# Vibratory Polishing of High-Purity Metals to Improve EBSD Results

EBSD experiments evaluated the band contrast of eighteen (18) high-purity (generally >99.95%) specimens mechanically prepared using five steps (four for Ti). These specimens varied from Mg (atomic number 12) to Bi (atomic number 83) and covered the range of metallic crystal structures: body-centered cubic (6), face-centered cubic (4), hexagonal close-packed (5), diamond cubic (1) and rhombohedral/trigonal (2). The table below lists the specimens prepared using our standard methods and analyzed. Results for six of these after vibratory polishing are shown in the second table.

Specimens of pure Sb, V and Zr were susceptible to SiC embedment, even though the grit size was coarse, e.g., 240- and 320-grit. Hence, grinding was repeated after coating the paper with paraffin wax. Attack polishing was used, mainly with 30% conc. $H_2O_2$, for the last step for preparing Cr, Nb, Ti, W and Zr. MasterMet® colloidal silica was used for the last step, except for preparing Fe (MasterPrep® alumina was used) and Mg (water-free MasterPolish® was used). Oil-based diamond suspensions (9-, 3- and 1-μm) were used to prepare the high-purity (99.999%) Mg. For the Bi and Pb pure specimens, grinding used four steps: 240-, 320-, 400- and 600-grit SiC paper coated with paraffin wax with low loads, followed by three polishing steps using 5-, 1- and 0.3-μm alumina slurries and a final polish with MasterMet® colloidal silica. All polishing steps used MicroCloth® synthetic suede cloth. Although the Bi produced an excellent EBSD pattern, none was obtained with the pure Pb specimen. A one-hour vibratory polish with MasterMet® colloidal silica using a MicroCloth® pad was required to obtain a diffraction pattern for Pb.

The result for pure Zr in the table below was obtained on the same specimen as illustrated here, but after an earlier preparation attempt with a less effective preparation method than used to generate the grain maps. The average band contrast for the high-purity Zr specimen using the improved preparation method was 92.34 and ~90% of the pixels produced indexable diffraction patterns. For the initial effort with high-purity Zr, which yielded an average band contrast of 77.3, only about 20% of the pixels in the SEM field yielded indexable pixels. Preparation methods are improved when they do not perform adequately. Zr is among the most difficult metals to prepare for EBSD.

Two maps of high-purity (99.99%), annealed Zr are shown. The first was constructed by adding an all Euler grain map with a band contrast map; the second shows an inverse pole figure map figure map, plus grain boundaries, with the grains with missing pixels (black spots in the first map) filled in. The band contrast averaged 92.34 for the area shown.

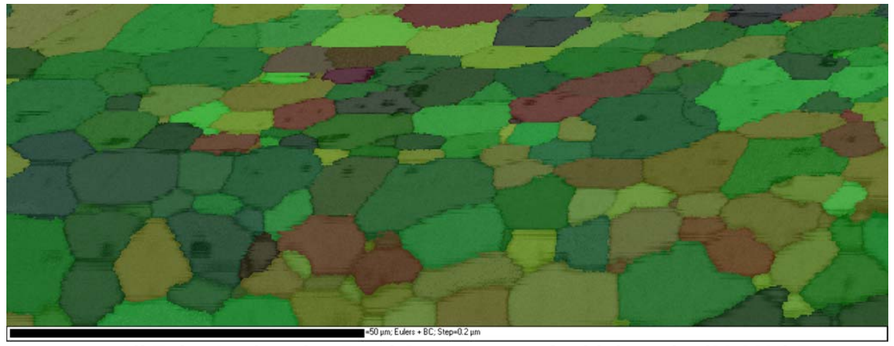
**Band Contrast Values for 18 Pure Metals**

| High-Purity Elements | Atomic Number | Crystal Structure | Band Contrast (0-255) |
|---|---|---|---|
| Mg | 12 | hcp | 161.2 |
| Al | 13 | fcc | 151.2 |
| Si | 14 | diamond cubic | 205.75 |
| Ti | 22 | hcp | 134.0 |
| V | 23 | bcc | 102.2 |
| Cr | 24 | bcc | 88.27 |
| Fe | 26 | bcc | 105.4 |
| Ni | 28 | fcc | 85.0 |
| Cu | 29 | fcc | 122.6 |
| Zn | 30 | hcp | 170.8 |
| Zr | 40 | hcp | 77.3 |
| Nb | 41 | bcc | 145.6 |
| Ru | 44 | hcp | 66.0 |
| Sb | 51 | rhombohedral | 180.2 |
| Ta | 73 | bcc | 122.8 |
| W | 74 | bcc | 91.6 |
| Pb | 82 | fcc | no pattern |
| Bi | 83 | rhomb./trigonal | 255 |

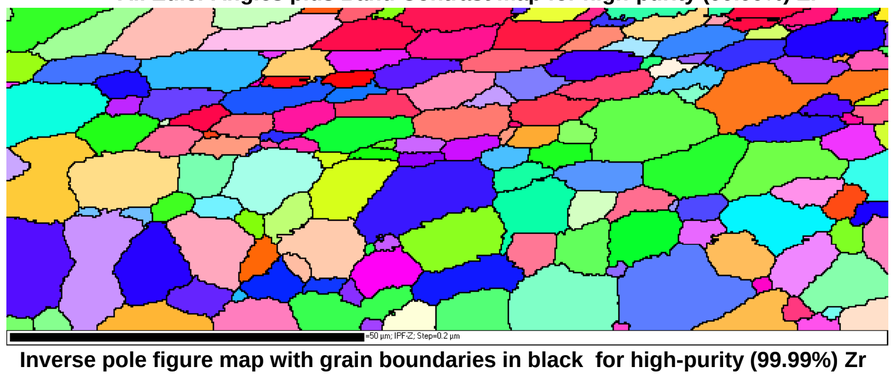
**Band Contrast Improvement Due to Vibratory Polishing (20 min.*)**

| High-Purity Element | Mean Band Contrast (0 to 255) | |
|---|---|---|
| | Standard Method | Standard + Vibratory Polish |
| Mg | 161.2 | 175.25 (+8.7%) |
| Si (single crystal) | 205.75 | 233 (+13.2%) |
| Ti | 134.0 | 146.2 (+9.1%) |
| Ni | 85.0 | 102.8 (+20.9%) |
| Nb | 145.6 | 151.2 (+3.8%) |
| Pb | No pattern | 108.0 |

* A 60 minute vibratory polish was used for the lead specimen.



**All Euler Angles plus Band Contrast map for high-purity (99.99%) Zr**

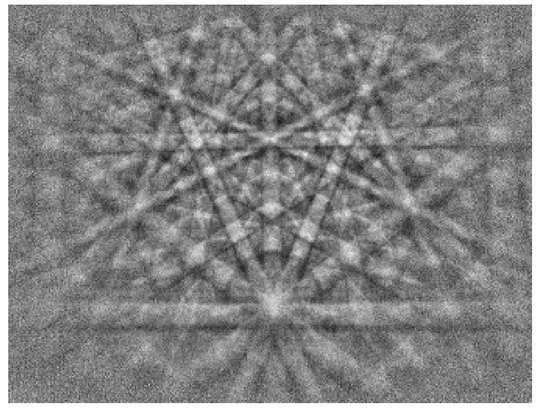


**Inverse pole figure map with grain boundaries in black for high-purity (99.99%) Zr**

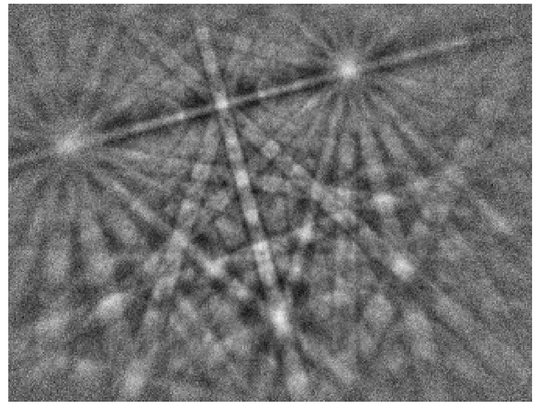
**Improved Preparation Method for Zirconium and Alloys**

| Surface | Abrasive | Lubricant | Force **(lbs)** | Speed **(rpm)** | Direction | Time **(minutes)** |
|---|---|---|---|---|---|---|
| SiC Paper **(wax coated)** | 240-grit | Water | 5 | 240 | Contra | Until Planar |
| SiC Paper **(wax coated)** | 320-grit | Water | 5 | 240 | Contra | 1 |
| UltraPol silk cloth | 9-μm Diamond Paste | MetaDi Fluid | 5 | 200 | Contra | 10 |
| TriDent polyester cloth | 3-μm Diamond Paste | MetaDi Fluid | 5 | 200 | Contra | 7 |
| TriDent polyester cloth | 1-μm Diamond Paste | MetaDi Fluid | 5 | 200 | Contra | 5 |
| MicroCloth pad | 0.05-μm Colloidal Silica* | None | 6 | 200 | Contra | 7 |

* Attack polish with 1 part $H_2O_2$ (30%) to 5 parts Colloidal Silica



**EBSD Pattern for annealed 99.99% Zr**



**EBSD Pattern for EB Melted (>99%) Zr (hot extruded and cold drawn)**

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