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Recent Metallurgical Applications of Electron Diffraction Techniques in TEM

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Abstract: A summary of the current efforts in the field of transmission electron microscopy (TEM) for the high spatial resolution characterisation needs of some of the metallurgical applications using electron diffraction techniques is presented. The aim is to highlight the improvement and the introduction of different methods in the TEM that made it possible to gain new insights in structural analyses of metals and alloys. Techniques like precession electron diffraction, electron diffraction tomography, and four-dimensional scanning transmission electron microscopy are shown to be capable of allowing an unprecedented amount of information to be studied about these materials with the help of improved detector technology. This review will showcase the benefits and some of the examples of the most recent electron diffraction applications for metals and alloys.

Keywords: Transmission electron microscopy, metals, alloys, imaging, diffraction, tomography.

Metalurji Alanında Kullanılan Güncel Geçirimli Elektron Kırınımı Teknikleri

Özet: Metalurjik uygulamaların yüksek çözünürlüklü karakterizasyon ihtiyaçları için geçirimli elektron mikroskobunda (TEM) elektron kırınımı teknikleri kullanılarak gerçekleştirilen güncel çabaların bir özeti sunulmaktadır. TEM kullanılarak metalik malzemelerin ve alaşımların yapısal karakterizasyonlarında yeni iç görüler kazandırmak için geliştirilen farklı yöntemlerin vurgulanması amaçlanmıştır. Presesyon elektron kırınımı, elektron kırınım tomografisi ve dört boyutlu taramalı geçirimli elektron mikroskopisi gibi tekniklerin yeni dedektör teknolojilerinin yardımıyla bu malzemeler hakkında benzeri görülmemiş miktarda bilginin incelenmesine olanak sağladığı gösterilmiştir. Bu derlemede, metalik malzemeler ve alaşımlar için güncel elektron kırınımı uygulamaların avantajları ve bazı çalışma örnekleri sergilenecektir.

Anahtar Kelimeler: Geçirimli elektron mikroskopisi, metaller, alaşımlar, görüntüleme, kırınım, tomografi.

Review

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1.Introduction

The field of metallurgy relies heavily on structural characterisation, both at the bulk and micro-scales to understand the property-structure relationships. Since the development of first commercial TEM, metallurgists used these microscopes to understand the defects and the overall nature of metals and alloys that they have been working on. The most popular and straightforward application of electron diffraction in TEM is collecting selected area electron diffraction (SAED) patterns. In this approach the selected area aperture is placed after the sample plane to select an area and only allow the beams from that area to produce the diffraction pattern. Since parallel beam illumination is needed to produce spot patterns, often times large areas are illuminated when collecting a diffraction pattern, which could create complex diffraction patterns depending on the number of crystals in the field of view. SAED approach mitigates this problem efficiently. It should be noted that the size of the parallel beam can be made smaller by utilising a smaller C2 aperture, though SAED offers a more practical solution (Williams & Carter, 2009).

Over the last decade there have been tremendous advancements in detector technology which allowed new and improved techniques to be developed which increased the level of insight one can gain from using such systems. Electron diffraction techniques in particular have benefited from the development of direct electron detectors (MacLaren et al., 2020). These highly sensitive detectors allow researchers to shorten the acquisition times which helps with beam damage and the overall experiment time. Due to high dynamic range and low noise of these detectors the collected data is of very high quality, which opens new pathways to be explored.

Precession electron diffraction was introduced as a new technique by Vincent and Midgley in 1994 that allows to collect kinematical-like intensities (Vincent & Midgley, 1994). Conventional electron diffraction experiments, especially zone-axis patterns suffer greatly from dynamical diffraction. Dynamical diffraction can be explained as the re-diffraction of a Bragg diffracted electron beam. Furthermore, because of this phenomenon there is a transfer of intensity between reflections. Since the zone axis patterns have many reflections simultaneously, there are many dynamical pathways which will result in transfer of intensity. The kinematical approximation of diffraction intensity ($I \propto |F|^2$) allows researchers to use the structure factor to identify the locations of the atoms in the unit cell and solve the crystal structure entirely. However dynamical intensities mean that the kinematical approximation cannot be used to accurately identify locations of the atoms. Precession electron diffraction involves rocking and de-rocking of the electron beam before and after the sample (Figure 1). Through this process multiple diffraction patterns are integrated and the resulting reflections have less dynamical effects (Midgley & Eggeman, 2015).

Electron diffraction tomography (Kolb et al., 2019) is a rapidly growing approach to identify crystal structures where a high spatial resolution is required. The basic idea is to use the TEM as a diffractometer and collect diffraction patterns over a range of sample tilts. This can be achieved through a couple of different ways. First method to collect tomographical electron diffraction data is simply tilting the sample by an increment over a range and collecting still diffraction patterns at each step. An appropriate sample holder needs to be used while conducting these experiments since the standard TEM holders do not offer large tilt angles. After collecting step-wise diffraction patterns the reciprocal lattice of the sample is reconstructed and unit cell parameters are determined.

However, this method will sample only a part of the reciprocal lattice of the sample and the intensities of the collected reflections will suffer from dynamical effects. Therefore, assisting this step-wise tomography approach with precession massively helps with structure solution. The precession allows to integrate an additional part of the reciprocal lattice so the intensities are more kinematical which aids in structure solution. Another tomography method is continuously tilting the sample over a range and collecting diffraction patterns as the sample is mechanically tilting (Gemmi et al., 2019). This allows some integration of the intensities similar to the precession assisted tomography and the data collection times can be quite fast, depending on the detector. However, the mechanical stability of the goniometer is the limiting factor for continuous rotation, the illuminated crystal can easily move outside of the beam if the goniometer is not very stable while tilting. All of the tomography methods have a missing wedge of the reciprocal lattice which comes from the mechanical tilting limit of the sample holder and the column geometry.

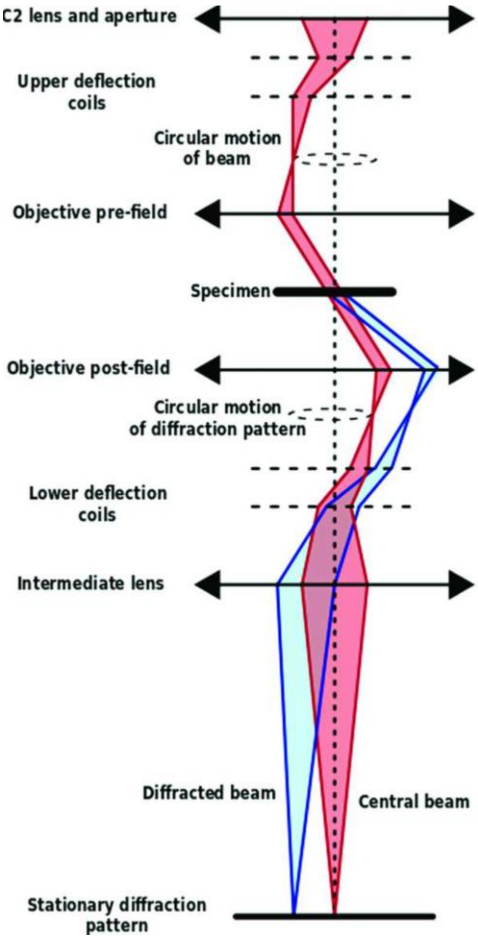

Figure 1. Schematic representation of how precession electron diffraction works. Deflection coils rock and de-rock the electron beam before and after the sample which creates a hollow cone of precession. Adapted with permission from Midgley & Eggeman (2015) (Midgley & Eggeman, 2015).

Şekil 1. Presesyon elektron kırınımı tekniğinin şematik gösterimi. Numunenin üstündeki ve altındaki elektron demeti saptırma bobinleri elektron demetini belirli bir açıyla eğerek içi boş bir presesyon konisi oluşturur. Midgley ve Eggeman'dan (2015) izin alınarak uyarlanmıştır (Midgley & Eggeman, 2015).

Both precession electron diffraction and electron diffraction tomography have been around for some time, though their application has become more popular in the last decade. There is another method that has been developed very recently, which is called four-dimensional scanning transmission electron microscopy (4DSTEM) (Ophus, 2019). This technique collects 2D electron diffraction patterns in STEM mode across a 2D sample area, hence the name 4DSTEM. It allows to collect large datasets that can be analysed offline.

This technique requires fast readout speeds from the detector, therefore only with the advances in detector technology (e.g. direct electron detection (MacLaren et al., 2020)) this method was made available. The amount of information that can be analysed in a 4DSTEM dataset is tremendous, methods such as virtual dark field (MacLaren, Fraser, et al., 2024), virtual bright field (Shao et al., 2019), orientation (Jeong et al., 2021) and strain analyses (Mahr et al., 2021) and ptychography (Li et al., 2022) can be performed on these datasets.

In this review, some of the recent applications of precession electron diffraction, electron diffraction tomography and 4DSTEM techniques on metals and alloys will be explored. Advanced electron diffraction techniques like these are becoming readily available due to widespread adoption of direct electron detectors and it is believed that the field of metallurgy will surely benefit from the implementation of these techniques to further our understanding of metallic materials.

2. Precession electron diffraction

As introduced previously, precessing the electron beam before and after the sample reduces dynamical effects, which makes the intensities of the reflections more kinematical and usable for crystallographic analysis. This method is also capable of dealing with coherent nano-sized phases within a matrix. Kverneland et. al. used precession electron diffraction to analyse the crystal structures of nano-participates found in an Al-Zn-Mg alloy (Kverneland et al., 2006). Even though they did not use a high dynamic range, low background detector, they were still capable of analysing the structure of the embedded nanoprecipitates. With the developments in direct electron detection, the precession electron diffraction technique becomes very powerful for analysing nano-features found in alloys.

Precession electron diffraction patterns can be collected using the parallel beam illumination condition from a region of interest on the sample. This would result in a single spot pattern with fewer dynamical pathways which can be used for identification of crystallographic information about the material. However, precession electron diffraction can also be done while a convergent electron beam is being scanned across the sample. That experiment would create a dataset with thousands of convergent beam electron diffraction (CBED) patterns. This method is called scanning precession electron diffraction (SPED) (Eggeman, 2019) and it is similar in principle to the 4DSTEM technique. SPED analysis can be very useful for determining the precipitation characteristics in alloys. For instance, the aging mechanism of 7050 Al alloys have been characterised using SPED, which provided phase maps and orientation analysis of the sample (Xiao et al., 2022). Analyses like these can be routinely performed when using the SPED technique.

Due to the high spatial resolution of TEM, SPED analyses can be performed with nanometre resolution. Furthermore, highly sensitive direct electron detectors increase the effectiveness of this technique, for instance Maclaren recently reported on crystallographic analyses of alpha-beta titanium alloy (MacLaren, Frutos-Myro, et al., 2024). Figure 2 demonstrates the capability of producing virtual ADF imaging as well as orientation matching of the selected points in the scan. Analyses like these are becoming routine with SPED experiments. It is clear that the quality of data is crucial for any meaningful analysis with this technique, here (Figure 2) the direct electron detector allowed for the collection of high dynamic range, low-noise diffraction patterns, which made it possible to pattern match with high correlation index for the orientation analysis.

3. Electron Diffraction Tomography

Electron diffraction tomography involves collecting a series of electron diffraction patterns while tilting the sample, which are then used to reconstruct the reciprocal lattice of the crystal (Gemmi et al., 2019). Since the rotation axis is known the collected diffraction patterns can be analysed using a dedicated software (e.g., PETS2 (Palatinus, Retrieved December 8, 2024)) and a 3D reciprocal lattice can be reconstructed. Tilt steps are generally around 0.5-2° and the tilt range is the maximum that the geometry of the goniometer can allow. Figure 3 represents a step-wise electron diffraction tomography data collection where the blue mesh is the reciprocal lattice of the illuminated crystal, red dots are the reciprocal lattice points that the Ewald sphere intersects (i.e., where the Bragg's law is satisfied) and the green lines are the tangents of the Ewald sphere. Even with a tomography holder there will be a missing wedge, but the remaining parts of the reconstructed reciprocal lattice can be generated using the symmetry of the crystal. However, the lattice parameters can be accurately identified even for a low symmetry crystal using this method.

Step-wise electron diffraction tomography is collecting still diffraction patterns at every tit step, but the goniometer can be continuously tilted while collecting diffraction patterns as well. This continuous rotation method will integrate a part of the reciprocal lattice rods, depending on the frame time of the patterns and the goniometer speed.

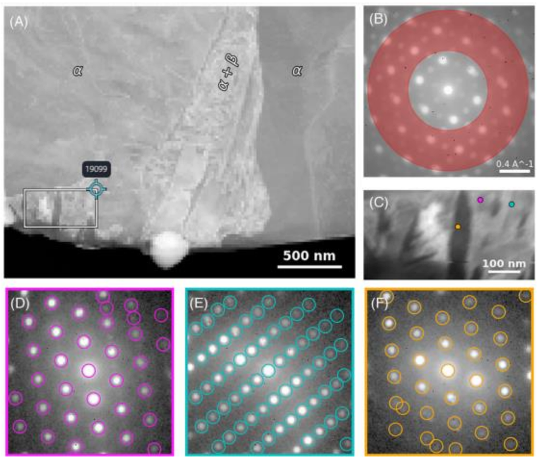

Figure 2. Data collection area for the SPED analysis and the virtual imaging and orientation analysis capabilities. (A) shows the larger field of view area of the sample, (B) shows the average diffraction pattern from the entirety of the scan and the virtual ADF detector in red, (C) is the resulting virtual ADF image, and (D), (E), and (F) shows the diffraction pattern from specific points in the scan with orientation pattern matching overlaid. Reproduced from Maclaren et al. (2024) (MacLaren, Frutos-Myro, et al., 2024).

Şekil 2. SPED analizi için veri toplama bölgesi, sanal görüntüleme ve oryantasyon analizi kabiliyetlerinin gösterimi. (A) numunedeki veri toplanan bölgenin etrafını, (B) bütün verinin ortalama kırınım desenini ve sanal ADF dedektörünü, (C) oluşturulan sanal ADF görüntüsünü ve (D), (E), ve (F) oryantasyon analizinde kullanılan verideki spesifik noktalara ait kırınım desenini göstermektedir. Maclaren et al.'dan (2024) alınmıştır (MacLaren, Frutos-Myro, et al., 2024).

Reconstructed reciprocal lattice allows to identify the lattice parameters and the symmetry elements of the structure, but in order to fully solve the crystal structure the intensities of the reflections must be used. However, the intensities of the reflections would be dynamical without using preventative measures such as precession. Precession cannot be applied to a continuous rotation electron diffraction tomography but it is possible (and very beneficial) to apply it to a step-wise tomography approach (Duran & Eggeman, 2021). That way the intensities can be used according to the kinematical approximation and the structure can be solved using the phase retrieval algorithms such as direct methods (Burla et al., 2015) or charge-flipping (Palatinus & Chapuis, 2007).

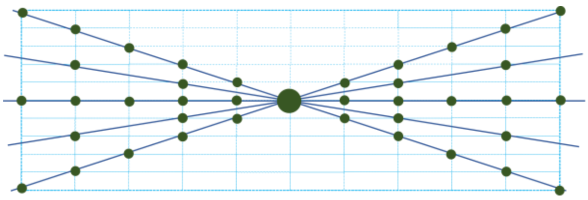

Figure 3. Schematic representation of step-wise electron diffraction tomography. The blue mesh represents the reciprocal lattice, the green dots represent the reciprocal lattice points that satisfy the Bragg's law, and the dark blue lines represent the tangents of Ewald Sphere.

Şekil 3. Sabit adımlı elektron kırınımı tomografisinin şematik gösterimi. Mavi ağ karşıt latisi, yeşil noktalar Bragg kanununu sağlayan karşıt latis noktalarını ve koyu mavi çizgiler de Ewald küresinin tanjantlarını temsil etmektedir.

Recently, Klein et. al. reported on the crystal structures of sub-micron precipitates of complex aluminides, such as θ'-Al_2Cu and $Al_{4.5}FeSi$, using a precession assisted step-wise electron diffraction tomography (Klein et al., 2022). The determination of crystal structure is extremely important for alloys since they can directly affect the mechanical properties of the material. Furthermore, the high spatial resolution of the TEMs becomes a necessity when dealing with sub-micron or nano-sized crystals. If the precipitate is embedded in a coherent matrix, then the problem becomes more complex, however it is still possible to utilise electron diffraction tomography techniques to solve crystal structures of embedded precipitates.

4. Four-dimensional Scanning Transmission Electron Microscopy

With the advancements in detector technology, another powerful electron diffraction technique was developed in recent years in the form of 4DSTEM. In this method, a STEM probe is rastered along a region of interest on the sample and a diffraction pattern collected for every probe position. Figure 4 shows a representation of collecting diffraction patterns for each probe position during 4DSTEM (Ophus, 2019). Depending on the step size and the area, very large datasets (>20 GB) can be collected relatively quickly. With a 4DSTEM dataset virtual imaging can be performed without a physical access to the microscope. Depending on the camera length used while collecting the dataset, an ADF image from the Bragg reflections or even a HAADF image from high-angle scattered electrons can be produced virtually. This approach has parallels to the SPED technique with the difference being that the microscope is in STEM mode for 4DSTEM, and it is in TEM mode for SPED, in other words the nanoprobe mode is used for 4DSTEM and the microprobe mode is used for SPED. In theory it is also possible to do SPED with a parallel beam but for 4DSTEM the probe must be fully converged.

The choice of C2 aperture is extremely important for 4DSTEM experiments, since it affects the convergence angle and thus the size of the diffraction disks. Overlapping disks are essential for ptychographical reconstruction, however isolated disks are usually needed for structural analysis such as for producing phase maps, orientation maps, or strain field maps. With this amount of data there are many analyses that can be done on metals and alloys.

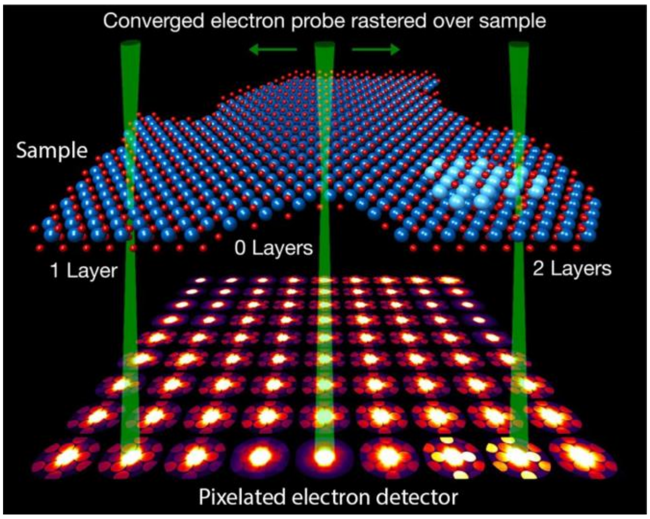

Figure 4. Data collection procedure for 4DSTEM. At every probe position an electron diffraction pattern is collected. Reproduced from Ophus (2019) (Ophus, 2019).

Şekil 4. 4DSTEM için veri toplama prosedürü. Her elektron prob pozisyonunda bir elektron kırınım deseni toplanmaktadır. Ophus'dan (2019) alınmıştır (Ophus, 2019).

One of the most widely performed analysis using 4DSTEM data is to measure the strain fields around a nanoparticle. In order to perform a strain analysis, a reference pattern is selected from an unstrained area of the scan and the pattern from the region of interest is analysed against it. Since the strain will change the lattice parameter and hence the (d*) of the relevant (hkl) reflection, it can be measured in the form of the difference between the unstrained position of the reflection and the strained position.

Large datasets of the 4DSTEM and SPED techniques require improved data processing to do accurate and fast analyses. There are software solutions such as Topspin (NanoMEGAS, Retrieved 14 Jan 2025) data analysis program from NanoMEGAS that provide some of the necessary processing for these datasets. For instance, Rottmann & Hemker utilised Topspin software to measure the strain around dislocations in 1% strained polycrystalline Mg and Figure 5 demonstrates the strain map, BF image and the strain profiles from that study (Rottmann & Hemker, 2018).

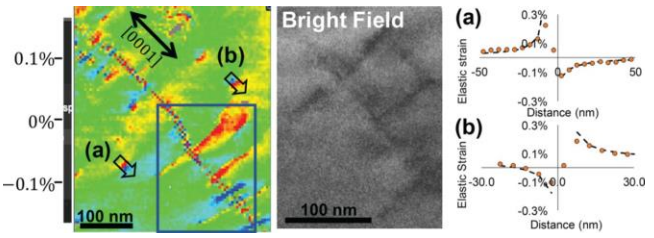

Figure 5. Strain map of strained Mg, BF image of the area shown in the blue rectangle and strain profiles. Reproduced from Rottmann & Hemker (2018) (Rottmann & Hemker, 2018)

Şekil 5. Gerinime uğratılmış Mg'a ait gerinim haritası, mavi kare ile gösterilen bölgenin BF görüntüsü ve gerinim profilleri. Rottmann ve Hemker'den alınmıştır (2018) (Rottmann & Hemker, 2018)

The application of machine learning approaches on electron diffraction tomography, SPED, and 4DSTEM datasets are becoming routine as well, with new workflows are being reported frequently (Kalinin et al., 2022). The simultaneous acquisition of different signals (such as energy dispersive X-ray spectroscopy (EDS) and 4DSTEM) also opens new areas to be explored in terms of materials characterisation (Duran et al., 2023) .

5. Conclusions

The field of metallurgy at the fundamental level, requires the study of the structure to improve the understanding of the material and to optimise it further. Unique material challenges such as embedded coherent phases in alloys, can be overcome with the current state of the art. These techniques can be extremely useful for identifying coherent phases, measuring strain fields with high spatial resolution, and for full structural solution and refinement of unknown structures. Thanks to the improvement and the development of new electron diffraction techniques, researchers have been able to open up new insights into the characterisation of materials in recent years. With faster and more sensitive detectors and precise control of the microscope through dedicated softwares, structural characterisation at high spatial resolution became almost routine for metals and alloys. On that note, it would be appropriate to mention that most of these techniques require advanced and often expensive instruments as well as specific expertise in implementation. However, there are exceptional efforts in the community to automate some of these techniques and thus the expertise in the data collection/ analysis may not be a crucial requirement in the future.
Performing structural solutions of coherent phases, orientation analyses at nanometre resolution, strain analysis, and virtual imaging are now possible thanks to the precession electron diffraction, SPED, electron diffraction tomography, and 4DSTEM. The speed of improvement with these methods is definitely exciting and it will be very intriguing to see the what more can be done in terms of materials characterisation in the near future.

6. Conflicts of Interest

The author declares no conflict of interest.

7. References

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