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## "Demands of European neutron users in materials science for the instrumentation of the upcoming PIK neutron science centre"

A workshop taking place in Kiel, Germany, 22.–23. September 2016, organized by Helmholtz-Zentrum Geesthacht, Germany, in the framework of Work Package 4 of the EU project CREMLIN

#### Workshop summary

by Peter Staron

#### Institute of Materials Science, Helmholtz-Zentrum Geesthacht, Max Planck-Str. 1, 21502 Geesthacht, Germany

18. November 2016

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#### 1. Introduction

The workshop was organized within the framework of the EU project "Connecting Russian and European Measures for Large-Scale Research Infrastructures" (CREMLIN; www.cremlin.eu). CREMLIN is a HORIZON 2020 Coordination and Support Action (proposal No. 654 166), coordinated by Martin Sandhop of DESY. Work package 4 (WP4), led by Alexander loffe from FZJ, is about "Science cooperation with the PIK research reactor in the field of neutron sources". The partners in WP4 are Forschungszentrum Jülich (FZJ), Petersburg Nuclear Physics Institute (PNPI), NRC Kurchatov Institute, Helmholtz-Zentrum Geesthacht (HZG), Technische Universität Münschen (TUM), Institute Laue-Langevin (ILL), and The European Spallation source (ESS). Task 4.1 is "Mapping the needs of the European user community".

The purpose of the workshop was to identify the research interests and needs of EU and Russian partners in the PIK project (<u>http://www.pnpi.spb.ru/eng/facil/pik.htm</u>). Since a large variety of scientific questions can be tackled with neutron techniques, which cannot be covered all in one workshop, the workshop was devoted to materials science.

#### 2. Participants

The workshop was organized as a satellite meeting following the German national neutron scattering conference (Deutsche Neutronenstreutagung, DN 2016) that took place in Kiel from 20<sup>th</sup> to 22<sup>th</sup> September 2016. The satellite meeting also took place in Kiel on the campus of the Christian Albrechts-Universität (CAU). European users were not only represented by participants from France, Italy and Sweden, but also by the chairwoman of the European Neutron



Scattering Association (ENSA), Professor Christiane Alba-Simionesco. The connection with the DN 2016 naturally lead to a large number of German participants, but it also brought directors of the leading European neutron facilities (ILL, MLZ, ESS) and other renowned experts to participating in the workshop. In total there were 45 participants, 12 talks, and a fruitful discussion leading to recommendations for the PIK team.

#### 3. Summary of the talks

There were 12 invited talks in the workshop (see program). After an introduction of the project, the first three talks reported the state of the art in the instrumentation for materials science, followed by the ENSA view on the demands of European users, completed by a report on the plans for the instrument suite and about another activity supporting the Russian user community. The second day was devoted to scientific demands for the PIK instruments, expressed in four talks. The following summary is grouped into these sections.

#### **3.1 The CREMLIN Project**

Alexander Ioffe from Forschungszentrum Jülich, the WP4 leader, started with an introduction to the CREMLIN project.

The CREMLIN project is an EU-funded networking activity with a total budget of 1.2 MEUR and duration from 01.09.2015 to 31.08.2018. The CREMLIN consortium is formed by 6 Russian and 13 European research Infrastructures. The 6 Russian research infrastructures are planning or hosting the Russian Megascience projects; the 13 European research infrastructures maintain close scientific collaboration links to the six Russian partners. CREMLIN shall improve and strengthen the relations and networks between European and Russian research infrastructures both at a scientific level and at a research policy level.

The six Russian Megascience projects are:

- 1. Nuclotron-based Ion Collider Facility **NICA** at Joint Institute for Nuclear Research, Dubna (WP3)
- 2. Scientific and Research Reactor Complex **PIK** at NRC "Kurchatov Institute" PNPI, Gatchina (WP4)
- 3. Fourth Generation Special-purpose Synchrotron Radiation Source **SSRS-4** at NRC "Kurchatov Institute", Moscow (WP5)
- 4. Exawatt Center for Extreme Light Studies **XCELS** at Institute of Applied Physics RAS, Nizhniy Novgorod (WP6)
- 5. Super Charm-Tau Factory **STC** at Budker Institute of Nuclear Physics, Novosibirsk (WP7)
- 6. **IGNITOR** Fusion Project at NRC "Kurchatov Institute", Moscow

The objectives of WP4 are:

- To identify the research interests and needs of EU and Russian partners in the PIK project.
- To develop guide lines for a general instrumentation concept for the PIK reactor in cooperation with international partners.
- To help in the development of a state-of-the-art supporting structures at the PIK reactor (sample environment and supporting laboratories, user access system, data management and storage, etc.).

A powerful reactor is only one part of a successful science centre, the supporting structures and services are important as well. The ESS is another upcoming neutron source, built in an exemplary European effort, which can serve as a model for creating an international science centre. It would be wise to strive for complementarity between PIK and ESS instrumentation.

#### **3.2 The PIK reactor project**

The PIK reactor project was introduced by Sergey Grigoriev from PNPI. The reactor had first been critical in 2011, attracting the attention of the Russian government. In 2013 the experimental hall was ready and the idea of an "International Centre for Neutron Research" (ICNR) was approved.

The PIK reactor has a power of 100 MW and it provides a maximum flux in the moderator of  $1.3 \cdot 10^{15}$  n cm<sup>-2</sup> s<sup>-1</sup>. In "phase 1", ranging from 2014 to 2018, a cold neutron source and 12 experimental stations should be build. In "phase 2", ranging from 2017 to 2021, a second cold source and an ultra-cold source together with 20 experimental stations were planned,

for which money had been promised by the Russian government. After the reactor commissioning, planned for 2018, operation will most likely be started with 7 instruments from HZG, 2 own instruments and 3 instruments for nuclear and particle physics. The 7 instruments from HZG were formerly operated at the FRG-1 reactor at HZG and transferred to PIK after the shut-down of FRG-1 in 2010. The first regular operation in user mode is expected for 2019, for which friendly users will be welcome. At that time, there will be a user office and guesthouse available.

There are two research reactors (8 and 10 MW) with a user program running in the Russian Federation, serving the Russian user community. The Russian Neutron Scattering Society (ROSNEITRO) had been established in 2004 and the corresponding conferences have about 150 participants. The European Conference on Neutron Scattering (ECNS) 2019 will take place in St. Petersburg on July 1–5, 2019.

Evgeny Moskvin from PNPI presented current plans for the instrument suite at the PIK reactor. There are plans for 22 instruments to be built until 2024:

- high-resolution powder diffractometer, high-flux powder diffractometer, four-circle diffractometer for thermal neutrons, high-resolution powder diffractometer for cold neutrons, polarized single-crystal diffractometer (POLDI, HZG), texture diffractometer (TEX-2, HZG), residual stress diffractometer (ARES-2, HZG)
- thermal triple axis spectrometer, ditto with full polarization analysis, cold triple-axis spectrometer, multi-functional TOF spectrometer, spin-echo spectrometer
- high-resolution polarized SANS, high-intensity SANS, TOF-SANS, Wide q-range SANS, USANS (DCD), SESANS
- high-resolution reflectometer, high luminosity reflectometer, TOF reflectometer, high luminosity reflectometer with vertical geometry

There had been several workshops dealing with the different instrument classes.

First neutrons are expected in 2018. The ready-to-run instruments are ARES-2, DCD and NERO, while SANS-2 and TEX-2 are still under work. These are former instruments at FRG-1 that had been delivered to PNPI by HZG. However, while the neutron guide design already has been calculated, neutron guides are not yet ordered. In total, 600 m of neutron guides will be needed. An estimated 150 meter per year could perhaps be obtained, describing a bottleneck for finally running the instruments.

#### 3.3 Current state in instrumentation for materials science

Helmut Schober from ILL, Winfried Petry from MLZ and Andreas Schreyer from ESS presented the current state-of-art in instrumentation for materials science at the leading European neutron sources.

The most important instruments for materials science were identified as: 1. powder diffractometer, 2. SANS instrument, 3. reflectometer, 4. strain scanner and texture diffractometer, 5. spectrometer for inelastic neutron scattering, and 6. imaging instruments (radiography and tomography). Powder diffractometers are most important and used by a large part of the user community. The order of relevance of the other instrument types can be debated, but in principle all of them should be there. In addition, PGAA (Prompt Gamma Activation Analysis) and NAA (Neutron Activation Analysis) are also important techniques.

Since parametric studies are sometimes required, high throughput is important for powder diffractometers and SANS instruments. For example, 144 samples can be measured within two days at D20@ILL. As resources are limited, users can be motivated to build dedicated sample environment. Sample environment, service facilities and software are required to get the best out of the instruments; they are more important than the pure flux.

Winfried Petry cited Heinz Maier-Leibnitz who had built two neutron sources and said "*Do it differently!*" Thus, existing facilities should not simply be copied, but an own strength should be developed, becoming complementary to other facilities. Background suppression is important to obtain good signal-to-background ratios. Innovative neutron optics is needed to increase the measureable signal.

Combined techniques get more and more attention. Examples are combinations of diffraction and SANS (e.g. HEIMDAL, DREAM, BEER at ESS) or diffraction and imaging (e.g. HEIMDAL, ODIN, BEER at ESS). Also combinations of neutron and non-neutron techniques like X-rays, dilatometry, DSC or light scattering are possible. However, combined techniques need not be present from day one on, but can be an option at some instruments.

Dedicated sample environment is needed for advanced in-situ and in-operando experiments in engineering materials science. Such experiments are required to study not only materials but also systems like hydrogen storage tanks, batteries, fuel cells, and processes like joining, forging, casting, electrolysis, catalysis, etc.

Since ESS will be the other source coming up next years, it will be important to develop instrumentation and sample environment in a complementary manner to ESS. A focus should be put on techniques where a continuous beam is preferred over a pulsed beam.

#### 3.4 The ENSA view on the demands of European users

Christiane Alba-Simionesco from LLB, chairwoman of the European Neutron Scattering association (ENSA), presented numerous interesting facts about the European neutron scattering community. A few of them are the following:

- Europe has more than 8000 frequent neutron users
- there are 1900 articles per year coming from European neutron research
- there are 30000 instrument days per year in Europe
- 8...10% of European neutron beam time is directly related to industry
- the largest demand is on SANS and powder diffraction, ~40% of beam days, followed by spectroscopy, ~30% of beam days. A future increase in imaging and vibrational spectroscopy is expected.
- The use of neutrons becomes more and more multi and inter-disciplinary

In the European neutron landscape it can be seen that complementary capabilities can be provided by different sources. One aspect is that education and training can be done at small sources.

New sources are important since there will be a significant decline in existing neutron scattering capabilities in Europe within the next 5 to 15 years. Therefore, the use of beam time (*"science production"*) needs to be optimized; this can be achieved with better

preparation of experiments and better training of users as well as better follow-up of experiments. Not only good instruments are needed, but also experts who help the occasional user or the industrial user.

In the discussion, the point was made that strong in-house research is needed at the facilities to be able to provide complex experiments and techniques to external users. Moreover, training is important, not only for students, but also for engineers. Funding will be required for user access of the facilities since Universities often cannot afford the travel expenses.

#### 3.5 Science-driven demands for instrumentation

There were four talks with different examples of research with neutrons, demonstrating a wide range of specific demands for neutron instruments, sample environment and data handling and evaluation possibilities.

Heinz-Günter Brokmeier from TU Clausthal demonstrated the demands for texture analysis, a method important e.g. for materials scientists, engineers, geologists and mineralogists. There are cases where only neutrons enable a reliable and complete description of the texture and texture changes of a material, semi-finished part or workpiece; this is due to the possibility of studying large-grained material and large samples, to the high transmission, reducing the need for corrections and allowing determining complete pole figures. Moreover, non-destructive measurements are possible and sample environments like load frames and furnaces or a combination of both can be used.

Six-axis-robots are state-of-the-art at texture diffractometers. They can be used not only for positioning and rotating the sample, but also for sample changing, allowing for the efficient measurement of large sample series. Also large samples can be positioned, which do not fit into a Euler cradle, the conventional system for rotation a sample around two axes.

Large detector coverage is needed for fast texture measurements, which is required for time-resolved in-situ experiments.

In addition, a school is needed for training PhD students or users from industry in the methods for texture measurement and analysis.

Tobias Panzner from PSI introduced the POLDI instrument. A clever TOF setup is used at POLDI (Pulse Overlap Time-of-Flight Diffractometer) for exploiting pulse overlap for increasing the signal. In addition, a focussing neutron guide is used. POLDI is a materials science diffractometer with a focus on residual stress analysis. Various in-situ experiments can be carried out, among which the bi-axial testing machine is standing out because of its uniqueness. It enables true bi-axial in-situ testing with large samples. There is also a new scintillation detector with increased efficiency.

An important aspect is the software for data reduction and analysis; a MANTID-based software was implemented that is user-friendly and is a take-home software. Thus, users can do data evaluation in their home institute, which should be the standard at all instruments.

Roberto Coppola from ENEA demonstrated experimental needs in the investigation of advanced engineering materials with a focus on nuclear applications. Resistance to radiation

damage is an important topic, as well as welding and joining of metals and ceramics, production routes and thermo-mechanical treatments. Important methods in this field are SANS and GISANS, reflectometry, and residual stress and texture analysis. For all methods it is important that radioactive material can be handled.

For SANS on radioactive steels this means that also the possibility of inserting radioactive samples into a magnet is required. High flux is important since irradiated samples can be small due to dose rate limitations. Polarized SANS has proven to be extremely helpful for the analysis of steels and other metals because the nuclear-magnetic interference term gives additional information about nuclear and magnetic contrasts.

For residual stress analysis, the capability of handling heavy and large samples is important. In-situ experiments for the study of processes like heating, welding or casting are important.

For texture analysis, also the possibility of in-situ heating and mechanical loading is important.

Especially for residual stress and texture analysis, a close-by workshop for sample preparation is helpful. This is required, e.g. for cutting samples or reference samples. In the ideal case, electro-discharge machining would be available for cutting stress-free reference samples.

Andreas Meyer from DLR showed how neutron scattering improves the understanding of liquid alloys and their solidification. This knowledge is required, e.g., for improving near net-shape casting techniques or understanding diffusion of mass in liquid alloys.

An excellent way of studying melts is levitation (e.g. electromagnetic or electrostatic) because it is container-less, thus minimizing background scattering and gives access to extreme temperatures and large undercooling. With such a set-up, self-diffusion and viscosity can be studied.

Quasi-elastic neutron scattering gives valuable results on diffusion constants. Diffraction gives results, e.g., on the structure of the melt via partial structure factors.

A high flux, traded against angular and energy resolution, is essential for performing such insitu experiments with sufficient time resolution on small samples.

#### 3.6 Other forms of European-Russian cooperation

Andreas Magerl from FAU Erlangen introduced an activity he has been pursuing since several years: the so-called "Travelling Seminars". They have emerged from a cooperation with the URAL Federal University (Ekaterinburg) and the URAL Branch of the Russian Academy of Sciences and started in 2008 with the first seminar on Nanotechnology at German universities and research centres. Other seminars followed almost every year at different places in Germany, Russia, France and Sweden.

The idea of the Travelling Seminars is to combine scientific and cultural exchange to the benefit of all partners. One week of teaching materials research shall be combined with one week of travelling to large-scale facilities.

The next Travelling Seminar is planned for 2017 in Russia for up to 50 PhD students and postdocs.

#### 4. Summary of the final discussion

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At the end of the workshop there was a discussion about most important questions in the instrumentation at the PIK reactor.

• Instrument suite. It was generally agreed that among the first instruments there should be a powder diffractometer, a SANS instrument, a reflectometer, a strain/stress and texture diffractometer, an instrument for inelastic scattering, an instrument for imaging (radiography and tomography).

Irradiation facilities, which are already foreseen at PIK, could be attractive.

- **Imaging.** Imaging can be available from the beginning on. Also a cold neutron imaging station should be there quickly. The intensity available at a 100 MW source opens up possibilities.
- **Combined techniques.** On the one hand, combined techniques like e.g. simultaneous diffraction and SANS, combined diffraction and imaging, or a combination of neutron with non-neutron techniques seem to be a developing field and could thus be an interesting niche to fill up. On the other hand, however, in many cases it should be feasible to reproduce conditions so that simultaneous measurements are not really needed.

In addition, optimum sample geometries can be very different for two different methods (e.g. diffraction and SANS) so that it might not be feasible to combine them.

A combined technique can be an option for an instrument upgrade, but needs not be there on day one.

- **Background.** Achieving a low background at the instruments is very important for the performance of the instruments. The importance of low background cannot be over-estimated. The signal-to-background ratio can be increased when only useful neutrons are brought to the sample ("point-to-point optics").
- **Sample Environment.** Only the most common sample environments (SE) are needed to start the user program at PIK. It is important to have such a set of SE from day one to guarantee early success of the facility.
- User service. Users need assistance, e.g. for data analysis, that makes important to append the instrument scientists team persons for data analysis. This support with data analysis will pay off by increased user satisfaction and can pay off by an increased amount of publications.
- **Centres of competence.** When external centres of competence are to be established for providing support for activities at the instruments, like e.g. data analysis, they have to be considered to start early with it. Also the proximity of universities is an asset.
- **Model facilities.** To some extent, ILL can serve as a model facility for PIK. However, the boundary conditions are different and every facility is usually developing its own strategy.
- International facility. If PIK is considered to be a facility for international users ("International Centre for Neutron Research"), then
  - A plan and schedule for the start of the reactor, the user facility and the instruments should be available and published soon.

- Reliable beam time schedules will be very important, otherwise experiments and travelling cannot be planned well. As a consequence, user satisfaction would decrease.
- The local scientific culture will attract users.
- A good facility and good instruments will attract users.
- Local transportation will be important.
- Mail-in service. Such a service is a chance to attract more users. It allows for an efficient
  use of beam time and reduces time and staff needed for training and assisting users. It
  reduces travel costs (which cannot be afforded by some users). The quality of data can
  be increased because the measurements are carried out by experienced staff instead of
  first-time or occasional users.

Such a service will only be reasonable for standard experiments with standard sample environments like, e.g., powder diffraction, SANS measurements, texture analysis, residual stress analysis.

A disadvantage could be that the contact to the user is lost, but this is the case for any scientific analysis technique where samples are just sent by mail (e.g. chemical analyses).

Setting up a professional process chain for a mail-in service from sample reception and labelling over measurement, data reduction, data analysis to reporting and sample storing with full documentation and quality management requires a large effort at the beginning, which should not be underestimated, and only a professional service will be well accepted. However, in total it could pay off with significant additional publications.

• **Miscellaneous.** Handling of radioactive samples is an important issue, e.g. for stress analysis or SANS.

#### **5.** Recommendations for the PIK project

Based on the talks and the discussion, the following recommendations for the PIK team can be pronounced.

- 1. Attracting many users from the beginning on requires that the instrument suite available at day one of the user service includes at least one instrument for each of the following experiments: powder diffraction, SANS, reflectometry, residual stress analysis and texture analysis, inelastic scattering, imaging.
- 2. A low neutron background at the instruments is extremely important. Thus, it is suggested to consider all possible measures for achieving this.
- 3. A collection of standard sample environments should be available on day one of user service.
- 4. It is recommended to provide basic assistance with data analysis to users from day one of user service. In case that such services are planned to be provided by external centres of competence, it should still be a service for users, national as well as international, and it should be available from day one of the PIK user service. If it would be external, good accessibility of the service should be organized.
- 5. User-friendly take-home software for data reduction is one key to a successful user service.
- 6. User confidence in the facility can only be built up when the reactor schedules are reliable. Only reliable schedules enable experiment and travel planning.
- 7. Good local transportation and the quality of user office and guesthouse will be important to attract international users.
- 8. A professional mail-in service could generate more users and increase the output.

The PIK team is in the unique position of being able to adopt and improve existing concepts and include new concepts in their new International Centre for Neutron Research. They can learn from existing sources, copy the standard things that simply have to be there, and above that find their niche and sharpen their profile in complementarity to the other European facilities, especially pulsed sources like ESS. Although this requires significant effort before the user service even starts, it will pay off later.

The organizers of the workshop, the CREMLIN WP4 team and all speakers and participants of the workshop express their best wishes for the success of the PIK team.

#### Acknowledgements

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#### Disclaimer

This report, summarizing the workshop talks and discussions, has been created to the best of the author's knowledge. It may have happened that parts of a discussion have been missed or misunderstood and wrongly summarized here.

#### Workshop photo



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#### Appendix: Workshop program

# "Demands of European neutron users in materials science for the instrumentation of the upcoming PIK neutron science centre"

#### Workshop program

#### Thursday, 22 September 2016

Session 1: Welcome and introduction (Chair: Martin Müller)

14:00–14:15 Martin Müller (HZG, D), Welcome

14:15–14:30 Alexander Ioffe (FZJ, D), Introduction to the CREMLIN project

14:30–15:00 Sergey Grigoriev (PNPI, RU), The PIK reactor project and the Russian user community

Session 2: Current state in instrumentation for materials science (Chair: Sergey Grigoriev)

15:00–15:30 Helmut Schober (ILL, F), The instrument suite at the HFR at ILL with a focus on materials science

15:30–16:00 Winfried Petry (TU Munich/MLZ, D), The instrument suite at the FRM II at MLZ with a focus on materials science

#### 16:00–16:30 Coffee break

16:30–17:00 Andreas Schreyer (ESS, SE), Instrumentation for Materials Science with Neutrons at the ESS

17:00–17:30 Christiane Alba-Simionesco (ENSA, EU), The ENSA view on the demands of European neutron users

Session 3: Towards European users' demand (Chair: Heinz-Günter Brokmeier) 17:30–18:00 Evgeny Moskvin (PNPI, RU), The planned instrument suite at the PIK reactor 18:00–18:10 Andreas Magerl (FAU, D), The "Travelling Seminars" in Russia and Germany

18:20 Leave for dinner place 19:00 Dinner at Restaurant Längengrad

#### Friday, 23 September 2016 (Chair: Andreas Schreyer)

09:00–09:30 Heinz-Günter Brokmeier (TU Clausthal, D), Demands for texture analysis 09:30–10:00 Tobias Panzner (PSI, CH), The Pulse Overlap Time of Flight Diffractometer POLDI – A material science Strain scanner for in-situ uni- and biaxial mechanical testing

#### 10:00–10:30 Coffee break

- 10:30–11:00 Roberto Coppola (ENEA, I), Experimental needs for neutron scattering methods in the investigation of advanced engineering materials
- 11:00–11:30 Andreas Meyer (DLR, D), How Neutron Scattering improves the understanding of liquid alloys and their solidification

Session 4: Discussion (Chair: Martin Müller) 11:30–11:40 Peter Staron (HZG, D), Summary 11:40–12:50 Discussion: Recommendations for the PIK instrumentation 12:50–13:00 Closure

13:00–14:00 Valedictory lunch snack

### CREMLIN Workshop (22./23.09.2016) Participants

No.	Name	First Name	Affiliation	Country
1	Abrosimova	Elena	JCNS	Germany
2	Alba-Simionesco	Christiane	LLB	France
3	Brokmeier	Heinz-Günter	TU Clausthal	Germany
4	Chiappisi	Leonardo	TU Berlin	France
5	Coppola	Roberto	ENEA	Italy
6	Dovzhenko	Gleb	HZG	Germany
7	Eckerlebe	Helmut	HZG	Germany
8	Fenske	Jochen	HZG	Germany
9	Gan	Weimin	HZG	Germany
10	Grigoriev	Sergey	PNPI	Russian Fed.
11	Gutberlet	Thomas	JCNS	Germany
12	Haese	Martin	HZG	Germany
13	loffe	Alexander	JCNS	Germany
14	Konik	Petr	PNPI	Russian Fed.
15	Kononikhina	Victoria	HZG	Germany
16	Kopitsa	Gennady	PNPI	Russian Fed.
17	Krist	Thomas	HZB	Germany
18	Lehmann	Eberhard	PSI	Switzerland

19	Magerl	Andreas	U Erlangen-Nürnberg	Germany
20	Mezei	Ferenc	ESS	Sweden
21	Meyer	Andreas	DLR	Germany
22	Moskvin	Evgeny	PNPI	Russian Fed.
23	Müller	Hans-J.	кіт	Germany
24	Müller	Martin	HZG	Germany
25	Nowak	Gregor	HZG	Germany
26	Ohms	Carsten	JRC	Netherlands
27	Panzner	Tobias	PSI	Switzerland
28	Pavlov	Konstantin	PNPI	Russian Fed.
29	Petry	Winfried	FRM II - TUM	Germany
30	Pranzas	P. Klaus	HZG	Germany
31	Prokhnenko	Olexandr	HZB	Germany
32	Rouijaa	Mustapha	HZG	Germany
33	Sandhop	Martin	DESY	Germany
34	Sazonov	Andrew	RWTH Aachen/JCNS	Germany
35	Scheffzük	Christian	кіт	Germany
36	Schilling	Frank	кіт	Germany
37	Schober	Helmut	ILL	France
38	Schreyer	Andreas	ESS Sweden	
39	Senyshyn	Anatoliy	TU München	Germany

40	Shishkin	lvan	PNPI	Russian Fed.
41	Sokolov	Alexey	exey PNPI	
42	Staron	Peter	HZG	Germany
43	Tarnavich	Vlad	PNPI	Russian Fed.
44	То	Cyrus	CAU	Germany
45	Vezhlev	Egor	FZJ	Germany
46	Walter	Jens	U Göttingen	Germany

CREMLIN Workshop (22./23.09.2016) Participants						
No.	Name	First Name	Affiliation	Country	Signature	
1	Abrosimova	Elena	JCNS	Germany	Ahlen	
2	Alba- Simionesco	Christiane	LLB	France	And	
3	Brokmeier	Heinz- Günter	TU Clausthal	Germany	12/0	
4	Chiappisi	Leonardo	TU Berlin	France	Augo	
5	Coppola	Roberto	ENEA	Italy	Rohado Spre	
6	Dovzhenko	Gleb	HZG	Germany	G. Dovehento	
7	Eckerlebe	Helmut	HZG	Germany	Hell alle	
8	Fenske	Jochen	HZG	Germany	2.192	
9	Gan	Weimin	HZG	Germany .	-E-	
10	Grigoriev	Sergey	PNPI	Russian Fed.	P	
11	Gutberlet	Thomas	JCNS	Germany	Ito	
12	Haese	Martin	HZG	Germany	Mi Heale	
13	loffe	Alexander	JCNS	Germany	i.v. Joffe	
14	Konik	Petr	PNPI	Russian Fed.		
15	Kononikhina	Victoria	HZG	Germany	Breenoh	
16	Kopitsa	Gennady	PNPI	Russian Fed.	144201	
17	Krist	Thomas	HZB	Germany	Merrian Mart	
18	Lehmann	Eberhard	PSI	Switzerland	Ehlen	

	CREMLIN Workshop (22./23.09.2016) Participants					
No.	Name	First Name	Affiliation	Country	Signature	
19	Magerl	Andreas	U Erlangen- Nürnberg	Germany	aluger	
20	Meyer	Andreas	DLR	Germany	Illy	
21	Moskvin	Evgeny	PNPI	Russian Fed.	Moculi	
22	Müller	Martin	HZG	Germany	May 1	
23	Nowak	Gregor	HZG	Germany	Noule	
24	Ohms	Carsten	JRC	Netherlands	dis	
25	Panzner	Tobias	PSI	Switzerland	V	
26	Pavlov	Konstantin	PNPI	Russian Fed.	M.	
27	Petry	Winfried	FRM II - TUM	Germany	W. Petry	
28	Pranzas	P. Klaus	HZG	Germany	f francia/	
29	Prokhnenko	Olexandr	HZB	Germany	M	
30	Sandhop	Martin	DESY	Germany	V	
31	Sazonov	Andrew	RWTH Aachen/JCNS	Germany	Aut	
32	Scheffzük	Christian	кіт	Germany	lett.	
33	Schober	Helmut	ILL	France	Nella	
34	Schreyer	Andreas	ESS	Sweden	All	
35	Senyshyn	Anatoliy	TU München	Germany	MAS	
36	Shishkin	lvan	PNPI	Russian Fed.	Atten	

CREMLIN Workshop (22./23.09.2016) Participants						
No.	Name	First Name	Affiliation	Country	Signature	
37	Sokolov	Alexey	PNPI	Russian Fed.	Come -	
38	Staron	Peter	HZG	Germany	Haven	
39	Tarnavich	Vlad	PNPI	Russian Fed.	A	
40	Vezhlev	Egor	FZJ	Germany	s for the	
41	Walter	Jens	U Göttingen	Germany	A. Walter	
42	Shilling	Frank	<b>k</b> iT	Germ.	Saletts	
43	Mülles	Hans-T.	EIT	Germ.	7	
44	Roujaa	Mustap	Ra 112 G	Germ	12	
45	Mezei	Ferenc	ESS	Schw	F. plese	
46	To	Cyms	CAUS	HK	A18#	
47			19	Contractive		
48		Martin	1120	Germany		
49		Alexander	JOIN .	Barmen		