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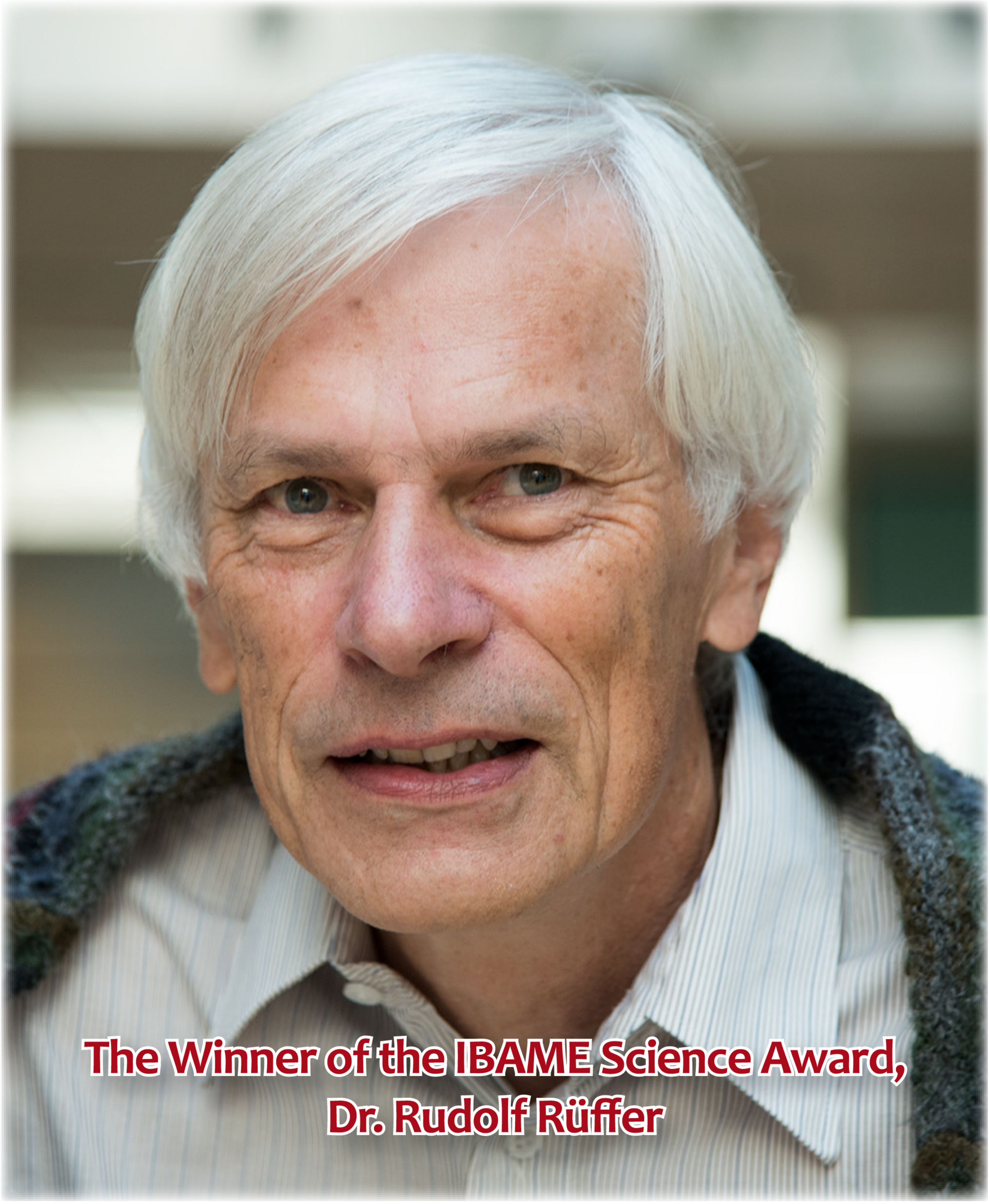

MÖSSBAUER SPECTROSCOPY NEWSLETTER

September 2016

Future challenges and opportunities in Nuclear Resonance Scattering with synchrotron radiation

Rudolf Rüffer (✉)
ESRF-The European Synchrotron, CS40220, 38043 Grenoble Cedex 9, France
Email: rueffer@esrf.fr

No doubt, the new 3rd generation synchrotron radiation facilities around the world have fostered Nuclear Resonance Scattering (NRS) with synchrotron radiation but vice versa their need and specifications were also driven by the requirements for nuclear resonance applications, *i.e.*, nuclear resonance scattering was one of the drivers for those dedicated 3rd generation facilities. To mention are (i) the special timing modes, which are mandatory for time differential applications and also allow for a highly efficient improvement of the signal-to-noise ratio and (ii) the high brilliance of the X-ray source, which allows for high flux at the Mössbauer transition energies and an efficient implementation of X-ray optics to cope with the extreme small energy bandwidth of the involved nuclear levels, *i.e.*, to improve the signal-to-noise ratio. Today we are again facing the challenge to go ahead for a new endeavor in nuclear resonance scattering. However, this time we are much more competing with an impressive number of other successful techniques developed in the meantime at the synchrotron radiation facilities and their active user communities. In the following we want to discuss the special situation of nuclear resonance scattering at a synchrotron radiation facility and to outline our view of possible routes on the future development of nuclear resonance scattering with synchrotron radiation.

Science: The science is the driver for any future developments of the research infrastructure. It is imbedded in the societal and environmental landscape and may be (in Europe) characterized by the following key areas: Health, environment and climate, information and communication technology, energy and economy. Based on those topics the synchrotron radiation facilities and specific the ESRF have identified several emerging areas for the future development such as (1) structural/functional biology, and soft matter – (2) science at extreme conditions – (3) pump-and-probe experiments and time-resolved science – (4) Nanoscience and nanotechnology – (5) X-ray imaging. Conventional Mössbauer spectroscopy already contributes significantly to those areas. Nuclear resonance scattering may add further areas and could play to its strengths, where it exploits the synergy of the outstanding properties of the Mössbauer effect and of synchrotron radiation, such as energy resolution and small size and collimation of the X-ray beam. However, there exist other constraints, which may influence the science applications. Those will be discussed in the following before coming back to the future perspectives in science.

Mössbauer isotopes: In the beginning one argument for Mössbauer spectroscopy at a synchrotron radiation source was the dream to have easily access to all Mössbauer isotopes and may be to find even new ones. It is certainly true that synchrotron radiation allows one to excite any Mössbauer isotope to some extend (depending on cross section) and normally to carry out an experiment. However, the efficient use of the isotope depends on the efficient extraction of the resonant quanta from the synchrotron radiation source such as the X-ray optics and the detector efficiency. During the past years big effort went in those developments and nowadays about 20 isotopes, *i.e.*, half of the known isotopes have been made available at the synchrotron radiation sources. As in conventional Mössbauer spectroscopy ^{57}Fe stands out also in the synchrotron radiation business. This is partly due to its convenient properties but as well due to the rich number of applications in a huge variety of scientific fields.

For those isotopes with low lying nuclear levels (< 30 keV) the necessary X-ray optics and detector systems have been developed in the past and are in principle available. They have to be tailored for the requested isotope. That is mainly a question of budget and priority.

The situation will get different for higher energies. Standard X-ray optics is no longer efficient enough for nuclear resonance techniques and so is the performance of the existing fast detector systems. The competing requirements for high efficiency and fast time response of the detector system seem to be insolvable for high energies. Nevertheless, there are currently several attempts to make those isotopes available. Those comprise new optical schemes such as (i) medium resolution optics for hyperfine spectroscopy, (ii) sapphire backscattering monochromators for hyperfine spectroscopy and in principle capable for nuclear inelastic scattering, (iii) energy analysis of the scattered radiation for hyperfine spectroscopy. On the detector side we find (iv) sophisticated arrangements for stacked assemblies and (v) the investigation of new detector materials. Even exotic ideas have been followed to overcome the problems such as the lighthouse effect by mapping the time to a spatial coordinate. You may imagine, even when one tries to optimize and bundle the efforts, it is a huge development programme. It means one has to present a sound scientific case with a user community behind for those developments in order to get the resources.

Focusing capabilities: The development of focusing devices is a huge endeavor by itself. However, in that case, nearly every technique and community at the synchrotron radiation facilities needs and has requested better and better focusing capabilities. Nowadays, we are able to get routinely focal spot sizes down to 10 nm. Adapting those achievements to the need of the Mössbauer community we may benefit from those developments and may afford and get the focusing systems we want. Due to the intrinsic problems of focusing radiation from radioactive Mössbauer sources we see in this field one of the biggest advantages and perspectives for nuclear resonance applications. It is my surprise that that has so far not really been used and only recently we see a new rush for those small beams in combination with the synchrotron Mössbauer source. I believe we are only at the beginning of the development and will see a huge increase of activities.

Energy resolution: The Mössbauer effect provides a superb energy resolution (*e.g.* 4.66 neV for the 14.4 keV ^{57}Fe resonance). This enabled not only, as in conventional Mössbauer spectroscopy, the access to quasi-elastic scattering, but also to phonon spectroscopy (phonon density of states). Important investigations have been carried out such as under high pressure (magnetism, geo-science, superconductors), on confined geometries (surfaces, interfaces, nanostructures), and on biological, chemical, and disordered samples. Those investigations allowed for a big step forward in the understanding of the underlying science. But they also brought up new questions necessitating the access to an energy regime (10 μeV – 500 μeV) inaccessible today. This regime is just in between that one accessible either by nuclear monochromators or by X-ray crystal optics.

Coherence: Coherence is a big issue at synchrotron radiation facilities and the new free electron lasers will provide a 100% coherent X-ray beam. Many techniques have been developed in order to carry out *e.g.* coherent imaging. This may become a playground for new ideas in nuclear resonance scattering.

Availability: Eventually, we have not to forget a more political aspect, the availability of synchrotron radiation sources. Due to the necessary brilliance nuclear resonance scattering may only successfully be conducted at the four 3rd generation synchrotron radiation facilities as APS (Argonne, USA), ESRF (Grenoble, France/Europe), PETRA III (Hamburg, Germany), and Spring-8 (Harima, Japan). That is quite unique compared to other synchrotron radiation based techniques where the choice of synchrotron radiation sources is much bigger and a suitable facility may be found quite often even on the national level. That has severe consequences regarding the availability of "beamtime" and by that on the research landscape. I still remember the phrase, after my presentation at ICAME 2001 in Oxford, "That are great techniques and results which you have presented, however, that is only for those few privileged people who get beamtime".

Compared to conventional Mössbauer spectroscopy nuclear resonance techniques consist no longer of a "cheap" and powerful lab-based set-up but of a highly expensive source, which is "sold" to the highest bidder in a pretty competitive research environment comprising a large variety of applications. Further, the special timing modes, not really appreciated by the other communities, create some additional headwind. In light of this fact the Mössbauer community needs, as the other communities do, to show up and to develop a roadmap for the needs and the use of the synchrotron radiation facilities. This gets even more important and urgent in the emerging discussion of the various upgrade programmes of the four synchrotron radiation facilities.

In summary, it is my firm belief that we need dreams and visions to move forward. However, as discussed above there are constraints, which may discourage us. In that respect the following view may look pretty conservative; however, it is challenging and will only come true provided the synchrotron radiation facilities undergo a major refurbishment. For that the community needs your active participation and the visions for the future science development. Even when we refer here to the ESRF the outlined science cases should be as well applicable to the other synchrotron radiation facilities.

The future science: The ESRF Upgrade Programme fosters the development of very small X-ray beams. We envisage a decrease of the beam size for nuclear resonance scattering from currently $5 \times 12\ \mu m^2$ to $0.15 \times 0.05\ \mu m^2$ (vertical × horizontal), *i.e.* about 10 000 times smaller, while keeping about 50% of the present intensity. This is a new quality and will help implementing nuclear resonance scattering in the following areas:

- Earth and planetary science: On the one hand we may simulate those conditions relevant for Earth and planetary science in the laboratory. Currently, the 100 GPa regime is routinely reachable by diamond anvil cells. With the smaller beam we may envisage pressures of 700 GPa recently been reported for X-ray diffraction or may even tackle the TPa regime. The combined conditions, high pressure and high temperature, in the inner of Earth and planets reachable by diamond anvil cells and laser heating are even more demanding, however they are in reach. Questions to answer cover the Fe^{3+}/Fe^{2+} ratio in minerals, carbon cycle, magnetic properties, and sound velocities. On the other hand we may investigate "real' samples such as inclusions in diamonds and meteorites. In both cases meaningful investigations need beam sizes of micrometer dimensions or even well below. Of course there exist "big" inclusions; however, most of them are in the micrometer range. Mapping of those are another challenge. In case of meteorites a striking example are the cloudy zones, which are thought to originate from phase transitions due to slow cooling. They may have dimensions well below micrometers. What are their composition and electric/magnetic properties?

- Magnetic properties: As mentioned above, magnetic properties are important for the understanding of Earth and its habitability. Recently, magnetic phases were found to reappear for well known compounds in Earth at higher pressure and temperature. But also the fundamental understanding of magnetism is still lacking in several cases. Nickel, compared to iron and cobalt, stays magnetic up to pressures well above 250 GPa and NiO even at higher pressures. How may we understand those findings and at what pressure the magnetic collapse may happen? Another example is superconductivity. The highest transition temperatures (*e.g.* H_2S at 203 K and 155 GPa) are found for materials exposed to high pressure. How to prove superconductivity under those conditions? To determine that the resistance gets zero might be still straight forward but to witness the Meissner-Ochsenfeld effect is a real challenge. An elegant way is the use of a non-magnetic Mössbauer probe imbedded to the superconductor, which may unquestionably probe that the magnetic flux lines are expelled from the superconductor. With a very small beam one may even investigate the vortex structure and the Meissner hole under those extreme conditions.

- New materials: On the one hand new materials are often synthesized at high pressure and high temperature. In the worst case they are only stable under those conditions. Mapping of the resulting products in the pressure cell is essential. Nuclear resonance scattering might again become the technique of choice to determine the various phases with their electric, magnetic, and dynamic properties. On the other hand new materials may come along as nanostructures and thin films. Besides the small beam the isotope selectivity of the nuclear resonance techniques is an asset for probing those properties with atomic resolution.

Further, we envisage an improvement of our current energy resolution by X-ray optics from 500 μeV to 10 μeV. It is our understanding that that will be achieved while keeping the present flux on the sample, *i.e.*, a spectrograph will replace the monochromator. By that we will cover the entire energy regime from neV with nuclear monochromators up to eV with "electronic" X-ray optics. Some pressing applications may be found in the following examples:

- Quasi-elastic scattering: With the currently available energy resolution we are certain about the extracted phonon density of states starting from about 3 meV. However, even in the simple case of α-iron we are not sure if there is something hidden at lower energies. A striking indication is the different Lamb-Mössbauer factor extracted from nuclear inelastic scattering and nuclear forward scattering/Mössbauer measurements. Meanwhile, nuclear inelastic scattering became an acknowledged technique to determine sound velocities especially under extreme conditions. When the investigated systems get more complicated with bigger unit cells and lower sound velocities like in geosciences and for disordered systems a better energy resolution is mandatory. That will allow escaping the low energetic vibrational modes and the reliable extrapolation to the zero-energy limit (sound velocity).

- Relaxation: The energy resolution of 10 μeV will allow for studies of relaxational dynamics, probably using inelastic X-ray scattering with nuclear resonance analysis. Despite of the limited count rate, these studies will be feasible even with an option of a moderate (~0.3 $Å^{-1}$) resolution in momentum space. This will, for instance, allow for decoupling relaxational dynamics of polymer chains. Other examples might be spin fluctuations, which are supposed to be related to the new iron-based superconductors; visco-elasticity of functional materials such as reinforced polymers. Finally, vibrations and relaxations in the range of GHz and THz are crucial in many bio-related systems, as it is very important in water. There, signatures of *e.g.* low- and high-density water and of interfacial water have been reported using light scattering.

- Dynamical heterogeneities: Dynamic heterogeneity refers to the existence of transient spatial fluctuations in the local dynamical behaviour. The domains of different mobility have no counterpart in the density fluctuations and only appear when dynamics is considered. Dynamic heterogeneity is observed in simulations for virtually all disordered systems with glassy dynamics. Intuitively, as the glass transition is approached, increasingly larger regions of the material have to move simultaneously to allow flow, leading to intermittent dynamics in space and in time. Nuclear resonance scattering, as a slow scattering process and solely determined by the incident wave vector rather than by the mean square displacement as in X-ray and neutron scattering, would allow tackling this question provided the energy resolution will become available.

Finally, like in the early Mössbauer days, there is the class of really "exotic" experiments, where the brilliant Mössbauer community comes up with ideas related to basic phenomena such as relativity theory, reciprocity, and quantum electrodynamics. Those investigations may not be the "daily bread" but highly appreciated and visible.

There is no doubt that the science with nuclear resonance scattering is very alive. Lots of new ideas come up every day in science and instrumentation. However, to let those ideas become reality and to actively participate in the various upgrade programmes of the synchrotron radiation facilities, the activity of the Mössbauer community is urgently needed. We hope that we will stimulate with this short description and outlook the discussion and readiness for future activities.

MEDC Interview

Dr. Rudolf Rüffer was recently honored by the IBAME Science award in recognition of his pioneering contribution to the development of nuclear resonance scattering of synchrotron radiation. On this occasion, he gave an invited lecture titled 'Nuclear Resonance Scattering - Expectations, Visions and their Challenges' at ICAME 2015. MERDJ took the opportunity to interview Dr. Rüffer after returning to his current activities at ESRF, France.

MEDC: Dr. Rüffer Greetings and warmest congratulations from MEDC for your IBAME science award! Receiving such a prestigious distinction is a true honor. How do you feel yourself?

RF: Thank you very much for the warm wishes. Yes indeed, it is a huge honor. I understand that also as recognition of all the great work and the efforts by my colleagues and the entire community engaged in the work with synchrotron radiation and finally that Nuclear Resonance Scattering (NRS) is generally accepted in the Mössbauer community. That makes me happy and eventually as well proud that our visions came true. We were on the right track. I am very grateful to my professor, Erich Gerdau, who had the vision and allowed me to start with an entire new field. I believe to have the chance to develop a new exciting field and technique is a unique experience, which does not frequently happen.

MEDC: IBAME has recognized outstanding achievements in Mössbauer research. Could you please tell us more about some recent highlights of your work, what drive you to the lab?

RF: Of course finally it is the science but for me that cannot be decoupled from the people. Growing up in a multi-national scientific environment and very much focused on development and methodology I am fascinated to interact with other scientist in different fields in order to get to the bottom of the problems and to see if we can solve them with new techniques. This synergy is great and allows for a successful competition also here at the synchrotron radiation facilities with their powerful and challenging techniques. Mössbauer spectroscopy is still unique in a couple of cases and combined with the outstanding properties of synchrotron radiation we can reach a new dimension. Exemplarily we may mention: (i) The focusing capabilities allow one for the first time to investigate in the laboratory not only the structural properties of the inner of the Earth but also their electronic, magnetic, and vibrational properties. Technical speaking it means we are able to apply pressures of several hundreds of gigapascal by diamond anvil cells and temperatures via laser heating of several thousands of degree to the sample in order to simulate conditions well down to the core of Earth. Carrying out those measurements you have to be aware to get each time unexpected surprises. Pushing the beam size further to the nanoworld we will be able to systematically explore, for example, inclusions in diamonds and magnetic properties of cloudy zones in meteorites. In both cases we have the chance to investigate "real" samples of the universe instead to deal with simulations in the laboratory. (ii) Staying with the nanoworld, the combination of different NRS techniques such as Nuclear Forward Scattering (NFS) and Nuclear Inelastic Scattering (NIS) allow for a thorough investigation of nanostructures, thin films and interfaces down to sub-monolayer coverage under UHV conditions. Examples are spin reorientation transitions during in-situ growth of iron layers, magnetic and vibrational properties of surfaces and sub-surfaces of iron and rare Earth layers. (iii) Another example is the better understanding of the so-called Boson peak, a 60 year old quest to understand the "anomalies" around 10 K in the thermal conductivity and the specific heat of glasses. Thanks to the NRS technique, in this case nuclear inelastic scattering with an excellent energy resolution, we could proof that there are no additional vibrational modes and show that glasses have higher specific heat than crystals not due to disorder, but because the typical glass has lower density than the typical crystal. We believe to have better understood the Boson peak. However, it is not the end of the story, new questions popped up, which require an even better energy resolution. You see the technical and methodological developments are driving the science and vice versa.

MEDC: Coming back to your earlier years, what has driven you to science and particularly to synchrotron radiation?

RF: That is a difficult question. When I am looking back I have always tried to do that what I liked most and where I thought to be good enough. Physics and mathematics belonged to those topics. Doing experiments with surprising results was fascinating. Looking then for the topic of my diploma thesis, synchrotron radiation was still in the very beginning and my hometown Hamburg was already at that time a ‘nucleation center’ for synchrotron radiation. I still remember all the pioneers in the field. To get involved in that activity was already an exciting perspective but to combine that with the Mössbauer effect was challenging to say the least. To start with something entirely new was very appealing but of course also very risky.

MEDC: As an active leader in the field, how do you see the evolution of synchrotron radiation related to Mössbauer spectroscopy? We were told it would be soon possible to access mostly all Mössbauer isotopes at a synchrotron. Could you comment on this exciting news?

RF: Sure, we and our colleagues from the other three facilities have demonstrated that in principle all known Mössbauer isotopes and may be even more (such as ^{187}Os) can be accessed. It is mainly a question of scientific needs and priorities. What fascinates me much more is the prospect to strike entirely new paths. And again I believe it is a subtle interplay between science and development. In that respect we are proposing a new nuclear resonance beamline for the new ESRF facility on the horizon in 2020. It shall allow for nanosized X-ray beams and energy resolutions spanning from the neV- to the eV-regime to allow for entirely new experiments. That will be fascinating and we are very much interested to have the community actively participating in this endeavor.

MEDC: Dr. Rüffer. Thank you and again warm congratulation for your IBAME science award on behalf of the whole MEDC staff!

RF: Thank you very much. I would like to take the opportunity to thank all my colleagues who joined me at the ESRF over the years and helped to make that happen. Especially, I want to thank A.I. Chumakov who is the key person since nearly the beginning of my endeavor at the ESRF. And as already mentioned it is a joined effort and thanks to all our collaborators. I believe we and my colleagues at the other three facilities are looking forward to an exciting time with all of you.

IBAME Science Award

The International Board on the Applications of the Mössbauer Effect

hereby grants the

IBAME Science Award

to

Rudolf Rüffer

in recognition of his distinguished work
in the field of the Mössbauer Effect
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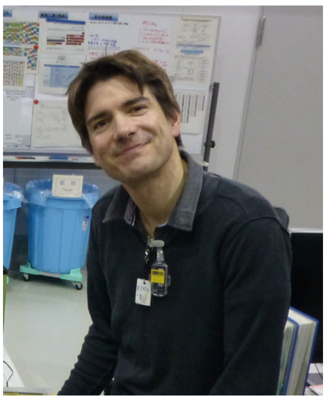
Reflections onthe success of Nuclear Resonance Scattering at the ESRF

Aleksandr Chumakov[1], Alfred Baron[2], Cornelius Strohm[3], Michael Krisch[1]
[1]ESRF-The European Synchrotron CS40220 38043 Grenoble Cedex 9 France
[2]RIKEN SPring-8 Center, 1-1-1 KoutoSayo, Hyogo 679-5148, Japan
[3]Deutsches Elektronen Synchrotron (DESY), Notkestrasse 85, 22607 Hamburg, Germany

Aleksandr Chumakov

Alfred Baron



Cornelius Strohm

Michael Krisch

In September 2015, Rudolf Rüffer received an award from the International Board on the Applications of the Mössbauer Effect "in recognition of his pioneering contribution to the development of nuclear resonance scattering of synchrotron radiation".We congratulate the winner! and hereby present a less official, more personal, appreciation of the winner's contributions, as seen by his colleagues. We feel this is especially important because Rudolf often tends to be quiet, not drawing attention to himself, or his accomplishments, unless there is need.

There are people who discover new phenomena, and there are others who bring them to fruition, making them into tools that are broadly used and appreciated by others. But not many people have done both. Rudolf Rüffer has. Rudolf, when he was Eric Gerdau's student in Hamburg, was the first to observe nuclear scattering using a synchrotron X-ray source, and he then went on to build the world's first dedicated nuclear resonance beamline at the ESRF, ID18. But, in fact, Rudolf did much more than that: his careful and constant effort and outreach have been crucial in allowing synchrotron nuclear resonance scattering to flourish. Long-time members of the community may recall the ICAME conferences in Nanjing, Vancouver, and Rimini, where Rudolf (even as a young postdoc) was able to gather Mössbauer experts for informal meetings and to get from them a detailed wish-list for Mössbauer spectroscopy at a synchrotron radiation source. Those early, and many subsequent, meetings did the job: the synchrotron Mössbauer community emerged from experts in conventional Mössbauer spectrsocopy and has flourished, attracting users from other fields, even those who never have done a conventional Mössbauer experiment.

Other parts of Rudolf's daily work are less visible outside of the ESRF, but have had immense importance, both within and outside the community. Rudolf's efforts to provide the best possible conditions for ID18 users was the motor that helped drive the decades-long work of the Accelerator & Source Division to develop and improve timing modes. The detailed reports and analysis by Rudolf's team on the bunch purity, beam stability, and emittance provided valuable feedback for the machine operation, with ultimately, an amazing (10^{-12}) suppression of spurious bunches. Most recently, on April 26th 2016 Rudolf had a broad and happy smile: that was the day the ESRF delivered 16 bunch mode in top-up operation. Today many other techniques profit from timing modes in scientific fields ranging from time-resolved X-ray diffraction and scattering, to picosecond resolved X-ray spectroscopy, to single-bunch imaging of materials.

Rudolf's interactions with users and staff exemplifies a low-key courtesy. There are no orders, and nearly no complaints. Even in busy times one rarely remembers him giving instructions. He kept external pressure away, giving time and freedom to people working with him. Often staff could choose their own way to contribute to the long term goals he defined. In brief, his attitude might be expressed as "beamline staff should be happy and beamline users should be delighted". Though this statement was never expressed literally, it is appreciated by everyone who has had a chance to work at ID18. Here, you would never hear "what you have measured makes no sense", but the message would be conveyed (still transparently) as "that is strange". Looking back, we also smile that we could have misunderstood a kindly expressed "it would be nice to do this and that" for anything but an express order.

Many came to appreciate Rudolf's understated leadership. Generations of users and young scientists are grateful for the way he would grant support when there was dedication. He would encourage genuine curiosity and careful science over short-term return. The ultimate success and significant new developments for the community reflect his understanding that not only does one need an interesting idea, but also a true passion to persevere. His approach to include people and collaborations at the center of scientific activities, made possible near continuous external funding and led to deeply rooted support of the beamline in various communities. The success of the NRS program emerged as a synergy of the expertise of honorable Mössbauer spectroscopists with the enthusiasm of new researchers entering the field. Generations, old a new, of scientists have passed through the beamline - staff, visitors, users, and friends.

One should also not forget some of the unofficial aspects leading to the success of nuclear resonant project. Science does not give its secrets away easily, and often requires hard work and long hours, even when families, friends, and the beautiful mountains around ESRF might suggest otherwise. A never ending supply of cookies, tea and cake in the 'meeting room' provided precious moments of relaxation, and sometimes exciting discussions among interesting personalities. And until not too long ago - every now and then - we would print and glue a sticker with a new resonance to one of those bottles of a very French beverage and celebrate. And, looking at the table of isotopes, there are opportunities for generations to come. Please come and add your own bottle!

The ESRF nuclear resonance program, under Rudolf's leadership, has continuously improved. There have been many generations of high-resolution monochromators, several generations of detectors and data acquisition systems, and more complex sample environments and in-situ setups than one can reasonably count. As a result, in about 20 years of operation, the program has accommodated more than 300 experiments, served more than 1000 users, led to publications of more than 400 papers - several per year in higher profile journals such Physical Review Letters, Nature, Science - and more than 30 diploma and PhD thesis. This would already be an impressive legacy, but, do note, Rudolf is not yet done!

Congratulations, indeed.

Future Conferences, Symposia, Workshops

November 13-18, 2016
XV Latin American Conference on the Applications of the Mössbauer Effect (LACAME 2016)
Panama City, Panama
http://www.viceipup.up.ac.pa/Lacame2016/

June 5 - 7, 2017
Mediterranean Cofference on the Applications of the Mössbauer Effect (MECAME 2017)
Jerusalem, Israel

September 3-8, 2017
International Conference on the Applications of the Mössbauer Effect (ICAME 2017)
Saint Petersburg, Russia

September 2019
International Conference on the Applications of the Mössbauer Effect (ICAME 2019)
Dalian, China