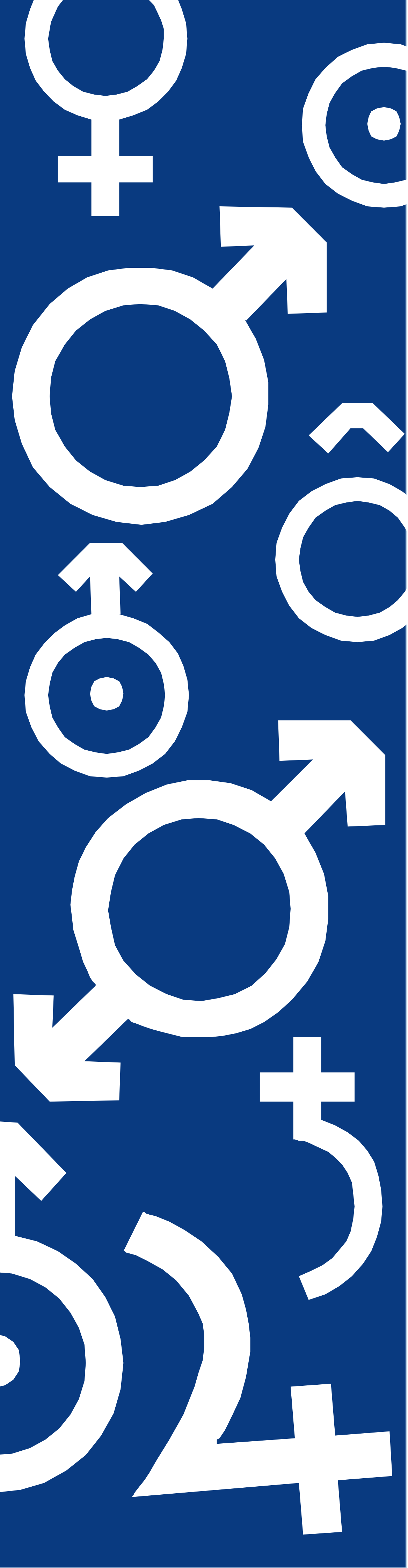

RESEARCH REPORT

Synchrotron radiation for industrial applications

David Malmsström & Jonas Gurell

11582

swerea | KIMAB

Swerea KIMAB AB ● Box 7047, SE-164 07 Kista, Sweden
Tel. +46 (0)8 440 48 00 ● Fax +46 (0)8 440 45 35 ● kimab@swerea.se ● www.swereakimab.se

Title: Synchrotron radiation for industrial applications
David Malmsström & Jonas Gurell: David Malmsström & Jonas Gurell
Report No: 11582
ISSN: 1403-848X

Date: 15.12.2014
Pages: 8
Language: English

Report – Swerea KIMAB AB
ISSN: 1403-848X
Report: 11582

Title: Synchrotron radiation for industrial applications

Author: David Malmsström & Jonas Gurell

Swerea KIMAB-Project number: 11582
Research department: Material analysis & monitoring

Distributions: Jernkontoret

Swerea KIMAB AB
Box 7047 SE-164 07 Kista, Sweden
Tel +46 (0)8 440 48 00, Fax +46 (0)8 440 45 35, kimab@swerea.se, www.swereakimab.se

Swerea KIMAB is a leading corrosion and metals research institute. Swerea KIMAB develops and improves materials and processes for material production and studies the behaviour of new materials in process engineering and mechanical structures.

Research and development work takes place in close co-operation with Swedish and international companies in the steel, metals, electronics, engineering, paper, vehicle, manufacturing, plastics and power industries. For our customers, the results of our work should be a good investment for future revenue.

swerea | KIMAB

Swerea KIMAB AB
Box 7047, SE-064 07 Kista, Sweden
Tel +46 (0)8 440 48 00, Fax +46 (0)8 440 45 35,
kimab@swerea.se, www.swereakimab.se

Synchrotron Radiation for Industrial Applications

David Malmström [a] [*] and Jonas Gurell [a]

Large investigations have been put into building and renovating new synchrotron radiation sources. With over 40 new facilities within the last 25 years, synchrotron radiation has shown to be an attractive technique in order to study the properties of steel materials *in situ*. Thanks to the visual 3D pictures cracks and corrosion could easily be seen and studied and to the power of the high energy bulk properties could investigated in their true nature. Despite the few industrial articles published, it can be said that there is an interest in synchrotron base light (especially X-ray spectroscopy) coming from the industry indicated by the many initiatives and enhancement programs taken by the different facilities around the world.

Keywords: Synchrotron Radiation; Industrial Applications; Steel, X-ray

[a] Swerea KIMAB, Isafjordsgatan 28 A, 164 40 Kista, Sweden
[*] Corresponding author, email David.Malmstrom@swerea.se

1. Introduction

Synchrotron light or synchrotron radiation is a source of brilliant light that scientists have used for 50 years to gather information about the structural and chemical properties of materials at a molecular level. As early as 1924 a Swedish physicist, Gustav Ising, proposed a theoretical concept of accelerating particles which was later shown as a proof-of-concept by the Norwegian Rolf Wideröe and is today known as the betatron. The first basic idea of a synchrotron was mentioned by Mark Oliphant working at the University of Birmingham, UK. The idea was to keep the beam in an orbit of constant radius by an annular ring of magnetic field. In 1945 new and important principles was discovered by Vladimir Veksler in the Soviet Union and Edwin McMillian in the United States which lay the ground to be able to build the first synchrotron. Frank Goward, a working physicist at Woolwich Arsenak Research Laboratory, UK, bought a betatron to inspect by X-ray the unexploded bombs in the streets of London following World War II. After reading about the synchrotron idea he converted the betatron into a synchrotron using a rudimentary accelerating electrode. However, the first synchrotron built as such was made by a team at General Electric Co. in New York, U.S. on April 24, 1947.[1]

Today, there are now about 50 operational rings in 17 countries, where of 10 are the latest third generation synchrotron radiation sources. Of the totally 50 rings in use, 40 are built in the last twenty years. Already since the pioneering work at DESY in Germany in the seventies, Europe has played the leading role in the development of the field by the construction of dedicated XUV sources at BESSY (Germany) and ACO (France), the third generation synchrotron radiation source in e.g. ESRF (France), ELETTRA (Italy), BESSY II (Germany) MAX II-IV (Sweden) and the construction of the free-electron X-ray lasers at DESY (Germany).

2. How does the synchrotron work?

Synchrotron light is produced by charged particles (typically electrons) when accelerated close to the speed of light. By combining several straight sections in a circular formation by bending the electronic path using arrays of strong magnets higher photon energies (the shorter wavelength) can be achieved. The radiation can contain energies in a broad spectrum from bright infrared (IR), ultraviolet (UV) to hard X-rays. Using a monochromator the photon energy of desire can be selected depending on application.
Except being tunable regarding photon energy the synchrotron is unique in many ways:

- The radiation intensity emerging from the storage ring can be up to a billion times greater than a typical laboratory X-ray source
- Extremely fine beams, down to just a few micrometers across
- Highly collimated beam

General synchrotron radiation sources consist of a few key parts (see Figure 1): electron gun, linear accelerator (linac), a booster ring, a storage ring, beamlines and the end cabins. The electron gun, as the name implies, creates electrons that are fed into the linac. Using radio frequency fields the electrons are accelerated to almost the speed of light. In the booster ring the acceleration of the electrons are then further increased (99.9999985 % of the speed of light). Using magnetic fields the electrons can be directed around the ring, as well as force the electron into a fine beam, and finally direct bunches of electrons to the storage ring ones they have enough energy to produce light. Once in the storage ring, the electrons will circulate between four to twelve hours and produce photons every time the dipole magnets changes.

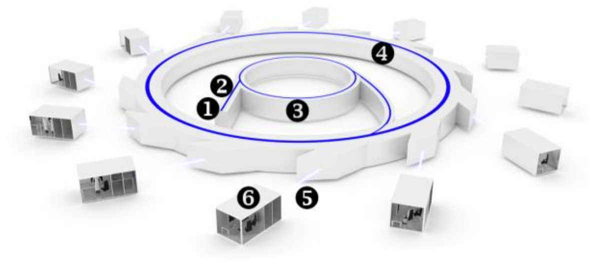

Figure 1. A schematic picture showing and overview of the synchrotron radiation source: electron gun (1), linear accelerator, linac (2), booster ring (3), storage ring (4), beam lines (5) and end stations, including the optics cabin, experiment cabin and control cabin (6).

Even though the ring looks circular, the storage ring consists of several straight sections, where each turn is followed by a photon port which allows the light to travel down the beamlines into the end stations. The end stations consist of a series of cabins, where the first one is the optic cabin containing slits, filters, mirrors and monochromators. Close to it is the experiment cabin with the sample of matter and the detector. Last in the line is the control cabin where the scientists can monitor and control the experiments and data acquisition.

3. Industrial applications

As the synchrotron provides high intensity light across a wide spectral range, it can be used to understand the underlying properties and structures of matters and physical, chemical, geological and biological processes, thus of immense importance in numerous of industries. The semiconductor industry, including lithography, uses synchrotron radiation to study the material interface and production issues. The chemical industries though, study properties such as stress or texture of various materials and products as well as the actual chemical reactions in situ[2,3]. Perhaps the most widely known industrial application of synchrotron based research is the work made on macromolecular biology, in particular proteins and pharmaceutical drug discovery [4]. With the power of x-ray diffraction atomic arrangements and 3D-structures of proteins and nucleic acids can be revealed and understood. It is especially the rapid data collection on very small crystals at higher resolution, in comparison with a standard laboratory x-ray source, that is appreciated.

Unfortunately, most of the commercial synchrotron radiation research carried out, by or for the industry, is confidential which makes it difficult to review the industrial work in a comprehensive manner. However, one might speculate that their work is mostly concentrated into what is called strategic research. That would include studies on product and processes rather than fundamental research.

The European Synchrotron Radiation Facility (ESRF) is supported and shared by 20 countries and is to date the most powerful synchrotron radiation source in Europe. Only on the micromolecular crystallography beamlines more than 10 000 industrial samples are processed. Non-destructive 3D X-ray images can be obtained in situ at temperature ranges from -60°C to +1800°C and compressive and tensile stress and fatigue can be investigated. However, only a few percentage of the total usage time are spent on industrial applications at ESRF. Though, a synchrotron radiation source named ANKA in Germany is purely specialized on the needs of industrial customers. The National Synchrotron Light Source (NSLS), located in USA, is another scientific research facility that focuses on industrial and commercial studies. A proof of this was shown by their industrial enhancement program that was launched in order to enhance industrial research at NSLS as well as address the needs from industrial and commercial users. The beamlines that are the most frequently used by industrial users, thus will have the most time set aside for industrial experiment (up to 10 %), are the powder diffraction, X-ray absorption spectroscopy, small angle X-ray scattering and thin film X-ray diffraction and reflectivity beamlines[5].

4. Synchrotron light used in steel research

Synchrotron radiation can, as previously mentioned, be used for many different applications and areas. One of the benefit with the technique, beyond its good sensitivity and concentration profile possibilities[6–9], is for instance its ability to directly observe the transformation of ferrite, austenite and sigma phases during heating and cooling of duplex stainless steel [4] and oxide dispersion strengthened steel[12]. X-ray diffraction from a synchrotron radiation source has the benefit over traditional optical metallographic techniques by being able to monitoring microstructural changes with e.g. temperature, strain/stress and time monitored in situ (in real time in situ), thus the full transformation lapse as they occur[10,11,13–15]. These type of investigations has shown that thermodynamic calculations falsely predict the actual dissolution temperature to a significant degree based on the amount of austenite, ferrite and sigma[10].

The *in situ* three dimensional (3D) high energy X-ray diffraction (HE-XRD) method could efficiently be used to measure and characterize the thermal stability of retained austenite and monitor martensitic transformation kinetics of individual austenite grains in transformation induced plasticity (TRIP) steel as a function of temperature and/or applied stress[16–18]. TRIP steel is widely used in many industrial applications due to its good combination of strength and plasticity in addition to the excellent corrosion resistance[19–21]. A high resolution X-ray diffraction *in situ* experiment with a far-field detector has just recently demonstrated that it is possible to obtain a full in-depth characterization of individual metastable austenite grains within a multiphase TRIP microstructure[22]. Due to far-field detector and the high resolving power, individual Bragg reflections could be studied which facilitates resolved subgrains in austenite grains prior to the martensitic transformation.

Synchrotron based X-ray computed tomography is excellent for indicating not only stress/strain but also internal cracks and microstructural damage (including inclusions and voids) by three dimensional visualization (*in situ*)[23–27]. In order to measure stress, deeper than just near the surface and to avoid overlapping reflection (as with conventional X-ray), high energy synchrotron based radiation in energy dispersive mode has successfully been applied cold-drawn pearlitic steel rods to determine residual stress profiles[14]. Residual stress could arise as a consequence of inhomogeneous plastic deformation associated to cold-drawing and may influence the mechanical properties of the steel material leading to e.g. corrosion cracking or fatigue. Rolling contact fatigue cracks in high strength steel could be observed by synchrotron radiation computed laminography (SRCL) of specimens with a thickness of 1.0 mm. SRCL imaging has shown to be a powerful technique for exposing, but also preventing, cracks and its initiation and propagation in bearing steel and plate specimens with different thickness[24].

Atmospheric corrosion give rises to maintain steel products and avoiding degradation of steel materials. Outdoor-exposure experiments have been performed during the last hundred years, showing that a rust layer formed on a steel surface often control the atmospheric corrosion behavior. However, still very little is fully known about the initial formation process of each rust phase during atmospheric corrosion. Yamashita *et al.*[28] showed how white X-rays from an energy dispersive X-ray diffraction spectroscopy facilitated by Spring-8 (Super Photon Ring – 8 GeV at Japan Synchrotron Radiation Research Institute) was powerful enough to capture initial corrosion products. Corrosion roughened surfaces can however be analyzed with other wavelength than X-ray, such as far-infrared (FIR). An Australian team showed how synchrotron-sourced FIR spectroscopy could be applied to study corrosion-roughened surfaces at variable incidence angles[29].

Figure 2. Corrosion could be a severe issue, however, energy dispersive X-ray diffraction spectroscopy and far-infrared X-ray spectroscopy has shown to be valuable synchrotron radiation techniques in order to study the phenomena.

The synchrotron based microfocused beam X-ray diffraction (µXRD) is another powerful tool for measuring local orientations and strain distribution inside individual grains of polycrystalline materials. In comparison to traditional XRD, µXRD has a high penetration depth thus can very well simulate bulk materials in their true serving condition and with the addition of very high spatial resolution. µXRD could therefore be used to study micromechanical behavior and reveal martensitic phase transformation mechanisms in various alloy systems[13]. Insight in the dynamics of phase transformation was also demonstrated by Hulme-Smith *et al.*[30] in their attempt to increase the thermal stability of a nanocrystalline bainite alloy. They showed how the thermal stability of the nanostructured bainite could be enhanced to above 500°C leading the way to new possibilities for high temperature applications.

5. Increasing the industrial usage

Since synchrotron radiation experiments can be carried out in different ways: either to be carried out at the synchrotron radiation facility, sending samples and buying the service or by sending sample and remotely control the experiments, the industrial usage of synchrotron sources could be increased with more automated pipelines (thus lower cost-per-sample analyzed), remotely accessible systems and offered data analyzing for fee. Both European Synchrotron Radiation Facility and American National Synchrotron Light Source have tried to optimize their offered services in numerous ways. NSLS introduced for instance a special industrial proposal review panel, an industrial program support and an industrial research tracking program[5]. The ESRF however, tried to optimize their services by using automated systems and developing standard methods to sustain the high-throughput demands[4]. In collaboration between academic and industrial researchers a reliable robotic sample changer, an automated data processing pipeline, a remote access system and a laboratory information management system has been developed at ESRF[4,31–34].

6. Upcoming radiation sources

Even more intense light is under construction (ready in 2016) in form of the European Free Electron Laser (European XFEL) which will generate ultra-short X-ray flashes with a brilliance that is a billion times higher than the top of the line conventional X-ray source. Just as the synchrotrons, XFEL will accelerate electrons using a linear accelerator (linac) that will wiggle the electrons inside large magnets (undulator) and coax them into emitting X-rays. This next generation source will be able to generate electron energies up to 17.5 GeV and 27 000 light flashes per second, which should be compared to todays 14.3 GeV and 120 flashes per second present at the Linac Coherent Light Source, USA or 6-8 GeV and 60 flashes per second at Spring-8 Angstrom Compact Free electron Laser, Japan. With the X-ray flashes from XFEL it will be possible to go further down in scale, to an atomic detail, and to take three-dimensional images of ultrafast processes such as the formation of molecules. However, the number of planned XFEL sources is low and according to some will most likely not be as common as the synchrotron sources of today[35].

7. Conclusions

This review of synchrotron radiation for industrial use has shown that the technique, in particular high energy X-ray spectroscopy, could be of great value to understand the process, structure and properties of steel products in a wide range of manufacturing industries. Thanks to the large investment in new and upgraded sources and industrial enhancement programs, more and more time slots will be available thus lower prices per analyzed sample which will therefor benefit the industrial usage - and ultimately result in higher quality materials.

8. References

1. Martins, M. N. & Silva, T. F. Electron accelerators: History, applications, and perspectives. *Radiat. Phys. Chem.* **95,** 78–85 (2014).

2. Buffiere, J.-Y., Maire, E., Adrien, J., Masse, J.-P. & Boller, E. In Situ Experiments with X ray Tomography: an Attractive Tool for Experimental Mechanics. *Exp. Mech.* **50,** 289–305 (2010).

3. Buffiere, J. Y., Ferrie, E., Proudhon, H. & Ludwig, W. Three-dimensional visualisation of fatigue cracks in metals using high resolution synchrotron X-ray micro-tomography. *Mater. Sci. Technol.* **22,** 1019–1024 (2006).

4. Malbet-Monaco, S., Leonard, G. a, Mitchell, E. P. & Gordon, E. J. How the ESRF helps industry and how they help the ESRF. *Acta Crystallogr. D. Biol. Crystallogr.* **69,** 1289–96 (2013).

5. Wang, J., Nasta, K. & Kao, C.-C. Industrial research enhancement program at the National Synchrotron Light Source. *Nucl. Instruments Methods Phys. Res. Sect. A Accel. Spectrometers, Detect. Assoc. Equip.* **649,** 19–21 (2011).

6. Wheeler, R. M., Chaturvedi, R. P., Duggan, J. L., Marble, D. K. & Braswell, D. Trace element analysis of steel samples with synchrotron radiation. *Nucl. Instruments Methods Phys. Res. Sect. B Beam Interact. with Mater. Atoms* **79,** 545–548 (1993).

7. Koepke, J. & Behrens, H. Trace element diffusion in andesitic melts: an application of synchrotron X-ray fluorescence analysis. *Geochim. Cosmochim. Acta* **65,** 1481–1498 (2001).

8. Hayakawa, S., Jia, X.-P., Wakatsuki, M., Gohshi, Y. & Hirokawa, T. Analysis of trace Co in synthetic diamonds using synchrotron radiation excited X-ray fluorescence analysis. *J. Cryst. Growth* **210,** 388–394 (2000).

9. Dyl, K. A. *et al.* Quantified, whole section trace element mapping of carbonaceous chondrites by Synchrotron X-ray Fluorescence Microscopy: 1. CV meteorites. *Geochim. Cosmochim. Acta* **134,** 100–119 (2014).

10. Elmer, J. W., Palmer, T. A. & Specht, E. D. In situ observations of sigma phase dissolution in 2205 duplex stainless steel using synchrotron X-ray diffraction. *Mater. Sci. Eng. A* **459,** 151–155 (2007).

11. Brauser, S., Kromm, A., Kannengiesser, T. & Rethmeier, M. In-situ synchrotron diffraction and digital image correlation technique for characterizations of retained austenite stability in low-alloyed transformation induced plasticity steel. *Scr. Mater.* **63,** 1149–1152 (2010).

12. Mo, K. *et al.* Synchrotron study on load partitioning between ferrite/martensite and nanoparticles of a 9Cr ODS steel. *J. Nucl. Mater.* **455,** 376–381 (2014).

13. Li, N. *et al.* In situ X-ray microdiffraction study of deformation-induced phase transformation in 304 austenitic stainless steel. *Acta Mater.* **64,** 12–23 (2014).

14. Martinez-Perez, M. L. *et al.* Residual stress profiling in the ferrite and cementite phases of cold-drawn steel rods by synchrotron X-ray and neutron diffraction. *Acta Mater.* **52,** 5303–5313 (2004).

15. Bellot, C., Lamesle, P. & Delagnes, D. XRD synchrotron study of carbide precipitation in martensitic steels during tempering. *Acta Metall. Sin. (English Lett.* **26,** 553–557 (2013).

16. Jimenez-Melero, E. *et al.* Characterization of individual retained austenite grains and their stability in low-alloyed TRIP steels. *Acta Mater.* **55,** 6713–6723 (2007).

17. Jimenez-Melero, E. *et al.* The effect of aluminium and phosphorus on the stability of individual austenite grains in TRIP steels. *Acta Mater.* **57,** 533–543 (2009).

18. Jimenez-Melero, E. *et al.* In situ synchrotron study on the interplay between martensite formation, texture evolution and load partitioning in low-alloyed TRIP steels. *Mater. Sci. Eng. A* **528,** 6407–6416 (2011).

19. Olson, G. B. & Cohen, M. A mechanism for the strain-induced nucleation of martensitic transformations. *J. Less Common Met.* **28,** 107–118 (1972).

20. Maxwell, P. C., Goldberg, A. & Shyne, J. C. Stress-Assisted and strain-induced martensites in FE-NI-C alloys. *Metall. Trans.* **5,** 1305–1318 (1974).

21. Eghbali, B. & Abdollah-zadeh, A. Strain-induced transformation in a low carbon microalloyed steel during hot compression testing. *Scr. Mater.* **54,** 1205–1209 (2006).

22. Blondé, R. *et al.* High-resolution X-ray diffraction investigation on the evolution of the substructure of individual austenite grains in TRIP steels during tensile deformation. *J. Appl. Crystallogr.* **47,** 965–973 (2014).

23. Zhu, L. K., Yan, Y., Li, J. X., Qiao, L. J. & Volinsky, A. A. Stress corrosion cracking under low stress: Continuous or discontinuous cracks? *Corros. Sci.* **80,** 350–358 (2014).

24. Shiozawa, D., Makino, T., Neishi, Y. & Nakai, Y. Observation of Rolling Contact Fatigue Cracks by Laminography Using Ultra-bright Synchrotron Radiation. *Procedia Mater. Sci.* **3,** 159–164 (2014).

25. Kaye, M. *et al.* A new parameter for modelling three-dimensional damage evolution validated by synchrotron tomography. *Acta Mater.* **61,** 7616–7623 (2013).

26. Hruby, P. *et al.* Fatigue crack growth in SiC particle reinforced Al alloy matrix composites at high and low R-ratios by in situ X-ray synchrotron tomography. *Int. J. Fatigue* **68,** 136–143 (2014).

27. Makino, T. *et al.* Evaluation of rolling contact fatigue crack path in high strength steel with artificial defects. *Int. J. Fatigue* **68,** 168–177 (2014).

28. Yamashita, M., Konishi, H., Kozakura, T., Mizuki, J. & Uchida, H. In situ observation of initial rust formation process on carbon steel under Na2SO4 and NaCl solution

films with wet/dry cycles using synchrotron radiation X-rays. *Corros. Sci.* **47,** 2492–2498 (2005).

29. Lepková, K., van Bronswijk, W., Pandarinathan, V. & Gubner, R. Synchrotron far-infrared spectroscopy of corroded steel surfaces using a variable angle of incidence. *J. Synchrotron Radiat.* **21,** 580–5 (2014).

30. Hulme-Smith, C. N., Lonardelli, I., Peet, M. J., Dippel, A. C. & Bhadeshia, H. K. D. H. Enhanced thermal stability in nanostructured bainitic steel. *Scr. Mater.* **69,** 191–194 (2013).

31. Arzt, S. *et al.* Automation of macromolecular crystallography beamlines. *Prog. Biophys. Mol. Biol.* **89,** 124–52 (2005).

32. Cipriani, F. *et al.* Automation of sample mounting for macromolecular crystallography. *ACTA Crystallogr. Sect. D-BIOLOGICAL Crystallogr.* **62,** 1251–1259 (2006).

33. Gabadinho, J. *et al.* MxCuBE: a synchrotron beamline control environment customized for rnacromolecular crystallography experiments. *J. Synchrotron Radiat.* **17,** 700–707 (2010).

34. Beteva, A. *et al.* High-throughput sample handling and data collection at synchrotrons: embedding the ESRF into the high-throughput gene-to-structure pipeline. *ACTA Crystallogr. Sect. D-BIOLOGICAL Crystallogr.* **62,** 1162–1169 (2006).

35. Werin, S., Thorin, S., Eriksson, M. & Larsson, J. Short pulse facility for MAX-lab. *Nucl. Instruments Methods Phys. Res. Sect. A Accel. Spectrometers, Detect. Assoc. Equip.* **601,** 98–107 (2009).