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AUTOMATED IN SITU EBSD EXPERIMENTS: CAPABILITIES AND CONSIDERATIONS

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1. ABSTRACT

Electron backscatter diffraction (EBSD) is an established technique to characterise microstructures of crystallographic materials that gives valuable information regarding grain structure, phase distribution, texture, and deformation mechanisms. There is a growing demand to make these observations under applied conditions (be it load or temperature) *in situ* within the scanning electron microscope SEM. Such experiments are not trivial, and hardware is only part of the complications. It is noted that there is a steep learning curve for any lab hoping to begin such testing without prior experience, with little consolidated advice on running such experiments. Original equipment manufacturers of both EBSD detectors and testing stages know their respective fields very well, but not necessarily how best to run experiments that combine them both. This paper discusses various tips, tricks, and things to bear in mind when planning, executing, and reporting on *in situ* experiments.

2. INTRODUCTION

The term '*in situ* testing' when related to SEM can cover a variety of different experiments carried out within the microscope from indentation and cantilever bending, to melting and solidification experiments. For the purposes of this paper, we will be focussing on the area most keenly worked on at our institute, and that which populates many of the recent publications – mechanical testing in the 1 - 10 kN range [1-3]. Electron microscopes excel at observing phenomena at the sub-micrometre scale so it may seem odd that we target such a high load, after all, even in the stiffest steel one would only require ~ 20 N to deform a sample with a 100 × 100 µm cross-section. However, as material scientists, we are often concerned with how materials behave in bulk, and a few grains across the deformation cross-section will behave very differently to a cross-section of many thousands of grains. Therefore, with this testing we use the resolving power of the electron microscope to observe the microstructural evolution at a sub-micrometre scale but on a sample showing close to bulk mechanical response. In addition to applied strain, we will also be discussing the considerations for testing at temperature within the SEM, both statically and under load.

Additional considerations for EBSD combined with in situ testing will be made throughout. Of the analytical techniques available within the SEM, EBSD gives some of the most complimentary information when it comes to thermo-mechanical testing. As an established technique for identifying the crystallographic orientation at both a high spatial and angular resolution, EBSD is particularly apt for measuring the grain shape changes and lattice rotations caused by external deformation. In addition, as the diffraction data can often be used to discriminate phases, it is a powerful tool to observe and measure phase transformations occurring due to temperature or load.

2.1. Why test in situ?

Testing *in situ* comes with its own set of challenges, but the benefits of a well-executed experiment justify the extra outlay. The main advantage of testing is-situ is that you get to observe the sample in the state it is being tested in. In many cases one could collect many snapshots in a traditional experiment by interrupting a test, transferring the sample to the microscope, carrying out the required analysis, returning the sample back to the external test apparatus to continue the test, and repeating this for the duration of the experiment. However, much is lost when removing the sample from its tested state. Removing load causes the elastic deformation to be lost and can cause cracks formed to close to the point that they can no longer be resolved. Returning the sample to room temperature may in many cases fix the microstructure in place, but in many others cause a transformation that means the observation no longer corresponds to the sample in its high temperature state.

A secondary benefit of testing in situ is that it often means that many more experimental steps can be carried out in a set time frame. Going between an external piece of testing equipment and the microscope can be time consuming and logistically challenging. Whereas having the entire experimental set up within one piece of equipment eliminates sample transportation time and that associated with pumping/venting the microscope, and analytical setup. A further benefit is that with less sample handling, there are fewer opportunities to damage the sample. EBSD is notoriously surface sensitive and repeatedly mounting and dismounting for SEM and (for example) a microtester risks damaging the sample at every step.

A final benefit, and one particularly relevant for EBSD, is that that when testing *in situ* the sample orientation relative to that of the detector and microscope is fixed (discounting movements caused by either applied deformation or thermal expansion). With EBSD we are capable of measuring orientation to a high degree of accuracy (often under 0.1°). It is unlikely that the sample will be correctly re-orientated to within this tolerance when mounting and remounting the sample between every step. The ability to keep the sample orientation the same throughout in situ testing means that orientations can be more confidently compared between steps.

2.2. Why automate these tests?

If an advantage of *in situ* testing is allowing one to collect more experimental steps than could be done with interrupted testing, then automation of the testing gives an even greater advantage. *In situ* testing has traditionally been a very manual undertaking. Operator input is needed at every step of an experiment; to setup the SEM beam conditions, trigger analytics, change the state of the sample (apply temperature or load), relocate the region of interest (which has likely moved due to loading or thermal expansion), refocus the electron beam, retrigger the analytics, and then repeat these steps for the duration of the experiment. Therefore, experiments are limited to the amount of time that the operator can attend the experiment and usually this will be working hours. By automating all the aforementioned steps, the operator is required only at the initial

stages of the experiment and then the system can run autonomously over evenings and weekends, acquiring data throughout. This additional data acquisition can be used to carry out more experimental steps (for example, collecting an EBSD map every 0.1 % strain rather than every 0.3 %) and improve the experimental resolution, or by using this additional time to collect larger datasets at each step for greater statistics.

A further benefit of automation is that with less operator input, there is less chance of operator error. Humans are prone to making unintentional mistakes, especially where repetitive tasks are concerned. With the increasing speed of analytics, it may be found that the same set of operations (find region, correct focus, set magnification, trigger EBSD map, change load and/or temperature) must be repeated every 5 - 10 minutes. It is easy in such a situation to miss any one of these steps or accidently repeat one (two maps at the same load/temperature for example).

A final benefit to automating these tests is data correlation. With a well implemented integrated system, there is no reliance on the operator to take detailed notes. Experimental logs are recorded throughout, and data collected on different systems can be stamped with the experimental conditions at the time of acquisition. For example, the metadata of the EBSD map including not only details relevant to map itself, but also the temperature/load of the sample at the time of acquisition.

3. WHAT IS REQUIRED?

3.1. Automation software

To the authors' knowledge, there are few only a couple of systems on the market that are capable of the level of automation already discussed. Building a system oneself is not beyond the realms of possibility (with enough scripting proficiency), however, this would require access from the original equipment manufacturers (OEMs) to the application programming interfaces (APIs) of the SEM, the microtester, and the EBSD system.

3.2. Hardware

3.2.1. Microscope. Although likely the largest outlay in terms of cost, the quality of the electron column is not the driving factor in the setting up a system for in situ EBSD experiments. A stable beam, that can be well focussed at reasonable kVs and high probe currents are what is needed, and this can be found with most modern field emission gun (FEG) SEMs. The often-quoted performance at 1 kV and under a mm working distance is largely irrelevant. What is a consideration, is the quality of the functions that can be called by an external software (auto-focus, auto stig, etc.) and how repeatable they are. Also of importance are physical parameters of the microscope. Microtesters tend not to be small so a large chamber is preferable, they also tend not to be light - so a capable microscope *xyx*/tilt/rotation stage with a high load tolerance is essential. Finally, multiple ports for feedthroughs are always beneficial.

3.2.2. Microtester. There are two main approaches when it comes to combining microtesting with EBSD. One is to have the sample pre-tilted within the microtester, the other is to tilt the microtester itself to an angle suitable for EBSD. Both have their advantages. Pre-tilting in many ways can be easier from a microtester design point of view. One of the biggest constraints when designing a micro-tester for within an SEM is the load limits, especially when at tilt. Microscope OEMs will often quote different load limits for their stages in the flat and tilted positions due to the concerns about how much harder the stage stepper motors have to work (particularly in the *y* axis) and concerns about stage accuracy and backlash. Pre-tilting the sample within the microtester means that the microtester can be a greater weight, allowing more design freedom and allowing construction from denser and stiffer materials. Stiffness is generally key when it comes to designing tensile rigs. As Newton's 3rd law insists on an equal and opposite reaction, while we deform our sample by pulling it apart, the sample resists by pulling back and deforming the microtester in turn. A large difference in stiffness between the sample and the tester means that the tester is only deformed minutely in comparison, and (hopefully!) only elastically. This elasticity from the tester affects the accuracy of the displacement measurements, and, therefore, the sample strain values predicted. It can be accounted for by applying a 'compliance correction', taking account of the elasticity of the tester, however the greater the difference in stiffness between the sample the less this is required, and the more accurate applied displacements will be.

A lighter microtester will inherently be more compliant, but the advantage of being able to tilt the stage is a compromise worth considering when it comes to EBSD. Yes, the displacement control will be inherently less accurate, but perhaps that is not so important if we are calculating the strain by another method (strain can be calculated directly from the EBSD maps with digital image correlation if required). We do gain flexibility – we are still able to rotate the stage. By pre-tilting the stage, you lose a degree of freedom, and you are no longer able to correct for small deviations in alignment. In EBSD we are always referencing our orientations to an external referencing system, and in an in situ test, we have our loading axis and we can ensure with stage rotation that our mapping is well aligned with it. A further advantage is experimental flexibility. We can go from the top-down imaging position to the EBSD position without remounting the sample and potentially having to reconfigure the microtester. This can be useful when, for example, finding a region of interest with BSE imaging, or printing a fiducial for region tracking (discussed later).

3.2.3. EBSD system. A fast, accurate, EBSD system is essential for in situ studies. This is not only as we are trying to maximise the amount of data we collect, but it is with the understanding that in many of the experiments the sample will not be in a static condition. Even if holding at a constant temperature or load, things may still be dynamically changing (such as recrystallisation or creep) and therefore time is a factor. EBSD maps need to be collected as quickly as possible to make them as time independent as possible.

It should be noted that EBSD detectors are sensitive to infrared (IR) radiation. If planning on experiments above ~ 500 °C, then an IR protection will be required. This takes the form of a thin alumina coating on the detector. Depending on the thickness of the coating and the material, then this may affect low kV sensitivity.

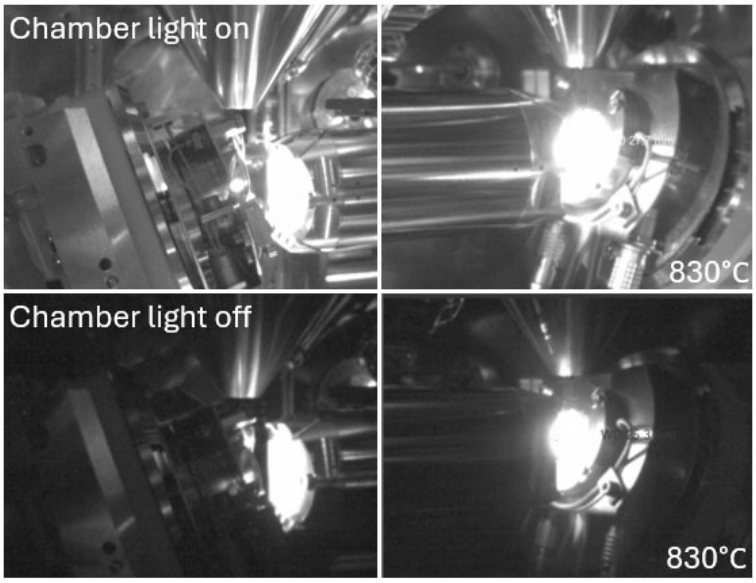

Figure 1. Chamber camera images of a sample at 830 °C. In the lower images the sample and heater are the only light sources in the chamber.

3.2.4. Additional recommendations. In addition to the above it is recommended that an in-chamber plasma cleaner be fitted. In the majority of in situ experiments one is likely to be analysing the same region of the sample repeatedly leading to the build-up of carbon on the surface due the beam interacting with residual hydrocarbons within the chamber. A plasma cleaner helps maintain cleanliness of the sample and chamber to reduce this, as do other endeavours to improve vacuum such as a cold trap.

A further recommendation is the ability to print a fiducial on the surface. Region tracking is described in more detail in the following section, but it is found that the addition of a gas injection system to allow beam assisted deposition of a heavy metal on the surface (as typically found on a FIB instrument) in a specific location can be useful in a range of experimental workflows.

4. REGION TRACKING

4.1. When region tracking is required

With almost all *in situ* experiments it can be expected that the region being investigated will shift during the testing. The most common microtester design is with a single screw, i.e., the test specimen is fixed at one end, and pulled from the other. Therefore, the region of interest can be expected to shift in the loading direction as the sample is stretched. In almost all situations this will be in 'x' as the long axis of the sample is aligned perpendicular to the EBSD detector. However, it is not just lateral movement that has to be contended with. The vast majority of materials obey Poisson's relationship, as they are extended in one direction, they contract in the opposing ones. In a practical sense this results in the sample thinning as pulled and, therefore, an increase in required working distance. A change in working distance will of course require a refocusing (autofocus!) but as we are at tilt for EBSD any change in z will have a component in in y and so will need to be adjusted for.

It is the only the change in working distance (which will have a component in y) that needs to be adjusted for in a stand-alone heating experiment. There can be little lateral movement expected without load applied, but thermal expansion and volume change due to phase transformation can both lead to a change in sample height.

4.2. Fiducials

Region tracking done automatically is carried out much as one would do manually. Find a recognisable feature on the surface, track its movement during a loading/temperature step, reposition the stage relative to the feature so it returns to its original position in the scanned frame. The feature used is known as a 'fiducial' and these can take several forms. It is possible to use an already present feature on the surface, however the chosen fiducial needs to fulfil three criteria:

1) Discernible. A fiducial should be clearly visible. Sharp contrast with the surroundings helps locate the feature. This can be from edges of a topographic structure if using SE, or atomic contrast with BSE.
2) Distinct. A fiducial should be unique. A small pore would make an excellent fiducial in many cases, but not if there are several similar pores in proximity that it could be confused for.
3) Durable. A fiducial should last the duration of the experiment. There is no use in using beam sensitive contamination that could break down with repeated scanning, or a feature that could decompose with temperature.

Often (especially on a polished metallic surface for EBSD) there may be no features that satisfy the above conditions. This is even more likely to be the case if the region is highly specific (investigating a particular grain boundary type for example) in which case it is necessary to create a fiducial marker.

There are many ways to make a fiducial with high spatial precision. The simplest is by placing an indent close to the region of interest. Many indenters are coupled with decent optical microscopes and x-y stages, however it may be tricky to identify regions of interest that require the SEM. The biggest downside to indents is that one is actively changing the material being observed. The indent deforms the material around it, locally work-hardening it before a tensile test, and potentially influencing recrystallisation behaviour during a heating experiment. A more convenient way (admittedly with a higher consumable cost) is fitting a gas injection system to the SEM to deposit a fiducial marker at the region of interest with e-beam patterning. One can rapidly move from the region identified by EBSD/EDS/BSE to the deposition position to print a fiducial on the region of interest. Caution needs to be made when considering temperature and the affinity of the deposited metal (commonly platinum) to diffuse into the sample. If chemical diffusion is a concern, then a marker can be directly milled into the material with a FIB. All the advantages of region finding of the SEM + Pt-Dep, but with the disadvantage of having to use a separate system. One should still be cautious of potential damage from the FIB beam such as implantation and amorphisation.

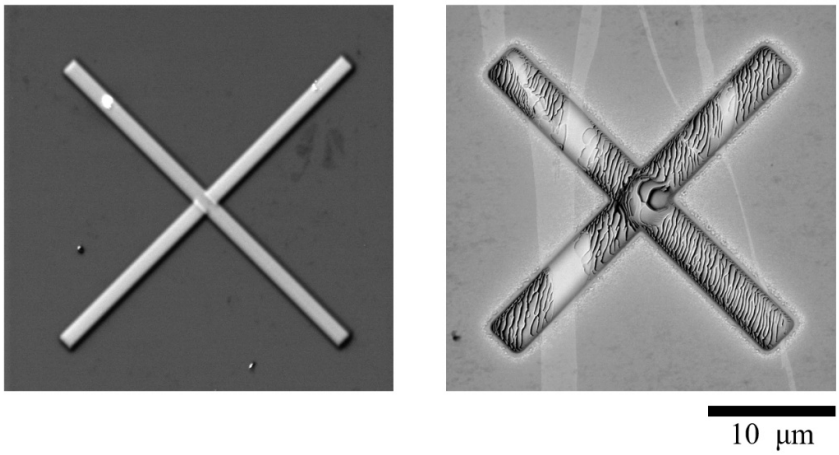

Figure 2. Fiducials made by e-beam Pt deposition (left) and FIB milling (right).

4.3. Methods of region tracking

As for the tracking itself there are numerous approaches that can be taken. Several image recognition techniques exist for recognising a feature in an image such as a fiducial. A number of these involve transforming the image into the frequency domain through a Fourier transform and locating the region through phase correlation or similar. These techniques can recognise the feature even if it has rotated, and more advanced algorithms can even identify the feature if it has changed size of has undergone an affine transformation [4]. As rotations are likely to be minimal, and changes in scale unlikely, we have found the simpler approach of template

matching to be more robust and sufficient. Changes to the fiducial during the test (due to either heating or strain) are best accounted for by always referring to the most recent image of the fiducial rather than the initial, as any changes are likely to be gradual.

Once the fiducial has been recognised, there are two approaches for relocating the imaging region back to the region of interest; through stage movements or scan shifts. Scan shifts are generally have a limited range, and we, therefore, often opt for a hybrid approach of a stage shift for coarse movements, followed by beam shifts for fine movements.

The biggest consideration when setting up the tracking is the window that should be searched to look for the fiducial. The window should be large enough to account for any shift during the testing step, but fine enough to include enough pixels in the fiducial that it can be reliably identified and the stage to be accurately repositioned. A large, high pixel count image could be taken for this at each step, but this will add significant time to the run. We have found that a 1024 × 1024 image with the fiducial taking up ~ 20 - 30 % of the image is sufficient in most cases. The tracking should be carried out before acquisitions, but additional tracking steps can be added if the fiducial can be expected to move out of the window (for large strain steps or temperature changes for example).

5. MECHANICAL TESTING CONSIDERATIONS

5.1. Sample preparation

As highlighted in previous EMAS contributions, surface preparation for EBSD is key [5]. This is particularly difficult for tensile specimens which are typically many cm's in length. Large samples by their nature are trickier to polish (especially hard materials) due to the greater amount of material being removed at each step, and it being difficult to maintain even pressure over the entirety of the sample. Sample handling is not helped by being unable to mount these large samples in resin. The lack of resin also increases the risk of rounding the edges of the sample. To minimise this, diamond polishing steps should be minimised as much as possible preferably going from a very fine grinding paper (#4000 grit) straight to a final polishing step of colloidal silica. For final polishing step (and diamond steps if needed) mats with a short/zero nap to minimise rounding.

5.2. Sample relaxation

With loading experiments there is a choice to be made concerning what to do with the sample while analysis is taking place. In a conventional tensile test elongation generally occurs at a constant loading rate or crosshead speed. This cannot generally occur with time consuming analysis steps such as EBSD as the state of the sample will have changed between the beginning and end of the acquisition.

In situ tests therefore tend to take place in a 'quasi-static' regime with the sample being held while the analysis takes place. There are two options here, each with their benefits and drawbacks. One can either hold by fixing the crosshead (displacement control) so no further straining can take place, or one can put the system under load control by allowing the crosshead to move to maintain a fixed load on the sample.

Fixing the cross head so that the sample is completely static may seem like the obvious choice, but this will be followed with a load drop measured on the sample. Despite deformation being paused, sufficient internal stresses to drive dislocation movement still exist, leading to annihilation or to arrange into cells, which may form new sub-boundaries and other stable structures. With the dislocation density decreasing (and/or forming stable cells) the internal back stresses are reduced and the macroscopic load drops [6]. Restarting the deformation will require an initial re-yielding while the internal stresses are re-established, and for hardening to continue at the rate before the pause.

The alternative is to hold the load. If the system is operated under load control then displacement will be applied to maintain the internal back stresses and prevent relaxation. Although this could give a 'truer' analogy to a conventional tensile test as it gives less opportunity for relaxation, the moving crosshead brings in further difficulties. With the crosshead moving then the region of interest will also shift, leading to potential difficulties with both region tracking and shear in the data acquisition. This can be accounted for by introducing a wait time for the load/displacement to reach a steady state, but it should be noted that for many creep prone materials and those above yield, no steady state will ever be reached as the material continues to deform under load.

Therefore, despite the material relaxing during analysis, the majority of tests are carried out under displacement control rather than load control.

5.3. Actual strain rate

With a microtester strain rates are generally lower than what can be achieved with benchtop systems, but many are capable of applying load by moving the crosshead at speeds up to ~ 10 μm/s (with the strain rate dependent on the gauge length of the sample). However, with these interrupted tests the actual strain rate is much lower than this. One has to factor in the amount of time held for region tracking and analysis where generally the crosshead is kept static. This is particularly true with EBSD, where depending on the quality of map required, the mapping may take in excess of an hour.

This difference between the instantaneous strain rate while loading and the effective experimental strain rate is also equally applicable to temperature, with the actual heating/cooling rate of the experiment having to consider the time spent on tracking/acquisition.

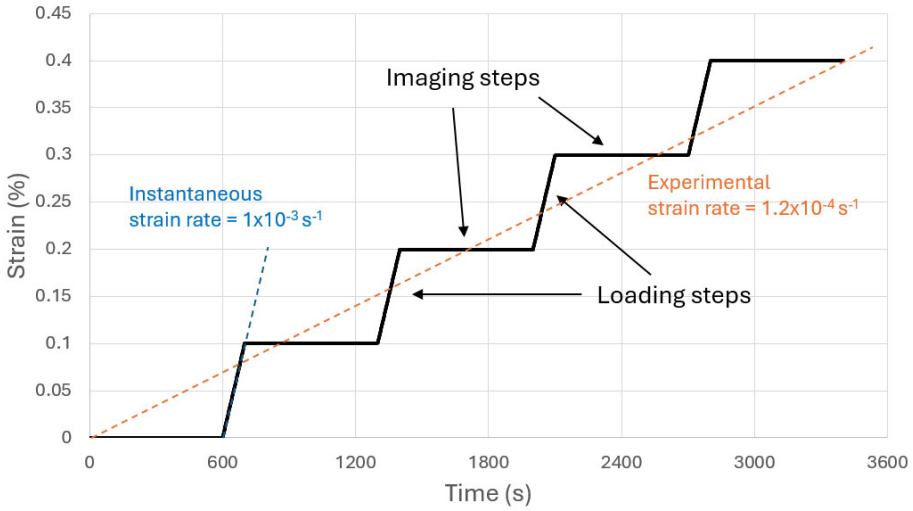

Figure 3. Schematic showing the difference in applied strain rate and effective strain rate accounting for analysis steps.

6. *TEMPERATURE CONSIDERATIONS*

6.1. *Temperature measurement and validation*

The type of microtesters discussed here are excellent for taking measurements at temperature, but they are not optimal for measuring the temperature at which things happen. With this type of testing it is notoriously difficult to measure the temperature of the surface being analysed. Typically, a thermocouple for regulating the power to the heater will be placed on the heater itself, however, the sample (especially the analysed surface) will likely be a lower temperature due to radiative and conductive losses. A thermocouple can be attached to the sample surface, but the accuracy of this is still debateable. On the scale of the samples being tested, the thermocouple itself can act as a significant heatsink conducting heat away from the sample, and under vacuum (where the majority of the temperature is being lost through radiation) the presence of the thermocouple alters the nature of the surface and local radiative losses. Instead, it is preferable to relate the observations made in situ to those made in a complimentary experiment. In an experiment observing a phase transformation in a material, it may be useful to relate the in situ observations to those from another method with more accurate sample temperature measurement, for example differential scanning calorimetry or dilatometry.

When reporting the data it is important not to refer to the sample temperature as this is unknown, but instead refer to the complimentary experiment if done, and/or report the temperature measured on the controlling thermocouple, describing the location of the measurement and the expected offset to the sample surface temperature.

6.2. Oxidation avoidance

Even in the vacuum of an SEM (typically in the order of ~ 10^{-6} mbar) materials liable to react at temperature can still do so with residual gasses present in the chamber. Oxygen ingress can affect the kinetics of the sample being observed – for example oxygen in solution in titanium will stabilise the alpha phase and push the beta transus temperature higher, or the presence of oxygen will lead to the creation of a thin oxide that can prevent EBSPs being diffracted from the surface.

Steps can be taken to achieve a better-quality vacuum, chief among them being chamber sample and cleanliness. Oils and greases will degas overtime and could react with the sample when at temperature. Good microscope etiquette of glove wearing whenever handling anything that goes within the chamber helps, as does suitable storage of the microtesters when not in use. Regular running of an on-chamber plasma cleaner will help maintain a clean chamber. Installation of a Peltier or liquid nitrogen cold finger can help improve the vacuum by trapping any residual gases onto its surface.

Additional pumps and better-quality seals can be used to achieve an even better ultimate vacuum, however this is likely impractical with a general-purpose SEM. However, a rudimentary getter can be made by placing something with a higher affinity for oxygen on the heater alongside the sample. Zirconium works particularly well for this purpose and is more efficient with a greater surface area such as roughened surface or a partially sintered powder.

6.3. Sublimation and eutectic points

Despite a high vacuum being desirable when it comes to minimising interactions with the sample, one has to be conscious that the sublimation point of elements drops with decreasing pressure as shown in Fig. 4. While elements strongly bonded (ionically/covalently) in ceramics and precipitates can be expected to hold to a higher temperature, those in solution in the material desorb from the surface. Depending on the diffusion rate at the test temperature, this can lead to significant depletion in the sample of the element(s) in question. This is not only a concern for the sample as one is fundamentally changing its composition, but is a risk for the microscope. Any evaporated metals will condense on the first surface they land upon. This deposition can reduce efficiency of detectors and risks damaging the electron column if it passes through the pole-piece.

Melting points of elements are not as affected by pressure as sublimation points [6], however, just being confident of the melting points of individual elements is not sufficient. Alloys will generally have a lower melting point that their constituent elements and therefore phase diagrams should be considered. Eutectic points are of particular concern, and a number of eutectic compositions and their temperature have been marked on Fig. 4.

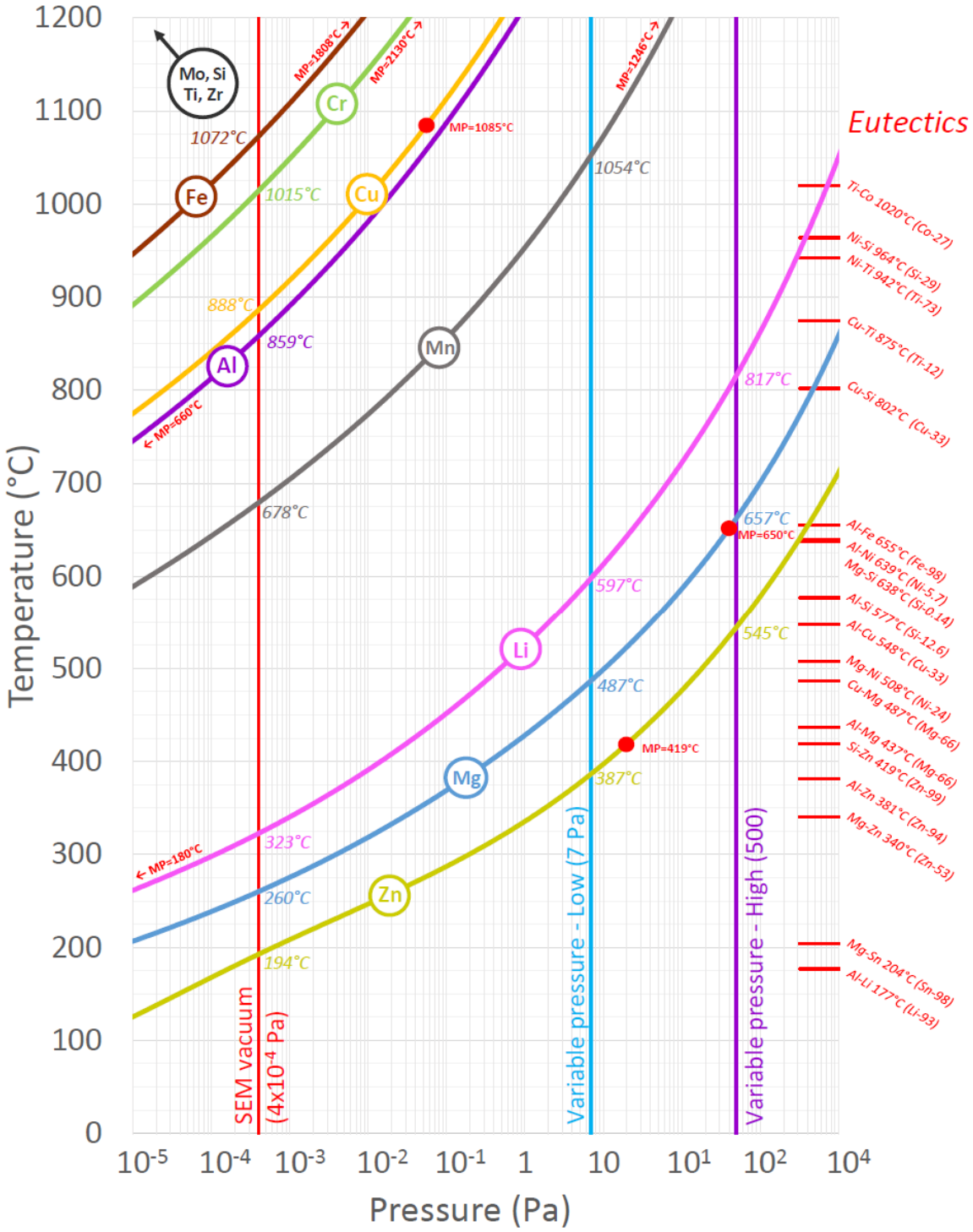

Figure 4. The vaporisation points of the elements under the pressures achievable in the SEM. Several eutectics of note are also indicated. Values plotted taken from the 'Vapour Pressure Calculator' by Michael Schmid of the IAP/TU Wien Surface Physics Group [7]. Due to lack of experimental data, and the estimations used, errors can be as great as ± 15 °C of stated temperature.

6.4. Dynamic experiments

As with loading experiments, temperature studies are not static, and in many circumstances one can expect more evolution of a microstructure at a held temperature than at a held load. This is not necessarily a negative, but one must be conscious of it when presenting results. Data can not be presented as a snapshot of what is occurring at a set temperature, but rather highlight that the changes are happening as the acquisition takes place. For example, in Fig. 5 below where the time of acquisition is indicated alongside the mapping.

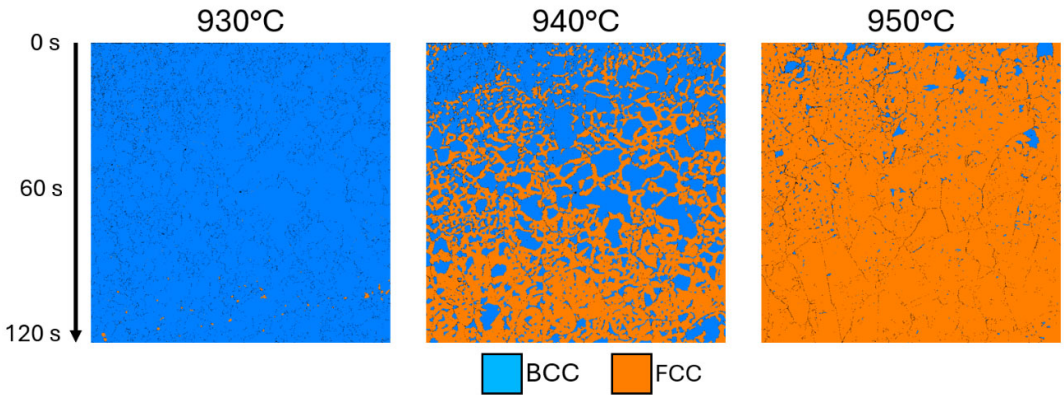

Figure 5. Ferrite to austenite transformation in a steel above 900 °C. The microstructure dynamically evolving within the map is highlighted by the time of acquisition indicated on the lefthand side.

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