Template for MAX IV Expressions of Interest

Administrative section

Which type of EoI are you submitting (tick one):

☒ Complete beamline

☐ Experimental stations, instrumentation, or upgrade

☐ Other infrastructure and capabilities: [If Other, please enter a brief explanation here]

Title of EoI:

OPERA – A hard X-ray operando diffraction beamline

Acronym or short name:

OPERA

Additional information (required)

Is there already a Conceptual Design Report or similar?

☒ NO ☐ YES – If yes, please provide a link to document here: [Enter URL]

Does the EoI relate to any areas mentioned in the MAX IV Strategy?

Transformative Science areas	Cross-Cutting topics
☐ Health and Medicine	☐ Imaging
☒ Tackling Environmental Challenges	☒ Dynamics
☒ Energy Materials & Technologies	☐ Data analysis, Machine Learning and AI
☐ Quantum and Advanced Materials	
☒ Ultrafast Science	☒ Other
☐ Accelerator Science	Industry

Is the intended item already funded?
(most should tick no here, unless funding is already approved)

☒ NO ☐ YES – If yes, by which entity?

Is there any cross-dependence between this EoI and others?

☒ NO ☐ YES – If yes, which one or ones?

Abstract

We propose a **flux optimized** beamline which combines the **extreme brilliance of MAX IV** with recent developments in **ultrafast detector technology** and flexible **infrastructure and software** for implementation of advanced **sample environments for in situ/operando experiments** across many research fields. This will constitute the first tailor-made beamline for ultrafast operando diffraction, and will cater to the needs of fundamental, applied and industrial research. Furthermore, a dedicated diffraction beamline will drastically increase the capacity of MAX IV to meet the need for powder and surface diffraction beyond the beamtime offered by DanMAX. Specifically, the strong industrial support for the proposal shows that its implementation will enable broad engagement of Swedish/ Nordic industry in MAX IV. We particularly foresee the development of a strong industry/institute/ university environment around the beamline and around operando diffraction in general. The science cases defined directly target industrial needs and are clearly aligned with MAX IV strategy to provide impact in terms of enabling sustainable development and tackling environmental challenges. The beamline will provide both complementarity and synergies with DanMAX, and special attention has been given to how it will complement the Swedish beamline P21 at PETRA III. As it largely builds on existing MAX IV technology, the technical risk is low and it is expected to yield early scientific results.

Background

There have been previous dedicated efforts towards realizing a hard X-ray diffraction beamline at MAX IV, DiffMAX. While the present proposal partly draws on discussions held during the DiffMAX workshops etc, it is a freestanding separate proposal developed by the group of proposing researchers without direct dependence on previous work.

Scientific Case

The proposed beamline will mainly draw on the extreme brilliance at low-to-medium energies provided by MAX IV. The high flux is critical to reach the microsecond (or even faster) time resolution aimed for, as complex sample environments are expected to provide access to only a small part of the diffraction cones, and the energy range provides general benefits as discussed for specific examples below. Here we list a number of broadly defined science cases, including specific examples of how some of these use the unique capabilities provided by MAX IV. However, beyond the proposed science cases, the beamline will enable possibilities for ultrafast science in many fields. In addition to providing capabilities for ultrafast operando diffraction, the beamline will significantly increase the general **capacity** (in terms of volume and throughput) for diffraction at MAX IV. This is critical for attracting industrial use, as well as providing beamtime matching the very large general need for X-ray diffraction beyond what can be met by DanMAX.

Energy materials: Operando diffraction during operation of energy storage materials and devices under relevant and realistic conditions. Performance of electrochemical devices is determined by highly complex and often inhomogeneous reactions taking place over multiple length and time scales. Powder diffraction is a highly versatile tool that reliably probes multiple proxies for the state of health of these devices. Of particular interest is monitoring the evolving reaction distributions during cycling [1], rapid diffractive mapping of commercial cells [2], high throughput testing of large sample matrixes [3] and long-term battery experiments [4]. The combination of new realistic operando cell designs [5] and a dedicated operando beamline will enable studies on a scale not previously achieved.

Catalysis/surface chemistry: Thermal and electro catalysis at model catalyst surfaces. The foundation of heterogeneous catalysis is the interaction of the catalysts surface with its liquid or gaseous surrounding. Crucial for this interaction is the surface structure. Surface X-ray diffraction (SXRD) is the only technique for investigating surface structures under realistic working conditions [6-9]. Presently, SXRD is limited to samples where the scattering is similar from all parts of the surface. The high brilliance in small X-ray beams available at MAX IV, in combination with a tomographic approach, will enable spatially resolved SXRD measurements of polycrystalline surfaces. This will open new possibilities in the investigation of the relation between surface structure and function in many processes and devices, such as catalysis, electrochemistry, corrosion, fuel cells and batteries, which are crucial for a shift to a sustainable society. The critical angle of total reflection scales inversely with the X-ray energy and hence the energy range of MAX IV beneficial compared to beamlines operating at higher energies, where a very small incidence angle is required to maximize the surface sensitivity giving a high sensitivity to sample misalignment. This sensitivity is significantly reduced at lower energies, which enable more complex in situ/operando experiments with e.g. varying temperatures. Furthermore, operando diffraction using nanoparticles, model powder catalysts or fully formulated industrial catalysts can be used to study catalytic processes under conditions highly relevant for industrial catalyst operation conditions [10,11]. With a proper gas system for transient gas environments, structural (synergistic) dynamics in, e.g., alloyed particles dispersed onto nanocrystalline active supports, can followed and linked to catalyst performance.

Surface processes: Surface/environment interactions, electrochemistry, hydrogen uptake and embrittlement, chemical and mechanical surface modification processes, oxidation and corrosion, tribomechanics/tribochemistry. As an example, combinations of surface sensitive diffraction/ reflectivity/fluoresce and electrochemistry enable operando measurements of metals to study formation, stability and breakdown of surface oxide films that are crucial for corrosion resistance of the metals [12,13]. Surface sensitivity, spatial resolution, and infrastructures for complex sample environments are required to push the boundaries of these multi-technique measurements.

Metals processing: Extremely rapid processes such as additive manufacturing (AM), welding, rapid thermomechanical cycles (e.g impact events), quenching. Successful implementation of AM in critical applications (aerospace, energy) requires extremely controlled processes. The very large number of variables makes process development and validation extremely challenging. Real-time tracking of processes allows fundamental understanding and knowledge/data-driven development. High energies require transmission measurements (which prevents realistic boundary conditions to be achieved) or low incidence angles (giving too large beam footprint relative to laser spot to be relevant), while it has been shown that experiments at lower energies allows exact replication of industrial processes during selective laser melting [14]. As the cooling rate can be as high has 10^3 to 10^6 °C/s, extreme time resolution is required to capture the processes in real time.

Thin films/coatings: Synthesis of thin films by physical vapour deposition (PVD), e.g for wear resistant coatings or thin film solar cell devices, phase/stress evolution during operation. The increased spatial resolution at lower energies will e.g. enable spatial mapping of film heterogeneity during deposition. For wear resistant coatings, the improved surface sensitivity enables studies of the thin layer where interactions between the tool and the work piece takes place during machining [15].

Complementarity with DanMAX: An energy range covering a larger span (7-45 keV) than DanMAX (15-35 keV) to allow studies of light elements, enhanced surface sensitivity and/or *Q*-resolution. Adding the possibility to exceed 35 keV enables operando diffraction of batteries as the high beam/specimen at lower energies can cause ionisation events directly affecting the electrochemical response in e.g. Li-ion batteries or, in a worst-case scenario, lead to electrolyte destruction and cell failure. The focus will be on applications where a limited range in *Q*-space will be tracked at extremely high time resolution, in contrast to the focus of DanMAX which targets acquisition of high-quality data over a large *Q*-range to enable structure refinements. In the proposed ultrafast experiments, the structures and phase transitions are typically known, and data collection over a smaller *Q*-range selected to contain the essential diffraction signals, is sufficient to follow the process.

Synergies with DanMAX: Common infrastructure, software, sample environment and beamline development (similar technology). Synergies in terms of competence and staffing, and a larger internal community at MAX IV.

Complementarity and synergies with P21. Beyond the obvious complementary energy range, OPERA mainly focusses on reflection geometry experiments, where the smaller penetration depth, larger incidence and diffraction angles are useful. However, the key impact of the proposal in this respect lies in the fact that it largely targets the same Swedish user community as P21. Engaging the same user community in both instruments will ensure that each beamline develops in the optimal directions and is therefore to be considered as a guarantee for securing complementarity and synergies between the facilities when it comes to in situ/operando diffraction.

User community and engagement

There is a large and active community in Sweden within the specific research fields targeted by the current EoI. Diffraction is a key technique within the more general “engineering materials” field, in particular connected to different aspects of metals processing, which is in focus across many departments and groups at the proposing universities, KTH, Linköping, Lund, Uppsala and Chalmers, as well as at several other universities. Industrial research projects within CAM2 (VINNOVA funded competence center for additive manufacturing of metals) have already used ultrafast operando diffraction capabilities at PSI, and large-scale infrastructures have been integrated into the next round application as a cross-cutting technique. There are many examples of industry related in-situ corrosion and hydrogen embrittlement research being performed using diffraction techniques. KTH is currently developing sample environments for in situ electron beam melting at DESY, and research groups at Linköping University are frequently performing advanced in situ/operando metal cutting [16] and coating deposition [17,18] research, and develop related sample environments at DESY. Research institutes such as RISE and Swerim are deliberately increasing the efforts to include large scale infrastructures into their strategies and implement it broadly in industrial collaborations, including supporting industry access to neutron and synchrotron facilities. Outside the proposing group in situ/operando synchrotron diffraction is frequently employed at e.g. Luleå University of Technology, Umeå University, Malmö University, Karlstad University, University West and Jönköping University.

Sweden has a long tradition of catalysis related research using synchrotron-based techniques, mostly based on spectroscopy. More recently, XRD and GIXRD have been used to study operando thermal catalysis especially by groups at Lund [19], Chalmers [8] and Malmö University [20]. The OPERA beamline will facilitate access for Swedish users, especially in bringing complex and bulky environments that the groups developed. GIXRD studies of electrochemical model electrodes relevant for electro catalysis is in its initial stages for Swedish users [21,22], but has seen an accelerated use especially at PETRA III. The increased interest in novel electrodes for electro catalysis and especially the electrochemical production of green H_2, would gain a significant boost by the construction of OPERA. Finally, GIXRD has been extensively used by the groups in Lund and KTH, at various synchrotrons to study corrosion [13] and hydrogen embrittlement [12], in collaboration with industry, and obviously OPERA would simplify such investigations.

The combination of structural chemistry and battery science has been a long running tradition in Sweden creating the current position as leaders in fundamental battery research. In particular, operando diffraction experiments have formed a critical part of the research profile in multiple Universities for understanding new cell chemistries [23], well established, commercialized materials [24] and for facilitating development of innovative electrochemical cells leading to spin off companies [25,26]. The relevance of operando diffraction to battery research is emphasized by its significant presence in the BIGMAP project within Battery2030+. Uppsala University has contributed to these efforts with a new cell design focused on long term, high rate and realistic electrochemical cycling [5]. Finally, groups such as the Ångström Advanced Battery Center (ÅABC) and Batteries Sweden (BASE) rely heavily on access to results stemming from operando diffraction studies to support other facets of battery research [27] and provide meaningful information to the various stakeholders.

In summary there is a very strong community and a large user base with pressing needs for both advanced operando experiments and traditional diffraction. Strong industrial support ensures relevance and opens possibilities for future platform development and engagement. Also note that the community in question is also actively engaged in P21 at DESY, which will enable efficient communication and development of complementarity/synergies.

Technical specifications

Insertion device (discussed in detail with Hamed Tarawneh at ID MAX IV group): We foresee an energy range of 7-45 keV in order to complement DanMAX and offer suitable conditions for both light elements and experiments where the lower beam/specimen interaction at high energies is required. The current in-vacuum undulators (IVU) at MAX IV enables access to the entire energy range, but with rapidly decreasing flux at higher energies. The lower flux (reduced time resolution) at high energy is likely acceptable with respect to the major science cases defined in this proposal, as the majority of the foreseen experiments using energies >35 keV would be related to operando diffraction of batteries, where charge/discharge cycles are sufficiently slow. However, increasing the flux at these energies would increase the usefulness for e.g. metals processing applications, and the ongoing development of short period cryogenic permanent magnet undulators (CPMUs) should be considered. CPMUs promises approximately 2-3 times higher flux, with increasing flux gain at higher energies, and reduced line width due to the use of lower harmonic number at a specific energy. Notably the flux gain/line width improvements are significant even in the 15-35 keV energy range and the use of a CPMU could offer further complementarity to DanMAX. In the future preliminary design work a decision must be taken as to which insertion device technology is most suitable considering cost-performance (-risk) trade-offs.

Optics (discussed in detail with Mads Ry Jørgensen at DanMAX): The ability to switch between high resolution-optimized (monochromatic) and flux-optimized (quasimonochromatic) modes to adapt to specific experimental requirements will be essential. DanMAX accomplishes this through a cryo-cooled high-resolution Si (111) double crystal monochromator (DCM, dE/E=1.7E-4@15keV–3.2E-4@35keV) and a water-cooled multilayer mirror monochromator (MLM, dE/E=0.3-1%) for high flux. Beryllium CRL transfocator focusing in the optics hutch as implemented at DanMAX is foreseen, which can focus down to 5x50 μm (at 34.8 keV). Further secondary focusing in the experimental hutch should be explored and the trade-off between secondary focusing and slits/pinholes should be discussed. The wide energy range calls for the use of a multi-strip mirror for higher harmonic rejection, in particular for the MLM. The mirror would also allow collimation of the beam in the horizontal direction for a more symmetric profile. Having all optics in the horizontal direction (enabled by the small horizontal divergence of the MAX IV source) will make for a very stable beam in the vertical direction, which is beneficial for surface diffraction experiments. Intensity losses at lower energies is probably acceptable in relation to the improved vertical stability but must be evaluated.

Experimental stations: The scientific cases defined above can be broadly divided into two categories, powder (PXRD) and surface diffraction (GI/SXRD). While the general requirements on the beam are the same in both cases, the two types of experiments call for different diffractometers. Exact positioning of the sample is less critical for powder diffraction experiment, where instead capabilities to handle large, bulky and heavy sample environments with medium precision is key. For surface or grazing diffraction, on the other hand, accurate alignment is required to maintain incidence angles in the order of 0.1° to 0.5° with an accuracy of 0.005°. We therefore propose two separate experimental stations to optimize the conditions for the two different cases. The PXRD station would greatly benefit from a heavy-duty hexapod combined with a robot (or another similarly flexible solution) for detector positioning. The SXRD/GIXRD station is more dependent on a diffractometer for accurate control of incidence angle and alignment. A branched beamline would be beneficial as it would allow parallel experiments as well as time-consuming setting up of the next operando experiment in parallel with the ongoing one. However, as a branched beamline is both more expensive and technically challenging, this must be evaluated during preliminary design.

Detectors: Detector capabilities is one of the key components of the OPERA beamline. Ultrafast area detectors with acquisition rates in the order of (tens of) kHz are deemed essential. Such detectors are becoming available, where e.g. a Dectris Eiger detector [28] have been operated at 20 kHz with an 8 bit counter depth for 1 s at SLS (PSI) to provide 50 μs time resolution during operando diffraction [14]. Due to the rapid development in detector technology combined with increasing demand for ultrafast detection we do not further define a specific detector or target specifications at this point, but the above 20 kHz is a first benchmark. At the lower energies available at MAX IV, the diffraction angles and signal absorption by sample environments is larger, compared to high energy beamlines. The covered Q-range will be significantly smaller which requires very flexible capabilities for detector positioning to allow the optimal placement for any specific diffraction geometry. This will likely demand more flexibility than can be achieved by a standard detector gantry, but could be achieved by use of an industrial robot (as implemented at NanoMAX).

Infrastructure: In addition to heavy-duty hexapod for PXRD and precision diffractometer for GI/SXRD, in combination with ultrafast detectors with flexible positioning, the scientific case for OPERA relies on the availability of advanced sample environments. Such sample environments require extensive and flexible infrastructure at the experimental stations, which should be considered from the very beginning. We propose that a certain budget is reserved for development of a limited suite of day-one sample environments, defined in collaboration with the user community. Given the long history of involvement of the Swedish user community in sample environment development projects at both MAX IV and other facilities we expect joint efforts to further develop the capabilities based on user-developed equipment. Dedicated efforts should be made to coordinate so that any new sample environment is compatible with other beamlines (in most cases probably DanMAX) to as large extent as possible to maximize the usefulness.

Data flow/visualization/analysis: To fully exploit the potential of operando measurements it is essential to allow close to real-time visualization and on-the-fly data analysis and evaluation. The mode of operation must allow interactive control during ongoing experiments. Closed-loop systems where continuous adaption of the sample environment parameters are automatically controlled based on the real-time feedback is envisaged. This is particularly critical for faster measurements where manual control is too slow to be useful. Again, such development should be considered from the start of the design process to ensure that integrated devices offer the possibility for such developments. These functions and their seamless integration into the physical infrastructure is a key point as this will ultimately define how well the capabilities of the sample environments can be exploited to offer possibilities well beyond existing instruments.

Limitations: The proposed beamline is fully dedicated to diffraction. We do not foresee capabilities for imaging or dedicated small angle scattering. SAXS could be envisaged as a secondary technique for upgrades if motivated, but the limited science case for day one capabilities. We do not expect sample changers etc for capillaries, as this will be available at DanMAX. High-throughput experiments would rather be performed using multi-sample translation stages.

Timeline: If included in the road map, parallel work on securing funding for construction and the exploration of possibilities to form an industry/institute/university consortium should start immediately. More detailed specifications and preliminary designs should start in parallel. The timeline largely depends on the success of the fundraising and consortium formation, but given the relative maturity of the proposed technical solutions (possibly with exception of the CPMU) and technical similarities with existing beamlines at MAX IV, relatively rapid progress is expected once the design part of the work is started. A realistic estimate for an operating beamline is 5-7 years counting from the start of the work to first experiments.

State of the Art / Benchmarking

While sample environments for advanced in situ/operando diffraction are available at many beamlines, there are no directly comparable instruments which are purpose built for ultrafast operando diffraction, including dedicated control software. The targeted capabilities (in terms of time resolution during relevant diffraction experiments) have to our knowledge only been demonstrated at the MS (X04SA) and microXAS (X05LA) beamlines at SLS.

Impact statement

Powder and surface diffraction are key techniques for scientific progress in a multitude of research fields, and the implementation of dedicated operando instruments at 4th generation synchrotron sources is expected to revolutionize the study of complicated processes in real time. While the proposed beamline will enable unprecedented experiments across many fields, the major impact is expected to come from the combination of ultrafast data acquisition combined with advanced sample environments and integrated control software. The ultrafast measurements tie into the cross-cutting **dynamics** theme defined in the MAX strategy draft, and will enable direct impact in the areas **tackling environmental challenges** and **energy storage and technologies**.

Time-resolved understanding of industrially relevant processes leading to accelerated, knowledge-driven innovation and development contributing to improving sustainability in the short and long-term perspective through e.g. (i) development of sustainable materials and processes as well as energy and resource efficiency in processing and application, (ii) enabling technology transitions for e.g. lightweight engineering and electrification, (iii) supporting development of batteries, energy devices and energy storage materials, (iv) surface engineering for catalytic processes and (v) development of materials and surface protection for e.g. biofuel combustion and hydrogen embrittlement mitigation in hydrogen infrastructures and fossil free steel production.

Furthermore, the proposal directly contributes to the quantitive goals for **industrial use** specified in the MAX IV strategy draft. Considering that close to 50 % of the pilot projects for industrial use of large-scale infrastructures related to synchrotron investigations funded by VINNOVA contained diffraction as (one of) the main technique(s), increasing the proprietary use to 5 % (from present 1.5 %) and collaborative industry/academia or industry/institute use to 40 % (from present 20 %) will undoubtedly require diffraction capacity beyond what is available at MAX IV today. Particularly in the context of impact and sustainable development goals it should also be acknowledged that short term impact can only be achieved through industrial R&D, which, according to the above, will require access to sufficient capacity for both conventional and advanced in situ/operando diffraction.

Proposers

SPOKESPERSON

Magnus Hörnqvist Colliander, Department of Physics, Chalmers University of Technology

PRINCIPAL INVESTIGATORS

As the proposed beamline represents a wide range of applications and science cases, we propose a larger PI group with different specialties, which will be coordinated by the spokesperson. This is expected to be subject to changes when a more formal and detailed structure can be set up. Nevertheless, the involvement of all listed PIs is critical for the initial concept development.

- **Magnus Hörnqvist Colliander**, Department of Physics, Chalmers University of Technology
  PXRD, metals processing
- **William Brant**, Department of Chemistry, Uppsala University
  PXRD, batteries, energy materials and devices
- **Johan Gustafson**, Department of Physics, Lund University
  SXRD/GIXRD, catalysis, electrocatalysis
- **Lina Rogström**, Department of Physics, Chemistry and Biology, Linköping University
  GIXRD, thin films/coatings
- **Jinshan Pan**, Department of Chemistry, KTH
  GIXRD, electrochemistry, metals/applied materials

TECHNICAL LEAD

At this early stage no individual technical lead is identified, this role will be assigned if the EoI is included in the roadmap and more dedicated efforts can be made towards a conceptual design. The general technical description has been discussed with and reviewed by **Mads Ry Jørgensen** (DanMAX) and the insertion device aspects has been discussed with **Hamed Tarawneh** (MAX IV ID group).

ADDITIONAL CO-PROPOSERS

In addition to the defined PI group the following group of co-proposers have been, and will continue to be, deeply involved in the development and implementation of the proposal:

- **Edvin Lundgren**, Department of Physics, Lund University
- **Jens Birch**, Department of Physics, Chemistry and Biology, Linköping University
- **Peter Hedström**, Department of Materials Science and Engineering, KTH Royal Institute of Technology
- **Martin Sahlberg**, Department of Chemistry, Uppsala University
- **Per-Ander Carlsson**, Department of Chemistry and Chemical Engineering, Chalmers University of Technology

SUPPORTING COMMUNITY

The EoI has been discussed with several partners at universities, institutes, and industries. The following organisations have expressed official support for the proposed beamline Organisations marked with * have chosen to supply official letters of support to emphasize the strategic importance. The signed support letters can be downloaded through this link:
https://chalmersuniversity.box.com/s/ky4mzmpeohn63hjca46jihg0p81nzvya

- GKN Aerospace Engine Systems*
- SECO Tools*
- Sandvik AB*
- SSAB*
- Siemens Energy*
- Linde Gas
- Volvo Car Corporation*
- NorthVolt*
- OVAKO
- Swerim*

- RISE Research Institutes of Sweden
- Competence Center for Additive Manufacturing – Metal (CAM2)*, which in turn represent close to 30 individual member organizations
- Competence Center for Catalysis* (KCK)
- Structural Chemistry, Uppsala University*
- DanMAX beamline at MAX IV*

A broad support from the intended community of academic researchers is shown through the list of almost 50 individual researchers who have registered their support for the EoI. The list of names and affiliations can be downloaded through this link:
https://chalmersuniversity.box.com/s/z4ltrxth7siaqewk2cyyv80idtcxkcet

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MOST RELEVANT PAPERS (1-10):

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