Nanoindentation testing of soft polymers:

Computation, experiments and parameters identification

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PREFACE

The actually presented work has been carried out during the years 2009-2013 at the Chair of Applied Mechanics, Saarland University, Germany. The research work has been funded by the DFG (Deutsche Forschungsgemeinschaft) with the grant number Di 430/14-1 and Di 430/14-2.

First of all, I would like to thank my Ph.D supervisor Prof. Dr.-Ing. Stefan Diebels, who offered me the unique opportunity to work in the field of applied mechanics and supervised my work with continuous encouragement and support. His expert scientific knowledge, his vivid explanation and his extraordinary patient resulted in an extremely stimulating guidance. I am greatly indebted for his trust and unselfish help, especially at the beginning of my Ph.D study, which motivated my passion for science. Secondly, my thank goes to Prof. Dr.rer.nat. Wulff Possart, who is my second Ph.D supervisor, for the fruitful discussion during the first stage of my Ph.D research study. His encouragement and sustained attention on my research give me a great sense of uplift.

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ABSTRACT

Since nanoindentation technique is able to measure the mechanical properties of extremely thin layers and small volumes with high resolution, it also became one of the important testing techniques for thin polymer layers and coatings.

This dissertation is focusing on the characterization of polymers using nanoindentation, which is dealt with numerical computation, experiments and parameters identification. An analysis procedure is developed with the FEM based inverse method to evaluate the hyperelasticity and time-dependent properties. This procedure is firstly verified with a parameters re-identification concept.

An important issue in this dissertation is to take the error contributions in real nanoindentation experiments into account. Therefore, the effects of surface roughness, adhesion force, friction and the real shape of the tip are involved in the numerical model to minimize the systematic error between the experimental responses and the numerical predictions. The effects are quantified as functions or models with corresponding parameters to be identified.

Finally, data from uniaxial or biaxial tensile tests and macroindentation tests are taken into account. The comparison of these different loading situations provides a validation of the proposed material model and a deep insight into nanoindentation of polymers.

ZUSAMMENFASSUNG

Da Nanoindentation die Messung der mechanischen Eigenschaften von dünnen Schichten und kleinen Volumen mit hoher Auflösung ermöglicht, hat sich diese Messmethode zu einer der wichtigsten Testmethoden für dünne Polymerschichten und -beschichtungen entwickelt.

Diese Dissertation konzentriert sich auf die Charakterisierung von Polymeren mittels Nanoindentation, die in Form von numerischen Berechnungen, Experimenten und Parameteridentifikationen behandelt wird. Es wurde ein Auswertungsverfahren mit einer FEM basierten inversen Methode zur Berechnung der Hyperelastizität und der zeitabhängigen Eigenschaften entwickelt. Dieses Verfahren wird zunächst mit einem Konzept der Parameter Re-Identifikation verifiziert.

Fehlerquellen wie Oberflächenrauheit, Adhäsionskräfte, Reibung und die tatsächlichen Form der Indenterspitze werden in das numerische Modell eingebunden, um die Abweichungen der numerischen Vorhersagen von den experimentellen Ergebnissen zu minimieren. Diese Einflüsse werden als Funktionen oder Modelle mit dazugehörigen, zu identifizierenden Parametern, quantifiziert.

Abschließend werden Messwerte aus uni- oder biaxialen Zugversuchen und Makroindentationsversuchen betrachtet. Der Vergleich dieser verschiedenen Belastungszustände liefert eine Bestätigung des vorgeschlagenen Materialmodells und verschafft einen tieferen Einblick in die bei der Nanoindentation von Polymeren ablaufenden Mechanismen.

LIST OF APPENDED PAPERS

This thesis consists of a summary and six appended papers:

Paper A Z. Chen, S. Diebels, Nanoindentation of Hyperelastic Polymer Layers at Finite Deformation and Parameter Re-identification, *Archive of Applied Mechanics* 82 (2012) 1041-1056

Paper B Z. Chen, S. Diebels, Parameter Re-identification in Nanoindentation Problems of Viscoelastic Polymer Layers: Small Deformation, *Zeitschrift für Angewandte Mathematik und Mechanik (ZAMM)* 93 (2012) 88-101

Paper C Z. Chen, S. Diebels, J. Schmitt, Frictional Nanoindentation of Hyperelastic Polymer Layers: A Numerical Study, *Proceedings of the 3rd ECCOMAS Thematic Conference on the Mechanical Response of Composites* (2011) 229-236

Paper D Z. Chen, S. Diebels, Modelling and Parameter Re-identification of Nanoindentation of Soft Polymers Taking into Account Effects of Surface Roughness, *Computers and Mathematics with Applications* 64 (2012) 2775-2786

Paper E Z. Chen, S. Diebels, N. J. Peter, A. S. Schneider, Identification of Finite Viscoelasticity and Adhesion Effects in Nanoindentation of a Soft Polymer by Inverse Method, *Computational Materials Science* 72 (2013) 127-139

Paper F Z. Chen, T. Scheffer, H. Seibert, S. Diebels, Macroindentation of a Soft Polymer: Identification of Hyperelasticity and Validation by Uni/Biaxial Tensile Tests, *Mechanics of Materials* 64 (2013) 111-127

CONTRIBUTIONS FROM CO-AUTHORS

The appended papers were prepared in cooperation with co-authors. The author of this thesis was in charge of the major work in all of the papers. The work of the co-authors is described in detail as following.

For **Paper A, Paper B and Paper D**, the sole co-author Prof. Dr.-Ing Stefan Diebels, who is my Ph.D supervisor, guided me to organize the ideas at the beginning. During the numerical computations, he supported me to develop the numerical model and the optimization procedure. Afterwards, he took part in planning the papers and correcting the English writing, which resulted in a qualitative improvement of these papers.

For **Paper C**, the co-authors, Prof. Dr.-Ing Stefan Diebels and Dr.-Ing Joachim Schmitt, supported me to implement the numerical model of nanoindentation taking into account the friction effects. The discussion of the simulation results with the co-authors is useful to organize the structure of the paper.

Paper E was prepared with three co-authors. My Ph.D supervisor Prof. Dr.-Ing Stefan Diebels supported me to develop a numerical model to simulate nanoindentation of viscoelastic polymers considering adhesion effects. He guided me to choose a suitable viscoelastic model and a contact model with a traction-separation interaction. B. Sc Nicolas Peter and Dr.-Ing Andreas Schneider performed the SEM and nanoindentation with in situ SPM tests to characterize the surface of the specimens and wrote the corresponding part to describe the experimental procedure. A qualitative improvement of the paper cannot be reached without useful suggestions of the co-authors and without their correction of my English writing.

For **Paper F**, Prof. Dr.-Ing Stefan Diebels brought forward the idea to characterize polymers with macroindentation and to compare the results to uni/biaxial tensile tests. Dipl.-Ing Tobias Scheffer and Dipl.-Ing Henning Seibert supported me to perform macroindentation tests and uni/biaxial tensile tests. They wrote the parts describing the experimental devices and prepared the illustrated sketches of the experimental devices and specimens. Fruitful discussion of the experimental data with all of the co-authors and their strategic suggestions lead to a qualitative improvement of the paper.

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