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# *05.02.11 МЕТОДЫ КОНТРОЛЯ И ДИАГНОСТИКА В МАШИНОСТРОЕНИИ*

# COMPUTER-ASSISTED SPIM-LIKE METHODS (SPIM, MSPIM, MUVISPIM & PIV/ LDV/ LDA/ LDF BASED ON SPIM-LIKE SETUPS) AS NOVEL TOOLS FOR DYNAMIC ANALYSIS OF SUPERCRITICAL FLUID FLUXES AND ORIENTED COMPLEX FLUIDS WITH SOFT MATTER STRUCTURES[1]

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**Abstract.** We propose a novel procedure for the supercritical fluid behavior visualization and measurements based on SPIM or similar microscopic techniques in the rotational micro-capillary tubes. We propose to replace a time-lapse registration by a super-fast registration (using high-speed cameras) for in situ fixation of supercritical fluid dynamics and for slow-motion representations of physical states and transitions, distinguishable using mathematical morphology tools. An elegant version of this method for our purposes includes also a cross-correlation spectroscopy for supercritical fluid behavior analysis Also we propose to use SPIM-like (Selective Plane Illumination Microscopy) setups with 2-objective, 3-objective or 4-objective registration schemes for this purpose in order to perform PIV (Particle Image Velocimetry), LDV (Laser Doppler Velocimetry) / LDA (Laser Doppler Anemometry) / LDF (Laser Doppler Flowmetry) and similar measurements using the above configuration.

**Key words:** SPIM (Selective Plane Illumination Microscopy), MSPIM, Supercritical Fluid Laser Optics, PIV (Particle Image Velocimetry), LDV (Laser Doppler Velocimetry), LDA (Laser Doppler Anemometry) / LDF (Laser Doppler Flowmetry).

# КОМПЬЮТЕРНО-ОПОСРЕДОВАННЫЕ МЕТОДЫ МИКРОСКОПИИ ПЛОСКОСТНОГО (ПЛАНАРНОГО) ОСВЕЩЕНИЯ (SPIM, MSPIM, MUVISPIM И PIV- / LDV- / LDA- / LDF-ИНСПИРИРОВАННЫЕ ТЕХНОЛОГИИ НА БАЗЕ МНОГООБЪЕКТИВНЫХ УСТАНОВОК SPIM-ПОДОБНОЙ ГЕОМЕТРИИ) КАК НОВЫЙ ИНСТРУМЕНТ ДИНАМИЧЕСКОГО АНАЛИЗА СВЕРХКРИТИЧЕСКИХ ЖИДКОСТНЫХ ПОТОКОВ, ОРИЕНТИРОВАННЫХ КОМПЛЕКСНЫХ ЖИДКОСТЕЙ И ЧАСТИЧНО УПОРЯДОЧЕННЫХ СРЕД[2]

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[1] This work was presented on the VIIIth Conf. «Supercritical Fluids (SCF): fundamentals, technology, innovation» and on the International Conference «Fluxes and structures in fluids» in 2015.

[2] Работа была представлена на VIII Научно-практической конференции «Сверхкритические флюиды: фундаментальные основы, технологии, инновации» (2015 г.) и международной конференции «Потоки и структуры в жидкости» (2015).

## COMPUTER-ASSISTED SPIM-LIKE METHODS (SPIM, MSPIM, MUVISPIM & PIV/ LDV/ LDA/ LDF BASED ON SPIM-LIKE SETUPS) AS NOVEL TOOLS FOR DYNAMIC ANALYSIS OF SUPERCRITICAL FLUID FLUXES AND ORIENTED COMPLEX FLUIDS WITH SOFT MATTER STRUCTURES

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**Аннотация.** Предлагается новая идеология объемной визуализации потоков сверхкритических флюидов при использовании установок с несколькими объективами (мультиобъективная микроскопия) в геометриях SPIM, MSPIM, MuViSPIM и им подобных. Измерения производятся в термохимически-стойких трубках / колонках / капиллярах, прозрачных для используемого диапазона излучений. Несмотря на то, что, по определению, сверхкритические флюиды, обладая достаточно высокой плотностью (как в жидкости) и низкой вязкостью, при отсутствии межфазной границы, не характеризуются наличием поверхностного натяжения, имеется ряд технических приемов, базирующихся на внедрении микрогетерогенных сред, а также диспергированных терморезистентных флуоресцентных меток в носитель в количествах, при которых существенного вклада в термические и аддитивно трактуемые физико-химические свойства и характеристики сверхкритической жидкости агент визуализации не вносит. Используя цейтраферные / time-lapse-техники регистрации с достаточными временами интегрирования и строя векторные поля с трассированием (отслеживанием траекторий) одиночных частиц и реконструкцией их характеристик с использованием математических принципов и алгоритмов оптической велосиметрии движения частиц в потоке (в особенности – PIV – Particle Image Velocimetry), возможно отслеживать динамику флюидов в отслеживаемом объеме в 3D-поле зрения мультиобъективной установки с достаточным временным и пространственным разрешением. Аннотируется применимость двух-, трех- и четырех- объективных конфигураций в описанном принципе измерений. Указывается на препятствие к внедрению подобных методов, заключающееся в существенном финансировании, необходимом для закупок необходимого числа специализированных объективов. Предлагается ряд качественно новых более дешевых схем, в частности – замена множества объективов в единой плоскости на вращаемый в этой плоскости один объектив, что заимствовано из более ранней разработки авторов, GMCC_CCI (Goniometric Microscopy in Cylindrical Coordinates for Chromatographic Column Imaging). При внедрении дополнительной схемы, обеспечивающей многоугловое позиционирование плоскости, в которой вращаются тубус и объектив, система преобразуется в многоосную, аналогичную MAGMCC_CCI (Multi-Axis Goniometric Microscopy in Cylindrical Coordinates for Chromatographic Column Imaging). Гибридизация методов велосиметрии и микроскопии, подобная имплементациям данного подхода, реализуемым в «microPIV» (на флюидном чипе), может быть реализована и в SPIM-подобных системах, когда чип может быть размещен между объективами и рассматриваться с нескольких сторон, а материал и геометрия чипа в целом не вносят оптических аберраций и иных артефактов в результирующее изображение и результат его обработки вышеуказанными алгоритмами.

**Ключевые слова:** сверхкритические флюиды, сверхкритическая жидкости, SPIM, MSPIM, MuViSPIM, PIV, LDV, LDA, LDF.

### 1. Introduction

#### *Case I*

In this part, we propose a novel procedure for the supercritical fluid behavior visualization and measurements based on SPIM or similar microscopic techniques in the rotational micro-capillary tubes (such as micro-columns for the supercritical chromatography [1; 2]; Fig. 1–4) or using a rotating multi-objective turret on an spindle (relative to the capillary tube axis). Very little attention has been paid to the supercritical fluid structure microscopy until now, since the major focus is on the confocal studying of the chemical products of supercritical fluid effects on the organic [3; 4] and inorganic matter [5] (but not in situ). The selective plane illumination microscopy method is intended for providing a realistic volume visualization of dynamical objects (such as living cells [6]; Fig. 5–7) as well as standard CLSM techniques. The above microscopic procedure is applied for in vitro embryological studies in time-lapse regimes.

We simply propose to replace a time-lapse registration by a superfast registration (using high-speed cameras) for in situ fixation of supercritical fluid dynamics and for slow-motion representations of physical states and transitions, distinguishable using mathematical morphology tools. An elegant version of this method for our purposes includes also a cross-correlation spectroscopy for supercritical fluid behavior analysis [7].

#### *Case II*

Cryogenic technologies play a significant role in the development of physics of the supercritical states, and particularly, the supercritical fluids.

**Fig. 1.** Old-type supercritical fluid chromatograph (1990)

**Fig. 2.** JASCO system for Preparative Supercritical Fluid Chromatography

**Fig. 3.** Separation using a 5-cm-diameter supercritical fluid chromatography column

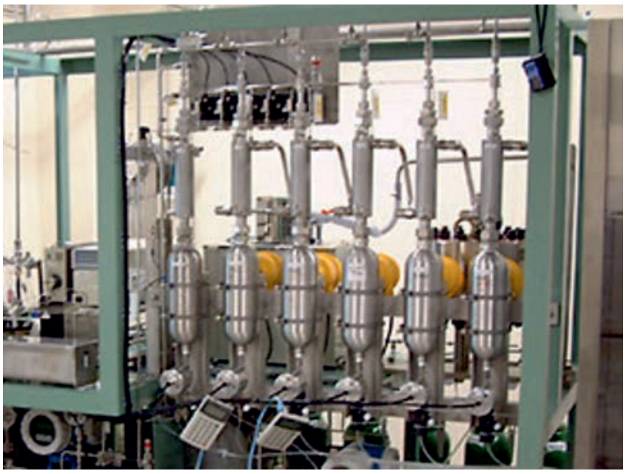


**Fig. 4.** This is liquid chromatography using supercritical carbon dioxide as the solvent. Since supercritical liquid has both gas and liquid characters, the resultant low viscosity allows very high flow rate making it suitable for small to larger (10 kg) separation

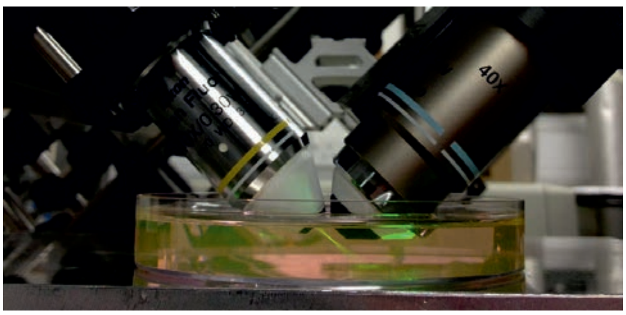



**Fig. 6.** SPIM geometry for non-capillary sample container reading/imaging

The early works considered the cryophysical aspects of the supercritical pressures [8; 9], while at the studies on the supercritical extraction the main attention was paid to the cryogenic cooling of the adsorbent [10] and related problems. From the early 2000-th the research focus was shifted to the cryogenic liquid injection technologies [11–13]. Later the strategy for simulating supercritical cryogenic jets using high-order schemes was proposed [14]. To date there is a clear boundary between the technologies of the cryogenic fluid injection into a supersonic flow field [15], supercritical pressure cryogenic injection [13], etc, and cryogenic spray-freezing technologies [16].

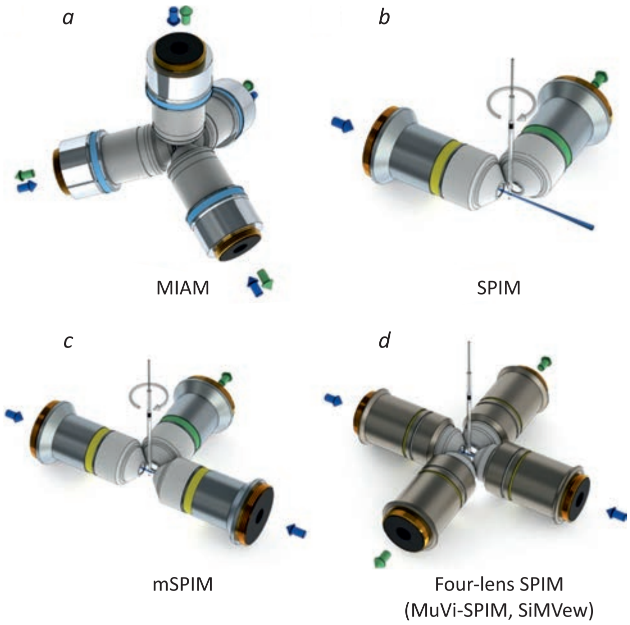
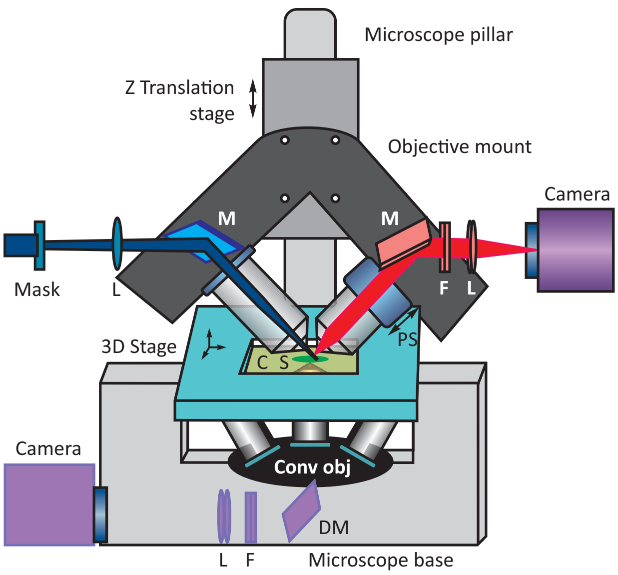



**Fig. 5.** Standard geometries of SPIM-like objective schemes (three-lens MIAM, two-lwnsSPIM, three-lens mSPIM, four-lens MuViSPIM / SiMView (also known as four-lens SPIM))



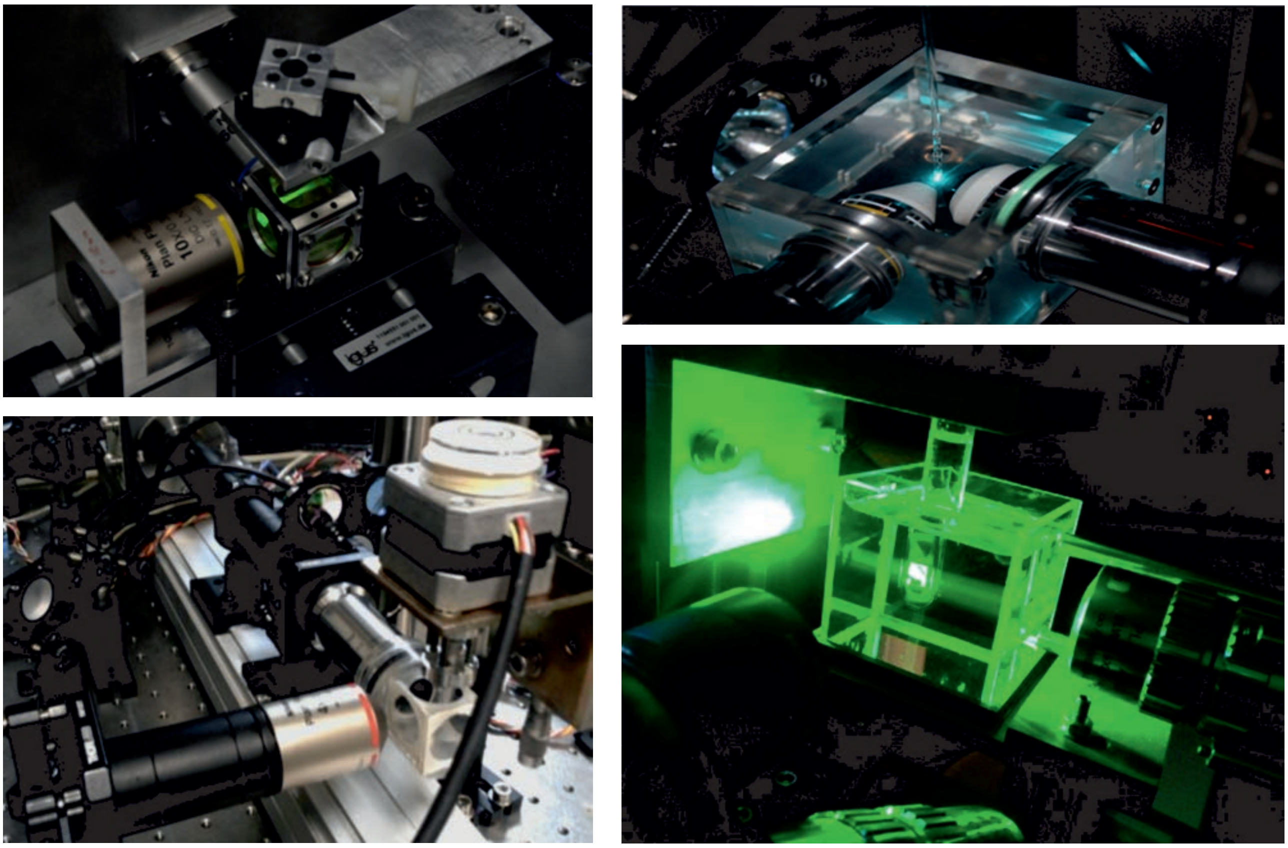
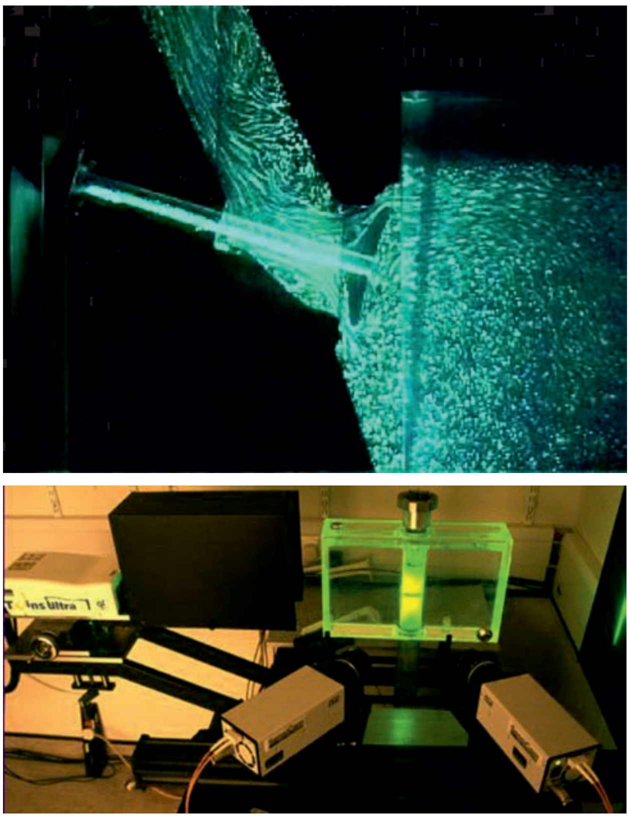


**Fig. 7.** Different geometries of capillary-assisted SPIM experiments and SPIM without capillary sapmle holders



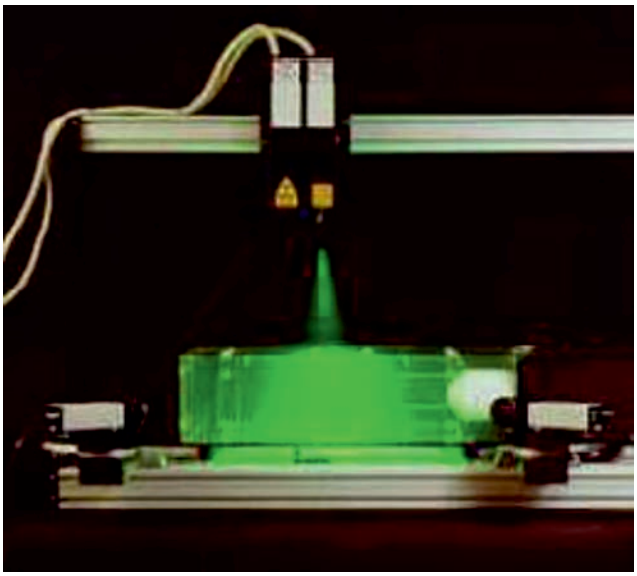


**Fig. 8.** Geometry and setups of/for PIV experiments

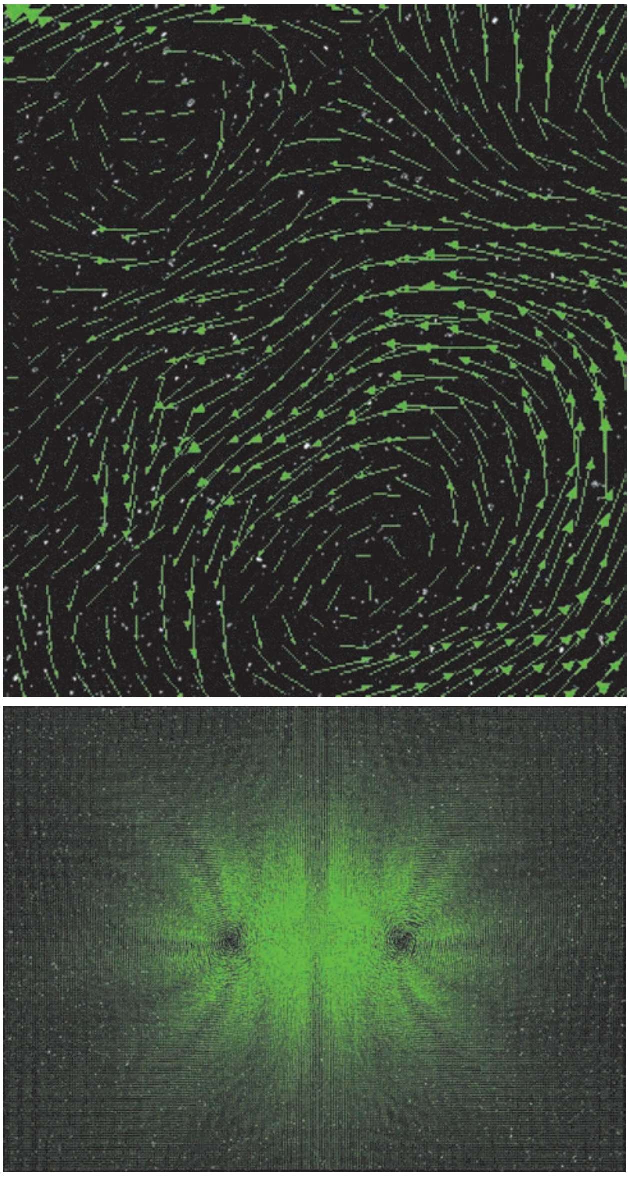


**Fig. 9.** Vector Field Motion Estimation Visualizations of PIV

To date there are no methods for the hydrodynamic jet microstructure 3D visualization and measurements for most of the above methods. We propose to use SPIM-like (Selective Plane Illumination Microscopy) setups with 2-3-4-objective registration schemes for this purpose in order to perform PIV (Particle Image Velocimetry), LDV (Laser Doppler Velocimetry) / LDA (Laser Doppler Anemometry) / LDF (Laser Doppler Flowmetry) and similar measurements using the above configuration.

### *Case 3*

It is well established that many gases, particularly ozone, which is known to be an unstable and poorly soluble agent, possess an increased solubility in supercritical fluids.

It has long been shown that using of supercritical $CO_2$ allows to vary ozone concentration within a wide range and to change its partial pressure [17], wherein $O_3$ concentration can be controlled specrtophotometrically due to its intense absorption band near 200–300 nm. It was also mentioned in the above cited paper that supercritical $CO_2$ effectively inhibits ozone decomposition and the rate of ozone thermal decomposition decreases with the increasing $CO_2$ pressure. There are also special techniques for $O_3$ monitoring in narrow spectral regions including its spatial localization with time resolution for biomedical purposes [18]. We propose to use similar methods for ozone determination in supercritical systems based on SPIM-like ozonometric microscopy.

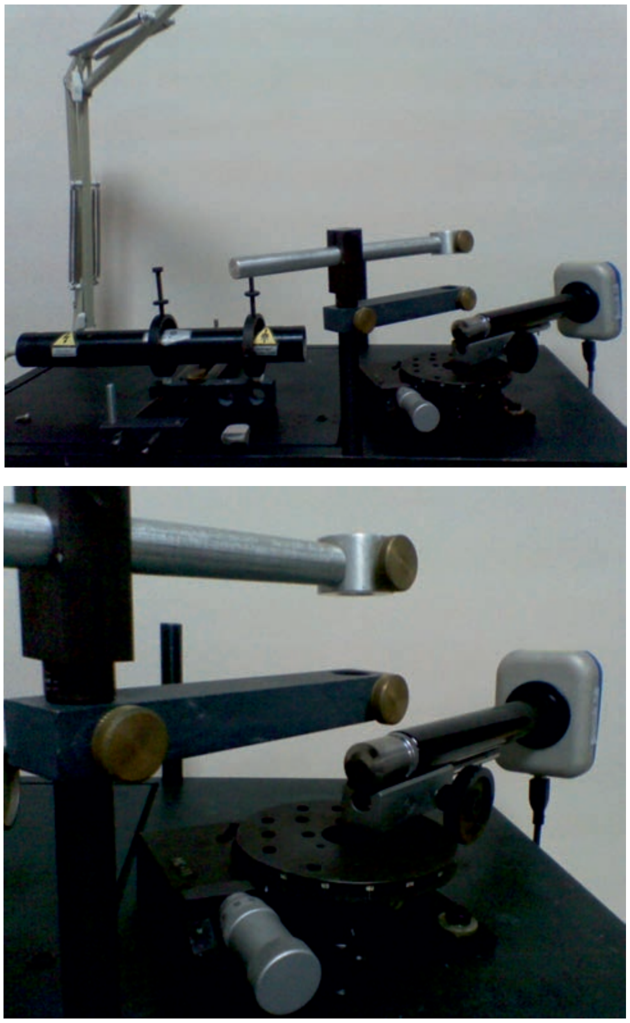


**Fig. 10.** GMCC_CCI (Goniometric Microscopy in Cylindrical Coordinates for Chromatographic Column Imaging)

## 2. Main body

Russian scientists from O.V. Gradow group was propose such concept systems for SPIM-like fluid analysis:

I. SPIM-PIV (Selective Plane Illumination Microscopy + Particle Image Velocimetry for SuperCritical Fluids);
II. SPIM-LDV (Selective Plane Illumination Microscopy + Laser Doppler Velocimetry);
III. SPIM-LDA (Selective Plane Illumination Microscopy + Laser Doppler Anemometry);
IV. SPIM-LDF (Selective Plane Illumination Microscopy + Laser Doppler Flowmetry);
V. mSPIM-PIV (Multidirectional Selective Plane Illumination Microscopy + Particle Image Velocimetry for SuperCritical Fluids);
VI. mSPIM-LDV (Multidirectional Selective Plane Illumination Microscopy + Laser Doppler Velocimetry);
VII. mSPIM-LDA (Multidirectional Selective Plane Illumination Microscopy + Laser Doppler Anemometry);

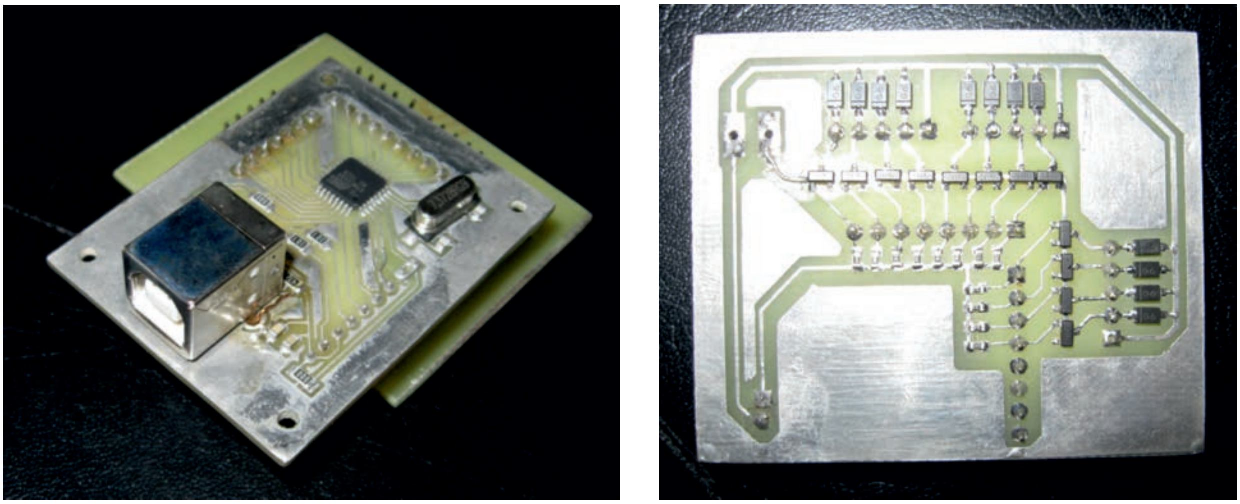


**Fig. 11.** Driver System for GMCC_CCI (author and developer – V.A. Oganessian [19-22]), goniometric optical tomography and neurogoniometry

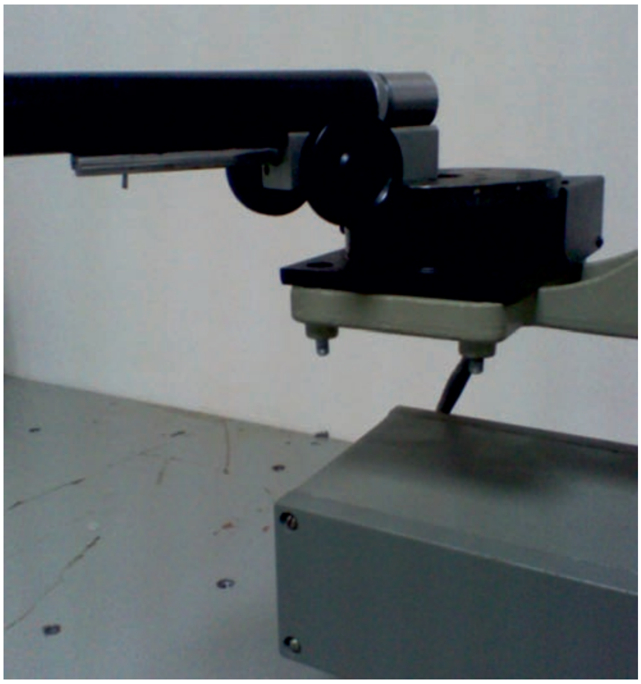


**Fig. 12.** MAGMCC_CCI – Multi-Axis Goniometric Microscopy in Cylindrical Coordinates for Chromatographic Column Imaging
Personal acknowledgment

VIII. mSPIM-LDF (Multidirectional Selective Plane Illumination Microscopy + Laser Doppler Flowmetry);
IX. MuViSPIM-PIV (MultiView Selective Plane Illumination Microscope + Particle Image Velocimetry for SuperCritical Fluids);
X. MuViSPIM-LDV (MultiView Selective Plane Illumination Microscope + Laser Doppler Velocimetry);
X. MuViSPIM-LDA (MultiView Selective Plane Illumination Microscope + Laser Doppler Anemometry);
XII. MuViSPIM-LDF (MultiView Selective Plane Illumination Microscope + Laser Doppler Flowmetry);
XIII. OPFOS-PIV (Orthogonal-Plane Fluorescence Optical Sectioning Microscopy + Particle Image Velocimetry for Super Critical Fluids);
XIV. OPFOS-LDV (Orthogonal-Plane Fluorescence Optical Sectioning Microscopy + Laser Doppler Velocimetry);
XV. OPFOS-LDA (Orthogonal-Plane Fluorescence Optical Sectioning Microscopy + Laser Doppler Anemometry);
XVI. OPFOS-LDF (Orthogonal-Plane Fluorescence Optical Sectioning Microscopy + Laser Doppler Flowmetry);
XVII. GMCC_CCI (Goniometric Microscopy in Cylindrical Coordinates for Chromatographic Column Imaging) (Fig. 10–13).


**Fig. 13.** Simplified Stage for MAGMCC_CCI – Multi-Axis Goniometric Microscopy in Cylindrical Coordinates for Chromatographic Column Imaging

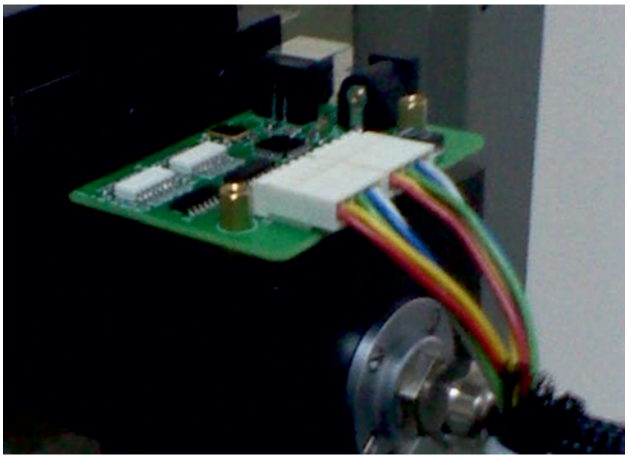


**Fig. 14.** Modernized Version of Driver System for GMCC_CCI and / or GMCC_CCI (author and developer – V.A. Oganessian [19–22]), goniometric optical tomography and neurogoniometry

## 3. Conclusion

It is possible to create some types of low cost setups for 3D optical measurements and characterizations of supercritical fluid fluxes and structures in different analytical processes and chemical technologies.

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