Final report

Tailored manipulation of fluids in functionalized highly integrated micro- and nanoscale fluidic systems

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Executive Summary

New technologies based on transport, actuation and manipulation of fluids and objects in the micro- and nanometer scale are rapidly developing. The enormous scientific and technological interest focuses on lab-on-a-chip approaches applicable in the fields of analytics and monitoring in medicine, biology and the environmental sector. The strong demand for size reduction of the highly integrated fluidic networks requires novel ideas for transport and tailored manipulation of fluids and particles in fluidic systems. Due to miniaturization, no moving mechanical parts are favourable but a contactless driven flow by electric, magnetic, thermal or microacoustic fields and their gradients as well as the combination of those are required. The project aims firstly on alternative concepts and a fundamental understanding of the impact of external forces on contactless pumping, fast and efficient mixing, controlled manipulation and sensing of species on a single particle scale and secondly on the application of relevant aspects in realizing proof-of-principle platforms.

Within the frame of the project firstly suitable microsystems and platforms were designed and manufactured. In addition, established as well as new measurement techniques were adapted for measuring velocity, concentration and temperature of the fluids on the microscale. This opens new possibilities for a detailed characterization of the influence of external forces within microfluidic devices. In conjunction with a novel approach for the in situ measurement of micro acoustic wave fields, the direct correlation between the external force originating from surface acoustic waves and the resulting induced 3D fluid flow could be revealed, for the first time. In that way, the derivation of a new numerical model was supported experimentally. This enables fast and easy 3D simulation of the acoustically-induced fluid motion within microfluidic devices using high-frequency surface acoustic waves. Besides, a strongly localized temperature increase, locally coinciding with the acoustically-induced fluid flow, was brought to light. Strong temperature gradients occur within short time, depending on frequency and power of the micro acoustic wave fields, fluid properties and the magnitude of the simultaneously induced fluid flow. Therewith promising new perspectives are opened for microfluidics using surface acoustic waves, not only for pumping or efficient mixing, but also for a tailored localized temperature controlling of the fluid based on acoustic energy absorption. With a SAW-based acoustofluidic setup it was possible to trap biological cells two dimensionally in fluidic solutions of different compositions and to hold them at a specific position independent of flow conditions inside the chamber. By using different inflow solutions the acoustic forces generated a gradient field allowing to expose the cells to different concentrations of staining solution and to observe their reaction over time. The device offers high functionality and indicates a promising path forward to a broad range in cell research.

Downscaling fluidic microsystems and platforms requires implementing electrodes and in particular magnetic systems with high energy density directly in the wall of the system to generate locally high magnetic field gradients. We succeeded to develop a magnet system consisting of a softmagnetic material (CoFe) and a hardmagnetic material (FePt L10) electrodeposited in lithographically designed micro- and nanostructures. Thereby two different approaches to prove the concept of fluid manipulation by overlaying the magnetohydrodynamic force and the magnetic field gradient force were studied. Firstly a redoxsystem, which is common for redox-MHD was used and combined it with magnetic field gradient templates. Secondly, a paramagnetic liquid was used and potential/concentration changes between the electrodes with magnetic field and its enhanced gradients were measured. The selective alteration of the fluid flow was visualized and supported by rigorous numeric simulation. For the first time it was experimentally shown and proved that the concentration of ions can change locally and fluid can move in desired directions by overlapping of the different magnetically driven forces. This opens new applications for sensing, mixing and sorting tasks on small scales.
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1. Starting position and project objectives

New technologies based on transport, actuation and manipulation of fluids and objects in the micro- and nanometer scale are rapidly developing and to be implemented in all branches of industry, analytics, medicine and biology. Downsizing of conventional fluidic systems and miniaturized highly integrated devices approaches physical limitations, for example the resulting very low Reynolds numbers restriction for the mixing of fluids or the transport of objects. Fundamental knowledge is required to understand the underlying physical and chemical phenomena that are linked to the complex interfacial interactions. Classical fluid mechanical approaches have to be combined with descriptions of the interfacial interaction between walls of the channels and particles or multiphase systems, with surface chemistry, and with electrokinetic effects, which alone or as cooperative effects impede the prediction for desired actuations. External driving forces based on electrical, thermal, magnetic and microacoustic fields and field gradients are promising tools to overcome problems affecting certain aspects of transport processes and may be tailored for specific tasks in micro- and nanoscaled systems. The challenge of this proposal is to cover both the engineering and manufacturing of micro- and nanoscaled fluidic systems and the fundamental understanding of fluid flow affected by near wall interaction. New interesting fundamental effects also with regard to future application are expected on the nanoscale when classical fluid dynamic approaches are reduced to the molecular scale. Besides the experimental part, theoretical modelling will be essential to discriminate single molecular effects and complex coupled behaviour in fluidic systems at the nanoscale and to quantify them.

The project aims at the fundamental understanding of the impact of external forces and boundary conditions on fluid flows in channels from the micrometer scale down to the nanometer scale. In highly integrated fluidic networks, a tailored manipulation of the fluid flow, the transport of particles, charged species or ions, and a quantitative description of the effects have to be established. The gained knowledge on fundamentals will be used to set up proof-of-principle hybrid micro- and nanofluidic platforms demonstrating three objectives, (i) controlled manipulation on a single particle scale, (ii) fast and efficient mixing for sensing and synthesis and (iii) constant laminar flow as required for high energy output of e.g. membrandfree fuel cells.

2. Development and realization of the work packages

Manufacturing, characterization and theoretical modeling were tackled in two work packages (WP) which interact and were subdivided in research topics (RT). WP 1 was focused on fundamental issues, which in all cases go along with carefully designed and well-controlled experiments accompanied with rigorous theoretical modeling. WP 2 should be transfer application relevant aspects realizing proof of principle demonstrators.

The main tasks in the work packages were:
(WP1-RT1) Manufacturing of micro- and nanosystems: We want to design and fabricate micro- and nanochannels (including the micro-and nanofluidic platforms used later in (WP2); micro- and nanoelectrodes that will be integrated in channel walls; particles and nanoobjects with defined size, functionality and physical properties.

(WP1-RT2) Investigation of fluid flow driven by external volume forces: Contactless transport of fluids will be generated by volume forces based on electric, magnetic, thermal and microacoustic fields and their gradients; the interaction of these different forces will be studied.

(WP1-RT3) Velocity and concentration profile measurements and analysis: (1) We will use novel measurement systems and (2) follow new concepts by implementation of nanometer sized electrodes (RT1).
(WP1-RT4) Theoretical modeling at the nanometer scale: The nanometer scale requires new concepts. Molecular dynamic approaches will be applied to resolve processes on small length and time scales.

(WP2-RT1) Hybrid micro- and nanofluidic platforms for controlled manipulation on the single particle scale: We will set up two types of experiments, (1) is based on microacoustic forces and (2) focuses on electro-and magnetophoretic manipulation.

(WP2-RT2) Fast and intense mixing for micro- / nanofluidic reactors: Electrochemically and magnetically active systems will be designed based on (WP2-RT1) and equipped with nanoelectrodes and nanosized magnets to generate mixing.

(WP2-RT3) Constant flow in micro membrane-free fuel cells: This aims at increasing the efficiency and life time by intelligent design of branched micro- and nanochannels with magnets to remove paramagnetic reaction products.

Already at the beginning of the project reasonable modifications of the tasks of the work packages were needed. Design and manufacturing of the micro channels and platforms for fundamental investigations were more comprehensive than expected. Following the tasks within the work packages new techniques were developed and established techniques were adapted to the lower dimensions of the micro systems investigated. Finally with the advanced design successful experiments were performed on the micro scale and supported by careful analysis of the results and extended numerical simulation to verify the theoretical models. In future proposals which are already planned and partially submitted in cooperation with partners the open questions related to the nanoscale are addressed.

3. Results and Discussion

3.1 Micro systems and platforms

Careful manufacturing of micro- and nanostructures is one of the main tasks to provide the platform for the further experiments. For the first fundamental investigations commercial available channels and systems from mm to µm scale were employed. It aims on the development of suitable methods measuring with high spatial and temporal resolution concentration, temperature and velocity profiles and detecting enrichment of particles, cells and ions. The techniques developed for sensing, actuation and measurement are unique and comprised a longer period than expected. In a second step 2D micro systems were designed from PDMS, SU8 and by lithographical methods equipped with electrodes, magnets, sensors and acoustic systems integrated in the wall of the micro systems and platforms to drive, manipulate or mix fluid with particles, cells or paramagnetic ions by external volume forces as are microacoustic fields, magnetic and electric fields and its gradients. A challenging task was also developing a magnet system consisting of a softmagnetic material and a hardmagnet electrodeposited in lithographically designed micro- and nanostructures.

Macroscopic cell to study fundamentals of the acoustic streaming-effect

For fundamental studies on the acoustic streaming-effect a simplified setup was employed, which is depicted in Figure 1. A combination of a large glass cuvette with an interdigital transducer (IDT) on a piezoelectric LiNbO3 chip was used. The glass cuvette has dimensions much larger than the acoustic length scales to be expected at the parameter range applied, e.g. size of the surface acoustic wave field and the damping length of the corresponding bulk acoustic wave (BAW) inside the fluid. Therefore, unwanted side-effects due to reflections of the BAW and of the SAW at microchannel sidewalls can be neglected, allowing studying the acoustically-induced fluid flow of three-dimensional characteristic and its temperature distributions inside the fluid, in detail. For this, a novel combination of a volumetric flow velocity measurement technique and planar laser induced fluorescence was applied, for the first time.
Inner size of glass cuvette: 
H x L x B = 97 mm x 143 mm x 50 mm

SAW parameter range:
- $\lambda = (20 \ldots 150) \mu m$
- $P_{IDT} = 10 \text{ mW} \ldots 1 \text{ W}$
- IDT aperture 2 mm

Two general IDT positions:
a) Loaded (as depicted)  
b) Unloaded (IDT not immersed in the fluid)

Fluid: water, mixtures of water / glycerin

Micro platform for investigation of fluid mixing

A microfluidic platform specifically designed for the active mixing of fluids with high efficiency was used for the investigation of SAW-driven fluid motion and subsequent mixing inside microchannels. An example of the setup comprising the fluidic component - the micro channel and the Y-shaped junction made of PDMS – as well as the microacoustic component – a double-sided polished piezoelectric substrate of 128° YX LiNbO₃ for SAW generation – is depicted in Figure 2. With this micro mixer, two fluids were brought together and mixed rapidly within a very short distance of about 500 µm given by the width of the SAW field. The optical access from the bottom through the piezoelectric substrate allowed for measuring the SAW-field and the 3D fluid motion induced by this field, showing, for the first time, the direct correlation between the induced fluid motion and the driving acoustic force for mixing purposes.

Fig. 1 Macroscopic cell to study fundamentals of the acoustic streaming-effect in fluids induced by SAW. A large glass cuvette was used to avoid side-effects of acoustic reflections and scattering at channel sidewalls, which are typically present at the microscale.

Fig. 2: (left) Sketch of the manufactured PDMS acoustic micro mixer, (right) flow measurement result of the induced fluid flow within the mixing zone and. The two fluids are going to be mixed efficiently by a lateral fluid flow induced by a surface acoustic wave
propagating perpendicular to the microchannel axis. The SAW parameter range applied: \( \lambda = (20 \ldots 60) \, \mu m, P_{\text{IDT}} < 10 \, \text{mW}, \text{IDT aperture} 500 \, \mu m, \text{Fluid: water-based solutions} \)

**Micro platform for particle/cell trapping and manipulation**

In order to investigate the capability for microacoustic particle manipulation inside two-dimensional (2D) fluid confinements like microfluidic chambers, a setup realizing inside the chamber 2D-patterned standing wave fields by means of 4 opposing main interdigital transducers aligned along the main SAW directions of a 128° YX LiNbO_3 chip was realized (Figure 3). Due to the differences of the SAW phase velocities along the X- direction and the X+90° direction the two active main IDTs pairs are operated at slightly different frequencies but at the same wavelength. In order to have additional possibilities to influence and corrugate the established main wave field pattern two inclined transducer pairs were also implemented in the setup.

**SAW parameter:**
- \( \lambda = 150 \, \mu m \)
- \( f_x = 24.3 \, \text{MHz} \)
- \( f_{x+90^\circ} = 25.8 \, \text{MHz} \)
- \( P_{\text{IDT}} = (80 \ldots 150) \, \text{mW} \)
- \( V = (0,1 \ldots 1) \, \mu L/s \)

**Fig. 3:** (left) Sketch micro chamber to trap living cells, (middle) photograph of the designed system, (right) SAW parameter

**Driving flow and ion enrichment in magnetic gradient fields by potential measurement**

For the first proof of principle experiments for enrichment of paramagnetic species in high magnetic gradient fields several types of PDMS based micro channels were designed, manufactured and tested used in 3.3.2. Figure 4 shows exemplarily a sketch and photograph of the designed structures. The length of the channels was in the range of 10 to 20 mm, the width was 1-2 mm and the height was 500 \( \mu m \). The micro systems used were equipped with Au electrodes. In one set of test channels the substrate below (Si or glass) was lithographically structured for electrodeposition of magnetic material (3.1.2). The Au electrodes were press-contacted and connected to the potentiostate IPS 100 mA-10 V with extremely high input impedance of \( 10^{15} \, \Omega \).

**Fig. 4:** (left) Sketch and manufactured PDMS micro channel with magnet and Au electrodes, (right) magnet system
3.1.2 Electrodeposition of micro electrodes and micro magnetic structures

In order to manipulate fluid flow in micro channels micro magnets should be electrochemically deposited directly into the wall or bottom of the fluid channel system as emphasized in (WP1-RT1 and WP2-RT2) and sketched in Figure 4. It is expected that fluid flow can be driven by the magnetic field gradient force provided by either magnetized soft magnetic structures with high saturation magnetization (CoFe) or/and hard magnetic L10 FePt structures. Therefore next to the design and manufacturing of different micro channels and lithographically structured systems deposition was performed. The structuring process was an iterative process to optimize the performance. The deposition of stress free soft magnetic CoFe and hard magnetic Fe50Pt50 with low oxygen content and high coercivity and remanence into micro structures was essentially and the last one was the most challenging and time consuming task for magnetic gradient field caused manipulation. Sensing the change of ion concentration by selective potential measurement in situ, additional electrodes are required. The enrichment was supported and verified by interferometric measurements as seen in Figure 8 and velocity measurements were realized by µ-PIV.

Electrodeposition of CoFe

A potentiostatic alternating deposition method of Co45Fe55 was developed and optimized based on simple sulfate electrolytes (Fe2+:Co2+ = 1:1.5) at -1.0 V \( t_{\text{on}} = 1 \text{ s} \) and \( t_{\text{off}} = 2 \text{ s} \) with addition of SDS (0.04 g/l) first for flat layers. The method was applied for deposition of CoFe in micro scaled lithographical structures to obtain smooth layers with high saturation polarization (2.3 ± 0.1) T and low coercitivity (0.008 ± 0.001) T, shown in Figure 5. The deposition in nanoscaled anodized aluminium oxid (AAO) structures has been performed successfully earlier and for nanomagnetic application within the project (E4, E5).

The obtained smooth and crack free structures are suitable in combination with hard magnetic layers for application in micro channels (Figure 5).

Electrodeposition of FePt (L10)

Concerning the FePt deposition three different electrolyte systems which rest upon three Pt sources have been investigated during this project. The used chemicals were iron(III) sulfate (Fe³⁺), sulfosalicylic acid (SSA), sodium citrate (Na₃Cit), and hexachloroplatinic acid or dinitrosulphatoplatinum (DNS-Pt) or the commercial Pt electrolyte JE18. The last one revealed the mostly homogeneous Fe50Pt50 films depositing potentiostatically with an optimized pulse regime on Au and into nanoscaled AAO structures, Figure 6. A heat treatment series reveals the transformation from a nanocrystalline as-deposited A1 phase to the L10 phase occurring at 500 °C and higher (E4, E5). This could be underlined with magnetic hysteresis loop measurements, showing an increase in the hard magnetic behavior. COMSOL simulations have been shown that the field gradient generated at the top of the layer system with the
optimized magnetic properties is suitable for application in highly integrated systems. The experimental evidence that the combined magnetic structure fulfill the demand could not provide within the time-span of the project but promising experiments are in progress.

3.2 Measurement techniques

In order to quantify the influence of external volume forces at the microscale, e.g. induced by micro acoustic wave fields or magnetic gradient forces, several methods have been used within the WP1-RT3. The methods applied can be subdivided not only into the quantity of interest, e.g. the velocity of the fluids, but also into the actual scientific question. The latter is either of fundamental nature according to WP1-RT2, or related to the application of external fields in tailored micro scaled devices manufactured in the course of the project, WP1-RT1, WP2-RT1 and WP2-RT2. The quantities of interest were the concentration, the velocity and the local temperature of the fluid as well as the vibrational amplitude of micro acoustic wave.
fields. For some tasks collaboration with other researchers were done, namely Prof. Cierpka’s group at Technische Universität Ilmenau, Prof. Czarske’s group at Technische Universität Dresden, Prof. Kähler’s group at the Universität der Bundeswehr München and TSI Incorporation (Shoreview, MN, USA).

3.2.1 Concentration

Variations of concentration cause variations of refractive index that can be measured using interferometric methods. For this, optical access from two opposing sides is usually required to determine the optical path length changes of an object beam propagating through the device using a reference beam superimposed on the camera chip, yielding the 2D distribution of the concentration averaged along the depth-direction. In case of micro magnetic structures embedded in the channel walls of micro devices (compare WP1-RT1) usually at the top or bottom sidewall, optical access is given only from one side. Therefore, a modified configuration has been tried in order to determine concentration changes induced by magnetic field gradients, see Figure 7. The laser light of 532 nm wavelength was split into the reference and the object beam with a beam splitter cube. The object beam was guided through the microscope via the camera port, while the reference beam passed several optical elements to adapt size and curvature of the reference beam. The object beam passed again the microscope in backward direction, after being reflected at the inner top wall of the micro channel coated with Au. Object beam and reference beam were finally superimposed onto the camera with an off-axis angle of about 2°, in order to discriminate between the diffraction orders and to allow for reconstructing the object beam. The reconstruction on the camera sensor was based on 2D FFT and spatial filtering of the +1. diffraction order.

Difficulties during the measurements arose due to reflections at internal optical components of the microscope and temporal changes of the wave front caused by slight movements, e.g. of the microchannel during the magnetization of the small CoFe structure (Figure 7 and 11c,d) with a permanent magnet placed behind the channel wall. These movements made interferometric measurements of concentration changes impossible, especially during the strong transient enrichment of paramagnetic ions, which immediately starts with the occurrence of magnetic gradient fields. Therefore, to investigate the evolution of the local enrichment of paramagnetic species in micro channels fluorescence intensity measurements have been done. For these measurements, the micro channels were filled with two solutions containing different concentration of the paramagnetic species. In addition, fluorescent dye (Rhodamine B) was dissolved in one of these solutions. Assuming negligible diffusion of the fluorescent dye within the short time of the experiments, the local distribution of the solutions can be visualized by measuring the local fluorescent intensity, allowing for quantifying the evolution of the local enrichment of paramagnetic species. The optical methods applied support and verify the enrichment of paramagnetic species in high magnetic gradient fields by local potential measurements in downscaled systems, see chapter 3.3.
Fig. 7: Setup for interferometric measurement of concentration changes due to magnetic gradient fields.

### 3.2.2 Velocity

For both aspects, fundamental research on the macroscopic scale and the application of micro acoustic wave fields on the microscopic scale, advanced optical methods based on particle image velocimetry (PIV) were used enabling the measurement and analysis of the acoustically-induced fluid flow with three-dimensional characteristic. For flow measurements inside the macroscopic cell (Figure 1) the V3V-Flex volumetric PIV was applied together with TSI Inc. This technique enables flow measurements with very high spatial resolution (in the range of 0.1 mm) in a comparably large measurement volume (up to 100 ml), which was required to gain fundamental understanding on the acoustically-induced fluid flow but also for comparison between fluid flow and local temperature rise induced by micro acoustic wave fields (Figure 1). For velocity measurements inside the manufactured acoustic micro mixer (Figure 2) the astigmatism particle tracking velocimetry (APTV) was used as this technique allows for 3D flow measurements with micron resolution at restricted optical access from only one side, as common for microfluidic devices. For the acoustic micro mixer, optical access was given through the birefringent piezoelectric material using a polarizing filter. As tiny particles are used that represent the local fluid velocity, extra care has been taken for selecting the tracer particles or adjusting a proper parameter range as acoustic impedance differs between fluid and particle. Parts of the experimental work did address this specific question to either determine particles that faithfully follow the acoustically-induced fluid flow or to properly set the range of frequency or acoustic power, in order to avoid any side-effects induced by the acoustic radiation force.

### 3.2.3 Temperature

To date, there is no well-established method available for reliable temperature measurements on the microscale that fulfils the requirements of three-dimensionality, spatial and temporal resolution necessary to investigate temperature effects induced by micro acoustic wave fields. For that reason, temperature effects were studied in the macroscopic cell (Figure 1) using the planar laser-induced fluorescence (PLIF) technique. This technique relies on dissolving fluorescent dyes in the fluid whose fluorescent intensity is highly dependent on the surrounding temperature. For measurements, a thin laser light sheet illuminates the dye, which absorbs the laser light and emits light of longer wavelength that is captured with a camera orthogonally aligned to the laser light sheet, providing 2D temperature fields within the thin laser light sheet by evaluating the actual fluorescent intensity, on condition that the fluorescent intensity
behavior is well-known by calibration. High spatial and temporal resolution can be achieved up to pixel size and frame rate of the camera (15 Hz), respectively. In addition, 3D temperature measurements with thermocouples were performed. As these measurements lack of spatial and temporal resolution, the results of these measurements have been used only for validation purposes. The results did fit well to the results obtained from PLIF measurements.

### 3.2.4 Amplitude distribution of micro acoustic wave fields

The amplitude of micro acoustic wave fields is of vital importance to gain detailed understanding of the influence of those fields within micro devices, e.g. inside the manufactured micro mixer (Figure 2). It is usually accessible from the top side of the piezoelectric crystal substrate and can be measured using the laser Doppler vibrometer (LDV, UHF-120; Polytec GmbH) at the IFW. The amplitude profile is strongly dependent on boundary conditions, meaning any micro structure or device placed on top of the piezoelectric material has influence on the micro acoustic wave fields, e.g. by diffraction, refraction and scattering effects as well as by damping at the channel walls. However, measurements from the top through the micro devices and the fluid are difficult to accomplish since changes of the optical path length occur due to vibrations of the channel walls or local changes of the refractive index originating from the SAW. Therefore, the amplitude of the SAW was measured from the bottom side through the birefringent piezoelectric crystal. To suppress double refraction a polarization filter was placed between microscope objective of the LDV and the LiNbO₃-chip to adjust laser light polarization corresponding to the polarization of the extraordinary wave. Test measurements were conducted with a piezoelectric substrate (128° YX LiNbO₃), at which no micro device was placed on top of the chip. In that way, the amplitude was accessible from the bottom as well as from the top side. By comparing the amplitude $A_{b}$, measured through the LiNbO₃ crystal, with the amplitude $A_{t}$ measured from top (reference measurements), a constant factor $n$ between both amplitudes was found ($A_{b} = n \times A_{t}; \ n = 2.24$) that corresponds to the refractive index of the extraordinary wave (angle between wave vector of laser light and c-axis of the 128° YX-LiNbO₃: 38°). As the optical path length depends on the refractive index, the actual SAW-amplitude is given by dividing the measured amplitude with this factor. Moreover, the factor n did not change for different electrical power levels applied to excite the micro acoustic wave. Hence, no significant influence of elasto-optical, electro-optical or temperature effects has to be expected for the measurement of the amplitude of micro acoustic wave fields at moderate electrical power levels ($P_{el} \leq 180$ mW) used in our studies. With this measurement approach, a first quantitative comparison between experimental and numerical results of the acoustically-induced fluid flow inside a micro mixer was possible [E 1]. In that way, improvements of the theoretical modelling of the acoustically-induced fluid flow originating from micro acoustic waves has been achieved (WP1-RT4).

### 3.3 External forces for manipulation, mixing and transport

#### 3.3.1 Surface and micro acoustic fields

**SAW-controlled particle trapping and manipulation**

For the investigation of microacoustic particle and cell manipulation effects inside microfluidic channels and chambers purpose-designed setups have been used deploying standing SAW wave fields. These wave fields with complex two-dimensional (2D) time-independent amplitude distributions were realized by symmetric arrangements of interdigital transducers (see Figure 3). By controlled balancing the electric input power levels of opposing transducers as well as their phase shift well-defined acoustic gratings with pressure nodes and anti-nodes could be established accompanied by forces sufficiently high to trap fluid-borne species like micro particles and cells. The acoustic contrast of these species with respect to the acoustic properties of the fluid filling the chamber is governing the kind of trapping position, i.e. whether a micro object is trapped in the nodal or anti-nodal positions of the three-dimensional bulk...
acoustic wave field established inside the cavity of the micro vessel. With such a SAW-based acoustofluidic setup it was possible to trap biological cells in fluidic solutions of different compositions and to hold them at a specific position independent of flow conditions inside the chamber. By phase-shifting the sine signals fed to each individual of the two pairs of IDTs arranged perpendicular to each other the 2D amplitude distribution of the wave field could be moved in a controlled manner. This enabled the specific movement of the cell pattern around the micro chamber exposing the trapped cells to different concentrations of a specific substance as depicted in Figure 8.

![Image](image1)

Fig. 8: Left: Multi-purpose SAW-based setup for comprehensive cell behavior studies. Right: SAW-induced gradient of two different fluids (for visualization dyed with red and yellow ink, resp.) [E32, E33].

The trapped cells could be observed during long-term experiments undergoing multiple alterations of the surrounding fluid. Such an observation even allowed to document the process of cell death after an exposure to different concentrations of culture medium providing fresh nutrients to the cells and the staining solution methylene blue used for live-dead-differentiation (Figure 9), [E32].

![Image](image2)

Fig. 9: Cells of Saccharomyces cerevisiae trapped in the SAW induced pressure field within the microfluidic chamber. Enlarged: focused cells over time when exposed to methylene blue solution. (dying cell indicated by arrow)

In conclusion the SAW-based device enabled a two dimensional trapping of yeast cells as well as a defined movement of the trapped cells within in the manipulation area. By using different inflow solutions the acoustic forces generated a gradient field allowing to expose the cells to different concentrations of staining solution and to observe their reaction over time. The device offers high functionality and indicates a promising path forward to a broad range in cell research.

**Acoustic Streaming**

Fundamentals of acoustic streaming originating from SAW were studied using the setup depicted in Figure 1. With this setup, fluid flow and temperature measurements were
conducted for two general configurations; unloaded and loaded IDT position. It was found that while frequency of the SAW and viscosity of the fluid influence the acoustically-induced flow according to Stokes law of sound attenuation, the electrical power applied exhibit unexpected influence on the velocity jets [E 3, E 11, E 18, E 20]. Besides a maximum velocity that scales with $\sqrt{P_{el}}$, as it also applies to the amplitude of the SAW, the penetration depth as well as the divergence of the velocity jets strongly increases and decreases with increasing $P_{el}$, respectively, see [E 11, E 20]. Meaning that for dimensioning microfluidic platforms using SAW, see WP1-RT2, not only the properties of the fluid and the frequency of the SAW but also the $P_{el}$ should carefully be taken into account. Furthermore, optical temperature measurements using the same experimental setup confirm that acoustic streaming and acoustic energy absorption occur at the same time, by showing that the temperature increase within the fluid locally coincides with the velocity jets. A local temperature rise of about 0.85 K with a strong local gradient 2 K/mm was determined, even at a very low power level of $P_{el} = 7$ mW. Time resolved temperature measurements indicate that most of the local temperature increase results from a local heating of the piezoelectric material, probably due to electro-mechanical losses [Fehler! Verweisquelle konnte nicht gefunden werden., E 9, E10].

Inside the acoustic micro mixer manufactured within WP1-RT1, three-dimensional flow measurements were conducted not only to study acoustic streaming at this particular microfluidic application, but also to support the development of a novel model for numerical simulations of acoustic streaming-induced fluid flow within microfluidic devices (see WP1-RT4). This new model allows for deriving the three-dimensional distribution of the corresponding body force by taking material-depending damping and the anisotropy of acoustic waves into account [E 3]. Based on experimental findings made during fundamental investigations, the acoustic micro mixer has been manufactured with proper dimensions and material as well as high-frequency SAWs excited at relatively low electrical power level $P_{el}$. Fully three-dimensional three-component (3D3C) flow measurements were performed using the micro APTV technique [E 1, E 12]. In addition, in situ measurements of the SAW amplitude profile were conducted as described in section 3.2.4, which allowed for a quantitative comparison between flow measurement and simulation. Details can be found in reference [E 1].

**3.3.2 Magnetic and electric fields**

The challenge of the workpackages WP1-RT2 and WP2-RT2 is to effectively combine magneto-hydrodynamics (Lorentz force, [3]) and magnetic field gradients [4] for advanced manipulation of (para-) magnetic fluids or species. To realize the objectives we planned collaboration with a highly successful team at the University of Arkansas (Prof. Ingrid Fritsch group). Teaming up with leading experts on magnetically induced micropumping we implemented designed magnetic field gradient templates below microelectrode arrays [1,2]. Successful prototype assembly and proof of principle in fluid flow manipulation and detection and confirmation of concentration distributions in simple aqueous solutions of paramagnetic ions were realized [E17].

**Magnetically induced pumping**

To prove the concept of fluid manipulation by overlaying the magnetohydrodynamic force and the magnetic field gradient force the following approach is presented: using a redoxsystem common for redox-MHD and combine it with magnetic field gradient templates. The additional gradients are generated by magnetic field gradient templates (gradB-templates) consisting of high moment soft magnetic CoFe stripes or sheets which are embedded in epoxy with 50 µm sheet thickness and 500 µm distance (Figure 11c and d). These samples are grinded to heights of approximately 1 mm and are used as field gradient templates. They are saturated by a permanent NdFeB-magnet placed underneath. The microfluidic chip on top of the magnetic field gradient template consists of the lithographically manufactured Au microelectrodes
(Figure 10b, schematically drawn in Figure 11a, photographed in 11b) in such geometry that a 550 µm wide “channel” exists. In a further step the implementation of the soft and hard magnetic layer system, developed in WP1-RT1 (3.1.2), in the experimental setup (Figure 4) is planned for further experiments realizing in a joint project.

Fig. 10: a) schematic and b) photographed assembly for microfluidic measurement involving MHD and magnetic field gradient force, including microscope and camera to visualize fluid flow, microfluidic chip on top of magnetic field gradient template and permanent NdFeB magnet according to [1,2].

Fig. 11: a) Microfluidic chip design with detailed view in b), electrode width 100 µm, length 250 µm, channel width 550 µm, CoFe c) single stripe and d) three stripes gradB-templates, CoFe sheet width 50 µm, length 4 mm, height up to 1 mm,

Experiments were conducted in a 0.1 M K3Fe(CN)6/0.1 M K4Fe(CN)6 redox couple electrolyte system with 0.1 M KCl supporting electrolyte. One of the redox couple ions, namely the [Fe(CN)6]3- is paramagnetic whereas the other one, [Fe(CN)6]4-, is diamagnetic. The solution for monitoring the fluid flow using video microscopy were prepared by adding 20 µL aliquot of the polystyrene latex microbead solution (diameter 10 µm) to 1 ml of the electrolyte. Then, a fluid flow can be controlled by on-off-switching of a set of 9 parallel micro electrodes (I = 10 µA) in combination with the magnetic field gradient template. A Lorentz force arises due to charge transport in magnetic field and give rise to the fluid flow. Video microscopy illustrates localized “channel-like” flow of the particles; velocity measurements quantify the effects further. The experiment is conducted with and without the field gradient template behind the microelectrode to analyze the influence and interactions of the magnetic forces. To clarify the impact of the gradient template they can be positioned in different distances between the electrodes facing each other to hinder or emphasize the fluid flow. Exemplarily, few summarized results are shown in Figure 12 which stands as a proof of principle for enhanced fluid manipulation by locally strengthened magnetic fields and gradients. Here, fluid velocity between two sets of brightly colored electrodes determined by particle
image velocimetry using “Dantec Dynamic studio” software is compared. In each case a constant current was applied between the electrodes to establish redox processes. The arrows in the pictures represent the fluid flow direction and the color and length their velocity. Highlighted with the black dotted ellipse is the region where the gradB-template is in its position.

Fig. 12: a) PIV analysis of redox-MHD on microfluidic chip with a) no gradB-template and b-d) increasing insertion of a CoFe single stripe (see Figure 11c) gradB-template in-between the gold electrodes highlighted by dotted black ellipse. The arrows represent the direction, their colour the velocity of the tracer particles due to the fluid flow.

Without addition of gradB-templates there is a fluid flow of up to 50 µm/s in the downwards direction (in plane on top of the chip). Using the single stripe gradB-template a local change in fluid velocity above the CoFe stripe is observed. On top of the stripe the fluid velocity is reduced. The increased B-field evokes regions of high velocity shifted to the electrode on the right. This becomes clear as the striped template is further moved into the channel (Figure 12 b,c,d) between the electrodes. Here further quantification is necessary, as these results represent integral values in which the velocity is averaged across the recorded chip segment. A detailed steady 3D numerical simulation for the magnetic field density, the resulting magnetic field gradient around the CoFe stripe and the resulting magnetically induced forces and fluid flow was performed by using COMSOL V.5.2a. The incompressible Navier-Stokes equation with two body forces, the Lorentz force and the magnetic gradient force, was solved in the flow cell. A no-slip boundary condition was applied to all walls including the electrodes and the upper lid. (Figure 13).

Finally the convection-diffusion equation for the tree species involved was solved to simulate the mass transfer in the limiting-current regime. The simulation was performed by our cooperation partner at the HZDR, Dr. Gerd Mutschke (Figure 14).
In conclusion, the change of fluid flow due to overlapping of the Lorentz force and field gradient force has been proved for the first time, and has been verified by numerical simulation.

Fig. 14: Comparison between experimental flow profile (left) and simulation (right) during redox-MHD in superimposed magnetic field gradient (Dr. G. Mutschke HZDR)

**Enrichment of paramagnetic ions**

An unexpected effect of enrichment of paramagnetic ions in regions of high magnetic field gradients has been shown recently in macroscopic cells [4]. The enrichment was studied by interferometry and revealed a time dependent but delayed acceleration of the paramagnetic ions whereby the mechanism is not fully understood. These finding has strong potential for separation of paramagnetic ions and local change of the velocity field as show before. Opposite to the finding before a fast and strong enrichment of paramagnetic ions in magnetic gradient fields without time delay was established qualitatively by fluorescence measurements developed for a micro channel and described in 3.2.1. To downscale the micro system another setup was employed and equipped with Au band electrodes similar to the PDMS channels shown in Figure 4 and 11. A smart approach is to measure the potential change between the electrodes and calculate the change of concentration via the Nernst equation locally and quantitatively. Such a micro channel with two Au band electrodes was filled with MnSO₄ solution. By moving a small permanent magnet underneath one electrode of the channel the high gradient attracts Mn²⁺ ions immediately resulting in measurable time dependent potential differences of about ΔE = 15 mV or time dependent concentration change of Δc = 0.25 M, respectively after 300 s, Figure 15. Switching off the magnetic field and the gradient the potential and concentration relaxes by time. Compared to the results obtained in macroscopic cells, the enrichment is one order of magnitude higher directly at the surface. The effect was slightly improved by fixing a single stripe CoFe gradB-template parallel to and below of one electrode. There is still a lack of explanation with classical physical models for attracting paramagnetic ions in high magnetic gradients. Although the magnetic field gradients are in the range of 100-1000 T/m only particles down to nm scale can be manipulated but not ions since the kinetic energy associated with Brownian motion are orders of magnitude higher than the magnetic energy. New concepts are in discussion to model these problems by atomistic approaches.
Fig.15: Enrichment of Mn$^{2+}$ ions in high magnetic field gradients depicted (left) by potential change and (right) by concentration change; on/off means the magnetic field and its gradients are switched on and off.

Nonetheless the findings to change concentration of ions locally and drive fluid in desired directions by overlapping of the different magnetically driven forces has been realized for the first time which open new applications for sensing, mixing and sorting tasks on small scales.


4. Economic usability

The scientific results, proof of principle concepts and findings obtained within the frame of the project belong to fundamental research. A direct commercial usability was not achieved during the time span of the project. From subsequent joint projects which are in preparation promising results may be expected for economic usability.

5. Cooperation

1. Technical University Ilmenau

Joint project based on the results obtained within the frame of the project to the action of surface microacoustic waves on transport, mixing and sorting results are in preparation.

Title of the DFG proposal: „Strömungs- und Temperaturuntersuchungen an einer mikroakustischen 2D Einzelzellenanordnung“ - principle investigation

Responsible Dr. H. Schmidt (IFW) and Prof. Ch. Cierpka (TUI)

2. University of Arkansas

DFG - Grant to Support the Initiation of International Collaboration, 10th - 22nd October 2016, (HA 8195/1-1)

Topic: Combining magnetohydrodynamic force and magnetic field gradient force for contactless manipulation in highly integrated microfluidic systems

The scientific objective of initiating a collaboration was combining the expertise of Prof. Dr. Fritsch’s research group in developing multifunctional, miniaturized analytical devices and MHD controlled pumping on a picoliter scale with IFW Dresden’s experience in magnetic field gradient force driven electrodeposition and the advanced high moment soft magnetic field gradient templates which shall result in a fruitful collaboration.

To initiate our international collaboration we applied for funding to realize a first proof of principle experiment in joint efforts to successfully obtain preliminary data. These initial results were necessary to substantially particularize a full joint project proposal for research grants and to contribute to preparative publications. Dr. Veronika Hähnel was hosted in the group of Prof. Ingrid Fritsch during the 10th - 22nd October 2016.

Responsible : Dr. M. Uhlemann / Dr. Veronika Hähnel (IFW)
Prof. Ingrid Fritsch, University of Arkansas, Fayetteville, AR, USA

3. Technische Universität Dresden:
Fakultät Maschinenwesen, Institut für Verfahrenstechnik und Umwelttechnik; Professur für Transportprozesse an Grenzflächen, Prof. Kerstin Eckert

The group of Prof. Kerstin Eckert has expertise in interphase phenomena. Joint experiments and discussion contribute essentially to fundamental knowledge. Main contributions arise in the field of enrichment of paramagnetic ions in magnetic gradient fields.

Faculty of Electrical and Computer Engineering, Laboratory of Measurement and Sensor System Techniques, Prof. J. Czarske.

4. Helmholtz-Zentrum Dresden Rossendorf (HZDR): Institut für Fluidodynamik, Dr. G. Mutschke

Due to the long standing and fruitful cooperation particular in the field of electrochemical processes in magnetic fields Dr. G. Mutschke supported the fundamental research performed in WP1-RT2. With his expertise in multiphase flow and interphase phenomena simulations were performed to analyze the influence of the different magnetic forces on flow profiles and enrichment of paramagnetic species in micro channels. Publications to the results of this fundamental research are in preparation.


6. TSI Incorporated, Fluid Mechanics Research Instrumentes, Shoreview, MN, USA.

6. Qualifications

The project was supported by the work students of the TU Dresden and HTW Dresden resulting in successful project theses as well as in different academic final degrees (bachelor and master).

Two PhD theses are in progress supervised by the project leaders and Postdocs.

The fundamental and experimental results of the PhD and the undergraduated work are incorporated in relevant scientific publications and presentations at conferences and workshops.

7. Publications

Peer - reviewed journals:


E 7. V. Haehnel, K. Foysal, G. Mutschke, I. Fritsch, M. Uhlemann: Combining magnetic forces for sensing and contactless manipulation of fluids in microfluidic systems, in preparation, Scientific Reports

E 8. J. Koenig, H. Schmidt, A. Boomsma, C. Kykal, Measurement of the three-dimensional viscous fluid flow and temperature distribution on the macroscopic scale originating from surface acoustic waves, in preparation

Proceedings:


Invited talks


Conferences / Workshops


E 24. F. Kiebert, S. Wege, J. König, R. Weser, H. Schmidt, “Comparison between acoustic streaming induced fluid flow in 3D-simulation and experiment”, Acoustofluidics 2016, Copenhagen, Denmark


E 29. Veronika Haehnel, Christoph Konczak, Jörg König, Margitta Uhlemann, Heike Schlörb, Kornelius Nielsch” Electrodeposited micromagnets for microfluidic applications“, 25. AKES workshop, Dresden

E 30. Veronika Haehnel, Xiao Ma, Christoph Konczak, Diana Pohl, Margitta Uhlemann, Heike Schlörb, „Fe- based Magnetic Alloy Electrodeposition For Thin Films and Template Based Nanostructures“, PRiME 2016/230 ECE Meeting, Hawaii

E 31. Veronika Haehnel, Xiao Ma, Christoph Konczak, Annett Gebert, Heike Schlörb, Margitta Uhlemann, „Electrodeposited magnets for microfluidic application“, EDNANO 11, 2016, Balaton Füred

E 32. Christine Faust, Erik Angermann, Andreas Winkler, Hagen Schmidt, "Multi-purpose SAW-based device for comprehensive cell behavior studies", Acoustofluidics 2016, Copenhagen, Denmark


Patents:

Patent application submitted to DPMA (Deutsches Patent- und Markenamt), Title: Brennstoffzelle (fuel cell), application number: 102017211149.6, 30. June 2017, applicant: IFW Dresden, Inventor: Jörg König, Sebastian Burgmann

Fuel cell with tailored surface acoustic wave fields, increasing the efficiency of fuel cells by exploiting acoustic streaming and acoustic radiation effects originating from surface acoustic waves.

8. Dissemination of the research results

The dissemination of the research results has been carried out by publishing in peer reviewed journals, presenting at international conferences and at workshops. Expected result coming out from ongoing work within the institute partners and in cooperation with the institutions named in chapter 5 will be published as well as in peer reviewed journals.