行政院所屬各機關因公出國人員出國報告書

（出國類別： 考 察）

赴歐洲參加表面檢測技術研討會暨考察真空微流量製程設備技術

出國報告

服務機關：國科會精密儀器發展中心

出 國 人：陳思翰　助理研究員

林郁欣　專案助理研究員

出國地點：德國

出國日期：九十年八月二十九日至九月七日

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赴歐洲參加表面檢測技術研討會暨考察真空微流量製程設備技術

主辦機關:

行政院國家科學委員會精密儀器發展中心

聯絡人／電話:

劉恭霖／03-5779911-522

出國人員:

陳思翰　行政院國家科學委員會精密儀器發展中心　研究發展組　助理研究員
林郁欣　行政院國家科學委員會精密儀器發展中心　研究發展組　專案助理研究員

出國類別: 考察

出國地區: 德國

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報告日期: 民國 90 年 10 月 16 日

分類號/目: ／　／

關鍵詞: 奈米科技,奈米檢測技術,IMT研究中心,IMM研究中心

內容摘要: 奈米科技被公認是本世紀最具發展潛力的科技產業技術之一，近來工研院擬成立一奈米技術中心，積極整合各方資源，以帶動國內相關技術發展。其應用領域涵蓋甚廣，包含電子、機械、航太、醫療、生化及通訊等，歐、美、日等先進國家的研究機構更是如火如荼地投入奈米技術之研發。 本中心也已進行奈米檢測技術之相關研究，並有了一些成果相繼發表於國際期刊，爲充分掌握國外科技的研究現況與發展趨勢、實地了解其研究環境與研究規劃，建立技術合作管道，加強彼此技術交流關係，特派員赴歐洲參加奈米技術研討會議，並考察其相關之真空方面製程技術。 這次的考察行程包括了，參觀IMT研究中心，IMT擁有相當完整的LIGA相關製程設備。接著趕往德國德勒斯登，參加第十四屆歐洲奈米表面檢測技術研討會，一連舉行四天，參加者來自世界各國、台灣參加者除本中心外，尚包含許多業界公司，會中邀請了世界上知名的理論與實驗專家，對於高分子奈米材料之製程及檢測技術做一完整系列的探討。最後我們於回國的前一天，趨車前往參觀位於法蘭克福附近的IMM研究中心。

本文電子檔已上傳至出國報告資訊網

考察行程表

日期	起	至	工　作　內　容
8/29~30 (三)~(四)	台 北	法蘭克福 (德國)	搭機前往 法蘭克福
8/31 (五)	法蘭克福		參訪 IMT 研究中心
9/1 (六)	法蘭克福		假日資料整理
9/2 (日)	法蘭克福 (德國)	德勒斯登 (德國)	轉程 註冊 **ESOPS** 14 研討會
9/3~4 (一)~(二)	德勒斯登		參加 **ESOPS** 14 研討會
9/5 (三)	德勒斯登 (德國)	法蘭克福 (德國)	轉程 參訪 IMM 研究中心
9/6~7 (四)~(五)	法蘭克福	台 北	搭機回國

摘 要

奈米科技被公認是本世紀最具發展潛力的科技產業技術之一，近來工研院擬成立一奈米技術中心，積極整合各方資源，以帶動國內相關技術發展。其應用領域涵蓋甚廣，包含電子、機械、航太、醫療、生化及通訊等，歐、美、日等先進國家的研究機構更是如火如荼地投入奈米技術之研發。

本中心也已進行奈米檢測技術之相關研究，並有了一些成果相繼發表於國際期刊，為充分掌握國外科技的研究現況與發展趨勢、實地了解其研究環境與研究規劃，建立技術合作管道，加強彼此技術交流關係，特派員赴歐洲參加奈米技術研討會議，並考察其相關之真空方面製程技術。

這次的考察行程包括了，參觀 IMT 研究中心，IMT 擁有相當完整的 LIGA 相關製程設備。接著趕往德國德勒斯登，參加第十四屆歐洲奈米表面檢測技術研討會，一連舉行四天，參加者來自世界各國、台灣參加者除本中心外，尚包含許多業界公司，會中邀請了世界上知名的理論與實驗專家，對於高分子奈米材料之製程及檢測技術做一完整系列的探討。最後我們於回國的前一天，趨車前往參觀位於法蘭克福附近的 IMM 研究中心。

目　　錄

壹、前言

由於人類對微小化元件的殷切需求，已由原來的微米（10^{-6} m）範圍進入了奈米（10^{-9} m）範圍的時代，在面臨二十一世紀高科技發展的競爭中，奈米科技的發展，將是國家高科技發展政策中不可或缺的一環，其應用層面涵蓋了機械、材料、光學、微電子、量測、生化醫藥及原子物理等。

奈米科技包括奈米材料製程、加工以及檢測三大部分，奈米材料和傳統的材料科學在研究的主要方向上有很大的不同。傳統上，材料科學是以冶金、陶瓷及高分子塑膠為基礎，是以塊材材料(Bulk Material)為對象。然而，以物理與化學前沿研究為基礎的奈米材料(Nanomaterials)研究的主要方向，是著重在化學、光電與機械在實際應用上所需的特殊及特定功能材料，它的發展需要基礎凝態、真空物理研究與化學合成做有效之整合。這樣的整合，近年來在研究前沿上已有突出的發展。它不只有應用的價值，更在基礎科學上開發了許多新領域。例如：在新開發的材料中，碳六十、碳管、半導體奈米晶體、中孔徑分子篩等都是非常出色的基礎研究，它們應用上的開發也是廣受注目。至於奈米加工與檢測方面，主要是以掃描探針顯微技術(SPM)為背景，所做的一些延伸功能與應用。

本中心為國內精密儀器的研發單位，在奈米檢測方面，已累積多年的寶貴技術與成功經驗。目前正積極規劃整合 SPM 之相關技

術，包括磁力顯微術(magnetic force microscopy, MFM)、靜電力顯微術(electrostatic force microscopy, EFM)、導電性原子力顯微術(conducting atomic force microscopy, CAFM)、近場光學顯微術(near-field scanning optical microscopy, NSOM)…等等，是目前國內少數具有完整奈米檢測技術能量之研究機構，未來將繼續規劃架構奈米表面電位與電容之量測機制，積極朝向整合磁、光、電之奈米檢測實驗室發展。

奈米系統技術是未來發展的方向，要獲得如此精密微小結構尺寸，大都需要在良好的真空下進行製程步驟，如薄膜沉積、電漿蝕刻和高精密壓模等等。本中心在類LIGA製程設備技術開發方面，已擁有各項高精密高真空製程設備，如壓膜機、鍍膜機、SEM及蝕刻機等等，於89年底建置之感應耦合電漿離子蝕刻系統（ICP），真空度更可高達10^{-6}至10^{-7} Torr，因應矽基微系統元件開發，提供良好的非等向性乾式矽蝕刻製程技術。本中心擁有各項高精密之製程設備及技術，且為國內製程設備與製程技術的研發重鎮。

此外本中心為進一步掌握國際上奈米檢測與分析技術之發展趨勢，故派員赴歐洲參加國際性會議，並考察相關的研究機構，以蒐集目前世界各國的重要研究成果與關鍵性技術，作為中心未來研究發展的重要參考依據。

貳、出國目的

ESOPS 14 是高分子材料工程技術發展的國際會議(14th European Symposium on Polymer Spectroscopy)，每兩或三年輪流在歐洲各個國家舉行，而今年是由德國主辦第十四屆，在德勒斯登一連舉行四天，這個研討會雖說只在歐洲地區舉行，參與者卻是來自世界各地的學術及研究單位，其所討論的議題主要是集中在高分子材料的製作與相關之應用，尤其是有機發光元件(organic light emitting diode, OLED)，由於具有低啟動電壓、高量子效率、使用旋轉塗佈之簡單製程，加上高分子材料本身具撓曲性，易合成加工，對未來大面積平面顯示器而言，具有革命性的影響。此次會議的探討重點包括：如何應用真空儀器設備進行新興材料合成與分析，以及其相關表面特性之研究，在目前炙手可熱的半導體與顯示器製程當中，均大量採用真空儀器設備，因而在會議演講安排時程中，亦不乏數位從事真空元件製程研究領域者。

本中心已開發完成導電性原子力顯微術，亦計畫進一步將其應用於高分子材料之電性檢測方面，在此次研討會中，我們獲知許多相關技術的發展，可供本中心相關研究作為參考。同時會中亦與其他各國與會先進交流相關技術與經驗，在技術專業和國際知名度的提升方面有重大的收

穫。

微機電是中心極力推展之重點計劃之一，德國 IMT 與 IMM 研究機構均在微機電領域上享譽盛名，這兩個單位人員均在兩百人左右，製程設備雖與中心並不完全相同，但其設備管理維護及在研發上之優越成果，均可以作為中心之參考，而奈米科技與生物醫學科技更是接下來之發展重點，在更小尺寸的領域範圍下，為獲得更精準之製程品質，在真空下製作已是必然的，因此此行也特別考察國外真空製程儀器設備作為研發的參考。

IMT 為 FZK (Forschungszentrum Karlsrule—Technik and Umwelt)的附屬機構，而 FZK 為德國最大的非營利研究機構，主要從事環保的高科技研究。FZK 下設有十五個研究所，從事四項研究計畫：核子融合(Nuclear fusion)、微系統技術(Microsystems technology)、核能安全(Nuclear safety research)、低污染低浪費程序(Low-pollutant, low waste processes)。

IMT 擁有相當完整的 LIGA 相關製程設備，包括電子束能量為 2.5 GeV 的同步輻射加速器 ANKA、可製作小至 150 nm 微結構的 100 kev 電子束直寫機(Electron beam writer)、電鑄及模造線。

IMM 研究機構是由德國 Rhineland Palatinate 州政府於 1990 年底成立。主要目的是在開發微結構的製程技術，包括：微精密工程技術、LIGA 技術、薄膜技術、準分子雷射微製造技術、材料開發研究、微光學系統、微反應器系統、SXM 奈米技術與量測、電鑄、壓模等。IMM 來自州政府的經費每年約僅佔總經費的 1/3，

其餘必須自籌，也因此開發產品的速度較快，也較工業導向。所開發的產品有：微馬達、微齒輪系統、微反應器、微混合器、微熱交換器、微幫浦、光纖開關與定位元件、整合光學分光器、微鏡片等。

參、參訪過程

一、參訪德國 Institute for Microstructure Technology (IMT)：

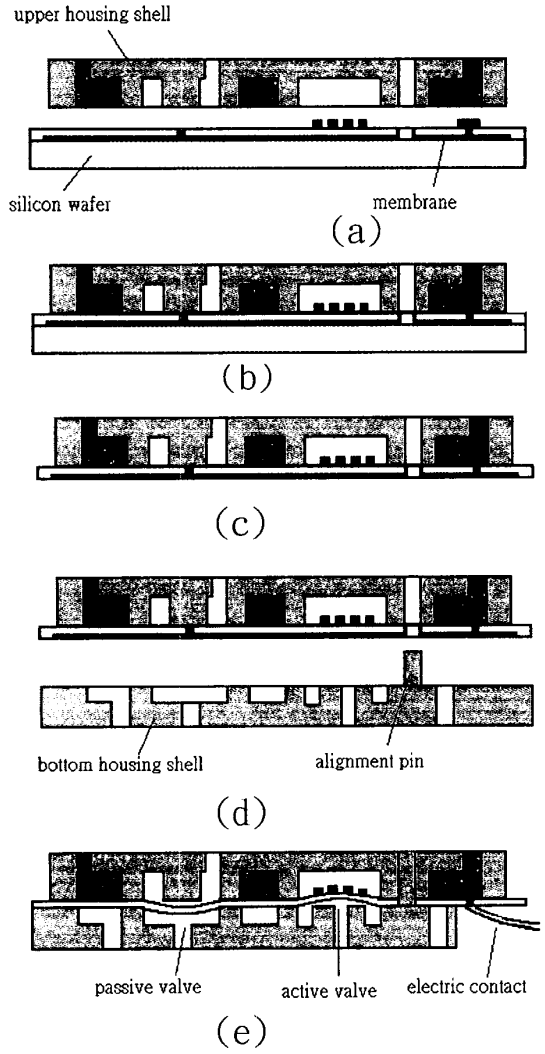
IMT 為 LIGA 的起源地，目前由 Dr. V. Saile. 負責，在世界上有名的一張圖片就是在一隻螞蟻的腳上套上一個齒輪，IMT 微系統技術計畫成立於 1992 年，主要在 IMT 執行，其餘的執行單位尚包括材料研究所(IMF)、放射性化學研究所(IRCH)、應用資訊研究所(IAI)、工程中心(HIT)、電子及資料處理中心(HPE)、實驗中心(HVT)等單位。微系統技術計畫將歷時十一年，其主要研究課題為：

圖一：IMT 利用 LIGA 製程製作之小齒輪

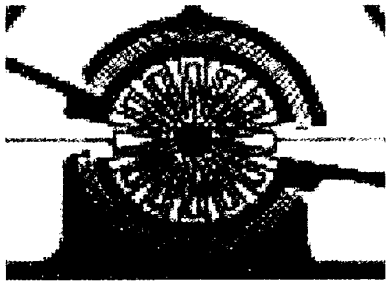
1. 微製造技術
   (1) LIGA 技術
   (2)精密加工
   (3)深光刻術
   (4)反應性離子蝕刻
2. 組裝及連結
3. 開發相關元件及系統雛形
   (1)機械式微感測器及致動器
   (2)微光學元件
   (3)微流體元件，如微閥、微泵
4. 模擬及系統技術

IMT 在提出 LIGA 製程後，繼而提出 AMANDA PROCESS 製程技術，AMANDA 製程技術應用於微流體，更是符合工業生產之製程技術。如藥劑的儲存、小量樣品之生物醫學分析、混合化學、微氣動力學應用與空氣品質監控等等。

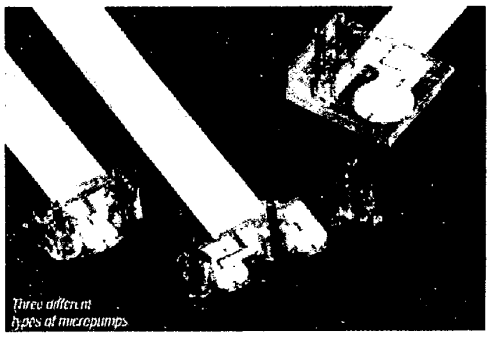
AMANDA 製程技術可製作多種微感測器與微致動器，是由表面微加工技術、模造技術與薄膜技術組成，製程步驟如下圖所示



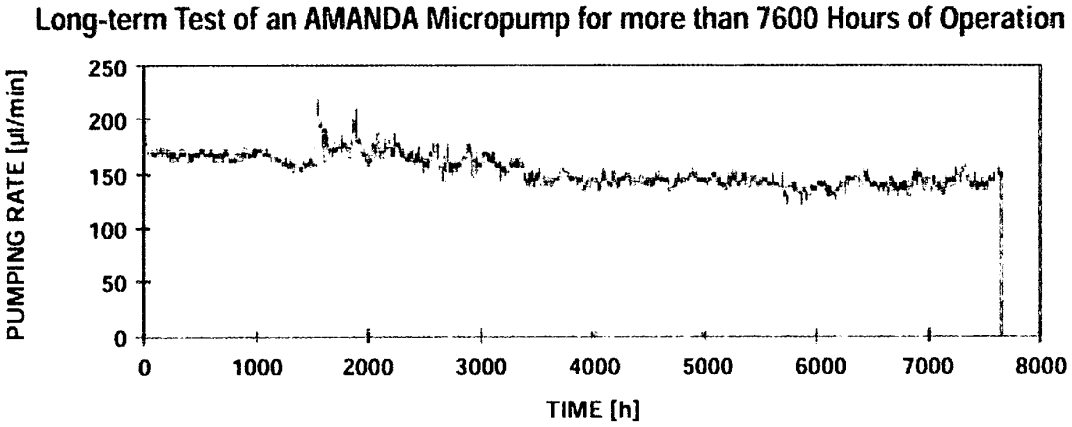
圖二：AMANDA 製程步驟



Strain gauge of a pressure sensor.



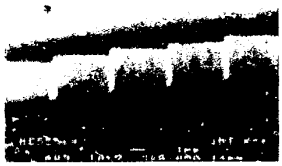
圖三：微流體成品



圖四：AMANDA 製程技術製作之微幫浦使用壽命測試

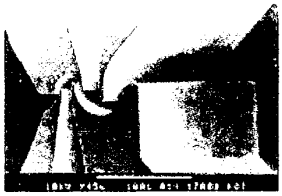
AMANDA 製程特點	
低製程成本	在 IMT 製作一個幫浦的費用為 150 馬克
可靠度高	幫浦的運轉可超過 7600 小時
高製程良率	70%的良率
材料選擇廣	Housings: PMMA、PSU、PVDF、PC、PEEK、PA、POM, etc. Membranes: PI、PTFE、FEP、Ti, etc. Conductor tracks: Au、Cr、Ti、Cu, etc.
高的結構	Housings:5 μm 到超過 1mm Membranes:1 μm 到 50 μm Conductor tracks:50nm 到 5 μm
對準精度	Housing-Membranes:50 μm Conductor tracks-Membranes:5 μm

high accuracy



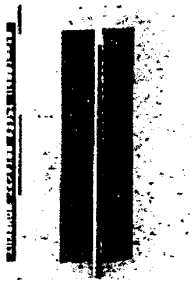
Resist structure of a reflection grating,
0.25 µm step height, 125 µm structural height.

any lateral shape

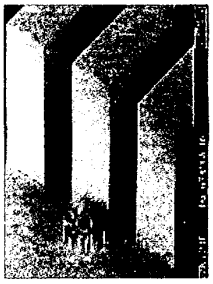


Separation nozzle as an example
of arbitrary lateral shaping.

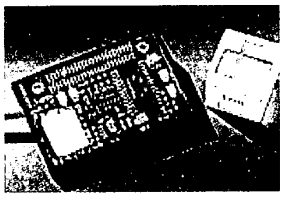
high aspect ratio



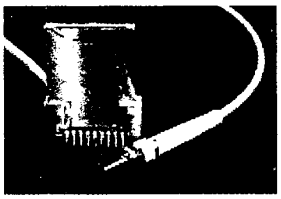
Bar structure 400 µm high,
with parallel sidewalls.



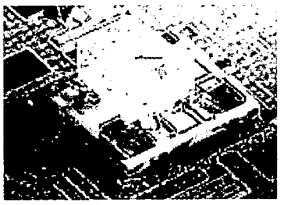
SEM micrograph of a PMMA
structure 1100 µm high and a
gearwheel made of nickel.



NIR array spectrometer system
with InGaAs array (156 pixels)
and pre-evaluation electronics
as OEM component with a single
electrical interface.
(Dimensions: 5 x 4 x 0.5 cm³)

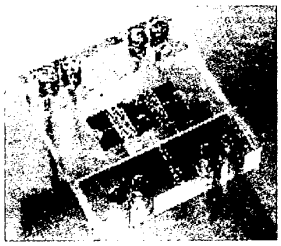


NIR spectrometer system with 16
parallel readout channels based on an
InGaAs detector array for identification
of polymer spectra.
Size of structure: 32 mm wide,
39 mm high (+ chip height), 23 mm long.

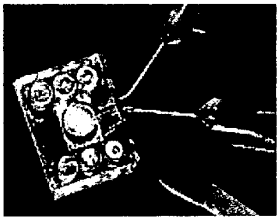


Optical Bench of a heterodyne receiver
for communication engineering:
On a ceramic substrate, positioning aids
for lenses, photodiodes, glass fibers,
and prisms are structured by means of
deep X-ray lithography.

Plug for 16 multi-mode fibers with
microstructured guiding ditches for
fibers and positioning pins. The plugs
are produced by injection molding (IMF III).
The mold used for this purpose is
produced by means of the LIGA technique
and mechanical micromachining (HVT).



Optical switch.
The dimensions of the linear actuators are 4 x 2 x 0.1 mm³.



Fluidic valve.
The diameter of the valve chamber is 2 mm.

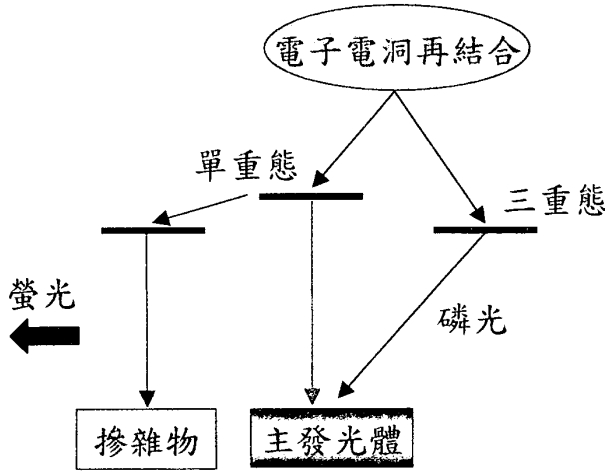
圖五：IMT 之研發成果

二、 參加第十四屆歐洲高分子材料工程技術發展研討會

歐洲高分子材料工程技術發展研討會今年輪由德國所舉辦，參加的人士來自歐、美、亞等三洲共三百多人，選在具古城風味的德勒斯登舉行，大會由德勒斯登大學校長親自主持，會中邀請歐、美和亞洲各個知名的研究團隊，針對高分子這種奈米材料之製程和檢測技術作一詳細的報告。發表的文章中，亞洲以日本為最多，國內參與盛會的團體包括精儀中心、清華大學、交通大學及部分從事 OLED 研究之私人公司。

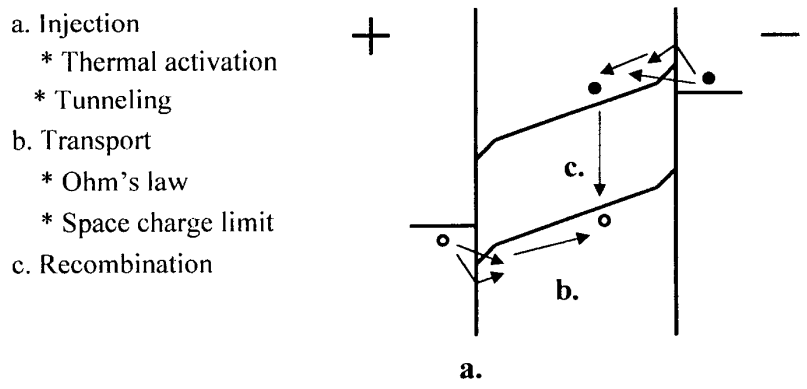
此次研討會將高分子材料製程、檢測、分析及其相關之應用做了通盤的討論，在 OLED 研究領域中具領導地位的日本，此次亦有兩位知名專家獲邀演講，其中來自東京大學的 Furukawa 教授發表"高分子電致發光光譜量測"中，不僅對於當前 OLED 發光機制做一深入淺出的探討，亦陳述其奈米級的光學檢測之重要性。

OLED 發光的原理是利用一正向外加偏壓，使電洞穿過電洞傳輸層(hole transport layer)，在一具有發光特性之有機層與來自電子傳輸層(electron transport layer)之自由電子相結合，並將能量以光的形式釋放出來的物理現象。而這種釋放出來的能量中，只有25%(單重態到基態)的能量可用來當作 OLED 的發光，其餘由三重態到基態 75%的能量，通常以磷光或熱的方式散發(如圖六所示)，由於選擇的發光材料的不同，可使此 25%的能量以不同顏色之形式加以釋放，而此一部份亦是奈米光學檢測相當重要的一環。



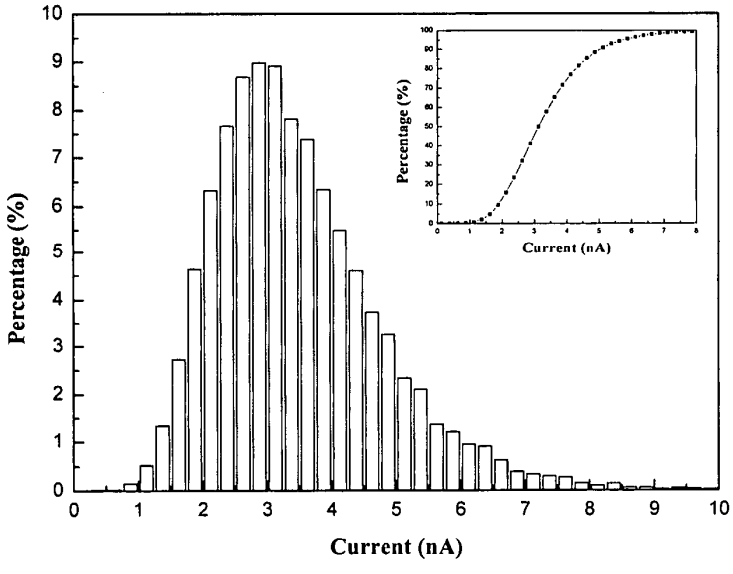
圖六：OLED 發光原理與效能

大會另一位演講者 Depecker 教授，目前任教於法國 Lille 大學，內容提及利用 AFM 量測 OLED 的奈米級電流分布。OLED 的電流特性可以一般固態電子理論解釋，演講者談及之電流公式以實驗試片為模型，即單層元件-電極/MEH-PPV/電極之結構。有機發光材料常是單極性物質，根據 B. K. Crone 和 I. H. Campbell 對 MEH-PPV 的研究，亦證明電洞的遷移率高於電子三個數量級，因此在 MEH-PPV 的元件中，常見的半導體理論公式中之遷移率應代入電洞之值，而不是一般的電子遷移率，也就是說電流的大小由電洞傳播主導。



圖七：OLED 元件中電子與電洞複合模型

電荷在元件中的行為包括界面電荷注入、電荷傳播與電子電洞結合（如圖七所示）。電荷注入的理論有熱激發(thermal activation)、穿隧效應(tunneling effect)、界面注入效率與鏡面效應(image effect)。電荷傳播的理論則包括歐姆定律(Ohmic's law)、空間電荷限制(Space Charge Limit, SCL)與缺陷填滿限制(Trap-Filled Limit, TFL)，其中 SCL 理論又因缺陷分佈的形式不同，而演化出各種電流與電壓的關係式。圖三為其主要之研究成果，實驗之方式與中心目前剛架設完成的導電性原子力顯微鏡類似，主要目的在於量測樣品表面之區域電流分布，並藉由上述之理論模型，對於元件中區域電荷的傳輸作深入的探討，尋求延長元件壽命有效的製程參數。



圖八：OLED 元件之區域電流分佈圖

全彩化的平面顯示器是 OLED 發展之最高目標，目前紅、綠、

藍三原色的摻雜材料均已成功的開發出來，但並未達成令人滿意的地步，尤其在藍光及紅光部分。此外白光材料也是近來研究之重點課題，主要是應用於液晶螢幕之背光源，預期可大幅減少目前 LCD 白光光源所佔的空間與重量。從這次會議演講者中發現，許多研究團體試圖以塑膠基板來取代玻璃基板，藉以製成可撓曲式的 OLED，此稱之為 FOLED(flexible OLED)，如果可順利研發成功，筆捲式螢幕的誕生將不再是電影中的科幻情節。

三、參訪德國 Institute for Microtechnology in Mainz(IMM)

我們繼續前往法蘭克福參觀著名的研發技術單位－IMM，Dr. W. Ehrfeld所領導的IMM研究機構，是由德國Rhineland Palatinate州政府於 1990 年底成立，是一個非營利的研究發展機構，接待人員告訴我們，他們的內部的成員約有兩百多人，並帶領我們參觀他們之研發成果，令我們印象深刻的是他們的人員並不是很多，但是研發成果卻是非常驚人，如花生大小之直昇機、微幫浦、商品化之微流量混合器、生物醫學微機電及微光學的研究等等，研究的方向是將創新的概念快速且有效率地轉成商品化。

IMM的重點工作包括：

(1)微系統製程技術的開發與最佳化

(2)發展製程與微組裝設備及周邊

(3)開展或改良微系統使用的材料

(4)提供微系統技術諮詢服務

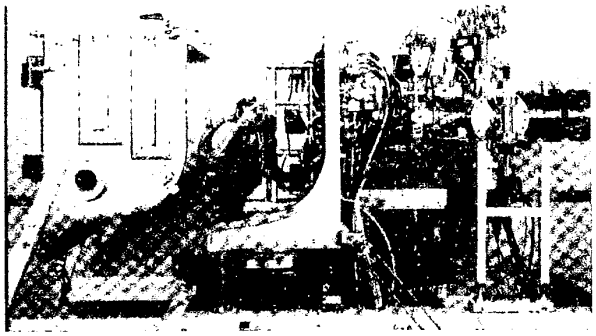
(5)提供微系統設備及技術支援

(6)協助合作對象建立市場活動

主要目的是在開發微結構、微流量的製程技術，包括：真空製程技術、真空鍍膜技術、真空鍍膜材料開發研究、微精密工程技術、準分子雷射微製造技術、微光學系統、微反應器系統等。IMM的製程技術成熟，並有許多真空微流量製品成功的產品，如已商品化之微流量計、微流量混和器、微馬達等等，其規劃發展真空

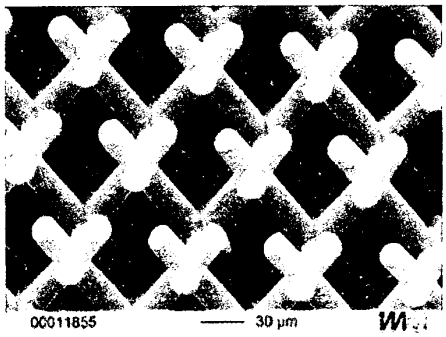
微流量製程技術的經驗及產品化的研究方向值得本中心學習。

IMM 所擁之 Deep X-ray lithography scanner，需在良好的真空度下作製程，並能調整曝光的角度，最大角度到 60 度，能做出 3D 的微結構。真空鍍膜方面，有 PECVD、LPCVD 與 MWCVD 三種鍍膜機台。



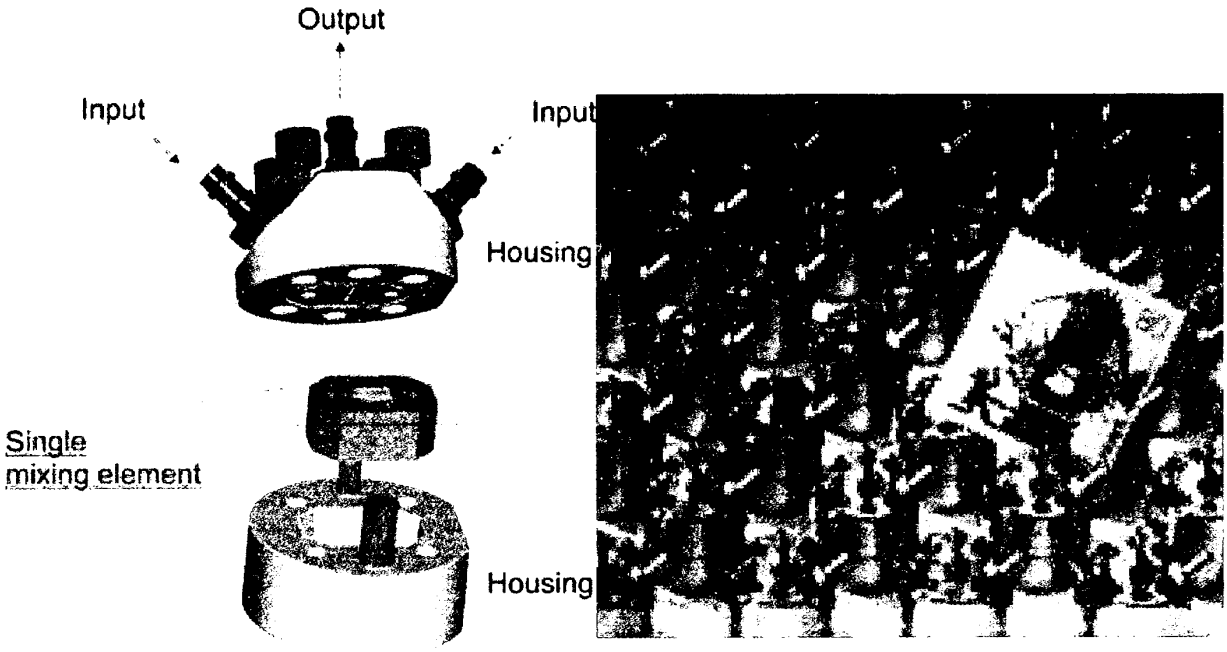
圖九：IMM Deep X-ray lithography scanner

圖十：IMM 薄膜沉積系統



圖十一：Deep X-ray scanner 做出的 3D 結構

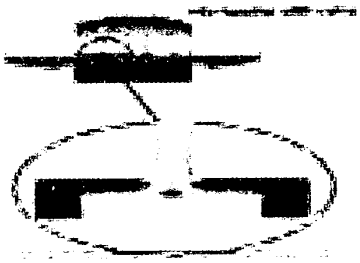
在微流量系統模組方面 IMM 除開發微幫浦外，更發展一個由混合器、反應器及分析系統所構成的連續流微反應器。並已成功發展成商品出售。



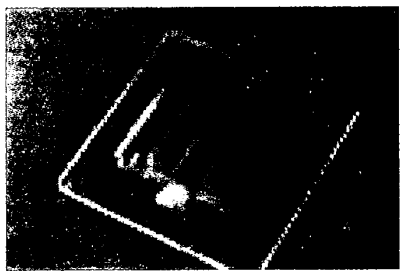
圖十二：IMM 所發展之微流量混和器商品

圖十三：微幫浦

圖十四：微流體

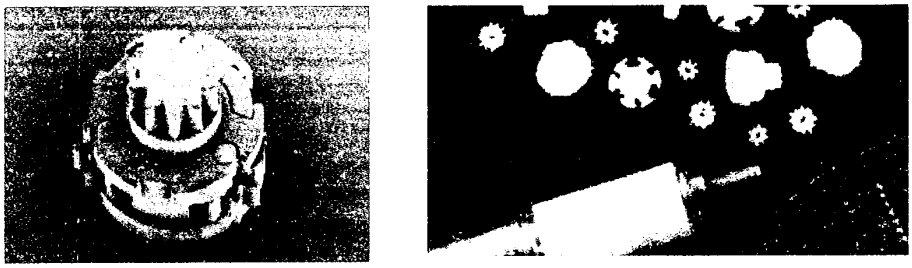


圖十五：模擬系統

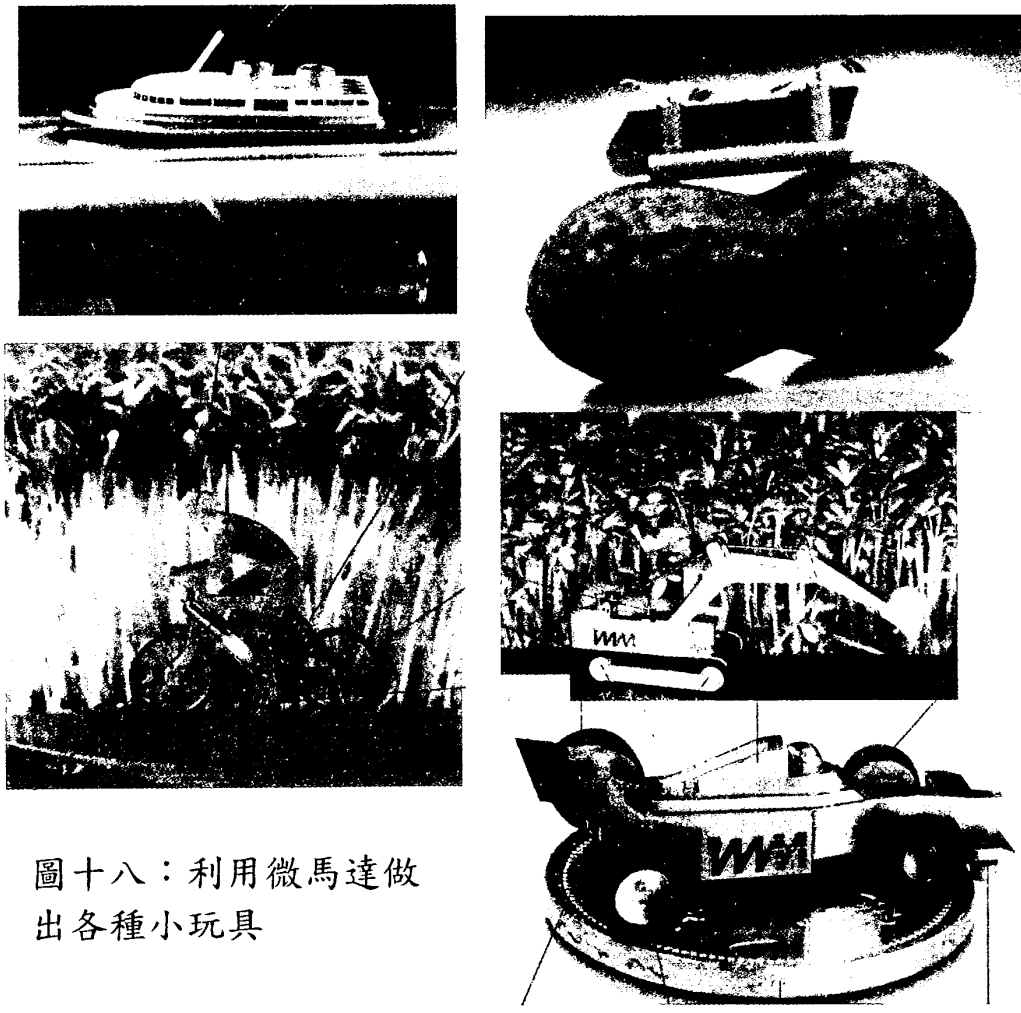


圖十六：流體分析系統

IMM 除在微流量系統有不錯的成果，在微機電 LIGA 也非常著名，如早些年所研發之微馬達，更利用所設計研發成功之微馬達作出一些應用，這些看似玩具之應用，代表著他們擁有高超的技術與能力，將枯燥繁重的研發工作轉變成娛樂，這是令我們感到最深刻的地方。

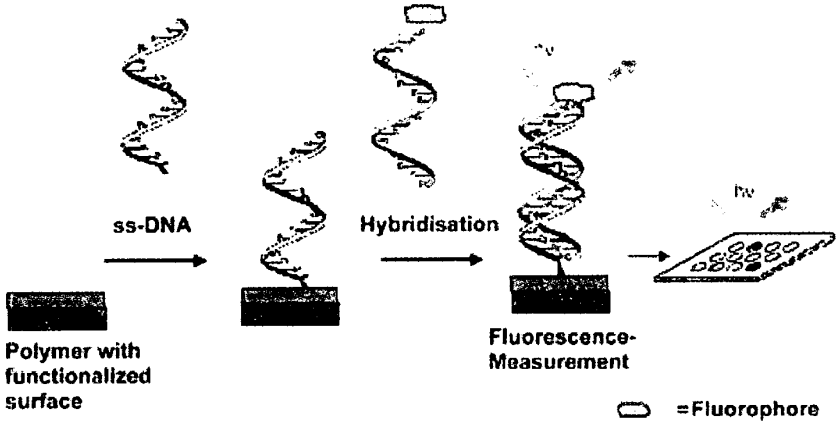


圖十七：IMM 發展成熟的微馬達



圖十八：利用微馬達做出各種小玩具

生物微機電也是目前發展的重點，IMM 發展的 DNA Chip ，經由生醫樣品 DNA 的前處理、DNA 的隔離，訊號的放大與分析來檢驗病人有病變的基因，為了與 ss—DNA 產生共價鍵結，選擇矽、玻璃和高分子等容易與 DNA 產生鍵結的材料，利用雷射來引發螢光的反應來作偵測。



圖十九：DNA Chip 鍵結與檢測原理

肆、達成之任務

1. 赴德國參加第十四屆歐洲高分子材料工程技術發展研討會(The 14th European Symposium on Polymer Spectroscopy)，瞭解目前先進研究奈米材料之製程及檢測系統方面的發展。高分子發光材料 OLED 製程技術上的發展，直接關係著未來發光元件的演進，對於現階段主流顯示器而言，亦具有革命性的影響。目前國內亦有為數不少之學術單位和業界團體致力於此相關產品的開發，如銖德科技旗下之子公司銖寶光電即發表了許多初步之成果。此外，工研院亦欲成立一奈米技術中心，預計在五年內投入約 100 億的預算，對於奈米材料、光學、電子、量測、生化醫藥及原子物理等相關技術加以統合，規劃成為國內唯一具有完整奈米技術之研究機構。

2. 參訪德國技術研發重鎮 IMT 與 IMM，了解德國微系統技術的研發現況及未來發展趨勢。對於其研發成果細部關鍵技術雖無法得知，但對發展的趨勢及方向有更深入的了解，參訪中除瞭解該單位對於高真空製程設備發展之過程及技術外，並蒐集其微系統製程設備之技術及研發之成果資料，可作為中心在此一領域技術發展方向的參考，提升中心在微系統技術的研發能力，規劃下一技術研發的方向。

伍、心得與建議

這次出國能夠圓滿達成任務，端賴事前的周密計畫以及相關人員的協助，帶回了許多豐富的資料，經過整理後提出一些心得與建議作為參考：

一、在德國德勒斯登參加研討會時，由於他們主辦非常成功而令人好評，在仔細體驗這個歐洲國家之生活後，你會發現他們非常注重所謂的時間觀念，舉搭火車為例，不管你搭車之距離有多遠，其班車之每一站的時間誤差絕對在一分鐘以內，可說令人印象深刻。九月的德國，氣溫約在攝氏20度左右，氣候宜人，地廣人稀且重視水土保持，因此每個人均具相當好的居家環境品質，記得小時後常唱的一首歌的歌詞寫道"我家門前有小河，後面有山坡"，此意境可說得到了完全的感受。

二、這次研討會，是由德國德勒斯登大學主辦，他們選擇了一棟傳統建築的三層樓房，二樓是開會的地點，三樓是壁報展示區，至於一樓則是德勒斯登有名的陶瓷藝品展覽館，此種安排方式，可讓遠道而來的國外學者，在開會的閒暇之餘，不僅認識當地的風俗民情，且鬆弛研討會嚴肅的氣氛，是相當值得國內仿效的一種開會方式。

三、 IMT 與 IMM 兩個單位的研究人員都不多，都約 2 百多人，但是他們的研究技術在世界上卻是赫赫有名，主要的原因是他們全都以 LIGA 為發展微系統技術的主軸，幾乎投入所有的人力與物力，專心致力於LIGA這個領域而全心全力地發展，同時在研發前就以商品化為研發方向，兼顧市場導向與經濟利益。

四、 研發是一非常耗時而且壓力沉重之工作，在製程步驟中常常一道製程步驟就要在實驗室裡花上半天一天，甚至幾天的製程時間，除了工作勞累外更要承受成功與否的壓力。而參訪 IMT 與 IMM 的過程中發現，在他們展示成果的櫥窗中，大部分看到的都是玩具，如微小直昇機、微小跑車、小型遙控船等等，當然也包括各式各樣的研發成品。而這些看似玩具的成果背後，代表著高科技、高技術能力。將繁重的研發工作以製作好玩玩具的心情來作，有愉快的心情作研究，必能做出更好的研發成果。

五、 在實驗室裡有各種高科技之儀器，而安全是非常重要的，外國實驗室的管理非常的嚴格，幾乎所有機台都有專門的人員負責維護管理。本中心開放研究生參與研究 ，並開放學生自行操作，除定期舉辦工安方面的訓練，對研究生的管理與訓練也不斷的在進行，唯有時時小心謹慎，才能確保安全。

陸、結語

在參加四天的國際研討會得到了許多寶貴經驗，和各國學者交換彼此經驗後，發現目前只是奈米科技發展的開端，其融合的領域相當廣泛，包括機械、電子、化工、材料、量測等，許多的相關技術正被積極的開發之中，非常值得中心作為規劃未來發展方向之參考。

參訪的最大收穫是國外的技術與人才養成是值得本中心參考借鏡的，技術的掌握及人才的運用更是不可少；參訪過程中深刻體驗到要在某領域中有所成果，必須掌握一關鍵之發展方向，專心於此領域中研發，儀器設備不在多而在充分運用所有的儀器，就能開創一片天地。

柒、附件

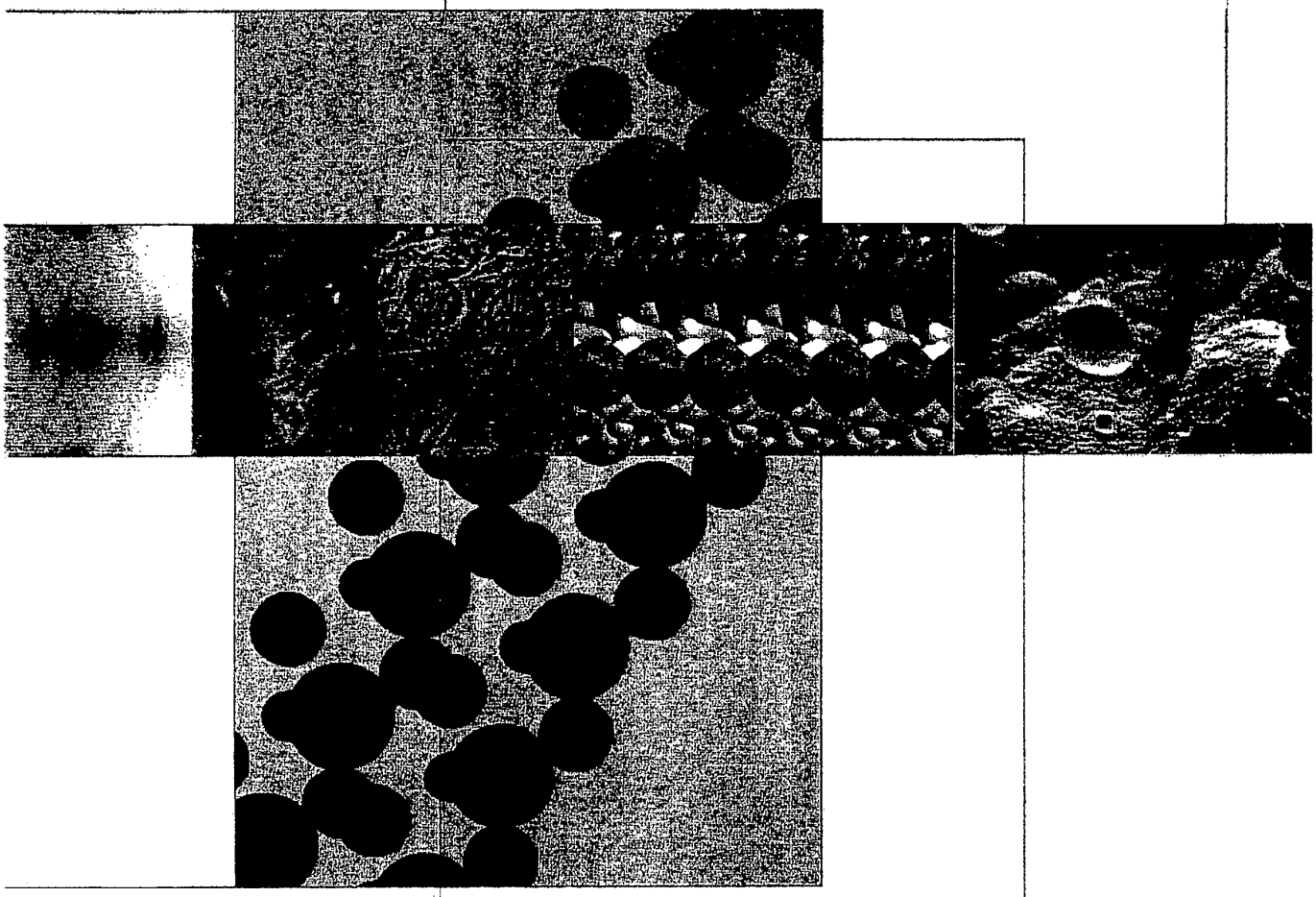
一、參加第十四歐洲高分子材料工程技術發展研討會相關參考文件

二、德國 IMT 研究中心微系統相關技術資料

三、德國 IMM 研究單位之相關微系統技術資料

一、第十四歐洲高分子材料工程技術發展研討會相關參考資料

Institut
für Polymerforschung
Dresden e.V.



Survey of research activities

Preface

The Institute of Polymer Research Dresden (IPF) is an independent research institute supported by the German federal government and the state government of Saxony. The institute is a member of the Wissenschaftsgemeinschaft G. W. Leibniz, and as all Leibniz institutes it has the mission to carry out research of supraregional importance and of interest for the whole German research community. The profile of the institute is characterized by a combination of both material and technology oriented studies ranging from application-oriented basic research to applied research projects. The work includes synthesis, modification and characterization of polymer materials as well as technological studies in polymer processing. It is related to three main fields which are divided into two priority topics each:

1. Defined polymer structures via polymer synthesis and melt modification
   - Synthesis of specific polymer architectures and functional polymers
   - Reactions in melts/Blends
2. Mechanisms of interactions at interfaces and their control
   - Characterization and design of polymer interfaces
   - Biocompatible interfaces
3. Polymeric materials/Functionalization and modification of surfaces and interphases
   - Fibre-matrix interfaces in composite materials
   - Functionalized surfaces/Polyelectrolytes

Investigations on the influence of interface-induced phenomena in multi-phase systems and in polymer processing as well as the design of tailored interfaces are a common feature of the research works in all fields. Basic funds for the activities of the institute are provided by the federal and the state governments in equal shares. A considerable amount of additional funds is acquired through projects supported by public institutions (e.g. Federal Ministry of Education and Research, Saxon State Ministry of Science and Arts, European Union, Deutsche Forschungsgemeinschaft, Union of Industrial Research Association) and through direct contracts with industrial partners.
Co-operation with industry – both large and medium sized enterprises – is a particular concern and a strong effort of the IPF. Medium and long-term co-operations in projects with scientific background and with an approach related to basic research are favoured; however, the institute supports innovations in industry also by contract research and consulting activities.
The IPF is firmly integrated in the scientific community and co-operates with all major polymer research institutions in Germany and with a large number of research groups abroad, partly on the basis of formal agreements. Very tight connections have been established to the Dresden University of Technology. They are due to the appointments of the heads of the institutes as professors at the university, by lectures, seminars and practical training courses provided by scientists of the IPF, and by collaborative research work, e.g. within a collaborative research centre of the Deutsche Forschungsgemeinschaft. In addition, the Materials Research Network Dresden links the IPF with a number of neighbouring institutes which are engaged in related areas of materials research. The combination of their facilities and competences makes Dresden to a leading centre of materials research in Germany.

Prof. Dr. Klaus Lunkwitz
Scientific Director

Günter Mateika
Administrative Director

Dresden, July 2000

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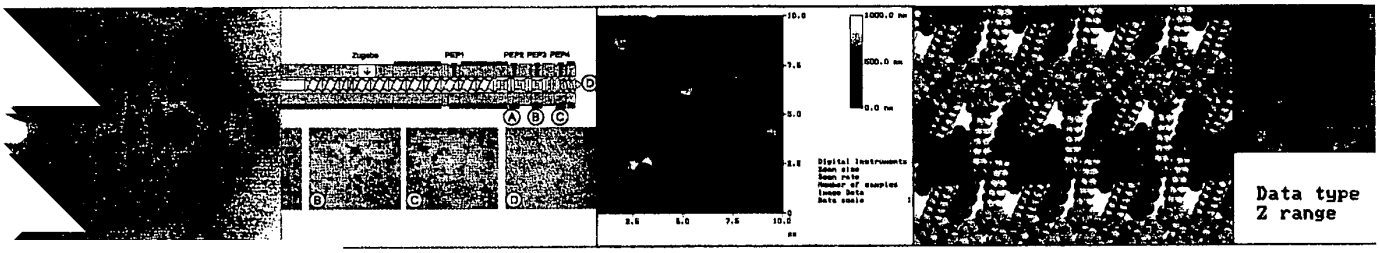
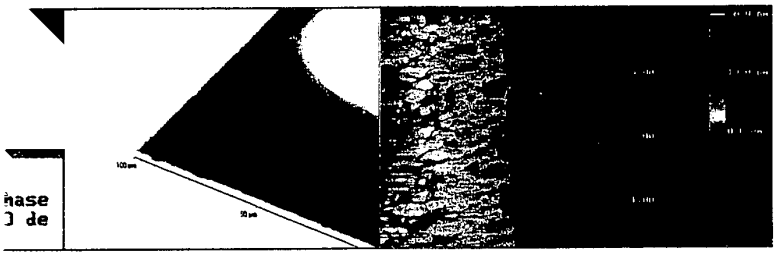


Projects within the main fields of research are, in general, handled by teams consisting of researchers affiliated to different institutes and departments. Therefore, the current research activities are presented in this brochure independent of the institute's organizational structure and under quite a large number of headlines. This shall help the reader to find easily a particular topic of interest.



1. **Synthesis of defined polymer architectures and functional polymers**
1.1 Dendritic polymers
1.2 Functional polymers
1.3 Multiblock copolymers

2. Reactions in polymer melts
2.1 Coupling and modification reactions
2.2 Reactive extrusion - Extrusion monitoring
2.3 Blends and blend morphology development

3. **Physico-chemical modification of polymer surfaces**
3.1 Modification of surfaces by surface-active agents and surface chemistry
3.2 Modification of surfaces by polyelectrolytes and polyelectrolyte complexes
3.3 Radiation-induced modification
3.4 Surface characterization

4. **Micro-/nanostructures and thin films**
4.1 Structure formation on micropatterned surfaces and polymer microstructures
4.2 Preparation, structure and properties of ultrathin organic layers
4.3 Modification of membranes

5. **Biocompatible materials**
5.1 Biointerfacial phenomena
5.2 Biosurface engineering
5.3 Low-pressure plasma treatment

6. **Development of novel polymer materials**
6.1 Compound materials and polymer processing
6.2 Composite materials
6.3 Fibre formation by melt spinning

7. Characterization of polymers
7.1 Polymer analysis
7.2 Structure and mechanical properties of polymers
7.3 Rheology

Research equipment development
Library

1.1 Dendritic polymers

Keywords

Hyperbranched polyesters, polyamides and poly(thio ethers)

Photolabile and thermolabile hyperbranched polymers

Perfectly branched polyamide dendrimers

Dendritic polymers in blends and coatings

Investigations of structures and rheology of hyperbranched polymers

Research activities

Synthesis of hyperbranched polyesters with repetitive units of different flexibility and their modification with specific functional end groups

- Incorporation of polymerizable or initiating groups for application in coatings and for the synthesis of star-like polymers
- Modification with alkyl chains or linear polymer chains of different polarities for generation of hyperbranched amphiphilic polymers
- Utilization of reactive end groups of hyperbranched polymers in reactive blends

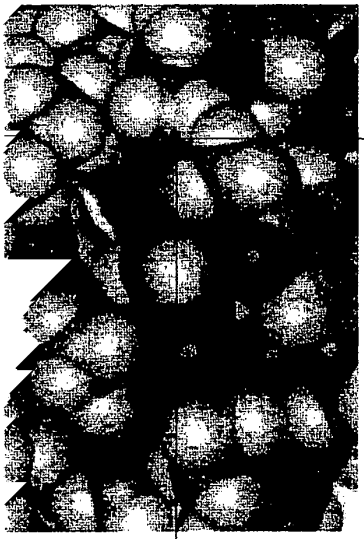
Hyperbranched polymers by unconventional reactions, e.g. synthesis of hyperbranched poly(ether amide)s via thermal ring-opening addition of nucleophiles to oxazolines or exploitation of thiol addition at double bonds to build up hyperbranched poly(thio ether)s. Investigations on the structural uniformity, the properties and potential applications of the polymers

Synthesis of photolabile and thermolabile globular polymers, e.g. via insertion of labile triazen units into the hyperbranched polymer framework. Molecular dispersed mixing of these labile polymers into a stable polymer matrix as nano-sized foaming agents

Synthesis and modification of perfectly branched polyamide dendrimer segments and dendrimers. Comparison of the perfect structures with the ones of less perfectly branched polymers. Investigations on surface modification with functional dendritic molecules by means of surface-sensitive methods

Detailed investigations on structure and properties of dendritic polymers, e.g. by high-resolution NMR spectroscopy (mainly for detection of different structural units), molar mass determination, and determination of the rheological properties and the reactivity. Support of synthesis and structural investigations by molecular modelling

1. Synthesis of defined polymer architectures and functional polymers



Functional and globular dendritic macromolecule

Prof. Dr. Brigitte Voit
Phone: +49-351-46 58-590
voit@ipfdd.de

Research activities

Polymeric layers with hydrophilic/hydrophobic, anionic/cationic and reactive groups for protein adsorption and coupling (biocompatible and biomimetic surfaces)

- Synthesis of alternating copolymers with functional and reactive side groups
- Synthesis of hydrophilic copolymers with reactive groups
- Reactions at surfaces to obtain functionalization and complexation properties

Surface modification using functional hydrogels built by radical polymerization with the aim of controlling mechanical properties as well as wetting and sorption behaviour in applications in medicine and in filtration processes

Polyamidines

- Influence of intermolecular and intramolecular interactions on structure and surface properties of polyamidines
- Structural influences on the formation of cyclic and macrocyclic amidines
- Acid-base interactions of polyamidines with low-molecular and polymeric agents

Functionalized polyolefines and polystyrenes

- Synthesis of functionalized polyolefines (e.g. -OH, -NH_2, -COOH) by metallocene-catalyzed copolymerization of ethene or propene with functional monomers
- Polymer-analogous reactions with polyolefines and polystyrenes functionalized with terminal or side groups
- Investigations on the structural influence of comonomers (short or long-chain branching) on the mechanical properties of polyolefines
- Grafting of polyolefines
- Synthesis of polymers with functional terminal or side groups by means of 'living' radical polymerization

Photolabile and thermolabile polymers

- Macroinitiators with azo and peroxide groups
- Block copolymers with thermolabile and photolabile groups
- Micro and nanopatterned materials
- Hyperbranched thermolabile and photolabile polymers

Keywords

Biocompatible and biomimetic surface modification

Polymers with acidic and basic groups

Macroinitiators

Photolabile and thermolabile polymers

Gas-phase polymerization of acrylic acid

Dr. Frank Böhme
Phone: +49-351-46 58-492
boehme@ipfdd.de

Dr. Volker Steinert
Phone: +49-351-46 58-351
steinert@ipfdd.de

Dr. Stefan Zschoche
Phone: +49-351-46 58-492
zschoche@ipfdd.de

1.3 Multiblock copolymers (segmented block copolymers)

Keywords

Synthesis and characterization of segmented block copolymers with polysulfone, polyether, poly(phenylene sulfide) and fluorinated polymeric segments

Design of polymers with tailor-made property profiles

Research activities

Synthesis of new segmented block copolymers preferably by polycondensation in the melt and controlled radical polymerization
- Characterization of structure and properties
- Up-scaling of polymer synthesis in a 1l-stirring autoclave or in a melt kneader

Structure-morphology-property relations in block copolymers, experimental and theoretical understanding using molecular modelling
- Molecular modelling of solid state structures of semicrystalline polymers
- Mean-field calculation of phase diagrams for estimation of phase separation in block copolymers and polymer blends

Segmented block copolymers in polymer blends
- Prove of the mechanism of compatibilization in immiscible polymer blends by block copolymers

Thermostable block copolymers with poly(diphenyl-phenylenoxide) segments

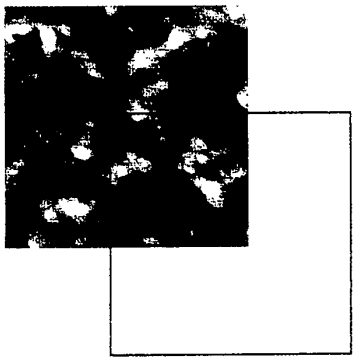
Modification of surface properties by incorporation of special surface-active segments in block copolymers
- Hydrophilization of polysulfone
- Investigation of the influence of surface segregation in fluorinated block copolymers with low surface energies
- Characterization of block copolymer surfaces (XPS, contact angle, AFM, XR)
- Investigation of the Lotos effect
- Investigation of the adsorption of model proteins on block copolymer surfaces

Special features of instrumentation

Hardware and software for molecular modelling and mean-field calculations
1l-polymerization autoclave
1l-pressure stirring autoclave
Spin coater

Work station for molecular modelling (RS/6000): computer simulation of a semifluorinated polyester

Surface morphology of a semifluorinated polyester (prepared by spin coating from trifluoroacetic acid/chloroform solution)



Dr. Doris Pospiech
Phone: +49-351-46 58-497
pospiech@ipfdd.de

Chain extension, branching and cross-linking of terminal group containing polycondensates by conversion with bifunctional and multifunctional coupling agents (such as oxazolines, oxazinones)

Utilization of selectively reacting multifunctional coupling agents for the in-situ compatibilization of polymer blends and synthesis of block copolymers

Functionalization of polymers by polymeranologous reactions in melt

Investigation on the influence of coupling reactions in interface-reactive two-component injection moulding

Thermally controlled curing reactions in melt (catalytic influence on single and multi-step reactions)

Keywords

Reaction mechanisms in polymer melts

Controlled coupling reactions and functionalization of polymers

In-situ compatibilization of polymer blends

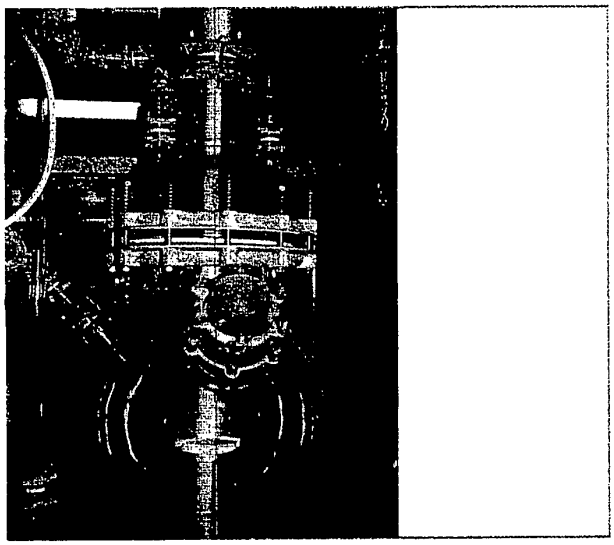
Synthesis of block copolymers by coupling reactions

Coupling reactions in the interfaces of heterogeneous polymer blends

Research activities

Synthesis and application of coupling agents with functional groups of different reactivities and investigation of their selectivities in chemical reactions

Reactions in polymer melts



Reaction vessels for large badges

Melt discharge from the DACA micro-compounder

Dr. Frank Böhme
Phone: +49-351-46 58-492
boehme@ipfdd.de

Dr. Dieter Lehmann
Phone: +49-351-46 58-392
lehmann@ipfdd.de

2.2 Reactive extrusion - Extrusion monitoring

Keywords

Reactive binary and ternary polymer blends

Defined degradation and build-up of polymers

Polymer-analogous reactions

Coupling reactions

In-line and on-line process monitoring

Research activities

Influence of processing parameters on the phase morphology of reactive blend systems based on polyamide melts with functionalized polyolefines, polytetrafluoroethylene, styrene maleic anhydride (SMA) copolymers and reactive elastomers in the extruder

Reactions of low-molecular substances (carboxylic acids, anhydrides) and of polymers containing carboxylic or anhydride groups with polyamides for build-up of block and graft copolymers in an extruder

Polymer-analogous reactions of polymers containing anhydride groups in the melt

Binary and ternary blend systems based on polyamide 6 mixed with SMA copolymers and/or reactive elastomers

In-line and on-line control of flow, mixing and chemical reactions simultaneously at different positions along the processing unit of a twin-screw extruder during operation:

- Labelling of pellets with radioactive nuclides and detecting the impuls rate of the extruder walls penetrating gamma radiation by use of scintillation counters (mean residence time, residence time distribution)
- Rapid sampling and quenching of melt probes from the running extruder and application of analytical methods for quantification of particle dynamics during plastification/melting and homogenization in blends

Reactive coupling of polymers within the interface using the heat of fusion to activate bonds

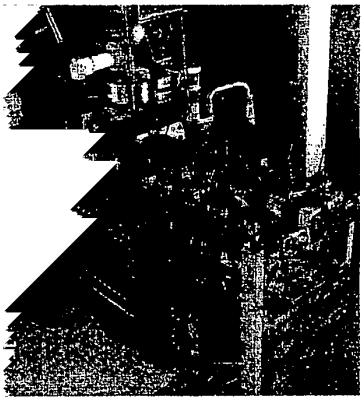
Quantitative determination of individual components in polymer mixtures in reactors and processing machines (mainly extruders) during operation and determination of conversion and endpoint of polymer reactions by means of IR and Raman high-temperature and high-pressure probes (mid IR/ATR, near IR/transmission, and diffuse reflectance)

Special features of instrumentation

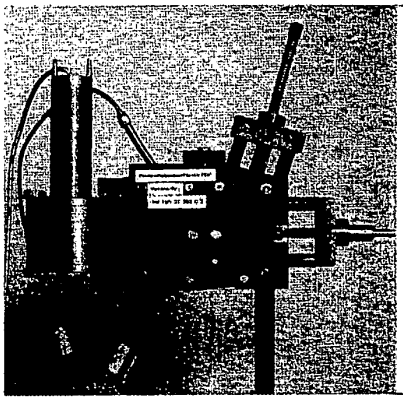
Experimental twin-screw extruder ZSK 40

- Gravimetric feeders for solid and liquid components, vacuum evaporation facility
- Special control and sampling plates for taking probes from the running extruder

IR process spectrometer for mid IR with an ATR dipping probe
IR process spectrometer for near IR with diffuse reflectance and transmission probes
Raman process spectrometer with high-temperature and high-pressure probe
All IR and Raman probes can be adapted to all extruders (cf. 6.1) and to different reactors.



Experimental pilot plant
twin-screw extruder ZSK 40



Special control and
sampling plate

Dr. Michael Stephan
phone: +49-351-46 58-314
stephan@ipfdd.de

Dr. Dieter Fischer
phone: +49-351-46 58-228
fisch@ipfdd.de

Research activities

Fundamental research on the creation of desired morphologies during melt mixing
- Melt rheology and morphology of heterogeneous polymer blends
- Modification and characterization of interphases
- Correlation between blend morphology and blend properties
- Realization and modelling of physical and chemical processes in screw reactors/ extruders (off-line and on-line analysis)

Improvement of the interaction energy at interfaces and phase morphology stabilization by
- Creation of covalent bonds by radically initiated graft reactions, addition of reactive coupling agents and compatibilizers, application of functionalized blend components utilising the functionalities for reactive blending
- Creation of interpenetrating polymer networks

Antistatic finishing of thermoplastics
- Modification with electrically conductive polypyrrole and carbon black
- Correlation of deliberately created morphologies with electrical and mechanical properties

Special features of instrumentation

Devices for mixing, modification and injection moulding of small material amounts:
- Miniatur mixing reactor EK-3-5C (volume 3 cm³)
- Microtruder RC-0250 (throughput: 10 to 160 g/h)
- Micro-compounder and Micro-injector, (volume 4.5 cm³)

Apparatus for angle-dependent laser light scattering measurements at thin layers

GNOMIX-pVT apparatus for the determination of pVT data and phase transitions up to 400 °C

Device for the determination of surface and interfacial tension of polymer melts by the pendant drop method at temperatures of up to 344 °C

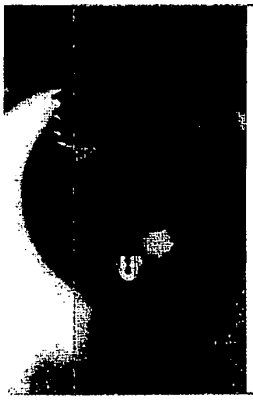
Keywords

Heterogeneous blends based on the following matrix polymers
- **Polypropylene**
- **Polyethylene**
- **Thermoplastic polyurethane**
- **Polyamide 6**
- **High performance polymers**

Interpenetrating polymer networks

Electrically conductive polymer composites

Light microscopic investigations for quantification of the phase morphologies in blends



Melt drop for determination of the surface tension according to the pendant drop method

Dr. Jürgen Pionteck
Phone: +49-351-46 58-393
pionteck@ipfdd.de

Dr. Petra Pötschke
Phone: +49-351-46 58-395
poe@ipfdd.de

3.1 Modification of surfaces by surface-active agents and surface chemistry

Keywords

Formation and structure of surfactant layers on polymer surfaces

Grafting of polymer layers on solid substrates

Research activities

Characterization of the interactions between polymers surfaces and surface active agents in aqueous solutions by atomic force microscopy (topography, direct force-measurements, e.g. by the force-volume mode), determination of the adsorbed amount by titration of the surfactant, in-situ ATR FTIR, and streaming potential measurements

Investigation of the structure of the adsorbed layer at the solid/liquid interface and, after drying, at the solid/gas interface using the methods mentioned above as well as X-ray photoelectron spectroscopy (XPS) and studies on the wetting behaviour

Investigation of the competitive adsorption at polymers from surfactant mixtures (ionogenic and non-ionogenic) or from emulsions (oil-in-water type) and characterization of the structure of the surface layers

Study of correlations between the interfacial parameters and the behaviour of the surfactant-modified solids during processing and use, derived from correlations between changes in the properties of the polymer surface (e.g. hydrophilicity/hydrophobicity, electrostatics, handle) and the structure of the surfactants adsorbed on the polymer surface or diffused in the bulk polymer. Application to practical problems of the polymer and textile industries

Grafting of weak polymer layers on solid substrates ('grafting-from' technique) to get specific changes in the surface properties, e.g. to produce reversibly switchable surfaces (for instance change from hydrophilic to hydrophobic behaviour)

3. Physico-chemical modification of polymer surfaces

Potentiometric determination of the concentration of ionic surfactants in aqueous solutions

Dr. Ingrid Grosse
Phone: +49-351-46 58 317
grossei@ipfdd.de

Dr. Sergei Minko
Phone: +49-351-46 58 271
minko@ipfdd.de

Modification of surfaces by polyelectrolytes and polyelectrolyte complexes 3.2

Research activities

Surface modification of polymer and inorganic materials of various geometries by polyelectrolytes (PEL) and polyelectrolyte complexes (PEC) for the creation of defined surface properties (polarity, charge, hydrophilicity/hydrophobicity, wetting, binding capacity for proteins and drugs), utilization of specific synergistic charge effects of PECs

Studies on correlations between the molecular structure of PELs and the properties of their PECs in solutions and at interfaces
- Conditions for the stability and defined particle size of dissolved or dispersed nanoscale PECs
- Thermodynamics und kinetics of PEL and PEC surface interaction
- Influence of molecular and external parameters on adsorption and complexation
- Polyelectrolyte conformationn in the dissolved, complexed and adsorbed state

Description and optimization of adsorption and flocculation processes by PEL or PEC in charged polymer/colloidal systems
- Application and exploration of new principles for the separation process, especially of dual systems, for the flocculation of industrially and ecologically relevant aqueous dispersed systems
- Stabilization of latex dispersions by PEL systems

Deposition and properties of organized polymer multilayer systems
- Defined control of vertical layer architectures and surface properties and description of interaction forces between the partial layers
- Exploration of applications in the biomedical and sensor field, creation of lateral microstructures at surfaces

Special features of instrumentation

Zetasizer for the combination of electrokinetic and particle size measurements
Lumifuge for rapid characterization of demixing processes
Fibre optical flocculation sensor for in-situ characterization of flocculation processes under practically relevant conditions
In-situ ATR-FTIR attachment with dual channel sorption cell operating by a pseudo-double beam principle (single beam sample reference – SBSR)

Keywords

Polyelectrolytes and polyelectrolyte complexes

Adsorption at the liquid/solid interface

Stabilization and flocculation of dispersed systems

Dual systems for flocculation

Multilayer systems

Investigation of flocculation by means of Labfloc®

In-situ ATR-FTIR attachment with dual channel sorption cell

Dr. Ulrich Oertel
Phone: +49-351-46 58-494
oertel@ipfdd.de

Dr. Martin Müller
Phone: +49-351-46 58-405
mamuller@ipfdd.de

3.3 Radiation-induced modification

Keywords

Electron beam irradiation of polymers

Radiation-modified fluoropolymers

Polytetrafluoroethylene (PTFE) micropowders

Research activities

Modification of fluoropolymers:
- Studies on the reaction routes and mechanisms initiated by irradiation of fluoro-polymers in presence of reactants (O_2, NH_3, SO_2)
- Preparation of functionalized PTFE micropowders by radiation-induced partial degradation of high molecular weight PTFE and studies on their incorporation into other polymers (POM, PA)
- Crosslinking of fluoropolymers by radiation

Long-chain-branching and crosslinking of polypropylene

Modification of polyaramide membranes

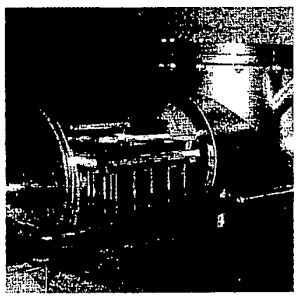
Preparation of membranes by irradiation of polyethylene/polymethacrylate

Service

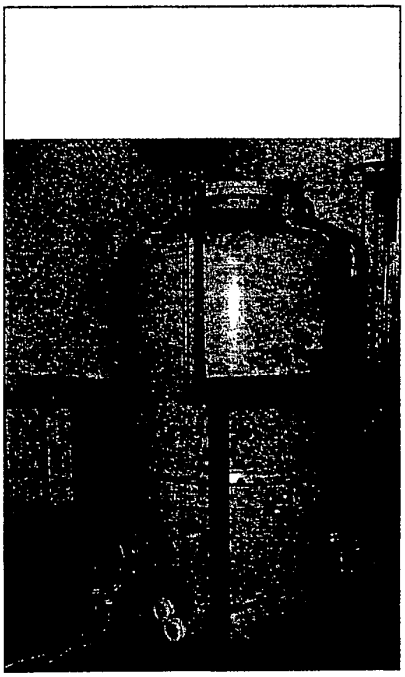
Among the polymer research institutions in the European community the IPF is the only one having an electron beam irradiation plant which is used both for IPF research activities and for service irradiation on a small scale. Electron radiation offers the unique possibility to provide energy for the initiation of chemical reactions inside the material within few seconds and independent of temperature and pressure.
Applications in the plastics industry are for example the radiation crosslinking of polyethylene and polyvinylchloride (hot water pipes, cable isolations, heat-shrinkable tubings and films), the vulcanization of elastomers and the degradation of PTFE to micropowders.

Special features of instrumentation

Universal electron beam irradiation plant with electron accelerator (electron energy: 0.6 to 1.5 MeV, beam power: 20 kW, continuous, batch and stationary irradiation procedures and experiments)



Irradiation vessel (open) with heatable sample support and clamping device for flat materials



Electron accelerator
ELV-2: pressurized tank

Dr. Uwe Lappan
Phone: +49-351-46 58-366
lappan@ipfdd.de

Helmut Dorschner
Phone: +49-351-46 58-231
dorsch@ipfdd.de

Research activities

Characterization of interfaces of polymeric materials including the development and adaptation of measuring techniques, polymer surface characterization with regard to their structures, chemical constitutions, reactivities and morphologies as well as their interactions with contacting solid, liquid, gaseous and solved substances

- Measurement of interface width and surface structure by means of X-ray and neutron reflectometry as well as by diffuse X-ray scattering under grazing incidence
- Direct determination of the interaction forces between solid surfaces in different media (gas, liquid, solution) by direct force measurements
- Indirect determination of interaction forces by physico-chemical measurements to estimate surface-energetic parameters such as reversible thermodynamic work of adhesion, surface (interfacial) free energy, and adsorption free energy
- Basic investigations on the influence of adsorption layers (ions, surfactants, polymers, polyelectrolytes, proteins) on the interaction forces in condensed systems
- Description of the reactivity of interfaces (polarity and acid-base properties)
- Determination of the surface charge and description of the charging process
- Characterization and interpretation of wetting phenomena at chemically and morphologically heterogeneous polymer surfaces
- Quantification of wetting and surface tension of polymer melts

Application of the results obtained with model systems to understanding and optimization of technological processes, e.g.

- Coating
- Development of composite materials
- Dispersion and coagulation processes
- Processes in textile finishing

Keywords

Surface-sensitive scattering methods (X-ray and neutron reflectometry)

Surface spectroscopy and force microscopy

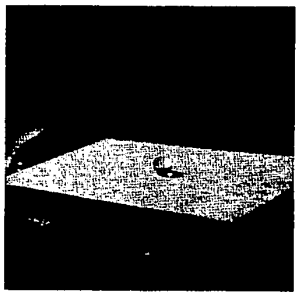
Direct force and adhesion measurements

Wetting and surface (interface) tension

Electrokinetics

Investigations on adsorption of substances from gaseous and liquid phases

Characterization of surface morphology



Water droplet at an ultrahydrohobically modified aluminium surface

Scanning force microscopic investigations (AFM) using the Nanoscope III Multimode with a cell for liquids

Dr. Frank Simon
Phone: +49-351-46 58-488
frsimon@ipfdd.de

Dr. Jochen Gutmann
Phone: +49-351-46 58-236
gutmann@ipfdd.de

Dr. Franz-Josef Schmitt
Phone: +49-351-46 58-222
schmfj@ipfdd.de

Methods and special features of instrumentation

Determination of interface width and surface structure
X-ray reflectometer with special monochromators (collimating multilayer optics, 2-bounce-channel-cut Ge-crystals) and oven,
Neutron reflectometers PNR and TOREMA in Geesthacht
X-ray scattering beam-lines at the HASYLAB synchrotron radiation laboratory in Hamburg for grazing incidence diffraction and diffuse scattering
Utilization of further equipments in Grenoble, Jülich, and Berlin (on request)

Surface spectroscopy
X-ray photoelectron spectrometer, under vacuum combined with a secondary ion time-of-flight mass spectrometer (XPS/ToF-SIMS)

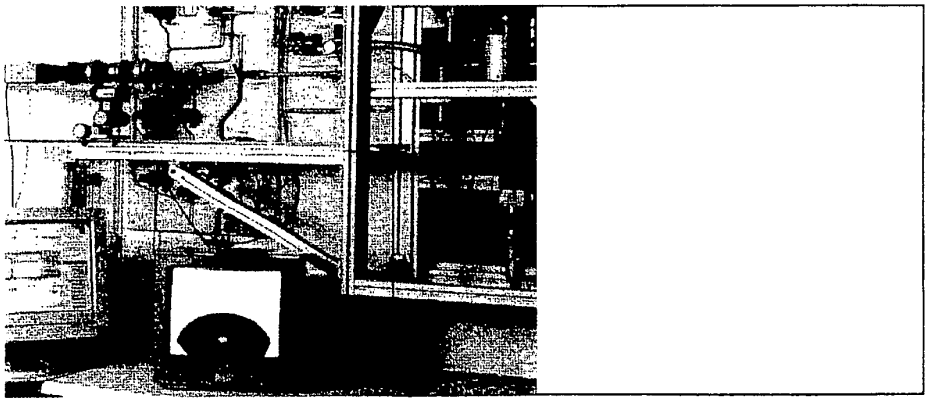
Direct force measurements/Characterization of the surface morphology
Surface force apparatus MASIF
Scanning force microscopes Multimode, Bio-scope

Electrokinetic methods
Electrokinetic analyzer for streaming potential measurements with powders, fibres and plates
Electro-osmosis device for powders, fibres and porous systems
Electrophoresis measuring system for dilute and concentrated dispersions and emulsions

Wetting and interfacial tension
Contact angle measuring devices
Tensiometers
Axisymmetric drop shape analysis - profile (ADSA-P) and axisymmetric drop shape analysis - contact diameter (ADSA-CD)
- Simultaneous determination of contact angle, surface tension, contact radius and volume of sessile liquid drops
- Cell for inverse contact angle measurements using air bubbles
- Determination of very small contact angles on heterogeneous and rough surfaces (< 20°)

Devices for determination of interfacial tension and wetting of polymer melts at elevated temperatures (up to about 400 °C)
Modified Wilhelmy technique with fibres to determine wetting and surface tension of polymer melts

Adsorption of substances from gaseous and liquid phases
BET-Liqui-Sorb-EL for low-temperature gas adsorption at finely divided solids and for sorption of vapours of different liquids
Microcalorimeter system TAM 2277 for titration calorimetry, flow-sorption calorimetry and solid-vapour phase interaction (perfusion cell)
Inverse gas chromatograph HP 6890



Device for simultaneous determination of density and surface (interfacial) tension of polymer melts at elevated temperatures

Dr. Cornelia Bellmann
Phone +49-351-46 58-327
bellmann@ipfdd.de

Dr. Karina Grundke
Phone +49-351-46 58-475
grundke@ipfdd.de

Research activities

Chemical heterogenization of surfaces in micrometer and submicrometer dimensions by soft-lithography, in particular by microcontact printing. Micropatterned surfaces are used
- to control wetting and dewetting processes including polymer film formation
- to control crystallization processes of both polymer and non-polymer systems in contact with tailored surfaces
- to study selective adsorption processes of polymers and biopolymers as well as cellular growth (cf. 5.1)
- to create microreaction environment

Preparation of both patterned and homogeneous ultrathin layers of surface-immobilized polypeptides by polymerization of activated amino-acids on initiator-modified surfaces: The obtained grafted polypeptide layers (Poly-(γ-benzylglutamate), Poly-(L-Lysine),Poly-(glutamicacid) with different surface properties (hydrophilic/hydrophobic, cationic/anionic) allow reversible conformational changes at the surface and therefore become interesting nanoscaled functional elements.

Preparation of nano-structured polymer templates from block copolymers and by ion radiation, nanowires and nanotubes by electrodeposition of metals and conducting polymers

Application of the tuning of surface properties in micrometer and sub-micrometer dimension to the design of tools for nanomanipulation units used for interaction with micrometer and sub-micrometer scaled objects in low-voltage and enviromental as well as in light microscopy

Design of molecularly tailored surfaces (e.g. with tunable adhesion or wetting behaviour) for nanomanipulation tools as an essential requirement for the control of matter by nanomanipulator sytems

Special features of instrumentation

Low-voltage scanning electron microscope with electron beam lithographic system
Reflective light microscope with integrated AFM
Inverse light microscope with multi-beam optical tweezers
Three-axes positionable nanomanipulation units

Keywords

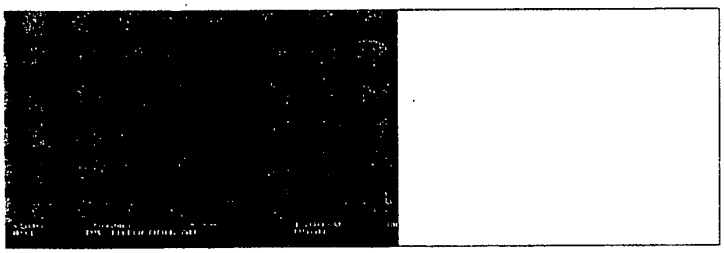
Microcontact printing

Surface polymerization

Wetting and dewetting of microheterogeneous surfaces

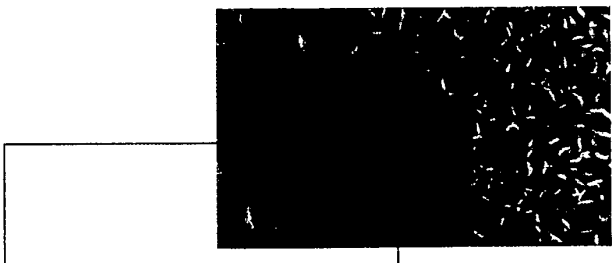
Nano-structured templates

Nanomanipulation techniques



Perforated polystyrene film produced by controlled dewetting of the polymer solution at a heterogeneous substrate

Polyethylene crystallization at a heterogeneous surface



Dr. Sergej Minko
Phone: +49-351-46 58-271
minko@ipfdd.de

Dr. Hans-Georg Braun
Phone: +49-351-46 58-548
braun@ipfdd.de

4.2 Preparation, structure and properties of ultrathin organic layers

Keywords

Functional Langmuir-Blodgett(LB) multilayers

Amphiphilic polymers

Polymer-metal complexes

Insulating layers

Adsorption of polyelectrolytes and polyelectrolyte complexes

Research activities

Basic investigations to obtain specific optical, electrical, dielectric or magnetic properties at solid surfaces, mainly by means of the LB technique

- Synthesis of functional amphiphilic polymers
- Investigations of electron and energy transfer in LB layers
- Coating of pre-structured substrates

Ultrathin organic insulating layers

Synthesis of model polyelectrolytes

- Diverse chemical functionalities by modification of a 'basic' polymer
- Application of sensor techniques, e.g. modification with fluorescent dyes

Application of LB multilayers as model surfaces to study basic problems of

- Adsorption of polyelectrolytes and polyelectrolyte complexes
- Influence of surface-near regions on the surface properties

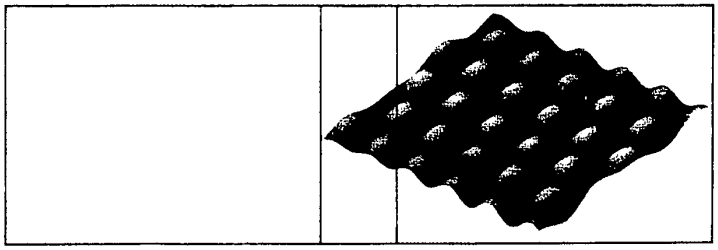
Build-up of LB multilayers from polymer-metal complexes and linear coordination polymers

- Investigation of their optical and electrical properties
- Study of energy and electron transfer processes
- Application in sensors

Special features of instrumentation

Langmuir-Blodgett troughs
Surface plasmon resonance spectrometer (SPR)
Brewster-angle microscope
Fluorescence, UV-VIS and FTIR spectrometers
In-situ measuring cells for investigation of adsorption processes by SPR and fluorescence spectroscopy

Molecular resolution of a Langmuir-Blodgett layer of dimethyldioctadecyl-ammoniumbromide (two monolayers) at mica



Measurement of surface plasmon resonance

Dr. Ulrich Oertel
Phone: +49-351-46 58-494
oertel@ipfdd.de

Research activities

Modification
- Optimization of selectivity, permeability and fouling behaviour of membranes by adjustment of polymer properties used for membrane formation (e.g. hydrophilicity, hydrophobicity, degree of swelling, surface charge, temperature and chemical resistance) by surface and bulk modification
- Membrane modification by electron beam irradiation and plasma (pre-)treatment
- Surface modification with polyelectrolytes and polyelectrolyte complexes
- Preparation and modification of membrane polymers by reactive extrusion

Characterization
- Determination of permeate flux, selectivity and retention
- Determination of characteristic values related to morphology, charge density, surface energy and chemical composition of the membrane polymer

Modelling of mass transport
- Determination of diffusion coefficient and interaction parameter between membrane polymer and feed component (Flory-Huggins parameter)
- Modelling of selective mass transport

Special features of instrumentation

Membrane-specific laboratory equipment for the determination of permeate flux and retention
Dead-end and crossflow filtration cells for testing porous and dense membranes in a pressure range from 0.05 to 10 MPa
UV-VIS spectrometer and densimeter for determination of concentrations and compositions

Keywords

Surface and bulk modification

Membrane characterization

Modelling of mass transport

Characterization of membranes in the pervaporation equipment

Dr. Siegfried Berwald
Phone: +49-351-46 58-396
berwald@ipfdd.de

Dr. Jochen Meier-Haack
Phone: +49-351-46 58-519
mhaack@ipfdd.de

5.1 Biointerfacial phenomena

Keywords

Electrosurface phenomena

Proteins at interfaces

Cell-surface interactions

Hemocompatibility testing

Research activities

Electrosurface phenomena
- Combined determination of zeta potential and interfacial conductivity at planar solid surfaces
- Asymmetrical ion adsorption and dissociation of sterically hindered surface sites
- In-situ characterization of adsorption of biopolymers by novel methods combining electrokinetic and spectroscopic techniques
- Microfluidics: Development of electrokinetic transport systems

Proteins at interfaces
- Qualitative and quantitative detection of human plasma proteins
- Characterization of adsorption processes
- Properties of protein layers
- Competition and exchange phenomena
- Adsorption-induced changes of three-dimensional structure and biological activity

Cell-surface interactions
- Effect of adsorbed proteins and biomolecular microstructures at polymer interfaces on cell adhesion and cell status
- Micromechanical adhesion experiments with individual cells
- Quantification of receptor expression, proliferation, and apoptosis

Hemocompatibility testing
- Static and dynamic incubation with human whole blood in vitro
- Determination of coagulation, complement activation, and activation and adhesion of thrombocytes

Special features of instrumentation

Microslit electrokinetic set-up for determination of zeta potential and interfacial conductivity

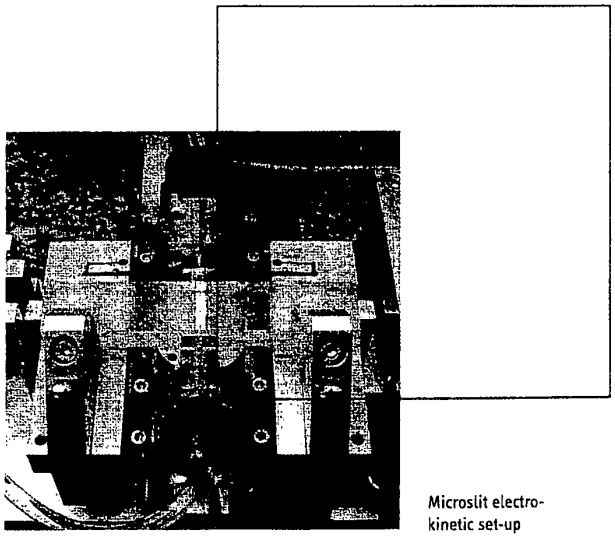
Combination of electrokinetic and spectroscopic techniques to study the formation of biomolecular layers

Spectroscopic ellipsometer, sub-monolayer ellipsometer to detect protein surface concentrations and the dynamics of adsorption processes

ATR-FTIR, micro-DSC and circular dichroism for conformational analysis of proteins

5. Biocompatible materials

Activated thrombocyte attached to a polymer film



Microslit electrokinetic set-up

Dr. Carsten Werner
Phone: +49-351-46 58-285
cwern@ipfdd.de

Isothermal microcalorimetry to measure protein adsorption enthalpies

HPLC with fluorescence detector and MS-TOF detector for analysis of proteins and amino acids

Fluorescence spectrometer, Lumi imager to analyze biological activity of enzymes and to evaluate immunoblots

Micromechanical cell adhesion experiments to quantify adhesion strength

Laser scanning cytometry, flow cytometry, confocal laser scanning microscopy, atomic force miscroscopy for analysis of cells at interfaces

Environmental scanning electron microscope for manipulation and study of microstructures at atmospheric pressure

Static and dynamic incubation systems for hemocompatibility assays

Research activities

Hemocompatible polymer interfaces
- Covalent immobilization of anticoagulant and fibrinolytic proteins; immobilization of synthetic anticoagulants at functionalized polymer surfaces
- Protein-resistant polymer interfaces by synthetic polymer structures

Control of cell surface interactions by designing polymeric substrates
- Functionalization of polymer materials for cell therapies by proteins of the extracellular matrix and other biomolecules
- Surface modification of resorbable polymers to direct the integration of tissue and the degradation behaviour
- Development of scaffolds for tissue engineering strategies

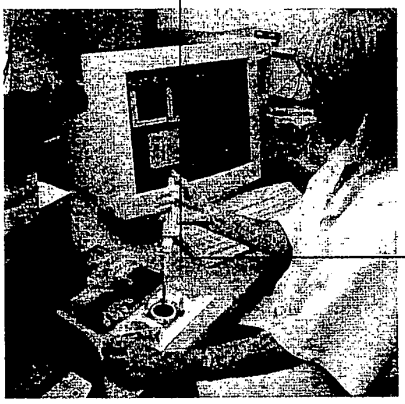
Keywords

Bio-inert modification of polymer surfaces

Biomolecular surface design of stable and resorbable polymer materials

Surface modification of polymers utilizing biomimetic molecules

Cell analysis by means of the Laser Scanning Cytometer



Dr. Carsten Werner
Phone: +49-351-46 58-285
cwern@ipfdd.de

5.3 Low-pressure plasma treatment

Keywords

Functionalization of polymer surfaces

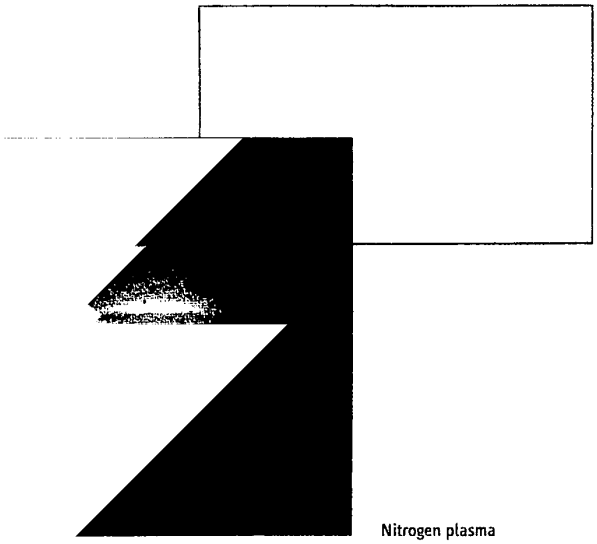
Chemical and physical surface characterization

Research activities

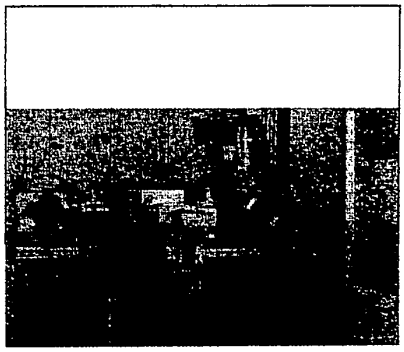
- Development of biocompatible polymer materials by chemical reactions on plasma activated surfaces and by plasma immobilization of surfactants
- Surface modification of fluoropolymers by plasma treatment followed by polyelectrolyte adsorption
- Fundamental investigations on plasma-polymer interactions
- Surface characterization by X-ray photoelectron spectroscopy, infrared spectroscopy, atomic force microscopy, electrokinetic measurements, contact angle measurements and in vitro hemocompatibility tests

Special features of instrumentation

- Multichamber high vacuum system, plasma excitation: 2.45 GHz microwave and 13.56 MHz radio frequency, gases: Ar, H_2, N_2, O_2, NH_3, SO_2, two separate lines for vapor phase media
- Plasma diagnostics by mass spectrometry and optical emission spectrometry
- Surface diagnostics without breaking the vacuum by X-ray photoelectron spectroscopy and infrared spectroscopy



Nitrogen plasma



High vacuum system for plasma treatment

Dr. Mirko Nitschke
Phone: +49-351-46 58-520
nitschke@ipfdd.de

Compound materials and polymer processing 6.1

Research activities

Contributions to the field of materials design and technological development by studies on the interrelations between the formulations and the process design on the one hand and the behaviour of the resulting material on the other hand

Design of compound materials and development of related technologies
- Physical and reactive compounding of thermoplastics and thermoplastic elastomers
- Polymer nanocomposites
- Plastics with novel and classical fillers and reinforcements
- Reactive compounding of biologically degradable materials

Investigations on extrusion processes
- On-line monitoring (NIR, MIR, ATR, Raman) of the process
- Distribution and dispersion processes of fillers and fibres
- Comparison of compounding units and processes

Short-fibre reinforcement of thermoplastics

Extrusion of semi-finished products (sheets, tubes, blown films, single and three layer flat films, testing profiles)

Applied tests (processability, fire behaviour, flammability, ageing, weathering)

Keywords

Design of compound materials

Reactive and physical compounding

Investigation of compounding processes

Extrusion of semi-finished products

Injection moulding of test specimens

Development of novel polymer materials 6.

Pilot plants for plastics processing

Processes and special facilities

Pilot plants for processing of thermoplastic materials

Compounding
Co-rotating twin-screw extruders (D = 25 to 30 mm, L/D 24 to 51, screw speed up to 1200 min^{-1})
Counter-rotating twin-screw extruder (D = 27 to 35 mm, L/D 17 to 28)
Co-kneader (D = 30 mm, L/D max. 18, with a choice of melt extruder or chill roll and cooling belt, e. g. for powder coatings)
Peripheral units for all compounding facilities:
- Gravimetric feeders of different sizes for pellets and powders, also for badly flowing materials
- Various feeders for liquids including pasts
- Side feeders for powders and pellets

Laboratory rolling mill and laboratory press

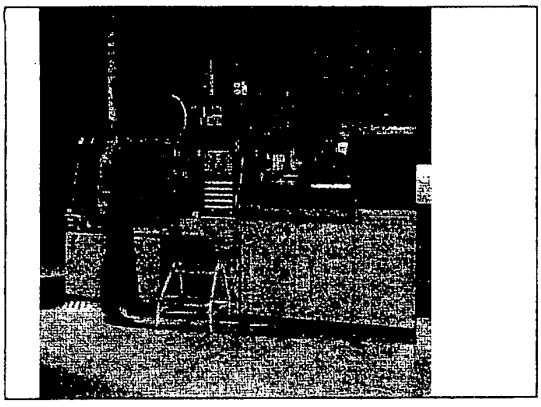
Process-analytical test stand with screw reactor 40/50 D (cf. 2.2)

Reprocessing
Fluid mixers 4 to 150 l
Pelletizers

Extrusion
Single-screw extruders (D = 30 and 32 mm, L/D 23 to 36)
Twin-screw extruders (cf. compounding)
Extruder downstream equipment:
- Sheet discharge unit up to 4 mm
- Flat film extrusion line (chill-roll and ABA-coextrusion)
- Blown-film discharge unit
- Pipe discharge unit
- Profile-bar discharge for test specimens

Injection-moulding
Injection moulding machines of up to 1000 kN clamping force
Two-components moulding machine
Units for thermosets and for unplasticized PVC
Injection-moulding tools for:
- Specimens according to CAMPUS
- Test sheets (variable thicknesses)
- Two-components tensile specimens
- Single-cavity mouldings for low amounts of material
- Spiral test
- Special specimens (DMA, etc.)

Cellular hollow microsphere (broken up) in polypropylene



Injection moulding machine for two-components injection moulding

Bernd Kretzschmar
Phone: +49-351-46 58-338
kretzsch@ipfdd.de

Dr. Udo Wagenknecht
Phone: +49 351 46 58-433
wagenknt@ipfdd.de

Research activities

Development of sizings for surface modification of E-glass filament yarns applied during the spinning process to improve the adhesion strength with thermosetting and thermoplastic matrices and characterization of the modified fibre surfaces using wetting measurements, streaming potential measurements, scanning electron microscopy, scanning force microscopy, inverse gas chromatography, and spectroscopic methods (FTIR, XPS, TOF-SIMS) in order to find relations between chemical structure and surface properties

Investigation of the influence of surface properties of reinforcing materials on the structure and properties of interphases, characterization of relations between the thermodynamic reversible work of adhesion and the work of adhesion determined by micromechanical tests

Characterization of the interphase properties (SFM, SFM in combination with thermal analysis or microthermal analysis μTA™ single fibre dynamic load test) and investigation of the influence of tailored interphases on the processing behaviour of composites. Investigation of relationships between surface, interphase and final composites properties

Investigation of differently modified interphases and composite properties influenced by hydrothermal treatments

Modification of polymer matrices to improve fibre-matrix interaction

Investigation of the influence of processing parameters on the resulting composite properties

Development of continuous-fibre reinforced thermoplastics from commingled yarns and from reinforcing filaments and thermoplastic split films

Dimensioning of composite parts

Calculation and formation of stress-field aligned fibre preforms for composite components by the tailored fibre placement technology (modified embroidery technique)

Keywords

Surface modification of E-glass fibres during the spinning process

Surface characterization of reinforcing fibres

Design and characterization of interphases

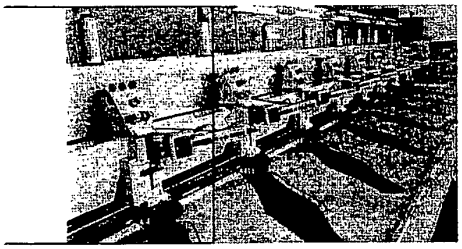
Reinforced thermoplastics

Reinforced thermosets

Composites testing

Textile preforms aligned to stress fields in composite components (tailored fibre placement)

Polymer-metal composites



Production of tailored fibre placement preforms by means of an embroidery machine with eight sewing heads

Melt discharge in glass-fibre spinning

Studies on the applicability of composite materials and their application-specific characterization

Fracture-mechanical characterization of composites and modelling of fracture behaviour (cf. Characterization of plastics)

Structuring and characterization of interphases between metal surfaces and reactive polymers using DMA, DSC and thermal microscopy

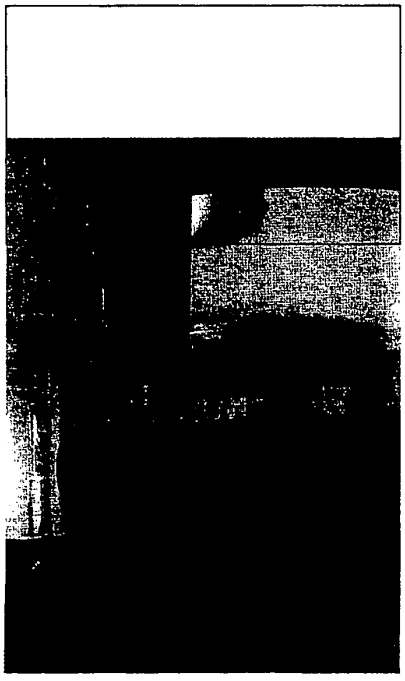
Methods and special features of instrumentation

Spinning and surface modification of E-glass filaments
Glass fibre spinning pilot plant equipped with multi-step sizing application

Characterization of interphase properties
Wetting measurements of single fibres (Wilhelmy technique)
Single-fibre pull-out (temperature range up to 500 °C, force range up to 1.5 N)
Single fibre dynamic load test for dynamic investigation of interphases in dependence on frequency and strain amplitude as well as microfatigue investigations

Composite processing
Air texturing machine DS 90 for manufacture of hybrid (commingled) yarns
Vertical prepreg plants for fabrics and unidirectional prepreg equipment
Filament winding unit for thermoplastics for manufacture of pipes and rings (up to 100 mm internal diameter)
Different laminating presses (clamping forces up to 1000 kN, temperature range up to 400°C, working areas up to 450 mm x 450 mm, PC-controlled)
Laboratory equipment for resin injection according to the RTM method

Manufacture of preforms with the tailored fibre placement technology
Electronically controlled embroidery machines (embroidery area per head 710 mm x 750 mm and 645 mm x 785 mm)
Robot with sewing head (3D ready making)
Equipment for ready making of 2D preforms



Single-fibre pull-out experiment

Dr. Horst Frenzel
Phone: +49-351-46 58-319
frenzel@ipfdd.de

Dr. Edith Mäder
Phone: +49-351-46 58-405
emaeder@ipfdd.de

Dr. Konrad Gliesche
Phone: +49-351-46 58-320
gliesche@ipfdd.de

Research activities

Basic investigations on spinning of polymer fibres in the velocity range between 500 and 6000 m/min

Mathematical modelling of fibre formation processes (including multi-filament melt spinning processes like cord, staple fibre, and spunbonded non-woven processes) with user-friendly model handling and presentation of results by means of visualized computer dialogue programmes

Spinning of special fibres (biologically degradable and biocompatible fibres, elastomeric fibres of thermoplastic polyurethanes and crosslinkable thermoplastic polyolefines, electrete fibres, fibres of higher meltable polymers)

On-line coupling of reactive extrusion and melt spinning using a twin-screw extruder

Basic investigations on the correlations between the rheological melt properties and high-speed shaping (spinnability) of thermoplastic polymers and their modifications

Application of dynamic process analysis devices and utilization of signal-theoretical methods of data evaluation and compression (spectral and correlation procedures) to process stages of fibre formation, of fibre temperature influence, of fibre orientation, and of fibre structure formation in different versions of the technological process

Special features of instrumentation

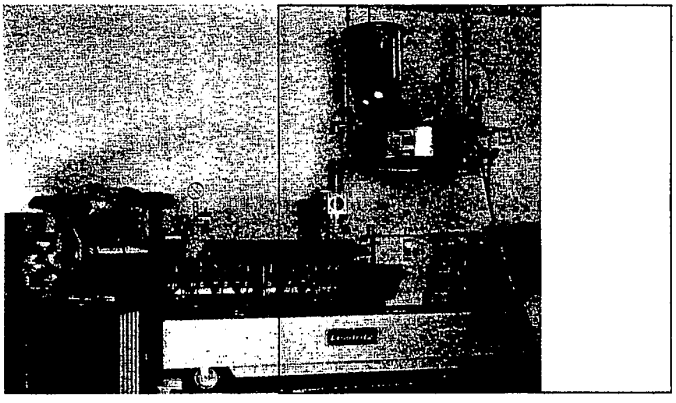
Laboratory high-speed spinning equipment designed according to demands of industrial practice
- Three heatable godet pairs (velocity up to 7200 m/min)
- High-speed wind-up device (velocity up to 6000 m/min)
- High-temperature single-screw extruder (temperature up to 450 °C)
- Twin-screw extruder (temperature up to 350 °C), throughputs each up to 3 kg/h

Plunger spinning device for low amounts of specimens (minimum amount required: 10 g, spinning velocity up to 1000 m/min, temperature up to 420 °C)
Laboratory drawing equipment
Measuring instruments for on-line measurements in the fibre line
- Fibre tensile strength
- Fibre temperature
- Fibre velocity

Measuring instruments for sonic residence time in polymer fibres

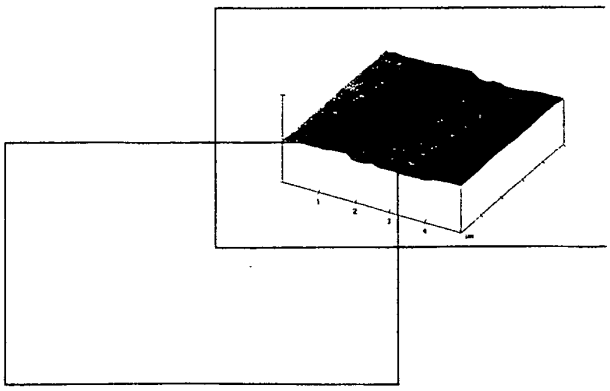
Keywords

Basic investigations on melt spinning of organic polymers including special and high performance polymers



On-line coupling of twin-screw extruder and melt spinning

Surface topography of PHB filaments: fibrillar structure



Prof. Dr. Roland Beyreuther
Phone: +49-351-46 58-301
probey@ipfdd.de

Keywords

Analysis of the chemical and physical structure and of the molecular weight of polymers:

Chromatography/MALDI-TOF-MS/elemental analysis

Molecular spectroscopy/ellipsometry

Thermal analysis

Light scattering

Research activities

Oligomeric model compounds and new polymer architectures: Characterization of the molecular structures and reactivities

Study of composition, morphology and properties of block copolymers, heterogeneous polymer blends, fibres and composites: Investigation of functionalities, phase behaviour, order, and orientation

Characterization of modified plastics surfaces and of thin surface layers by optical reflection methods

Development of fundamental concepts of on-line/in-line process analysis of polymer melts using infrared and Raman spectroscopy

Methods and special features of instrumentation

The following methods are used separately or in combination. According to the actual analytical task specific methods or experimetal set-ups are developped.

Chromatographic methods/MALDI-TOF-MS/elemental analysis
Size exclusion chromatography and viscosity measurements (operating range between 20 and 190°C) for characterization of molecular weight distributions of polymers

On-line coupling of liquid chromatography with FTIR or NMR

HPLC and SFC (supercritical fluid chromatography) for investigation of low molecular weight substances like additives and oligomers

Gas chromatography with mass-selective detection for investigation of volatile substances, coupled with Pyroprobe 2000 for characterization and identification of polymers and cross-linked polymers by thermal fragmentation

7. Characterization of polymers

Preparation of the measurement with the differential scanning calorimeter DSC 7

MALDI-TOF-MS (matrix-assisted laser desorption ionization time-of-flight mass spectroscopy) for characterization of oligomers and low molecular weight substances

Elemental analysis for C, H, N, S

Infrared and Raman spectroscopy
FTIR spectroscopy for qualitative and quantitative analysis of polymer materials including different methods of reflection (ATR, DRIFT, IRRAS) and the photoacoustic spectroscopy (PAS)

FT-Raman spectroscopy of polymers as complementary method for FTIR spectroscopy

FTIR and Raman microscopy for characterization of contaminations/inclusions and studies of heterogeneity

NIR spectroscopy for quantitative process analysis using various in-line-probes (transmission, diffuse reflection) and for determination of composition of mixed solid materials

Raman process analysis (holographic imaging spectrometer with CCD detector, excitation laser wave length at 785 nm)

NMR spectroscopy
^{1}H, ^{13}C, ^{19}F, and hetero-nuclei NMR spectroscopy for structural characterization of soluble polymers and low-molecular-weight substances

Solid-state NMR for the characterization of structure and dynamics based on high-speed MAS and multiple-pulse techniques. Special focus on fluorine-19

NMR imaging: pulsed field-gradient NMR (PFG-NMR) for the measurement of diffusion and flow
Electrophoresis-NMR system (in-house built)

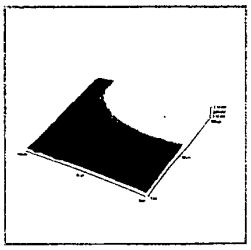
Characterization of thin layers
Spectroscopic ellipsometry (fast in-situ 44 wave-lengths ellipsometer)

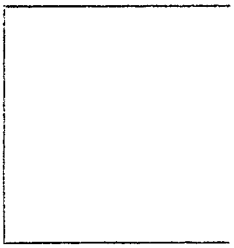
Thermal analysis
Thermogravimetry to study thermal degradation in inert or active atmosphere

Differential scanning calorimetry (DSC) to investigate melting and crystallization behaviour, glass transition behaviour, chemical reactions (e.g. crosslinking), and solid-solid phase transitions

Simultaneous thermal analysis (TG, DTA) coupled with mass spectrometry to study mass loss, heat flow and to detect the mass units of volatile products during the thermally activated processes (e. g. vapourization, degradation)

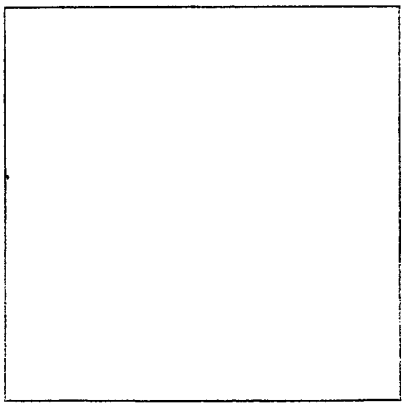
Micro-thermal analysis μTA™ 2990 (scan range 100 μm x 100 μm, resolution < 1 μm, 100 μm, modes of representation: topography, thermal conductivity)





Thermal conductivity of a sized glass fibre in an epoxy resin matrix studied by microthermal analysis μTA

FTIR laboratory



Dr. Klaus-Jochen Eichhorn
Phone: +49-351-46 58-256
kjeich@ipfdd.de

7.2 Structure and mechanical properties of polymers

Keywords

Structure-mechanical properties relationships in bulk and thin films

Structure, dynamics and conformation of polymers

Development of scattering techniques and X-ray microscopy

Influence of external fields on structure and phase transitions

Mechanical characterization of polymeric materials

Modelling of fracture behaviour of polymer composites

Testing of polymers, polymer-based composites, textiles, and fibres

Research activities

Establishment of relationships between the microscopic structure and macroscopic (mechanical) properties. Structural characterization by X-ray and neutron scattering as well as by static and dynamic mechanical measurements

Use of synchrotron scattering techniques at Hamburg, Berlin or Grenoble as well as a rotating anode set-up in Dresden for structure investigations at nanometer scale. Simultaneous wide and small angle scattering experiments, time-resolved diffraction, reflectometry for surface structures and thin film morphology, temperature and pressure jump experiments, in-situ shear or extrusion experiments as well as X-ray microscopy

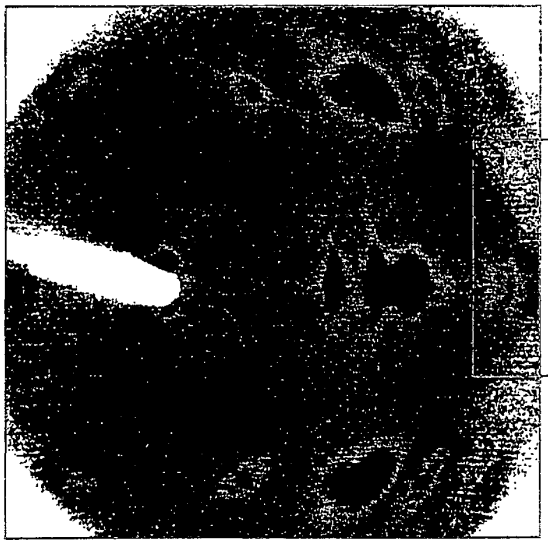
Small-angle neutron scattering for the investigation of chain conformation and grazing-incidence small-angle scattering from thin films (project in collaboration with the Research-Centre Jülich for the new research reactor FRM-2 in Garching) and neutron reflectometry at the research centre Geesthacht (in collaboration with GKSS Geesthacht)

Basic research in the field of miniaturized materials testing aiming at combinatorial investigations of materials properties. Transfer of miniaturized tests to selected industrially relevant applications

Investigation of molecular motions and interactions using dynamic-mechanical relaxation spectroscopy over a wide frequency, time, and temperature range. Elucidation and modelling of the influence of sample structure and thermo-mechanical history on material properties

Improvement of theoretical models describing the macroscopic mechanical and fracture-mechanical behaviour of short and long fibres as well as of particle-reinforced polymers, special emphasis on the inclusion of mixed-mode loading as well as incorporation of length and orientation distributions of fibres into the models. Combination of mechanical adhesion testing and structural investigations with high spatial resolution

Finite element modelling of (interfacial) crack propagation in polymer-based composites, including stress concentrations, formation of plastic zones and thermal stresses. Development of new custom-made testing methods for sophisticated mechanical tests, such as characterization of physical surface properties of modified fibres, measurements of fracture toughness under high load rates, and mechanical characterization of miniaturized samples



Two-dimensional X-ray diffraction pattern (WAXS) of poly(3-hydroxybutyrate) fibres showing high orientation of two crystalline modifications α and β

Prof. Dr. Manfred Stamm
Phone: +49-351-46 58-224
stamm@ipfdd.de

Dr. Dieter Jehnichen
Phone: +49-351-46 58-493
djeh@ipfdd.de

Dr. Jochen Gutmann
Phone: +49-351-46 58-236
gutmann@ipfdd.de

Techniques and special developments

X-ray diffraction (WAXS)
for the investigation of internal structure and short range order in polymeric materials using a wide-angle diffractometer equipped with area detector, structural analysis of polymeric phases, crystal structure, degree of cristallinity, orientation of crystalline domains, refinement of the crystal structure via molecular modelling and Rietveld analysis

Small-angle X-ray scattering (SAXS)
to determine supramolecular structures like long period, orientation, nanostructures, micro inhomogeneities, particle sizes and superstructures. The set-up is located at a rotating anode and incorporates focussing mirror optics and an area detector. Supplementary sample environment: furnace for high-temperature experiments, pressure chamber, rheometer for on-line orientation

Use of international large-scale research facilities
Synchrotron experiments are conducted at research laboratories in Hamburg, Berlin and Grenoble, neutron scattering experiments in Geesthacht, Jülich, Berlin, Grenoble or Garching. In addition to the use of X-ray scattering beamlines (at HASYLAB, Hamburg), a neutron reflectometer (at GKSS, Geesthacht) and a neutron small-angle scattering beamline (Garching, projected) are operated by the institute and scanning X-ray microscopy as well as photoelectron emission microscopy techniques are developed (at ESRF, Grenoble and BESSY II, Berlin).

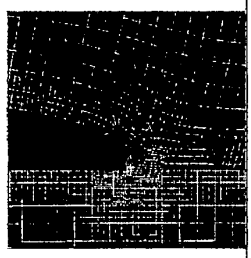
Mechanical characterization
- Universal mechanical test equipment (force range from 10 N to 100 kN, temperatures from -100 to 250 °C, optional inclusion of strain gauges and external sensors, video recording during experiments)
- Set-up for miniaturized tensile and bending experiments, e.g. on single fibres (1.5 N to 1000 N load, optional operation under an optical, scanning electron or scanning force microscope)
- Dynamic testing using a servohydraulic system with up to 50 kN load in a temperature range from -130 to 315 °C
- Acoustic emission analysis (6 channels)
- Micro indentation
- Equipment for impact bending and impact tensile test, density (helium micro pyknometer or buoyancy), ball indentation and shore-A hardness

Dynamic-mechanical spectroscopy
with various operational modes (torsion, compression/elongation, bending, shearing) in a temperature range from -150 to 600°C and in combination with a X-ray scattering set-up

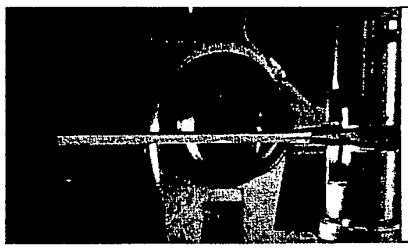
Thermo-mechanical analysis
employing different probes and optional static or dynamic loading of the sample

Dielectric spectroscopy
over a wide frequency range (10^{-4} to 10^{7} Hz) with various capacitor plate arrangements; resistance measurements (10^{6} to 10^{14} Ω).

Modelling
of mechanical and structural aspects with linear and non-linear behaviour of material properties. Modelling in two or three dimensions by analytical methods and via finite element modelling using the ANSYS software package. Modelling of sample structures on a molecular level with CERIUS



Finite-element modelling of a crack tip during interfacial fracture of a two-component material



Double-cantilever beam test with simultaneous observation by a stereo microscope

Dr. Konrad Schneider
Phone: +49-351-46 58-296
schneider@ipfdd.de

Dr. Bernd Lauke
Phone: +49-351-46 58-293
laukeb@ipfdd.de

7.3 Rheology

7.3 Systems under investigation

Polymer melts and solutions

Polyolefines

Polymer blends and their components

Filled polymer systems

Thermosets and prepreg materials

Basis materials for powder coatings

Special polymers for melt spinning processes

Research activities

Application of structural rheology to get answers to different questions in the development of polymeric materials and processing technologies

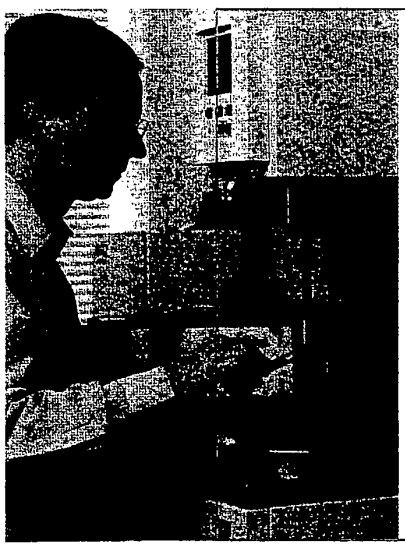
- Fundamental investigations on reactive compounding
- Characterization of polymer blends
- Investigation of the influence of synthesis conditions, fillers, modifications and other factors on the rheological material functions
- Detection of relaxation processes and thermal transitions in polymeric solids
- Determination of the crosslinking behaviour of thermosets (basis materials for powder coatings, prepregs)
- Studies to evaluate the processing behaviour of polymer melts in extrusion, injection moulding, and melt spinning
- Development of evaluation procedures to get information on structures from rheological investigations (e.g. invariant representations, calculation of relaxation spectra, models)

Characterization of flow and deformation behaviour of melts, concentrated solutions, and solids in case of very small deformations in order to obtain material parameters that permit conclusions on, e.g., molecular weight and molecular weight distribution, chemical and physical crosslinking, and blend composition

Investigation of the flow behaviour at large deformations and high rates of strain in shearing to draw conclusions on

- Flow behaviour in case of given processing conditions
- Estimation of accessible particle sizes in melt mixing of blends
- Die swell in melt spinning
- Influence of processing additives and water content on the flow behaviour
- Determination of critical flow conditions for the onset of flow instabilities
- Modelling of the deformation behaviour from the melt to the solid fibre under extreme extension and cooling

Sample positioning in the rotary rheometer ARES



Dr. Roland Vogel
Phone: +49-351-46 58-302
vogel@ipfdd.de

Research equipment development

Experienced engineers and technicians in the fields of
- Technical design
- Computer-based measurement, control and automation
- Mechanical manufacture

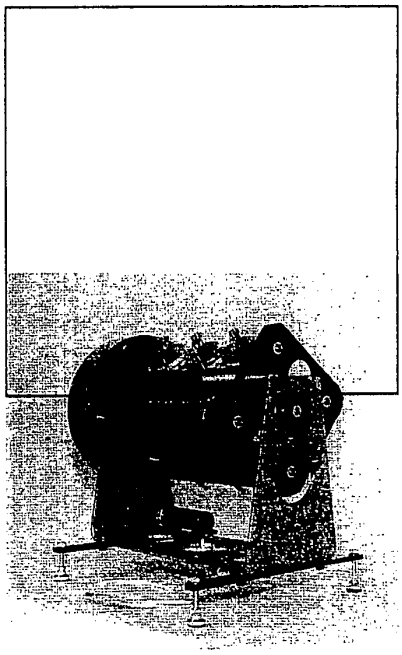
co-operate to provide the technological basis of the institute's research work and to develop special instrumentation.
Modern equipments like CAD-work stations and CNC-machines allow efficient design and manufacture.

Activities in the field of equipment making are:
- Development and manufacture of non-commercial measuring and testing devices
- Project, technical design, and manufacture of complete technological equipments on laboratory and pilot-plant scale
- Technical design and manufacture of accessory units for measuring and testing devices and machines in order to adapt commercial equipments to additional parameters and changed conditions
- Hardware conception and software development for measurement, control and automation in laboratory

The equipments are designed, manufactured and tested in close co-operation with the scientists, which allows very fast conversion of scientific ideas into novel techniques and devices.

Library

The specialized scientific library of the institute collects literature on chemistry, processing, modification, and characterization of polymers. Its inventory includes more than 14,000 publications and specialized collections of research reports and standard specifications. The library is subscribed to 150 specialist periodicals and series.
Expert knowledge can be obtained using the Current Contents and by on-line inquiries at STN International Karlsruhe, FIZ Technik Frankfurt on the Main. Special databases are available on CD-ROMs.
With its library, the institute is a member of the German Association of Libraries and of the Working Pool of Specialized Libraries.



Example of development of equipments: Rotation system for incubation cells for testing of biological materials

Rudolf Hanke
Phone: +49-351-46 58-221
hanke@ipfdd.de

Silke Thüm
Phone: +49-351-46 58-324
thuem@ipfdd.de

How to get to the institute

by train

Numerous direct connections with major German and European cities.
The institute is quite near the main railway station Dresden-Hauptbahnhof.
(Five minutes walk from the Southern exit 'Bayrische Straße' of Dresden-Hauptbahnhof: Leave the railway station to the right and follow Bayrische Straße and Hohe Straße)

by car

If you come from Frankfurt/Main, Hannover or Munich you should get on Autobahn A 4: Leave the highway at exit Dresden-Altstadt and follow the road signs to 'Zentrum'. When you pass beneath the railraod bridge turn right and follow Könneritzstraße and Ammonstraße. Turn right to the elevated roadway Budapester Straße following the sign to 'Freiberg'. Shortly after the brigde turn left to Schweizer Straße and again left to Hohe Straße (cf. detailed map).

If you come from Berlin or Poland you reach Dresden on Autobahn A4 via Bautzen or A 13 from Berlin
Leave the highway at exit Dresden-Hellerau and follow first the road signs to 'Zentrum' and 'Hauptbahnhof'. At the railway station Bahnhof Dresden-Neustadt turn right and follow Antonstraße, Marienbrücke, Könneritzstraße and Ammonstraße. Turn right to the elevated roadway Budapester Straße following the sign to 'Freiberg'. Shortly after the brigde turn left to Schweizer Straße and again left to Hohe Straße (cf. detailed map).

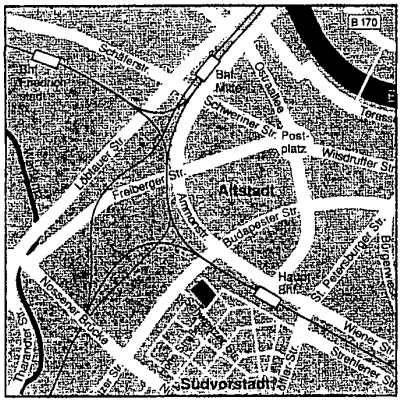
by airplane

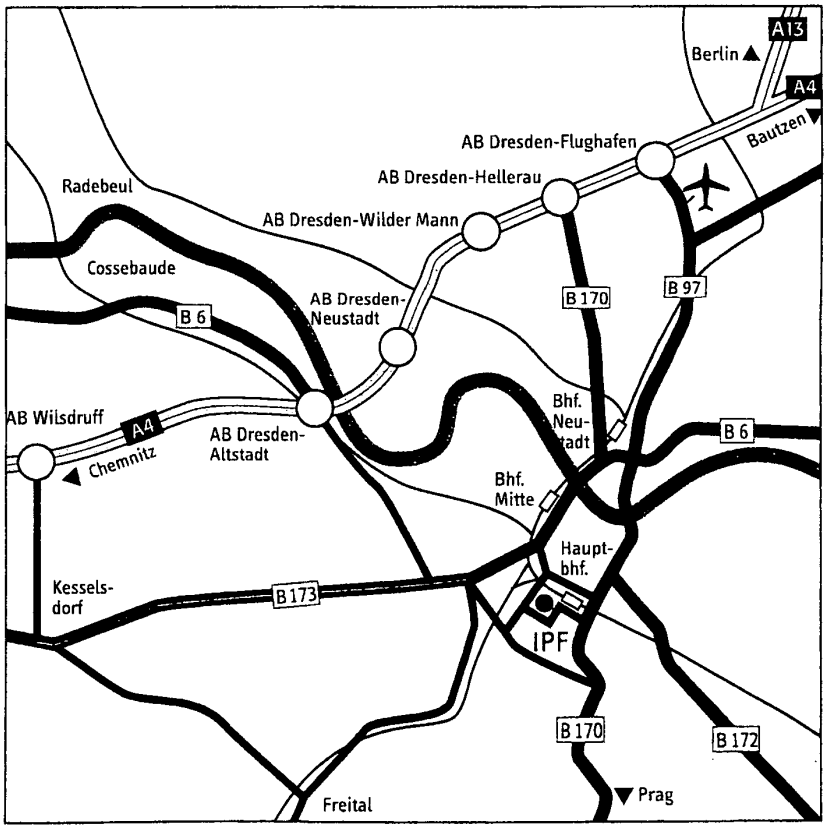
World-wide air connections to Dresden Airport, numerous direct flights between Dresden and major German and European airports.
From the airport to the institute:

Airport Cityliner to Dresden-Hauptbahnhof (duration about 30 minutes, departures every 20 to 30 minutes), from the bus stop five minutes walk along Bayrische Straße and Hohe Straße

or

Take a taxi/hire-car
(Follow the signs indicating direction 'Zentrum' and 'Hauptbahnhof' via Karl-Marx-Straße, Königsbrücker Straße, Albertstraße, St. Petersburger Straße. After having passed beneath the railroad bridge of Dresden Hauptbahnhof turn right to Bayrische Straße until Hohe Straße.)



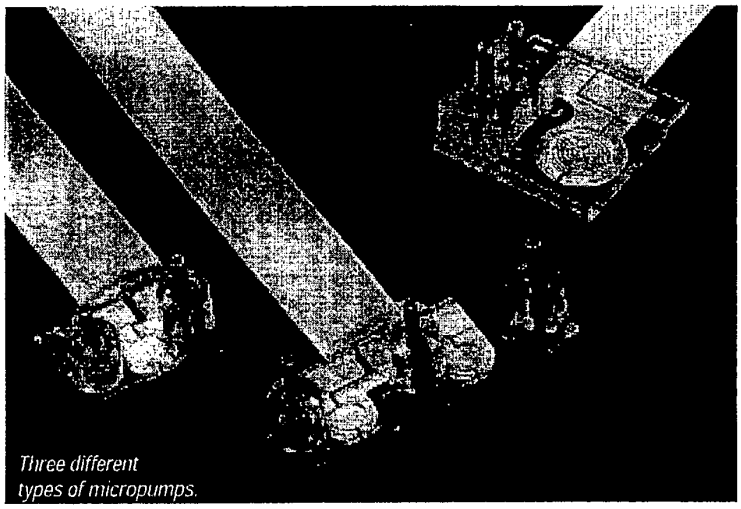


二、德國 IMT 研究中心微系統相關技術資料

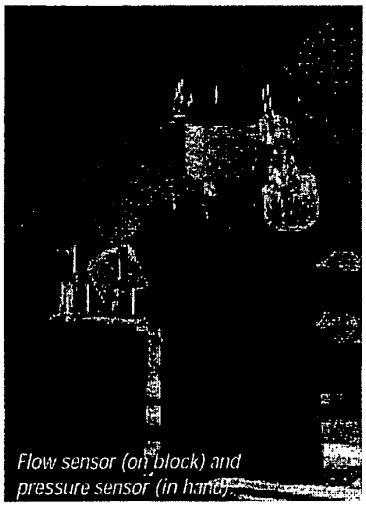
Forschungszentrum Karlsruhe
Technik und Umwelt
Microsystem Technologies Program (PMT)

AMANDA Process

Surface Micromachining, Molding and Diaphragm Transfer



Three different types of micropumps.



Flow sensor (on block) and pressure sensor (in hand).

AMANDA – a proven and reliable manufacturing technique on its way to industrial-scale implementation.

New AMANDA applications in microfluidics are being developed together with various industries.

Generate your own innovative product ideas, and move into the 21st century with AMANDA.

Applications

- **Drug dosage**
- **Biological and chemical analyses of small sample volumes**
- **Combinatorial chemistry**
- **Micropneumatics**
- **Air conditioning technology**

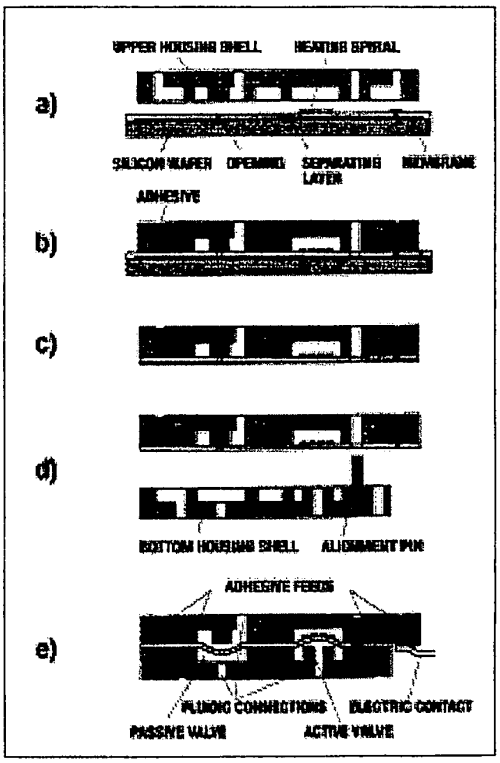
The AMANDA Process

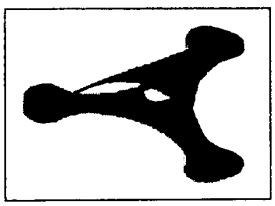
A variety of microsensors and microactuators can be manufactured by joining two shells of a housing with a diaphragm positioned in between. This diaphragm acts as a movable component, and it may be made out of a polymer or a metal. Depending upon the application envisaged, the diaphragm may be perforated or carry metal structures (such as conductor tracks, heating spirals, or contacts).

One particularly simple process of manufacturing such components in parallel at low cost is the AMANDA process in which surface micromachining, molding and diaphragm transfer are combined in one process. Processes are being developed within the Microsystems Technology Program of the Forschungszentrum Karlsruhe by which diaphragms only a few micrometers thick can be made out of a polymer or a metal and equipped with a housing. In the diagram below, the AMANDA process is explained by the example of an active and a passive microvalve:

- The upper shell of the housing is produced by molding (hot embossing or injection molding). The mold inserts required for molding are produced either by precision mechanics processes (such as milling) and/or by the LIGA technique, depending on product requirements (minimum lateral structure dimensions, number of planes inside the structures, etc.) (a).
- Methods of thin-film technology are employed consecutively, e.g., to deposite and pattern a separating layer, the diaphragm material, and a metal layer (a) on a silicon wafer as the auxiliary substrate.
- In this case, the injection of an adhesive into cavities made for this purpose joins the upper shell of the housing to the diaphragm (b).
- Low adhesion of the separating layer to the silicon wafer allows the composite consisting of the upper shell of the housing and the membrane to be mechanically separated from the substrate (c).
- The lower shell of the housing, like the upper shell, is produced by molding techniques and bonded in place. Positioned assembly can be simplified by alignment pins integrated into the housing (d). Next, the electric and fluidic contacts are attached (e).

In the AMANDA process, many components are manufactured side by side on one batch and diced later. This greatly reduces production costs.





FEM simulation of a passive microvalve.

Driving and evaluation electronics of a flow sensor.

The Microsystem Technologies Program has know-how not only in manufacturing the components listed above, but also in developing appropriate electronic driving and evaluation systems, in optimizing components by means of simulation calculations, and test installations for specific products. In this way, existing components can be made to match specific customer applications, and new areas of application can be developed.

Small series production by the AMANDA Process, e.g., of a Micropump

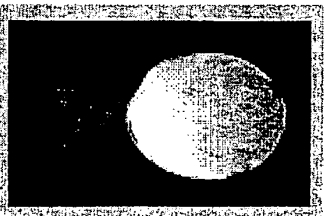
To demonstrate the capability of the AMANDA process for mass fabrication, more than 100 micromembrane pumps (cf. title photo) were manufactured in a small series at the Karlsruhe Research Center within one month.

The yield amounted to more than 70%. Twelve micropumps are manufactured in parallel per working step.

In continuous tests, these micropumps achieve more than 7600 hours of operation.

Left: Injection-molded polysulphone housing (bottom and top shells) for twelve micropumps each.

Right: Silicon wafer with structured polyimide membrane and heating spirals for 24 pumps.

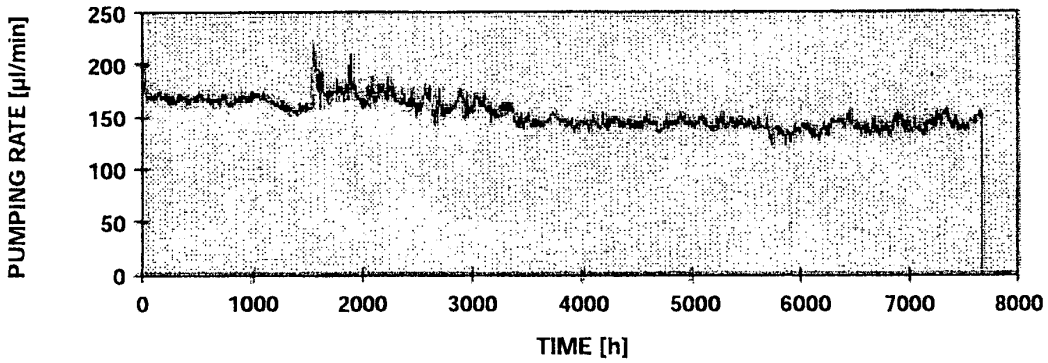


One batch with 12 micropumps after adhesive bonding.

Complete micropump and components for manufacturing the fluidic and electric connections.

Completed micropumps.

Long-term Test of an AMANDA Micropump for more than 7600 Hours of Operation

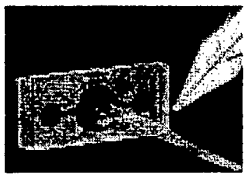


Typical Characteristics of AMANDA Products

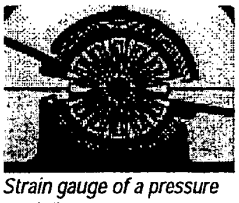
◗ Low manufacturing expense	Parallel manufacture of many components, e.g., micropumps at only DM 150 when manufactured in small numbers at the IMT laboratory	
◗ High reliability	Operating life in excess of 7600 hours in the case of micropumps	
◗ High manufacturing yield	70% at the laboratory of the Institute for Microstructure Technology (IMT)	
◗ Wide range of materials, e.g.	Housings,	PMMA, PSU, PVDF, PC, PEEK, PA, POM, etc.
	Membranes,	PI, PTFE, FEP, Ti, etc.
	Conductor tracks	Au, Cr, Ti, Cu, etc.
◗ Height of structures	Housings	5 µm to more than 1 mm
	Membranes	1 µm to approx. 50 µm
	Conductor tracks	50 nm to 5 µm
◗ Critical dimensions	Housings	50 µm in various planes
	Membranes	approx. 5 µm
	Conductor tracks	5 µm and less
◗ Alignment accuracy	Housings - membranes	50 µm
	Conductor tracks- membranes	5 µm

We Offer Know-how in These Areas:

- Microstructuring techniques (LIGA process; machining techniques, thin-film techniques, etc.).
- Membrane production and processing.
- Fitting and joining techniques (bonding techniques, positioning, etc.).
- Microelectronics for evaluation and driving circuits, respectively (thick-film hybrid, SMD, or ASIC technologies).
- Small-series manufacture of microcomponents (test equipment and quality assurance).



Bistable microvalve.



Strain gauge of a pressure sensor.

Fluidics laboratory.

Equipment Available

- Hot-embossing and injection molding plants for manufacturing pilot products or for small series production.
- Various fitting and joining techniques (e.g. bonding processes).
- Measurement and test installations for microfluidic components.
- Microelectronic components for signal processing and for driving AMANDA products.

Possibilities for Cooperation

Transfers of technology from research to industry can be managed in various ways, all of which can be individually adapted to the needs and boundary conditions of the partners. Here are some examples.

- Bilateral cooperative ventures: Process development, translation of processes to an industrial scale, and development of components and systems.
- Joint research: Common research and development projects pursued together with industrial partners.
- Contract research and development.
- Manufacturing small series of microstructure components and microsystems prototypes.
- Training of staff from industry in the course of small-series manufacture of prototypes.
- Services, such as mask production, exposure, resist substrates, mold inserts.
- Consulting in AMANDA, LIGA, and microstructure techniques.
- Acquisition of rights of use.

For contacts, please write to
Dr. W.K. Schomburg, Institut für Mikrostrukturtechnik, phone ++49-(0)7247-82-2745; facsimile -4331,
Dr. R. Mache, Stabsabteilung Technologie und Marketing, phone ++49(0)7247-82-4876; facsimile -4331,
Dr. J. Fahrenberg, Projektleitung Mikrosystemtechnik, phone ++49(0)7247-82-5589; facsimile -5579.

Forschungszentrum Karlsruhe GmbH, Postfach 3640, D-76021 Karlsruhe, Germany.

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Assembly of hybrid integrated micro-optical modules using passive alignment with LIGA mounting elements and adhesive bonding techniques

A. Gerlach, P. Ziegler, J. Mohr

Abstract Assembling hybrid integrated micro-optical modules for mono-mode applications requires alignment tolerances better than ±1 μm which, so far, were achieved by complex active alignment of the micro-optical components. This article describes a passive assembly concept employing high-precision LIGA structures to simplify assembly and reduce manufacturing costs. The advantages of this passive assembly concept are demonstrated in the micro-optical assembly of a heterodyne receiver. The micro-optical components (ball microlenses, prisms, glass fibers, photodiodes) were aligned passively by means of alignment structures made of PMMA, and were subsequently fixed by UV-curing adhesive. Photodiodes were additionally contacted with electrically conducting adhesive cured at 70 °C. The mono-mode glass fibers were mounted in fiber mounts into the fiber grooves of which the glass fibers were inserted and immobilized with UV-curing adhesive. Measurements of the optical quality of the heterodyne receiver indicated an accuracy of assembly better than 1 μm.

1 Introduction

Applications in telecommunications require more and more modules for transmitting and processing increasingly larger data flows. Transferring information by means of optical fibers offers a multitude of possibilities to meet present and future requirements in terms of transmission capacity. Optical networks make use, i.e., of hybrid micro-optical modules (such as optical switches, optical amplifiers, laser sources, receivers, optical insulators, couplers, wavelength filters, and optical heterodyne receivers).

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A. Gerlach (✉)[1], P. Ziegler, J. Mohr
Forschungszentrum Karlsruhe
Institut für Mikrostrukturtechnik
Postfach 3640, D-76021 Karlsruhe, Germany

Present address:
[1] Greiner Labortechnik GmbH
Maybachstrasse 2, D-72636 Frickenhausen, Germany

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The application and propagation of this technology will depend on manufacturing techniques being found which allow these modules to be manufactured at low cost [1]. The key to low-cost manufacture is passive alignment of the micro-optical components in such a way that they are aligned relative to each other by means of mechanical and optical alignment systems. In the much more sophisticated technique of active alignment, an optical assembly is aligned with the light source turned on, and the optimum position of the components is then immobilized. Low-cost manufacture of hybrid integrated micro-optical modules requires manufacturing techniques to be developed which allow passive alignment of the components to a precision better than ±1 μm [2].

This article describes the development of a micro-optical LIGA bench (see Fig. 1) for hybrid integration and passive assembly of the optical components along the optical axis by means of high-precision alignment structures of PMMA produced by the LIGA technique [3] and employing adhesive bonding [4]. The assembly concept was demonstrated in the design and construction of a heterodyne receiver [5, 6].

In conventional technologies, active micro-optical components (photodiodes, laser diodes) are integrated by the entire assembly being soldered or the components being soldered to the substrate individually by local heating. In substrates of high thermal conductivity (such as silicon) this is associated with the drawback of the heat being dissipated over the entire substrate, thus affecting every single bonded connection on the substrate. This may cause soldered connections to melt and, consequently, influence previous alignment of the components. Moreover, flux is used in soldering which could contaminate the optical interfaces during assembly. Also, the use of soldering fluxes may cause shrinkage during soldering which, in turn, may influence positioning of a component [7, 8].

In the assembly concept described in this paper, the micro-optical components were positioned passively on LIGA alignment structures of PMMA, and immobilized at room temperature by means of UV-curing adhesive. Electrical contacting of photodiodes was achieved with an electrically conducting adhesive cured at 70 °C. Glass fibers were mounted with mounting elements positioned on the alignment structures. The low thermal stress and the low amount of adhesive shrinking during curing did not impair alignment of the micro-optical components already mounted. As the micro-optical components were positioned by means of the alignment structures, and the adhesive only served to fix the position, shrinkage of the

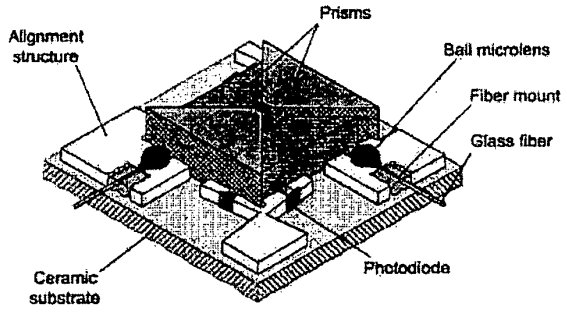

Fig. 1. Micro-optical LIGA-bench

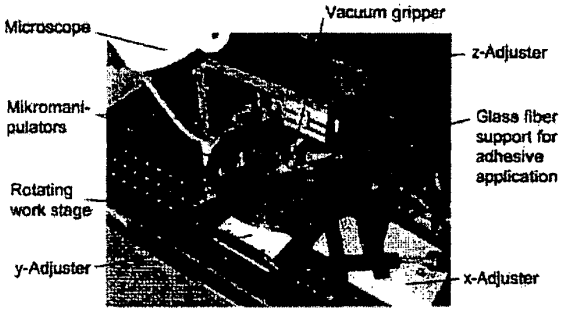

Fig. 3. Mounting device

adhesive caused no misalignment. The adhesives used were free from solvent which could have precipitated on the interfaces of optical components during curing, and thus could have impaired their optical function.

The principle of a micro-optical heterodyne receiver, in which an optical signal received is coherently superimposed the light of a local laser diode before it is detected by photodiodes, has been published before [5, 6].

The design of the heterodyne receiver is shown in Fig. 2. The signal and local oscillator light emerging from an optical single mode fiber is coupled into a free-space micro-optical system with ball microlenses to collimate the beams. Prisms as polarization beam splitters separate the collimated beams into the perpendicular and parallel polarization states. The polarized light is superimposed by means of prisms employed as 3 dB splitters. The light signals are detected in a balanced receiver with two photodiodes for each polarization state.

2
Mounting optical components on a micro-optical LIGA bench

2.1
Mounting device and tools

A computer controlled mounting device was set up for assembly and bonding of the micro-optical components (see Fig. 3). The work stage allowed horizontal displacement and turning in the x- and y-directions. A gripper was attached to the vertical z-adjuster which, in addition, allowed positioning by manual tilting. Due to the manufacturer (Physik Instrumente, Waldbronn, Germany) the reproducibility of the linear adjustment units was 0.5 µm, while the absolute accuracy was 3 µm; the reproducibility of the rotating adjustment unit was 0.3 arc s.

Manual micromanipulators were attached to the work stage to press the positioned micro-optical components against the substrate bottom and against the alignment structures of the micro-optical bench by means of spring loaded pins. This ensured exact positioning prior to adhesive bonding.

A support for a glass fiber (125 µm diameter) was attached to the z-adjuster of the mounting device; an appropriate reduction gear allowed it to be freely positioned manually. The glass fiber picked up from a reservoir one drop of UV-curing adhesive and deposited it at the bonding site of the micro-optical component in position. Subsequently, the adhesive was cured under UV light. Especially for assembly on the micro-optical bench of photodiodes with lateral dimensions <1 mm, a vacuum gripper with integrated leaf springs (see Fig. 4) was developed for moving the component under force control against an alignment structure and for lateral movement of the component on the gripper against a stop edge of the alignment structure (passive alignment). However, even larger micro-optical components, such as ball microlenses and prisms, were picked up by this gripper and mounted on the micro-optical bench.

The entire assembly device was contained underneath a clean-room flowbox and was grounded electrically to avoid dust generation and electric charges, especially when picking up the sensitive photodiodes.

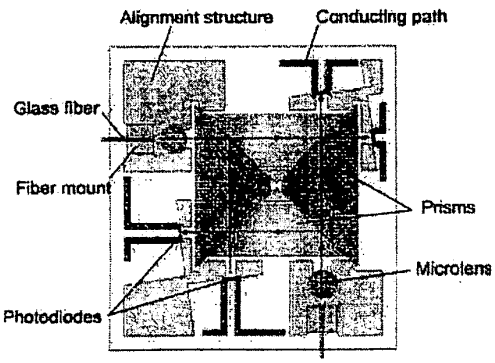

Fig. 2. Design of the heterodyne receiver (⊥ denotes perpendicular polarized light, || denotes parallel polarized light)

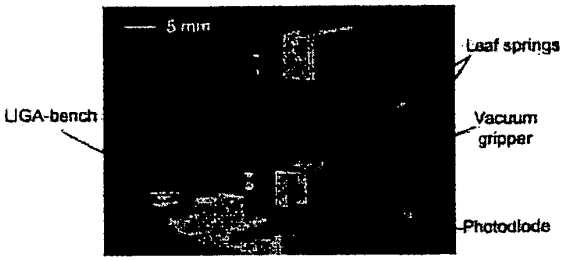

Fig. 4. Vacuum gripper with a photodiode close to a micro-optical LIGA-bench

2.2
Assembly of ball microlenses and glass fibers

Ball microlenses with a diameter of 900 µm were picked up with the vacuum gripper and pressed against the bottom of the substrate at the 500 µm high ball microlens alignment structures made of PMMA. Each lens was aligned by way of point contacts relative to the alignment structure and the substrate bottom. Subsequently, droplets of adhesive were applied laterally between the ball microlens and the PMMA alignment structure, and the adhesive was cured under UV light.

Applying adhesive, e.g. to the bottom of the substrate, prior to assembly of the lens was avoided. In this way, reproducible positioning of the lens by means of the point contacts was ensured. As the ball microlenses were mounted right on the substrate, the optical axis ran 450 µm above the substrate surface.

Consequently, high-precision LIGA mounts of PMMA were used to position the glass fibers onto the optical axis (see Fig. 5). The convex mounts contacted the substrate on only two lines and were positioned in the PMMA alignment structure by means of lateral point contacts. The convex shape of the mounts facilitated assembly into the alignment structure and made alignment of the fiber mounts less sensitive to dust particles on the substrate bottom and on the PMMA alignment structures than would have been the case if the glass fiber mounts had been rectangular in shape. At the same time, the biconvex shape of the mounts generated an adhesive channel fixing the mounts with UV-curing adhesive.

Manufacturing the mounts by the LIGA technique allows both the contour of the mounts and that of the fiber groove to be freely chosen and, consequently, to be optimally adapted to the respective micro-optical design.

The glass fiber was inserted into the fiber groove of the fiber mount installed, pressed into the fiber groove by means of a spring-loaded pin of a micromanipulator, and then fixed with UV-curing adhesive. The position of the glass fiber was determined laterally by the fiber groove and axially by a fiber stop in the PMMA alignment structure (see Figs. 6 and 7).

2.3
Assembly of photodiodes

To avoid unwanted reflections, the photodiodes were installed at an angle of 8° relative to the optical axis. Prior to assembly of the photodiodes, the substrate with the micro-optical bench and the vacuum gripper were aligned parallel to each other and at an angle of 8° relative to the optical axis. The photodiodes (see Fig. 8) were picked up by the vacuum gripper (see Fig. 4) and first moved in the direction of the longitudinal axis of the gripper as far as to the stop of the alignment structure on the micro-optical bench. Then the assembly tool was displaced normal to its longitudinal axis until the photodiode had been aligned to the lateral stop of the PMMA alignment structure. Next, the gripper was lowered until the photodiode contacted the bottom of the substrate. A manual micromanipulator was used to immobilize the photodiode in its position temporarily by means of a spring-loaded pin; a drop of

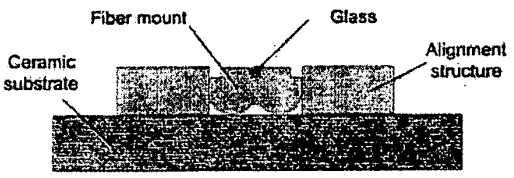

Fig. 5. Alignment of single mode fibers with so called fiber mounts

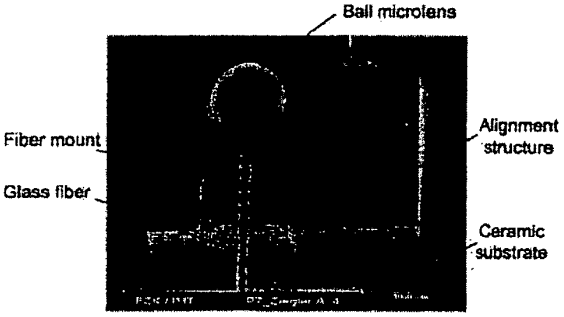

Fig. 6. Assembled ball microlenses (∅ 900 µm) and single mode fibers. The optical axis runs 450 µm above the substrate surface

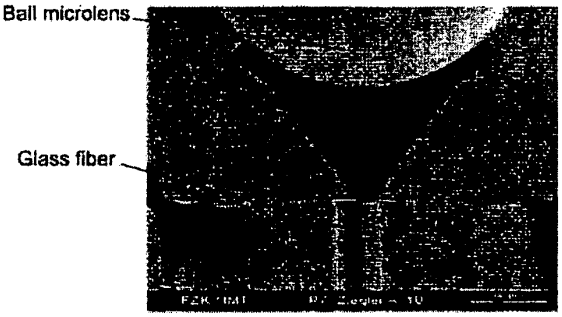

Fig. 7. Passive alignment of single mode fibers along the optical axis with fiber stops formed on the micro-optical LIGA-bench

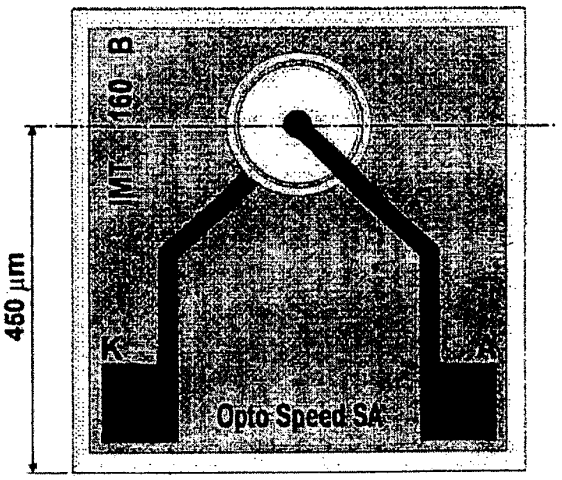

Fig. 8. Design of the photodiodes

UV-curing adhesive was applied to each adhesive channel of the PMMA alignment structure (see Figs. 9 and 10), and the adhesive was cured under UV light.

Electric contacting of the photodiodes relative to the gold conducting paths on the ceramic substrate was achieved by low-viscosity conductive adhesive filled with Ag particles which allowed adhesive droplets of less than 100 μm in diameter to be applied. The conductive adhesive cured at a temperature of 70 °C (see Figs. 9 and 10). As immobilization of the micro-optical components was achieved at temperatures below the softening temperature of PMMA, the thermal load to which the entire assembly was subjected during the bonding stage was negligible.

2.4 Assembly of prisms

Four prisms were mounted on the micro-optical bench as beam splitters for the superposition of two light signals. First, the two larger prisms, which were optically coated on their short faces, were put on the substrate from above with the vacuum gripper and pressed against the substrate bottom and the stops of the PMMA alignment structures by the spring-loaded pins of a manual micromanipulator. The entire assembly was fixed with UV-curing adhesive.

The two smaller prisms were put in place in a second step in which the two prisms mounted first served as mechanical stops (see Fig. 12). Prior to immobilization

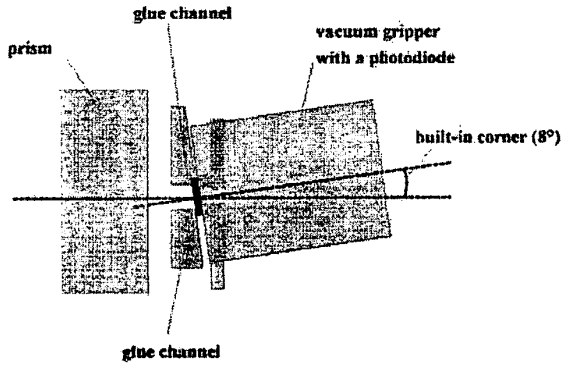

Fig. 9. Assembling of the photodiodes with a vacuum gripper

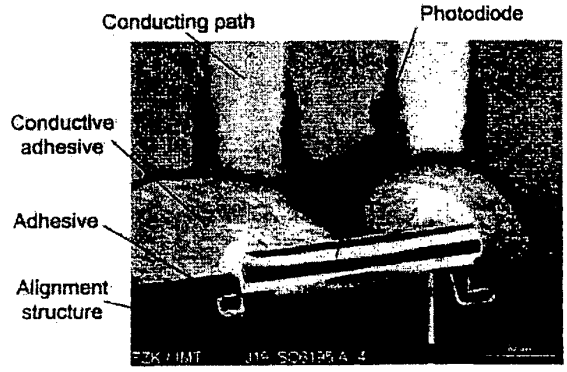

Fig. 10. Passive alignment and fixing of the photodiodes

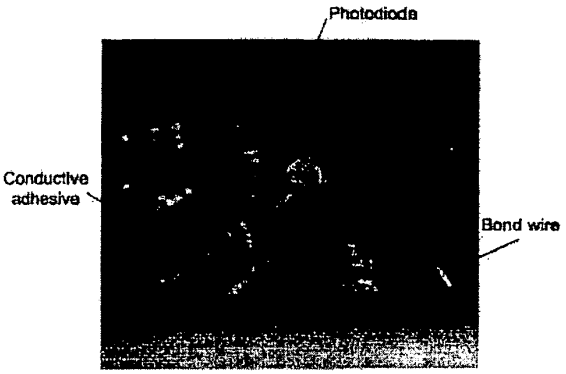

Fig. 11. Electrical interconnection of a photodiode with conductive adhesive and wire bonding

with adhesive, also the small prisms were pressed against the substrate bottom and against the larger prisms by spring-loaded pins. The short faces of the smaller prisms were wetted with a special UV-curing adhesive prior to assembly which had the same index of refraction as the BK7 glass of the prisms. This adhesive avoided a loss of intensity of the optical beam as a result of multiple reflection at the interfaces between glass and air and, at the same time, ensured immobilization of the smaller prisms.

2.5 Accuracy achieved in assembly

For measurement of the accuracy achieved in assembly, the mounted heterodyne receiver was characterized optically with two external laser diodes whose light was supplied through the two glass fibers. One laser diode acted as the signal laser, while the other served as the local oscillator. Figure 13 shows the image recorded by a camera of the intensity distribution generated at the position of a photodiode by superposition of the signal laser light with the local oscillator light. The intensity distribution of the superimposed signal at the position of the photodiode shows

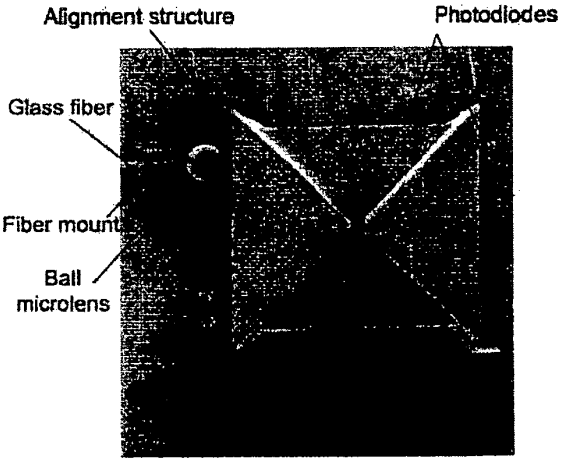

Fig. 12. Assembled microoptical LIGA-bench with ball microlenses, glass fibers, photodiodes and prisms

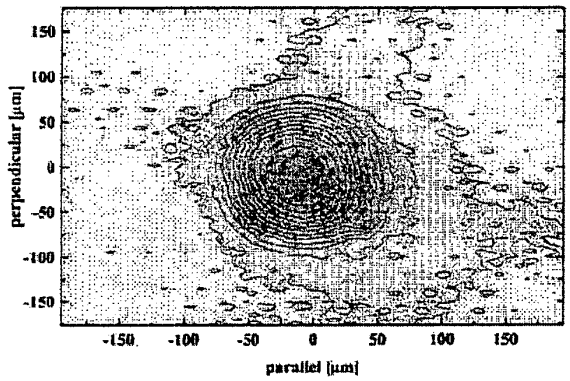

Fig. 13. Overlayed intensity distributions of the signal and local oscillator light at the position of a photodiode

that the micro-optical assembly ensures superposition of the signal laser light upon the local oscillator light.

As the intensity distribution was detected by the camera always with the local laser diode turned off and the signal laser diode turned off, quantitative analysis of the superposition is possible by measuring the distance between the centers of the intensity distributions. Figure 14 shows the profile of the three intensity distributions measured. The peaks of the two intensity distributions originating from the signal laser and the local laser are spaced 5.1 µm apart. According to calculations of the beam path [5], a relative lateral fiber misalignment by ±1 µm causes a beam misalignment by ±6.4 µm at the position of the photodiode. As the measured beam misalignment is within these bounds, the measurement confirms that passive assembly by means of LIGA structures and immobilization with adhesive allows alignment of the micro-optical components to be achieved to the required precision of better than ±1 µm.

3 Conclusions

The concept of passive assembly of hybrid integrated micro-optical modules by means of LIGA alignment structures and adhesive bonding techniques was demonstrated in the assembly of a micro-optical heterodyne receiver. Compared to active assembly techniques it allows major simplification and cost reduction in assembly. The micro-optical bench produced by the LIGA technique allows the micro-optical components (ball microlenses, glass fibers, prisms, photodiodes) to be positioned along, and normal to, the optical axis in the substrate plane. Vertical positioning of the glass fibers was achieved with mounts produced by the LIGA technique which, in turn, were positioned in the optical beam path by means of precise alignment structures on the micro-optical bench.

The micro-optical components were attached to the alignment structures with UV-curing adhesive. The photodiodes were additionally contacted with electrically conducting adhesive. The curing temperatures for the adhesives were below the softening point of PMMA. This ensured that alignment of the micro-optical components was not impaired.

The precision of passive assembly was demonstrated by measurement of the optical quality of the heterodyne receiver, resulting in an accuracy of assembly of less than 1 µm.

The assembly concept can be converted into an automated assembly procedure. For this purpose, the appropriate automatic assembly systems [2, 9] need to be developed, for instance, to mount the micro-optical components with force sensing.

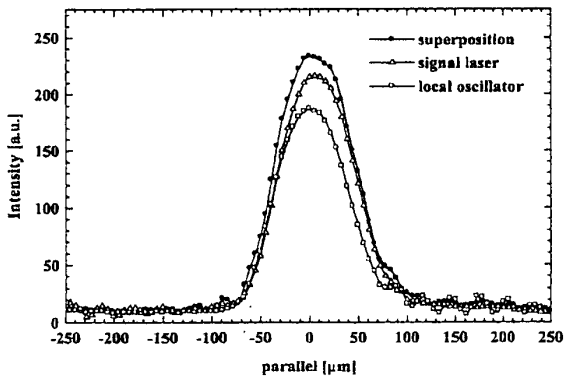

Fig. 14. Section through the center of the intensity distributions in direction parallel

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Gas permeability of adhesives and their application for hermetic packaging of microcomponents

A. Gerlach, W. Keller, J. Schulz, K. Schumacher

Abstract The use of adhesives with low gas permeability in packaging microcomponents provides a simple, low-cost alternative to the standard techniques for hermetic packaging. The permeability to helium of various epoxy resin adhesives was determined by means of a mass spectrometer in reference specimens of defined dimensions. The adhesive with the lowest permeability to helium was chosen to bond an evacuated LIGA gyrometer package (flatpack, volume = 1.4 cm^3). For comparison, a few packages were closed by laser welding. Subsequently, the pressure rise in the sealed packages due to gas permeation and desorption from the walls was measured by the integrated microsensor, and the permeation rate was determined. The permeation rates of as low as 10^{-17} m^3/s achieved in bonded packages are comparable to those measured in laser welded packages.

1 Introduction

Many applications in microelectronics, micro-optics, and microsensors require hermetically sealed packages either to protect microcomponents from chemical reactions with gases penetrating from the environment [1] or in order to maintain a low overall pressure in an evacuated package, e.g., to eliminate the attenuation by air of movable microcomponents [2].

Where standard techniques are used for hermetic sealing, such as glass sealing, soldering, welding [3], anodic or direct bonding [4, 5], a specific choice of material is required. These techniques, as a rule, are associated with high process temperatures and high process costs. Adhesives can be employed in packaging technology at low process temperatures and with a large variety of materials [6, 7]. Process costs can be reduced also because of the ease of handling.

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A. Gerlach* (✉), W. Keller, J. Schulz, K. Schumacher
Forschungszentrum Karlsruhe
Institut für Mikrostrukturtechnik
Postfach 3640, D-76021 Karlsruhe, Germany

Present address:
*Greiner Labortechnik GmbH, Maybachstrasse 2
D-72636 Frickenhausen, Germany

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As with standard technologies, the required hermeticity of the package is achieved, on the one hand, by the use of hermetic packaging materials, such as metal, ceramics, and glass. On the other hand, the hermetic tightness of the respective sealing interfaces of the materials so joined is a decisive factor.

Modern epoxy resin adhesives have very low gas permeabilities and outgassing rates and, consequently, can be used to join materials for hermetic packages if the package has been designed properly.

In this study, the permeability to helium of various epoxy resin adhesives was measured by means of a mass spectrometer in reference specimens of defined dimensions, and adhesives were used to seal an evacuated LIGA gyrometer package (flatpack, volume = 1.4 cm^3). For comparison, a number of packages were also sealed by laser welding. The overall pressure rise in the sealed packages due to gas permeation and desorption from the walls was measured by the integrated microsensor, and the permeation rate was determined from these readings.

2 Permeation of gases through polymers

The permeation of gases through polymers can be considered a three-stage process:

- Absorption and subsequent dissolution, of the gas at the gas/polymer interface.
- Diffusion of the gas towards the polymer interface with the lower gas concentration.
- Desorption of the gas from the polymer surface into the vacuum and ambient gas, respectively.

As a rule, polymers have permeability coefficients for gases which are between 10^{-17} and 10^{-11} $m^2/(s\ Pa)$, while those for glasses or ceramics are between 10^{-19} and 10^{-17} $m^2/(s\ Pa)$, and those for metals or semiconductors are between 10^{-20} and 10^{-22} $m^2/(s\ Pa)$ [8]. In some polymers, such as silicones (see Table 1), high permeability to water vapor is caused by the capability of the polar gas to interact strongly with polar polymer groups. In this way, a large fraction of the gas is dissolved in the polymer and can be desorbed again on the opposite surface. Among the polymers used in adhesives, especially epoxy resins are characterized by low gas permeabilities. Other authors than us measured mainly the permeation of water vapor through epoxy resins (see Table 1).

Gas permeability is determined in adhesive polymers not only by the fraction of reactive polymer groups but, especially, by the degree of crosslinking, the filler content,

Table 1. Measured permeability coefficients, P, in 10^{-17} $m^2/(s\ Pa)$ for various polymers and gases at 25–30 °C

Polymers	He	N_2	O_2	CO_2	H_2O	Ref.
Silicones	175	170	76–460	460–2300	8000–32 000	[11, 13]
PU	–	0.4–1.1	1.1–3.6	10–30	260–9500	[11]
PC	7.5	–	1.1	6.0	720–1050	[8, 13]
PMMA	5.2	–	0.12	–	480–1900	[12, 13]
PI	1.9	0.03	0.1	0.2	431	[11, 13]
PI with 1 μm Al or Cu	0.004–0.01	–	–	–	–	[14]
Fluoroelastomers	9–16	0.05–0.3	1.0–1.1	5.8–6.0	40	[11]
Teflon	–	0.14–1.2	0.04–3.7	0.12–9.5	13–27	[10, 11, 13]
KEL-F	5.1	0.002–0.004	0.02–0.03	0.04–0.16	0.22	[11, 13]
Epoxides	0.21–2.3*	–	–	–	40–300	[8, 12]

* Measurements conducted within this study

and the degree of polymer wetting of the filler. A high degree of crosslinking is able to reduce the gas permeability of an adhesive polymer quite considerably. A high content of filler material in particles of glass, ceramics or metal can further reduce gas permeability. The permeation rate, Q, through a material in equilibrium is a function of the area, A, the thickness, d, and the differential pressure, Δp, of the permeating gas at opposite interfaces; P denotes the permeability coefficient:

$$Q = P\frac{A\Delta p}{d} \tag{1}$$

In dry gases, such as helium or air, the development of the permeation rate, $Q(t)$, as a function of time can be described approximately by Fick's 2nd law of one-dimensional diffusion. The approximate solution for long diffusion times results in

$$Q(t) = Q(t=\infty)\left(1 - \exp\left(-\frac{\pi^2}{4}\frac{Dt}{d^2}\right)\right) \tag{2}$$

where D indicates the diffusion constant of the gas in the respective material, and $Q(t=\infty)$ is the equilibrium value of the permeation rate after an infinitely long diffusion time. The diffusion constant, D, is proportional to the permeability coefficient, P. According to Eq. (2), the diffusion time, t, quadruples, at the same permeation rate, Q, when the thickness, d, of the material is doubled.

This fact may become significant especially for hermetic packages of limited lifetime. In order to obtain the lowest possible permeation rates, e.g., into an adhesively bonded package, the design should provide not only for the proper choice of adhesive for the joining area, but also for a minimum permeation area, A, and the largest possible thickness (permeation length), d, of the layer of adhesive.

3 Experimental

3.1 Preparation of adhesive samples and measurements of helium permeation

Reference samples of 13 different commercial epoxy resin adhesives were prepared for measurements of helium permeation. One epoxy resin, Vitralit 1507 (manufactured by Panacol-Elosol, Oberursel, Germany), was a one-component adhesive filled with SiO_2 nanoparticles and curing in ultraviolet light. All other epoxy resins (Polytec, Waldbronn, Germany) were two-component adhesives, partly filled with Al_2O_3 and BN particles cured at temperatures between 65 °C and 180 °C. The curing temperatures and curing times were chosen so as to achieve a maximum degree of crosslinking of the adhesive polymer in accordance with information provided by the manufacturers.

The adhesives studied were poured into aluminum dishes (29 mm inner diameter, 2 mm inner depth, 0.2 mm wall thickness), cured, and then the aluminum bottom was milled off from the back side. Then the front side was leveled parallel to the back side and milled down to an adhesive sample thickness of 1–2 mm.

For measurement of the permeation of helium, the adhesive sample was held between two Viton ring seals screwed between a reservoir permeated by a gas flow and a vacuum flange (see Fig. 1). The reservoir offered the possibility of flooding either with helium or with nitrogen at a partial pressure of 10^5 Pa. The vacuum flange was connected to a mass spectrometer which, after evacuation of the chamber and calibration of the mass spectrometer by means of a calibration leak, allowed the rates of helium permeation through the adhesive sample to be measured directly. The measurement setup with two Viton seals

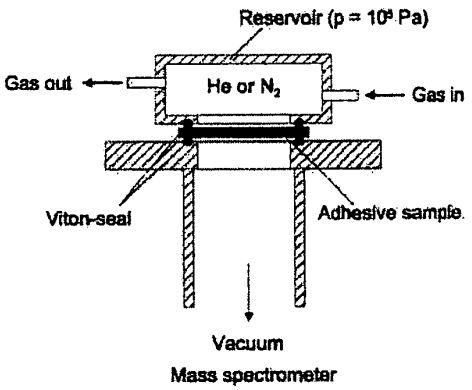

Fig. 1. Schematic representation of the test setup for He permeation measurements of adhesive samples

attached to the adhesive sample ensured that helium permeated from the reservoir into the vacuum only through the adhesive sample, not through the ring seals.

Reference measurements with adhesive samples poured into Teflon dishes and then removed indicated that mechanical working does not produce any microcracks in the samples which would add to the gas permeability.

3.2
Packaging technology for the LIGA gyrometer

Optimum operation of a LIGA gyrometer [9] requires an evacuated package in order to eliminate attenuation by air of the oscillating mass.

The packages used were gold plated standard flatpacks (volume = 1.4 cm^3) in which the sensors to be packaged were first glued to the bottom by means of an epoxy resin adhesive. After cleaning in isopropanol, the wire bond connections between the bond pads of the respective sensor and the electric leads of the package were produced (see Fig. 2). A borehole was drilled into the gold plated lid, into which an oxygen-free copper tube was soldered. The other end of that tube was soldered to a vacuum flange (see Fig. 3).

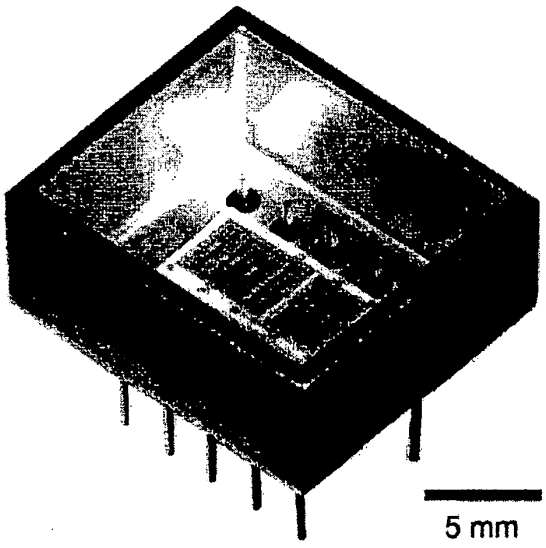

Fig. 2. LIGA gyrometer adhesively bonded and wire bonded into a flatpack

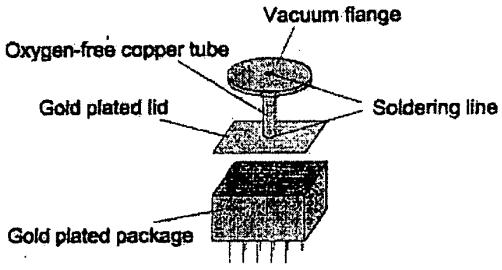

Fig. 3. Design of the gyrometer package. The lid is adhesively bonded or laser-welded to the package

The lid was bonded to the package by means of the H72 epoxy resin adhesive which had been measured to show the lowest permeability to helium (see Fig. 4). Alternatively, the package was welded with a Nd:YAG laser (see Fig. 5). After the package had been evacuated for two days at a pressure of 2×10^{-3} Pa and a temperature of 100 °C, the copper tube was cut with special tongs so that it fused at the cut. In a few samples, the cut at the copper tube was additionally soldered, in order to be on the safe side (see Fig. 5), or the copper tube was additionally glued to the lid (see Fig. 4).

Gold coating of the package and of the lid was able to reduce the adsorption of gases onto the inner surface of the package.

3.3
Pressure measurement in the gyrometer package

As the quality of the oscillator of the LIGA gyrometer was strongly dependent on ambient pressure, this measured signal was used for pressure measurement in the

Fig. 4. Adhesively bonded, evacuated gyrometer package

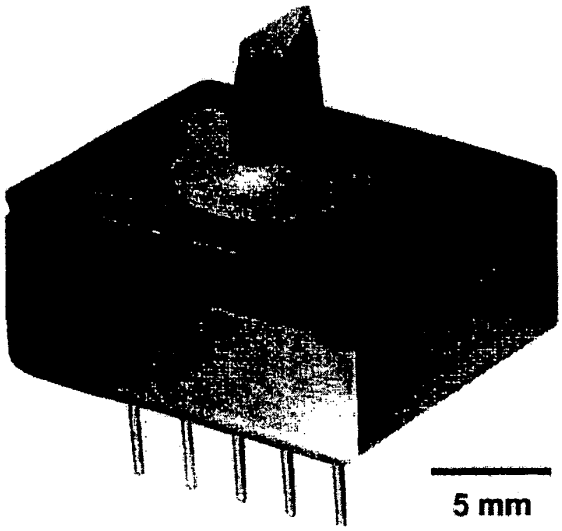

Fig. 5. Laser-welded, evacuated gyrometer package

sealed flatpack. For this purpose, the sealed gyrometer pack was connected to the vacuum pump with a pressure sensor, and before the copper tube was cut, the resonant rise was measured at various ambient air pressures, and the Q-factor was determined from that measurement. The decrease of the Q-factor with rising air pressure resulted in a measurable pressure range of approx. 3–3000 Pa (see Fig. 6). The reference curve then allowed the pressure rise to be determined from the measured oscillator signal in the package separated from the vacuum pump.

4
Results and discussion

4.1
Permeation of helium through adhesive samples

Before the permeation of helium was measured in adhesive samples, an aluminum plate of the same dimensions as the adhesive samples was put between the ring seals of the test setup, and the permeation of helium was measured (see Table 2). The very low permeation rate measured was near the detection limit of the mass spectrometer, thus confirming that there was no measurable permeation of helium through the ring seals.

The results of the helium permeation measurements of adhesive samples are also shown in Table 2. For the diffusion times, the time t_{98} until 98% of the equilibrium value of the permeation rate, $Q(t = \infty)$, had been reached, was indicated. The permeability coefficient, P, was determined in accordance with Eq. (1) from the measured permeation rate, Q, with the helium partial pressure, the thickness, d, and the area to be permeated, $A = 6.6\ cm^2$, of the adhesive sample taken into account. As a rule, filled adhesives turned out to have lower helium permeabilities and longer diffusion times than unfilled adhesives. A high filler content, however, is not the sole factor determining a low helium permeation rate. This can be seen, e.g., from the T7109 adhesive in which, despite a high content of filler, the highest helium permeability constant, $P = 0.88 \times 10^{-17}\ m^2/(Pa\ s)$, of all filled adhesives was measured. Probably, polymer wetting of the filler was not as good in this adhesive as, e.g., in the H72 adhesive, which has a lower content of filler so that helium permeation occurred mainly along the interface between the polymer and the filler. The lowest helium permeability coefficient, $P = 0.21 \times 10^{-17}\ m^2/(Pa\ s)$, was measured with the filled H72 adhesive, which also exhibited the longest diffusion time for helium ($t_{98} = 54$ h at $d = 2.0$ mm). The unfilled Durapot 862 adhesive exhibited the highest measured helium permeability coefficient $P = 2.3 \times 10^{-17}\ m^2/(Pa\ s)$.

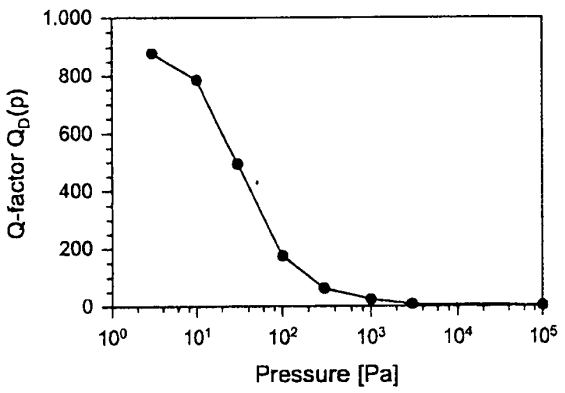

Fig. 6. Measured reference curve for pressure measurement in the gyrometer package. Q-factor of the oscillator, Q_D, as a function of the ambient air pressure, p

The approximate proportionality of the diffusion time to the square of the thickness according to Eq. (2) is confirmed by the measurement of the diffusion time, t_{98}, of an adhesive sample of various thicknesses, d. For the H74F adhesive, the deviation from this square dependence, as determined from the measured values, is 4.1%, while it is 4.9% in the 302-3M adhesive, and only 0.5% in the Vitralit 1507 adhesive.

For various sample thicknesses of the same adhesive, comparable permeability coefficients were measured (see Table 2). These results document the dependence on the sample thickness of the permeation rate as shown in Eq. (1). Two samples of the Vitralit 1507 adhesive were produced and measured one after the other. In the case of the H74F and 302-3M adhesives, the same sample was measured with two different thicknesses. Prior to the second measurement, the thickness of the samples was reduced by milling.

A typical measurement of the helium permeation is shown in Fig. 7 for the 301 adhesive.

4.2
Vacuum stability in adhesively bonded and laser-welded gyrometer packages

Table 3 indicates the results measured for the pressure rise in evacuated gyrometer packages sealed by adhesive bonding and by laser welding. The pressure rise, Δp, in the time Δt was used to calculate, under the approximate assumption of a linear pressure rise, the total permeation rate, Q_{total}, resulting from the permeation of the ambient air and desorption from the walls into the package.

Both the bonded and the laser welded packages attained tightnesses of down to $Q_{total} = 10^{-17}\ m^3/s$.

If the adhesive geometry on the package is taken into account, i.e., the thickness of the adhesive layer, $d = 1.5$ mm, as well as the area to be permeated, $A = 0.005\ mm^2$, this, together with the total permeation rate, Q_{total}, according to Eq. (1) results in a permeability coefficient, P_{total} (including wall desorption), for the two packages sealed with the H72 adhesive (see Table 3) which is approximately a factor of 1.4–5.4 lower than the helium permeability coefficient of $P = 0.21 \times 10^{-17}\ m^2/(Pa\ s)$ measured with the reference sample (see Table 2). This is understandable, as it may be assumed that, as for other polymers (see Table 1), also for epoxy resins the permeability to air is lower than that to helium, and the higher permeability of epoxy resins to water vapor, because of its low partial pressure in air, results in a low permeation rate through the adhesive.

Table 2. Measured diffusion times, t_{98}, permeation rates, Q, and the resultant permeability coefficients, P, of helium through various samples of epoxy resin adhesives and through aluminium, respectively, arranged by rising permeability coefficients, P

Sample (Curing)	Filler particle size weight fraction	Sample thickness, d (10^{-3} m)	t_{98} (h)	Q (10^{-14} m^3/s)	P (10^{-17} m^2/(Pa s))
Aluminium	-	1.8	-	<0.005	<0.00014
H72 (100 °C, 30 min)	Al_2O_3 < 60 µm 55%	2.0	54	7.0	0.21
H70E (80 °C, 135 min)	Al_2O_3 < 60 µm 72%	2.0	23	8.4	0.25
H70E-2 (80 °C, 135 min)	Al_2O_3 < 60 µm 77%	2.0	23	8.5	0.26
H74F (1)	Al_2O_3 < 20 µm 71%	1.5	23	21	0.48
(100 °C, 30 min) (2)*		1.2	15	25	0.45
Vitralit 1507 (1)	SiO_2 < 1 µm 20%	1.8	12	24	0.65
(UV, 270 s) (2)		1.1	4.5	34	0.57
H77 (150 °C, 45 min)	Al_2O_3 < 16 µm 20%	1.7	25	29	0.75
T7109 (100 °C, 12 h)	BN < 20 µm 63%	2.0	17	29	0.88
353ND (80 °C, 30 min)	-	1.8	2.7	25	0.68
301 (65 °C, 90 min)	-	2.0	2.7	23	0.73
377 (150 °C, 90 min)	-	1.9	2.5	39	1.1
302-3M (1)	-	1.5	2.3	50	1.0
(65 °C, 135 min) (2)*		1.1	1.2	52	0.93
Durapot 863 (<180 °C, 2–4 h)	-	1.3	1.5	56	1.1
Durapot 862 (<180 °C, 2–4 h)	-	1.8	2.5	85	2.3

* Milled sample

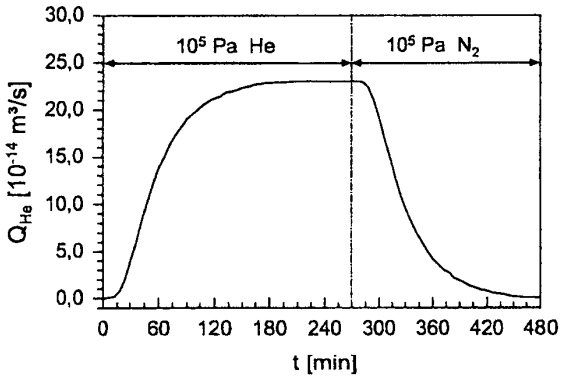

Fig. 7. He permeation rate, Q_{He}, as a function of the measuring time, t, for epoxy resin adhesive sample 301

Table 3. Pressure rise, Δp, in adhesively bonded and laser welded LIGA gyrometer packages, and the resultant total permeation rates, Q_{total}, and permeability coefficients, P_{total}

Sensor no. packaging	$\Delta p(\Delta t)$ (Pa)	Δt (days)	Q_{total} (10^{-16} m^3/s)	P_{total} (10^{-17} m^2/(Pa s))
0473 Bonding, H72	<3	38	<0.13	<0.039
1075 Bonding, H72	20	62	0.51	0.15
1054 Laser-welding	6	121	0.08	0.024
1051 Laser-welding	20	129	0.25	0.075

5 Conclusions

Epoxy resin adhesives, because of their low gas permeability, can be used for simple and low-cost sealing of packages requiring high levels of tightness for microcomponents. The helium permeability measured of epoxy resin adhesives filled with ceramic and glass particles, respectively, as a rule was lower than that of unfilled epoxy resin adhesives. The use of H72 epoxy resin adhesive, in which the lowest helium permeability coefficient had been measured in sealing evacuated LIGA gyrometer packages, resulted in total permeation rates of the ambient air and wall desorption into the package of a minimum of 10^{-17} m^3/s. At these permeation rates, a partial pressure of 100 Pa will be attained in the gyrometer package only after approximately 3.4 years. These permeation rates can be compared to those measured with laser welded packages. Conclusions about the long-time stability of adhesively bonded and laser welded packages could be provided after the application of temperature cycles and humidity.

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三、德國 IMM 研究單位之相關微系統技術資料

..... X-Ray Scanner for Deep X-Ray Lithography.

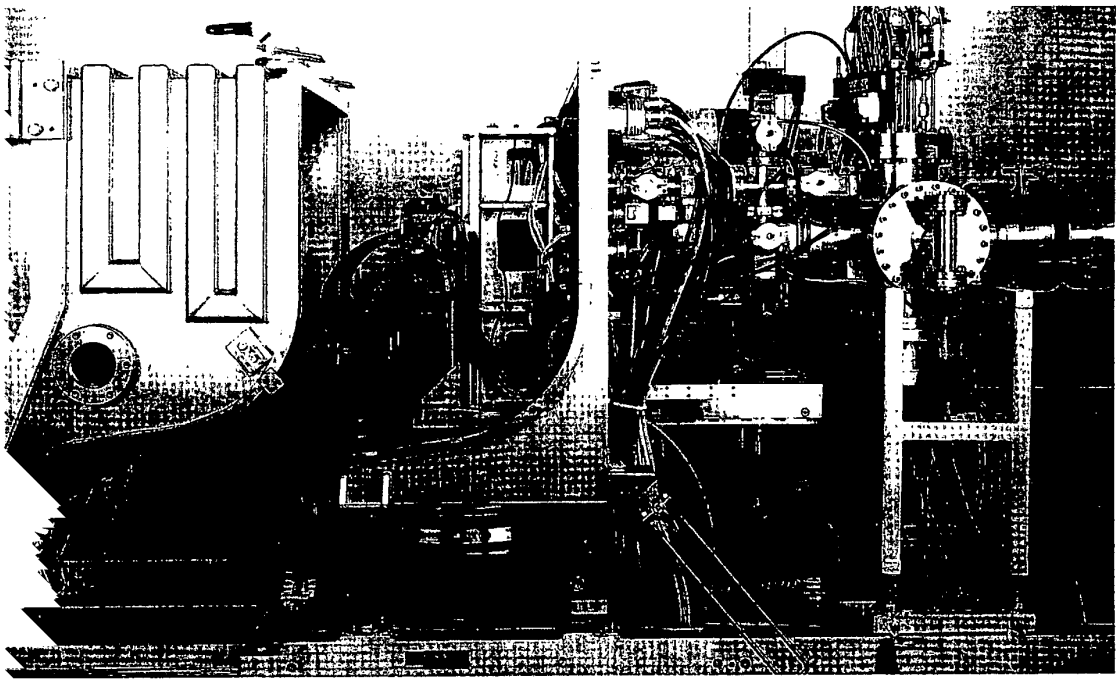

Fig. 1
X-ray scanner with internal optical alignment system

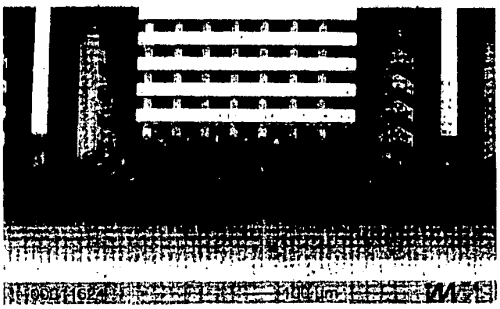

Fig. 2
Three-dimensional microstructure, fabricated by dual oblique exposure of a thick X-ray resist layer and subsequent development

Constituents of the apparatus:

- Scanning table with mask and resist holder
- Integrated tilt and rotation module
- Internal optical alignment system
- Evacuable work chamber
- UHV-compatible prefilter chamber with automatic filter change
- Flexible underframe
- Control unit
- Interlock system

Features of the apparatus

- Modular and compact, 4", 5"and 6" mask and substrate holders
- Exposure under vacuum or any inert atmosphere
- Standard exposure of substrate/resist thicknesses up to 15 mm
- Adjusted exposures up to 1000 µm resist thickness
- Fully automatic exposure

Technical data:

- Vertical exposure range 125 mm
- Table speed 1-50 mm/s
- Tilt error less than 100 µrad
- Horizontal and vertical apertures

Optical alignment system:

- Alignment accuracy of the instrument +/- 0.3 µm

Tilt and rotation module:

- Tilt angle for oblique exposure max. 60° (in 0.1° increments)
- Angle of rotation +/- 180° (in 0.1° increments)

JENOPTIK
JENA
Mikrotechnik

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Mode of operation

Since synchrotron radiation is emitted as a narrow horizontal fan and illuminates only a small part of the mask surface vertically, it is necessary to move the mask-resist system evenly through the ray. IMM and the company JENOPTIK Mikrotechnik GmbH have developed radiation equipment that allows to transfer mask structures very precisely into the depth of a resist material by way of shadowing. Through an integrated optical alignment system, adjusted multiple exposure on prestructured substrates is possible. A tilt and rotation module enables oblique exposures and a rotary movement around the incident beam axis. The radiation equipment is also available in a basic version, which additionally can be equipped with an external alignment system and/or an external tilt and rotation module ●

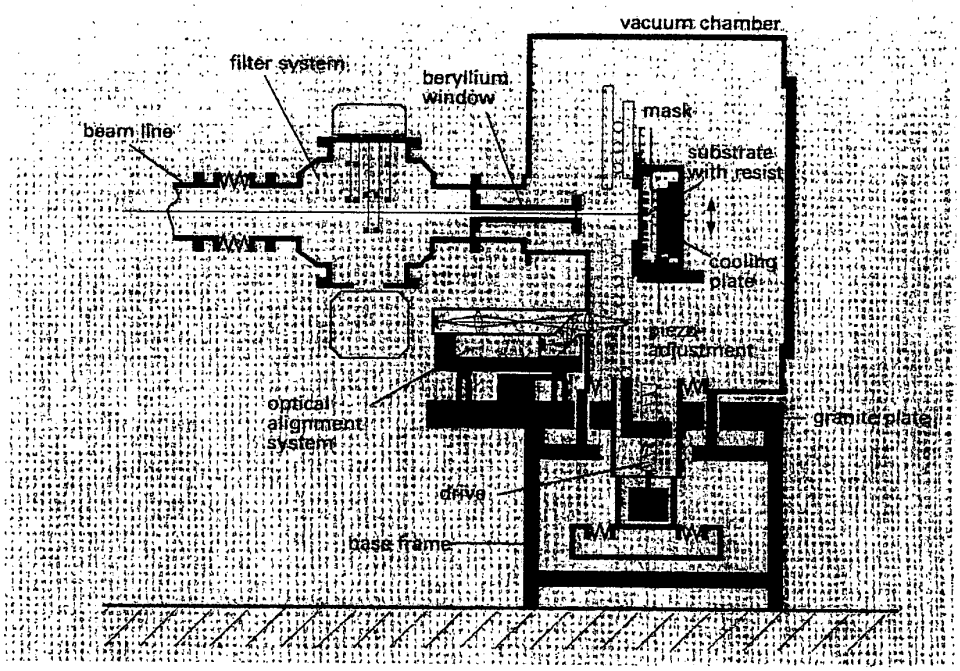


Fig. 3
Sketch showing the principle of the radiation apparatus at the storage ring. The synchrotron radiation fan is generated by the deflection of electrons or positrons in a magnetic field. The homogeneous illumination of mask/resist ensemble is achieved by a vertical movement of the scanning table.

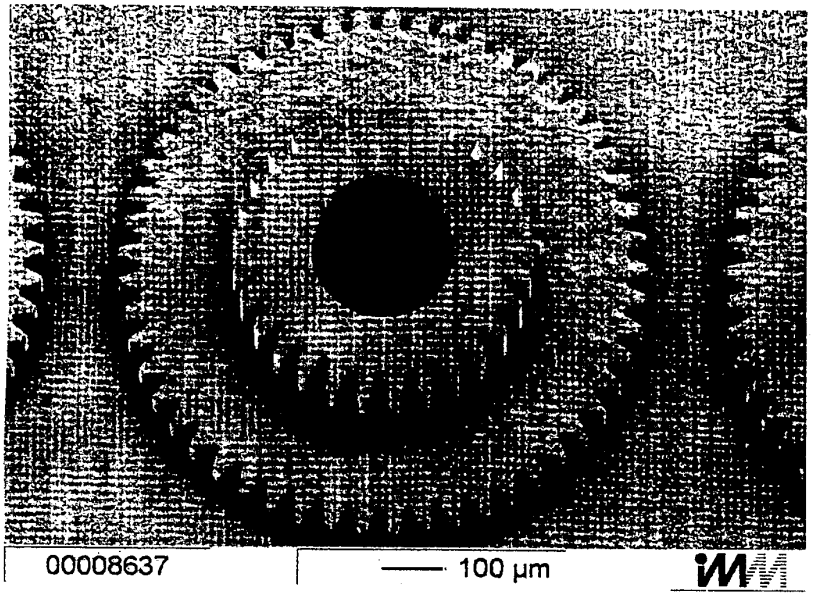


Fig. 4
Double gear wheel for a microgearing unit, fabricated by adjusted multiple exposure. Material: nickel

Your contact at IMM:
Public relations
Phone: +49 6131/990-117
Fax: +49 6131/990-205
Institut für Mikrotechnik Mainz GmbH
Carl-Zeiss-Strasse 18-20
D-55129 Mainz
http://www.imm-mainz.de
or
Hartmut Blum
Phone: +49 36 41 / 65 33 38
Fax: +49 36 41 / 65 35 62
Jenoptik Mikrotechnik GmbH
Prüssingstrasse 41
D-07745 Jena

IMM 6 12/0799 E. Roth & Gerisch, Darmstadt

Cantilever with integrated distance sensor

In order to get the full advantage of an AFM tip it is necessary to develop position sensitive systems that are precise enough to control the tip down to the atomic level. To use such a system even under extreme conditions like in an ultra high vacuum chamber, IMM in collaboration with Omicron Vakuumphysik GmbH realized a detection unit based on a Fabry-Perot interferometer that is incorporated in the cantilever head (Fig. 1, Fig. 6).
A laser beam that hits the cantilever head from the back is reflected into a detector unit. The intensity registered is a well defined function of the cantilever tip position with respect to its holder and is used to control the distance between the tip and the substrate to the desired precision. This unit can simply be exchanged with a new one if the tip is damaged and does not require any additional adjustment. Prototypes showed a sub Angstrom resolution.
A small scale production of this sensor is now in progress.

Standards for scanning probe microscopy

The tip radii of scanning probe microscopy (SPM) probes often vary due to wearing or fabrication tolerances. The determination of the tip radius can be achieved by measuring calibration standards with well defined dimensions. The quality of the tip can be judged by comparison of the picture with the known shape of the standard. Most standards provide small pyramidal indents which are anisotropicly etched into silicon. IMM has developed a process that allows to mould these silicon standards by electroforming. In contrast to silicon standards, the moulding provides protruding pyramids, which reveal better information about the tip shape (Fig. 3, Fig. 7).

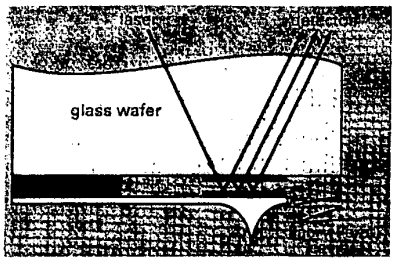
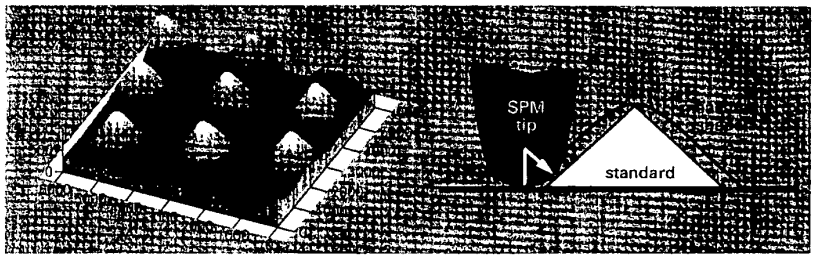


Fig. 6: Schematic view of a cantilever with integrated Fabry-Perot distance sensor

Fig. 7: Scanning probe microscopy standard tip radius determination



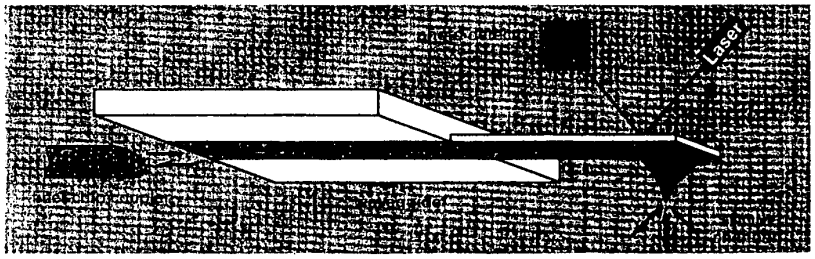


Fig. 8: Concept for a multifunctional AFM-SNOM probe based on thin film technology. The waveguiding cantilever combined with an aperture SNOM tip makes simultanous measurements of optical and atomic force images possible.

Combination of AFM and SNOM

Optical resolution with conventional lens based microscopes is limited due to diffraction to about one half of the wavelength that is used, typically 500 nanometers. If a tiny metallic aperture is placed close (less than 25 nm) to the subject under investigation it is possible to overcome the diffraction limit. This technique is called near field optical microscopy (SNOM). This technique will open the field of applications towards medicine and biology, since it will be possible to get fluorescence and infrared data on the molecular level.

Since the amplitude of the optical near field varies strongly with distance the spacing between the aperture and the sample has to be controlled precisely.

A logical step is to combine AFM and SNOM into one device to get at the same time, topological and optical information from one machine (Fig. 8).

Micromachined SNOM Tips

Whereas most AFM-SNOM probes are based on a fiber technology that has problems with production and lateral resolution, IMM developed a concept that is based on thin film technology. The advantage of this approach lies in the better control of tip geometry, a bigger range of possible materials and the fact that one production cycle can produce a big number of identical tips on a silicon wafer (Fig. 10). By optimizing these technologies we are currently able to produce a Si_3N_4 tip on a standard AFM cantilever with a well defined aperture in the 100 nanometer range (Fig. 9). Due to the high refractive index of Si_3N_4 this system shows transmissions between 10^{-3} to 10^{-4} in the visible light range. This is up to 100 times more light that can be transmitted than by conventional fiber probes.

SNOM Tip Arrays

Our way of making SNOM tips also allows the fabrication of tip arrays (Fig. 10). IMM was the first to produce such an array on a glass substrate that opens industry the way to new concepts in optical parallel data processing ●

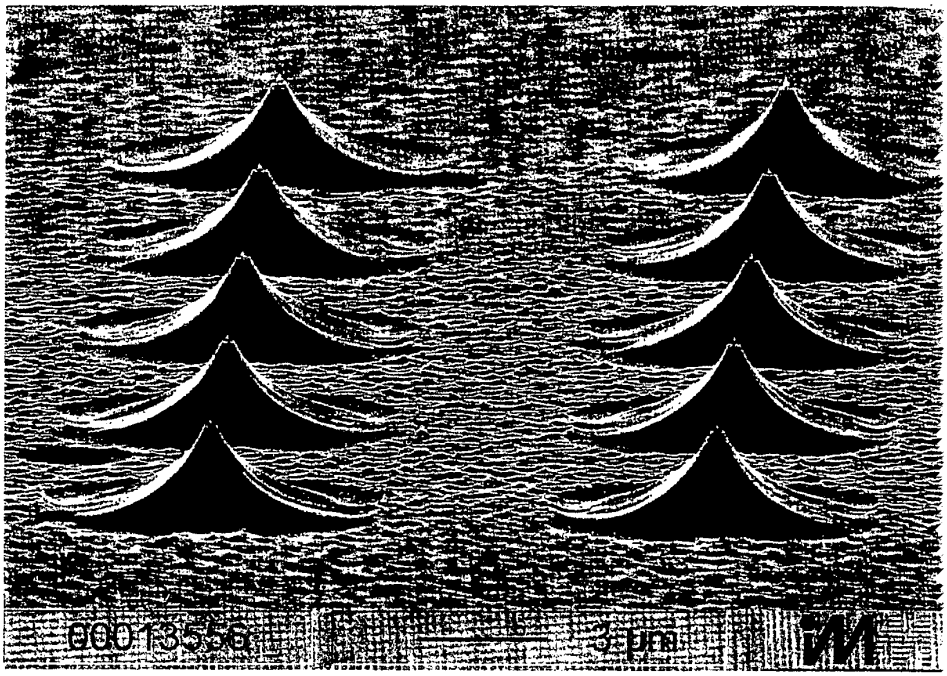


Fig. 10: Micrograph of SNOM tip array

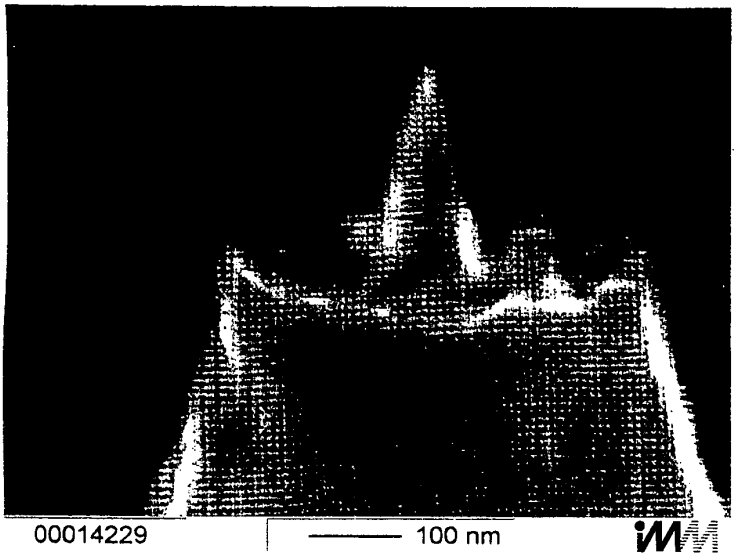


Fig. 9: SNOM tip with 120 nm aperture

Your contact at IMM:
Public relations
Phone: +49 6131/990-117
Fax: +49 6131/990-205
Institut für Mikrotechnik Mainz GmbH
Carl-Zeiss-Straße 18-20
D-55129 Mainz
http://www.imm-mainz.de

Micromoulding

Fig. 1
Microstructures produced by injection moulding

Varieties of micromoulding

- Injection moulding of plastics, ceramics and metallic powders
- Embossing and extrusion of plastics
- Tapecasting and embossing of metallic and ceramic powders
- Casting of pre-ceramic suspensions
- Slurry casting of ceramics
- Embossing of aluminium

Technological requirements

- Adapted injection moulding and embossing equipment
- Evacuable cavities
- Variothermal process management for high aspect ratios
- Ejection of moulded parts without lateral offset
- Complete process control
- Use of small plastification units during injection moulding

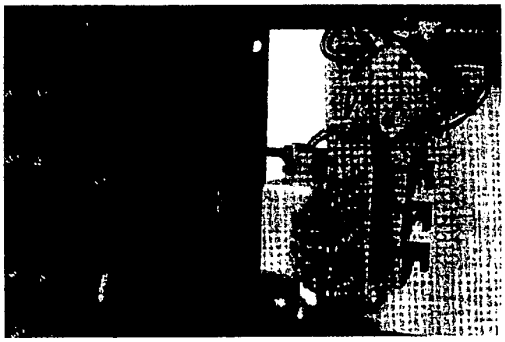


Fig. 2
Injection moulding and unloading system for the micro-injection moulding of flow sensors

Areas of application

- Optics
- Environmental technology
- Medical technology and biotechnology
- Electrical and optical packaging and interconnection
- Measurement technology and sensor systems
- Actuator systems
- Telecommunications

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Mould inserts

For the fabrication of high quality but economically priced moulded microstructural components, the choice of a suitable mould insert is paramount. IMM has numerous highly developed processes immediately available or under development with its partners. The differences between mould inserts lie in the size of structure that can be obtained, the aspect ratios and the attainable dimensional tolerances. The principal commercial considerations are fabrication costs, reproducibility, and access time. Table 1 gives an overview of the processes most often deployed at IMM for the production of micromould inserts.
Fig. 3 shows arbitrarily shaped surfaces for a mould insert of a mixer used in the area of chemical micro-reactors. The mould insert is fabricated by the combination of excimer laser ablation and electroforming in nickel.

Processes for the fabrication of mould inserts	Typical lateral structure dimensions	Typical aspect ratios	Typical material	Availability at IMM
Micromechanical treatment (milling)	100-1000 µm	10-50	Steel	available
EDM	100-1000 µm	10-100	Steel	available
Laser ablation and electroforming	5-500 µm	1-10	Nickel and nickel alloy	available
E-Beam Lithography and electroforming	100-500 nm	1	Nickel and nickel alloy	In cooperation
UV Lithography and electroforming	5-100 µm	1-10	Nickel and nickel alloy	available
X-Ray Lithography and electroforming	1-1000 µm	10-100	Nickel and nickel alloy	available
Wet chemical etching of silicon and electroforming	5-500 µm	1	Nickel and nickel alloy	available

Table 1
Processes deployed at IMM for the production of micromould inserts

Fig. 3
Micromould inserts produced by laser ablation and electroforming

Engineering

In the area of precision engineering, computer simulation both for design functions and for processes is state-of-the-art. An example is the prediction of filling behaviour in injection moulding by flow simulation and the consequent design of mould inserts and tools.

Studies at IMM have shown that in three-dimensional structures in the range of a few hundred micrometers, a high-quality prediction of filling behaviour is possible with the aid of conventional injection moulding software. Figure 5 shows the example of the simulation result for structures of a micropump. The predicted incomplete filling of a side was subsequently confirmed by experiment (see also Figure 6).

However, in the area of microtechnology, factors such as the small dimensions of the structures, the high ratio of surface to volume and the three-dimensionality of the moulds have a limiting effect on the application of existing FEM simulation modules. To overcome these problems, IMM is cooperating with its partners on the development of simulation software suited for microstructures, and with a special eye to micro injection moulding.

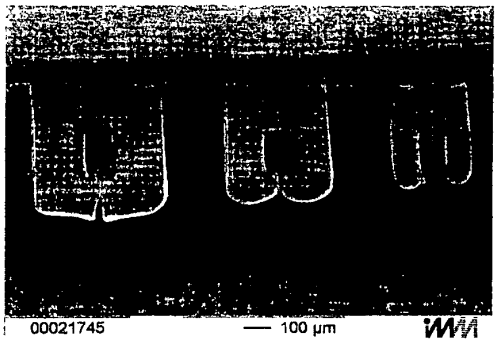
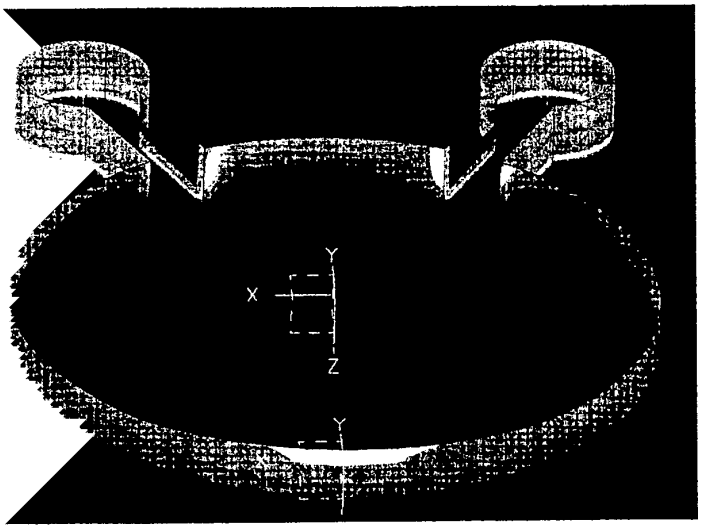


Fig. 4
Fundamental research on materials characteristics, formation of seams during bypassing of obstacles in micro injection moulding

Fig. 5
Filling sequence in the injection moulding simulation of a micropump. Result: incomplete filling in the area of a valve side with a width of 150 µm



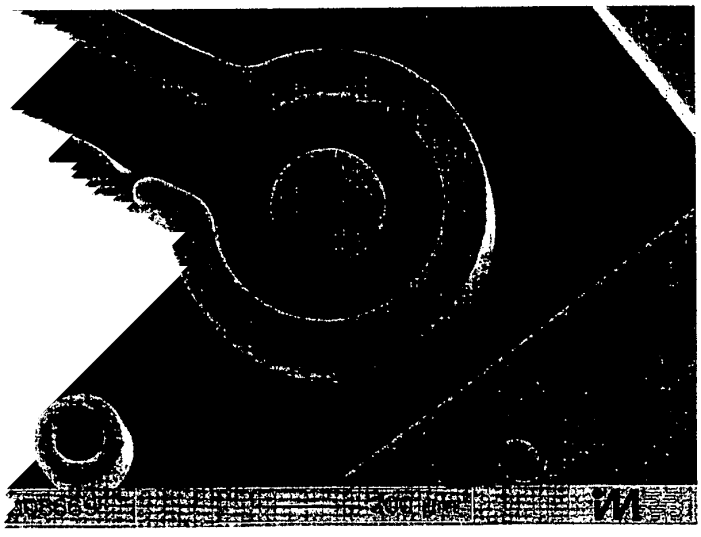


Fig. 6
Injection moulding experiments on a micropump. Result: incomplete filling in the area of a valve side

Materials

The choice of the materials to be moulded is mainly governed by the functions of the products. For this reason, at IMM potentially interesting thermoplastic polymers and, increasingly, powder ceramics and metals too are being developed for microtechnology in special screenings. Typical quality aspects which are used for the categorization and evaluation of the materials include the mould filling behaviour, the attainable flow path length, shrinkage and warpage, the surface roughness which can be obtained and the reproducibility.

The example shown in Figure 4 is the formation of seams during the bypassing of obstacles in micromoulds fabricated by LIGA technology.

Quality control

Moulding processes potentially offer the possibility of low cost mass production of ultraprecise micro-components. For this, properly developed quality control is essential. Because of the small structural dimensions involved and the high aspect ratios, here it is necessary to open up new avenues. IMM is working in this area on the further development of existing and, together with its partners, the realization of new measurement technologies. Another avenue being pursued at IMM with regard to the fabrication of large quantities is to replace quality control of the moulded products, which involves a high input of measurement technology, with registration and analysis of the processing parameters of well-understood moulding processes.

The process of product development in the area of micromoulding technology is already standardized. Statistical experiments planning and the coordinated registration and analyis of processing parameters and mould quality have replaced "trial and error". At present, the way is also being prepared to proceed to the fabrication of microstructured products primarily with micro injection moulding •

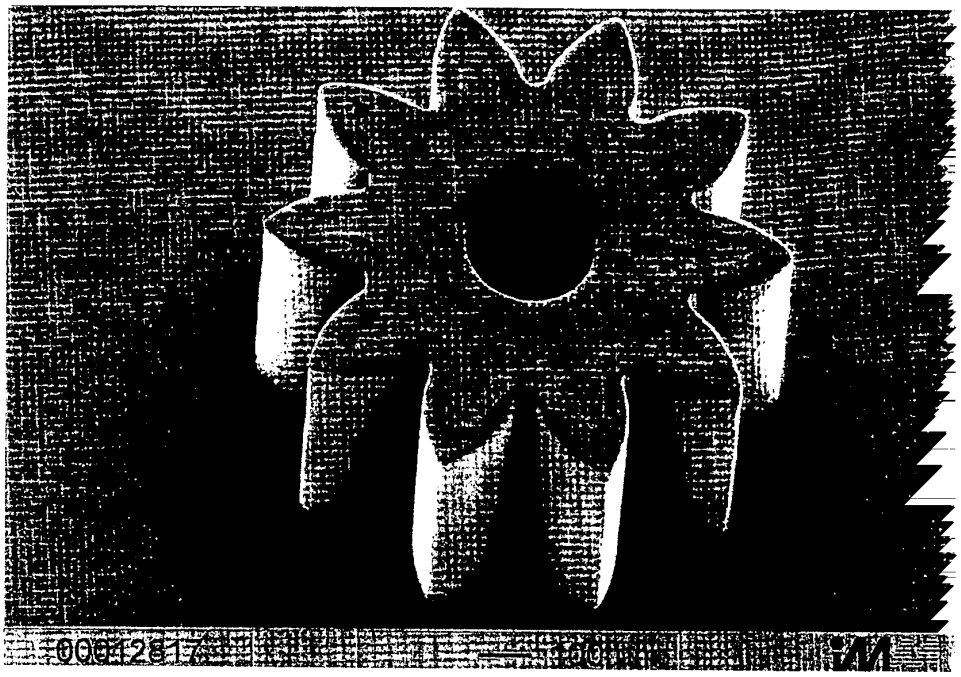


Fig. 7
Micro gear wheel for motor gearing; manufactured by injection moulding

Applications

- Diffractive and refractive optical components and integrated optical waveguides
- Ultraprecise components for optical packaging and interconnection technology and lens brackets (see Fig. 9)
- Gear wheels (see Fig. 7) and brackets for microgears
- Optical lenses and lens arrays
- Mixers, grooves and reaction vessels for microreactors
- Micro heat exchangers and components for molecular biotechnology
- Micropumps

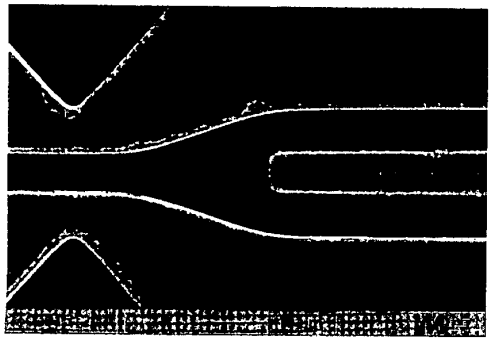


Fig. 8
Integrated optical waveguide structures; manufactured by hot embossing

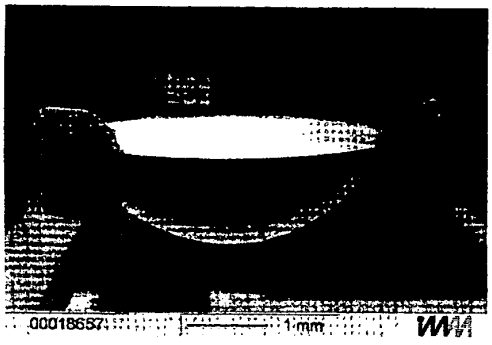


Fig. 9
Precision bracket for endoscope lenses; manufactured by injection moulding

Your contact at IMM:
Public relations
Phone: +49 6131/990-117
Fax: +49 6131/990-205
Institut für
Mikrotechnik Mainz
GmbH
Carl-Zeiss-Strasse 18-20
D-55129 Mainz
http://www.imm-mainz.de

IMM 8 02/0797 E. Bailey & Giesasch. Darmstadt

.....Electro Discharge Machining.

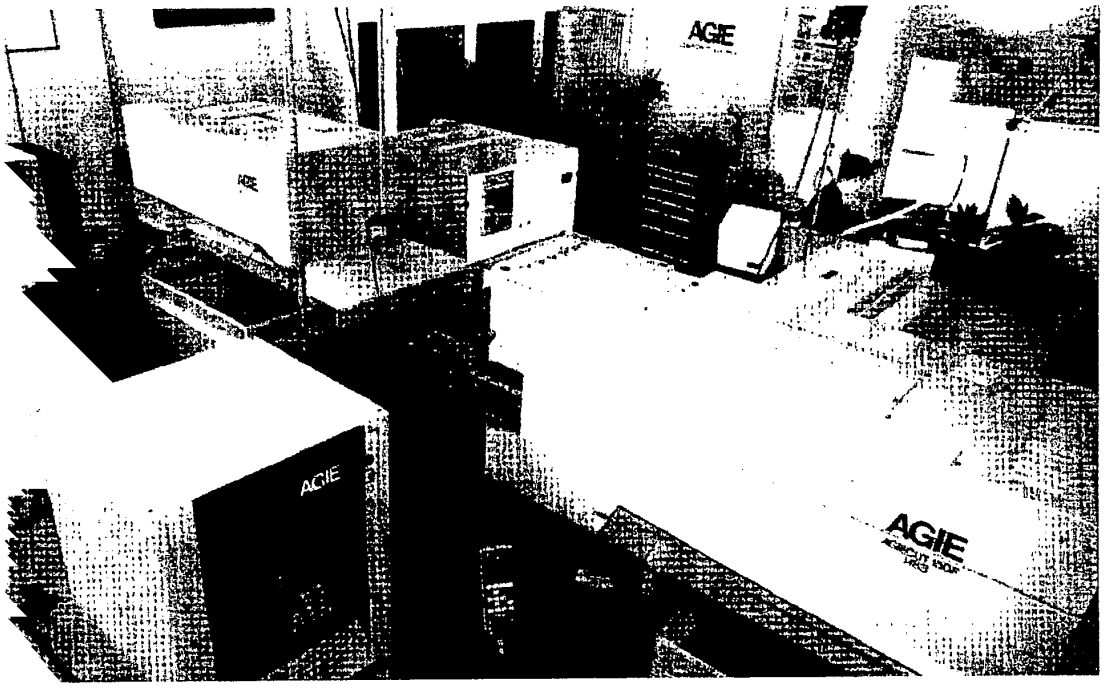
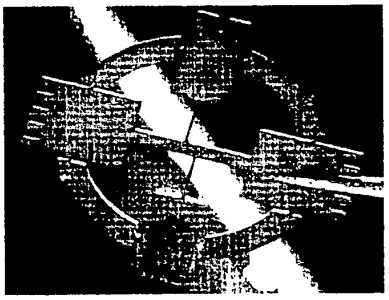


Fig 1:
Air conditioned EDM-center at IMM with AGIE die sinking and wire-EDM machining facilities

Research and Development in Micro Electro Discharge Machining for Industries

- Micro die sinking
- Thin wire-EDM
- Application work
- EDM-research
- Development of devices and equipment



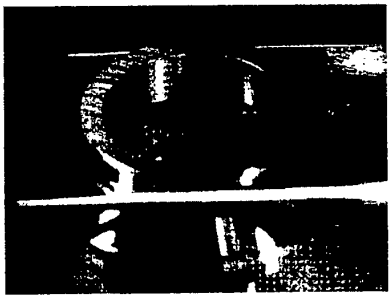


Fig. 4:
Microreactor component made of stainless steel with microholes

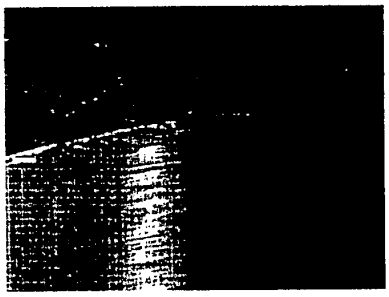


Fig. 2:
Catalyst for micro-reaction technology, fabricated by die sinking and wire-EDM; material: platinum

Fig. 3:
Detailed view of microholes with diameters of 60 µm, machined by EDM-drilling

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Mainz

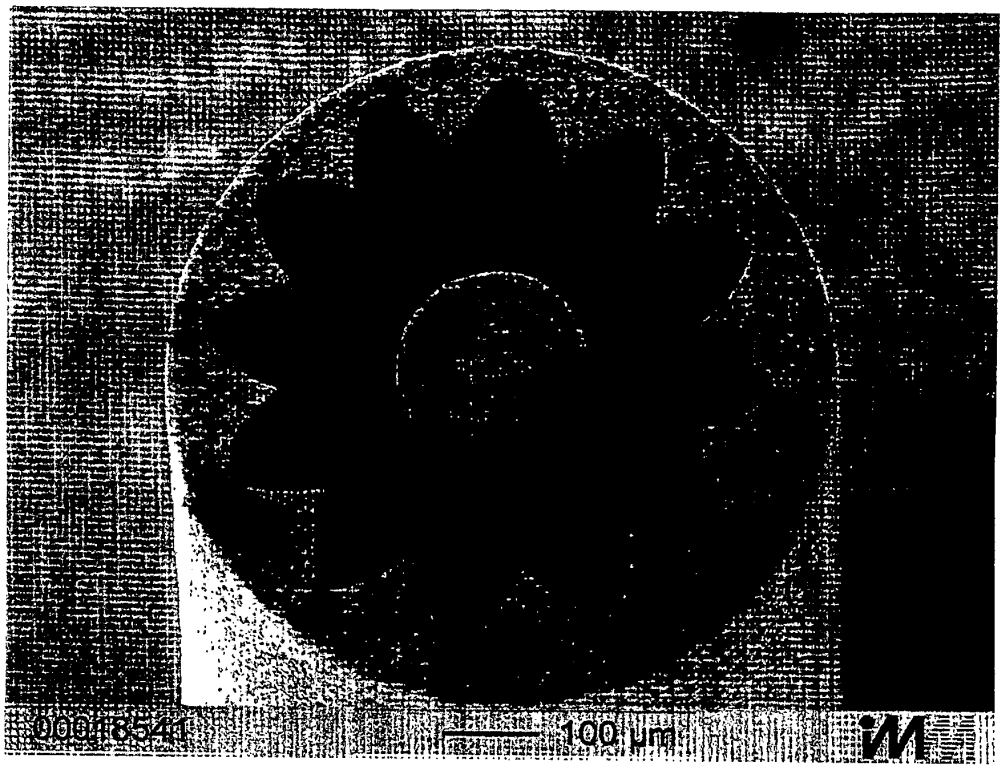


Fig. 5:
Hollow wheel of tungsten carbide, generated by using LIGA-electrodes

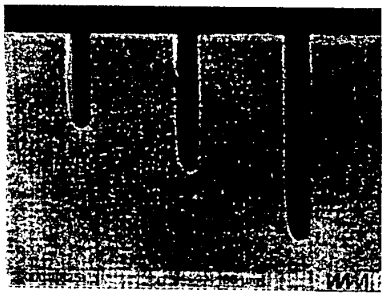


Fig. 6:
Cross section of microfluidic channels with high aspect ratio in stainless steel, machined by EDM-grinding using rotating tungsten discs as electrodes

Micro-EDM

The recent progress in generator development and machine control technology has led to highly sophisticated wire-cut and die sinking machines resulting also in the introduction of EDM as fabrication method in microsystem technology.

Precise impulse generation, highly dynamic CNC-machine control and process specific know-how result in minimized surface layer modifications as well as significant reduction of machining time. Furthermore, real-time process control provides stable and uniform working conditions and highest repeatability of production results.

Negligible small forces as well as the independence from mechanical properties of the workpiece material are some other EDM-process advantages. This also qualifies the technology to be flexibly applied in combination with other microtechniques, like LIGA, embossing or micro injection moulding, which offers a new variety in design and economic production of microcomponents.

Numerous micro-EDM applications indicate the performance of EDM-technology being well prepared for future demands in microsystem development and fabrication.

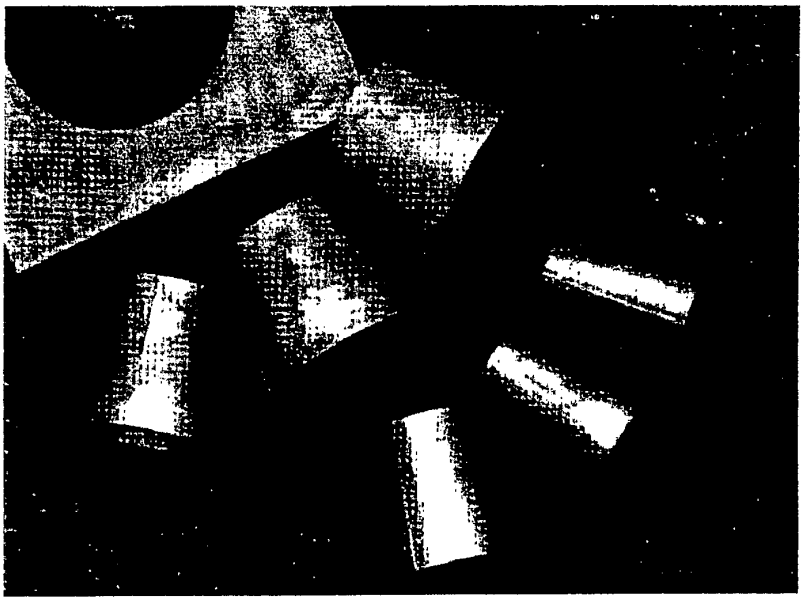
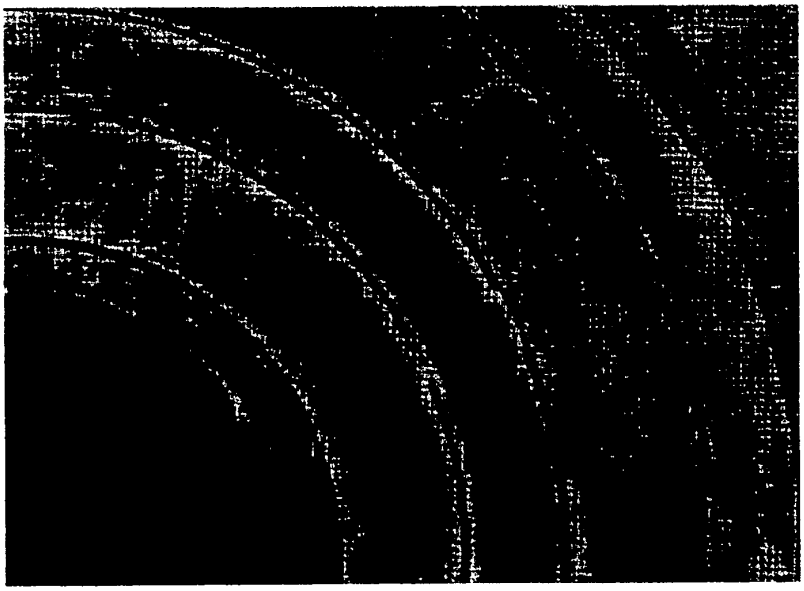


Fig. 7:
Conical slit system, machined by thin wire-EDM; demanded geometrical accuracy ± 1 µm

Fig. 8:
Mounted conical slit system; top view



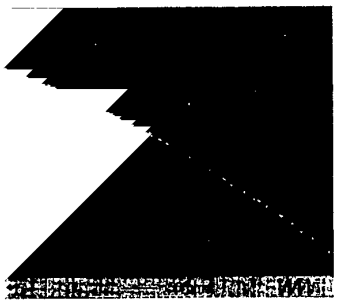


Fig. 9:
Detail view of spacers to assure a constant slit width

Thin wire-EDM

Thin wire EDM mostly uses wire electrodes made from tungsten or molybdenum with diameters down to 30 µm. Modern wire EDM machines therefore are equipped with extremely sensitive and precise wire handling systems.

This enables the generation of very fine and complex geometries down to a minimum width of 50 µm and a maximum height varying between 3 mm and 7 mm depending on the actual working conditions. The characteristic shape tolerance which can be obtained is in the order of ± 1 µm. Sophisticated surface finish strategies provide a surface quality up to R_a = 0,1 µm. The taper angle can be varied within ± 15°.

Besides highly precise tool making for microoptical and micromechanical devices, thin wire-EDM at IMM is directly applied for the fabrication of actuators and microtools, like microgrippers or components for microreaction systems. EDM processes are furthermore well suited for use in combination with other techniques, like LIGA, laser cutting processes or micro die sinking.

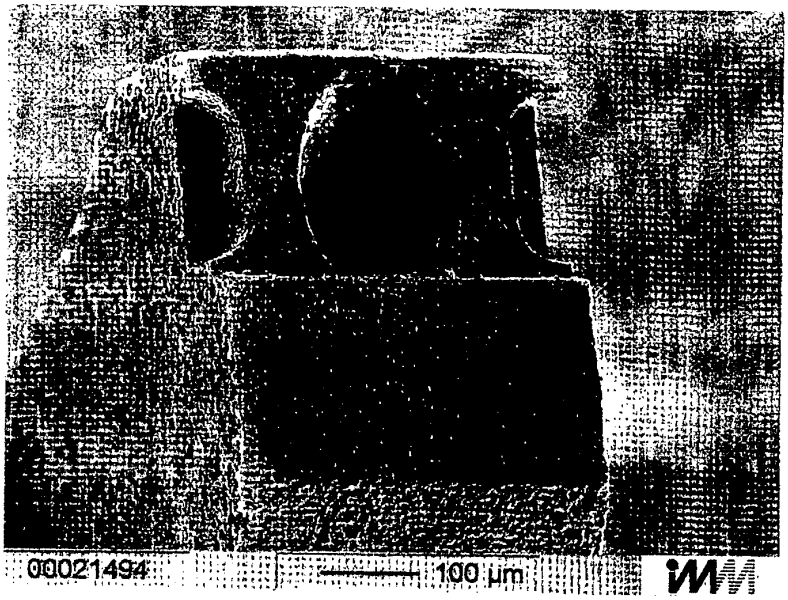
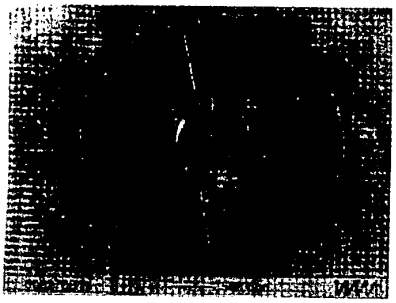


Fig. 10:
Gripper for micro-assembly of highly sensitive semiconductor elements, machined by die sinking



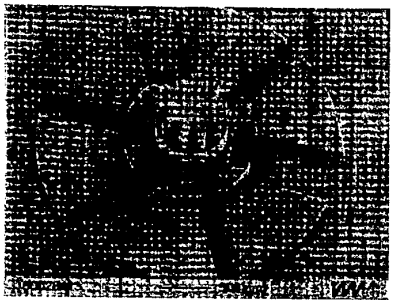


Fig. 11:
Die sinking electrode, machined by EDM-milling

Fig. 12:
LIGA mould insert, finished by die sinking

Fig. 13:
Part of a microgear, fabricated by injection moulding

Application work

Fast response times and immediate technological integration of systematic process research, which is continuously performed at IMM, are essential for best results.

The work for both internal and external clients is mainly focused on components for applications in microassembly, microfluidics and microoptics.

Moreover, the generation and modification of mould inserts for injection moulding, embossing and extrusion even enhances the variety of applications for µ-EDM.

Electro Discharge Machining

Expert center µ-EDM
Qualified personnel
Highly precise machining facilities
Various measuring instruments
Direct access to precision- and micromechanical machining facilities
Development of devices and equipment

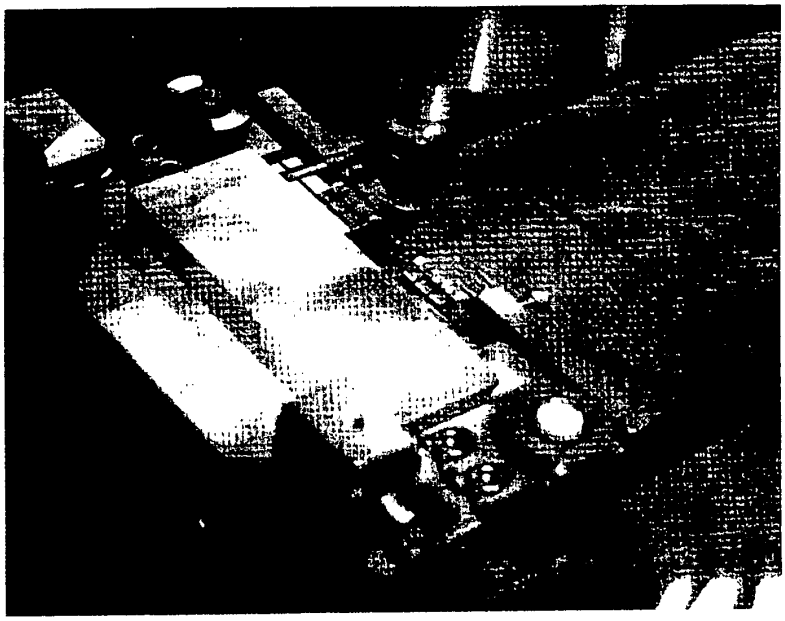


Fig. 14:
Guiding unit of an IMM designed device for electrode generation during a roughing process

Micro die sinking
Micro die sinking can be subdivided into three categories with respect to the electrode manufacturing method.

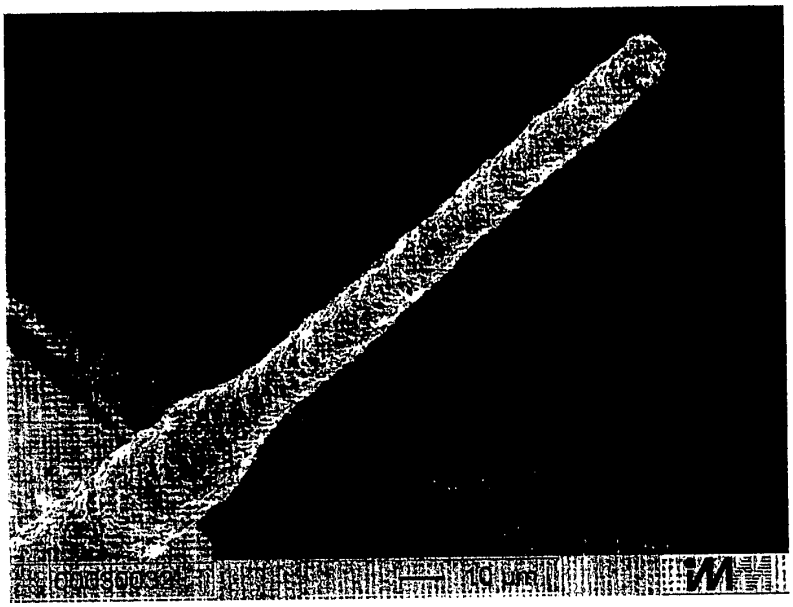
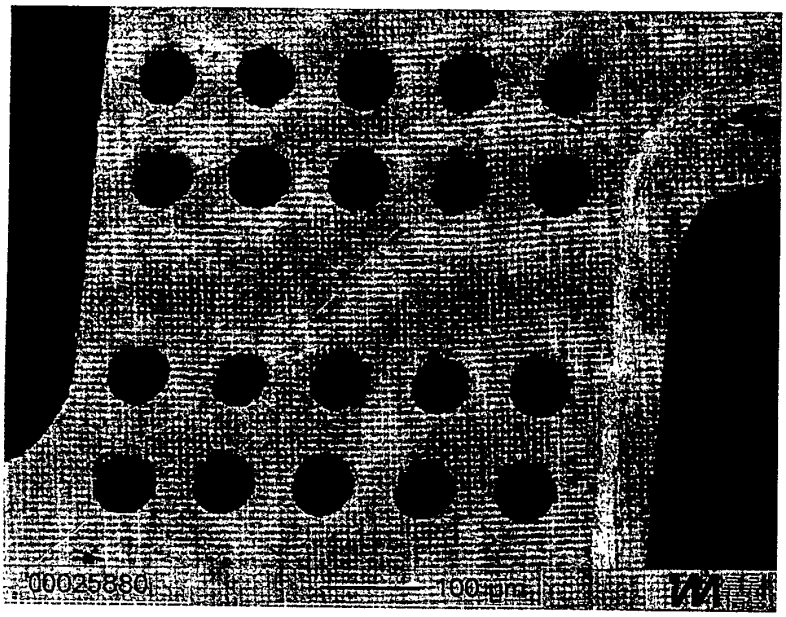


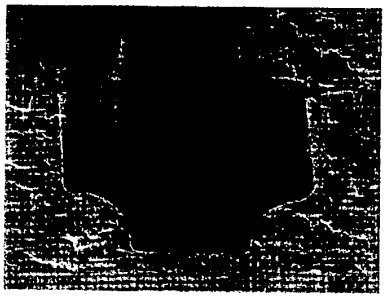
Fig. 15:
Cylindrical electrode for die sinking with a diameter of 12 µm; obtained by applying wire- electro discharge grinding, improved by IMM

① Small electrodes with precise structures can be machined by conventional methods using precision engineering techniques like milling, turning, or laser based technologies. Here, also high speed cutting becomes a machining method of growing importance, mostly used to structure graphite or copper electrodes.

② Loss of precision due to clamping, tool changing actions and adjustment operations can be avoided by generating the electrodes on the EDM-machine itself. For this purpose, IMM has developed an optimized system for electrode generation, reducing the above mentioned problems and enabling the fabrication of e.g. small cylindrical electrodes down to diameters between 5 µm and 20 µm.

③ Moreover, IMM has developed an EDM-technique applying micromachined electrodes for die sinking purposes. Structures made by LIGA are the best choice here, providing low surface roughness down to R_a = 0,2 µm as well as highly precise structures with submicron accuracy from standard electrode materials like copper or silver. Errors due to tool changing and adjustment can be neglected here. Additionally, the process time can be significantly reduced.





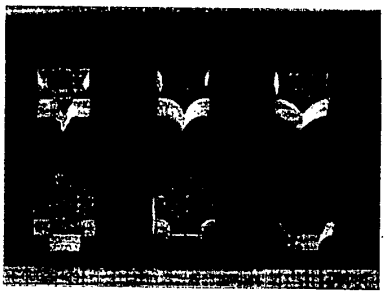


Fig. 16:
Array of microholes with diameters of 60 µm in a micro-reactor component

Fig. 17:
Spinning nozzle, machined by micro die sinking using LIGA electrodes

Fig. 18:
Nozzle for microextrusion machined by LIGA-EDM applying LIGA electrodes

Fig. 19:
LIGA electrodes for µ-die sinking

EDM-research and development at IMM

The application of EDM as a machining method in microsystem technology demands an adapted machine concept as well as sophisticated rules for electrode design and process parameters. Therefore, research in workpiece and electrode handling, dielectric flushing, adjustment and process control is required.

The improvement of measurement facilities, at IMM realized e.g. by integration of optimized equipment into the machine environment, essentially enhances process performance in EDM.

Further development at IMM is performed with respect to electrode generation and process performance. As an example, a device for electrode generation was designed, allowing fast and precise fabrication of electrodes for µ-EDM drilling or micro die sinking with smallest diameters between 5 µm and 20 µm.

Precision by Innovation

Highest precision as well as reliable results can only be guaranteed by qualified and experienced machine operators and a sophisticated machine park working under best process conditions.

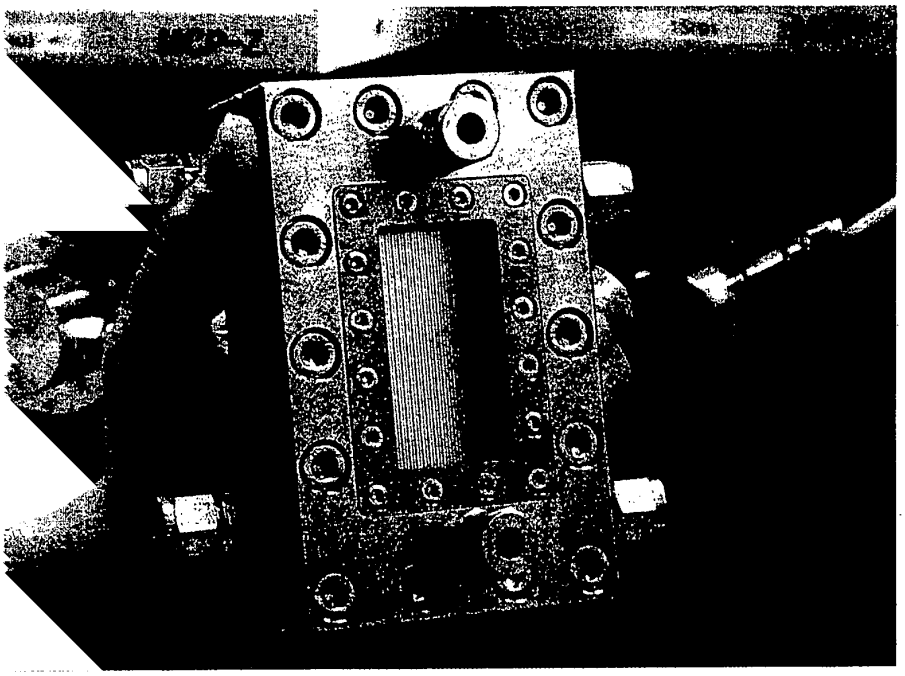
Air conditioned machine laboratories and various measurement facilities like profile projectors, scanning electron microscope, laser interferometer, ellipsometer and the white light interferometer are essential to provide continuously reproducible results.

A research and development cooperation with a leading EDM manufacturer ensures permanent technological improvement, providing minimal spark gaps for the realization of smallest structures with complex geometries in micro component fabrication ●

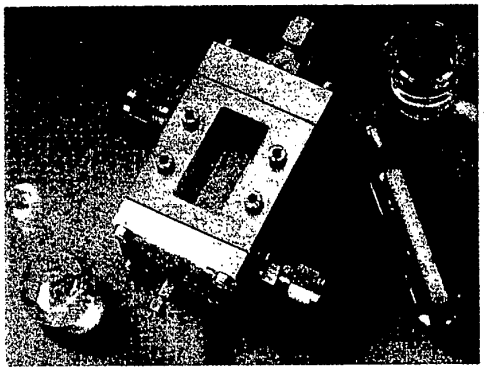
Your contact at IMM:
Public Relations
Phone: +49 6131 / 990-117
Fax: +49 6131 / 990-205
Institut für Mikrotechnik Mainz GmbH
Carl-Zeiss-Straße 18-20
D-55129 Mainz
http://www.imm-mainz.de

IMM 6.12/0698 D; Bailey & Gerasch, Darmstadt

Microreactors



Doing a first step towards microreaction technology, you may desire to have assistance in yours company's phase of technology implementation.
IMM has the tools, microfabrication and chemical engineering know-how, and – last not least – a young, creative and flexible team, which is eager to stand by your company.



Falling Film Microreactor and Micro Bubble Column

Development of a new microstructured reactor being tailor made to your field of application.
We will do it step by step, starting with feasibility study, check the success with precisely defined milestones, and finally validate the new device before delivering to you.

Solution of your chemical problem, outpacing traditional technology and searching for innovation with hindsight.

We know about the real potentials of microreactors by our wealth of experience. You will receive a discerning expert opinion, outlining the innovation, but not forgetting to bench-mark. We will built your microreactor plant and carry out the process development with it.

Institute of
Microtechnology
Mainz

Manufacture and Design of Microreactor Compone and Construction of Microreactor Setups

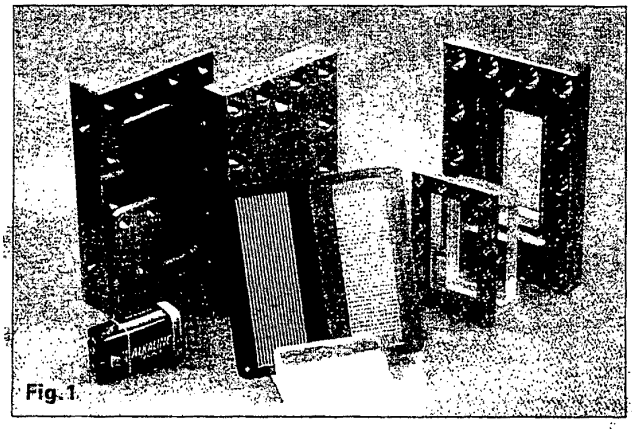


Fig.1

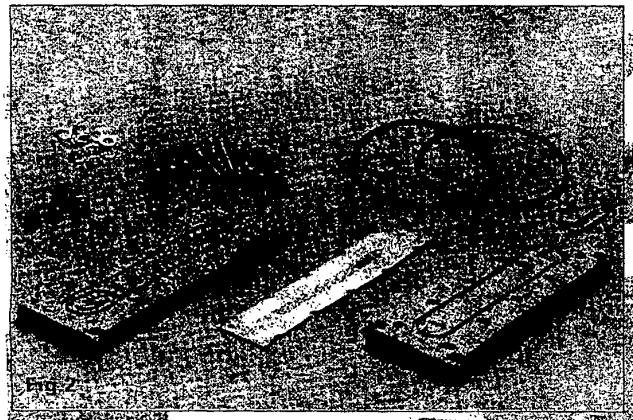


Fig.2

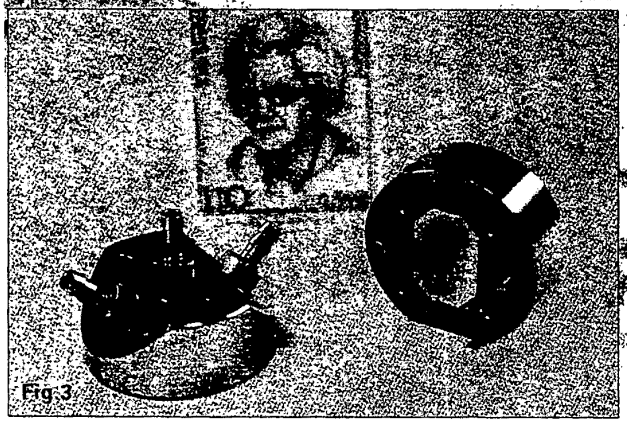


Fig.3

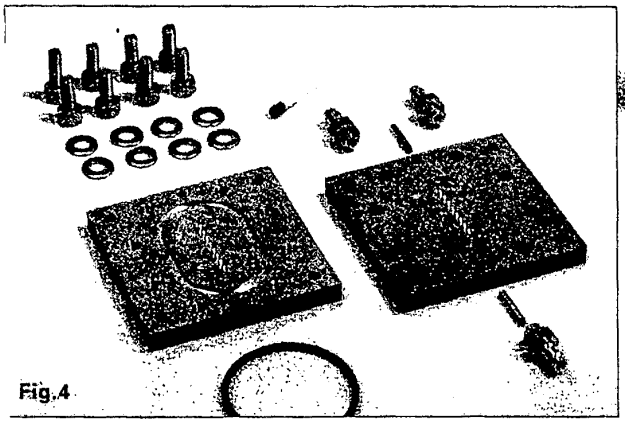


Fig.4

1. Falling Film Microreactor
2. Liquid/Liquid Reactor/Heat Exchanger
3. Interdigital Micromixer
4. Caterpillar Micromixer
5. Cross-Flow Heat Exchanger
6. Heating Module/Monolith Reactor
7. Micro Bubble Column
8. Counter-Flow Heat Exchanger
9. Your Individual Application-unique Device

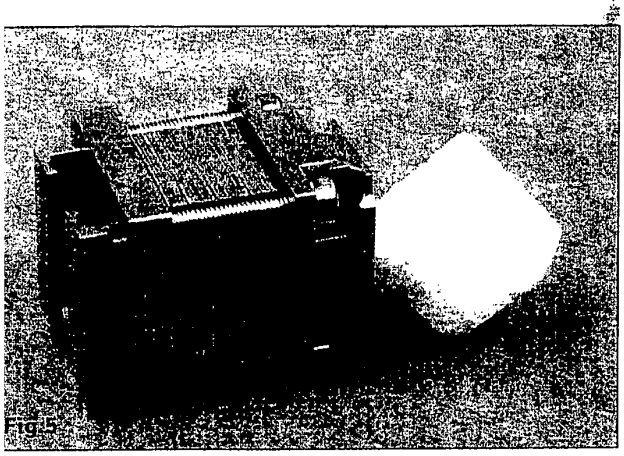
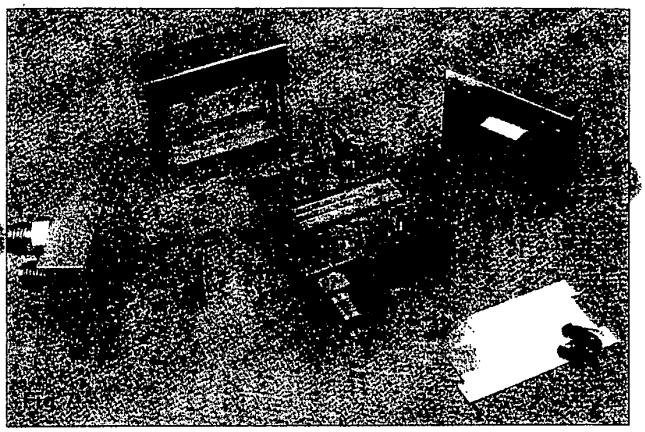
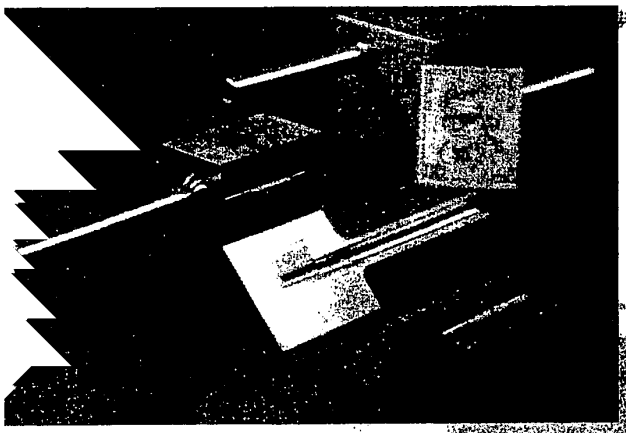


Fig.5





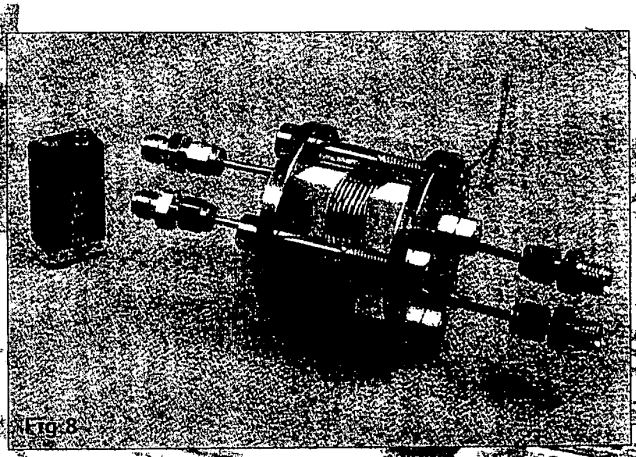


Fig.8

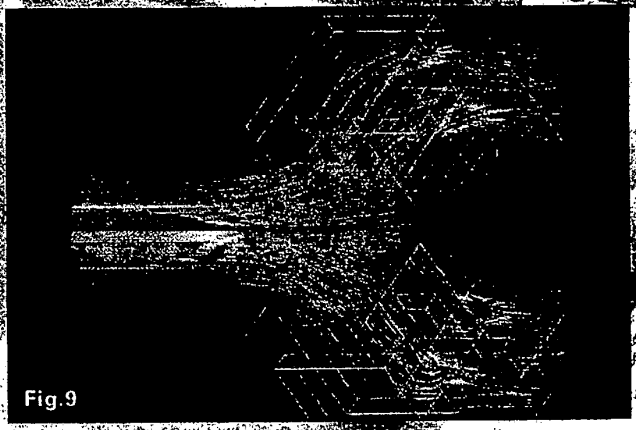
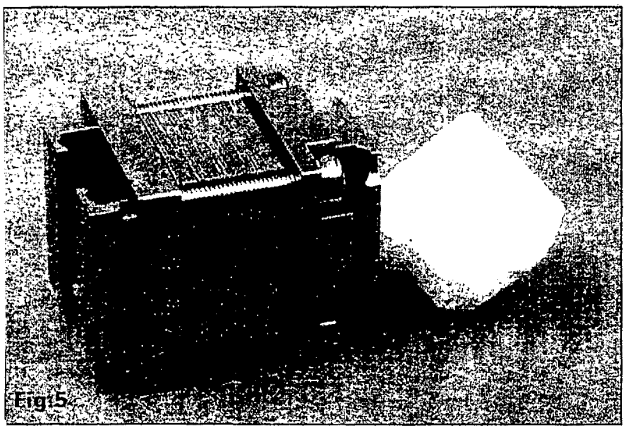


Fig.9

Get Your Individual Application-unique Device.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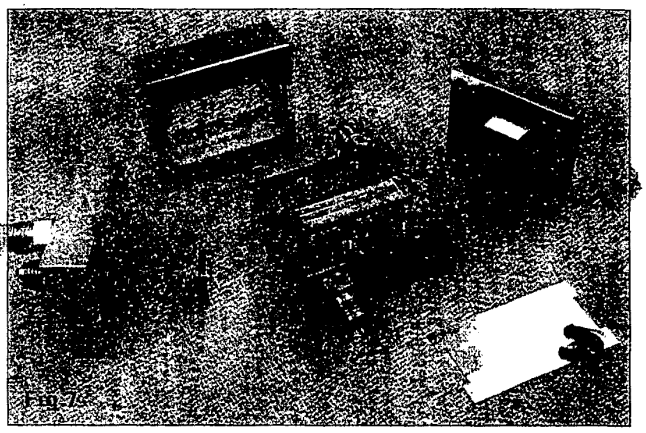
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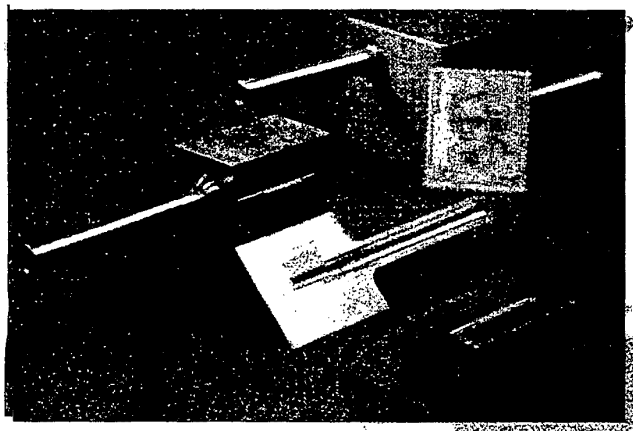

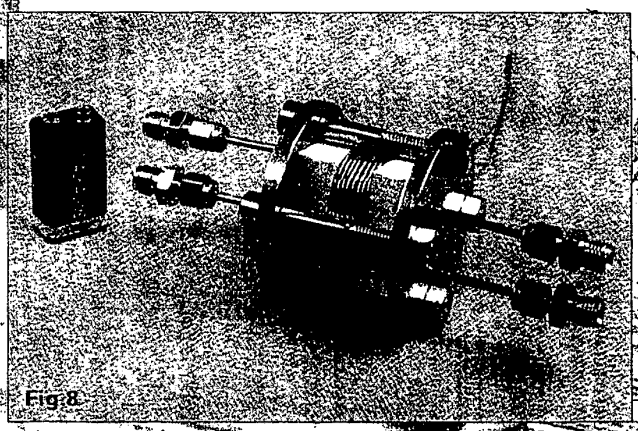
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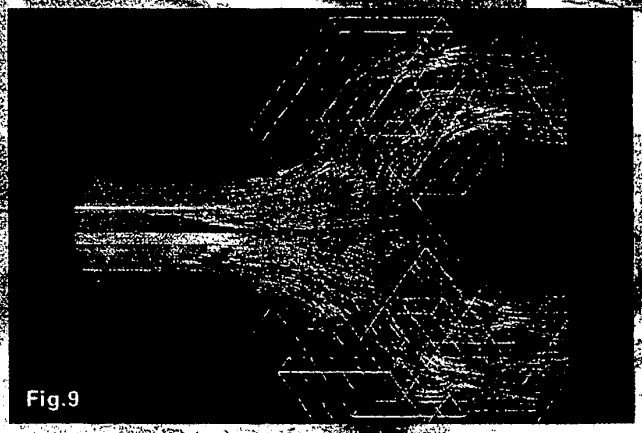
Fig.9

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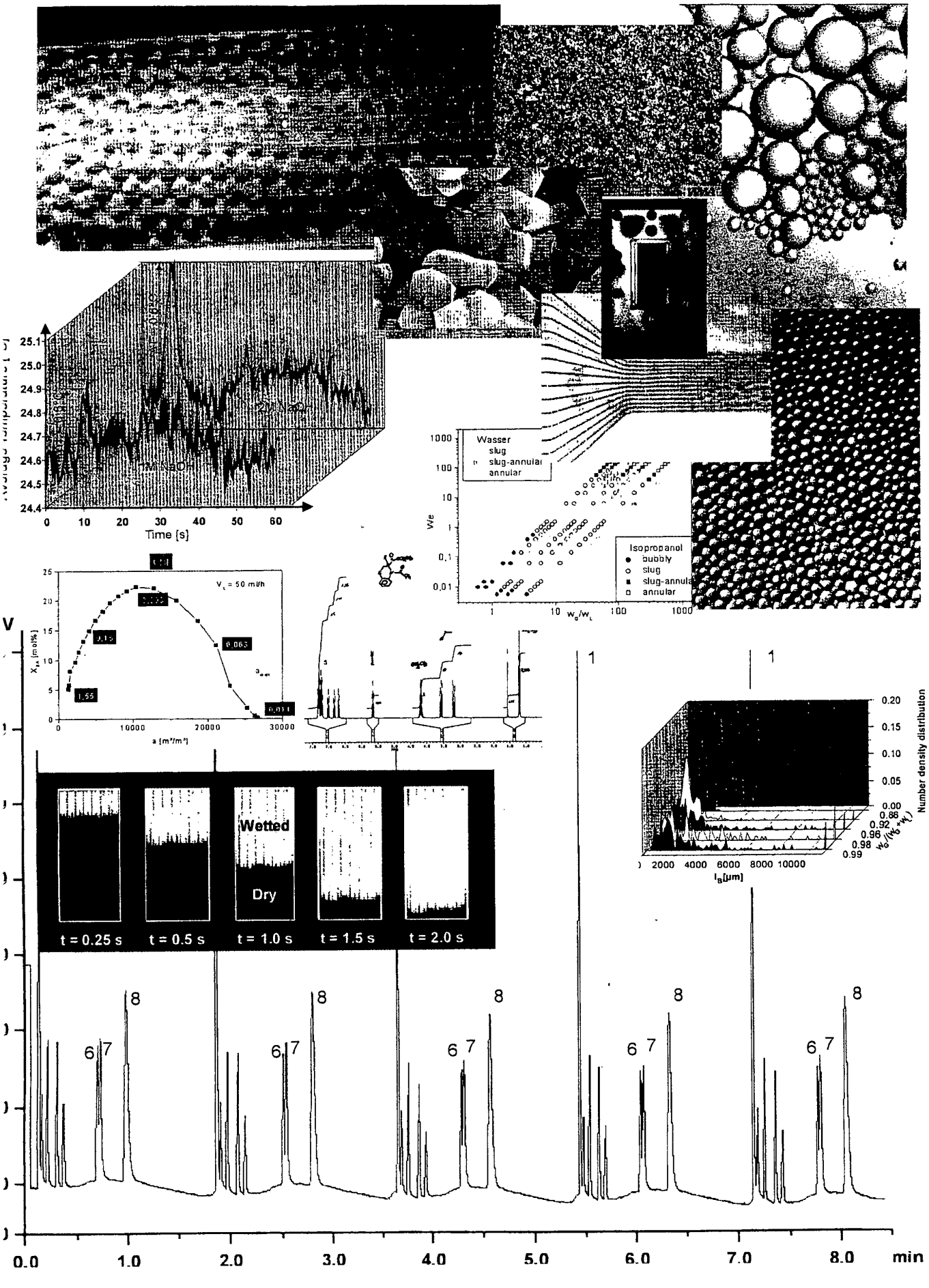
Microreactor Based Chemical Process Development



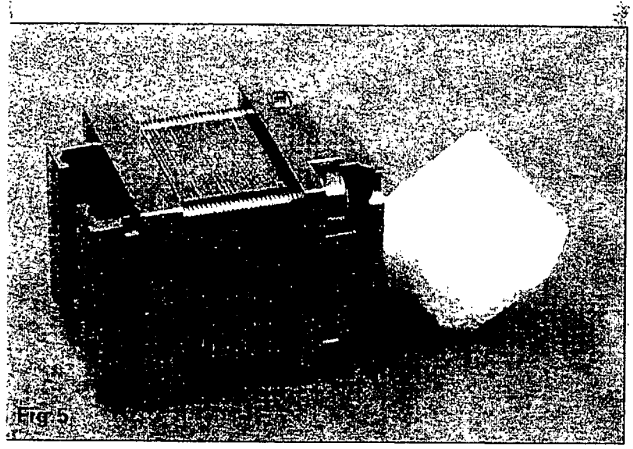
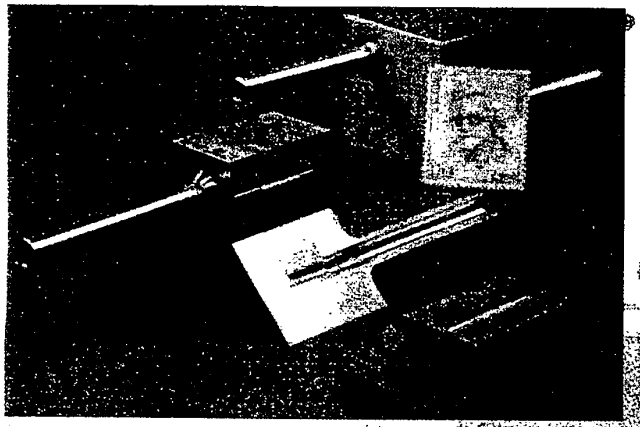


Fig.5



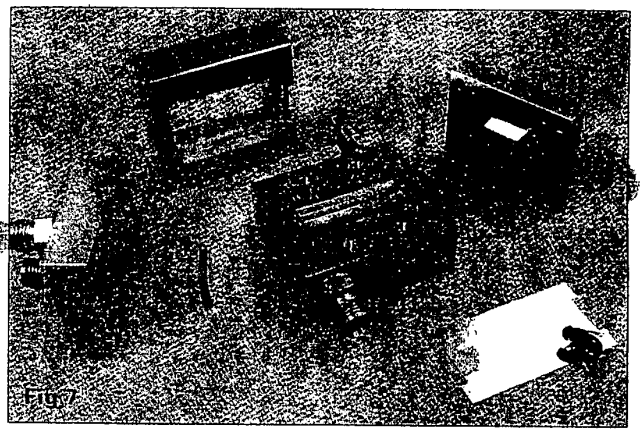
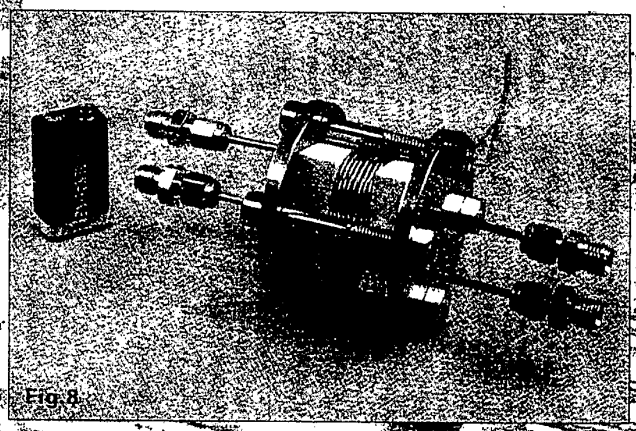


Fig.7



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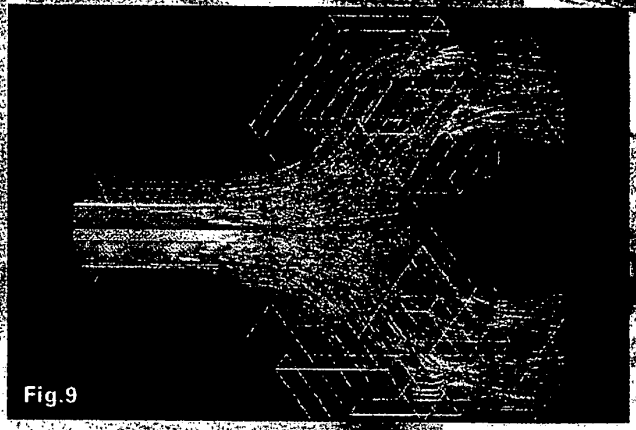
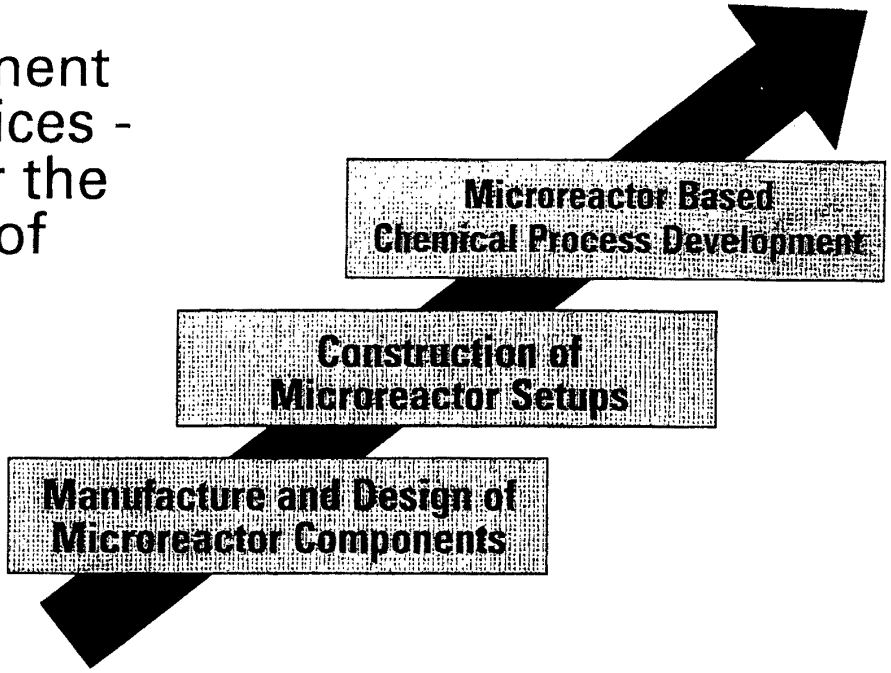


Fig.9

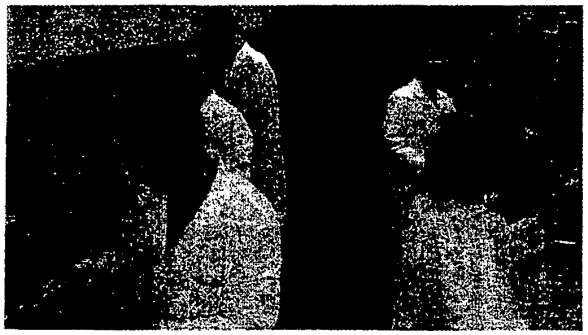
Get Your Individual Application-unique Device.

Product Development and Services - All Under the Same Roof

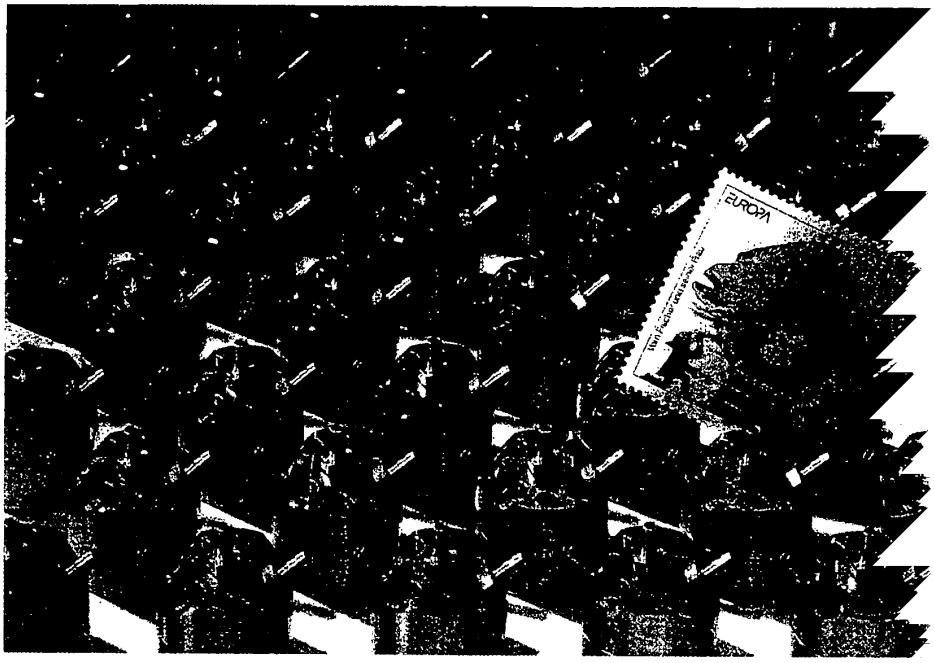


„We take care for proper product development and our strategy to meet the requirements of today's market. We are familar with our business customers and their fields of application, helping them to consolidate their lead. Besides providing excellent technology, service and logistics are a must in modern B2B marketing. Moreover, we create the intangible part of the products being developed for you – production of intellectual rights and property, access to background technology, and your personally designed technology contract committed with the scope-of-work you desire."

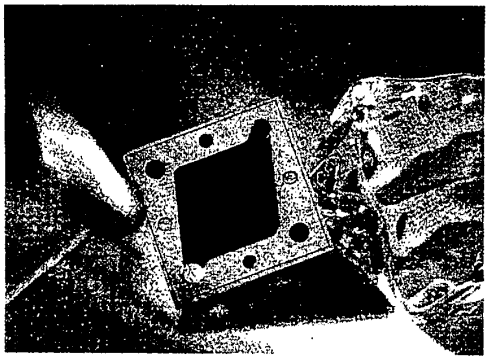
„Only the best technology platform will guarantee you to jointly seek for an innovation with hindsight and, thereby, to achieve a fast and efficient improvement of your present state of the art. Consequently, we are used to balance vision and technical feasibility. We are constantly developing new microreactors, know about appropriate fluidic peripherals and control units, and are familiar with modern simulation and CAD tools. Our engineering craft stands for service/quality leadership."



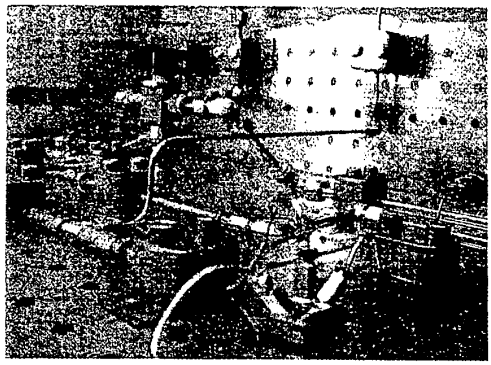
„Our daily routine regarding handling microreactors and our wealth of experience shows that real success can only be achieved by tailor-made chemical process development, at best using individually designed microreactors. We know how to construct set-ups, handle chemicals and carry out chemical processes as well as to analyse them. We run your process in our laboratory, either as a first step for your company to learn about microreactors or in the framework of a long-term cooperation with you as a key customer."



Micromixers in a serial production



Micro heat exchanger platelet



Chemical process development

References
W. Ehrfeld, V. Hessel, H. Löwe
Microreactors,
Wiley-VCH (2000)
and literature cited therein
W. Ehrfeld, V. Hessel, V. Haverkamp
Ullmann's Encyclopaedia of Chemical Industry, electronic version,
Wiley-VCH (1999)

We provide offers both for the newcomer and persons already experienced with microreaction technology. Step by step you will become familiar with our services.

Please contact:

Arrange a first contact, become more familiar with our services:

Dr. Ursula Eul
Head of Marketing Department
Tel.: 00 49-61 31-99 01 92
eul@imm-mainz.de

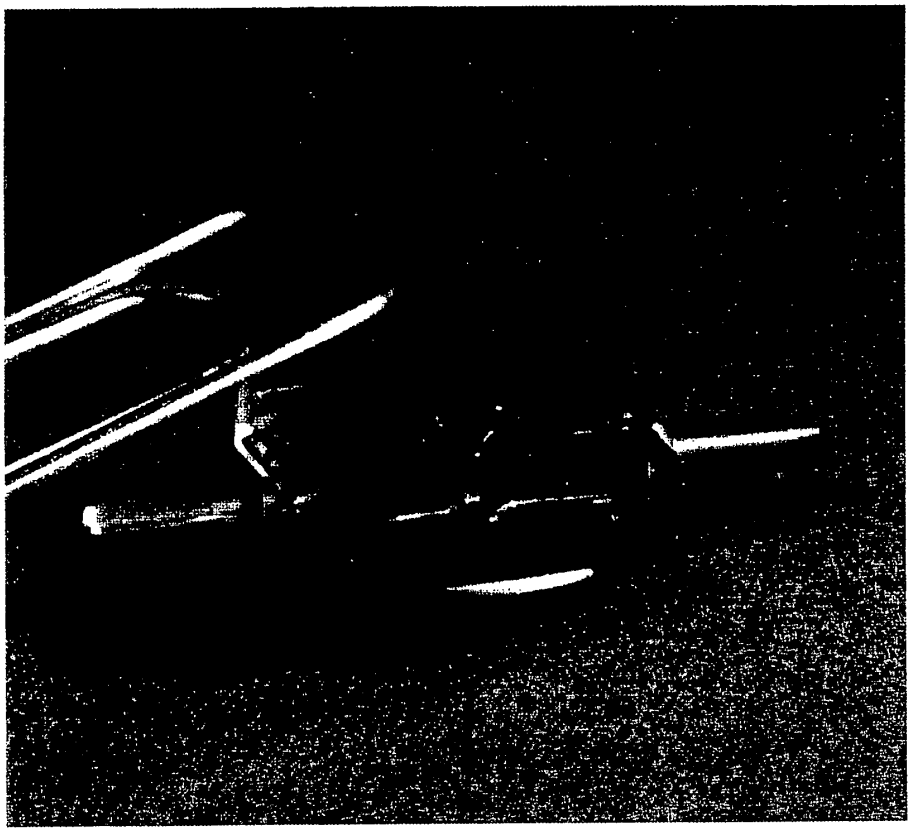
Have detailed questions or want to specify a certain application:

Dr. Volker Hessel
Head of Microreaction Technology Department
Tel.: 00 49-61 31-99 04 50
hessel@imm-mainz.de

By means of either contact route, you will meet our marketing and technology experts and get their opinion.

IMM 6.13e/0401 E

µl-Flows and Dosages



µ The IMM-Micropump

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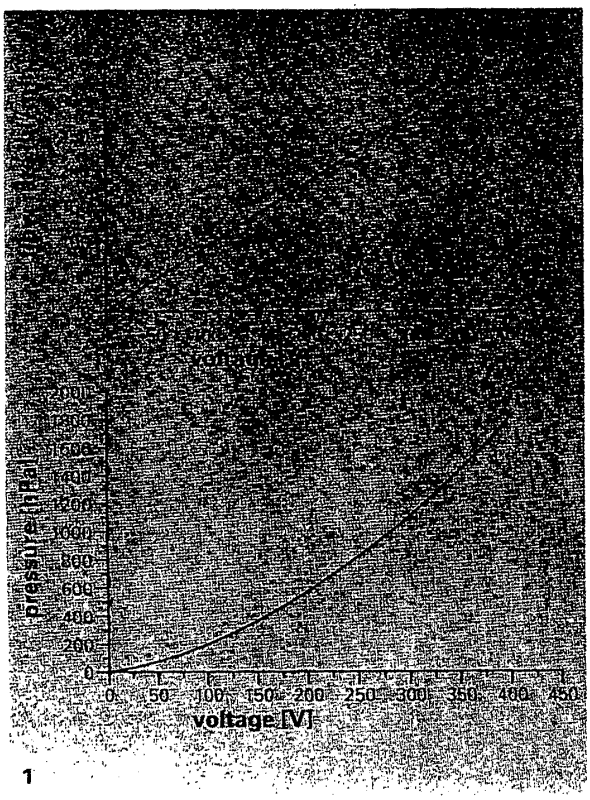


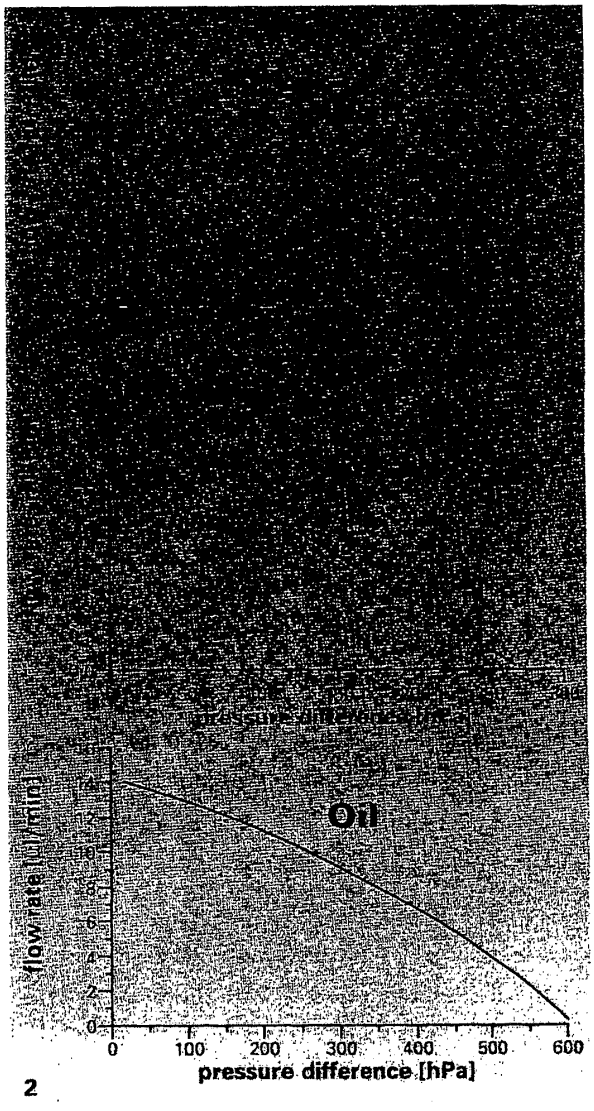
1

In the laboratory you are concerned about the **ease of handling** equipment. When it comes to pumps priming can be laborious. It isn't with the IMM-micropump*. The IMM-micropump is self-priming, since it draws a vacuum of 350 hPa(air). Flows and dosages are conveniently set using IMM's voltage modulating controller (for details on the IMM micropump control unit please refer to the inserted page). The typical correlation of flow and back pressure with voltage is depicted in diagram 1.

Laboratory Use:

- **self-priming pump**
- **biocompatible and inert materials**
- **no trouble from sealants**
- **linear pressure / flow characteristics**

Independently from minor changes to your experimental set-up you want to rely on a **steady, predictable flow**. The IMM-micropump exerts linear behaviour between its maximum flow rate of 310 µl and its maximum back pressure of 1820 hPa with water (diagram 2: water, gas and oil (Viscosity 10mm²/s, measured at ambient temperature)).



2

Reliable Operation requires stability of your pump against the media processed.
In addition, many biochemical media require non-toxic biocompatible materials. Any IMM-micropump is made from just one material. Advanced micro-connection technology enabled us to avoid glues and other sealants. The pump refered to in this document is IMM's polycarbonate-micropump. For information on other available materials please contact us.

* Patent application pending in USA

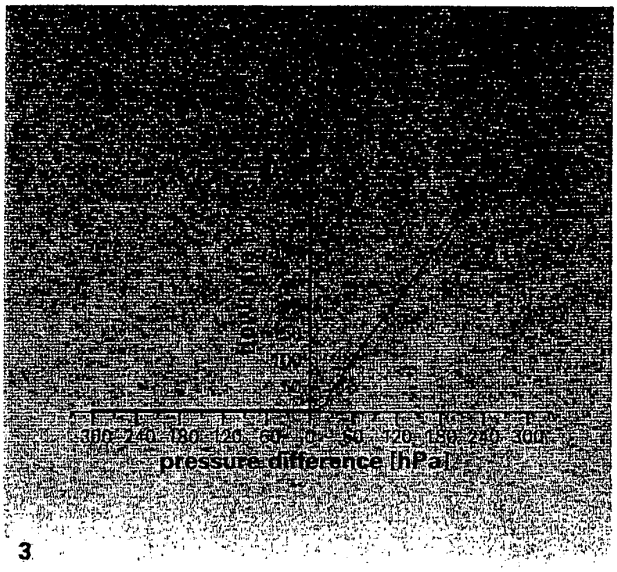
System Integration:

- **self-priming pump**
- **quick qualification**
- **high availability**
- **customisation to your requirements**

As equipment manufacturer you need an **economical solution**. The IMM-micropump yields significant space, weight and material savings due to its miniature dimensions of 12 · 12 · 3 mm.

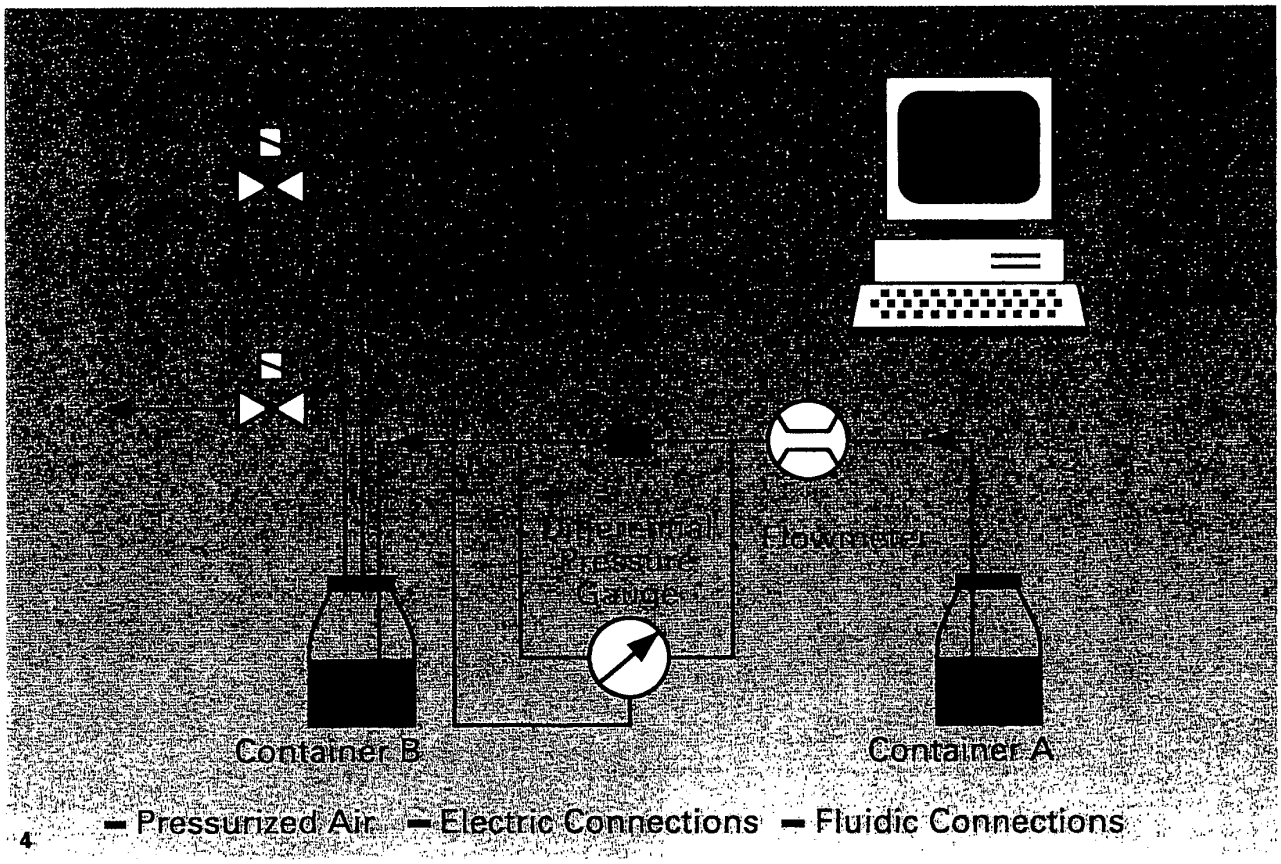
Since any IMM-micropump is comprised of just one material it is rapidly qualified in your application development process.

You are concerned about **system reliability**, notably about the avoidance of leakages. IMM's all welded micropumps take the trouble of backward leakage from your equipment, as exemplified in the valve characteristic of the built-in passive valves (diagram 3). And when it comes to **availibility**, you will appreciate your choice of IMM's injection moulded micropump for both responsiveness and throughput of the manufacturing processes utilised.



3

And of course, we are delighted to accomodate to your specific requirements through **customised designs**. Just call us at (49) 6131-990-117.



4

Pump Characteristics **	
Maximum Flow Rate: (Water)	310 µl/min Average, 250 µl/min Minimum
Maximum Flow Rate: (Gas)	3,5 ml/min Average
Maximum Back Pressure: (Water)	1820 hPa Average
Maximum Back Pressure: (Gas)	275 hPa Average
Material:	Polycarbonate
Dimensions:	12 · 12 · 3 mm
Life Cycle:	> 10 E 9 Pulses
Particle Tolerance:	< 5 µm
Voltage:	<+340/-100 V
Frequency:	0-80 Hz

** Preliminary data, subject to change (11/97)

Procedures:

Filtration of Fluids: To avoid contamination of the pump with particles filtration via conventional 5 µm syringe filters was found to be effective.

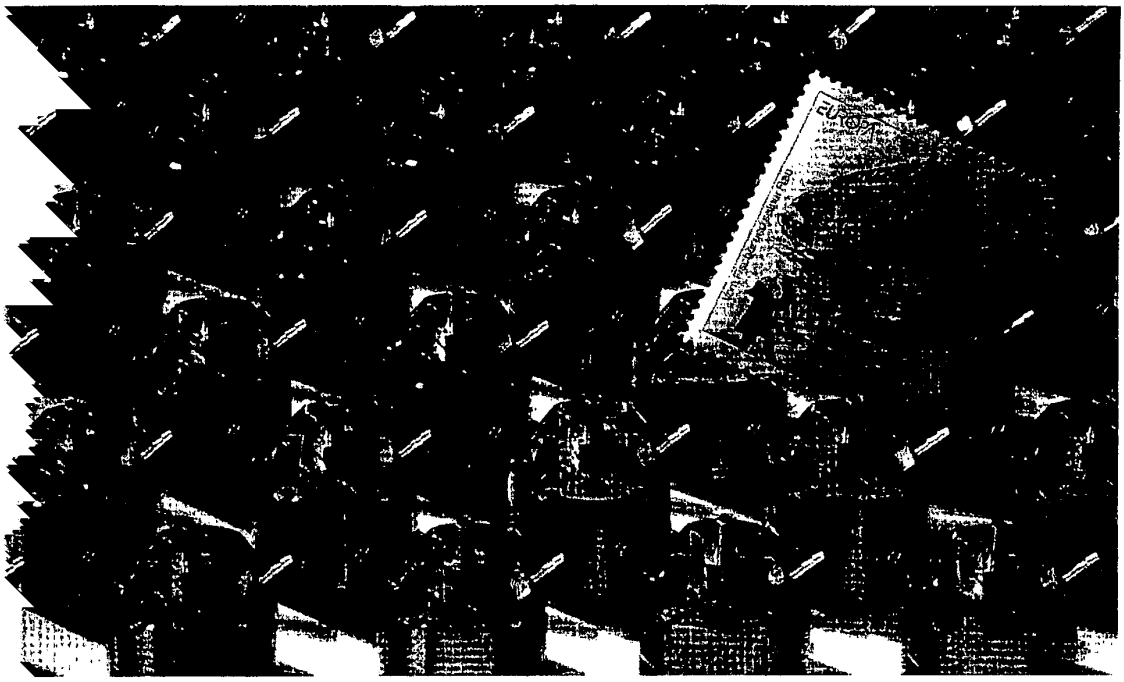
Flow measurement: IMM uses the set-up depicted in diagram 4 to monitor performance of micropumps.

Tubes: Good experience was made using silicone tubing for water and gas, and TYGON™ tubing for oils.

In the development process of IMM's micropump a wide selection from the set of methods for structuring and replicating microstructures mastered by IMM was drawn.
Such methods are LIGA, thin film technologies, plasma technology, µ-EDM, micro-injection moulding of plastics, ceramics and metals, microembossing and cutting, laserprocessing, technologies for silicon and glass structuring, microfabrication techniques.

Institut für Mikrotechnik Mainz GmbH
Carl-Zeiss-Straße 18-20
D-55129 Mainz-Hechtsheim
phone: +49 6131/990-117; fax: +49 6131/990-205
http://www.imm-mainz.de

Micromixer



The IMM micromixer, based on a static mixing principle, is suitable for a wide variety of applications in chemistry, chemical engineering, medicine and biology.

First application examples prove process intensification and process features completely different from those of state-of-the-art mixing devices.

Micromixer characteristics

- width of channels: 25 or 40 µm
- width of slit: 60 µm
- material (LIGA device): nickel, nickel on copper, silver
- material (housing): plastics, highly alloyed stainless steel or titanium
- maximum pressure: up to 30 bar
- flow rates of 10 to 1500 ml/h at pressure drops of 10 to 1000 mbar

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Mainz

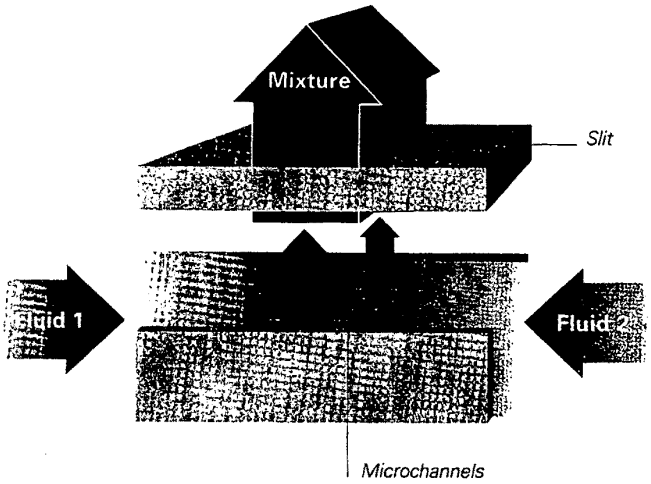


Fig. 1:
Multilamination of fluid layers

Mixing principle

By means of the LIGA process micromixing devices are fabricated which use flow multilamination with subsequent diffusional mixing.

The fluids to be mixed enter the mixing element as two counter-flows which stream into an interdigital channel configuration with corrugated walls.

The lamellar flow leaves the device through a discharge slit which is perpendicular to the direction of the feed flows and thus forces interpenetration within a defined contact zone.

Because of the small thickness of the lamellae, fast mixing takes place through diffusion. Typical values of the channel widths are e.g. 25 µm or 40 µm. The corrugated shape of the channel walls increases the contact surface of the lamellated streams and improves the mechanical stability of the separating walls.

Considering fluids with different properties, a simple adjustment of the flow and mixing conditions is obtainable by variation of the width of the corrugated interdigital channels and of the discharge slit.

The LIGA micromixing device is inserted into the bottom part of the housing. The top part of the housing contains the two inlets of the feed streams, the outlet of the mixture as well as the discharge slit.

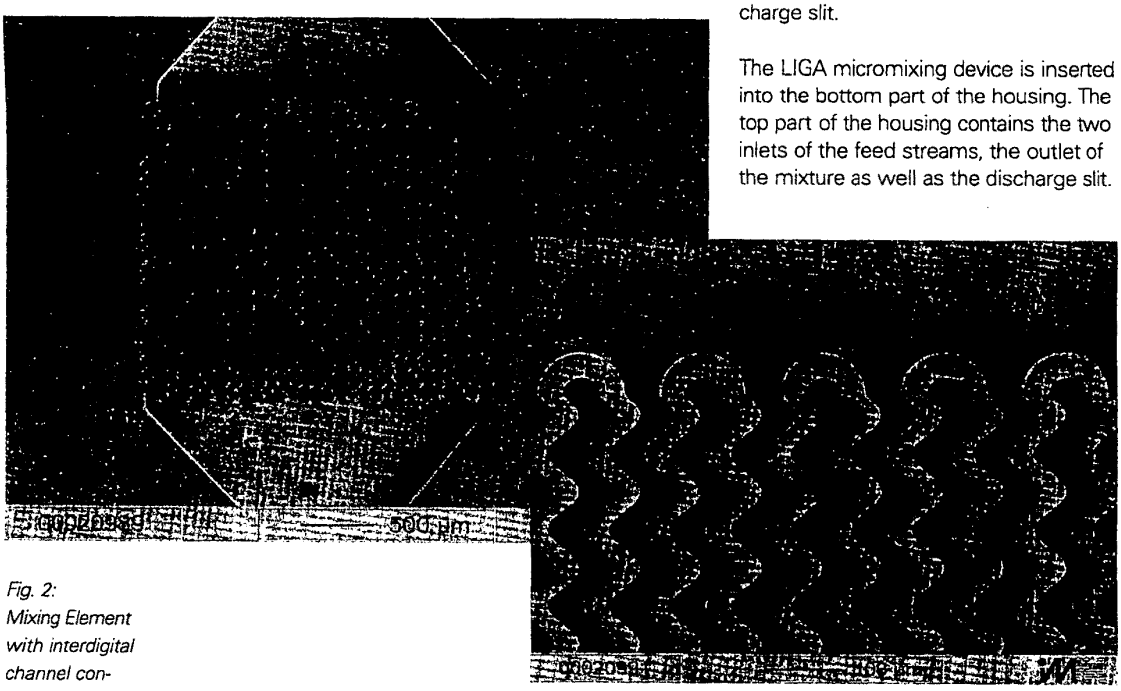


Fig. 2:
Mixing Element with interdigital channel configuration

Fig. 3:
Details of the Microchannels

The LIGA device can be made of various metals, e.g. nickel, nickel on copper or silver. The materials of the top and bottom parts of the housing are plastics, highly alloyed stainless steel or titanium.

Fig. 4:
Assembled housing with micromixing elements

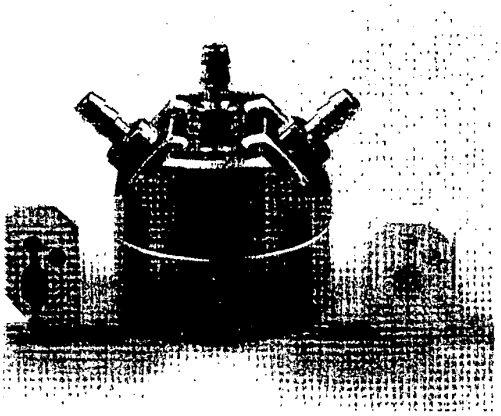
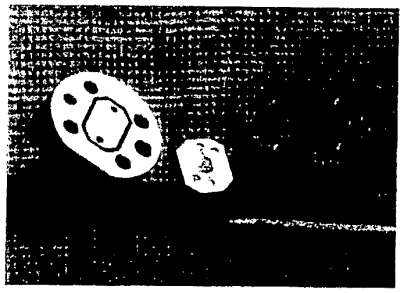


Fig 5:
Disassembled micromixer



Flow rate

The flow rate of the micromixing device ranges from 10 to 1500 ml/h at pressure losses of 10 to 1000 mbar. Higher flows are achieved by numbering-up via parallel integration of ten micromixing elements in an array configuration, the so-called micromixing array. This leads to a significant increase in throughput up to 6000 ml/h at similar pressure losses. The throughput of the micromixing device and array can be even further increased by using pumps capable of feeding fluids at pressure loss higher than 2 bar.

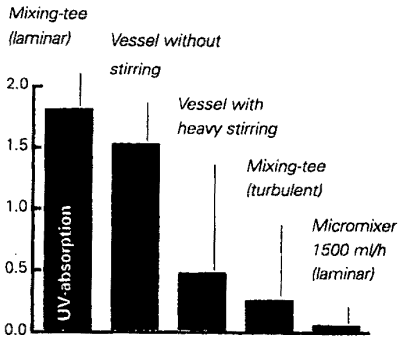


Fig 6:
UV-absorption of different mixing-devices (inverse to mixing quality)

Mixing of miscible fluids

A test reaction was developed in order to quantitatively characterise the mixing quality at different operating conditions, e.g. flow, temperature and devices with various channel and slit widths. Two different feed fluids react after mixing via two different pathways, a fast one and an ultrafast one, resulting in different colours of the reaction product. For comparison, the mixing qualities of the micromixing device, of a mixing tee with corresponding sizes of feed and withdrawal channels and of a heavily stirred mixing vessel have been determined.

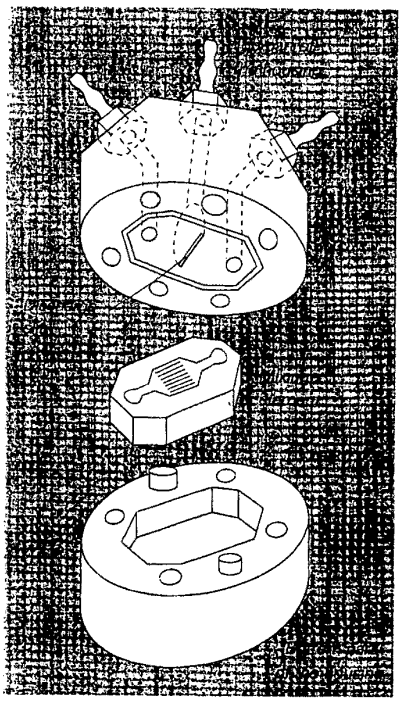
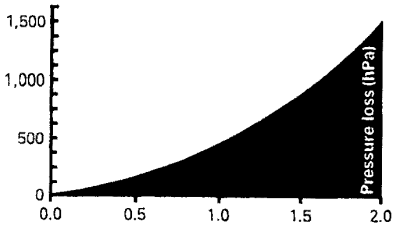


Fig. 7:
Scheme of micromixer

Fig. 8:
Pressure loss



By measuring the UV-absorption, which is inversely related to the mixing quality, it turned out that such standard devices cannot compete with the mixing quality of the LIGA micromixer.

Emulsions and gas/liquid dispersions
Emulsions as well as gas/liquid dispersions may be generated by the micromixing device. Small droplets and bubbles, respectively, are obtained although neither moving parts nor high pressure need to be applied. In a number of experiments very narrow distributions of the droplet or bubble size were found.
For a number of solvents the formation of dispersed liquid/liquid systems is achieved in some cases even without aid of standard surfactants (emulgator).

The emulsions or dispersed liquid/liquid systems made by IMM micromixers are characterised by a

- droplet size down to 800 nm
- small deviation from the average droplet size and
- formation of liquid/liquid dispersions without aid of standard emulgators (for various solvents).

Using IMM micromixers, dispersions are obtained with

- bubble size down to 100 µm and
- small deviation from average bubble size (aid of surfactants is generally necessary).

Chemical reactions on the laboratory scale
In addition, micromixers were tested by IMM and its customers to perform fast reactions (with external heating or cooling) in the liquid and gaseous phase like phase transfer reactions, metal organic synthesis or anionic polymerisation.

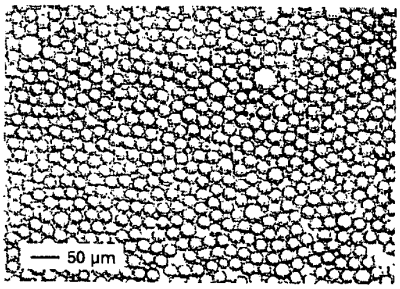


Fig. 9: Dispersed liquid/ liquid systems

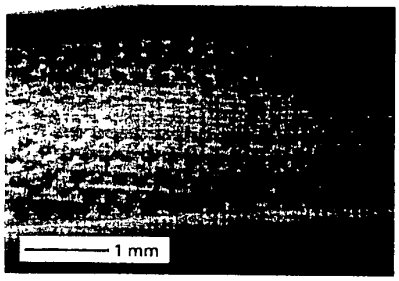


Fig. 10: Gas/liquid dispersion

IMM provides the possibility to adapt the micromixing device according to your demands. Furthermore, we are prepared to develop prototypes of other components of microreactors or complete microreaction systems in co-operation with you. Our common goal is your success on the market ●

The following publications give detailed information about the performance of the microfabrication and assembly of the micromixing system and its application:

Microfabrication
W. Ehrfeld, V. Hessel, V. Haverkamp:
Microreactors.
Ullmann's Encyclopedia of Industrial Chemistry, Sixth Ed., Wiley-VCH, Weinheim (1999) *electronic release*

Microfabrication, Assembly, Mixing of miscible liquids
W. Ehrfeld, K. Golbig, V. Hessel, H. Löwe, Th. Richter:
Characterisation of mixing in micromixers by a test reaction: Single mixing units and mixer arrays.
Ind. Eng. Chem. Res., 38(3), 1075-82 (1999)

Gas-liquid dispersions
V. Hessel, W. Ehrfeld, K Golbig, V. Haverkamp, H. Löwe, Th. Richter:
Gas / liquid dispersion processes in micromixers: The hexagon flow.
Process Miniaturisation: 2nd International Conference on Microreaction Technology, March 9-12 1998, New Orleans, LA, USA.

Topical Conference Preprints, The American Institute of Chemical Engineers (1998), pp. 259 - 266

Emulsions
V. Haverkamp, W. Ehrfeld, K. Gebauer, V. Hessel, H. Löwe, T. Richter, C. Wille:
The potential of micromixers for contacting of disperse liquid phases.
Fresenius' Journal of Analytical Chemistry, (1999) 364: 617-624

Your contact at IMM:
Public Relations
Phone: +49 6131 / 990-117
Fax: +49 6131 / 990-205
Institut für Mikrotechnik Mainz GmbH
Carl-Zeiss-Straße 18-20
55129 Mainz-Hechtsheim
http://www.imm-mainz.de

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...Optical Backplane based on integrated-optical Star Coupler for Computer Applications •

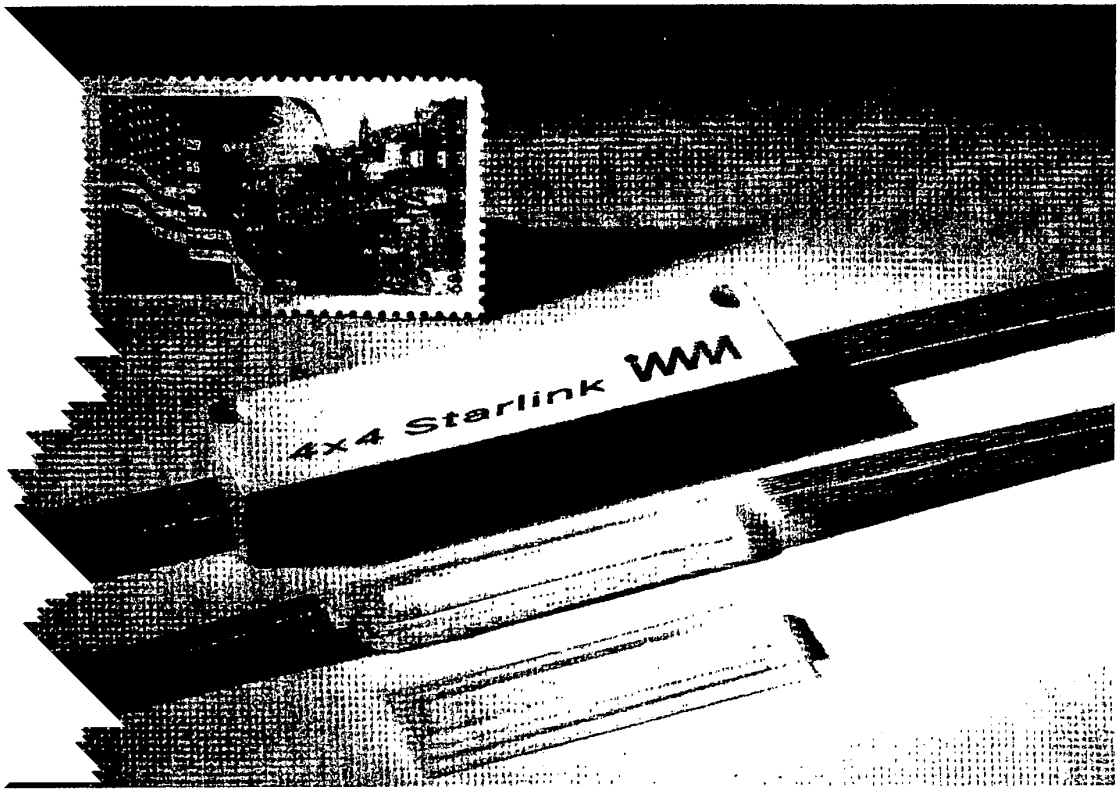

Fig. 1:
Integrated-optical star coupler based on polymer materials for interconnecting processor boards of high-speed computers: Fully assembled star coupler, 60mm x 9mm x 16mm (top), pigtailed component (middle), microstructured moulded part (bottom).

Specifications

The star couplers are pigtailed with 125 µm / 62,5 µm GRIN multimode fibers. Using a wavelength of 830 nm the following performance is obtained: Average excess loss: 2,4 to 2,9 dB. Uniformity of the splitting ratio: better than 2 dB. Fig. 1 shows the fully assembled and packaged component.

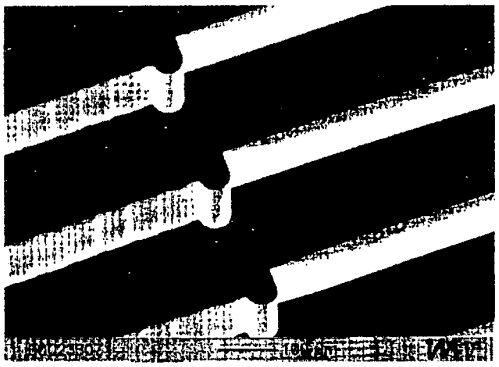

Fig. 2:
SEM-image of the mould insert detail used for the replication of highly precise fiber-chip-coupling schemes allowing passive pigtailing.

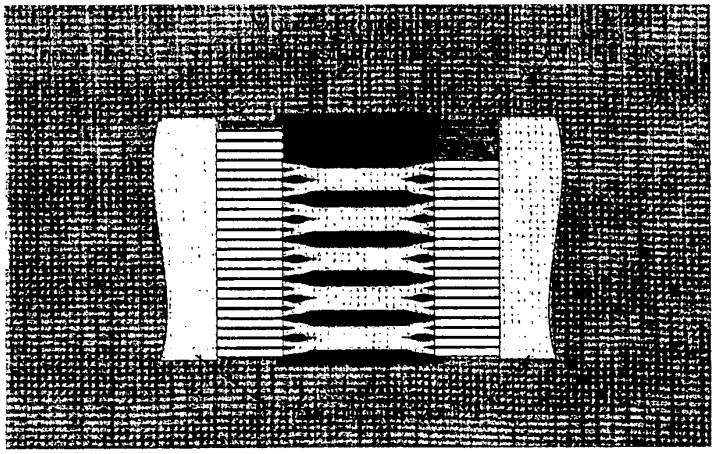

Fig. 3:
Compact arrangement of six 4x4 star couplers on a single moulded component (40mm x 9mm).

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Optical communication technology

Our so called "information society" produces a continuously increasing demand for higher data storage capacities, higher data rates, and faster data processing. HDTV and Internet are only two examples which push these demands forward. Based on the immense bandwidth and the absence of EMI effects optical fibers are increasingly used for optical data transmission over long distances (telecom) and in local area networks or even inside computers (datacom). These applications long for very precise, cost-efficient and, therefore, mass-producible passive components, e.g. splitters and star couplers.

Computer backplane with integrated-optical star coupler

With multi-processor high-speed computers, the limits of copper wiring, basis of traditional interconnection technology, have already been reached. The most difficult bottleneck in these cases is the backplane used for data interchange between the various processor boards.
In the near future such computers will be equipped with optical bus systems and soon PCs will follow this development, too. For a bus system passive components are necessary that split, route, and combine optical signals.
One possible backplane architecture uses the "star" system for board-to-board communication: A signal sent by one "talker" has to be split very uniformly to all the "listeners".
Core component of this kind of optical backplane is a star coupler, like the one recently developed at IMM. Fig. 3 schematically shows this component, with six 4x4 star coupler elements on it, built to manage the interaction of four processor boards via six optical channels. All star coupler units are fabricated in parallel on the same substrate, resulting in a high packing density.

Fabrication using LIGA technique

The realization of these components is only possible with means of a high-precision technology able to yield a cost-efficient product. The LIGA technique can fullfil these demands and allows high-volume production. Using microfabricated replication tools the high accuracy achieved in an X-ray lithography process can be transferred to a plastic product. To realize multimode waveguides, for example, rectangular grooves with a cross-section of 50 µm x 50 µm are moulded into a polymer substrate. Filled with a UV-curable resin of a higher refractive index these grooves act as optical waveguides.
By combining LIGA with high-end precision milling methods moulding tools have been realized that show three different height levels. In addition to the waveguide grooves, deep fiber alignment trenches can be fabricated during the same moulding step (Fig. 4). Fiber grooves and waveguides are exactly aligned to each other by the lithography process. Therefore, easy passive pigtailing is possible.
Fully assembled star couplers are shown in Fig. 5 ●

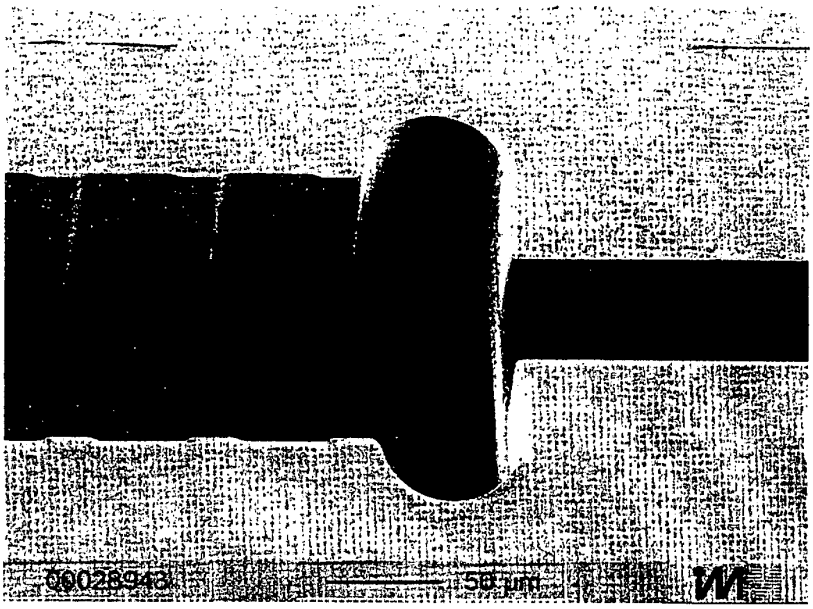


Fig. 4:
SEM-image of the moulded fiber-waveguide-coupling region. The preformed waveguide shows a cross section of 50 µm x 50 µm.

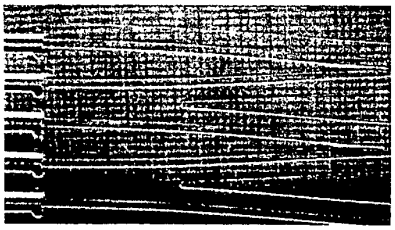


Fig. 5:
Fully assembled star coupler (detail of microscope image) with "filled" waveguides and fixed fiber ends.

Your contact at IMM:
Public relations
Phone: +49 6131/990-117
Fax: +49 6131/990-205
Institut für Mikrotechnik Mainz GmbH
Carl-Zeiss-Strasse 18-20
D-55129 Mainz
http://www.imm-mainz.de

Surface treatment in low temperature plasmas

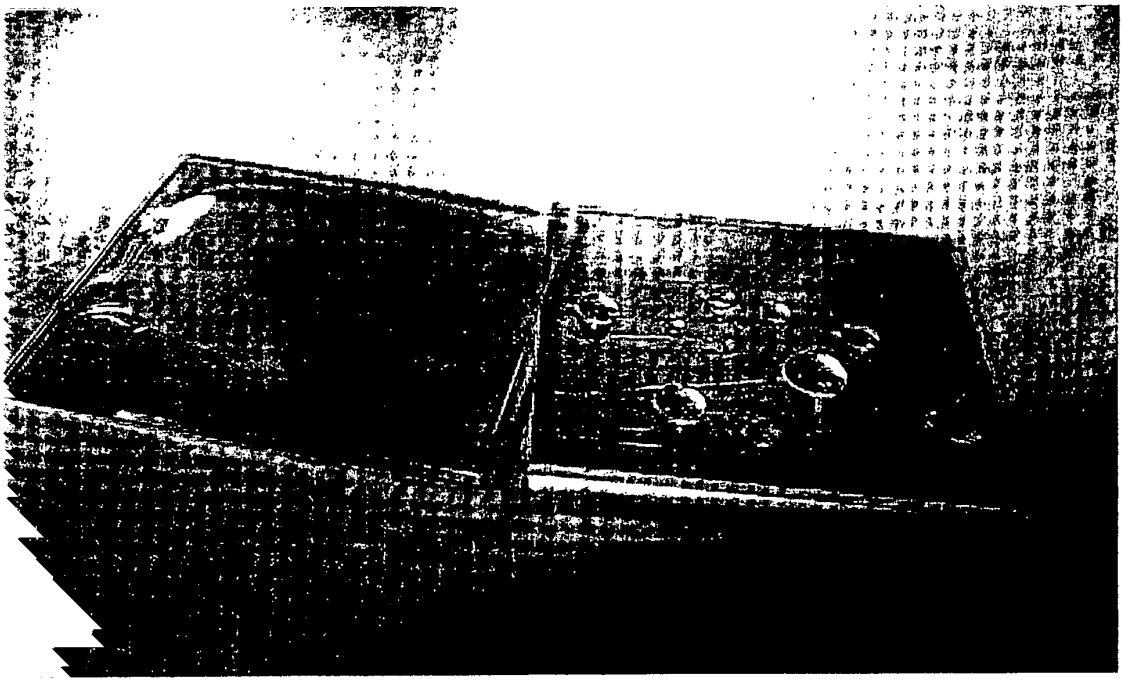
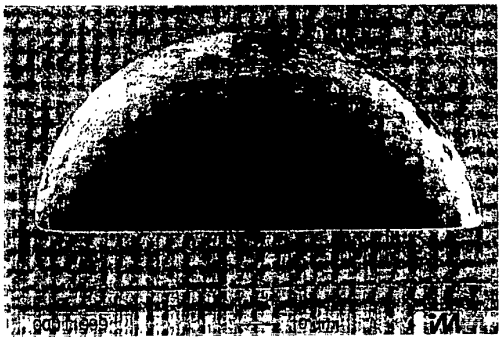


Fig. 1
Wettability of polycarbonate; on the left, plasma-treated, on the right, untreated

Work areas of IMM:

- Microtechnology and sensor systems
- Textile refinement and finishing
- Membrane separation technology
- Medical technology and pharmaceutical diagnostics
- Mechanical engineering



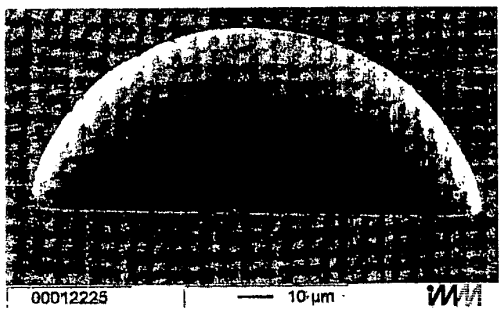


Fig. 2
LIGA mould insert before and after plasma cleaning

Applications:

- Plasma cleaning of metallic workpieces
- Hydrophilic and hydrophobic coatings
- Quartz-like, optically perfect transparent hard protective coatings
- Diffusion and migration barriers, anti-corrosion coatings
- Diamond-like, chemically and mechanically extremely robust layers on plastics, metals and ceramics
- Functional layers for the specific coupling of biomolecules for medical and diagnostic purposes

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Low temperature plasma processes open up a wide spectrum of possibilities for changing the physical and chemical properties of material surfaces and adapting them to the intended use. Since the processes run at close to room temperature, they are especially suitable for coating and surface modification of polymers and other materials which cannot be subjected to much thermal stress.
The range of applications of low temperature plasma processes extends now from microsystem technology to car manufacture. Among the most well-known applications in industrial fabrication are the hard coating given to plastic lenses for glasses and anti-corrosion layers on headlamp reflectors for road vehicles.
The processes may be divided into three categories:

Plasma cleaning processes
enable an environmentally acceptable, solvent-free removal of organic and anorganic contaminations, for example, to ceramic, metal and plastic surfaces.

With **plasma-induced surface modification**, a new chemical surface structure can be given to plastic materials by additional cross-linking or the insertion of new functional groups.
This enables the surface properties of materials, e.g. their adhesion behaviour, wettability, permeability, biocompatibility or chemical and physical durability, to be adjusted to the requirements of a specific application.

Plasma polymerization
enables the deposition of thin organic or anorganic coatings on workpieces which are already closed and free of defects from thicknesses of only a few millionths of a millimetre.
The spectrum of available processes ranges from organic polymer-like layers with hydrophilic or hydrophobic characteristics and hard quartz or diamond-like anti-abrasion and anti-corrosion layers to special functional layers, suitable for use, for example, for the binding of biomolecules on diagnostic devices.

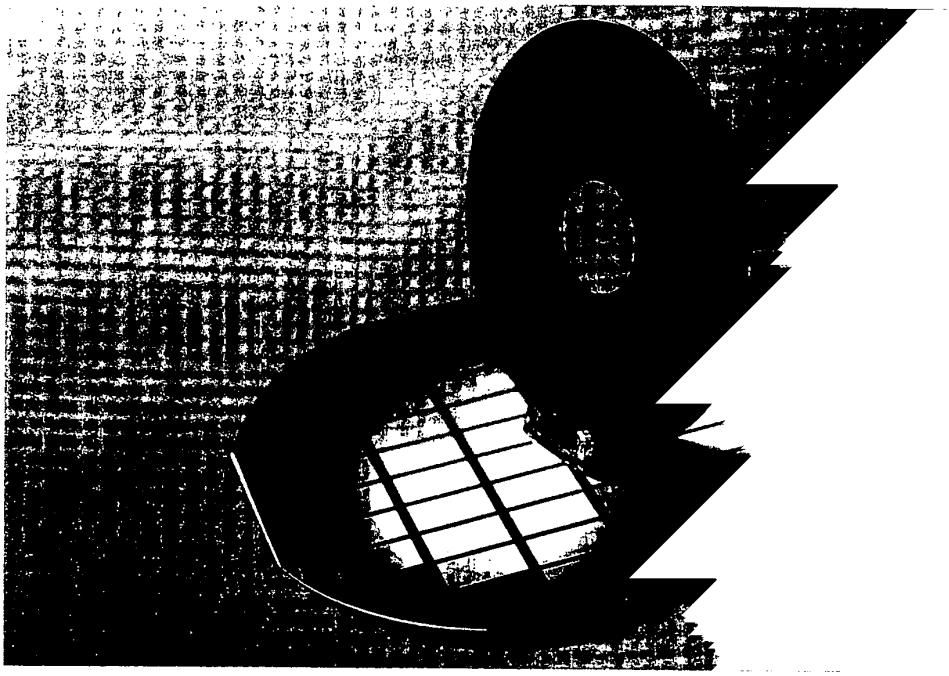


Fig. 3
Plasma-coated silicon wafer with sensor structures and magnetic hard disk with protective coating

Environmental compatability
Plasma processes are environmentally friendly. They display high efficiency with low energy and media consumption. Since the processes run at underpressure in a closed reaction chamber, only small quantities of process residues are generated, which can be disposed easily. Plasma-treated products can be recycled straightforwardly following separation into their constituent materials.

IMM has a range of high-frequency and microwave plasma equipment with which workpieces of various materials and shapes can be cleaned, modified or coated. Mass spectrometry and optical spectroscopy are used to monitor the processes. For determining surface and layer properties, surface profilometers, contact angle goniometer, FTIR-spectrometer, ellipsometer and both atomic force (AFM) and secondary emission (SEM) microscope are available. ●

Do you have fabricating or applications-related problems with the surface properties of your product? We should be pleased to develop application specific plasma processes to improve the quality of your product and work out concepts for the integration of the processes into production.

Your contact at IMM:
Public relations
Phone: +49 6131/990-117
Fax: +49 6131/990-205
Institut für Mikrotechnik Mainz GmbH
Carl-Zeiss-Strasse 18-20
D-55129 Mainz
http://www.imm-mainz.de

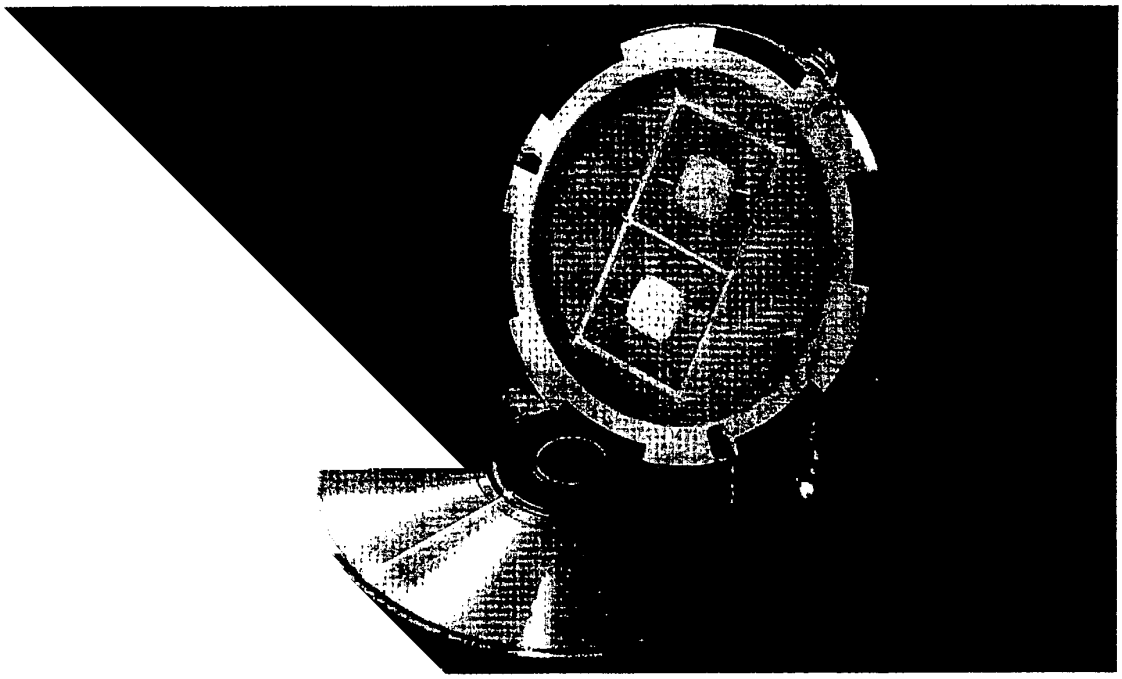


Abb. 1:
Au/Si_3N_4 -Maske für Röntgentiefenlithographie, Strukturhöhe 2 µm, Membrandicke 4 µm.

Materialien:
- Gold, Silber
- Nickel, Nickel/Kobalt, Nickel/Eisen
- Nickel/Wolfram, Nickel/Phosphor, Kupfer
- Kompositschichten (Metall+Hartstoffpartikel)

Fertigungsausrüstung:
- Reinraumbedingungen:
  12 Mikrogalvanikanlagen
- Übliche Laborbedingungen:
  4 Anlagen

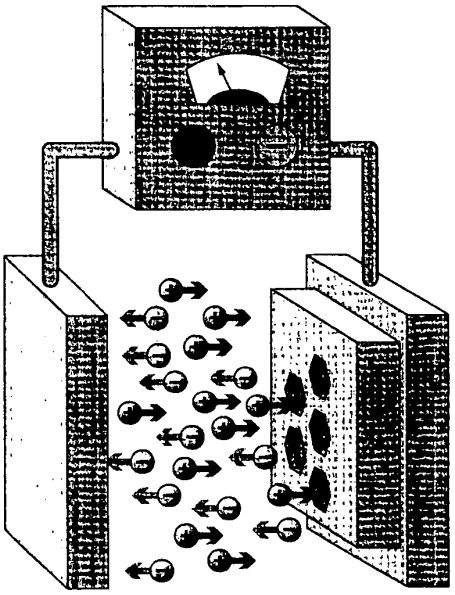


Abb. 2:
Schema der kathodischen Metallabscheidung in Mikroformen (Mikrogalvanoformung)

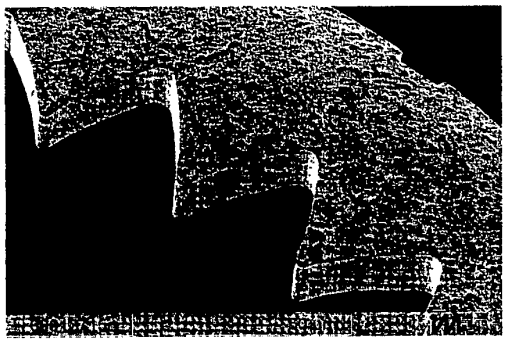


Abb. 3:
REM-Aufnahme eines Mikrozahnkranzes mit einer Mikrohärte von 600 HV aus einer kathodisch abgeschiedenen Nickel/Wolfram-Legierung.

Institut für
Mikrotechnik
Mainz

Der LIGA-Prozeß

Die Galvanoformung ist einer der wichtigsten Prozeßschritte in der LIGA-Technologie (Lithographie, Galvanoformung, Abformung) zur Herstellung von Mikrostrukturen oder Formeinsätzen für die Massenfertigung mikromechanischer, mikrooptischer oder mikrofluidischer Elemente mit extremer Präzision und hohen Aspektverhältnissen. Präzision ist eine zentrale Anforderung an die Mikrogalvanoformung, die in Kombination mit geeigneten Photoresists an mehreren Stellen im LIGA-Prozeß realisiert wird. So wird die grundsätzliche Präzision des Prozesses schon bei der Herstellung der Röntgenmasken durch Übertragung des CAD-Maskenlayouts auf die Tiefenlithographie festgelegt. Die nachfolgende Galvanoformung von Gold (Abb. 1) reproduziert diese Präzision vollkommen. Fehlerhafte Masken würden zu gravierenden Strukturdefekten führen. Die Mikrogalvanoformung ermöglicht selbst mehrfaches Umkopieren von Masken ohne Beeinträchtigung der Qualität.

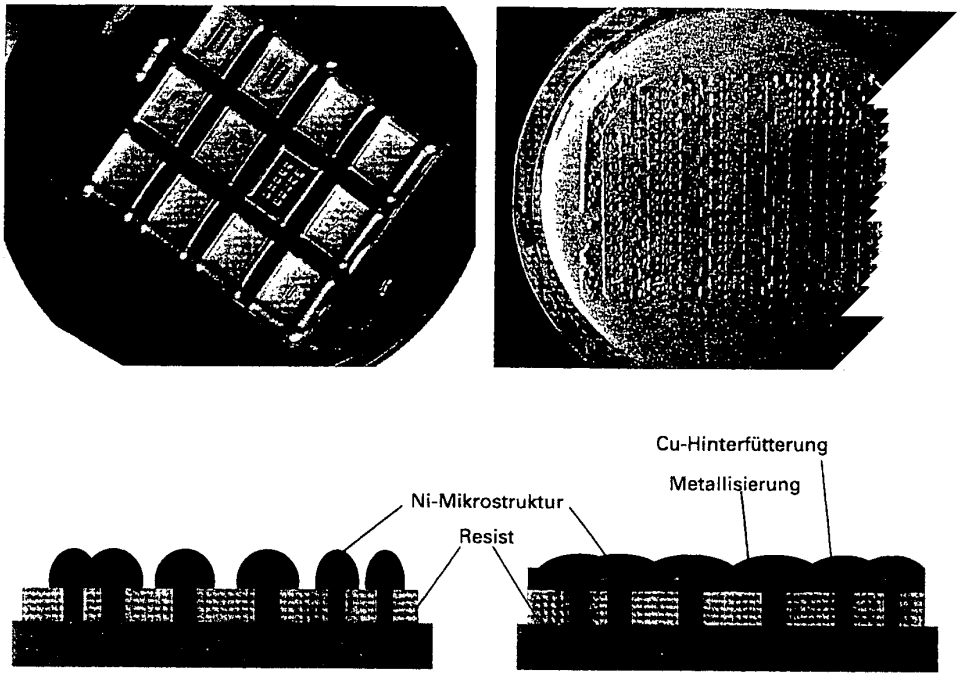


Abb. 4:
Herstellung von Ni-Formeinsätzen durch galvanische Hinterfütterung mit Kupfer.

Eigenschaften

Über die Wahl der abzuscheidenden Metalle kann auf die resultierenden Werkstoffeigenschaften entscheidend Einfluß genommen werden. So erfordert die Verwendung als Abformwerkzeug für Spritzguß- oder Heißprägeverfahren eine hohe Resistenz gegen Abnutzung, um die geforderte Präzision bei langen Standzeiten einhalten zu können.

Der Prozeß der Galvanoformung

Geeignete Resistmaterialien werden auf ein elektrisch leitfähiges Substrat in vorbestimmter Schichtdicke (1 µm bis >1 mm) aufgetragen und röntgenlithographisch mikrostrukturiert. Durch Galvanoformung (kathodische Metallabscheidung, Abb. 2) werden die Resiststrukturen mit Metall aufgefüllt. Da die kathodische Metallabscheidung ein isotroper Prozeß ist, lassen sich prinzipiell beliebige Formen realisieren. Daneben existieren Verfahren zur galvanischen Beschichtung nichtleitender Substrate wie Mikrostrukturen aus Kunststoff.

Absorberstrukturen werden durch Gold-Galvanoformung auf röntgentransparenten Substraten wie Beryllium in einem Goldsulfitelektrolyten hergestellt.

Zur Herstellung von Formeinsätzen wurde bisher vornehmlich Reinnickel aus Nickelsulfamatelektrolyten verwendet (Abb. 4). Zur Steigerung der Verschleißfestigkeit können statt dessen auch Hartlegierungen wie Nickel/Wolfram oder Kobalt/Wolfram aus schwach alkalischen Sulfatelektrolyten kathodisch abgeschieden werden. Bei einem Wolframgehalt um 10% weisen die erzeugten Schichten eine Mikrohärte von 600HV auf (Abb. 3). Diese läßt sich noch durch Diffusionsglühen bei 650°C auf 800HV erhöhen. Durch Optimierung der Elektrolytzusammensetzung und der Arbeitsbedingungen werden innere Spannungen minimiert, was den Aufbau von Mikrostrukturen mit Höhen von mehr als 1 mm bei hoher thermischer und mechanischer Belastbarkeit erlaubt.

Die Abscheidung von Nickel/Eisen-Legierungen (Ni/Fe) ermöglicht die Herstellung von Mikrostrukturen mit spezifischen magnetischen Eigenschaften. Abhängig von den Prozeßparametern kann der Eisengehalt in der Schicht eingestellt werden. Ni/Fe-Legierungen niedrigen Eisengehaltes können als Material für Federn dienen, während Legierungen mit einem Eisengehalt von bis zu 50% interessant für elektromagnetische Bauteile sind.

Harte Legierungen aus Nickel/Phosphor (NiP) können sowohl außenstromlos (chemisch reduktiv) als auch elektrochemisch abgeschieden werden.

Weitere Modifizierung der Materialeigenschaften kann durch den Einbau von Diamant-, Bornitrid-, Aluminiumoxid- oder Siliciumcarbid-Nanopartikeln (Kompositschichten) erfolgen. Hierdurch werden besonders die Härte und Verschleißfestigkeit erhöht ●

Ihr Ansprechpartner im IMM:
Öffentlichkeitsarbeit
Tel: 0 61 31 / 990-117
Fax:0 61 31 / 990-205
Institut für Mikrotechnik Mainz GmbH
Carl-Zeiss-Straße 18-20
55129 Mainz-Hechtsheim
http://www.imm-mainz.de

IMM 6 04 / 0797 D. Bailey & Gerasch, Darmstadt

Advanced Silicon Etching

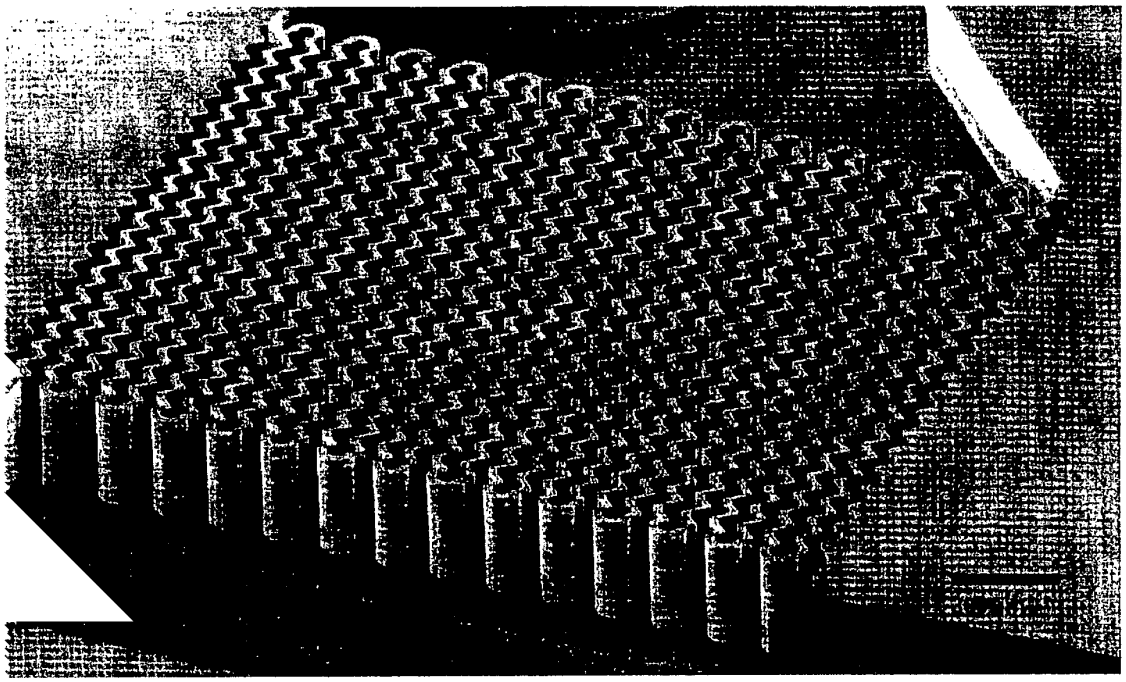


Fig. 1:
Static mixer made from silicon for chemically aggressive media (thermally oxidized, depth 300 µm, aspect ratio 12). Application example: chemical and biochemical reaction technology

ADVANCED SILICON ETCHING – ASE™
A three-dimensional fabrication technique for silicon microstructures:

By using a special plasma etching technique it is possible to fabricate silicon structures with depths of several hundred micrometers with vertical walls and high aspect ratio. This technique is compatible with almost all semiconductor and thin film processes and with common bonding techniques.

Fig. 2:
Three-dimensional silicon cutting tool. The blades are fabricated by defined undercut

Application areas structures made by ASE:

- Medical applications
- Information technology
- Environmental control
- Chemical and biochemical process
- Engineering and analysis
- Space technology

Examples of components:

Micromechanics:

- bridges
- cantilevers
- springs
- membranes
- comb structures
- resonators
- electrostatic actuators
- piezoresistive sensors (accelleration, pressure, angular rate,...)

Microfluidics:

- valves
- flow channels
- mixers
- nozzles
- fluidic amplifiers and switches
- heat exchangers
- microreactors

Microoptics:

- alignment structures
- fiber guidance
- micromirrors
- lens holders
- microoptical benches

Deep silicon etching

Anisotropic wet etching techniques for single crystalline silicon substrates are known for several decades. These etching techniques use the dependence of the etching rate of the crystal orientation. Three-dimensional silicon structures (e.g. V-grooves, flow channels, 45°-mirrors, etc.) are defined in a wet chemical etching process using a structured masking layer. These etching techniques are limited by the orientation of the crystal planes in the substrate and lead to restrictions with respect to the achievable structure geometries.
Advanced Silicon Etching (ASE™) is a new deep etching technique using a high density flourine plasma, which allows the fabrication of silicon structures with almost vertical walls independent of the orientation of the crystal planes.

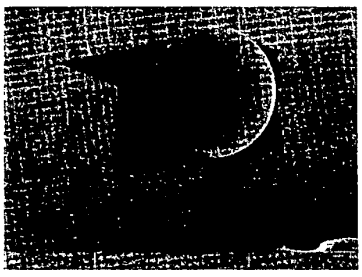


Fig. 4:
Silicon microvalve with a ruby ball. Application example: miniaturized pneumatic and fluidic systems within µl-range

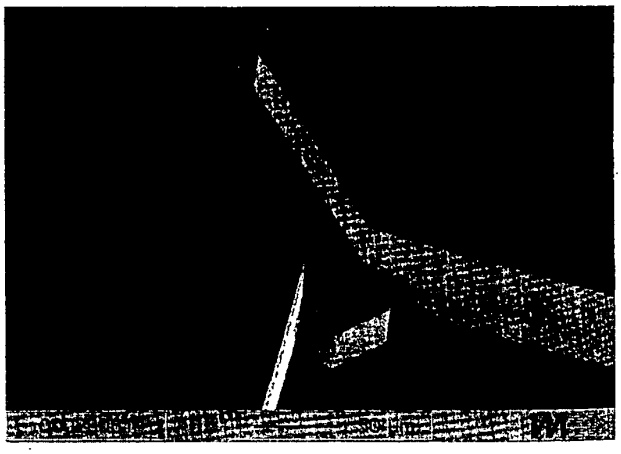


Fig. 5:
Anisotropically etched, step like silicon structure with dimensions in the µm range, width of the channel: 40µm, depth of the structure:
Level 1: 40µm
Level 2: 80µm

Advanced Silicon Etching ASE™

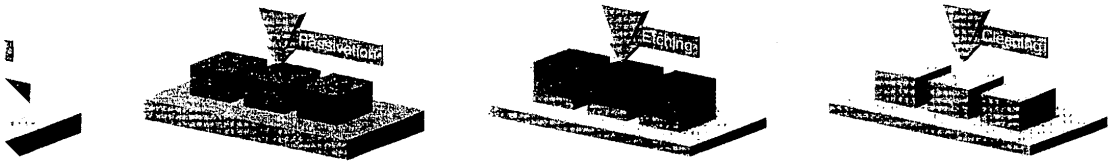


Fig. 3: Scheme of the ASE-Process

The Advanced Silicon Etching process - ASE™

High density fluorine plasmas are used to etch silicon isotropically with high etching rates. To achieve almost vertical sidewalls in the range of 80°- 90° the addition of monomer gases induces the deposition of sidewall polymers, which diminish or stop etching of the sidewalls (side wall passivation). These competing processes can be separated in time. During the so-called Advanced Silicon Etching- process (ASE™) an alternating switching between a bias enhanced plasma etching process and a conformal deposition of a teflon like polymer leads to a deep etch process with a high etching rate (several µm/min) and a high anisotropy (Fig.3). Vertical sidewalls with a sidewall angle of 90°(±1°) are possible as well as an undercut with a maximum angle of 7° relative to the vertical axis.

Combinations with other technologies

This deep etch process for silicon structures opens up plenty of combination possibilities with other technologies:

- integration of thin film devices such as sensors and electronic devices
- etch stop and sacrificial layer techniques (e.g. using SOI-wafers)
- steplike silicon structures as mold inserts for hot embossing and injection molding
- anodic bonding of structured silicon wafers with glass
- fabrication of free-standing thin film sensor and actuator devices such as membranes, bridges and cantilevers
- high precision shadow masks for coating processes
- integration of CMOS devices

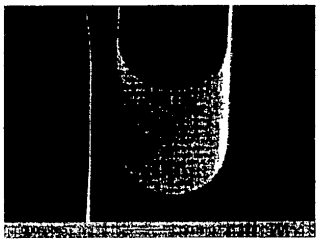


Fig. 6 (top): Three-dimensional silicon structure with defined undercut. Application example: minimal invasive surgery

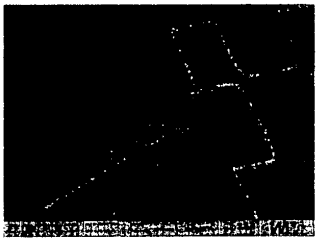


Fig. 7 (bottom): Deep, narrow channels within the µm-Range. Application example: densification of particles in fluids

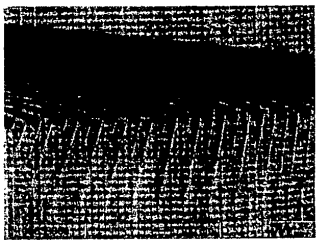


Fig. 8 (top): Undercut silicon structure for fabrication of silicon tips (e.g. for AFM), length of the tip: 160µm. Application example: surface analysis

Fig. 9 (bottom): Flow channels with typical depths of 50µm to 300µm. Application examples: microreaction technology, capillary electrophoresis, gas chromatography

The described technology allows to structurize silicon three-dimensionally from the µm range up to millimeters. Furthermore it is possible to combine the fabrication of silicon microstructures with many other technologies. This allows fabrication of complex multifunctional microstructures on wafer scale very cost effectively and to apply them in interdisciplinary fields such as environmental control, medical technology, process engineering and information technology.

Fig. 10:
Micro vessels with SiO_2-membranes. Application examples: microreaction technology, combinatorial chemistry

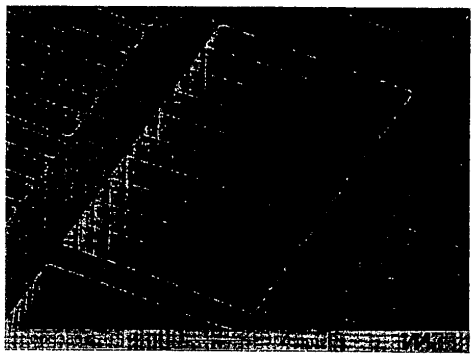


Fig. 11:
Micro bridges across an opening. Application example: mass flow sensors

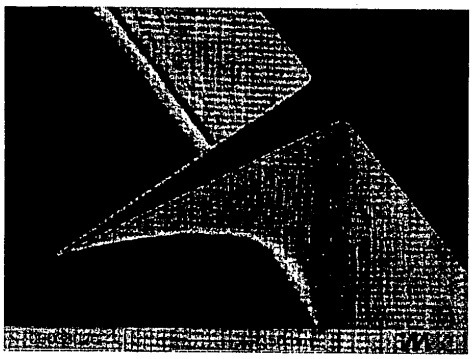


Fig. 12:
Silicon needles as support structure for fluidic components and sensors. Application examples: micro dosing, flow and viscosity measurements

Material properties of Silicon

- Youngs modulus 130-188 GPa, crystal orientation dependent
- fracture strength < 6 GPa
- no plastic deformations below 400°C
- high heat conduction (146,5 W/mK)
- high temperature resistance (S_m=1417°C)
- easy fabrication of insulating barriers and etch stops by thermal oxidation
- high chemical resistance
- semiconductor with indirect band gap of 1,106 eV
- high piezoresistive effect

Your contact at IMM:
Public relations
Phone: +49 6131/990-117
Fax: +49 6131/990-205
Institut für Mikrotechnik Mainz GmbH
Carl-Zeiss-Strasse 18-20
D-55129 Mainz
http://www.imm-mainz.de

LIGA Technology

from the initial letters of the German words for
Lithography, Electroforming and Moulding

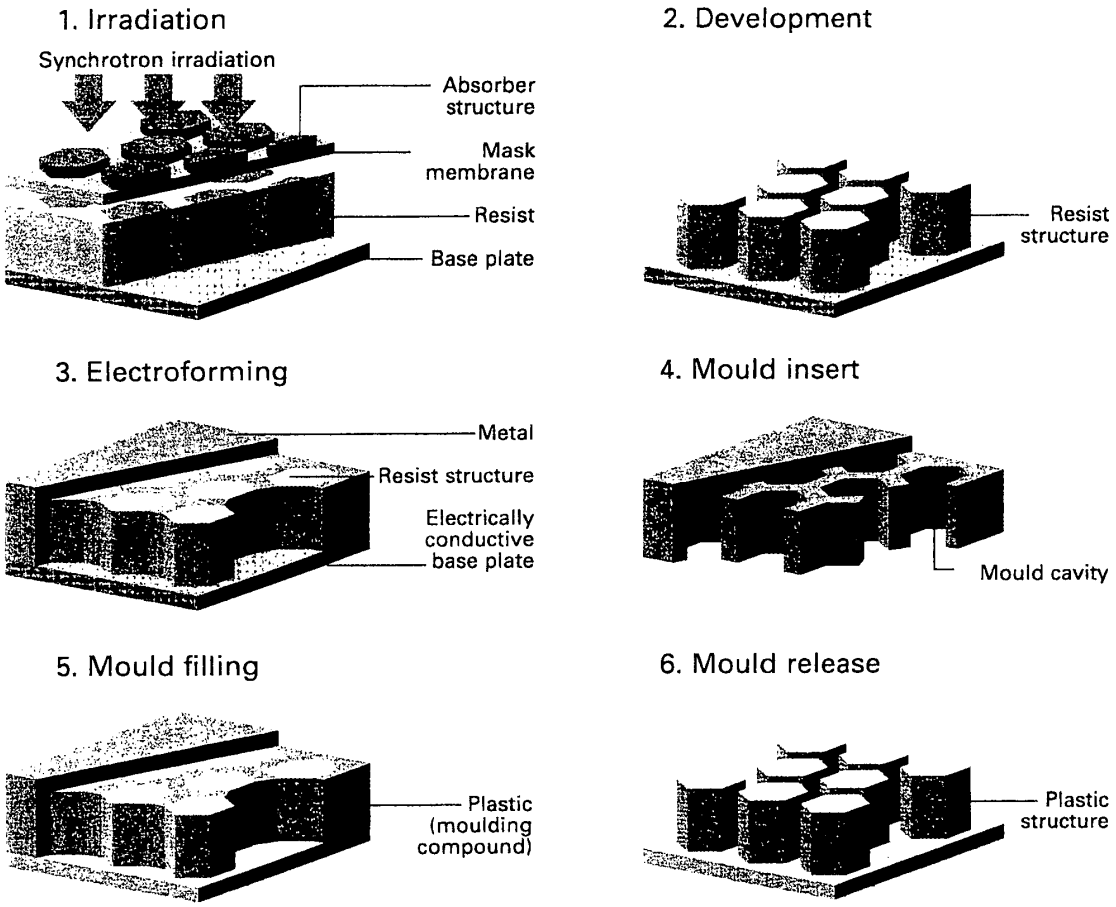


Fig. 1
Manufacturing steps in the LIGA process

Advantages of the LIGA technology

- Large range of materials: metals, alloys, plastics, ceramics
- Great variety of possible designs
- Structures over 1 mm high
- High precision into the sub µm range
- Low cost mass production

LIGA projects at IMM

- High precision positioning structures for glass fibres
- Miniaturized motors and gears
- Waveguide components
- Switches for fibre-optical applications
- Micro-optical components
- Micro-fluidic components such as pumps, heat exchanger structures and micro-reactors
- Optically-based sensors

Institute of
Microtechnology
Mainz

Deep lithography

The actual forming process deployed in LIGA technology involves the structuring of a radiation sensitive polymer material.
This may involve direct writing processes (laser ablation or electron and ion beam techniques), optical lithography or X-ray lithography. Lithographic techniques use the shadow projection of an absorber relief into a radiation-sensitive resist material (Fig. 1.1). The best results are achieved with synchrotron radiation. Lateral dimensions in the micron range and structure heights over 1 millimeter can be achieved.
The highest structure fidelity in copying is obtained by coating the absorber structure (usually gold) onto a stable substrate with good X-ray transparency. For this, IMM uses in particular beryllium. Also used at IMM are diamond membranes which possess optical transparency for multiple exposures and which are unproblematic to process. By using different materials and finely graded process steps to produce the primary structures, it is possible to reduce considerably the high costs of the mask manufacture.

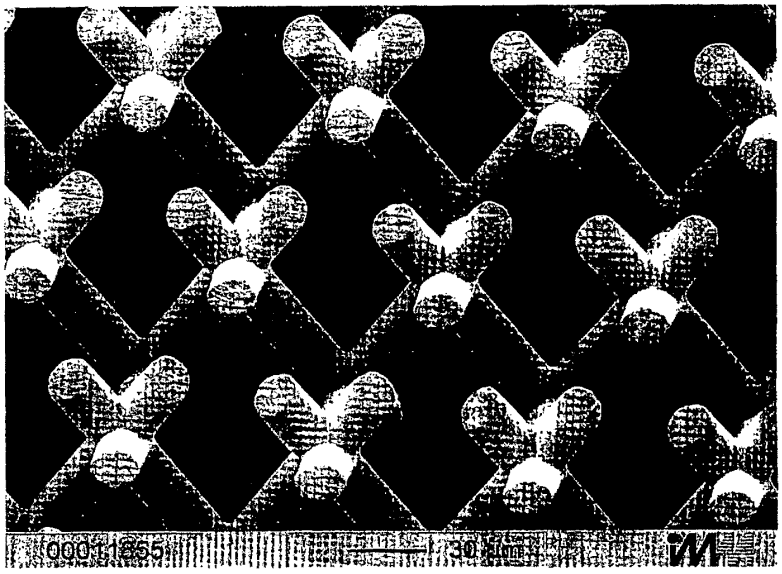


Fig. 2:
Initial structure for "Photonic Band Gap Materials", fabricated by threefold oblique exposure in a newly developed negative resist.

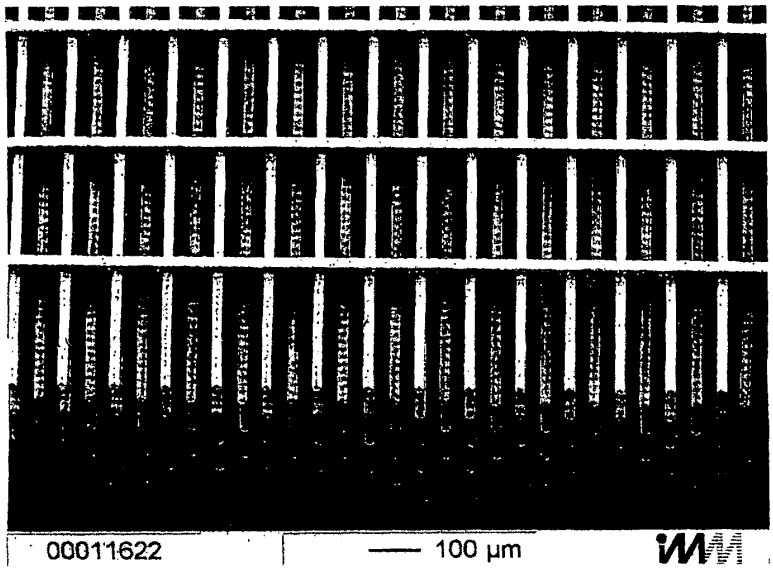


Fig. 3:
Test structure, produced by double oblique exposure.

New X-ray resists

Depending on the resist material, following the exposure process and the development of the irradiated area, either there is a direct three-dimensional reproduction (positive resist, see e.g. Fig. 1.2) or there is a tonal inversion through the use of a negative resist. Besides PMMA, which has been used as a resist material for many years, BASF AG has developed a further positive resist, poly-(lactide-coglycolide), which displays roughly two or three times the sensitivity of PMMA, together with a higher process reliability. IMM has moreover developed a negative resist which possesses a sensitivity fifteen times greater than PMMA. Now low cost manufacture of polymer structures is hence possible not only through moulding processes (microelectroplating, polymer mould techniques), but also by means of deep X-ray lithography.

Technology and structural variety

Mass production

The polymer structures can, depending on the application, either constitute the end product or serve for filling in slip casting so that, after firing, ceramic microstructures emerge. For mass production of microstructures from such different materials as metals, polymers, ceramics or glass, the resist structure is filled with a pure metal or an alloy by electroplating. This renders the negative of the original structure which may subsequently serve as embossing tool or as mould insert in injection moulding. The whole process sequence is illustrated in Figure 1.

Three dimensionality

Besides the often used simple projection technique shown in Figure 1, IMM is developing multiple exposure processes to manufacture stepped microstuctures (Fig. 5) and for structuring sacrificial layers (Fig. 5). Here, with the aid of an optical adjustment system, the exposure is performed on pre-structured substrates. With a mask using an optically transparent silicon nitride membrane, it is possible to produce masking precisions in the submicron range. Completely novel structural geometries arise when exposures are performed under oblique radiation and defined orientation of the mask to the resist unit (Figures 2 and 3). The structural variety thus achieved can be extended by additional irradiation techniques, such as defined displacement of the mask between two exposures, application of differently structured masks and multiple rotation of the resist.

The application of these processes is possible through the use of an X-ray exposure apparatus developed by IMM and Jenoptik Mikrotechnik GmbH and also by the use of new mask, resist and micro-electroforming techniques. For the further development of LIGA technology and its acceptance, it will be of paramount importance that future process-adapted apparatuses are made commercially available. At present, further manufacturing equipment is being developed to industrial maturity at IMM.

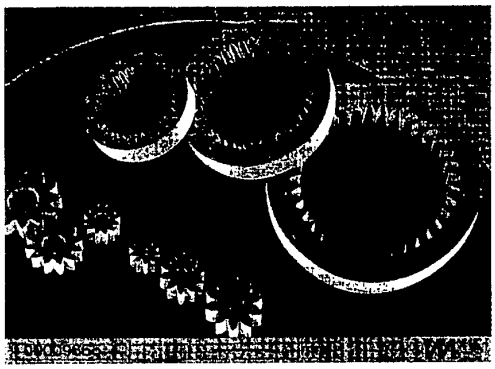


Fig. 4
Components of an epicycloidal gear, metallically moulded in nickel.

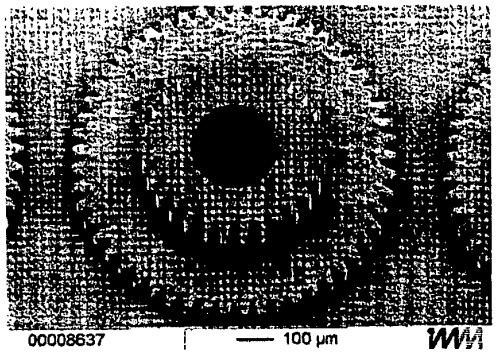


Fig. 5
Double pinion for a micro-gear.

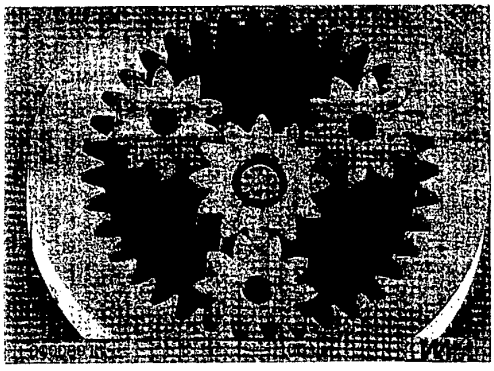


Fig. 6
Miniaturized epicycloidal gear for application with an electro-magnetic motor. The pinions are produced by electroforming of a resist structure.

Industrial use of synchrotron radiation

IMM offers its partners the use of all processes involved in LIGA. The deep X-ray lithography steps are conducted periodically at the storage rings in Berlin (BESSY I/II) and Orsay (DCI), the number of industrial users of synchrotron radiation is steadily increasing worldwide. Up-to-the-minute information about new trends in LIGA technology can
be accessed at our Internet site.

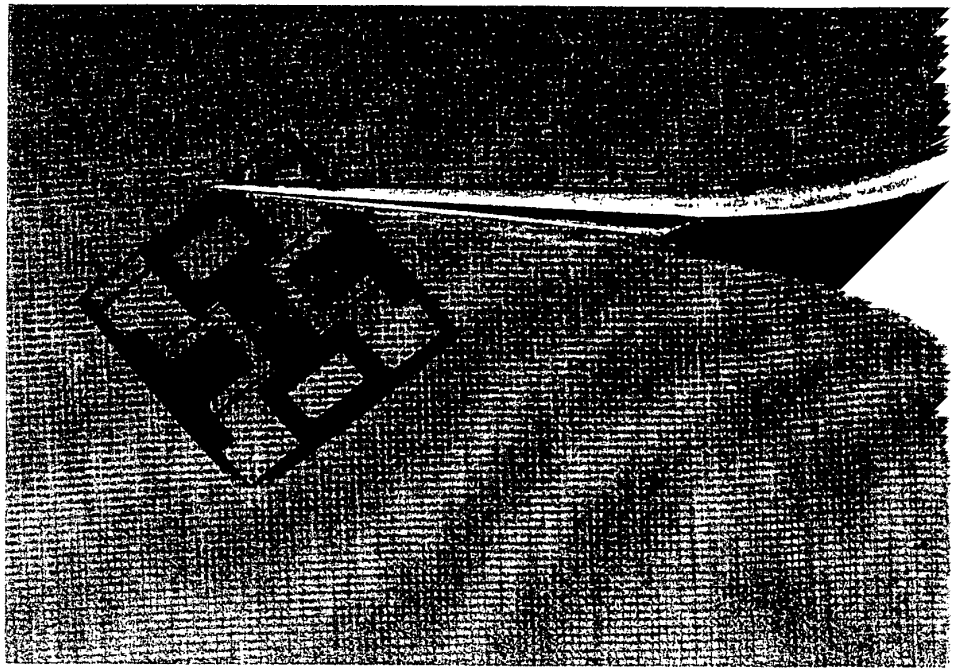


Fig. 7
Ultra-precise metal frame for an electromagnetic glass fibre switch. Material: electroplated ferronickel alloy.

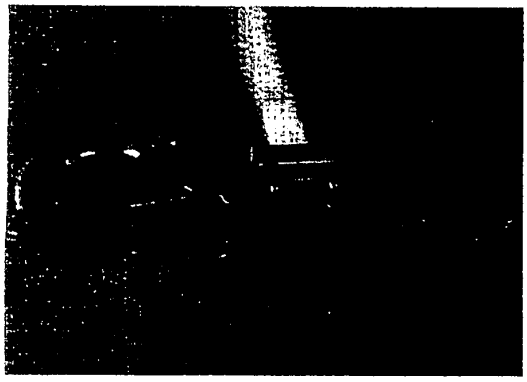


Fig. 8
Optical multiple channel fibre plug for applications in telecommunications. The plug is produced by injection moulding on the basis of modular micro-moulds.

Manufacturing equipment and materials for LIGA technology

- Further development of X-ray scanners (Commercially available from Jenoptik Mikrotechnik, Jena)
- Clean room electroforming equipment (in cooperation with R. KISSLER GmbH, Speyer)
- Provision of commercially available electrolyte baths and resist materials (SURTEC MICRO, Mainz)
- Equipment for micro-assembly

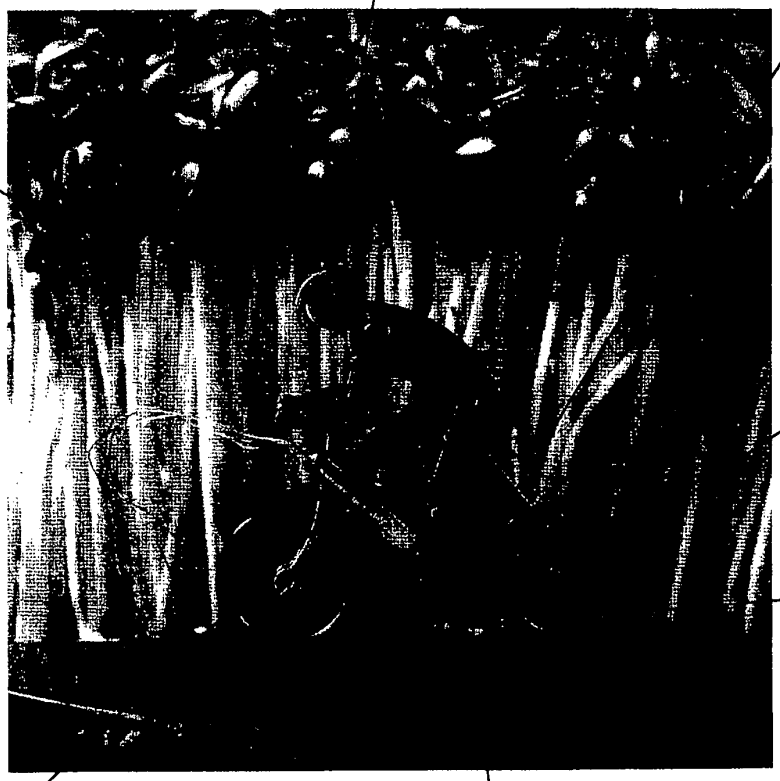
Your contact at IMM:
Public relations
Phone: +49 6131/990-117
Fax: +49 6131/990-205
Institut für Mikrotechnik Mainz GmbH
Carl-Zeiss-Strasse 18-20
D-55129 Mainz
http://www.imm-mainz.de

IMM 6.01/0499 E; Bailey & Gerasch, Darmstadt

„Strampeln“ mit Mikrotechnik aus Mainz
“Biking” with Microtechnology from Mainz

Radfahrer aus Edelstahl,
25 mm x 25 mm inklusive Fahrrad
Cyclist made of stainless steel,
25 mm x 25 mm including bicycle

Kresse,
handelsüblich
Garden-cress
(standard commercial size)



Riemen aus
Elastomer
Elastomer
belt

Räder aus PMMA
Wheels made
from PMMA

Gleitlager mit
Buchse aus
Messing
Slide bearings
and socket all
made of brass

Mikromotor mit dreistufigem Planetengetriebe:
Durchmesser 1,9 mm, Länge Motor 5 mm
Material der Getriebeteile: Kunststoff
Untersetzungsverhältnis des Getriebes: ca. 50:1
Micromotor with a three-stage planetary gear system: diameter 1.9 mm, length of motor 5 mm
Material used for the gear system: plastics
Reducing ratio of the gear system: approx. 50:1

Übersetzungsverhältnis vom
Tretlager auf das Hinterrad: ca. 1,3:1
Transmission ratio from pedals
to rear wheel: approx. 1.3:1

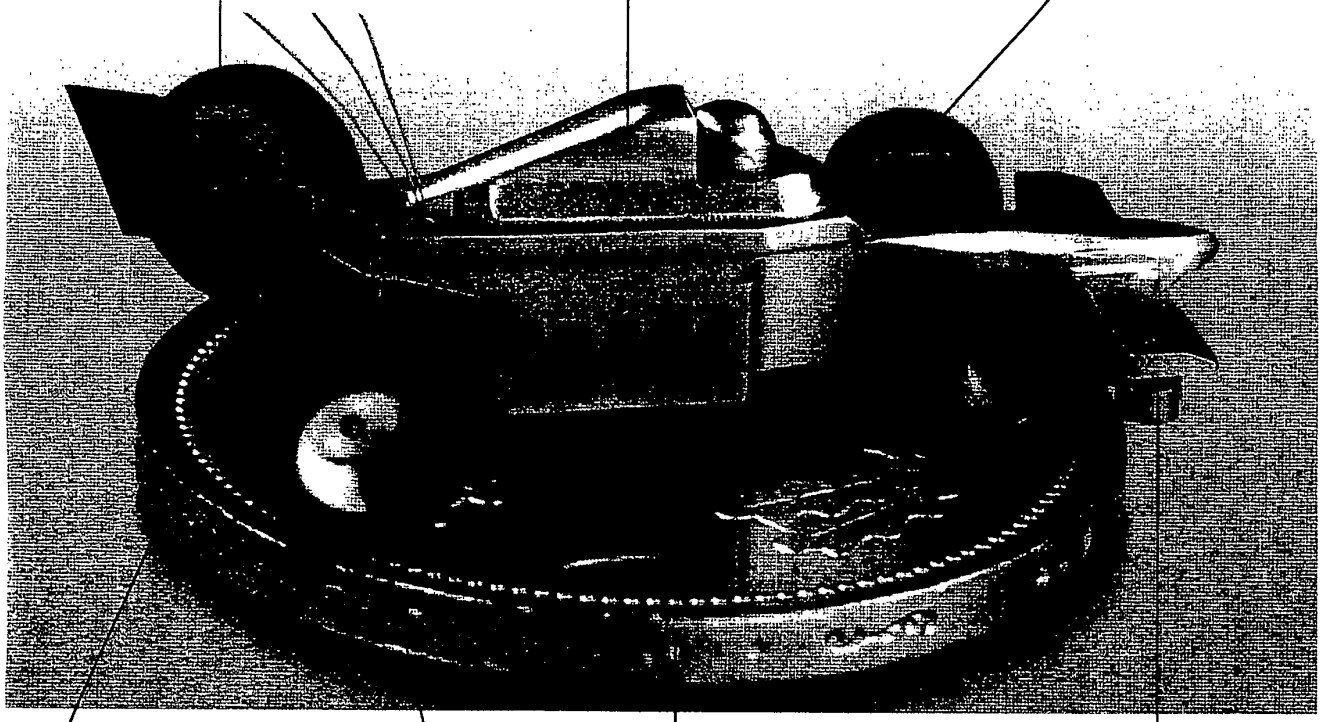
Institut für Mikrotechnik Mainz GmbH
Carl-Zeiss-Straße 18-20
D-55129 Mainz
Tel.: +49-6131-9900
Fax: +49-6131-990205
http://www.imm-mainz.de

Bahn frei für das High-Tech-Rennen
IMM auf der Pole-Position
Ready-steady-go for the High-Tech Race
IMM in the Pole-Position

Profillose Rennreifen aus Kunststoff
Plastic racing tyres

Vernickeltes Messingchassis des kleinsten Rennwagens der Welt, Länge ca. 25 mm
The world's tiniest racing car, made from brass and coated with nickel, length approx. 25 mm

Einzelradaufhängung vorne
Independant front wheel suspension



Winkelgetriebe an der Hinterachse, bestehend aus Ritzel und Kronenrad
Angular gear at the rear axle consisting of pinion and contrate gear

Alufelgen
Aluminium rims

Münze in deutscher Währung, Wert DM 1,–
German coin, value DM 1,–

Passive Lenkung mit Rubinlagerung
Passive steering with ruby bearing

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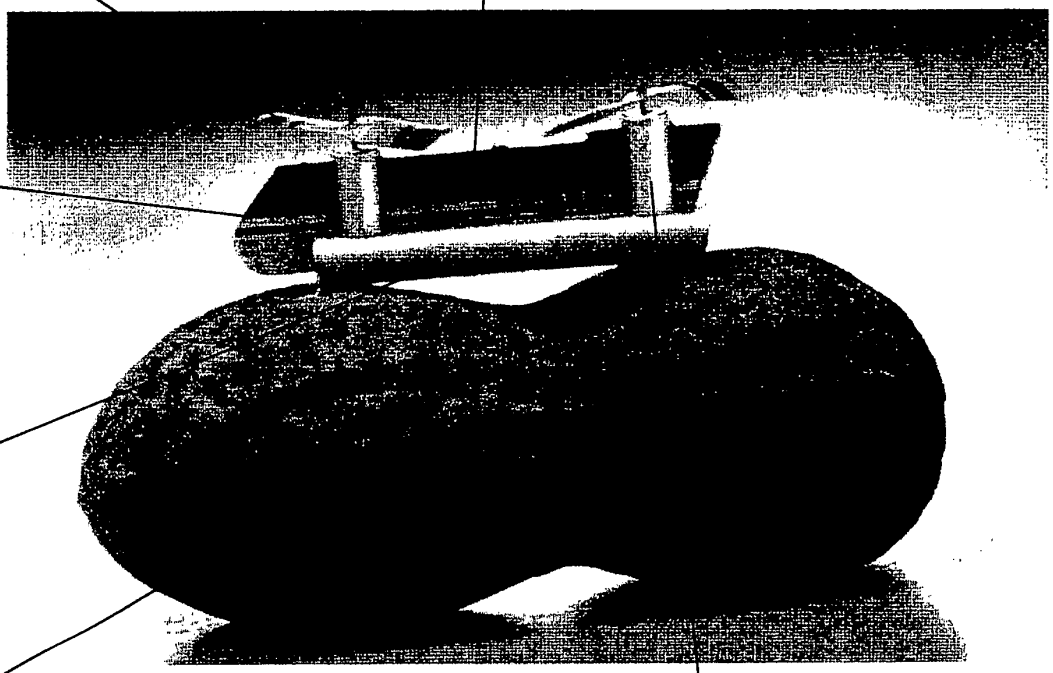
Mikromotor mit 3-stufigem Planetengetriebe:
Durchmesser ca. 2 mm, Länge 5 mm
Material der Getriebeteile: Kunststoff
Untersetzungsverhältnis des Getriebes: ca. 50:1
Micromotor with a three-stage planetary gear system:
diameter approx. 2 mm, length 5 mm
Material used for the gear system: plastics
Reducing ratio of the gear system: approx. 50:1

„Kleinster fliegender Hubschrauber der Welt“
“Tiniest flying helicopter in the world”

„Kleinster fliegender Hubschrauber der Welt“, reale Größe:
Länge 24 mm, Höhe 8 mm inklusive Rotoren
“Tiniest flying helicopter in the world”, actual size:
length 24 mm, height including rotors 8 mm

Rotor: lackiertes Papier, Durchmesser 13 mm
Rotor: painted paper, diameter 13 mm



Gewicht
ohne Erdnuß:
400 mg
Weight
(without peanut): 400 mg

Gehäuse:
Aluminium
Body:
aluminium

Erdnuß, handelsüblich
Peanut (standard commercial size)

Motor: Durchmesser ca. 2 mm, Länge 5 mm
Motor: diameter approx. 2 mm, length 5 mm

Leistung der Motoren:
bei 32.000 Umdrehungen pro Minute
hebt der Hubschrauber ab,
100.000 Umdrehungen pro Minute
werden mühelos erreicht
Motor performance:
airborne at 32,000 revolutions per minute,
capable of 100,000 revolutions per minute

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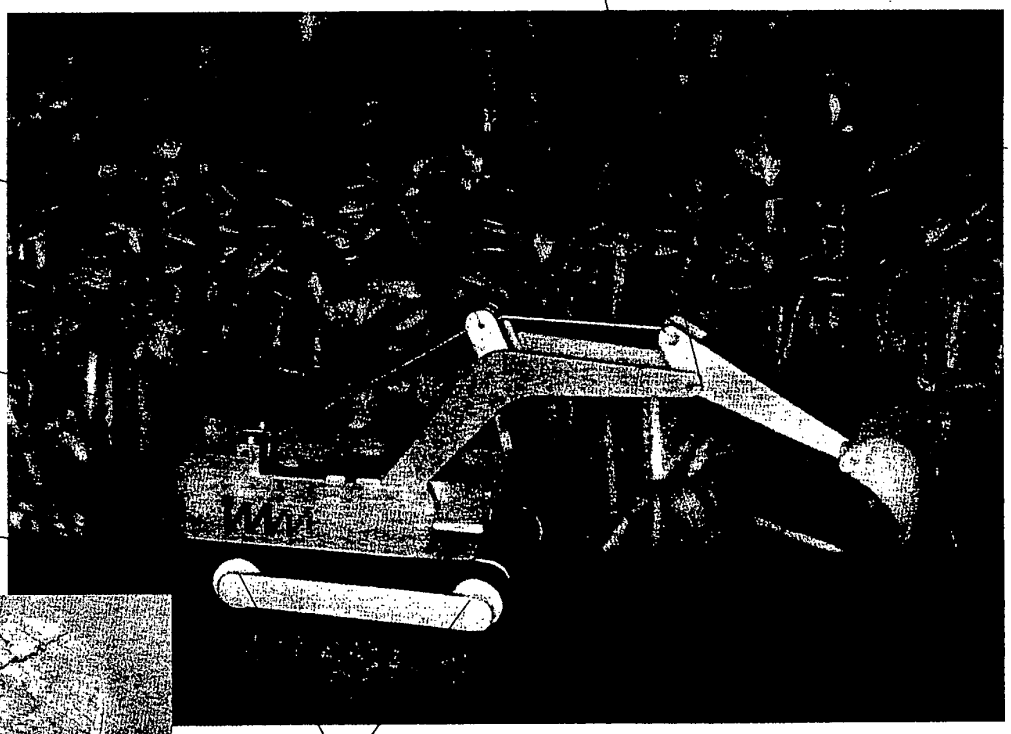
r Welt kleinster Bagger – mit Mikrotechnik
e world's tiniest digger – by Microtechnology

Baggeraufbau aus PMMA:
Gesamtlänge 63 mm, Gewicht 12 g
Digger made of PMMA:
total length 63 mm, weight 12 g

Ausleger aus Messing
Boom made from brass



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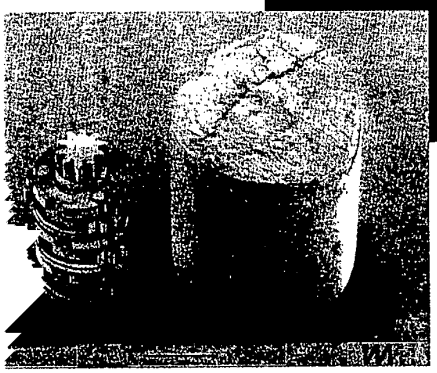

reistufiges Planetengetriebe,
pritzgegossen aus Kunststoff
it einem Granulatkorn
hree-stage planetary gear
stem with a plastic pellet

2 Mikromotoren mit dreistufigem Planetengetriebe:
ca. 2 mm Durchmesser, Länge 5 mm
Material der Getriebeteile: Kunststoff
2 Micromotors with a three-stage planetary gear system: diameter approx. 2 mm, length 5 mm
Material used for the gear system: plastic

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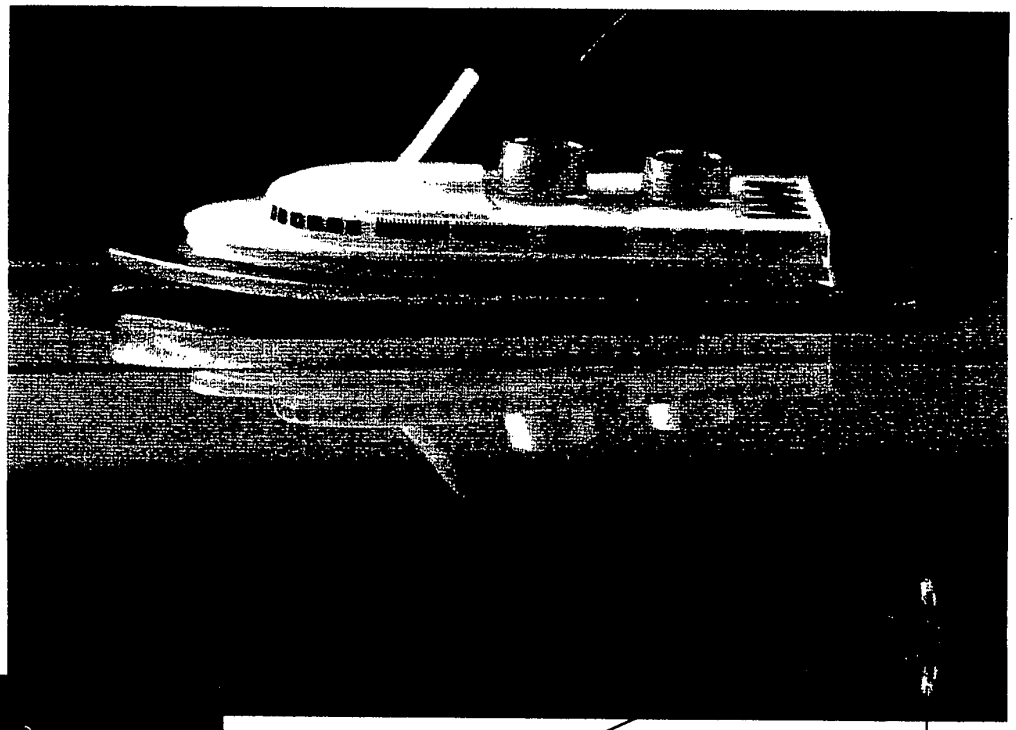
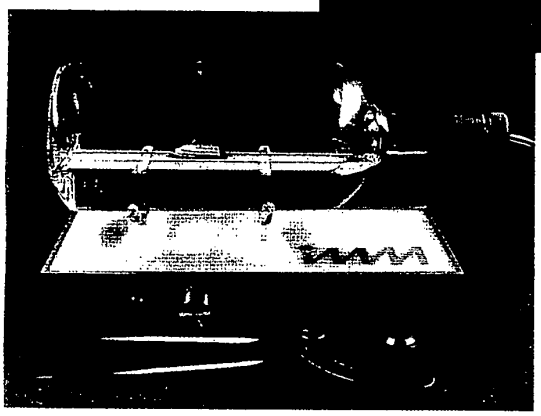
Kurs Innovation – mit Mikrotechnik
Course Innovation – of course Microtechnology

Rumpf des Mikroschiffs aus Polyurethan:
Länge 38 mm, Breite 12 mm, Höhe 9 mm
The hull of the microship is made of polyurethane:
length 38 mm, width 12 mm, height 9 mm

Deckaufbauten: 8 mm
Superstructure: 8 mm





Mikromotor:
ca. 2 mm Durchmesser,
Länge 5 mm
Micromotor:
diameter approx. 2 mm,
length 5 mm

Zweiflügelige
Schiffsschraube:
3,8 mm Durchmesser
Twin propeller blades:
diameter 3.8 mm

High-Tech message in a bottle

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