

Precautions when texture of cold rolled hcp metals is measured by ACOM.

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Automated Crystal Orientation Measurement (ACOM) by evaluating backscatter Kikuchi patterns in the SEM has become a well-accepted technique in materials science. Other acronyms for this method are BKD and Automated EBSD. It is not only used for individual grain orientation measurement on a meso-scale, but has also some benefits over conventional x-ray pole-figure measurement when the orientation density function (ODF) has to be determined from large sample areas¹.

Three types of deformation texture are found after unidirectional cold rolling of hcp metals, depending on the c/a ratio (Figure 1): For metals with $c/a < 1.633$, such as Ti or Zr, the basal pole (or the c axes) is inclined by about 15° to 25° from the sheet normal (ND) to the transverse direction (TD). Metals with $c/a \approx 1.633$, such as Mg or Co, have their basal pole pointing in the sheet normal direction. The rolling texture has almost axial symmetry. In metals with $c/a > 1.633$, such as Zn or Cd, the basal pole is inclined by typically 15° to 25° from the sheet normal direction to the rolling direction (RD).

A Kikuchi pattern with the $[0002]$ zone axis (basal pole) in the center consists of a central "star" of six-fold symmetry that is formed by an alternation of three $\{10\bar{1}0\}$ and three $\{11\bar{2}0\}$ bands, rotated about the zone axis at 30° from each other. The ratio of the band widths is $d_{10\bar{1}0}/d_{11\bar{2}0} = 1/\sqrt{3}$. If only the angular positions of these 6 bands are considered for indexing, a 30° ambiguity about the c -axis remains for the calculated orientation. That means that one out of two measurements will likely lead to a systematically false orientation value. Therefore, some precautions have to be taken when measuring the rolling texture of hcp materials by ACOM.

The following means are recommended to remove this ambiguity in calculated orientation values: 1. Since the sample surface is inclined by about 20° from grazing beam incidence in the SEM, the sample should be placed with RD in horizontal direction for $c/a > 1.633$, but with TD in horizontal direction for $c/a < 1.633$ materials, to avoid $[0002]$ zone axes at the pattern center. 2. To enable consistent indexing, one or more additional bands from non-collinear lattice planes outside the $[0002]$ pole should be added to the set of central bands. It is advisable to use about 8 to 12 bands for indexing, if the patterns are sufficiently clear for locating so many bands (Figure 2). 3. The band positions and the band widths should be taken into account. 4. The intensities of the bands may be further used to discriminate possible orientation variants.

The system has to be well calibrated, since small deviations of the specimen-to-screen distance ("diffraction length L ") or the position of the pattern center from the correct values may lead to gross errors in pattern indexing and in the calculated grain orientations (Figure 3). Scanning at low SEM magnifications thus requires dynamic system calibration on the fly².

Erroneous indexing is hardly noticed neither in crystal orientation maps (COM), nor in the conventional representation of the rolling texture of hexagonal materials by one symmetrized basal (0002) pole figure only that has been recalculated from the ODF under the assumption of orthorhombic sample symmetry. If triclinic sample symmetry, however, is used and additional pole figures are recalculated, the differences become quite apparent. A further indication of false indexing is the inconsistency between the shape of twins in COM and their misorientations.

The following examples of indexing a backscatter Kikuchi pattern with the basal pole close to the center have been made with a cold rolled titanium sheet sample at 20 kV accelerating voltage on the ORKID BKD system.

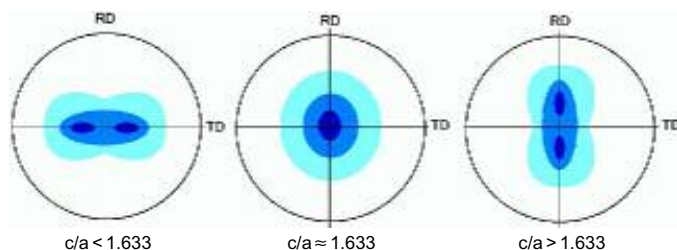


Figure 1. Schematic representation of (0002) pole figures of cold rolled hcp metals for the three classes of c/a ratio.

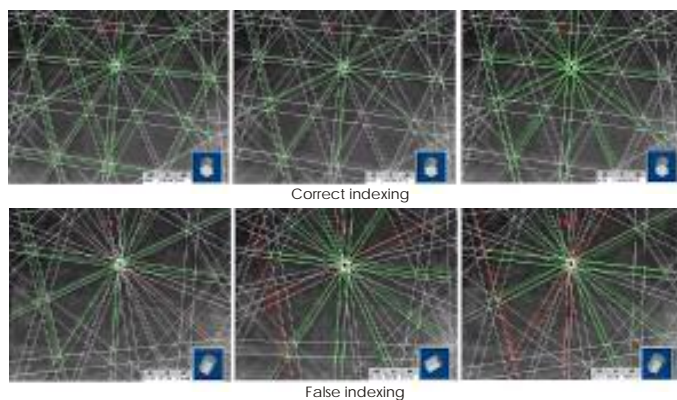


Figure 2: Well calibrated system. For correct indexing, the considered bands should not all belong to the same pole. Reliability depends on the number and combination of bands.

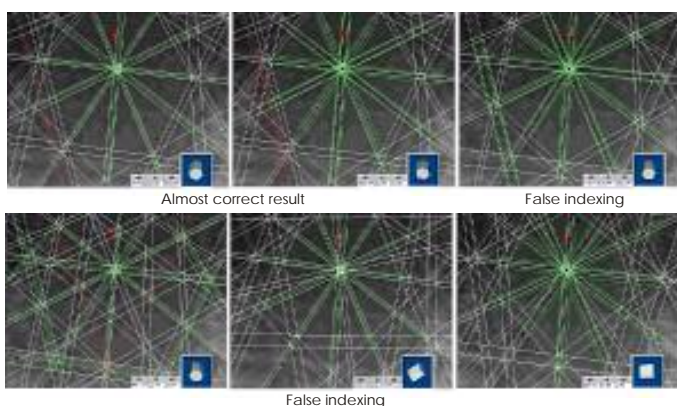


Figure 3: A slightly mis-calibrated system may lead to false indexing. The pattern center (red dot) has been shifted by about $\vartheta_{(11\bar{2}0)}$ and the diffraction length has been increased by 5 %, as compared to the correct values, for indexing.

- [1] R.A. Schwarzer: Measurement of macrotexture by automated crystal orientation mapping: an alternative to X-ray diffraction. *Mat. Sci. Technol.* **16** (2000) 1384-1388
- [2] R.A. Schwarzer: Automated Crystal Lattice Orientation Measurement Using a Computer-controlled SEM. *Micron* **28** (1997) 249-265

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