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Maik Rudolf Johann Scherer

Double-Gyroid-Structured Functional Materials

Synthesis and Applications

Doctoral Thesis accepted by
University of Cambridge, UK

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*Insanity: doing the same thing over and over again and obtaining different results.*¹

¹ Original quotation by Albert Einstein: “Insanity: doing the same thing over and over again and expecting different results.” North American Wildlife and Natural Resources Conference, Volume 71, p. 54, Wildlife Management Institute, 1975.

Dedicated to my family

Supervisor's Foreword

The detailed control of material assembly on nanometer length scales is an important strategy in the optimization of energy-related technologies. Electronic processes in organic and organic–inorganic hybrid solar cells, in batteries and in super-capacitors take place at interfaces. An optimal device architecture therefore has to simultaneously fulfil two requirements: the maximization of interfacial area per volume, and the direct connectivity of each interfacial element with the outside circuit. This is a considerable scientific and technological challenge since it requires the local control over materials assembly on the 10 nm length scale in layers that are many micrometers or even millimeters thick.

Maik Scherer's doctoral theses explore the use of polymer self-assembly for the manufacture of energy materials with well-defined nano-morphologies and structural connectivities. To this end, the self-assembly of block-copolymers into the double-gyroid morphology was optimized for use as a 10-nm scaffold for material synthesis. This addresses the challenge in the field of polymer self-assembly of using these highly periodic morphologies in the synthesis of functional materials. It is demonstrated in a range of different ways. Metals and metal-oxides were introduced into voided gyroid channel networks by electroplating from a substrate or by atomic layer deposition; single crystals were grown within the mesoporous network; and conjugated polymers were electropolymerized within the gyroid void channels.

This opened-up a number of strategies for the creation of high-performance functional materials whose morphology is controlled on the 10-nm length scale, which Maik used to create materials with unprecedented electrochromic properties, a world-leading supercapacitor, and 3D 10 nm structured conjugated polymer assemblies that cannot be manufactured in any other fashion.

Finally the strategies developed in this doctoral thesis present a generic route to the synthesis of a very wide array of materials—both inorganic and organic—with periodic 3D morphologies on the 10-nm length scale, allowing the structural optimization of a large range of functional materials.

Cavendish Laboratory, Cambridge April 2013

Prof. U. Steiner

Preface

Inspired by the work of Edward Crossland et al. on using a thin porous polymer film with double-gyroid morphology for the template-assisted precision patterning of inorganic materials on the 10 nm length scale, [1–5] and due to the fact that all of this polymeric material which was custom synthesized in small quantities by Prof. Dr. Marc Hillmyer had been used up, I established a recipe for preparing thin gyroid films using commercially available polymers during my graduate studies [6, 7]. However, the latter system had some shortcomings as will be discussed below, thus the supply of double-gyroid templates for the patterning of functional materials remained a challenge.

Neat diblock copolymers form the desired double-gyroid morphology during microphase separation only for a very narrow range of block volume ratios. Nevertheless, Prof. Hillmyer succeeded in synthesizing a pure diblock copolymer, poly(4-fluorostyrene)-*b*-poly(D,L-lactide) (PFS-*b*-PLA), which adopted the gyroid morphology in thin films during temperature annealing [1–5]. In contrast, the system I investigated was a binary blend of a lamellae forming diblock copolymer, poly(styrene)-*b*-poly(isoprene) (PS-*b*-PI), and a homopolymer, poly(styrene) (hPS), which forms a gyroidal film during slow solvent-casting from toluene [6, 7]. The solvent evaporation and thereby the film formation is controlled by sandwiching the polymer solution between a teflon foil and the substrate.

The main advantage of the PS-*b*-PI/hPS system is that commercially available copolymers of a wide block composition range can be used since the final blend composition, which determines the microphase separated morphology, can be altered by addition of hPS homopolymer. However, compared to the neat PFS-*b*-PLA the PS-*b*-PI/hPS blend has some major drawbacks. The preparation of templates using PS-*b*-PI/hPS is very time-consuming and involves a drying process that typically takes several days, while the PFS-*b*-PLA films only require brief temperature annealing for less than an hour. Further, film areas of homogeneous thickness are limited to a square centimeter, while PFS-*b*-PLA templates of arbitrary dimensions can be easily fabricated by spin or blade-coating methods. But most importantly, in contrast to PFS-*b*-PLA, the blend system formed non-porous wetting layers at the air/polymer and polymer/substrate interfaces. Especially, the latter layer prevents the usage of the films as templates for the

electrodeposition of functional materials, which requires the voided network to be continuous all the way down to the conducting substrate.

The objective of this work, concerning the template-assisted patterning of functional materials on the nanoscale to enhance specific material properties, is fivefold. First, to develop a highly reproducible synthesis pathway for large quantities of double-gyroid-forming diblock copolymers. Second, to replace the previously used fluorinated monomers with an inexpensive and environmentally sustainable alternative making large-scale applications feasible. Third, to establish a reliable and fast fabrication technique for fully porous gyroidal templates. Fourth, to replicate these templates with inorganic as well as organic functional materials using electrochemical and atomic layer deposition. Finally, to apply and characterize these novel double-gyroid structured materials in devices such as photovoltaic devices, electrochromic displays, and supercapacitors.

The first part of this thesis introduces the different techniques of modern nanotechnology employed in three-dimensional nanopatterning of functional materials and seeks to highlight the possibilities and advantages of using self-assembled, mesoporous polymeric templates for this purpose. [Chapter 2](#) explores the fascinating gyroidal intermaterial dividing surfaces formed by self-assembling systems, such as microphase separating diblock copolymers. [Chapter 3](#) focuses on the synthesis of double-gyroid-forming diblock copolymers consisting of poly(4-X-styrene) and poly(lactic acid) via atom transfer radical and organocatalytic ring-opening polymerization using a bifunctional initiator. The preparation of mesoporous templates with double-gyroid morphology based on a rapid thermal annealing protocol is described in [Chap. 4](#). Importantly, it is shown that copolymers prepared from inexpensive, non-substituted styrene can yield fully porous thin films.

[Chapter 5](#) reports the template-assisted electrodeposition of metal oxides, such as V_2O_5 , using the previously prepared voided styrenic templates. Further, the performance of these materials as electrodes in electrochromic displays and supercapacitors is tested. [Chapter 6](#) describes an alternative approach to obtain double-gyroid-structured metal ceramics via the post-nanostructuring thermal oxidation of electroplated metals. Nickel is chosen to demonstrate this highly versatile fabrication route and to study the nanoscale Kirkendall effect of three-dimensional structures. Further, the electrochromic performance of NiO is investigated. [Chapter 7](#) deals with yet another electrodeposition technique, namely the electropolymerization of conjugated polymers. Identification of suitable solvents for the templated electrosynthesis and the subsequent template dissolution enable the three-dimensional nanopatterning of PPy, PEDOT, and PT. The refilling of the organic template via atomic layer deposition, including the necessary chemical modification of the styrenic scaffold for an improved thermal resistance and surface functionality, is discussed in [Chap. 8](#).

This thesis is the result of work carried out between May 2009 and August 2012 with the Thin Films and Interfaces Group at the Cavendish Laboratory, Cambridge, UK, and was only made possible by generous funding through Nokia and with the kind help and support I received from numerous people.

First of all I would like to thank Prof. Dr. Ullrich Steiner who gave me the unique opportunity to join his group and to work in a superb scientific environment. I am deeply grateful for the constant help, support, and excellent supervision. It was a very inspiring and exciting time in Cambridge and great fun participating in the three winter schools in Austria and Switzerland.

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August 2012

Maik R. J. Scherer

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Abbreviations

[BMIM][TFSI]	1-Butyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide
[EMIM][BF ₄]	1-Ethyl-3-methylimidazolium tetrafluoroborate
[SET3][TFSI]	Triethylsulfonium bis(trifluoromethylsulfonyl)imide
A(R)GET	Activators (re)generated by electron transfer
AFM	Atomic force microscopy
ALD	Atomic layer deposition
ATR-FTIR	Attenuated total reflection Fourier transform infrared spectroscopy
ATRP	Atom transfer radical polymerization
Bipy	2,2'-Bipyridine
CMC	Constant mean curvature
CV	Cyclic voltammetry
DBU	1,8-Diazabicycloundec-7-ene
DCM	Dichloromethane
DEZ	Diethylzinc
DG	Double-gyroid
DI water	Deionized water
EBIB	Ethyl α -bromoisobutyrate
EDLC	Electrochemical double layer capacitor
EDOT	3,4-Ethylenedioxythiophene
EDTA	Ethylenediaminetetraacetic acid
EDX	Energy-dispersive X-ray spectroscopy
FTO	Fluorine-doped tin oxide coated glass
HMDS	Bis(trimethylsilyl)amine
HMTETA	1,1,4,7,10,10-Hexamethyltriethylenetetramine
HOMO	Highest occupied molecular orbital
HPL	Hexagonally perforated lamellar
IMDS	Intermaterial dividing surfaces
IO	Inverse opal
ITO	Tin-doped indium oxide coated glass
Lactide	3,6-Dimethyl-1,4-dioxane-2,5-dione
LiTFSI	Bis(trifluoromethane)sulfonimide lithium
LUMO	Lowest unoccupied molecular orbital
Me ₆ TREN	Tris[2-(dimethylamino)ethyl]amine

MO	Metal oxide
MTBD	N-methylated 1,4,7-triazabicyclodecene
MTS	Methyltrichlorosilane
NKE	Nanoscale Kirkendall effect
NMR	Nuclear magnetic resonance
ODT	Order-disorder transition
OTS	Octyltrichlorosilane
P(F)S	Poly(4-fluorostyrene)- <i>r</i> -poly(styrene)
P3MT	Poly(3-methylthiophene)
PBrS	Poly(4-bromostyrene)
PBT	Poly(bithiophene)
PCBM	[6,6]-Phenyl-C ₆₁ -butyric acid methyl ester
PCIS	Poly(4-chlorostyrene)
PDI	Polydispersity index
PEDOT	Poly(3,4-ethylenedioxythiophene)
PEN	Poly(ethylene naphthalate)
PFS	Poly(4-fluorostyrene)
PGMEA	Propylene glycol monomethyl ether acetate
PhOPh	Diphenyl ether
PI	Poly(isoprene)
PLA	Poly(D,L-lactide)
PMDETA	N,N,N',N'',N''-pentamethyldiethylenetriamine
POZ	Primary ozonide
PPy	Poly(pyrrole)
PS	Poly(styrene)
PT	Poly(thiophene)
PTFE	Poly(tetrafluoroethylene)
PXS	Poly(4-X-styrene)
RAFT	Reversible addition fragmentation transfer
RCA	Wafer cleaning procedure developed
RIE	Reactive-ion etching
ROP	Ring-opening polymerization
RTIL	Room temperature ionic liquids
SAXS	Small angle X-ray scattering
SEC	Size exclusion chromatography
SEM	Scanning electron microscope
SG	Single-gyroid
Sn(EH) ₂	Tin(II) 2-ethylhexanoate
SOZ	Secondary ozonide
SU-8	Epoxy-based negative photoresist
TBD	1,4,7-Triazabicyclodecene
TCM	Chloroform
TEM	Transmission electron microscopy
THF	Tetrahydrofuran
Thiourea	Bis(3,5-trifluoromethyl)phenyl cyclohexylthiourea

TIP	Titanium isopropoxide
TMA	Trimethylaluminium
TPMS	Triply periodic minimal surfaces
TRIS	2-Amino-2-hydroxymethyl-propane-1,3-diol
UV	Ultraviolet
XPS	X-ray photoelectron spectroscopy
XRD	X-ray diffraction