When used with a Scanning Electron Microscope (SEM), Energy Dispersive Spectroscopy (EDS) offers the analyst many interesting results. For example, the operator gets images colored according to elements or phases which give additional information about the composition of the material which was visible in the SEM images. If more analytical details are needed, it is necessary to analyze the original X-ray spectra which were acquired by EDS detector. These are the base for the element distribution images. This process is called ‘spectra evaluation’. Sophisticated physical models, algorithms, and computer programs are needed to do this. There are always two questions; the first is which elements are in the sample, and the second is how much of each is present (the concentrations).

Element identification is the first step towards getting a proper view of the sample composition. No Quant model would be able to calculate the element concentrations properly if the assumptions about the elements in a given sample were incorrect or wrongly evaluated. This is why EXpertID [1] is used with all EDAX spectra evaluation for the automatic identification of elements.

There are two different base Quant models, based on evaluation of the measured net-counts of element peaks (‘yellow’) or based on Peak-to-Background (P/B) ratios (‘yellow’ divided by ‘purple’), shown in Figure 1.

![Figure 1. Two different base Quant models, based on evaluation of the measured net-counts of element peaks (yellow) or based on P/B ratios (yellow divided by purple).](image-url)
In both cases, one can say that the measured values are higher with higher element concentrations in the sample. But there is usually no linear relationship between element concentration and measured signal, and also other elements can cause huge inter-element effects. However, the measured input values for the quantification calculation (net intensities or P/B) are extracted from a measured spectrum. For these reasons a background-spectrum approach is needed to subtract the continuous X-ray radiation (Bremsstrahlung) and to determine the background measured signal for the P/B-ratio. If element lines overlap, a deconvolution of the peaks is required. EDAX uses a full physical algorithm-based Bremsstrahlung calculation [2,3] or a pure mathematical background approximation method (SNIP). The peak-deconvolution is always performed with a probability-theory based and very robust Bayesian algorithm [4,5]. Both are also core components of the EXpertID qualitative analysis of measured spectra. Additional corrections are required, for example pile-up correction, which is a special effect of detectors and pulse processing electronics.

Following the two base types for quantification inputs, two models are available in EDAX software for SEM applications with measurement of bulk samples, the net-counts evaluation eZAF and the P/B based PeBaZAF. ‘ZAF’ usually stands for Atomic number correction (Z), Absorption correction (A) and Fluorescence correction (F). The classical (very simplified) formula where $N_i$ are the measured net-counts and $c_i$ are the unknown concentrations, is:

$$N_i^{obs} = \frac{\Delta \Omega_{i,t} \cdot e_i}{4 \pi} \cdot c_i \cdot [ZAF_i]^{gb}$$

$e_i$ is the detector efficiency. The index $i$ is for all elements in sample (used line).

$\frac{\Delta \Omega_{i,t}}{4 \pi}$ is the product of solid angle, the Electron Microscope beam-current and the measurement time.

The measurement time is known, but it is not usually possible to determine the beam-current and solid angle exactly. There is one unknown parameter more than available equations, therefore it is required to use the additional constraint of the sum for all concentrations must equal 100%, to solve the equation system.

eZAF is using the Z correction which follows the fundamental work by Love/Scott [6]. The used absorption correction is based on the Sewell/Love/Scott ‘Quadrilateral’ model (Figure 2) [7] with additional special modifications with using proprietary measurements at very high tilt angles.

Additionally, a more recent database by Tim Elam et al. [8] is used, which contains additional correction, especially with mass absorption coefficients, which dominate the results of absorption calculation.

PeBaZAF is a P/B-method following the ZAF approach with an additional term for Bremsstrahlung. The basic relation is given by dividing both equations, where (P/B)$_i$ are the number of net counts divided by measured Bremsstrahlung of the same energy:

$$\frac{(P/B)_i}{N_i^{obs}} = \frac{e_i \cdot c_i \cdot [ZAF_i]^{gb}}{N_i}$$

Compared to the eZAF formula, one can see some parameters have been removed so that the number of unknown variables is equal to the number of equations, and the solution does not require the composition normalization equation. This model has been developed and improved from the 1980s until today [9-11].

Both approaches have advantages and disadvantages. The original design goal with the introduction of the first model was that the P/B is always the method of choice if the sample surface is not flat (particles, rough surfaces). The challenge with PeBaZAF is to extract the proper P/B values from a measured spectrum. An accurate background determination is required to interpolate the values at the peak energies, and the EDAX software provides this. Because the measured Bremsstrahlung statistics influence the P/B values much more than net-counts, the PeBaZAF comes with worse base precision compared to eZAF. On the other hand, the P/B-model based model-corrections are low compared to eZAF. Higher corrections also mean higher model uncertainties and therefore the PeBaZAF has advantages in mean accuracy.

PeBaZAF

<table>
<thead>
<tr>
<th>Symb. Line</th>
<th>P/B</th>
<th>Cts</th>
<th>Atom</th>
<th>Error</th>
<th>R</th>
<th>A</th>
<th>F</th>
</tr>
</thead>
<tbody>
<tr>
<td>18 P Kα</td>
<td>586.9</td>
<td>82.14</td>
<td>61.83</td>
<td>7.44</td>
<td>0.0050</td>
<td>0.0477</td>
<td>0.0000</td>
</tr>
<tr>
<td>31 Ga Kα</td>
<td>238.1</td>
<td>67.84</td>
<td>81.97</td>
<td>5.97</td>
<td>0.0035</td>
<td>0.0033</td>
<td>0.0092</td>
</tr>
</tbody>
</table>

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Basically, both models have similar uncertainties. eZAF has more systematic errors but good precision (repeatability of measurements). → PeBaZAF has fewer systematic errors and so has good mean accuracy but it has worse precision due to higher influence of counting statistics (which is bad for comparison tasks).

The eZAF error is not reduced much with more and more counts, because the model errors dominate. But the positive influence on PeBaZAF error can be huge when more counts are available. These are the results with same sample but 100 times longer acquisition time or 100 times higher count rate (e.g 200 kcps SDD vs 2 kcps with old Si(Li) count rate):

PeBaZAF

<table>
<thead>
<tr>
<th>Atomic Number</th>
<th>Deviation</th>
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<tbody>
<tr>
<td>15</td>
<td>2.4</td>
</tr>
<tr>
<td>31</td>
<td>33.9</td>
</tr>
<tr>
<td>55</td>
<td>55.4</td>
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eZAF

<table>
<thead>
<tr>
<th>Atomic Number</th>
<th>Deviation</th>
</tr>
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<tbody>
<tr>
<td>15</td>
<td>2.4</td>
</tr>
<tr>
<td>31</td>
<td>33.9</td>
</tr>
<tr>
<td>55</td>
<td>55.4</td>
</tr>
</tbody>
</table>


It is possible to improve the error factor for the PeBaZAF model with more counts in spectrum. The use of SDDs has supported this.

It is known for net-count based standardless models like eZAF that a measured database is required to get the best accuracy.

The PeBaZAF disadvantage that it required higher than usual acquisition times was reduced with the introduction of SDD detectors, which offer more than 2 orders of magnitude higher count rates compared to old Si(Li) detectors in analytical mode. This ability to offer more counts in the same collection time was a benefit to the PeBaZAF method.

The major uncertainties with net-count based approaches are systematic errors due to the correction model and due to the parameters used (e.g. MACs). The example has shown about one order of magnitude higher corrections are applied with the eZAF model compared to PeBaZAF. But it is possible to improve eZAF by including a good measured database (“remote standards” standardless [12]), if it is not possible to perform real standards comparison measurements (because the standards are not available or because of timing issues).

Finally, it is now possible to achieve standardless EDS quantification within + 10% relative deviation for 95% of all results (standard deviation about 5%).

For PeBaZAF a reasonable number of counts are required in spectrum; a modern SDD detector provides the user with high count-rate use, while eZAF will get the accuracy by support of a measured database.

References

High-Speed EBSD Mapping with the Velocity™ EBSD Camera Series

In the 25 years that Electron Backscatter Diffraction (EBSD) mapping has been commercially available, the adoption of new technology has continually enabled faster acquisition rates for more efficient data collection. Initial analog video cameras were limited to operating rates of 30 frames per second and were often frame averaged to collect at speeds of one pattern per second. The first digital CCD-based cameras were introduced at about 40 patterns per second. The first high-speed CCD-based cameras were introduced at 200 patterns per second and were continually improved to reach speeds of 1,500 patterns per second. The recent introduction of the Velocity™ Plus EBSD Detector, utilizing a high-sensitivity and low-noise CMOS imaging sensor, again provided a significant improvement of collection speeds to 3,000 indexed patterns per second. Now, with the latest Velocity™ Super EBSD Detector, data collection speeds up to 4,500 indexed points per second are available.

To make these fast acquisition speeds practical and usable, the entire Velocity™ EBSD detector and system has been optimized for performance. This includes a customized for EBSD CMOS-based camera, a custom-designed lens for highest sensitivity, and optimized software for indexing speed and performance. The results of this design are shown in Figure 1. This image shows an EBSD Image Quality (IQ) greyscale map combined with a colored Inverse Pole Figure (IPF) map (this map type will be subsequently referred to as an IQ + IPF map), where the colors correspond to the crystallographic orientation aligned to the sample surface normal direction, collected from an Inconel 600 superalloy at 3,000 indexed points per second. The beam current used for this acquisition was 11 nA, which demonstrates the high-sensitivity of the Velocity™ system, which combined with the 99.6% indexing success rate, also shows that the system is fast and accurate.

The Velocity™ EBSD System is available in two configurations: the Velocity™ Plus, with a collection speed up to 3,000 indexed points per second, and the Velocity™ Super, with a collection speed up to 4,500 indexed points per second. To enable this collection speed, the Velocity™ Super has a dedicated high-speed mode designed for the fastest collection speeds. Beam currents of 25 nA or higher are necessary to achieve these collection speeds with 99% indexing success on standard samples, although lower currents can be used if this indexing success is not required. Figure 2 shows an IQ + IPF map from an additively manufactured Inconel 718 alloy collected at 4,500 indexed points per second with a 98.2% indexing success rate using a beam current of ≈30 nA. This orientation information helps users to understand the solidification rates and mechanisms during the additive manufacturing process. These microstructures can contain information both over a large area and with fine detail. The high-speed collection capability of the Velocity™ Super is ideal for characterizing these 3D printed structures.

One advantage of the Velocity™ CMOS-based EBSD detectors is that the EBSD pattern resolution used for high-speed collection is 120 x 120 pixels. In comparison, the Hikari CCD-based detector uses a 30 x 30 pixel pattern for 1,500 points per second acquisition and has an acquisition speed of around 500 patterns per second at a comparable 120 x 120 pixel image resolution. Because of this, the Velocity™ detector can be used on a range of materials (both in terms of material state and crystal structure) without having to optimize either the indexing or band detection settings. In the following examples, the Velocity™ was set up to acquire patterns at 3,000 points per second and was indexing around 2,500 points per second using the default Hough parameters with the EDAX TEAM™ software and a beam current of ≈30 nA. The ability to achieve these high collection speeds without specific optimization, which requires a higher level of operator knowledge, allows users to get more real performance on their instruments with a shorter learning curve compared to traditional detectors.
Figure 3 shows an IQ + IPF map from a deformed ferritic steel sample. The deformation is visualized through the subtle changes in color within the individual grains. The orientations within a grain change as much as 30°, but the precision of the measurements at these conditions allows detection of the small rotations within the microstructure. There are more defects within the crystal lattice in a deformed material, which results in EBSD patterns that are less sharp. This can reduce band detection efficiency and indexing performance. However, with this example, an indexing rate of 98.3% was achieved. This shows that deformed materials can be measured at high speeds with the Velocity™.

All these examples have been single phase materials. When analyzing multiple phases, the system needs to determine both the correct phase for a given EBSD pattern and the correct orientation. This increases the computational requirements for the data collection process. Figure 4a shows an IQ + IPF map from a dual-phase (BCC Ferrite and FCC Austenite) steel sample and Figure 4b shows an IQ + Phase map, with an indexing success rate of 97.3%. These phases are clearly resolved, and the orientation is correctly determined. These types of steels are used in severe environments to resist corrosion and understanding the phase distribution helps to optimize performance.

These examples have all had cubic crystal structures, which have a high degree of symmetry. This increased symmetry reduces the scope of the orientation determination process. Figure 5 shows results from an additive manufactured titanium medical implant. Figure 5a shows the IQ + IPF map, and 5b shows the IQ + Phase map, with an indexing success rate of 94.3%. This sample is primarily alpha titanium, which has a hexagonal crystal structure. There are also small pockets of beta titanium, which has a BCC crystal structure. This beta phase is what is retained as the titanium cools during the additive manufacturing process. Specific orientation relationships measured here confirm the phase transformation mechanism. Measuring the size and fraction of the beta phase can give insight into the cooling rates within the material.

These examples show that the Velocity™ cameras can provide high-quality data collected at high-speeds with reasonable beam currents without the need for expert level software optimization. This makes the Velocity™ practical for traditional EBSD scans, but also ideal for 3D serial sectioning and in-situ experiments, where minimizing acquisition time is very important.
For those who attended Dr. Stuart Wright’s recent webinar ‘Texture Analysis via EBSD’, he emphasized the need for sampling enough orientations for statistical reliability in characterizing a texture. Many forming processes are symmetric in nature and produce a texture that shows symmetry beyond crystallographic symmetry. In crystallographic symmetry, after a symmetry operation is applied, the crystal is left in an orientation indistinguishable from the original. For example, consider a crystal with cubic symmetry. A 90°, 180° or 270° rotation about any of the face normals of a cube would leave the cube in an orientation that is indistinguishable from the original. However, sample symmetry is different. The classic example is rolled sheet, where there are mirror planes in the vertical and horizontal directions. In the bulk of the material (where surface effects due to friction between the material and the rolls are negligible) we expect the set of lattice orientations shown in Figure 1 to be present in the material with the same frequency. This sample (or statistical) symmetry thus differs from crystal symmetry as the orientations are distinguishable from each other. To reach “statistical” symmetry in EBSD measurements, many grain orientations must be sampled.

There are several tools in OIM Analysis™ to explore sample symmetry. Figure 2 shows the workflow for calculating a texture in the standard way with triclinic symmetry. The same steps should be repeated to calculate a texture with sample symmetry with the exception of changing the sample symmetry to orthotropic where highlighted. An example (111) pole figure for rolled copper shows the effect of applying orthotropic symmetry (top) compared to a pole figure calculated without enforcing sample symmetry (below).

There is a way in OIM Analysis™ to quantitatively compare how similar these two textures are. This is done using a Texture Difference Index (TDI) Chart as shown in the following sequence (Figure 3). Pressing the “Edit >>” button allows you to choose the two textures you want to compare. The resulting plot shows the TDI as a function of the rank of the series expansion used to calculate the textures. As the rank increases the resulting texture captures finer details and increases the TDI. For more details on the TDI, read Dr. Wright’s EDAX Blog post ‘Old Eyes?’.

Figure 1. Four orientations of a cube related through vertical and horizontal mirror planes.

Figure 2. Screen shots from OIM Analysis™ demonstrating how to calculate a texture with Triclinic (None) or orthotropic symmetry with accompanying (111) pole figures for rolled copper.

Figure 3. Screen shots from OIM Analysis™ demonstrating how to generate a texture difference index plot to compare two different textures.
### 2019 Worldwide Events

**April 1-3**  
**RMS EBSD 2019**  
London, United Kingdom  
**May 22-24**  
**Microscopical Society of Canada (MSC)**  
Vancouver, Canada  

**April 30-May 1**  
**Ceramics Expo 2019**  
Cleveland, OH  
**June 2-7**  
**Lehigh Microscopy School**  
Bethlehem, PA  

**May 6-7**  
**FIB SEM Meeting**  
Washington, DC  
**June 10-14**  
**Inter/Micro 2019**  
Chicago, IL  

**May 19-23**  
**European Microbeam Analysis Society**  
Trondheim, Norway  
**June 11-14**  
**SCANDEM 2019**  
Gothenburg, Sweden  

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### 2019 Worldwide Training

To help our present and potential customers obtain the most from their equipment and to increase their expertise in EDS microanalysis, WDS microanalysis, EBSD/OIM™, and Micro-XRF systems, we organize a number of Operator Courses at the EDAX facilities in North America, Europe, Japan, and China.

### EUROPE

| EDS Microanalysis (TEAM™ EDS) | April 8-10 | Wiesbaden*  
|--------------------------------|------------|  
|                                 | June 24-26 | Wiesbaden*  
|                                 | September 23-25 | Wiesbaden*  
|                                 | October 8-11 | Wiesbaden*  
|                                 | October 21-23 | Wiesbaden*  

### JAPAN

| APEX™ EDS | April 11 | Osaka  
|-----------|---------|  
|           | October 10 | Tokyo  
|           | November 14 | Osaka  

### TEAM™ EDS

| June 13 | Tokyo  
| July 11 | Osaka  

### OIM School

| April 23-24 (Entry) | Tokyo  
| May 27-28 (Basic) | Osaka  
| July 18-19 (Entry) | Tokyo  
| October 24-25 (Advance) | Osaka  
| November (Basic) | Tokyo  

### NORTH AMERICA

| APEX™ EDS | November 20-21 | Mahwah, NJ  
|-----------|----------------|  

### APEX™ and TEAM™ EDS

| May 14-16 | September 10-12 | Mahwah, NJ  
|-----------|-----------------|  

### TEAM™ EDS

| October 21-23 | Draper, UT  
|-------------|  

### EBSD OIM Academy

| June 18-20 | Mahwah, NJ  
| October 23-25 | Draper, UT  

### TEAM™ Pegasus (EDS & EBSD)

| October 21-25 | Draper, UT  

### XRF

| May 21-23 | Mahwah, NJ  

### CHINA

| EDS Microanalysis | June 11-13 | Shanghai (ACES)  
|------------------|------------|  
|                  | September 3-5 | Shanghai (ACES)  
|                  | December 3-5  | Shanghai (ACES)  

### TEAM™ EBSD OIM Academy

| June 18-20 | Shanghai (ACES)  
| September 10-12 | Shanghai (ACES)  
| December 10-12  | Shanghai (ACES)  

Please visit [https://www.edax.com/support/training-schools](https://www.edax.com/support/training-schools) for a complete list and additional information on our training courses.

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[http://edaxblog.com](http://edaxblog.com).  

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Matthew Mumford

Matthew joined EDAX on September 6, 1994 as a Production Planner in the Mahwah, NJ office. Today as the Master Scheduler, his many tasks include forecasting the company’s material requirements and keeping the inventory supply in balance with its demand. Matt is a past co-recipient of the company’s annual “Dr. Lux” Quality Accomplishment Award, and this year he will join the EDAX 25 Year Club!


Matthew earned his A.A.S. in Business Administration from Rockland Community College SUNY in 1983. He is a past President, Engineer, and Life Member of the New City Fire Department in Rockland County.

Matthew and his wife Julie, a high school Special Ed teacher, reside in Warwick NY with their canine “Petey”. They enjoy gardening and they each hold leadership roles within their local church. The three grown “children” (each now 30-somethings) have gifted them with two adorable granddaughters, and four handsome grandsons, with more on their way. In his spare time, Matthew enjoys watching the New York Yankees and New York Islanders, playing the drums, and following politics.

Weimin Xia

Weimin joined EDAX in February 2001. He has been an Application Specialist in the Beijing, China office since 2011. Weimin’s responsibilities include user training, application and sales support, as well as, some equipment installation and service. From 2001-11, he was an Account Manager for EDAX.

Prior to EDAX, Weimin was a Sales and Application Engineer at Everyone Enterprises Ltd from 1998-2001. He served as a Concurrent Post Teacher for a computer course at the CIBT School for Business from 1996-98. Weimin was a Consultant for Carl Zeiss Far East Co. (1994-96) and an Assistant Research Professor in the State Key Laboratory of Tribology at Tsinghua University (1987-94). From 1984-87, he worked as an Engineer at the Ministry of Energy Resource’s Electric Power Construction Research Institute in Beijing.

In 1984, Weimin earned a bachelor’s degree from Tsinghua University. He is married to his wife, Yun Song. In his spare time, Weimin enjoys music, reading, and taking trips in his car.
The Institut de Chimie Moléculaire et des Matériaux d’Orsay (ICMMO) at the Université Paris-Sud/Université Paris Saclay is one of the largest chemistry institutes in France. The ICMMO is a pioneer with regards to comparing texture analysis at the multiscale level using Electron Backscatter Diffraction (EBSD), X-ray, and neutron diffractions. At the institute, engineers, researchers, PhD students, Master students, and scientists run experiments for many other research centers and industrial companies. The material team at ICMMO specializes in materials science, optic fiber development, mechanical and magnetic properties and the link between texture and microstructure in metallic alloys, as well as monocrystal orientated grain growth.

The metallurgical team at the ICMMO was one of the first users of EBSD in France. In 1988, Dr. Richard Penelle hired Dr. Thierry Baudin from the Centre National de la Recherche Scientifique (CNRS) to use EBSD to study both deformation processes and recrystallization in metallic alloys. The first EBSD system at the ICMMO was a Dingley EBSD System (Figure 1) installed on a Camebax microprobe (CAMECA). The system was soon moved to a W-SEM (Zeiss 940) in order to get 1,000 grains with 1 orientation per grain on a Fe-3%Si alloy. The acquisition used to take about a week to complete. Eventually, the computer, software, and camera were upgraded to get 1,000 grains with approximately 5,000 orientations within 22 hours.

In order to speed things up, the institute purchased an EDAX DigiView EBSD System and added beam control to the microscope. This allowed the users to acquire the same orientation maps in 20 minutes. In 2006, the ICMMO purchased its first FEG-SEM (Zeiss Supra 55VP). Engineer Dr. François Brisset bought an EDAX Hikari EBSD System that enabled him to obtain combo scans and perform combined EDS-EBSD analysis, in addition to working in low pressure mode when non-conductive materials were studied with EBSD. A few years later, the ICMMO upgraded to a Hikari XP camera allowing users to run at speeds up to 500 frames per second. The lab is now thinking of upgrading to the EDAX Velocity™ EBSD camera with CMOS technology, which can run up to speeds of over 3,000 frames per second; it would become the ICMMO’s fifth camera.

“Fast acquisition and sensitivity are becoming required more often, either to scan at faster speeds or to use less energy or current for sensitive samples,” said Dr. Brisset. “We chose EDAX because the power of the EBSD analysis software is advanced compared to its competitors. We also like the capability of combining EBSD and EDS quant maps and the ability to extract data. Last but not least, EDAX’s support has been very good from both Europe and the United States, over the years.”

In addition to the camera updates, the ICMMO acquired a hot in-situ stage for EBSD analysis that can run at temperatures up to 700°C. This allows users to work on samples like Mg, Al, Cu, or steel or Ni alloys. For example, the researchers have been able to describe recrystallization and grain growth mechanisms caused by twinning in a copper sample. They have also identified which twins disappeared at high temperatures during grain growth after recrystallization (Figure 2).

For more information about the ICMMO, please visit: https://www.icmmo.u-psud.fr/en/.

**Figure 1.** The ICMMO’s first EBSD system in 1988.

**Figure 2.** Determination of the coherent twin boundary (a), and the incoherent twin (b) boundary. The coherent twin boundaries are not mobile for geometrical reasons but the incoherent twin boundaries can still continue to migrate as observed in Figures 2a and 2b. It is easy to notice the migration of the incoherent boundaries (plain marks b) and the stability of the coherent boundaries (dash marks a) that generate the disappearance of the twins. Here, the pole figures are given to highlight the coincidence planes.