of the rope interconnect

Experimental results of multiaxial loading ROPE interconnect are presented here in order to compare the deformation of real microfabricated samples against the multiaxial simulations presented in the previous section. All samples have a height of 50 µm, beam width of 2 µm, thickness of 0.3 µm and a gap (in-between any two beams) of 1 µm, as seen in Figure 3.7 and identical to the dimensions used for the ROPE interconnect simulations (shown in Figure 3.2). Unfortunately, only samples with 8 or 10 beams (instead of 6 beams as used in the simulations) were available. However, as samples with more beam members have an equivalently larger footprint width, to the first degree, the global strain values should not be affected. FEM simulations of 6, 8 and 10 beam member interconnect geometries show a global strain of 454.4%, 492% and 516%. Thus, a maximum increase in global strain of ~19% for each added beam member. Details on the test setup and the experimental methodology are explained in the 3.4.

The microfabricated ROPE samples were tested for three loading cases: 1) in-plane normal extension, i.e., \( \varepsilon_{xx} \) (see Figure 3.7a), 2) in-plane shear, i.e., \( \varepsilon_{xy} \) (see Figure 3.7b) and out-of-plane shear, i.e., \( \varepsilon_{xz} \) (see Figure 3.7c). During the experiments, the samples were incrementally loaded to increasing values of applied global strain and subsequently unloaded to the original configuration to assess if any permanent shape change could be visualized, which would indicate the onset of plasticity. As seen in Figure 3.7a, for \( \varepsilon_{xx} \) loading, the sample exhibits signs of plastic deformation after a global strain of 474.6%, which is in adequate agreement with the FE simulations (see Figure 3.4). Similarly, for in-plane shear (\( \varepsilon_{xy} \)), the sample can conveniently buckle out-of-plane and attain the configuration predicted by the FE simulations (see Figure 3.4a) leading to a high global strain of 435.2%. To visualize the deformation in the out-of-plane shear case (\( \varepsilon_{xz} \)), the sample stage of the SEM was tilted by 5°. The experiment shows that the interconnect can sustain a global strain of 708%, before plastic deformation occurs. This is again in good agreement with the FE simulations shown in Figure 3.5 and Figure 3.6. In conclusion, the experiments confirm the predictions of the FE simulations for the ROPE interconnect, validating that this interconnect type can be omnidirectionally stretched in the xyz Cartesian space to relatively high values of global strain.

3.4 Discussion and conclusions

Typical advanced stretchable electronics applications such as high-density smart patches and balloon detectors, etc., require interconnect which can not only provide high in-plane normal global strains but also significant in-plane and out-of-plane global shear strains...
Figure 3.6: (Top left) limit curves plotted for the serpentine, ROPE and IPE interconnects in the xz-plane at 7 different loading angles, along with snapshots of the interconnects at selected limit points marked by black lettering in the limit curves. The experimental results for the ROPE interconnect at two different loading angles have also been plotted (with blue filled circles). Furthermore, the global strain values required by interconnects in the balloon detector simulation (shown in Figure 3.1) are plotted (with green unfilled circles) on top of the limit curves. The sub-figures (a-d) show the out-of-plane displacement field $u_z$ plotted on top of the deformed configuration of the particular interconnect, for: (c) serpentine interconnect at 75° loading, (b) ROPE interconnect at 75° loading and (a, d) IPE interconnect at (a) 60° and (d) 75° loading and inset showing parts of the interconnect making self-contact.
Figure 3.7: Experimental results for multiaxial loading of the microfabricated ROPE interconnect with the samples being cyclically loaded-unloaded to increasingly higher strains to study the onset of plasticity. Note that in sub-figure (b) the greyed-out areas indicate the neighboring interconnects. (a) Sample loaded in normal in-plane extension along the x-axis ($\varepsilon_{xx}$). (b) In-plane shear ($\varepsilon_{xy}$) along the y-axis and (c) out-of-plane shear $\varepsilon_{xz}$ along the z-axis. Note that for the z-loading, the SEM stage was tilted by 5° around the y-axis to better visualize the out-of-plane displacement.
during regular operation. Moreover, these devices inevitably experience unwanted vibrations during handling, which due to their desired compliant natures would trigger complex multiaxial deformations including in-plane and out-of-plane shear deformation to the interconnects. In this paper, the multiaxial loading performance of three typical freestanding interconnect geometries has been studied in this context. The three geometries are: 1) the serpentine interconnect which is a buckling type interconnect and it is the conventional interconnect geometry studied most in the literature, 2) the ROPE interconnect which is also a buckling type interconnect with a very high stretchability [55] and 3) the IPE interconnect which is a non-buckling type interconnect, designed to have a very compact shape with the maximum number of beam members within a specific footprint area.

Multiaxial FE simulations of the traditional serpentine interconnect show promising results in the respect that the interconnect can easily buckle out-of-plane for all loading angles, exhibiting a nearly isotropic yield curve in xy-, xz-, and yz- loading planes. However, the typical magnitude of the global strains for the serpentine geometry is relatively low (~40%) and almost an order of magnitude smaller than the required values for the balloon detector application presented. The limited global strain magnitude of the serpentine interconnect originates from its spatial configuration, containing a footprint length that is only half of the length of the other two geometries with the same footprint width. Clearly the footprint length has a large influence on the global strains that can be achieved in all three interconnect choices. Furthermore, due to its inefficient shape, only one repetition of the serpentine fits in the prescribed footprint width, compared to the multiple repetition of the unit cells in the ROPE and the IPE interconnect geometry. Consequently, with only a single meander, the whole serpentine structure deforms as a combination of torsion and bending. This results in a higher stiffness of the structure in the loading direction compared to, e.g., a serpentine structure with multiple repetitions, where the torsion for the out-of-plane rotation of the whole structure is taken up by the corner sections of the structure, while the inner meanders would be relatively torsion free and consequently more compliant, resulting in a higher bending deflection of the inner meanders and higher global strains. The distinction between torsion and bending is even more apparent in the case of the ROPE interconnect where only the corner beams provide torsion, whereas the inner beams provide bending. Furthermore, even though the large radius of curvature of the serpentine structure results in a lower stress concentration compared to the ROPE and the IPE interconnects, the overall improvement on the global elastic strain is greatly diminished due to its increased footprint and thus initial length. This is illustrated in Figure 3.10 (in the Appendix section), where for both structures having the same height of 50 $\mu m$, the serpentine interconnect needs to displace by 14.2 $\mu m$ to result in a global strain of 75%, while the ROPE interconnect only needs to displace over 4.5 $\mu m$ for the same global strain. Consequently, at the same global strain, the serpentine interconnect exhibits approximately an order of magnitude higher von Mises stress compared to the ROPE interconnect. Additionally, for the loading conditions in Figure 3.10, which mimics deformation of the inner (bending) members (providing the major contribution to the global strain), the ROPE interconnect has an ~80% lower stiffness in the loading direction compared to the serpentine interconnect. Based on these results it is concluded that the serpentine geometry, while
allowing for omnidirectional stretchability, does not result in sufficiently high global strains as required in advanced high-density stretchable electronics applications.

In contrast, the IPE interconnect at first appears to be an ideal geometry to achieve very high interconnect global strains, due to the thin direction of the slender beams aligned with the loading direction along with its initially small footprint width. FE simulations have shown that the interconnect provides large global strains in the normal opening direction ($\varepsilon_{xx}$), yet as soon as the in-plane shear component ($\varepsilon_{xy}$) increases with a corresponding loading angle beyond 15° in the xy-plane, the interconnect makes self-contact at very low global strains, which is highly undesired at the nm/µm scale as it typically leads to permanent adhesion and ultimately failure. The tendency of the structure to trigger self-contact under shear is caused by the fact the long beams of the IPE interconnect can only rotate in the plane to provide the $\varepsilon_{xy}$ component of the global strain while the central beams, being less constrained than the corner beams, rotate more resulting in contact with the corner beams. Moreover, loading in the yz-plane leads to contact at all loading angles at very small values of applied global strains. Increasing the gap between the beams in the IPE interconnect only leads to a modest increase in the global strains at which self-contact takes place during shear. An FE simulation with the gap increased from 1 µm to 2.56 µm, resulting in 6 beams (with the original beam cross-section) that fit in the original footprint of 19 µm, yields in an increase of approximately two times in global strain (i.e., $\varepsilon_{xx} = 20\%$ and $\varepsilon_{xy} = 11.5\%$) at which contact takes place for a loading angle of 30° in the xy-plane. This value is still far too small for a typical application such as the balloon detector. Note that for this increased gap the originally high global strain at 0° and 15° loading angles would approximately reduce to half as only half of the original number of beams now contribute to the opening. Furthermore, it should be emphasized that while there have been some interconnect structures reported in literature with high aspect ratios and a high thickness, fabricating such a structure on the small scales using traditional thin film process is not trivial and can be very challenging. Therefore, it is recommended that, while the IPE geometry might be ideal for applications with very well-defined loading conditions that are restricted to uniaxial in-plane opening, it should be avoided in applications where even minute shear strains are to be expected that would lead to self-contact, and a high probability for device failure. Alternatively, at a larger scale where self-contact is typically not a concern, if the interconnects are well insulated, this design can still be considered as long as it can be feasibly produced.

Finally, the ROPE interconnect FE simulations demonstrated that this geometry exhibits a ten times higher global strains than the serpentine interconnect. Yet in normal extension it has a three times lower global strain than the maximum global strains sustained by the IPE interconnect. In order to provide stretchability, the initially planar interconnect buckles out of plane and stretches by bending when loaded in the uniaxial in-plane opening direction. For in-plane shear, the structure can buckle out of plane resulting in almost the same magnitude of global stain for $\varepsilon_{xy}$. For the loading along the out-of-plane z axis, the beams are already ideally aligned for stretching without the need for buckling. Importantly, the structure can buckle and stretch in any direction in the xy-, xz- and yz- planes without making self-contact, thus constituting an omnidirectionally stretchable interconnect, with a high stretchability in all directions. However, the buckling does result in the long beams sticking out-of-plane by up to half of the
beam length, which should be taken into consideration in the device design. Finally, multiaxial experiments were performed on microfabricated ROPE interconnect samples for the case of $\varepsilon_{xx}$, $\varepsilon_{xy}$ and $\varepsilon_{xz}$ global strains and the results adequately match with the corresponding FE simulations.

Acknowledgements

This work was supported by the Vidi funding of J.H. (project number 12966) within the Netherlands Organization for Scientific Research (NWO).

Determination of 3D Strains and rotations

Due to large 3D displacements and rotations experienced by the detector islands of the inflatable detector array shown in Figure 3.1, that calculation of strains and rotations at the level of the interconnects connecting the islands is not trivial. Here, the calculation of these strains and rotation angles between two such islands, as shown in Figure 3.8 (with a 2D projection of deformation for ease of illustration), is presented.

![Figure 3.8: Schematic showing the displaced and rotated islands with reference to Figure 3.1 with the corresponding displacement vectors and the local coordinate axes used for calculating the strains and the rotation angles.](image)

The in-plane normal linear strain (under large shear strains and rotations), $\varepsilon_{xx}$, is calculated as:

$$\varepsilon_{xx} = \frac{\vec{l}_{ab} \cdot \vec{m}_x}{\|\vec{l}_o\|} - 1,$$

(3.2)
where, $\vec{l}_{ab}$ is the displacement vector between the two displaced and rotated islands, $\vec{m}_x$ is the normal vector of the mid-plane and $\|\vec{l}_0\|$ is the initial gap between the two islands. The in-plane shear strain, $\varepsilon_{xy}$, is given by:

$$\varepsilon_{xy} = \frac{\vec{u}_{ab} \cdot \vec{m}_y}{\|\vec{l}_0\|}, \quad (3.3)$$

and the out-of-plane shear strain, $\varepsilon_{xz}$, is given by:

$$\varepsilon_{xz} = \frac{\vec{u}_{ab} \cdot \vec{m}_z}{\|\vec{l}_0\|}. \quad (3.4)$$

The rotation angles between the two islands a and b are denoted by $\theta_x$, $\theta_y$ and $\theta_z$ and are the rotations of the local Cartesian coordinate axes $\vec{b}_x$, $\vec{b}_y$ & $\vec{b}_z$ of the displaced and rotated island b with respect to the local Cartesian coordinate axes $\vec{a}_x$, $\vec{a}_y$ & $\vec{a}_z$ of the displaced and rotated island a. The rotation angles are extracted as intrinsic Euler angles in the order of $\theta_z$, $\theta_y$ and $\theta_x$ from the rotation matrix of the local coordinate systems of the two islands. The rotation matrix $R$ provides the transformation of the local Cartesian coordinate axes of island b $(\vec{b}_x, \vec{b}_y, \vec{b}_z)$ to the local Cartesian coordinate axes of island a $(\vec{a}_x, \vec{a}_y, \vec{a}_z)$, i.e:

$$A = RB^T \quad (3.5)$$

where, $A$ and $B$ are matrices whose column vectors are the local Cartesian coordinates of island a, i.e., $(\vec{a}_x, \vec{a}_y, \vec{a}_z)$ and the local Cartesian coordinates at island b, i.e., $(\vec{b}_x, \vec{b}_y, \vec{b}_z)$, respectively. The rotation matrix $R$ is determined as [63]:

$$R = \begin{bmatrix} \vec{a}_x \cdot \vec{b}_x & \vec{a}_x \cdot \vec{b}_y & \vec{a}_x \cdot \vec{b}_z \\ \vec{a}_y \cdot \vec{b}_x & \vec{a}_y \cdot \vec{b}_y & \vec{a}_y \cdot \vec{b}_z \\ \vec{a}_z \cdot \vec{b}_x & \vec{a}_z \cdot \vec{b}_y & \vec{a}_z \cdot \vec{b}_z \end{bmatrix}. \quad (3.6)$$
Table 3.1: Calculated values of relative displacements and relative rotations between adjacent islands in Figure 3.1. These displacements and rotations result in the loading of the interconnects between adjacent islands. The maximum values for each column are in red text color.

<table>
<thead>
<tr>
<th>interconnect no</th>
<th>( \varepsilon_{xx} ) [%]</th>
<th>( \varepsilon_{xy} ) [%]</th>
<th>( \varepsilon_{xz} ) [%]</th>
<th>( \theta_z ) (deg)</th>
<th>( \theta_y ) (deg)</th>
<th>( \theta_x ) (deg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>241.8</td>
<td>1.5</td>
<td>-0.3</td>
<td>0.5</td>
<td>-10.4</td>
<td>0.0</td>
</tr>
<tr>
<td>2</td>
<td>248.4</td>
<td>3.3</td>
<td>-0.6</td>
<td>0.5</td>
<td>-11.1</td>
<td>0.0</td>
</tr>
<tr>
<td>3</td>
<td>259.5</td>
<td>5.6</td>
<td>-0.9</td>
<td>0.5</td>
<td>-12.3</td>
<td>0.1</td>
</tr>
<tr>
<td>4</td>
<td>275.3</td>
<td>8.1</td>
<td>-1.1</td>
<td>0.5</td>
<td>-13.9</td>
<td>0.2</td>
</tr>
<tr>
<td>5</td>
<td>295.1</td>
<td>14.9</td>
<td>-1.0</td>
<td>0.3</td>
<td>-15.8</td>
<td>0.7</td>
</tr>
<tr>
<td>6</td>
<td>311.0</td>
<td>30.0</td>
<td>-0.4</td>
<td>0.1</td>
<td>-17.2</td>
<td>2.4</td>
</tr>
<tr>
<td>7</td>
<td>235.3</td>
<td>4.8</td>
<td>0.3</td>
<td>-1.4</td>
<td>0.0</td>
<td>10.3</td>
</tr>
<tr>
<td>8</td>
<td>222.3</td>
<td>8.8</td>
<td>0.4</td>
<td>-2.4</td>
<td>0.0</td>
<td>10.1</td>
</tr>
<tr>
<td>9</td>
<td>202.6</td>
<td>13.5</td>
<td>0.4</td>
<td>-3.5</td>
<td>-0.1</td>
<td>9.9</td>
</tr>
<tr>
<td>10</td>
<td>172.8</td>
<td>20.0</td>
<td>0.2</td>
<td>-4.6</td>
<td>-0.4</td>
<td>9.3</td>
</tr>
<tr>
<td>11</td>
<td>127.9</td>
<td>36.7</td>
<td>-1.0</td>
<td>-5.6</td>
<td>-1.3</td>
<td>7.8</td>
</tr>
<tr>
<td>12</td>
<td>88.7</td>
<td>66.6</td>
<td>-1.6</td>
<td>-7.0</td>
<td>-2.6</td>
<td>8.7</td>
</tr>
<tr>
<td>13</td>
<td>242.1</td>
<td>10.3</td>
<td>-0.7</td>
<td>1.5</td>
<td>-11.1</td>
<td>0.1</td>
</tr>
<tr>
<td>14</td>
<td>253.1</td>
<td>17.4</td>
<td>-0.9</td>
<td>1.6</td>
<td>-12.3</td>
<td>0.4</td>
</tr>
<tr>
<td>15</td>
<td>209.9</td>
<td>23.2</td>
<td>-1.3</td>
<td>1.8</td>
<td>-14.0</td>
<td>0.5</td>
</tr>
<tr>
<td>16</td>
<td>297.7</td>
<td>26.7</td>
<td>-1.6</td>
<td>1.9</td>
<td>-16.5</td>
<td>0.3</td>
</tr>
<tr>
<td>17</td>
<td>345.5</td>
<td>66.7</td>
<td>-0.1</td>
<td>0.7</td>
<td>-18.2</td>
<td>5.1</td>
</tr>
<tr>
<td>18</td>
<td>229.1</td>
<td>18.7</td>
<td>0.7</td>
<td>-2.6</td>
<td>-0.4</td>
<td>11.0</td>
</tr>
<tr>
<td>19</td>
<td>211.8</td>
<td>26.8</td>
<td>1.1</td>
<td>-3.8</td>
<td>-0.6</td>
<td>11.0</td>
</tr>
<tr>
<td>20</td>
<td>187.2</td>
<td>30.6</td>
<td>2.0</td>
<td>-5.5</td>
<td>-0.7</td>
<td>11.1</td>
</tr>
<tr>
<td>21</td>
<td>153.7</td>
<td>11.9</td>
<td>5.0</td>
<td>-7.5</td>
<td>0.4</td>
<td>12.4</td>
</tr>
<tr>
<td>22</td>
<td>238.3</td>
<td>34.2</td>
<td>-0.8</td>
<td>2.6</td>
<td>-12.2</td>
<td>0.9</td>
</tr>
<tr>
<td>23</td>
<td>249.0</td>
<td>49.0</td>
<td>-1.1</td>
<td>2.6</td>
<td>-13.8</td>
<td>1.6</td>
</tr>
<tr>
<td>24</td>
<td>254.1</td>
<td>73.2</td>
<td>-0.6</td>
<td>2.6</td>
<td>-15.0</td>
<td>3.5</td>
</tr>
<tr>
<td>25</td>
<td>223.2</td>
<td>55.8</td>
<td>0.8</td>
<td>-4.2</td>
<td>-1.8</td>
<td>12.4</td>
</tr>
<tr>
<td>26</td>
<td>195.5</td>
<td>85.0</td>
<td>-0.2</td>
<td>-5.5</td>
<td>-3.0</td>
<td>12.6</td>
</tr>
<tr>
<td>27</td>
<td>176.4</td>
<td>134.3</td>
<td>-1.7</td>
<td>-7.7</td>
<td>-5.3</td>
<td>14.5</td>
</tr>
<tr>
<td>28</td>
<td>239.1</td>
<td>75.7</td>
<td>-1.7</td>
<td>4.2</td>
<td>-14.2</td>
<td>2.9</td>
</tr>
<tr>
<td>29</td>
<td>272.6</td>
<td>132.6</td>
<td>-1.0</td>
<td>3.4</td>
<td>-16.3</td>
<td>7.0</td>
</tr>
<tr>
<td>30</td>
<td>212.3</td>
<td>106.0</td>
<td>2.7</td>
<td>-6.7</td>
<td>-4.4</td>
<td>14.6</td>
</tr>
</tbody>
</table>

Experimental Testing Methodology

The ROPE interconnect samples were processed from pure Al, on top of a silicon test chip shown in Figure 3.9. The test chip essentially consists of two rectangular silicon clamping islands for fixation on a tensile stage. Six parallel freestanding interconnects of the same dimensions are situated at the centre of the test chip, i.e., in a single test six samples are tested in parallel. Initially, during fabrication and handling, up to the
point of actuation, the test chip is fixed rigidly with the help of sacrificial Si beams connecting the two Si clamping islands at the top and bottom, to ensure the integrity of the samples and to avoid any preloading. Each sacrificial Si beam has three notches, where the beam is locally thinned down in the in-plane and out-of-plane directions. Once the test chip has been clamped, the notches allow the sacrificial beam trigger localized stresses requiring only a minute (point) force at the central notch of each beam to break it with negligible energy. Furthermore, the test chip has been designed in order to allow multiaxial loading in the x, y and z directions. A detailed description of the microfabrication steps for the test chip can be found in Ref [64]. A home-built multiaxial micro tensile stage shown in Figure 3.9 is used to apply actuation to the samples. The stage is based on two commercially acquired closed-loop piezo stages, each formed by the stacking of x, y and z platforms, with a range of 200 $\mu$m and a resolution of $\sim 1$ nm along each of the three directions. Clamps added on top of the piezo stages allow to achieve the desired alignment of the samples with respect to the loading axes. The test chip is clamped by gluing it to removable insets fixed in the clamps, such as to keep the top easily accessible, e.g., for high resolution (low working distance) optical microscopy. A LABVIEW based GUI was setup to apply displacement controlled loading in the xyz Cartesian space.

**Comparison of Serpentine and ROPE interconnects**

The ROPE interconnect geometry is compared against the serpentine interconnect geometry by loading a section of each geometry in the out-of-plane direction, reflecting the bending mode (which provides stretchability), in these interconnects as shown in Figure 3.10. Both structures have the same beam cross-sectional area (i.e., $2 \mu m \times 0.3 \mu m$) and the same height (i.e., 50 $\mu m$). The interconnects are loaded to the same global strain of 75%, which entails a global displacement of 4.5 $\mu m$ for the ROPE interconnect, while the serpentine interconnects, due to its larger footprint width requires a global displacement of 14.2 $\mu m$ to reach the same global strain. In Figure 3.11, the reaction forces at the prescribed nodes are summed up and plotted against the prescribed displacement to evaluate the stiffness of each interconnect in the loading direction. The $\sim 5$ times higher stiffness of the serpentine interconnect (due to its suboptimal; geometry) and its higher footprint area compared to the ROPE interconnect result in the serpentine interconnect having almost an order of magnitude higher von-Mises stresses compared to the ROPE interconnect at the same applied global strains as shown in Figure 3.10.
Figure 3.9: Schematic of test setup showing: (a) multiaxial tensile tester consisting of two xyz closed-loop piezoelectric stages, (b) an illustration of the test chip with the sacrificial Si beams that can be broken with a minute force before loading the interconnect samples, and on its right a magnified view of the six parallel interconnects at the centre of the test chip and (c) SEM micrograph of a ROPE interconnect sample.
Chapter 3.

Figure 3.10: The serpentine and the ROPE interconnect loading in the out-of-plane z direction at the same global strain of 75%. von Mises stress ($\sigma_{vm}$) plotted on the deformed geometry. Both interconnect have the same length (i.e., 50 $\mu m$) and the same cross-sectional area.

Figure 3.11: Reaction force in the z direction vs. displacement in z direction for the serpentine and the ROPE interconnect loaded in z direction (Figure S 3). The stiffness of the structures in the z direction is estimated with a linear fit.
Part II

Experimental characterization techniques
Cool, dry, straightforward, sub-micron nebulization, DIC patterning of delicate, heterogeneous, non-planar specimens

Abstract

Application of patterns to enable Digital Image Correlation (DIC) at the small-scale (µm/nm) is known to be very challenging as techniques developed for the macro-scale such as spray painting or screen printing, etc. cannot be scaled down directly. Furthermore, the often delicate nature (physical and/ chemical) of the specimens that need to be patterned puts various constraints on the chemicals, physical steps, temperatures, etc. such samples can be exposed to for patterning without damaging them. Herein, a technique specially developed for patterning highly delicate samples is presented. The technique consists in a well-controlled nebulized micro-mist, containing predominantly no more than one nanoparticle per mist droplet, which is dried, resulting in a flow of individual nanoparticles that are subsequently deposited on the sample surface at near-room temperature. By having single nanoparticles falling on the sample surface, the notoriously challenging task of controlling nanoparticle-nanoparticle and nanoparticle-surface interactions as a result of the complex droplet drying dynamics, e.g., in drop-casting, is circumvented. The technique is used here to deposit a number of challenging cases of physically and chemically delicate samples with nanoparticles from 1 µm down to 50 nm in diameter. While the technique has an upper limit of particle size (∼1 µm), due to the maximum droplet size, there is strictly no lower limit to the smallest particle size it should be possible to deposit particles sizes below 50 nm. Thus, the technique is easily scalable within this range, which is of special interest for micromechanical testing using imaging techniques such as, high-magnification optical microscopy, optical profilometry, atomic force microscopy, and scanning electron microscopy etc. In short, the (delicate) samples can be patterned at a near-room temperature (∼37 °C), without exposure to chemicals or physical contact, while the pattern density can be easily tuned by changing the pattern application duration.

4.1 Introduction

During the last few decades Digital Image Correlation (DIC) has emerged as a leading technique for measuring shape, displacement and deformation due to its non-contact
nature, high (sub-pixel) accuracy [65], applicability with a wide range of imaging setups from cheap webcam base image acquisition [66] to state-of-the-art microscopy techniques, such as High Resolution Transmission Electron Microscope (HRTEM) [67] and applications ranging from health-monitoring of megastructures [68] down to atomic scales for studying the deformation fields resulting from electrochemical reactions [67]. Recently, there has been increasing interest in integrating DIC with high resolution microscopy techniques such as Atomic Force Microscopy (AFM) [69, 70] and especially Scanning Electron Microscopy (SEM) [71–74] to understand micromechanical deformation mechanisms as well as performing accurate meteorology on micron-sized structures, such as micro-electromechanical systems (MEMS) [75], microelectronics, etc. However, one of the most important requirements for the successful application of DIC is the requirement of an (ideally speckle-like) dense, non-continuous pattern on the specimen surface, which the DIC algorithm can effectively track, either globally (in the case of global-DIC [76]) over the whole field of view or locally by dividing the image into smaller subsets (as in local-DIC [65]) to provide the displacement fields. While numerous uncomplicated techniques exist in literature to create high quality patterns at the meso and macro scales, achieving a similar pattern quality at the micron scale remains challenging [77]. A number of very useful small-scale patterning techniques have recently been developed to meet this challenge, each with its own set of advantages and disadvantages. A detailed review of such techniques can be found in Ref [78]. Nevertheless, with all these techniques it remains challenging to pattern fragile small-scale samples which are sensitive to physical contact, such as (freestanding) MEMS specimens, or exposure to heat or chemicals, such as biological samples, while both types of samples also typically possess a complex three-dimensional topography. Additionally, multiphase or multi-material samples pose a challenge for achieving a uniform pattern over the whole sample due to varying surface properties, which strongly affect pattern application at the small-scale. In light of these challenges, existing patterning techniques exhibit some limitations. Techniques which are based on the thin-film remodelling principle typical require elevated temperature treatment to modify the thin-film into individual islands (speckles) [79], while the nanoparticle self-assembly technique described in Ref. [80], requires immersing the sample in chemical solutions to first functionalize the sample surface and then to attach Au nanoparticles (NPs) onto the surface. While relatively thick samples can often be immersed in solutions without damage, miniaturized and especially freestanding samples such as MEMS specimens as well as biological specimens are often damaged by submersion in any liquid, especially harsh chemicals. Furthermore, the techniques described above are of a specialized nature while it is not straightforward to apply them on a wide range of sample types. Therefore, this work aims to develop a patterning technique with which it should be possible to pattern delicate samples. Secondly, it should be a generic technique to allow patterning of most of possible cases where a small-scale pattern is required because in most research facilities various sample types are of interest for analysis therefore with a generic technique, only a single technique needs to be mastered. Therefore, the following requirements are set for this new patterning technique:

- Pattern application should be:
  - dry
  - at/near room temperature
  - contactless
• The resulting pattern should be:
  – non-continuous, homogeneous distribution of random speckles of high density
  – sample surface and material independent
  – scalable, without the need for time consuming optimization steps
• And, the patterning setup should be:
  – inexpensive
  – made of readily available non-specialized parts

Herein, we present a patterning technique based on the requirements set above. The methodology uses (commercially available) NPs as the pattern. Similar NPs have been utilized in other patterning techniques [77, 80–82] due to a multitude of inherent advantages including, (i) availability in a wide range to particle sizes from few nm hundreds of \( \mu \text{m} \) (ii) variety of material compositions and surface properties, (iii) easy availability and (iv) low cost due to numerous applications and mature synthetization technologies. However, the key challenge is to invent an application method to apply a homogeneous, high density pattern a with such particles, since the particles have a strong tendency to agglomerate due to complex interactions based on the van der Wall and electrostatic forces being dominant at their intrinsic scales and dictating their behaviour (as discussed in Ref. [80]). These particles can be homogeneously distributed, e.g., in an aqueous suspension (in which they are typically commercially available) by optimizing parameters such as the pH level (using pH buffers) and surface tension (using an appropriate surfactant). However, when this suspension is applied on a sample and dried as in drop-casting [77], complex droplet dynamics phenomena, highly dependent on the surface properties and other factors, dictate the type of pattern achieved on the sample [83]. For example, during coffee-stain pattern formation, the liquid inside the droplet is believed to flow to its periphery as the droplet dries and along with it the liquid drags the NPs which come in contact with each other and form uncontrolled agglomerates. This is undesirable for a high quality DIC pattern as a homogeneously distributed, random pattern ideally consisting of individual particles is desired. It becomes even more challenging to achieve a homogeneous pattern on a 3D or multi-material surface. A solution to this issue is discussed in Ref. [80], which involves first chemically treating the sample surface by silanizing it and subsequently dipping the sample into a bath a freshly synthesized Au NPs. The Au NPs stick to the organosilane molecules (thus immobilized) and finally the sample is rinsed in water. While this method has been employed to apply high quality NPs DIC patterns [80], it requires immersing the sample in liquid baths, therefore it cannot be used for fragile samples as discussed above. Moreover with a change in sample compositions, the chemical treatment steps have to be optimized as discussed in Ref. [82], where a modified procedure is described for patterning corrosion susceptible metallic alloys.

In this work, the NPs diluted in an inert suspension of an ethanol/water mixture are nebulized into an ultrafine mist generated by an ultrasonic nebulizer and carried to the sample surface by a low velocity inert gas (Ar, \( \text{N}_2 \) or air) flow. In order to inhibit NPs agglomeration on the sample surface, ideally a single NP per droplet (of the mist) is desired, which is controlled by keeping the density of the NP in the suspension sufficiently low. This way the droplet drying problem of a single large droplet with thousands of NP in drop-casting leading to agglomeration is converted to the case of millions of microdroplets each predominantly containing one (or zero) NP. This makes
Chapter 4.

50

The patterning sample surface/material independent with a rather simple and straightforward process. Furthermore, the microdroplets mist is dried up before it reaches the specimen with the help of a heated passage which ensures that the solvent evaporates with only dried particles reaching the sample and thus circumventing the possibility of condensation of solvent on the sample surface, or mobility of prior deposited NPs. The choice of the ultrasonic nebulizer comes from the fact that it can provide a narrower micro-droplet distribution in the mist when compared to, e.g., compressed air spray nozzles [84]. Due to the tighter control on the mist droplet size the ultrasonic nebulizers have gained popularity in some applications as a replacement of compressed gas nozzles, notably in aerosol-based drug delivery devices [85, 86], spray pyrolysis based thin-film [87–89] and powder/NP synthesis [84, 90, 91]. Furthermore, due to the compact nature of the ultrasonic nebulizers consisting of a piezo transducer connected to a simple driving circuitry, compared to a nozzle attached to a compressor or pump, the ultrasonic nebulizers have gained popularity in portable commercial applications such aerosol-based drug delivery devices, household humidifiers, etc. Consequently, a wide range of nebulizer kits are available from a number of vendors for a low price. For instance, the nebulizer kit (TKD nb-59s-09s-0) used in the setup described here costs $60.

In the proceeding section, details of the patterning setup are presented for reproduction. Subsequently, in the results section high density patterns utilizing NPs of 50 nm, 300 nm, 500 nm and 1 µm size on various types of delicate samples are shown to show the feasibly of patterning technique.

4.2 Setup Design

The design of the setup developed for patterning is described here in detail for the purposes of reproduction. It was intended to keep the design of the setup simple for ease of reproduction by fellow researchers. Therefore, even when specialized components were available, inexpensive and more readily available alternatives were used instead, such that the total cost of the setup was kept well below $1000.

A schematic drawing and photograph of the setup with all the main components are shown in Figure 4.1, and Figure 4.2, respectively. The setup essentially consists of three main sets of parts. The first set marked by lettering A in Figure 4.1, consists of the main nebulizer tube [A1] and associated parts where the NP suspension [A4] is kept at low temperature and nebulized with the help of a piezoelectric ultrasonic nebulizer [A2]. The next set of parts marked by lettering B in Figure 4.1, generates a flow of clean air with a controlled flowrate and this airflow carries the nebulized droplets of the suspension toward the sample out of nebulizer tube. Lastly the nebulized mist is passed through a passage (marked by lettering C), part of which is heated to completely vaporize the liquid component of the mist, resulting in dried NPs. The NPs are deposited on the specimen in a turbulent flow [D4].

The nebulizer tube [A1] is machined from aluminium, with a relatively long length, such that there is enough height available for the jet resulting in micro-droplet mist to form freely. An air inlet hole at the top (in which the syringe filter [B3] fits) was drilled at a slanting downwards angle to allow a flow of air to be injected into the nebulization tube that lifts the NPs containing mist out of the tube. A piezo actuator [A2] is fitted
at the bottom of the nebulizer tube through a simple adapter plate, while a seal cut out from a 0.5 mm Teflon sheet is used as a seal between the adapter plate and the bottom of the nebulizer tube. Apart from a few Teflon components the setup is completely free from polymers, restricting the choice of materials to metals and glass, since any polymers leeching or dissolving into the solution would end up as hydrocarbon residue on the sample. Teflon is highly chemically inert, so where the use of polymers was unavoidable, Teflon based components were used. A commercially acquired ultrasonic nebulizer (TDK nb-59S-09S) was used here. The transducer has an operating frequency of 1600-1720 kHz, which results in a mean droplet size of $\sim 2 \mu m$, using the analytical equation in Ref. [92], and using values of density and surface tension for a mixture of 25% ethanol by volume in DI water from Ref [93]. The nebulizer kit are provided with the driving electronic circuitry, therefore, the transducer only needs to be installed onto the bottom of the nebulizer tube for operation. The electrical circuitry, and more specifically a power-transistor on the circuit can heat up to high temperatures over time, therefore it is advisable to have a heatsink attached to the power-transistor and keep in a well-ventilated location to avoid an electrical burnout. Ultrasonic nebulizers operate most efficiently at a specified liquid column height above the transducer. For the specific transducer used here, the optimum liquid column height is found to be 13 mm above the transducer surface for the 25% ethanol by volume in DI water mixture. The nebulizer tube can contain only a small volume of liquid, therefore to maintain the liquid level constant over the transducer to approximately 13 mm for a few hours of deposition, a reservoir [A7] is attached to the nebulizer tube with the help of a siphon established between the nebulizer tube and the reservoir. At the base of the nebulizer tube, a u-shaped bent tube from soft copper tube (O.D. 8 mm) is attached which in turn is attached by means of Teflon tubing with the tubing end residing at the bottom of the reservoir. Alternatively, a float-switch or level sensor placed in the nebulizer tube and connected to a valve between the nebulizer tube and the reservoir can be utilized to maintain the liquid level at a constant height. The temperature of the NP-suspension inside the nebulizer tube can reach up to 50°C, within a few minutes of operation, to high vibrational energy generated by the actuator. While a higher liquid temperature can be beneficial for achieving a higher micro-mist output from the nebulizer, here it is undesirable as higher temperatures provide more energy to the NPs in the suspension leading to agglomeration already in the suspended state. Depending on their constituent material, NPs should be kept at a specified temperature to ensure their stability in liquid suspension and thus avoiding agglomeration. Many NP suspensions, e.g., silica NPs in DI water or ethanol are stable around room temperature, while certain particle suspension such as Au in DI water typically require refrigeration for stability. Therefore, the nebulizer tube is cooled to stay close to room temperature for the choice of particles used here. The easiest solution would be to attach cooling fins around the nebulizer tube. However, the cooling fins were not found to be efficient enough without a strong air flow from a fan and a strong air flow in the vicinity of the output nozzle is disruptive for the flow of the dried NPs reaching the sample surface. Therefore, liquid cooling was used here instead and a cooling coil was made by bending a readily (commercially) available 8 mm soft copper tubing around the nebulizer tube [A5]. The water was recirculated through the cooling coils and a fan-cooled radiator (Enermax LiqFusion) using a simple in-line pump (Comet 1005.02.00). Using this assembly, the
temperature of the suspension inside the nebulizer tube could be kept to 30 °C, which was enough to prevent temperature induced NP agglomeration. For suspensions, requiring refrigeration temperatures, the cooling coil can be attached to a compressor based liquid chiller, e.g., using such a recirculating chiller (Caron 2050W) a temperature of 4°C was easily reached inside the nebulizer tube.

The transport gas (air) to push the NP mist out of the nebulizer tube and onto the sample is obtained from a miniature diaphragm pump (VWR VP 86) marked as [B1] in Figure 4.1. It is important to use an oil free pump to avoid any contamination of the setup and the sample. Alternatively, other inert gases such as N₂ or Ar, typically available in laboratories can be utilized here. However, it is important to take into account the flow rate conversion factors to recreate the same results from different gases. The flow rate is measured and controlled with the help of a rotameter (Omega FL-2013) [B2] for flows in the range of 0.4 to 5 litres per minute (LPM). A disposable syringe filter (PTFE membrane, VWR 514-0067) with a pore size of 45 µm is subsequently used [B3] to filter out any residue such as dust before the air flow is injected into the nebulizer tube. The airflow drags the nebulized mist containing the NPs out of the nebulizer tube upwards towards the specimen. The nebulized mist is subsequently heated to vaporize it such that dry particles arrive at the sample surface. The first part of this passage way is a 24/40 join, glass inlet adapter [C1] with a 90° bend (VWR 201-0348). The inlet adapter fits into the nebulizer tube with the help of a bushing adapter with 24/40 inner female joint and 34/45 outer male joint (Laboy HMA012011). The tube connector end of the inlet adapter is attached to a soft copper tubing of a total length of 45 mm bent to an L shape [C2]. A 400 Watt flexible rope heater (Omega FGR-080/240V) with a total length of 8 feet is wrapped around the copper tubing [C3]. Typically such a heater is attached to a temperature controller, however, for ease of design, a simple AC dimmer (IVT DR-2000 Hand-Dimmer), offering PWM-based power (temperature) control, was used instead [C4].

4.3 Methodology

To start the procedure, the setup is rinsed with ethanol to remove any dried and agglomerated particles from previous depositions. Next a low-concentration suspension of the NPs in a volatile solvent needs to be prepared, because the particles typically come in a highly concentrated suspension (typically DI water) and thus need to be diluted. The exact concentration depends on the particle size and initial solution concentration. A too low suspension concentration results in an excessively long deposition time, while a too high concentration leads to an agglomerated pattern, as a result of NPs coming in contact with each other in the suspension and/or because of nebulized droplet containing multiple NPs are formed. Here, the concentration of the solution is optimized by starting with a low concentration and increasing until the first large spherical agglomerates can be seen. Before this point, it is assumed that the average micro-mist droplet carries only one (or zero) particle.

The choice of the liquid in the suspension depends on a number of factors. Firstly, the liquid should be volatile at relatively low temperatures such that in the micro-mist the liquid can be easily evaporated. Secondly, it is desired that the liquid possesses a
Figure 4.1: Schematic drawing of the patterning setup.
low surface tension for higher dispersibility of the NPs in the suspension and thus less agglomeration. Considering these factors, alcohols would be an ideal choice, however, in our experience it led to a ‘hydrocarbon’ like residue accumulating over the sample surface. We hypothesize that this might be resulting from less volatile hydrocarbons being dissolved in the suspension from the setup (although care was taken not to have any dissolvable materials in the setup) or the hydrocarbon residue from the ambient air being dissolved in the air stream. DI water is a better option to avoid hydrocarbon solubility, however, it possesses higher surface tension leading to a more agglomerated pattern and a higher boiling point. A solution was found by using a mixture of ethanol and water since addition of 25% ethanol by volume significantly reduces the surface tension of DI water from $72.08 \times 10^{-3} \text{ Nm}^{-1}$ (for pure DI water) to $\sim 38.2 \times 10^{-3} \text{ Nm}^{-1}$, while the surface tension of pure ethanol is, at room temperature and atmospheric pressure [93]. The ethanol-water mixture (25% V/V) was found to result in a good dispersibility of NPs in the suspension and thus significantly reduced agglomeration on the sample, while a hydrocarbon residue is prevented. It should be noted that laboratory grade DI water should be used. In our experience, significant microplastic residue consisting of microspheres ranging from a few nm to hundreds of µm were found in several sources of DI water, including DI water from laboratory filtration stations, as found by drop-casting and analysis in SEM. The DI water used here was obtained from VWR with MDL number: MFCD00011332 and CAS number: 7732-18-5.

A wide variety of NPs of different material compositions are commercially available, including metals (Au, Ag, Cu, etc.) ceramics and polymers, with silica, and polystyrene particles being most widely available. Furthermore, different dyed and fluorescent colors and surface functionalization are available. Here, silica and polystyrene NPs were used since both can be dispersed easily in water or alcohols without the need for a surfactant and can be commercially obtained easily for a low price.

Typically, a solution of 200 ml was used and during an hour of deposition approximately 20 ml of the solution would be consumed. As suggested by the NP manufacturer, prior to use, the suspension was sonicated for around 30 minutes in a bath sonicator to break any agglomerates and to distribute the particles homogeneously in the suspension. During sonication over tens of minutes the temperature of the liquid can increase up to 50 °C, therefore, to avoid elevated temperature induced agglomeration, it is important to keep the bath temperature in check. Finally, the sonicated solution is added to the nebulizer tube and a siphon is established between the setup and the reservoir, by first completely filling up the nebulizer tube with the suspension and then lifting it up till the liquid passes to the reservoir beaker and all the air is forced out. The height of the liquid in the nebulizer tube is set to the optimum height of 13 mm by adjusting the lab jack below the siphon reservoir beaker ([A7], Figure 4.2 (4)).

Subsequently, the nozzle to sample distance [D2] is set by adjusting the height of the lab jack beneath the sample. This distance, together with suspension concentration is the most important parameter to obtain good pattern quality. One may intuitively assume that it would be best if specimen is kept as close as possible to the nozzle to ‘catch’ all the particles and thus obtain a high-density pattern. However, in this configuration numerous planar particle agglomerates are typically noticed, as seen in Figure 4.11 (shown in the Appendix section). It was found that this type of agglomeration typically points to the case that the agglomeration is happening on the specimen itself, likely due
to an enhanced particle mobility at specimen surface induced by forced airflow. Such planar agglomerates, especially in long chains could break under localized deformation or partially slide on the surface to comply with the local deformation, thus leading to erroneous results. Another observation was made that close to the nozzle the flow appears to be laminar and further away from the nozzle the flow become turbulent (see Figure 4.10 in the Appendix section) and if the sample is kept in the turbulent region a more homogeneous NP pattern is achieved. Therefore, it is important to adjust the sample to nozzle distance \[D2\] such that the sample lies in the turbulent regime. Based on the relationship for determining the Reynolds number of the free-jet flow [94], the flow can be made turbulent in a number of ways, including increasing the nozzle diameter,
increasing the flow rate (velocity) or being further away from the nozzle. Increasing the diameter would decrease deposition time, since the particles would be spread over larger area on the sample. Increasing the flow rate is beneficial to a certain degree since while making flow turbulent earlier, it also brings in more droplets and thus particles from the nebulizer tube thus decreasing deposition time. However, after a certain point this effect of flow rate increase starts to be detrimental, as it can carry the very large nebulized droplets leading to large spherical agglomerates. In our experience, we found a flow rate of around 3LPM of air with a sample to nozzle distance of 7.5 mm yielding good results for a nozzle width and inner diameter of 6 mm. It is suggested to calibrate the sample to nozzle distance while the heating is turned off since then the flow of nebulized mixture can still be visualized as faint translucent flow, as shown in Figure 4.10 in the Appendix. This way of visualization of the flow pattern is importance as it allows to determine the optimal sample location, which is approximately in the middle of the turbulent regime. If the sample is kept at the end of the turbulent regime or further away almost no particles land on the sample surface and it can give the impression that the technique is not working. Likewise, when the specimen is placed besides the flow diameter (of ∼15 mm for the settings used) very few particles land on the sample surface also giving the impression that the technique is ineffective.

After ∼15 mins of operation, a steady state temperature of ∼200 °C (measured with thermocouple) is reached for a 75% input power. It should be noted that while that temperature of the outflow at the nozzle is quite high, by the time it reaches the sample, it drops to ∼37 °C, which was confirmed in many experiments. Finally, it is important to note that at intermittent intervals the sample can be taken out of the patterning setup and checked under a microscope to see if the desired density has been achieved and if not, the sample can be placed again under the setup to increase the density which is shown in the next chapter.

A sample to nozzle distance of 7.5 mm was found to be optimum for deposition along with a flow rate of 3 LPM. These values were used for depositing all the cases described in the proceeding section while the suspension concentration was optimized for each NP size and is reported correspondingly. Furthermore, the suspensions were made in an ethanol-DI water mixture with 25% ethanol (V/V). All NPs were obtained from micromod Partikeltechnologie GmbH. It was found that for very small sizes such as the 50 nm particles, NP agglomeration was the determining factor for achieving a good pattern and silica particles provided the best results in terms of an unagglomerated pattern. While, at larger sizes such as the 1 µm NP diameter, while the silica particles provided a high quality unagglomerated pattern, the density could not be increased with longer deposition time beyond a certain pattern density. On the other hand, the polystyrene (1 µm) NP provided a high quality pattern as well, while the density would keep on increasing with longer deposition. Therefore, in our experience at the larger particle diameters, close to 1 µm, polystyrene NPs seem to provide the best results in terms of reaching a high density, while at the lowest end of the spectrum, i.e., close to 50 nm, silica NPs provided a higher quality pattern. In the medium particle diameter range, i.e., around 500 nm, both silica and polystyrene NPs seem to result in a similar type of pattern and can be used interchangeably.

Please note that the information provided by NP manufactures states that prolonged inhalation the NPs (silica) can be hazardous, therefore it is important to operate the
setup in a fume hood.

4.4 Experiments: Pattern test cases

To show the applicability of the patterning technique, a wide range of patterning examples are given here, including patterns of a range of particle sizes, samples with different surface characteristics and various delicate samples.

4.4.1 500 nm, 50 nm and 1000 nm NP pattern on polished steel

The first example is of 500 nm silica particles (with a plain, non-functionalized surface) deposited on top of flat mirror polished steel surface. The particles were originally provided in a suspension of DI water without surfactant with a concentration of 50 mg/ml. The suspension was further diluted into with a ratio or 1:4000. To reach a pattern density shown in Figure 4.3, the sample was patterned for 110 mins. In Figure 4.3a microscopic image of the sample color coded with deposition time is shown to show the increase in pattern density with deposition time. After each time interval the sample was imaged under optical microscopy. This also highlights an advantage of this technique over other pattering techniques, as the density of the sample can monitored and increased additively. As it can be seen in Figure 4.3b and c, a many particles land next to the already deposited particles on the sample without agglomeration. A few spherical agglomerates, which are inevitable due to the larger than mean sized nebulized droplets containing multiple NPs, can be seen in Figure 4.3.

For many high-resolution DIC applications, such as quantifying sub-grain strain fields, obtaining a DIC pattern with speckle sizes below 100 nm is of special interest. Therefore, DIC pattern with 50 nm plain silica NPs was applied on a polished steel sample for 210 mins, as shown in Figure 4.4. It can be seen that a homogeneously high-density patter is deposited. To this end, the original NP suspension was diluted with a ratio of 1:40000. Although not tested it should be possible to deposit smaller sized NPs down to few nm, which are still commercially available, however, the source solution would need to be diluted further of course because due to smaller size, the chance on having more than one NP in a nebulized droplet increases for the same concentration.

As shown in Figure 4.5, 1 µm polystyrene NPs have been deposited on a polished steel sample essentially to show the upper limit of the particle size that can still be used. The original suspension was diluted by a ratio of 1:100 and the sample was deposited for 480 mins. As mentioned earlier, the average droplet size of the mist for the nebulizer used here is estimated to be ~ 2µm. Therefore, it becomes difficult to deposits larger particles.

4.4.2 LED cross-section: (varying material composition sample)

A major challenge in small-scale pattering is the surface dependent adherence of the speckles onto the sample surface. While at larger scales, techniques such as airbrushing can be utilized to obtain a reproducible patterns on samples with almost any material composition, at the µm/nm scales, the adherence of the patterning material on the sample is highly dependent on the physical and chemical interactions of the patterning materials with the sample surface material. For instance, while the self-assembled
Figure 4.3: Color-coded optical microscope image of a polished steel sample deposited with 500 nm silica nanoparticles. Insets show a magnified image of a region marked by dashed rectangle, with (b) a colour-coded image and (c) the original dark field image. Images were taken at four time intervals and the colours represent the particles deposited in each of the four intervals.
Figure 4.4: SEM image of 50 nm silica NPs deposited on top of polished steel sample. The SEM image was taken in high-vacuum mode with an acceleration voltage of 8.5 kV in secondary electron mode.

Figure 4.5: 1 µm polystyrene NPs deposited on top of polished steel sample deposited for 8 hrs. The SEM image was taken in low-vacuum mode (at 1.3 mbar) with an acceleration voltage of 20 kV in secondary electron mode.
nanoparticles technique can be applied on metals as well as non-metals, it requires modifications based on the sample substrate, e.g., for corrosion susceptible specimens as discussed in Ref. [82] to successfully functionalize the sample surface with organosilane molecules to which the Au NPs attach. Similarly, in the Au thin-film remodelling technique, the interaction of the Au layer with the underlying substrate is of key importance for condensation of the Au layer into separate islands [74]. Optimization to a specific specimen surface becomes even more complicated if a sample is composed of multiple materials/phases, such as the cross-section of a light emitting diode (LED), as seen in Figure 4.6, which is composed of an epoxy lens, a ceramic (AlN) substrate, metal (Cu) electrodes on a polymer printed circuit board (PCB). For such a specimen DIC can provide useful insight in, e.g., thermo-mechanical induced shear strains at the interfaces leading to decohesion induced failure. For this specimen, optimization of a patterning technique to achieve suitable pattern over the whole specimen is not only challenging due to the highly heterogeneous surface properties, but also due to the sensitivity to chemical reactions (e.g., corrosion of Cu in DI water) and sensitivity to high temperatures (e.g., of the epoxy lens and plastic PCB).

The patterning technique described here can be very convenient for such samples, since only dry NPs come in contact with the specimen and adhere to the surface due to physical interactions, e.g., van der Walls forces. Importantly, it was consistently observed that all the deposited particles, ranging from 50 nm to 1 µm once deposited cannot be blown off the surface even with pressurized air. In contrast, when a droplet of water or ethanol is added to the patterned surface, the NPs disperse in the liquid and subsequently rearrange on the surface as the liquid dries, leading to, e.g., coffee-staining [83].

The LED sample shown in Figure 4.6 was patterned using 500 nm plain silica NPs and the suspension was diluted with a ratio of 1:4000 and the sample was patterned for 360 mins. Importantly, the different material composition areas show a very similar pattern quality.

4.4.3 Synthetic hydrogel fiber for biological applications: (chemically delicate sample with topography)

Applications of DIC on biological samples typically involves dealing with samples that are highly delicate in terms of interactions with chemicals. Moreover, such samples are typically non-planar or highly textured, and planarization can affect their structures and properties that are to be investigated using DIC. Additionally, they can be easily damaged under (high) vacuum conditions by loss of moisture and even moderately high temperatures. Therefore, patterning such samples requires a dry technique, where the sample does not come in contact with any chemical, even water. Moreover, the patterning needs to be isotropic over the non-uniform three-dimensional topography.

An example of such a sample is a synthetic hydrogel fibre used in 3D printing of bioreactive scaffolds that can be employed for in-situ tissue regeneration [95]. The hygroexpansion of the hydrogel material is used as a means for dynamic actuation of the overall structure [96, 97]. Therefore, quantifying the hygroexpansive swelling behaviour of such materials, e.g., using Digital Height Correlation (DHC) together with surface profilometry or AFM imaging is of special interest [98–100].
Figure 4.6: Patterning of LED cross-section with 500 nm silica particles, with (a) Back Scatter Electron image of the LED cross-section, and Secondary Electron images of (b) Cu electrode, (c) plastic PCB, (d) epoxy lens, and (e) aluminum-nitride substrate. Note that subfigures (b)-(e) have the same magnification.
The cylindrical hydrogel fibre sample shown in Figure 4.7 was patterned using 500 nm plain silica particles for 360 mins. It could be clearly seen that the fibre material is very sensitive as electron beam exposure for \( \sim 1 \) min during SEM imaging (in low-vacuum mode) resulted in the samples surface being damaged (Figure 4.7b). Therefore, another region of the fibre, previously unexposed to the electron beam, is shown in Figure 4.7c to demonstrate that the patterning technique itself did not damage the fibre. Moreover, the fibre material is fragile to chemical exposure, as all attempts to delicately pattern the fibre by airbrush spraying the same NP suspension resulted in extensive fiber damage, as shown in Figure 4.7d. Both Figure 4.7b and c show that the cylindrical surface of the fiber was covered well with the NPs, even at very steep side angles.

### 4.4.4 Freestanding microtensile specimen: (EBSD transparent pattern on a physically delicate sample)

Performing simultaneous EBSD (crystallography characterization) along with DIC measurement can be of high interest, especially where it is important to understand the connection between crystallography and microscale deformation. Examples of that include analysis of fracture at the microstructural scale [101], change in grain orientation with deformation [101], twinning [102] or phase-transformation mediated deformation [103], or the role of complex microstructure on deformation [104, 105]. While application of DIC in such cases requires a high-density pattern, simultaneous EBSD imaging requires a pattern that is transparent to the backscatter electrons (BSE). Furthermore, it is important to have the surface as visible as possible between the pattern speckles, e.g., in BSE or SE imaging mode, such that the strain fields can be directly superimposed on the underlying (visible) microstructure and related to surface phenomenon such as slip lines and damage. Therefore, a solution such as deposition of a textured thin-film on top of the sample does not suffice as it impeded visualizing of the sample surface.

A test case of a freestanding microtensile polycrystalline Al specimens of 5 \( \mu m \) thickness was chosen here (see Figure 4.8a). Details on the sample are available in Ref. [75]. The freestanding nature of the sample makes it very challenging to pattern since, e.g., it cannot be come in contact with any liquids. The as processed (microfabricated) sample was patterned using 50 nm silica NPs for 270 mins and the patterned sample surface is shown in Figure 4.8b. Note that it would have been beneficial to perform a plasma cleaning step to remove hydrocarbon contamination on the sample surface before deposition however, this was not available to the project. Silica NPs are ideal for their EBSD and BSE transparency, as due to their lower atomic mass compared to noble metal NPs with higher atomic mass and thus shallower electron penetration depth, making it more challenging to achieve EBSD and BSE transparency, as argued in Ref. [106]. Thus, the EBSD is typically performed post-mortem after removing the DIC pattern, e.g., by rising the sample in a cleaning agent, mechanically polishing the surface or performing FIB milling [80, 102].

After deposition, EBSD data was collected for this sample was collected on FEI Serion SEM, using an acceleration voltage of 25 kV and spot size of 5. The EBSD scan was performed using EDAX OIM data collection software, with a \( 5 \times 5 \) binning. As seen in the image quality (IQ) map in Figure 4.8c, only at the grain boundaries the IQ is poor, which is unrelated to the NPs, while within the grains, the NPs do not reduce the
Figure 4.7: Patterning of an ultra-delicate hydrogel fiber. (a1) Un-patterned initial fiber surface and in the figure. (a2) Topographical profile of the fiber highlighting the approximately cylindrical shape of the fibre. (b1) The same region as (a1) after patterning for 360 min with damage visible on the surface due to previous electron beam exposure. (c1) An adjacent region of the fibre after 360 mins of patterning, not previously exposed to the electron beam, with no visible damage, which demonstrates that the damage is not caused by the patterning operation. (b2 and c2) The magnified view at the right shows the same density of the pattern at the steep side surface. (d) A fibre from the same material damaged after patterning a suspension of NPs using an airbrush, using the most delicate airbrush setting which that the sample can easily be damaged. Note that (a)-(d) have been imaged at the same magnification in secondary electron mode in low-vacuum operation (1 mbar) at an acceleration voltage of 8.5 kV.
IQ, as can be seen by comparing the zoomed inset of Figure 4.8b with the corresponding zoomed inset of Figure 4.8c. The poor IQ at the grain boundaries is caused by the fact that they are highly etched due to the microfabrication processing, creating so-called “grain boundary grooves” with steep side slopes, which results in a poor EBSD signal from those regions. A similar trend can be seen in the inverse pole figure (IPF) shown in Figure 4.8d. It should be noted that the IPF is shown in the as-recorded state without any postprocessing data cleaning. Therefore, these measurements demonstrate that the EBSD transparent silica NP pattern enables simultaneous DIC and EBSD data acquisition.

### 4.4.5 Freestanding microtensile specimen: (EBSD transparent pattern on a physically delicate sample)

In the field of micro- and nano-electromechanical systems (MEMS and NEMS), material characterization is of key importance. Due to the likelihood that mechanical size-effects are active, the mechanical properties of the material cannot be assumed a priori form bulk material properties [107]. Furthermore, such size-effects typically depend on the processing conditions, therefore, either the same processing needs to be used for fabricating dedicated samples, or ideally, characterization should be performed on the MEMS/NEMS structure itself. Full-field displacement data from DIC can be invaluable in such cases, especially when characterizing inhomogeneous displacement fields, as in the case of, e.g., strain localization due to plasticity, or a complex sample geometry. However, due to the highly delicate nature of such structures, DIC patterning becomes very challenging. Therefore, typically the native surface pattern due to roughness, grain boundaries, voids, etc. is relied upon [75, 108, 109].

A delicate freestanding MEMS structure designed as highly stretchable interconnect for stretchable electronics applications is shown in Figure 4.9a. A pattern is applied to the MEMS structure to enable Digital Height Correlation (DHC), which is a special form of DIC that yields three dimensional surface displacement fields by correlating surface topography images, here measured with an optical confocal profilometer. The microfabricated sample is 50 \( \mu m \) tall and micromachined from a 300 nm layer of a sputtered layer of 99.99999% Al [64]. The samples as discussed Ref. [55], exhibited a significantly enhanced yield strength due to the likely presence of mechanical size-effects. Therefore, the displacement fields were characterized using DHC, to be used in conjunction with an inverse characterization method (integrated DHC), for quantifying the yield strength of the material, using the as processed sample.

Since in regular operation, while stretching, most of the beams of the interconnect rotate by 90° out-of-plane impeding the applicability of DHC, a modified loading sequence was applied. The interconnects was loaded in the out-of-plane direction and subsequently unloaded multiple times, after each incrementally increasing displacement load. A high magnification 100x lens of 0.9 NA and 1 mm working distance was used to measure the surface (height) topographical images. The sample beams exhibit high curvatures in the out-of-plane loading state, resulting in the data not being captured over most of the whole structure. In contrast, in the unloaded states after each load-unload sequence the beam curvature due to the initial residual stress and subsequent plasticity is not too high and the topography can be easily captured. Therefore, the topographical
Figure 4.8: Pattern of a delicate freestanding microtensile specimen (polycrystalline Al of 5 µm thickness with an EBSD transparent pattern of 50 nm silica NPs. (a) SEM image of the freestanding microtensile sample in secondary electron mode under high-vacuum and an acceleration voltage of 7.5 kV. (b) A magnified view over the width of the sample, marked in (a) with a red rectangle. A further magnified view of a smaller region of interest marked by a yellow rectangle is shown as an inset to show the density of the pattern. (c) Image quality (IQ) map from an EBSD measurement (at 25 kV) of the same area as (b), showing low image quality at the grain boundaries due to grain boundary grooves with steep slopes and some variation in (IQ) in the grains related to the surface texture. However, the particles have negligible effect on the image quality. (d) An inverse pole figure (IPF) showing grain orientations corresponding to (c) without any post-processing (data cleaning). The IPF shows the same trend as the IQ with un-indexable areas limited to the grain boundary grooves.
images were only captured in the unloaded configuration after each load-unload sequence to quantify the plastic deformation in the structure, which manifests itself as change in curvature of the unloaded beams.

Since the sample did not possess enough natural contrast, a pattern using 300 nm silica particles was applied. The original suspension was diluted by 1:9000 and the sample was patterned for 3 hrs. This was a particularly challenging case since the in-plane optical resolution of the optical profilometer is \( \sim 255 \) nm thus it was important to use particles larger in size than this value, while the width of each beam is 2 \( \mu m \). Therefore, the choice on pattern size was quite constrained. As seen in Figure 4.9a, the density of the pattern is not as high as previous examples, which is mainly due to the fact that the pattern could not be optimized due to scarcity of available highly-stretchable interconnect samples yet it does fulfill the requirement of having quite a few particles over the sample length that is needed for the used global Non-Uniform Rational B-Splines (NURBS) based DHC algorithm. The sample was meshed with four elements over the length and one element over each beam element with 2\textsuperscript{nd} order shape functions in the beam length direction and 0\textsuperscript{th} order shape function on the beam width direction. Further details on the NURBS based DIC algorithm can be found in Refs. [110, 111]. Using DHC, the three dimensional displacement fields of the stretchable interconnect sample could be successfully mapped in the in-plane and out-of-plane directions (see Figure 4.9(b) – (d)).

4.5 Concluding remarks

Herein, a new technique for patterning the otherwise challenging small-scale fragile samples, to enable DIC-based micromechanical analysis of such structures, is presented. The technique consists of generating a well-controlled mist of a (water/ethanol) volatile suspension containing the nanoparticles (NPs), which is dried up resulting in ideally single particles landing on the samples surface. The technique circumvents the numerous issues with application of NPs for random distributions directly from a volatile suspension, using the so-called ‘drop casting’ method. To show the applicability of the method a number of examples of patterning of delicate samples are presented. Silica particle from 1 \( \mu m \) down to 50 nm in diameter have been applied successfully yielding a high quality pattern with high density, homogeneous distribution and good contrast. It should be noted that while smaller particle sizes have not been tried but it should be possible to deposit them as well. There are a number of advantages of this technique, namely:

1. **Dry and chemically inert and near-room temperature process**

   A key advantage of this technique is that it is dry in nature, which is of essence when dealing with small-scales freestanding structures such as the microtensile sample or the 300 nm thick stretchable interconnect patterned here, for which contact with liquids can easily lead to damage. Furthermore, the chemically inert nature of the used airflow means that chemically fragile samples, such as the hydrogel fibres, as dealt with here, or other biological samples can be easily patterned.

   Moreover, the deposition can be performed at near-room temperature of \( \sim 37 ^\circ \)C. For applications that might require operation at lower temperatures, the heating
Figure 4.9: Patterning and subsequent digital height correlation (DHC) results for a highly delicate freestanding stretchable electronics sample with a height of 50 µm and a thickness of 300 nm. (a) Topographical image of the interconnect patterned with 300 nm silica NPs. A magnified image of part of the sample showing the added roughness due to the NPs is shown in an inset. (b, c,) The in-plane displacement field in (b) x- and (c) y- direction. (d) The out-of-plane displacement field in the z-direction overlaid on an image of the freestanding interconnect.
tube temperature in conjunction with the nozzle to sample distance and the flow-rate can easily be varied and optimized.

2. **Sample material and topography independent deposition**

This technique is effectively independent of the sample surface properties and can be used to pattern samples with varying sample composition, as demonstrated by patterning of a multilayered LED cross-section sample. Similarly, samples with varying surface topography can be patterned as is the case for cylindrical hydrogel fibers shown here.

3. **Scalability**

It has been shown that the technique is scalable in the range from 1 µm and lower speckle sizes, which is the ideal regime for micromechanical testing. The only parameter that needs to be optimized with the change in particle size is the concentration of the suspension. Note that, for scales larger than 1 µm, applying microspheres or ink based spray using an airbrush is a good option, as already reported in literature [78]. Other advantages include the relatively low setup and operation costs. The setup parts approximately cost < $1000, while the microspheres particles are relatively low priced. For instance, the 500 nm silica particles were approximately $6 per deposited sample.

A limitation of the technique is that very high speckle size density, such as reported for thin-film remodelling based techniques, might not be possible [78, 79]. The density of the pattern seems to saturate with an average particle to particle spacing (centre to centre) of ∼4-6 times the particle diameter. A solution for cases where very high density is required can be to use smaller particles sizes relative to the field of view. However it should be noted that for accurate DIC application, each speckle (NP) should span over at least 3 pixels as discussed in Ref. [112], to avoid aliasing errors.

**Acknowledgements**

This work was supported by the Vidi funding of J.P.M.H. (project number 12966) within the Netherlands Organization for Scientific Research (NWO). The authors would like to acknowledge Sandra van de Looij - Kleinendorst for the digital height correlation, Varun Shah for performing EBSD scans, Lucien Cleven for fabricating the setup, Lambert Bergers, Andre Ruybalid and Niels Vonk for providing test specimens, Weijia Zhu, Nick Verschuur and Inge van der Kuil for assistance with experiments and Marc van Maris for technical support.

**Appendix**
Figure 4.10: Photograph of the flow of the nebulized mist from the nozzle, showing laminar and turbulent flow regimes. Note that rope heater was powered down to be able to visualize the flow.
Figure 4.11: Example of deposition in the laminar regime, resulting in planar agglomeration on the sample. The sample was deposited at a sample to nozzle distance of 4 cm instead of the optimized 7.5 cm distance.
CHAPTER 5

Multi-axial electro-mechanical testing methodology for highly stretchable freestanding micron-sized structures

Abstract

Recent advances in MEMS technology have brought forward a new class of high-density stretchable/flexible electronics as well as large displacement MEMS devices. The in-situ electro-mechanical characterization of such devices is challenging since it requires: (i) highly delicate sample handling, (ii) controlled application of large (hundreds of μm) multi-axial displacements to mimic service conditions, (iii) integrate electrical testing and (iv) fast actuation for cyclic testing. Techniques already developed for small-scale testing in literature fall short to meet the combined set of requirements. To this end, a characterization methodology that fulfils all these requirements is developed and presented here. The technique is based on a piezo-driven micro-tensile stage, which provides large multi-axial displacements with high resolution and fast actuation (4000 μm/s). This is combined with a method for sample microfabrication on a test-chip to warrant delicate sample handling. Proof-of-principle experiments are shown for multi-axial mechanical characterization, electrical characterization and high cycle fatigue testing of micron-sized highly stretchable interconnects. Experiments are conducted under in-situ microscopic observation using optical microscopy, scanning electron microscopy, and high resolution profilometry. The generic platform proposed here can be used for other problems where similar requirements are faced, e.g. other miniaturized, large displacement electro-mechanical applications that are currently being developed.

5.1 Introduction

By imparting flexibility and stretchability to intrinsically stiff and brittle (micro)electronics, numerous novel breakthrough applications have been reported. The diverse applications of stretchable electronics (SE) include smart health monitoring body patches [52, 53, 113], bio-mimicking wide angle cameras (detectors) [50], conformal neural implants [114], to name a few.

A typical (hybrid) approach to realize SE devices consists of placing an array of application-specific integrated circuit (ASIC) islands on an elastomer substrate, whereby the islands are electrically connected by metal, stretchable interconnects typically embedded in the elastomer substrate [6]. For many applications, especially detector arrays
[9], increasing the (currently low) density of the ASIC islands to match the density of conventional microelectronics is strongly required for improved device performance. To this end, these interconnects need to be miniaturized (with decreased footprint area), which entails an even larger interconnect stretchability to preserve the overall stretchability of the device [55]. Furthermore, in most applications, the interconnects experience in-plane and out-of-plane shear on top of the uniaxial stretch.

To achieve ultra-stretchability, interconnects that are suspended freely between the connecting ASIC islands (i.e., no embedded in the substrate) have been introduced [22, 36, 55]. Furthermore, employing standard microfabrication recipes for processing the interconnects allows their miniaturization to the micron scale [55, 115] and beyond, while providing straightforward integration with ASIC island fabrication.

Electro-mechanical characterization of such ultra-stretchable interconnects, which are the critical components in such systems, proves to be challenging, since it requires: (1) multi-axial actuation to mimic real service conditions; (2) large displacement actuation in the range of 100s of microns, due to their ultra-stretchable nature; (3) delicate sample handling, since undesired forces or vibrations during processing and handling can easily result in 1000s of percent (unintentional) stretching that may damage the interconnects; (4) integrated electrical monitoring, which can be challenging since conventional bulky probe stations suitable for on-wafer testing need to be integrating with the tensile stage for continuous monitoring; (5) fast actuation for low-cycle and high-cycle fatigue testing to ensure reliability and lifetime requirements, especially for medical devices.

Numerous novel small-scale testing techniques have been developed over the last couple of decades with rapid developments in MEMS technology, notably on-chip testing techniques [116, 117]. While these techniques adequately tackle some of the challenges listed above, e.g., delicate sample handling, with the sample being co-fabricated in a test device, they fail short to meet other challenges, e.g., high displacement actuation [118]. This is mainly due to the fact that these methodologies have been developed for material testing (requiring relatively small strains to failure) and not for testing highly stretchable MEMS structures. The latter have emerged only recently with the advent of highly stretchable and large displacement micro-actuators, e.g., for optical scanning applications [119]. Similarly, at the larger scales, various techniques exist to apply complex, multi-axial deformation [120], [121], [122], which however cannot be downscaled to micromechanical tests, especially when both an out-of-plane displacement and in-plane displacements need to be imposed. Therefore, in this paper, a new experimental methodology tackling the combined challenges of multi-axial loading, large displacement actuation, delicate sample handling, integrated electrical testing and fast actuation is presented. The methodology consists in a miniaturized, fast actuation, multi-axial tensile stage with integrated electrical probing, along with a test-chip designed to ensure delicate handling of the samples at each stage from fabrication to testing.

5.2 Setup design and experimental methodology

The actuation of the miniaturized loading stage is based on two high-resolution piezoelectric nanopositioning stages (see Figure 5.1). The vacuum compatible nanopositioning stages (MCL Nano-3D200) each have three (translations) degrees of freedom (along x, y
Figure 5.1: Configuration of the multi-axial tensile stage, based on two piezoelectric stages (a). (b) Zoom-in of the test-chip with 6 samples. (c) The applied multi-axial displacement is illustrated in the (simulated) deformation of one of the 6 interconnects.

and z axes), enabled by the assembled stack of three stages with a resolution of 1 \textit{nm} and a range of 200 \textit{µm}. The nanopositioning stages are equipped with internal displacement sensors and a control loop to eliminate the creep/drift effects arising from hysteresis in the piezo actuators, for stable actuation even on long time scales. Furthermore, the piezo actuators offer a fast response time, which is needed for (high-cycle) fatigue testing. Combined, the two nanopositioning stages provide a sample actuation range of 400 \textit{µm} along all three axes, enabling complex multi-axial loading.
Figure 5.2: Schematic of typical sample clamping mechanisms for micromechanical tensile testing, with (a) sample clamped at both ends by screw clamps, (b) sample fixed at one end and actuated at the other end through a hook inserted into an alignment hole in the sample, (c) a bow-tie shaped sample that fits into self-aligning clamps at both ends and (d) the sample clamped by the downward force of leaf springs at both ends.

The setup has been designed for in-situ, high-resolution Scanning Electron Microscopy (SEM) as well as optical microscopy testing. High-resolution optical microscopy necessitates small working distances, e.g., a 150x lens with 0.95 Numerical Aperture (N.A.) typically has a working distance of 300 \( \mu m \). This along with the requirement of being able to actuate the samples in x, y and z axes and ensuring a rigid enough clamping such that the interconnects would not be preloaded when a mechanical force is applied to break the sacrificial Si beams (shown in Figure 5.1 and explained later), puts a constraint on the type of sample clamping mechanism that can be used. Therefore, typical clamping techniques used in micromechanical tensile testing, as illustrated in Figure 5.2, cannot be adopted here. Considering these three factors, the test-chip is clamped by applying a low contraction, UV-curable glue to the side faces of the test-chip resting on top of the inset faces which have earlier been levelled in height under a profilometer (see Figure 5.3 and Figure 5.4). Using a UV curable glue allows to first ensure that the sides of the test-chip are properly wetted with the glue and any glue overflowing to the top of the sample is cleaned off. Once a good alignment has been achieved the glue is irradiated with a UV lamp for 30 minutes. After completion of the test the insets together with the samples are unscrewed from the clamps and sonicated in an acetone bath to remove the glue from the reusable insets.

An important aspect to consider in any mechanical test is the proper alignment of the sample with respect to the loading axes to avoid any spurious loading, as elaborated in detail by Saif et al [123][124] in the context of micromechanical testing. This is ensured here by first aligning the test-chip, containing the freestanding interconnects (Figure 5.4), with the clamps. The test-chip is a rectangular platform processed in a
150 mm Si wafer and formed by etching through the wafer thickness. The test-chip consists of two Si islands, for clamping, each supporting the freestanding interconnects at the opposite ends. Sacrificial Si beams are processed into the test-chip to keep the two islands rigid during processing, handling and test-chip clamping. Microfabrication makes it possible to perfectly align the interconnects with the test-chip itself. Etched (flat) faces of the test-chip provide ideal features to align the test-chip with respect to the clamp. The right (or alternatively left) edge face of the test-chip is positioned against the corresponding (finely milled) vertical edge face of the corresponding clamp to align the samples with respect to the clamp (Figure 5.4a). The clamps in turn are aligned with the corresponding loading axes (of the actuators) by placing the (finely milled) vertical edge face of a step machined at the bottom of the clamp against the vertical face of stacked z-stage protruding out from the top of the nanopositioning stage (Figure 5.4b₁). Since the nanopositioning stage was machined using electrical discharge machining (by the manufacture) it provides low-roughness, flat faces, appropriate for alignment purposes. Finally, the two nanopositioning stages (and hence the corresponding loading axes) are aligned with each other by positioning and fixing the vertical edge faces of the stages against the vertical edge face of the placement cavity milled into the base plate (Figure 5.4b₂). This results in a complete alignment of the whole setup and hence the alignment of the interconnect samples with respect to the loading axes.

After clamping, to start loading the interconnect samples, the sacrificial Si beams (Figure 5.1b), processed to keep the two islands fixed with respect to each other need to be broken. The sacrificial Si beams have been designed on the basis of prior Finite Element (FE) simulations to render a high in-plane and out-of-plane stiffness to the test-chip, preventing forces arising from processing, handling, or clamping to be transferred
Figure 5.4: Alignment of the tensile stage components with respect to each other and of the test-chip with respect to the tensile stage, with: a) test-chip placed on the insets (in grey) and pushed against the corresponding face of the clamp (in blue), b) vertical face at the bottom of a clamp, pushed against the corresponding vertical face of island protruding out of the piezo stage and, c) the piezoelectric stages pushed against the vertical face of the base plate cavity to align both piezo-stages with respect to each other.

to the interconnect samples. Whereas, after the silicon islands of the test-chip have been clamped, it is desired that only a small force is needed to break the sacrificial Si beams, to make sure that the loads that are transferred to the interconnects remain negligible. To this end, the sacrificial Si beams contain sharp notches (see Figure 5.5) created by thinning the sacrificial Si beams through thickness as well as the in-plane direction, to trigger localized high stresses and initiating local fracture. Due to the brittle and single crystalline nature of the test-chip material the local crack(s) propagate easily all the way through the cross-section of the sacrificial Si beams. Here the beam width has been aligned along $<100>$ crystalline direction of the Si wafer. Using 3 notches in the sacrificial Si beam significantly reduces the required load for bending, while more than 3 notches would only negatively affect the test-chip stiffness during handling and clamping. Exploiting FE simulations, various deformation modes of the beam are explored to identify the lowest energy to fracture mode. If the test-chip is deformed as a whole, e.g., in tension (see Figure 5.5a), or even in-plane (see Figure 5.5b), or out-of-plane shear (see Figure 5.5c), a high amount of energy would be required to reach the fracture stress of Si ($\sim1.2$ GPa [125]) in the notches. If, on the contrary, a local point force is applied
Figure 5.5: FE simulation to identify the smallest energy to fracture the sacrificial Si beams. a) Tensile loading along x-axis, b) In-plane shear loading along y-axis, c) Out-of-plane shear loading along z-axis and d) Point load applied at the central notch at angle $\theta$ in the yz plane. The corresponding work done by the applied force/displacement till the fracture stress of 1.2 GPa is reached is displayed to the right of each sub-figure. Note that the deformation has been magnified by a factor of 10 in each sub-figure for visualization.

downwards (along the z-axis), on the central notch of a sacrificial Si beams it requires only 2 $\mu$J to reach fracture. This value is even lower if the force is applied at an angle of 60° with respect to the notch width.

To apply this localized force, a miniature load assembly is made, with razor a blade edge at an angle of 60° with respect to the notch width attached to a miniature linear x,y stage (DT12XY stage by Thorlabs) as shown in Figure 5.6a. The razor blade edge is first aligned with the central notch and then carefully inserted into the notch to conveniently fracture it in a well-controlled manner.

Additional to the mechanical characterization, the electrical characterization of such interconnect structures (or other MEMS structures), i.e., the evolution of electrical resistance with deformation, is essential. This is particularly pertinent here since a high interconnect stretch is required and a significant change in electrical characteristics with large deformation can affect the performance of the SE device. To this end, an approach for in-situ electrical characterization during mechanical loading is developed. This allows for less cumbersome testing compared to ex-situ testing using probing stations, often...
used in semiconductor industry for intermittent quasi-static testing. Furthermore, continuous electrical monitoring allows to detect interconnect failure during fatigue testing as shown below. The approach adopted here is to have miniaturized custom built (12 needles) wedge probes directly mounted on the clamps (see Figure 5.6), such that there is no relative movement between the probe needles and the test-chip during tensile testing. The probe wedges are mounted on the clamps through a miniaturized custom-built vertical manipulator. The wedge can be moved vertically with the help of two sleeves attached to each wedge, sliding along lubricated vertical shafts attached to a bottom plate, which itself is screwed to the clamp. The vertical displacement is controlled with the help of a screw of an extremely fine pitch of 200 $\mu m$ (by Thorlabs), providing a high resolution of $\sim 6$ $\mu m$ (assuming $10^\circ$ minimum rotation) (see Figure 5.6c). A miniature bearing is glued to the bottom side of the manipulator screw head to isolate the torque applied on the screw from being applied to the probe wedge. Leaf springs attached to the bottom plate ensure that the wedge and consequently the probe needles can be moved down in a well-controlled fashion and provide a vertical force to retract the wedge after the test. On the test-chip, two wires from each per interconnect fan out to a total of 12 contact pads, on either side of the test-chip, to allow 4-point probe electrical resistance measurements (see Figure 5.6b). The 4-point probe measurement is needed due to the low magnitude of the interconnect resistance for which the contact and lead resistance would have a significant influence if a 2-probe resistance measurement would have been used. The electrical measurements and data acquisition are performed using an integrated multimeter and data acquisition system (Keithley 2701) with a multiplexer module (Keithley 7700), to be able to measure the resistance of all six interconnects in parallel.

The whole setup is vacuum compatible and designed for high vacuum conditions (up to $10^{-6}$ mbar), enabling high-resolution in-situ SEM testing. The miniaturized setup can easily fit inside the SEM chamber, even with the possibility to tilt the SEM stage. Furthermore, the flat top design is instrumental for imaging at small working distances for high-resolution imaging. An image of the setup placed inside the chamber of FEI Quanta 600 SEM is shown in Figure 5.7.

To start a test, first, the test-chip is extracted from the Si processing wafer (Figure 5.8). The test-chips are suspended in the wafer with the help of polyimide tabs (see Figure 5.8b), which can be conveniently removed using, e.g., a low-cost CO2 laser cutter or a razor blade mounted on a manual manipulator. The PI tabs help to avoid the use of, e.g., mechanically aggressive saw dicing, which could damage the freestanding interconnects, or the use of an expensive dicing laser. Next, with the help of a wafer tweezers the sample is placed on the top surface of the clamp insets. The left (or alternatively right) face of the test-chip is pushed against the corresponding face of the clamp. Subsequently, UV-curable glue is applied on the top and bottom edges of the test-chip while ensuring that the glue does not rise above the top surface of the test-chip, which could impede the use of low working distance lens. Finally, the reinforcement bars (see Figure 5.6a) are placed on the clamps on the top and bottom sides and then glued to the clamps by apply air-drying silver paint.

Once the clamping procedure has been completed, the knife-edge of the beam breakage assembly (see Figure 5.6a) is heightened to be at the level as the test-chip with the help of adjustment screw and delicately moved with the miniaturized linear stage
Figure 5.6: Illustration of: (a) electrical resistance probe assembly integrated with the piezoelectric stage, and: (b) a magnified view of the electrical probes in contact with the Al contact pads on the test-chip, and (c) details of the electrical probe assembly components.
into the central notch to conveniently break it. Note that as explained earlier, only a minute force is needed to break the sacrificial Si beam. After breaking the notched silicon sacrificial Si beams, the glued temporary reinforcement bars are also removed before specimen can be initiated. This is done by locally applying a few drops of acetone with a syringe to dissolve the dried silver paste. The temporary reinforcement bars are then removed by retracting the sacrificial Si beam breakage assembly, with the slanted edge of the assembly frame, pushing the reinforcement bars backward. Now the loading can be applied to the interconnects. Since six parallel interconnects are processed in each test-chip, six of them can be simultaneously tested in one experiment. A custom-built LabVIEW interface is used to apply the loading in displacement control, enabling complex loading paths representative of interconnect loads in real devices.

5.3 Proof of principle experiments

A typical uniaxial tensile loading test is shown in Figure 5.9. Initially the loading is applied in small increments to study the onset of buckling (see Figure 5.9d). At each loading step, the interconnect is unloaded to the initial configuration to compare the
shape of the interconnect with the initial shape, in order to determine the onset of plasticity in parts of the interconnect. Beyond the engineering elastic limit (Figure 5.9e, f), which is the point where the interconnect still recovers its original shape, the interconnect is incrementally stretched further beyond the elastic limit, reaching a high axial displacement, till finally fracture occurs (Figure 5.9g).

For such structures, besides uniaxial stretching, multi-axial characterization is equally important, i.e., application of in-plane as well as out-of-plane shear loading to mimic the interconnect loading in real service conditions. Similar to tensile loading, for in-plane shear loading large displacements can be conveniently applied with the piezoelectric stages since the resolution and displacement range along all three axes is identical. As in the case of the uniaxial tensile loading, initially, small displacements steps in increments of 1 $\mu$m are applied to study the onset of buckling, while later on larger steps may be applied with subsequent unloading of the sample to its original position to study the onset of plasticity, as shown in Figure 5.10.

A unique feature compared to other small scale testing setups is the out-of-plane (shear) loading based characterization can also be easily performed. In order to visualize the out-of-plane displacement better, the setup is tilted by 5° with respect to the electron...
beam with the help of the SEM tilt stage. Since for these interconnects, there is no buckling transition occurring in the out-of-plane opening mode, the initial displacements are not chosen to be small as in the previous two tests.

The change in the electrical resistance of the samples with applied deformation is studied with the help of the 4-point probe resistance measurement assembly described earlier. The electrical characterization during mechanical loading can be performed for any type of multi-axial (xyz) loading path. To start the test, after fixating the sample and before removing the sacrificial Si beam, the electrical probes are aligned with the contact pads under an optical microscope (Figure 5.12) and constrained in the in-plane directions with the help of fixation screws. The probe tip diameter is 50 $\mu$m, while the contact pad pitch, i.e., the centre to centre distance between two adjacent pads is 640 $\mu$m, which is large enough to allow the in-plane alignment between the probes and pads manually without an additional XY translational stage. Once fixed, the probe wedge is moved downwards by rotating ultra-fine threaded screw, while visualizing the probes through the microscope. Contact between the probes and the pads manifests itself through an increased reaction moment on the screw, which is simultaneously verified.
Figure 5.10: Proof-of-principle test showing 3 of the 6 parallel interconnects loaded in in-plane (xy) shear with: (left) the initial configuration to, (middle) loaded to maximum elastic in-plane shear of 495 % and, (right) being unloaded after the maximum elastic stretch.
through the microscope. Upon contact, the probe wedge is moved further down by \( \sim 25 \, \mu m \), i.e., a quarter rotation of the screw, to ensure that all probes make good contact with the contact pads. The titanium probes allow for a 100 \( \mu m \) elastic deflection, i.e., the deflection. The electrical connection is verified by an initial resistance measurement for which the current is known to break down the native oxide layer on the Al contact pads and also stabilize the probe-pad contact junction. Finally, the cables connecting the probe card to the multimeter are chosen to be very compliant, in order to not pose any asymmetric load to the relatively compliant flexure stages. In order to measure all (six) interconnect resistances in parallel, the probe wedge cables are connected to the multimeter through a Keithley 7700 multiplexer module, capable of supporting 10 4-point probe resistance measurements. At each loading stage (multiple) resistance measurements are acquired for each of the 6 interconnects, as shown in Figure 5.13. The experiments were performed in temperature-controlled conditions with a maximum variation of 1\( ^\circ \)C, to reduce the influence of ambient temperature on the resistivity.

For application purposes, it is important to characterize the repeated usage and fatigue behaviour of stretchable interconnects and MEMS devices considering the cyclic loading in their applications. The actuation stages, with the fast reacting piezo elements and their flexure mechanism based design enable convenient fatigue testing, which is considerably more difficult with the geared, motorized systems. In order to achieve the maximum actuation frequency (for feasible testing durations), a predetermined square waveform (see Figure 5.14) is sent to the controller through the LabVIEW GUI, with which a maximum actuation frequency of 10 Hz is achieved. While this frequency is large enough for fatigue testing, the inertia effects in the compliant sample structures, such as the highly stretchable interconnects are still negligible. The electrical resistance of the interconnects is monitored as well during the cyclic loading, with open-circuit resistance serving as a convenient criterion to detect interconnect failure. More importantly, a systematic resistance change occurs due to plasticity and/or defect evolution during cyclic loading. For the results displayed in Figure 5.14, optical microscopy images are captured every 1 million cycles to complement the electrical resistance results. The results shown here are for uniaxial tensile loading of the 100 \( \mu m \) tall interconnects

Figure 5.11: Proof-of-principle tensile test results with an interconnect loaded in the out-of-plane z-direction from (a) the initial configuration to (b) a stretch of 813\%. To better visualize the out-of-plane displacement the SEM stage has been tilted by 5\( ^\circ \).
Chapter 5.

Figure 5.12: Illustration of the 4-point probe electrical resistance measurement setup, with: (a) tensile stage with integrated testing probes, under optical microscope, (b) the probe tips aligned on top of their corresponding contact pads of the test-chip and (c) a magnified view of a probe tip in contact with the corresponding contact pad.

Figure 5.13: The evolution of the electrical resistance with applied stretch, displaying only a small variation till $\sim 1000\%$ stretch (beyond the elastic limit) and a steeper increase to a maximum of $\sim 3\%$ before interconnect failure.
stretched up to 1000% (half of the plastic stretch value). All interconnects survive up to 10 million cycles without failure. The variation of the interconnect resistance lies within a range of 1%, which is mainly attributed to variations of the ambient temperature and a systematic increase or decrease of the resistance cannot be observed.

Due to the sub-micron characteristic length (thickness in this case), the material properties of the interconnect and similar microfabricated samples can greatly vary from bulk properties as a result of mechanical size-effect being active and consequently the material properties cannot be assumed a priori [24]. A material strengthening effect was also observed in the case of uniaxial tensile tests performed on the freestanding interconnects. A qualitative estimation of the strengthening was made by fitting FEM simulations to the experiments [55]. Typically, dedicated test samples with the same sizes and processing condition are fabricated and tested (e.g., by tensile test) to characterize the material behavior. In case of the freestanding interconnects another consideration needs to be made, i.e., the size effect might also be dependent on the deformation modes of interconnect, more specifically bending induced strain gradient effects [46]. Therefore, testing on the original sample is highly preferred. A stretching (uniaxial tensile test) on the freestanding interconnect sample to characterize its material behavior however entails force resolution of few nano-Newton (due to highly compliant structure), along with very delicate sample handling and load transfer functionalities as discussed in Ref.
Alternatively, inverse methods such as FEM updating (FEMU) or integrate digital image correlation (IDIC) [126] can be utilized to circumvent some of these complications. A variation of IDIC which utilizes surface topographical images (developed by Kleinendorst et al. and presented in detail elsewhere) and termed as integrated digital height correlation (IDHC) was utilized for the sample characterization. A confocal optical profilometer (Sensofar S neox) with a 100x magnification lens having a numerical aperture of 0.9 and working distance 1 mm was used to obtain topographical images. The sample were loading out-of-plane in the z direction with incrementally higher displacement-controlled loads and subsequently unloaded after each load step. Ideally the topographical maps of the interconnects in the out-of-plane loaded condition would be utilized for the IDHC. However, even at relatively small out-of-plane displacements of tens of µm, interconnect beams bend with large gradients and the topography cannot be captured even with a high numerical aperture lens. The topography of the sample in the unloaded configuration could however be captured and was recorded after unloading from each incrementally increasing displacement load. With the onset of plasticity, the sample geometry would change in the unloaded configuration as a result of accumulated plasticity and can be quantified by correlating the unloaded (deformed) shape with the original unloaded (undeformed) shape.

To enable DHC on the freestanding interconnect sample, a pattern is needed to be applied as the native pattern was insufficient. A pattern consisting of 300 nm silica nanoparticle was applied the sample using a dried micro-mist technique. Due to the scarcity of the available samples the pattern could not be optimized and only a sparse pattern (of unclustered nanoparticles) was applied. However, in this case essentially the change in curvature of the interconnect beams as a result of plasticity was required to be captured, for which a few particles over the beam length were deemed sufficient. Furthermore, a global-DHC approach [111] was used here, which reduced the requirement on pattern density due to higher order continuity. The sample was meshed with isogeometric elements with four elements over the interconnect beam length and one element over the beam width. Non-Uniform Rational B-Splines (NURBS) shape functions were used to capture the displacement fields with 2nd order shape functions in the beam length direction and 0th order shape function on the beam width direction. The in-plane and out-of-plane displacement fields obtained from application of DHC are shown in Figure 5.15a-c. As shown in Figure 5.15d the image residual has a very low average value with no systematic variations, indicating an accurate correlation. Results form the IDHC are out of scope of this work and will be presented by Kleinendorst et al. elsewhere. The experimental characterization procedure mentioned here can be utilized where similar constrains are present, e.g., in the case of freestanding interconnects or MEMS devices.

5.4 Conclusions and recommendations

This paper presented an experimental methodology for electro-mechanical characterization under in-situ microscopic observation of highly stretchable micron-sized structures such as interconnects for stretchable electronics or other MEMS applications where large multi-axial displacements and a high resolution at the micrometer scale matters. The
design of a multi-axial test setup is presented, along with sample handling and clamping procedures based on a test-chip platform on which the micron-sized samples are microfabricated. As a proof of principle, in-situ multi-axial tensile test experiments with 4-point probe electrical characterization, are shown for static conditions and high-cycle fatigue.

The setup is based on two commercially available piezo flexure position stages for actuation, a technology which has matured over the last few decades for nanopositioning systems, providing fast, multi-axial and high-resolution actuation. Typical systems also come with drivers, e.g., for LabVIEW, making it possible to build GUIs with rich and customizable functionality with relative ease. For such piezo-ceramics based systems, creep, hysteresis and thermal drift are well known issues [127]. Therefore, it is important to use piezo stages with integrated closed loop control systems. Different displacement sensing options are available for such systems, each with its own advantages and disadvantages. Strain gauge sensors typically provide a cheaper solution with low noise levels, while capacitive sensors provide better long-time stability. Therefore, when choosing piezo stages for actuation it is important to choose the appropriate sensing solution and verify the performance of the control system based on the particular test of interest, e.g., quasi-static testing, creep testing or high-temperature testing, each
with its own set of requirements. Another important aspect to consider is that the piezo stages are relatively compliant when compared with geared-motorized stages due to their flexure based design. Therefore, appropriate measures, such as discussed here need to be taken to ensure that the samples are not pre-loaded during the sample clamping procedure. Moreover, typical piezo flexure actuation stages are not designed to handle large loads, e.g., the system used here is rated at 10 N, which is enough for many small-scale testing cases, however, for larger forces a specialized piezo stage or geared systems may be needed instead. For electrical characterization with simultaneous mechanical loading, commercially available customized probe wedges were mounted directly on the clamps with a custom-built miniaturized manipulator as a simple, yet accurate probing solution. The Si based test-chip on which the samples have been fabricated is obtained by regular microfabrication processing, typically used for the fabrication of MEMS structures. The test-chip design ensures that it is rigid during processing and handling while easily set free by minute mechanical force to start testing. Finally, it is important to note that the testing methodology is not suitable for all applications, e.g., small-scale material testing which involves relatively small scale gauge displacements to sample failure and thus requiring extremely fine sample handling would require other small scale testing techniques such as ones detailed in Refs [117, 123, 128–130]. The methodology discussed here is, however, meant for small-scale tests where large multi-axial gauge displacements are required for characterization along with electrical probing and fatigue testing, which is a necessity for stretchable electronics interconnects and large displacement MEMS devices.

Acknowledgements

This work was supported by the Vidi funding of J.H. (project number 12966) within the Netherlands Organization for Scientific Research (NWO).
A bulge test based methodology for characterizing ultra-thin buckled membranes

Reproduced from:
S. Shafqat, O. van der Sluis, M.G.D. Geers, and J.P.M. Hoefnagels.

Abstract

Buckled membranes become ever more important with further miniaturization and development of ultra-thin film based systems. It is well established that the bulge test method, generally considered the gold standard for characterizing freestanding thin films, is unsuited to characterize buckled membranes, because of compressive residual stresses and a negligible out-of-plane bending stiffness. When pressurized, buckled membranes immediately start entering the ripple regime, but they typically plastically deform or fracture before reaching the cylindrical regime. In this paper the bulge test method is extended to enable characterization of buckled freestanding ultra-thin membranes in the ripple regime. In a combined experimental-numerical approach, the advanced technique of digital height correlation was first extended towards the sub-micron scale, to enable measurement of the highly varying local 3D strain and curvature fields on top of a single ripple in a total region of interest as small as $\sim 25 \mu m$. Subsequently, a finite element (FE) model was set up to analyse the post-buckled membrane under pressure loading. In the seemingly complex ripple configuration, a suitable combination of local region of interest and pressure range was identified for which the stress-strain state can be extracted from the local strain and curvature fields. This enables the extraction of both the Young’s modulus and Poisson’s ratio from a single bulge sample, contrary to the conventional bulge test method. Virtual experiments demonstrate the feasibility of the approach, while real proof of principle of the method was demonstrated for fragile specimens with rather narrow ($\sim 25 \mu m$) ripples.

6.1 Introduction

The bulge test methodology has become the standard technique for mechanical characterization of thin films [131] especially for freestanding membranes. This is due to (1) the possibility of precise sample processing facilitated by recent developments in microfabrication technology; (2) the need for minimal sample handling, which is especially challenging at small scales; (3) the relatively simple data processing for determining the
membrane stress and strain values, needed to extract the mechanical properties. The method essentially involves fixing a freestanding membrane over a small window opening and applying a known pressure to it, while measuring the resulting membrane deflection (or curvature). Various models have been developed, based on the sample geometry, to convert the pressure-deflection data to the a (elasto-plastic) stress-strain curve, which is used to determine mechanical properties such as Young’s modulus, Poisson’s ratio, residual stresses, and plasticity parameters [131–133]. During the last 30 years ample research has been devoted to improve the accuracy of the bulge test by studying the underlying assumptions such as the influence of bending stiffness [134] and initial conditions, e.g., initial film thickness and residual stress [131, 135, 136]. Among the different varieties of the bulge test method, the plane-strain bulge test is most popular [137], where it was shown that for rectangular membranes with in-plane aspect ratio larger than 4, the stress state in the centre of the membrane reduces to a plane strain condition. This means that the stress and the strain are given by [134]:

\[
\kappa_{tt} = \frac{2\delta}{a^2 + \delta^2} \quad (6.1)
\]

\[
\sigma_{tt} = \frac{P}{h\kappa_{tt}} \quad (6.2)
\]

\[
\varepsilon_{tt} = \frac{1}{a\kappa_{tt}} \sin^{-1} (a\kappa_{tt}) - 1 \quad (6.3)
\]

where \( \kappa_{tt} \) is the curvature in the transverse direction, \( a \) is half of the width of the membrane, \( \delta \) is the deflection of the apex of the membrane, \( P \) is the applied pressure and \( h \) is the membrane thickness. \( \sigma_{tt} \) and \( \varepsilon_{tt} \) denote the normal stress and strain in the transverse direction, respectively. As a consequence of the plane strain condition in the centre of the rectangular membrane, the transverse stress and the transverse strain can be related by the following constitutive equation:

\[
\sigma_{tt} = \left( \frac{E}{1 - \nu^2} \right) \varepsilon_{tt} \quad (6.4)
\]

where \( \frac{E}{1 - \nu^2} \) is the plane strain modulus. To extract the Young’s modulus \( E \) and the Poisson’s ratio \( \nu \) separately, an additional test needs to be performed, e.g., a bulge test on a circular or square membrane for which a different stress-strain equation holds, resulting in the biaxial modulus \( \frac{E}{1 - \nu} \) [99, 133].

However, such freestanding membranes often buckle as a result of processing induced (compressive) residual stresses in combination with their small out-of-plane bending stiffness, particularly for ultra-thin membranes. In some cases, the buckling is exploited as a functional part in devices, e.g., in bi-stable micro actuators [138–140]. Moreover, the buckling phenomenon in freestanding thin membranes has gained a lot of attention in Micro Solid Oxide Fuel Cells (µSOFC), where stacks of freestanding membranes serving as electrodes or solid electrolytes, are often buckled as a result of the processing. While initially buckling was considered an issue [141], recent literature suggests that it can actually be beneficial to have these membranes in a buckled state to enhance their functional properties. It has been shown that buckled membranes are mechanically more stable at elevated temperatures, i.e., lower thermomechanical tensile stresses develop
Chapter 6.

Figure 6.1: Examples of applications of buckled membranes: (a) design and placement of electrodes to locally strain-engineered ionic transport of the solid electrolyte free-standing membrane for $\mu$SOFC application [15] (reproduced with permission), (b) SEM micrograph showing ripples in a bilayer suspended graphene membrane [16] (reproduced with permission).

compared to a ‘flat’ membrane, often having significant tensile stresses already at room temperature [142, 143]. Mechanical models have been developed to exploit the behaviour of such $\mu$SOFC membranes in the post-buckling regime and consequently expand the design space into the low-stress post-buckling regime [141, 142]. Recently, controlled buckling patterns in $\mu$SOFC solid electrolyte membranes (Figure 6.1a) using ‘strain engineering’ have been employed to demonstrate local tuning of ionic conductivity of the electrolyte as an alternative of solid solution doping [144]. Furthermore, in the exciting field of graphene, where buckles and ripples are intrinsically present in the suspended configuration (Figure 6.1b), these phenomena are receiving considerable attention [145] to be exploited in various applications [146], such as improved hydrogen absorption on a rippled graphene surface (due to local curvature), for future efficient hydrogen-based fuel cells [147].

The presence of such buckling patterns in test samples, as shown in Figure 6.2, prevents the application of the conventional bulge test. All available literature to date confirms that the conventional bulge test methodology cannot be used to characterize buckled membranes since even at high pressures, buckling patterns typically do not disappear in the membranes and some stress components near the edge of the membrane stay compressive [133, 148]. Only in very few cases, the samples can be pressurized, beyond the point where the buckling pattern completely disappears, where the bulge eq. 6.1 - 6.4 might apply [133, 148]. Alternatively, the sample may deform plastically before entering the cylindrical regime. Therefore, such buckled samples are typically discarded and the processing needs to be modified to prevent the buckles to occur, in order to mechanically characterize the membranes accurately. Such processing modifications can be time consuming, costly and sometimes even infeasible or undesired. Moreover, any processing change could influence the actual properties to be determined.

Clearly there is a need for a convenient characterization methodology to determine the material properties of the buckled samples in their original (buckled) state. This paper introduces a characterization methodology for testing buckled samples, which builds on the bulge test theory and thus exploits its aforementioned advantages. The approach adopted here is to numerically model the bulge test-like pressure loading of the buckled membrane in the rippled regime, to which the meandering pattern (see
Figure 6.2: Typical ‘flat’ freestanding bulge test membrane. (Bottom) Buckled membrane meandering/telephone cord type pattern [148] (reproduced with permission).

Figure 6.2 bottom) starts transitioning as soon as even minute pressure is applied [149], to understand the mechanics and provide relations for the relevant membrane stress and strain components. To relate the stresses and strains using simple constitutive equations for extracting the material properties, regions of interest (ROI) with simplified stress states are identified and explored. Furthermore, to accurately measure the complex non-uniform three-dimensional displacement field of the buckled membranes, recent advances in bulge test methodology involving integration of Global Digital Image Correlation (GDIC) with conventional bulge test theory to [99] are exploited.

6.2 Methodology

6.2.1 Digital height correlation based bulge test

In conventional bulge test theory, the stresses and the strains obtained using eq. 6.2 and 6.3 are based on the assumption that they are homogeneous over the membrane, i.e., an infinitely long cylinder or full sphere is assumed. This assumption does not hold any more when the bending effects at the boundaries play a significant role for films with a relatively large thickness [99]. Inhomogeneous fields are also expected in the case of buckled membranes, even for a very small thickness, and in the pressure loaded case. This challenge is addressed by adopting a recently developed Digital Height Correlation (DHC) based bulge test technique [99], to capture the non-uniform 3D displacement fields.

The fundamental concept underlying this extension is to apply digital image correlation on the topographical (height) maps of subsequent load increments (pressure increments), resulting in corresponding displacement fields. Curvature fields can subsequently be computed from the displacement fields. Based on the sample geometry, relations such as eq. 6.2 can be used locally to obtain stress from curvature data, while the local strain can be obtained directly from the displacement fields. Therefore, the key assumption of a uniform cylindrical shape and uniform deformation adopted in the conventional bulge test methodology does not need to be fulfilled. Consequently, more accurate, local stress and strain fields can be obtained.
Digital Height Correlation is a variant of Global Digital Image Correlation (GDIC) and it is based on the conservation of height, instead of brightness, between a (topographical) reference image \( f(\vec{x}) \) and a corresponding deformed image \( g(\vec{x}) \), where \( \vec{x} \) is the in-plane position vector, i.e., \( \vec{x} = x\vec{e}_x + y\vec{e}_y \), with \( \vec{e}_x \) and \( \vec{e}_y \) denoting the Cartesian unit vectors. The conservation of height is written as:

\[
f(\vec{x}) = g(\vec{x} + u_x(\vec{x})\vec{e}_x + u_y(\vec{x})\vec{e}_y) - u_z(\vec{x}),
\]

(6.5)

where \( u_x \) and \( u_y \) are the in-plane displacement components in \( x \) and \( y \) direction respectively, while \( u_z \) is the out-of-plane displacement component. The image residual \( r(\vec{x}) \) is defined as:

\[
r(\vec{x}) = f(\vec{x}) - g(\vec{x} + u_x(\vec{x})\vec{e}_x + u_y(\vec{x})\vec{e}_y) - u_z(\vec{x}) + n_0(\vec{x}),
\]

(6.6)

\[
\approx g(\vec{x}) + (\vec{\nabla}g \cdot \vec{e}_x) u_x(\vec{x}) + u_y(\vec{x}) + (\vec{\nabla}g \cdot \vec{e}_y) u_y(\vec{x}) - u_z(\vec{x}) + n_0(\vec{x}),
\]

(6.7)

where \( n_0 \) is the image noise and \( \vec{\nabla}g \) is the gradient of the deformed image \( g \). The square of the image residual is minimized over the region of interest in the GDIC algorithm,

\[
\gamma^2 = \int_{ROI} r(\vec{x})^2 d\vec{x},
\]

(6.8)

where \( \gamma \) is the global residual. The 3D displacement vector is given by:

\[
\vec{u}(\vec{x}) = u_x(\vec{x})\vec{e}_x + u_y(\vec{x})\vec{e}_y + u_z(\vec{x})\vec{e}_z.
\]

(6.9)

To make this a well-posed optimization problem, the displacement field is parametrized as a sum of basis functions \( \varphi_i(\vec{x}) \) that act over the ROI and weighted by a discrete set of degrees of freedom \( \lambda_i \). The choice of the basis and shape functions is based on the expected deformation complexity. It should be noted that different shape functions may be required in the \( x, y \) and \( z \) directions to capture the deformation. It has been shown that the shape of square and rectangular bulged membranes are well described by polynomial functions \[150\].

\[
\vec{u}(\vec{x}) = \sum_i \lambda_i \varphi_i(\vec{x})\vec{e}_x + \sum_i \lambda_i \varphi_i(\vec{x})\vec{e}_y + \sum_i \lambda_i \varphi_i(\vec{x})\vec{e}_z,
\]

(6.10)

where the basis functions \( \varphi_i \) are chosen here to be polynomial functions, given by:

\[
\varphi_i = x^\alpha y^\beta.
\]

(6.11)

Subsequently, the strain fields can be computed from the extracted displacement fields using an appropriate strain definition. To determine the relevant stress components, the curvature field is obtained with the same DHC measurement. The curvature tensor \( \kappa \) is determined by taking the spatial gradient of the outward normal vector \( \vec{u} \) field as:

\[
\kappa(\vec{x}) = \vec{\nabla} \otimes \vec{u}(\vec{x}),
\]

(6.12)
where the outward normal vector is the normalized gradient of the position field \( z(\vec{x}) \), as given by:

\[
\hat{n}(\vec{x}) = \frac{\vec{\nabla} z(\vec{x})}{\|\vec{\nabla} z(\vec{x})\|}.
\]  

(6.13)

The curvature fields in transverse direction is given by:

\[
\kappa_{tt}(\vec{x}) = \vec{t}_x(\vec{x}) \cdot \kappa(\vec{x}) \cdot \vec{t}_x(\vec{x}),
\]  

(6.14)

where \( \vec{t}_x \) is a vector tangent to the membrane surface along the x direction. These curvature fields will be used to determine the local stress components. For instance, for a local region with a plane strain state (in the middle of a non-buckled rectangular membrane), the curvature can be related to the hoop stress by:

\[
\sigma_{tt} = \frac{P}{t\kappa_{tt}},
\]  

(6.15)

where \( P \) is the applied (uniform) pressure and \( t \) is the thickness of the membrane.

### 6.2.2 Experimental setup and procedure

A custom-made, gaseous pressure medium based bulge test setup (similar to the setup reported in Ref. [151]) is used (see Figure 6.3b) for the experiments. The setup consists of a sample holder block attached to a pressurized \( \text{N}_2 \) reservoir through a pressure regulator. A commercially available pressure regulator (MFCS-EZ by Fluigent) was used with a range of 0-200 kPa and a resolution of 6 Pa (0.03% of full range). The pressure regulator and consequently the bulge test setup has a fast response and settling time, which is an advantage over liquid based setups. While liquid pressure-medium setups reach higher pressures, as needed for testing stiff (thick or narrow) films or plates, they can suffer adversely from pressure build up problems in case of a minute leakage or the presence of gas bubbles in the pressure medium. This is usually not a problem in gaseous pressure-medium setups, since any drop in pressure due to leakages is directly compensated through the connected large reservoir. Furthermore, the setup is less sensitive to pressure changes due to ambient temperature variations compared to liquid based setups.

The setup is placed under a commercially available optical profilometer, Sensofar Plµ 2300. The profilometer is used in confocal mode to obtain full-field topographical images. The highest magnification lens, with a magnification of 150x and Numerical Aperture (N.A.) of 0.95 is used to obtain high resolution images of the narrow ripples which are 20-30 \( \mu \text{m} \) wide. The resulting field of view is 84x63 \( \mu \text{m}^2 \) with in-plane spatial sampling (pixel size) of 0.11 \( \mu \text{m} \), while the effective height resolution obtained is in the range of 25 nm.

Rectangular bulge test samples used for the proof of principle experiment were fabricated by deposition of a (proprietary) multi-layered stack consisting of transition metals and oxides on a mono-crystalline 650 \( \mu \text{m} \) thick silicon wafer. The freestanding window was created by wet etching from the back side of the wafer with a 1x5 mm\(^2 \) window. The sample has a total thickness of 64 \( \mu \text{m} \) and (volume averaged) Young’s modulus (estimated by volume averaging using the rule of mixtures) of 217 GPa and Poisson’s
Figure 6.3: Schematic of experimental setup for performing bulge test experiments: (a) high-resolution optical profilometry object, (b) bulge test setup, (c) pressure regulator to control pressure loading with regulator \( \text{N}_2 \) supplied with through a cylinder reservoir, (d) magnified view of cross-section of a typical bulge sample with a thin film deposited in a silicon frame with an etched window in the centre. The sample is fixed by gluing it onto the bulge tester sample holder.

ratio of 0.35. In general, application of DIC requires a good pattern providing sufficient image contrast. In case of digital height correlation this contrast is achieved by local differences in height on the sample surface. Since the native surface roughness of the samples is in the order of a few nanometres, below the resolution of the profilometer, a ‘height’ pattern is applied. For this purpose, 500 nm mono-dispersed polystyrene microspheres (by micromod\textsuperscript{®}) were used. The microspheres were applied using the drop-casting method. The particles are provided in a dense suspension and are further diluted (by a dilution factor of \( \sim 40 \)) in ethanol to achieve the required particle density on the sample surface (see Figure 6.4). A relatively dense pattern is required to capture the expected inhomogeneous displacement fields. Since the particles do not form a continuous layer, adhering to the sample surface (upon contact, without the need for an adhesive) as single particles or homogeneously distributed aggregates composed of few particles, their influence on the mechanics of the membrane is assumed to be negligible. Moreover, during the pressure loading step, the particles adhere well to the sample and no pattern change or degradation is observed, which is important for reliable application of DIC.

6.2.3 Numerical modelling

A non-linear FE model was set up to simulate the pressure loading of the test sample using the commercial FE program, MSC Marc/Mentat\textsuperscript{®}. Since in the test specimens the meander profile starts transitioning to a ripple profile as soon as a slight pressure is
applied [149] [see Figure 6.6], the analysis is focused on ripple and the cylindrical regime. As the substrate is many orders of magnitude stiffer than the film, it is modelled with rigid boundary conditions at the edges of the film. The rectangular membrane was meshed uniformly with quadrilateral 4-node thick-shell elements having three translational and three rotational degrees of freedom at each node. A mesh convergence study was performed and it was found that the solution becomes mesh independent at a size of 80×400 elements (used as the mesh size for the model).

Four batches of multi-layer test samples were available for testing, with varying material stack thickness ratios and hence different overall membrane thickness and volume averaged Young’s modulus. The focus here is on developing a methodology for obtaining thickness-averaged mechanical properties of the buckled membranes. This is also the case for conventional bulge test which also provides thickness-averaged data only. Therefore, the Young’s modulus and Poisson’s ratio in the FE model were set to 153.45 GPa and 0.351, respectively, based on a batch of samples planned for fabrication but was never produced. Note, however, that the exact properties used for the FE model are unimportant for the analysis and conclusion made and the model serves as a general platform for virtual experiments. The dimensions of the membrane in the FE model were taken as 1x5 mm$^2$ in accordance with the size of the test samples, while the membrane thickness was set to a value of 50 nm based on the median thickness batch.

To induce buckling in the membrane in-plane compressive load is required. The residual compressive stress was simulated through a thermal loading step resulting in an in-plane stress applied by the substrate (frame) on the freestanding membrane. This is caused by higher contraction of the substrate w.r.t the membrane on cooling down from a high temperature. Since the substrate was modelled by rigid membrane boundaries, the difference in coefficient of thermal expansion ($\alpha$) of the substrate and the membrane ($-2.6 \times 10^{-6} ^\circ\text{C}^{-1}$) was assigned to the membrane, to simulate this effect.

In order to avoid typical numerical instabilities at the bifurcation point (i.e., suddenly going from a flat to meandering shape in a single increment), a method used...
Chapter 6

Figure 6.5: Three-step thermo-mechanical loading procedure for gradual onset of buckling, initially as a ripple profile, finally settling into a meandering profile at the last load increment. In load step 1, the pressure is increased while temperature is kept constant to result in an inflated cylindrical profile. Subsequently, in load step 2, the temperature is decreased while the pressure is kept constant, resulting in thermal stresses that (gradually) manifest themselves as increasing residual compressive stress while the pressure is reduced (as the temperature is kept constant) in load step 3, triggering the onset of ripples. Note that the top sub-figures represent membrane deflection for the corresponding load increment with a line profile of half of the longitudinal centre line.

to model strongly buckled square membranes from Ref. [152] was adopted here. This involves bypassing the bifurcation point by adopting the three-step loading procedure illustrated in Figure 6.5. First the membrane is bulged by application of a pressure load (Figure 6.6a3). Then, compressive residual stresses are induced in the bulged membrane by applying the thermal loading step. In the final step the pressures is gradually decreased to let the membrane slowly settle down into a rippled profile (Figure 6.6a2). If the pressure is completely removed the membrane transitions from a rippled to a meandering configuration (Figure 6.6a1). Using this method, in addition to bypassing the bifurcation point, the strong and sudden geometrical nonlinearities expected at the transition from planar to meandering configuration are avoided by gradually settling from the cylindrical to the rippled configuration.

6.3 Numerical analysis

6.3.1 Simulation results

Using the three step loading procedure, explained in the previous section, the numerical model adequately captures the three different regimes seen in the experiments, i.e., the rippled, meandering regime, and the cylindrical regime. Moreover, the evolution of the rippled regime, with increasing pressure as well as the transitions between the different regimes seems are well captured. Comparison with experimentally observed regimes shown in Figure 6.6 provides a qualitative validation of the model.

In order to also quantitatively validate the FE model, the Energy Minimization Method (EMM) based model reported by Kramer et al. ([24]) is exploited here. This model was developed to describe the rippled regime for a similar rectangular membrane
Figure 6.6: Comparison between numerically modelled (a) and experimentally observed [152] (b) buckling regimes: meandering regime (a1, b1), rippled regime (a2, b2) and cylindrical regime (a3, b3), with membrane size 1x5 mm$^2$ (a) and 0.65x4.5 mm$^2$ (b). Note that similar buckling (meandering and rippled) regimes as in Ref. [152] are observed in our samples (see Figure 6.4), however, since cylindrical regime is not reached, the images are not shown here.

Figure 6.7: Centre line profile of (half of) the membrane along the longitudinal direction, extracted from FE simulation at increasing pressure values loads, with the middle profile (in blue) corresponding to $\overline{\varepsilon}_0 = -500$ and $\overline{p} = 7.48 \times 10^4$ displaying relevant parameters. The three profiles display the evolution of the buckling pattern with increasing pressure.

with width $a$ and thickness $h$, as discussed here. The model is able to predict the reduced ripple wavelength $\lambda = \frac{\lambda}{a}$, the reduced peak-to-peak amplitude $\Delta \bar{w} = \frac{\Delta w}{h}$ and the reduced ripple free amplitude $\bar{w}_{ps} = \frac{w_{ps}}{h}$ (see Figure 6.7), as a function of the reduced prestrain $\overline{\varepsilon}_0 = \frac{\varepsilon a^2}{2h^2}$ and the reduced pressure $\overline{p} = \frac{p(1-\nu^2)a^4}{Eh^4}$.

For realistic values of the applied pressure and residual stress (due to thermal loading) the results for our test specimens lie significantly outside the boundaries of the plotted results presented in Ref. [153], due to their very small thickness. In order to validate the FE model, smaller temperature-load and pressure values are applied to enable a comparison with the EMM results. Based on the values of $E$, $\nu$ and the coefficient of thermal expansion ($\alpha$), used in the FE model, a temperature difference of 0.347 °C and a pressure load of 0.0818 Pa is calculated, which corresponds to a reduced strain, $\overline{\varepsilon}_{test}$ of -500 and a reduced pressure $\overline{p}_{test}$ of 7.4811 $\times 10^4$, respectively, thus bringing the $\overline{\varepsilon}_0$ and $\overline{p}_{test}$ values within the bounds of the reported EMM results available (in Ref. [153]).
Table 6.1: Comparison of $\bar{\lambda}$, $\bar{w}_{ps}$ and $\Delta \bar{w}$ predicted by EMM [153] and by the present FE analysis.

<table>
<thead>
<tr>
<th></th>
<th>FE</th>
<th>EMM</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\bar{\lambda}$</td>
<td>0.242±0.01</td>
<td>0.250±0.005</td>
</tr>
<tr>
<td>$\bar{w}_{ps}$</td>
<td>20±0.4</td>
<td>19.7±0.2</td>
</tr>
<tr>
<td>$\Delta \bar{w}$</td>
<td>0.23±0.4</td>
<td>0.26±0.1</td>
</tr>
</tbody>
</table>

A line profile along the longitudinal axis in the middle of the membrane from the FE simulations is displayed in Figure 6.7 with the calculated reduced parameters which are given in Table 1, along with the corresponding parameters from the EMM results at the same $\bar{\varepsilon}_0$ and $\bar{p}$ values.

As can be seen in Table 1, the FE and EMM results agree for all three parameters within readout error from the FE and EMM results. However, the deviation for $\Delta \bar{w}$ is significant. On the one hand, the higher variation for $\Delta \bar{w}$ can partially be attributed to a higher readout error from the EMM plots, reflecting in the error bar in Table 1. On the other hand, the peak-to-peak amplitude varies over the length of the line profile in the FE results (see Figure 6.7), thus an average value of a relatively small magnitude is taken thereby, possibly contributing to the relatively high deviation. Based on the adequate agreement for all three values, FE model is considered valid.

6.3.2 Numerical analysis of a suitable regions of interest

As the model adequately captures the mechanics of the pressure loaded buckled membrane, it is well suited to be used as a numerical framework to test the methodology developed here, i.e., serving as a virtual experiment. Various ROIs are analyzed, where, with suitable modifications, bulge test analysis can be applied. The criteria for choosing a suitable ROI are:

1. The presence of a simplified stress or strain state, e.g., plane-strain
2. Membrane stresses can be conveniently calculated using an analytical equation, e.g., eq. 6.2
3. Membrane strains (using DHC) can be accurately determined

While the cylindrical regime may seem promising from the analysis point of view as classical simple plane strain bulge equations may be applicable there, most initially-buckled membranes (of various types) fracture before reaching the cylindrical regime, while those membranes that can sustain a high enough pressure typically go into plasticity before entering the cylindrical regime. Alternatively, the meandering regimes is not interesting form a practical point of view, as it is only accessible at minute pressures [149]. Since the rippled regime exists throughout almost the whole pressure loading cycle, only the rippled regime is here considered for further analysis. Therefore, the analysis must be performed in the ripple regime.

There are multiple reason for choosing the ROI along the longitudinal centre line of the membrane. First, it is observed in the FE simulations and the experiments that the magnitude of the ripples close to the longitudinal edge is lower than that of the same
ripples in the centre of member, see Figure 6.8. This is due to the boundary constraints provided by the edge (on the edge ripples can, of course, not form). Second, considering the third criterion of accurate determination of the membrane strains using DHC keeping the ROI always in the field of view is important. Due to symmetric deformation, an ROI in the centre of the membrane experiences the least rigid body motion, while closer to the membrane edge the large out of plane rotations result in large rigid body motion. Furthermore, these large out-of-plane rotations can affect the image residual, due to the change in effective viewing angle of the pattern, as discussed below. Finally, the boundary conditions at the edge are never as perfect as assumed in a Finite Elements simulation, especially when a dry etch is used to free the membrane, therefore, it is best to do the analysis far away from the edge, where these local boundary effects are negligible due to the well-known Saint-Venant’s principle in solid mechanics. Therefore, the most suitable ROI is identified as an ROI along the longitudinal centre line far away from the edges.

It can be assumed that the membrane is in a state of plane stress with respect to the thickness direction, since the thickness of the membrane is very small relative to the other two dimensions (with free contraction in the thickness direction). Indeed, the stress in the thickness direction due to the applied pressure is negligible compared to the in-plane stress. Furthermore, isotropic material behaviour is assumed (which is common for thin films produced with thin film deposition techniques). Therefore, the isotropic linear elastic plane stress equations (eq. 6.16 and eq. 6.17) apply for the present analysis.

$$\varepsilon_{yy} = \frac{1}{E} \sigma_{yy} - \nu \frac{1}{E} \sigma_{xx},$$  \hspace{1cm} (6.16)

$$\varepsilon_{xx} = -\nu \frac{1}{E} \sigma_{yy} + \frac{1}{E} \sigma_{xx},$$  \hspace{1cm} (6.17)

where $\varepsilon_{yy}$ and $\varepsilon_{xx}$ are the normal membrane strains in the longitudinal and transverse direction, respectively, while, $\sigma_{xx}$ and $\sigma_{xx}$ are the membrane stresses in the longitudinal and transverse directions, respectively.
Chapter 6.

Figure 6.9: Calculated transverse and longitudinal stress at the center in transverse direction and near the center in longitudinal direction, i.e., in the middle of a ripple where the curvature is zero, as a function of the applied pressure in the ripple and cylindrical regimes, revealing that the longitudinal stress stays close to zero in the rippled regime.

and transverse direction respectively.

The FE analysis shows that in the cylindrical regime the magnitude of the transverse stress $\sigma_{xx}$ is much larger than the longitudinal stress $\sigma_{xx}$ as expected for a rectangular geometry. In the last loading step, as the pressure is being reduced, at a certain stage (labelled point 3 in Figure 6.5), the longitudinal stress becomes compressive while the transverse stress is still tensile. As soon as the stress state in the longitudinal direction becomes compressive, the membrane releases the compressive stress by buckling, in the form of a rippled pattern. Given its small thickness and consequently negligible bending stiffness, the membrane does not possess the ability to support a compressive stress, which is therefore released in a buckling pattern. This phenomenon is illustrated in Figure 6.9. After the emergence of the ripples, as the pressure is further decreased, the longitudinal stress remains almost zero. The transverse stress however is still tensile and it keeps on decreasing with decreasing applied pressure. The small longitudinal stress varies over the width of a ripple due to the bending induced stress, and is therefore most compressive at the valley of the ripple while being least compressive at the peak of the ripple. At the crossover point in the middle of a ripple where the curvature is zero (marked with point C in Figure 6.11a) however, where bending effects do not contribute, the compressive stress is only due to the residual stress and $\sim 300$ times lower than $\sigma_{xx}$ (at the ripple transition point), i.e., negligible.

Accordingly, this configuration is close to uniaxial tension, i.e., there is only a non-zero stress in the transverse direction while the deformation in the longitudinal direction
Chapter 6.

is governed by free contraction. Therefore eq. 6.16 and eq. 6.17 reduce to:

\[ \varepsilon_{yy} = -\nu \frac{1}{E} \sigma_{xx}. \]  
\[ \varepsilon_{xx} = \frac{1}{E} \sigma_{xx}. \]  

Inserting Eq. (19) into Eq. (18):

\[ \varepsilon_{yy} = -\nu \varepsilon_{xx}. \]  

eq. 6.19 and eq. 6.20 provide a direct (explicit) relationship between the relevant stress and strain components, which is linear, similar to the conventional plane strain bulge test. Furthermore, unlike the plane strain bulge test, where the Young’s modulus and the Poisson’s ratio are coupled in the plane strain modulus \( \frac{E}{1-\nu^2} \), here \( E \) and \( \nu \) can be obtained separately from a single experiment.

At this point it is important to note that in the regions where the ripples have locally diminished along the longitudinal direction, e.g., point A in Figure 6.7, neither plane strain, nor plane stress condition holds with respect to the in-plane directions due to which eq. 6.16 and eq. 6.17 cannot be reduced to a simpler form to yield \( E \) and \( \nu \), even in a coupled form. Therefore, the analysis is best performed at the true center of the membrane (i.e., the middle in longitudinal direction), where the ripples disappear for the highest pressure, making the above-mentioned analysis valid for the largest pressure regime (another reason is that the rigid body motion is lowest at the true center as discussed above).

The validity of the uniaxial tensile state (eq. 6.19 and eq. 6.20) is assessed with a virtual experiment. The stress and strain state is extracted from a node at the cross-over (zero curvature) point (denoted as point C in Figure 6.11) in the ripple in the centre of the membrane. The Young’s modulus is extracted from the gradient of the \( \sigma_{xx} - \varepsilon_{xx} \) plot (see Figure 6.10a), whereas the Poisson’s ratio is extracted from the \( \varepsilon_{yy} - \varepsilon_{xx} \) plot (Figure 6.10b). The Young’s modulus (with a value of 154.5 GPa) is extracted with an error of 0.68 %, while the resulting Poisson’s ratio (with a value of 0.357) reveals an error of 1.7 % with respect to the respective reference (input) values in the FE model.

Note that eq. 6.15 is still valid in the rippled regime, allowing to relate the local curvature and applied pressure to the transverse stress at the crossover point. This is the case, since at the crossover point the local curvature in the longitudinal direction is almost zero, whereas \( \sigma_{xx} \) is negligible \( \text{w.r.t} \ \sigma_{xx} \) (as illustrated in Figure 6.9), thus the pressure applied to the face of the membrane is entirely balanced by the transverse stress. The validity of eq. 6.15 in the rippled regime is numerically assessed by computing the transverse stress \( \sigma_{xx} \) from \( \kappa_{tt} \) (extracted from the FE simulation) and the applied pressure \( P \) (known in the simulation). The resulting value was plotted against the transverse strain \( \varepsilon_{xx} \) extracted from the FE simulation to determine the Young’s modulus, which has an error of 1.4 %.

Based on the relatively small errors in the extracted values, it is concluded that the proposed method is promising. This analysis sets the basis of the characterization methodology. In a real experiment, \( \sigma_{tt} \) can be determined using eq. 6.12 - 6.15 from the position field in the deformed configuration. While \( \varepsilon_{yy} \) and \( \varepsilon_{xx} \) can be obtained from the 3D displacement fields using eq. 6.26 - eq. 6.29, as will be shown in the next section.
Figure 6.10: Stress and strain values extracted from FE results in the rippled regime to obtain Young’s modulus ($E$) and Poisson’s ratio ($\nu$): (a) Transverse stress ($\sigma_{xx}$) vs. transverse strain ($\varepsilon_{xx}$) with a linear fit to provide $E$, and (b) Longitudinal strain ($\varepsilon_{yy}$) vs. transverse strain ($\varepsilon_{xx}$) with a linear fit to provide $\nu$.

Furthermore, for the membranes that have not ruptured before reaching the cylindrical regime at higher pressure, this method can be used in conjunction with the plane strain equation. In that case, the same ROI analyzed in the rippled region can also be analyzed in the cylindrical plane strain state.

Practical application of DHC to the bulged buckled membranes requires highly accurate determination of 3D displacement fields as well as the position field in the reference configuration. This procedure is first tested in a virtual experiment. To this end, the displacement fields are extracted from the FE simulation in a typical ROI, from peak to valley in the longitudinal direction (marked in Figure 6.11a point A to B).

These displacement fields and the resulting analysis correspond to the membrane mid-plane. In DHC however, only the membrane surface (top or bottom, see Figure 6.11a) can be analysed and thus only displacements at the surface are determined. This causes a discrepancy of less than 0.5 % between the transverse displacements extracted from the mid-plane and the outer surface (top/bottom) plane in the FE simulation. However, for the longitudinal displacement, due to the significant curvature in that direction, the displacement at the surface is found to be considerably different with respect to the displacement at the mid-plane. Nevertheless, the average of the longitudinal strain over a full and half ripple width (as considered here), on the top surface should be the same as on the mid-plane, since the difference in the length of a line profile from the peak to the valley of the ripple in the top surface, middle and bottom planes (Figure 6.11a) will be negligible, especially for small strains. This has been validated by the FE simulation. Therefore, the average longitudinal strain over half the ripple width is computed, and plotted against the transverse strain in Figure 6.13a, resulting in a value of Poisson’s ratio with an error of only 0.3 %.

Another important observation can be made from Figure 6.12b, which shows that the longitudinal displacement is in the order of a few nanometres. The best optical in-plane resolution is approximately 250 nm. Even considering a subpixel resolution of $\sim$0.01 pixels that can be captured with DIC techniques, the longitudinal displacement cannot be captured accurately due to the physical resolution limit. Furthermore it has been discussed in literature [154] that for large out-of-plane rotations (high curvature
changes), a systematic error in the displacement fields might be introduced. To address this shortcoming, the longitudinal (in-plane) strain is disregarded and only the rigid body motion in the longitudinal direction is used, together with the height displacement field for determining the longitudinal membrane strain $\varepsilon_{yy}$, see Figure 6.13b. The resulting Poisson’s ratio still has an error limited to 0.3%. Therefore, only the rigid-body motion will be included as the displacement description in DHC to capture the longitudinal displacement field.

## 6.4 Proof of principle experiment

Here, the results of a successful test serving as a proof of principle experiment to show the feasibility of application of the method in a real experiment are shown. Pressure increments of 2.5 kPa were applied to the sample and kept constant while the topographical images were acquired with the profilometer.

An ROI shown in Figure 6.4 from the peak to valley of a ripple is selected for DHC analysis. As discussed in Ref. [99], a limited number of degrees of freedom (dofs) has to be used to capture the deformation kinematics accurately. Too few dofs restrict the kinematics while too many can cause the correlation to diverge while making it sensitive
Figure 6.12: Displacement fields and line-profiles extracted from FE simulations from peak to valley along the longitudinal axis in the rippled regime: (a) out-of-plane displacement field ($u_z$), (b) longitudinal displacement field ($u_y$), (c) transverse displacement field ($u_x$), (d) mean-subtracted line-profiles of $u_y$ and $u_z$ at $y = 33\, \mu m$.

Figure 6.13: Graphs of the longitudinal strain ($\varepsilon_{yy}$) as a function of the transverse strain ($\varepsilon_{xx}$) linear fits to extract the Poisson’s ratio, where $\varepsilon_{yy}$ is averaged over a line-profile that ranges from ripple peak to valley, for (a) $\varepsilon_{yy}$ obtained from both $u_y$ and $u_z$ (from FE simulations) and (b) $\varepsilon_{yy}$ obtained only from $u_z$.

to image noise. Here, the expected order of displacements is determined from the out-of-plane and transverse displacement fields extracted from the FE simulations (Figure 6.12), defining the initial set of shape functions to be used for the DHC analysis. More shape functions are subsequently added to optimize the correlation until the residual fields
The number and order of shape functions needed to capture the out-of-plane displacement is significantly higher than those required for the in-plane (transverse) displacement field, see eq. 6.21 and eq. 6.23. This is expected since the predicted (from FE simulations) displacement field in the height direction is considerably more complex than the in-plane (transverse) displacement field, see Figure 6.12. This is not a problem since the optimization of the dofs related to out-of-plane shape functions is more robust than the in-plane shape functions, since the out-of-plane displacement field directly affects the image residual, whereas, the in-plane displacement field affects the image residual through the (noisy) image gradient (see eq. 6.7).

The quality of the correlation in DHC is typically assessed by analysing the residual fields. Figure 6.14a, and Figure 6.14b show the residual before and after correlation, respectively. The signature of the pattern is vaguely visible after convergence, due to the increasing rotation of the membrane during bulging, resulting in an altered viewing.
angle. However, the residual field has a very low RMS value indicating that the correlation was successful. Moreover, a good qualitative agreement can be seen between the shape of the displacement fields extracted from the simulations (Figure 6.12) and the displacement fields obtained from DHC analysis (Figure 6.15), confirming that the deformation kinematics has been adequately captured.

Subsequently, the required membrane strain $\varepsilon_{xx}$ and $\varepsilon_{yy}$ are calculated from the obtained 3D displacement fields. This involves determination of strain with respect to a non-flat (rippled) initial configuration ($f$). The stretch ratios and principal logarithmic strains resulting from the deformation are given by:

\[
\varepsilon_{xx} = \ln (\lambda_t) \\
\varepsilon_{yy} = \ln (\lambda_l)
\]  

(6.24)  

(6.25)

where $\lambda_t$ and $\lambda_l$ are the stretch ratios ($\lambda = \frac{\text{current length}}{\text{original length}}$) in the transverse and longitudinal directions, respectively, and given by:

\[
\lambda_t = \frac{L^g_t}{L^f_t} \\
\lambda_l = \frac{L^g_l}{L^f_l}
\]  

(6.26)  

(6.27)

$L^g_t$ and $L^f_t$ are denoted in Figure 6.11b and defined as:

\[
L^g_t = \sqrt{(\Delta x (x + u_x))^2 + (\Delta x (Z_f + u_z))^2} \\
L^f_t = \sqrt{(\Delta x (x))^2 + (\Delta x (Z_f))^2}
\]  

(6.28)  

(6.29)

where $Z_f$ is the height map of the reference image (i.e., $f(\vec{x})$), while $\Delta x (\cdot)$ denotes an operator that acts on a field to produce the finite difference of the field in the x-direction. Since the reference surface is expected to be non-smooth due to the applied pattern and its intrinsic surface roughness, the height map and resulting displacement fields will be non-smooth. A surface polynomial fit of the reference image is used to smoothen this field. Similarly, for determining the surface normal and curvature values using eq. 6.13, a smoothed position field $(Z_f + u_z)$ is adopted instead of the deformed configuration (topographical image) $g(\vec{x})$. Likewise, $L^g_l$ and $L^f_l$ are given by:

\[
L^g_l = \sqrt{(\Delta y (y + u_y))^2 + (\Delta y (Z_f + u_z))^2} \\
L^f_l = \sqrt{(\Delta y (y))^2 + (\Delta y (Z_f))^2}
\]  

(6.30)  

(6.31)

where $\Delta y (\cdot)$ is the operator that produces the finite difference of the specified field in the y-direction. Subsequently the transverse stress is determined using eq. 6.12 - eq. 6.15. The transverse stress $\sigma_{xx}$ is plotted again the transverse strain $\varepsilon_{xx}$ in Figure 6.16a resulting in a Young’s modulus of 211±8 GPa. In order to obtain the Poisson’s ratio, the mean longitudinal strain $\bar{\varepsilon}_{yy}$ (instead of $\varepsilon_{ll}$, as discussed in the previous section) is
Figure 6.16: Stress and strains obtained from DHC results plotted to determine Young’s modulus $E$ and Poisson’s ratio $\nu$: (a) transverse stress ($\sigma_{xx}$) vs. transverse strain ($\varepsilon_{xx}$) with a linear fit to obtain the Young’s modulus ($E$) and (b) mean longitudinal strain ($\bar{\varepsilon}_{xx}$) vs. transverse strain ($\varepsilon_{xx}$) with a linear fit to obtain the Poisson’s ratio ($\nu$).

plotted against the transverse strain $\varepsilon_{xx}$ resulting in a value of 0.36±0.12. These values are in adequate agreement with the predicted (volume averaged) Young’s modulus and Poisson’s ratio of respectively 217 GPa and 0.35, respectively. Therefore, the proposed methodology can be applied to obtain the elastic properties from a buckled membrane.

6.5 Conclusions and recommendations

It is well known from literature that conventional bulge testing, which is frequently used to characterize freestanding membranes, does not apply to the particular class of buckled membranes. In this paper, the conventional bulge test methodology has been extended to characterize the elastic properties of buckled membranes. This has been achieved by developing a validated FE based numerical model, which captures the complex mechanics of the (pressure-loaded) buckled membrane. A recently developed DHC approach has been employed to obtain local, complex 3D displacement (strain) and curvature fields.

Interestingly, the virtual experiments revealed that, embedded in the seemingly complex ripple configuration, a simplified state of uniaxial stress exists in the transverse direction. This implies that the transverse stress can be directly related to the transverse strain, yielding Young’s modulus. Poisson’s ratio can be extracted directly from the ratio of the longitudinal strain and the transverse strain. This is a significant advantage over the conventional bulge test theory where Young’s modulus and Poisson’s ratio are intrinsically measured in a coupled manner, either through the plane strain modulus or the biaxial modulus. As a result, tests on two sample geometries, typically rectangular and square shape, are needed to obtain Young’s modulus and Poisson’s ratio independently.

The numerical model also showed that applying DHC to measure the displacements in the longitudinal direction would require high-order shape functions and a displacement resolution that cannot be achieved with an optical system. This problem was solved by exploiting the fact that the contribution of the ‘in-plane’ longitudinal displacement to the longitudinal strain is negligible, allowing to obtain an accurate value of Poisson’s ratio by only considering the height displacement to determine the longitudinal strain.
Proof of principle experiment clearly show that the method is applicable on real samples, even with rather narrow ripples dimensions (20-30 $\mu$m) and only few data points, as the fragile samples were very susceptible to deformation induced failure. Residual maps indicate proper convergence and the shape of the displacement fields captured with DHC adequately match the predicted displacement fields extracted from the FE simulation, confirming that the buckled membrane kinematics are being properly captured. Moreover, the resulting values of Young’s modulus and Poisson’s ratio are consistent with the expected values based on the material stack.

As both the stress and strains are measured under uniaxial tension, the method is not necessarily confined to the elastic regime and should work as well in the plastic regime (yielding plasticity parameters), if the specimens would plastically deform. Furthermore, by applying cyclic pressure loading, fatigue testing, as suggested in Ref. [155] for plane strain loading, can be applied with uniaxial tension. Using a feedback loop to maintain a constant membrane stress, as suggested in Ref. [156], a creep test in uniaxial tension can be performed.

Acknowledgements

This work was supported by the Vidi funding of J.H. (project number 12966) within the Netherlands Organization for Scientific Research (NWO). The authors would also like to greatly thank Johan Klootwijk from Philips Research for proving the samples, Jan Neggers for sharing his digital image correlation code, Roel Donders for upgrading the bulge test setup and help with experiments and Marc van Maris for technical support in the laboratory.
Conclusions and outlook

7.1 Conclusions

The objective of this thesis was to develop highly stretchable, miniaturized (micron-sized) interconnects that can stretch beyond 1000% which are compatible with industrially standardized microfabrication-based processing methods to enable high-density, highly stretchable electronics applications.

7.1.1 Interconnect design and performance

To this end, a freestanding slender beam-based interconnect was developed. The interconnect consists of two corner torsion beams and the remaining inner beams flex in bending providing high stretchability to the structure. This interconnect is termed here as the Rotation Out-of-plane Elongation (ROPE) interconnect.

In-situ electro-mechanical characterization of the interconnects revealed a maximum elastic stretch of $\sim 2000\%$, and an ultimate (plastic) stretch beyond 3000% with $<0.3\%$ electrical resistance change and a cyclic response of $>10$ million cycles with $<1\%$ electrical resistance change. This is the highest value of stretchability utilizing metallic interconnects as reported in a recent literature review [4]. Furthermore, the interconnect offers a very compact initial footprint area, important for high density applications, while the measured resistance results in a sheet resistance of $0.11 \ \Omega sq.^{-1}$, which is the lowest value when compared to the information on metallic interconnects reported in Ref. [3], where the sheet resistance values for various types of stretchable interconnects.

An integral part of the design was its manufacturability, i.e., it should be possible to be manufactured as a part of a standard microfabrication processing scheme, to enable miniaturization of interconnects as well as the future routine integration of the highly stretchable interconnects with the CMOS-processed functional electronics. To this end, the interconnects were designed to be fabricated using the ‘polymer-last’ [23] approach by project partners at Philips Research.

The slender beam-based structure provides a relatively simple design for which a simple analytical expression relates the geometrical parameters such as beam width, thickness, length, etc. to the interconnect stretchability, without the need for geometry specific FEM modelling. Thus, once the system requirements such as interconnect stretchability, electrical resistance, footprint area, etc. are known, the interconnect design geometry (beam dimensions and spacing) can be easily determined using a simple analytical equation.
7.1.2 Freestanding interconnect multiaxial stretchability

In real applications, however, the interconnects not only experience uniaxial tension but they can also be subjected to complex multiaxial deformation. This was highlighted here through a FEM simulation of a stretchable detector array going from a flat to a hemispherical configuration, showing that the interconnects at the periphery of the circular detector array are subjected to significant shear strains. Therefore, emphasis was put on characterizing the multiaxial loading performance of the interconnects as well. The multiaxial loading performance of the ROPE interconnect was compared with the help of FEM simulations to two other freestanding geometries: the traditional serpentine interconnect made freestanding and a new freestanding non-buckling interconnect. The results show that the ROPE interconnect exhibits a high stretchability with an omnidirectional response, i.e., similar levels of stretchability in in-plane and out-of-plane loading along all angles, which makes the mechanical response of the interconnect very reliable. The serpentine interconnect also provides an omnidirectional response but at approximately an order of magnitude lower stretchability than the ROPE interconnect. The non-buckling interconnect, however, while providing an even higher stretchability in the uniaxial loading direction, fails to perform even at low magnitudes of in-plane shear. Multiaxial loading experiments were performed on the ROPE interconnect samples and an adequate agreement was found with the FEM simulations. The main conclusion of the analysis is that even for an application such as the inflatable detector array, where uniaxial loading of the interconnect is intended, significant multiaxial strains are present, ruling out the non-buckling interconnect. For a more complicated functional island placement, e.g., in a non-uniform grid, the requirement on multiaxial stretchability would be even higher. In such a case, a buckling type interconnect (e.g., serpentine or ROPE) can provide the desired omnidirectional stretchability, while the ROPE interconnect provides the high stretchability as well, required in high density applications due to the improved design.

7.1.3 Experimental testing methodology

Due to the small-scale nature of the samples, a delicate sample handling and testing approach had to be developed to enable the above mentioned characterization of the µm-sized freestanding samples. A miniaturized tensile tester was developed which allows application of multiaxial loading of the samples with a resolution of \(\sim 1\,\text{nm}\) and a range of 400 nm in the x, y and z directions. The tensile tester was integrated with electrical probe wedges which allow 4 probe electrical resistance measurement simultaneously with mechanical loading. Furthermore, since piezoelectric stages were utilized here for actuation, cyclic loading could be applied for fatigue testing with a maximum of 10 Hz loading at maximum displacement. Furthermore, due to the flat-top design, high resolution optical microscopy with a working distance as low as 300 µm could be used. Finally, the tensile tester was designed to be compatible with high-vacuum SEM.

Alongside the tensile tester, a smart test chip on which the samples are processed was designed to ensure that the samples do not experience any spurious loading during sample processing and handling prior to mechanical testing. Based on insights from FEM simulations, notches were introduced in the monolithic silicon chip, which ensure a high stability of the chip. Once the test chip has been clamped in the tensile stage a
small controlled localized applied force results in breaking two notched sacrificial beams, releasing the chip for actuation of the interconnect samples. Six parallel interconnects could be processed on one chip allowing parallel testing of multiple samples in the same test. The developed testing approach may be utilized for other small scale-testing cases, where high (global) strains need to be applied to a structure.

7.1.4 A novel DIC pattern technique for ROPE interconnects and other delicate samples

An on-sample characterization approach based on integrated digital height correlation (IDHC) was adopted in the parallel project, focused on development of numerical methods for characterizing the highly stretchable interconnects. As the microfabricated ROPE samples do not possess enough (natural) pattern necessary for the application of digital image correlation (DIC) and consequently IDHC, the ROPE samples needed to be patterned. The delicate nature of the ROPE samples makes it extremely challenging to pattern them and existing techniques in the literature are inadequate. Similar to the ROPE sample other small-scale delicate samples suffer from the same problem, limiting the use of DIC in analyzing such specimens in general. To solve this problem, a new patterning technique was developed here that is based on the generation of a very fine mist of nanoparticles (50-1000 nm in diameter) from a suspension in a volatile solvent. The mist is dried up to create a flow of dry, individual particles, which are subsequently delivered to the sample surface, resulting in an ultra-delicate pattern application process. Several cases of delicate specimens were patterned using this technique, without any visible damage. Moreover, the ROPE specimens were patterned for IDHC based characterization. The sample were tested in out-of-plane loading under an optical profilometer and the resulting displacement fields obtained from digital height correlation (DHC) are presented here. The generic patterning technique developed here can be utilized for a wide range of delicate samples and is of interest in particular for patterning MEMS structures for analysis with DIC.

7.1.5 A bulge test based approach for analysing thin films with compressive and tensile residual stresses

In the design of microfabricated highly stretchable devices, e.g., with the polymer-last technique, the mechanical behavior of the constituent thin films needs to be known. The Bulge test is a well-known technique to characterize thin films. However, often such thin films include compressive residual stresses which induce buckling, rendering the conventional bulge test method inapplicable. Here, a new bulge test based technique was developed to characterize freestanding thin-films under compressive residual stresses. As a result of the development made here, the bulge test method can be used in the project to test the whole range of thin-film materials, including the regular flat samples under tensile residual stress as well as buckled samples resulting from compressive residual stresses. This makes the bulge test method ideal for testing various thin-film material options available which can be used for the interconnect as well as the various thin-film parts required in an SE device. Due to time limitations, the method could not be utilized to test and explore different materials. However, currently it is being utilized in
a parallel project that focuses on the design and microfabrication of an SE demonstrator device based on the here-developed highly stretchable interconnects.

7.2 Outlook

The work done in this thesis shows the proof of principle operation and reliability characteristics of a miniaturized highly stretchable interconnect design. However, to integrate such interconnects in actual commercial applications a number of challenges remain to be addressed. Moreover, deeper analysis and testing can help in better utilization of these interconnects. Some key aspects in this regard are discussed below:

7.2.1 SE Device Packaging

One of the key challenges with realizing SE devices is device packaging. This is even more important and challenging in the case of SE devices with freestanding interconnects, as embedded or substrate-adhered interconnects are more stable and protected from the environment. Similar challenges were faced and solved during the development of MEMS devices, that have similar moving parts which need to be protected from the environment, including harsh operating conditions [157–159].

In SE devices, a strategy developed for large-area SE devices with freestanding interconnects involves the freestanding interconnects suspended in a cavity between a top and a bottom elastomer layer while the cavity is filled with a dielectric fluid such that upon contact on the top layer, load is not locally or directly transferred to the freestanding interconnect [26]. For µm-sized interconnects however, the dielectric liquid would most likely damage the interconnects, due to the significance of the forces applied by the liquid on the interconnect at the small-scale relative to the stiffness of the interconnect. However, at the small-scale in presence of sufficient space between the elastomer layers and the interconnect, e.g., tens of µm, it is very difficult for the elastomer layers to contact the interconnect upon an external local contact force. Therefore, such a such a fluid is likely not necessary.

A top layer or capping layer is, however, necessary to protect the SE device. With the polymer-last approach an elastomer substrate can be easily integrated in the process flow as demonstrated by Savov et. al. [39] and Pakazad, et. al. [160]. Application of the top capping layer requires additional steps and can be done by first increasing the height of the silicon islands, e.g., with polyimide (PI) posts and then applying an elastomer layer by lamination. Alternatively, another wafer with dummy silicon islands processed on an elastomer substrate with the same configuration as the active islands wafer could be attached by wafer bonding, typically utilized in MEMS packaging.

7.2.2 Freestanding interconnect Passivation

Another important challenge along with packaging is the passivation of the interconnects. Typically, exposed IC interconnect layers are passivated, i.e., covered, with a PI or Paralyne conformal coating to act as a dielectric barrier to avoid short circuits as well as a moisture barrier to avoid corrosion. Such conformal coatings are however typically a few µm thick which can impede the onset of buckling in the freestanding interconnect
depending on the interconnect thickness. For instance, if the ROPE interconnect with a thickness of 300 nm (as used here) would be coated with 1 µm of passivation coating on either side, it will most likely prevent the interconnect from buckling, resulting in the interconnect opening in in-plane deformation, leading to plasticity at a very low global stretchability. Therefore, thinner passivation coatings need to be explored. One possible solution in the case where pure aluminium is utilized (as used here), would be to evaluate if the native oxide layer of aluminium can serve as an adequate passivation layer, especially if the interconnects are protected on both the top as well as the bottom sides with elastomer layers. In this respect, further growth of the native oxide can be explored to enhance the passivation effect as discussed in Ref. [161]. Alternatively, for other interconnect materials, deposition of an ultra-thin passivation layer of a few nm should be pursued.

7.2.3 Electromigration, thermal migration and stress migration based degradation of interconnects

Electromigration might play a role in defining the reliability of miniaturized freestanding interconnect due to the relatively small thicknesses involved. Similarly, thermal gradients due to joule heating or stress (gradients) due to interconnect stretching can result in migration effects and ultimately interconnect failure. These effects are however highly dependent on the system requirements, such as interconnect current densities, interconnect material, etc. [162]. Therefore, such effects need to be studied using electro-mechanical testing similar to the tests discussed in chapter 5. It should be noted, however, that the dimensions of the back end of line interconnects (0.3x2 µm$^2$) discussed here are still large compared to the front end of line nm-sized features that are known to be susceptible to the above-mentioned migrations effects.

7.2.4 Analytical buckling analysis of ROPE interconnect

While the interconnect geometrical design parameters can be rather easily determined from the interconnect requirements (stretchability, electrical resistance, footprint area, etc.), it is not straightforward to precisely predict whether the interconnect geometry will buckle and thus provide the desired stretchability, especially when approaching a square beam cross-sectional area. In this work, finite element simulations were employed to confirm whether the structure would buckle or not, before having the designs processed. However, this process can be tedious if a large design space needs to be evaluated. An analytical analysis of the interconnect buckling can be an alternative approach. The critical buckling strain for a particular geometry can be derived as a function of the geometric and material parameters of the interconnect. Furthermore, an analytical description of the maximum pre-buckling strains in the interconnect as a function of the geometrical parameters can be used to determine whether buckling in the structure occurs before the onset of plasticity.

7.2.5 Further miniaturization

To enhance the stretchability and reducing the footprint area, further miniaturization should be considered to reduce the interconnect beam width, thickness and gap. A
higher resolution lithography technology needs to be utilized compared to the one available and used here. Naturally, upon further miniaturization, the electrical resistance of the interconnect will increase and migration effects discussed earlier become more important. Furthermore, interconnect warping due to process induced residual stress would likely increase with a reduced beam width and smaller interconnect thickness and excessive warping could be problematic in interconnect operation and strategies to manage residual stress would need to be explored.

7.2.6 Size effect investigation

As discussed in chapter 2, results from ROPE interconnect experiments suggest that that mechanical size-effects are likely active due to the sub-micron interconnect thickness. This generally provides strengthening to the material and thus an enhanced stretchability. An in-depth investigation into the identification of the possible mechanisms can greatly help in exploiting this strengthening effect reproducibly and possibly enhancing the interconnect stretchability even further.

7.2.7 Utilization in 2.5/3D permanently deformed circuits

One possible utilization of the irreversible (plastic) stretchability of the ROPE interconnect can be in 2.5/3D circuits [11], where an initially flat-processed circuit processed on a mouldable substrate is permanently moulded into a 3D shape, e.g., by thermoforming. It was shown in chapter 2 that the ROPE interconnect can be stretched (plastically) into a straight wire (or a ‘ROPE’), resulting in an irreversible stretchability of 3000%. Since such circuits are morphed into 3D shapes, such as a hemisphere, the highly stretchable ROPE interconnects can help in boosting the device fill factor. Furthermore, the key challenge in the utility of freestanding interconnects, i.e., packaging, is already met in the case of permanently deformed 2.5/3D circuits, since during the moulding process the interconnects can be encapsulated by the molding material. However, the influence of mouldable substrate on the ROPE interconnect buckling would need to be investigated. It is of essence that during the molding process the ROPE interconnects can buckle out-of-plane to provide the high stretchability.
Bibliography


Publications

Below is a list of publications related to the thesis


Other publications


Word of Thanks

During the course of this long Ph.D. journey, which seems to have gone by in the blink of an eye, I had the opportunity to work with and meet a lot of people, whom I would like to acknowledge here and pay my sincere gratitude to. First of all, I would like to thank my supervisors, Marc, and Johan for giving me this opportunity. Marc, thank you for all your help, support and guidance during the project. It still amazes me how you can be responsible for so many things and still respond to emails so fast, even in late evenings and your holidays. Johan, I want to sincerely thank you for all the freedom you gave me during the Ph.D., without which I would not have made it. On the other hand, you were always there to support me when I needed it, whether on technical, administrative or personal issues, especially during the tough times at the end of the journey. While we have very different personalities, still we could work together very well, starting from my Master’s thesis until the end of the Ph.D. So, I want to thank you for that experience. Olaf, although I wasn’t your student anymore after finishing my Masters, yet you always had time for me when I needed advice. Thank you for all your support; you are a great mentor. Ronald, I wish I had the opportunity to work more closely with you, yet the limited interactions we had were a highlight of my Ph.D. Your enthusiasm was infectious and every meeting with you was a motivation booster. I really want to thank you for that and your support. Angel and Shivani, getting to know both of you was probably the best part of my Ph.D. Working with both of you, which was mainly during the beginning of the Ph.D. was a great experience. I got to learn a lot from both of you and without your commitment, hard work and expertise we would not have been able to achieve these results. Furthermore, I want to thank you for your friendship which blossomed from our work relationship and all of your support during this time that I have known both you. I wish to have the opportunity to enjoy many more of our ‘stretchable-victims’ get-togethers in the future. Sandra, while we didn’t end up working very closely together, I would like to thank you for the pleasant interactions we always had. It was great to have you as a conference buddy, especially during the SEM conference road trip, together with Johan. I wish you the best of luck for the future. Lambert, I got to learn a lot from you in the multiscale lab during my Master’s because of your vast knowledge and mentoring personality. I wish that we had more of an opportunity to work together during this project, yet the limited interactions we had were great, so thank you for that. Lucien, I would like to thank you for all your help in the workshop and most importantly your enthusiasm. Marc (from the lab), thanks for your help and support in the multi-scale lab, especially at the end, and for bearing me in the lab for all these years. Alice and Rachael, you two were the best secretaries and the coolest people to have around during all the events. The MoM group is lucky to have you as the mother hen(s).

I would really like to offer my sincere thanks to my Ph.D. defense committee for taking out the time to read my thesis, offer comments and being a part of the defense.
Anastasija, thanks for patiently going through all the iterations for the cover and for the great job.

I want to thank all the people from the amazing 4.13 office: Stefan, Majid, Maqsood, Steyn, Mirka, Siavash, Mash Mohsin, Vahid, Luv, Rody, Sandra, Jim, Nilgoontje, Mary, Emanuela, and Jasna. You all made it fun to come to the office every day. It was a beautiful time I got to spend with you in our office family and I will cherish all those memories, especially the Sinterklaas parties, the after 6 talks and more. To all the people who joined the office while I was in the process of finishing, I wish the same for all of you. Also, I want to especially thank Jim and Rody for patiently answering all my ‘numerical’ questions. I would like to thank ‘our batch’ of MoM PhDs/Postdocs for the nice and friendly atmosphere: Andre, Ashwin, Hector, Awital, Franz, Aslan, the yin and yang Varuns, Priscilla and others. Prakhyat and Varuns, thanks for forcing me to speak Hindi/Urdu during our fun and chaotic meetups, although my lungs are probably not as happy from all that secondhand smoke.

I guess every Ph.D. journey has its difficulties and for me, most of those came at the end. I honestly could not have gone through those tough times without the help and support of some amazing people that I had around me at that time and I cannot thank you all enough for that. Mirka, you practically adopted me as a kid and with you and Francesco it felt like I was with my own family. The world needs more people like you. Varun, you are one of the most patient and selfless persons I have met and the world also needs people like you, although you really need to stop with scaring people multiple times a day and with all the made-up stories, the world doesn’t need that. Andre, it was great to have you as a buddy all the way from Masters through Ph.D., it feels like we grew up (old) together. Also, Priscilla, Prakhyat, Nilgoontje, Varun Raj, Maqsood, Siavash (Sishmish), Rody, Kaipeng, and many more, thanks for being there for me and for being great friends.

I want to thank my parents and my brother for all the love and support. Lastly, Mary, you have been my rock in this journey. As things got tougher your love and support only grew stronger and for that and more, I cannot thank you enough.
Curriculum Vitae

Salman Shafqat was born on 17-06-1988 in Bhaun, Pakistan. He obtained his Bachelor’s in Mechatronics Engineering from National University of Science and Technology, Pakistan in 2010. He received the TSP talent scholarship in 2010 to pursue a Master’s degree in Eindhoven University of Technology (TU/e). He finished his Master’s thesis in the Mechanics of Materials group in 2014 at TU/e under the supervision of Dr. Olaf van der Sluis and Dr. Johan Hoefnagels. He continued in the same research group at TU/e to pursue a PhD on a project titled ‘Stretching the limits of IC stretchability’ under the supervision of Dr. Johan Hoefnagels and Prof. Dr. Marc Geers. Since September 2019 he is employed at Philips as a Research Engineer through Bright Society.