ANALYTICAL SERVICES

Sample Preparation of Copper for EBSD Analysis: How Far Do You Really Need to Go?

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Introduction

Copper is used in a multitude of ways: plumbing, electronics, heat exchangers, antimicrobial surfaces, and the occasional piece of strange clothing that makes bold claims about arthritis relief. That broad range of uses comes straight from copper's defining traits. It is ductile, conductive, and soft. Those same great traits are exactly why preparing for EBSD can be a real pain.

As a service lab, we routinely receive customer samples in a "mirror finish," claimed to be EBSD-ready because they appear flawless under an optical microscope. Then we put them in the SEM, and the data is completely unusable. That is copper holding onto prep damage below the beam's interaction volume, right where the EBSD patterns are generated. The surface looks perfect. The layer the patterns actually come from is wrecked.

In this quick study, we examine several mechanical prep workflows and how each affects EBSD data quality. We used two generic copper plumbing fittings from a certain large, orange-branded hardware store. One sample we left as-is, straight out of the box. The other we annealed to give us a low-deformation reference. That gives us a surface where anything we see in the maps is prep damage, not intrinsic strain from cold working. Running both side by side in the same mount tells us whether we are looking at real deformation or prep deformation.

We prepared the samples four ways, from a quick by-eye "mirror" polish all the way up to a 5-hour vibratory run. We left ion milling out of this note since it gets its own in the near future. For each state we looked at indexing rate, band contrast, GND density, and KAM, plus the maps themselves. The goal is a quick, practical look at how much prep you actually need based on the data you want out of it, with an eye on the prep time you are spending to get there.

Material

Both samples were generic C12200 (DHP) copper plumbing fittings. One was left in its as-received, drawn-tube condition. The other was annealed at 500°C for 2 hours and then air cooled by simply opening the furnace door, which gave us a recrystallized structure with large grains and plenty of annealing twins. Both fittings were sectioned and mounted together in a single epoxy puck, then prepared side by side through every workflow. Mounting them together is the whole point. Both samples see the exact same prep, so any difference between them is real and not sample differences.

One note on the annealed structure. Copper forms abundant annealing twins during recrystallization, so the recrystallized grains contain numerous twin lamellae. EBSD grain detection treats the high-angle twin boundaries as grain boundaries and splits each parent grain into the parent plus its twins. As a result, the raw grain statistics report a finer and more elongated structure than is physically present. The parent grains are essentially equiaxed once the twins are accounted for.

Table 1 lists the four preparation workflows compared in this study. Each was prepared from scratch as an independent surface, with the mount ground back between states.

State	Steps	Final Step	Time
Mirror Polish	240 → 600 → 800 grit SiC, 3 µm diamond, 1 µm alumina, polished to a mirror by eye	1 µm alumina	~15 min
Standard Prep	240 → 400 → 600 → 800 → 1200 grit SiC, 3 µm diamond, 1 µm alumina (~2 min a step), 3 min OP-S	3 min OP-S	~20 min
Extended OP-S	Same as Standard Prep through 1 µm alumina, final OP-S extended to 25 min	25 min OP-S	~45 min
Vibratory	Standard Prep through 1 µm alumina, then 5 hr on a VibroMet 2 (30% amplitude first hour, 20% the rest)	5 hr vibratory	~20 min + 5 hr

Table 1. The four mechanical prep workflows compared in this study.

A Few Terms

A quick reference for the metrics used throughout this note:

- OP-S (oxide polishing suspension): a colloidal silica polishing suspension that acts as both a fine mechanical abrasive and a mild chemical etchant. The chemo-mechanical action removes subsurface deformation that purely mechanical polishing leaves behind.
- Band Contrast (BC): a measure of how sharp and well-defined the EBSD pattern is at each point. Higher is better. It is a good proxy for surface quality, since damaged or deformed material produces weaker, fuzzier patterns.
- KAM (Kernel Average Misorientation): the average misorientation between a point and its immediate neighbors. It picks up local lattice distortion, so both prep damage and real deformation raise it. Reported here from a 3×3 first-nearest-neighbor kernel.
- GND Density (Geometrically Necessary Dislocations): the dislocation density required to accommodate the local lattice curvature, calculated from misorientation gradients between neighboring points. High GND means a strained lattice, whether from prep damage or genuine cold work.

EBSD Acquisition

We ran every scan in AZtec's Resolution mode, the highest-resolution and slowest-speed camera setting. This lets us collect the best-quality data we can, so we do not confuse a poor surface with poor scan settings. For a prep study, that is the right call. We did not want to compromise the data through the camera settings.

All scans were collected on a Thermo Fisher Apreo S SEM with an Oxford Symmetry S2 EBSD detector. Settings were identical across every scan: 20 kV, 13 nA, ~15 mm working distance, 70° tilt, detector at 164.6 mm. Patterns came in at 1244 × 1024 (Resolution mode), 2.12 ms exposure, 2-frame averaging. Indexing was optimized BD against the Copper phase (68 reflectors), Hough resolution 60, 12 bands. Each scan was a 1024 × 704 raster at a 0.272 µm step size, covering about 282 × 188 µm (~720,000 points), and took roughly 80 minutes at ~146 Hz. KAM and GND were calculated afterward in AZtec Crystal.

Results

Here is each state in turn. For every prep state we show the band contrast (BC) and GND density maps for both samples, then say what we are looking at. All the numbers are in Table 2 at the end.

Mirror Polish

Both samples came off this prep looking like mirrors. Optically you would call them ready. The EBSD disagreed.

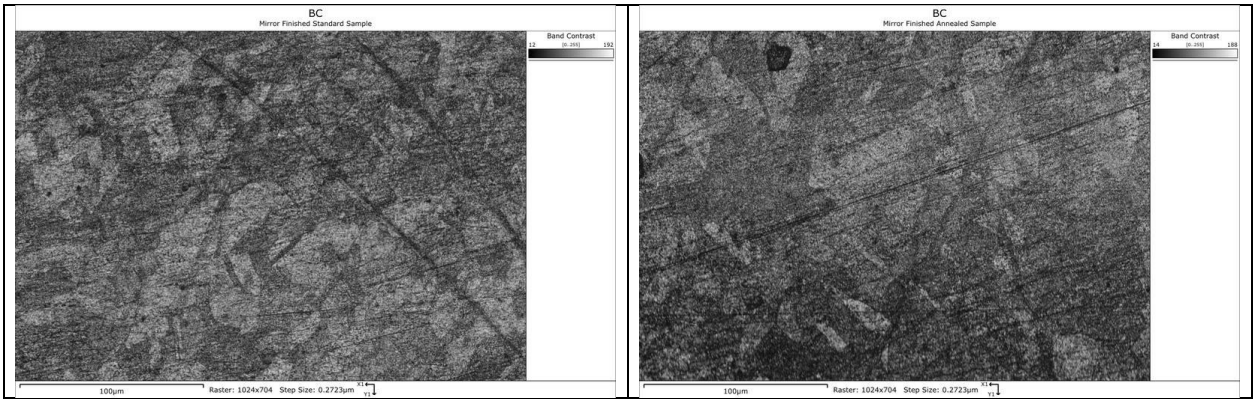

Figure 1. Band contrast after Mirror Polish. Left: as-received (57.6% indexing). Right: annealed (51.7%). Directional smear and broad noise dominate; grain structure is barely there.

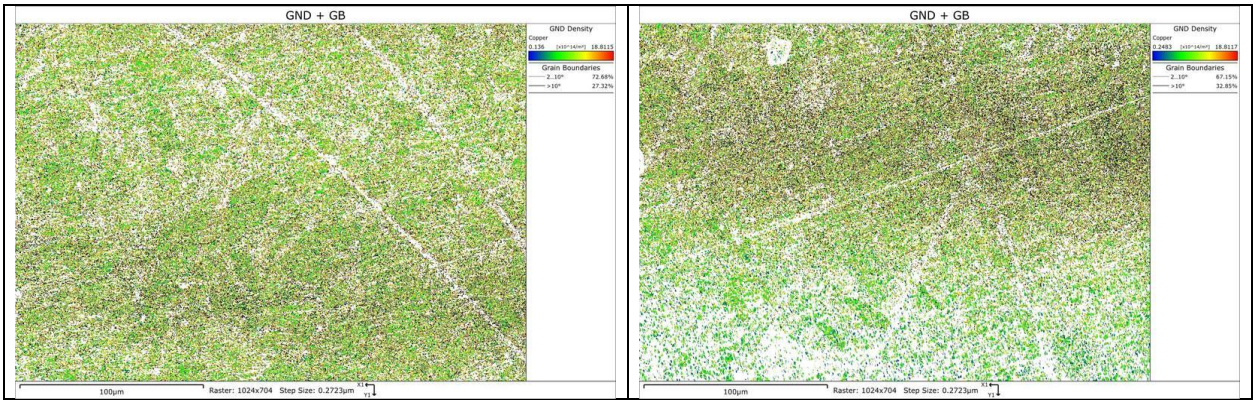

Figure 2. GND density after Mirror Polish (left: as-received, right: annealed). Both maps are showing elevated values across the whole field. The prep damage layer is dominating everything.

Indexing fell below 60% on both samples (57.6% and 51.7%) and band contrast was poor (92 and 88). The clearest sign of the problem is in the KAM. Both samples gave the exact same mean KAM, 1.64°. The prep damage is so heavy that it completely buries any real difference between the cold-worked and annealed structures, so the two come out indistinguishable. Grain detection also chopped each scan into more than 5,000 “grains,” almost none of them real. That is just the damage layer breaking the orientation data into fragments. Mirror Polish is not a usable EBSD prep for copper, no matter how good it looks under the optical scope.

Standard Prep

This is the textbook approach. Full grit sequence, diamond and alumina, short 3-minute OP-S finish. Most people would call this a perfectly thorough EBSD prep.

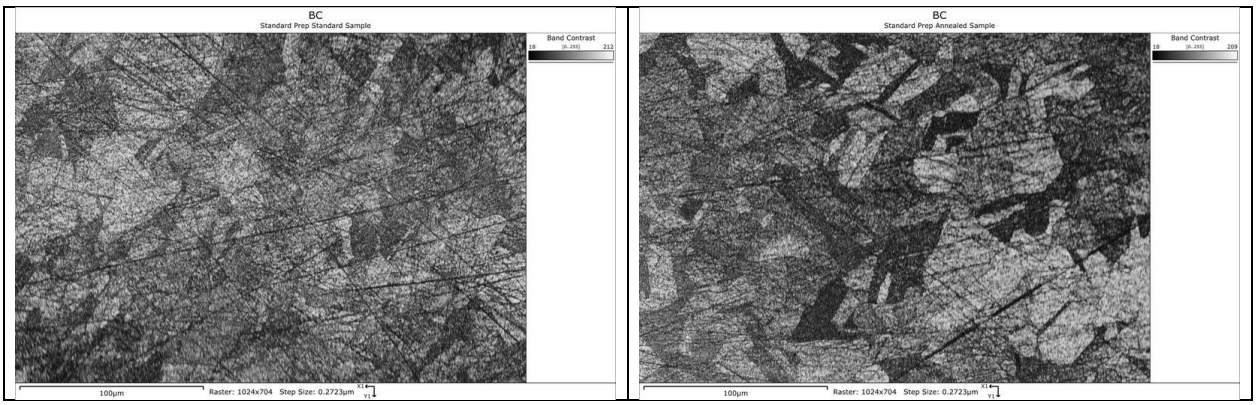

Figure 3. Band contrast after Standard Prep (left: as-received, 70.6%; right: annealed, 68.1%). Grain structure shows up now, but scratches and intra-grain noise are still all over it.

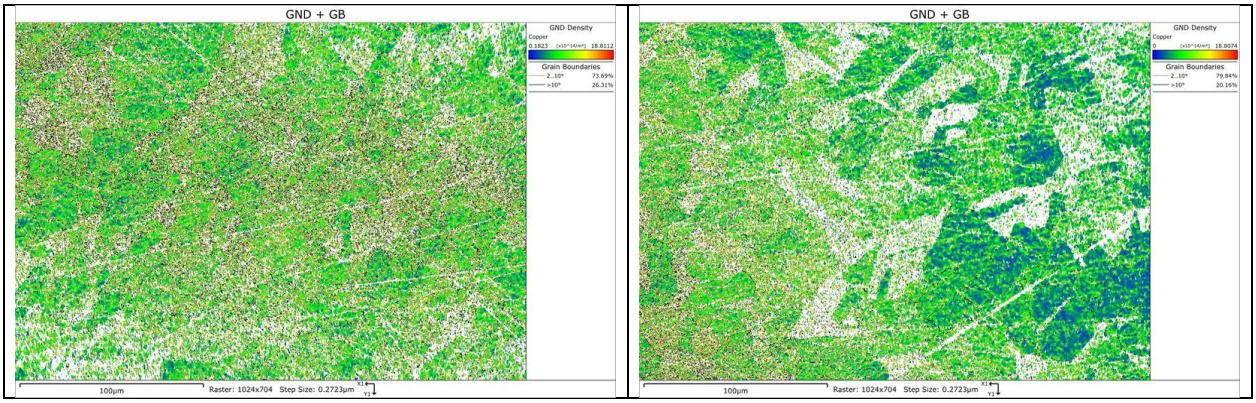

Figure 4. GND density after Standard Prep. Better, but still plenty of GND through both samples.

Standard Prep is a real step up. Indexing climbed to about 70%, mean band contrast rose from the Mirror Polish values of 88 and 92 up to 105 and 110, and grain structure is visible. But both maps still carry a lot of residual damage. Mean GND on the annealed sample sat at 6.62 $\times 10^{14}/m^2$, about an order of magnitude higher than what well-prepped annealed copper should read. Grain count dropped to ~2,000, better but still inflated by damage fragmentation.

Worth a note here. If all you need is grain sizing and orientation stats, Standard Prep plus a generic noise-reduction cleanup in AZtec will get you statistically valid grain data. You do not strictly need to go further for that. But the jump from here to Extended OP-S costs you about 20 extra minutes and buys you an enormous quality improvement, so for most work, why not just take the jump?

Extended OP-S

Same prep as Standard through the 1 µm alumina step, but the final OP-S goes from 3 minutes out to 25. Colloidal silica works as both a fine abrasive and a mild chemical etchant, and the extra time lets that chemo-mechanical action clear out damage that mechanical polishing alone just cannot reach.

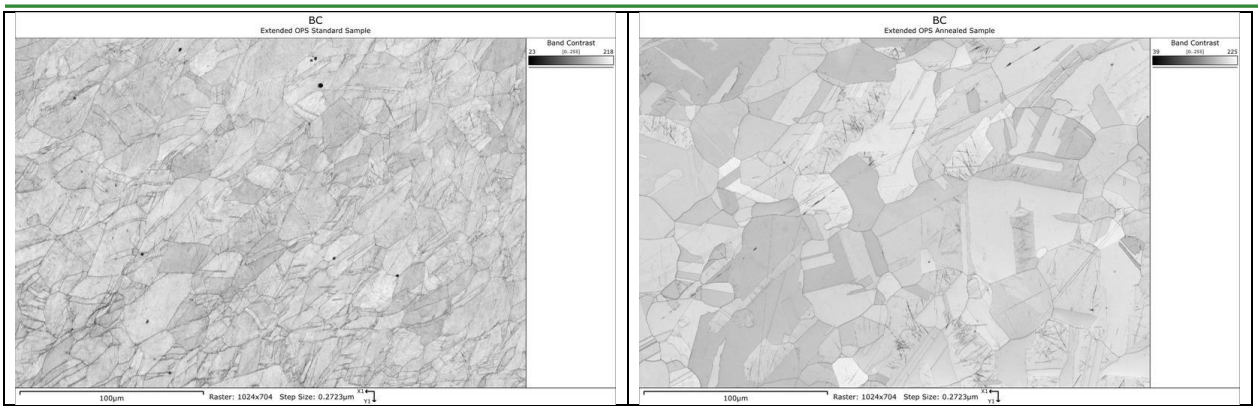

Figure 5. Band contrast after Extended OP-S (left: as-received, 99.7%; right: annealed, 99.8%). Grains and twin boundaries are sharp now. The grain-scale contrast in the as-received map is real cold-work deformation, not prep artifact.

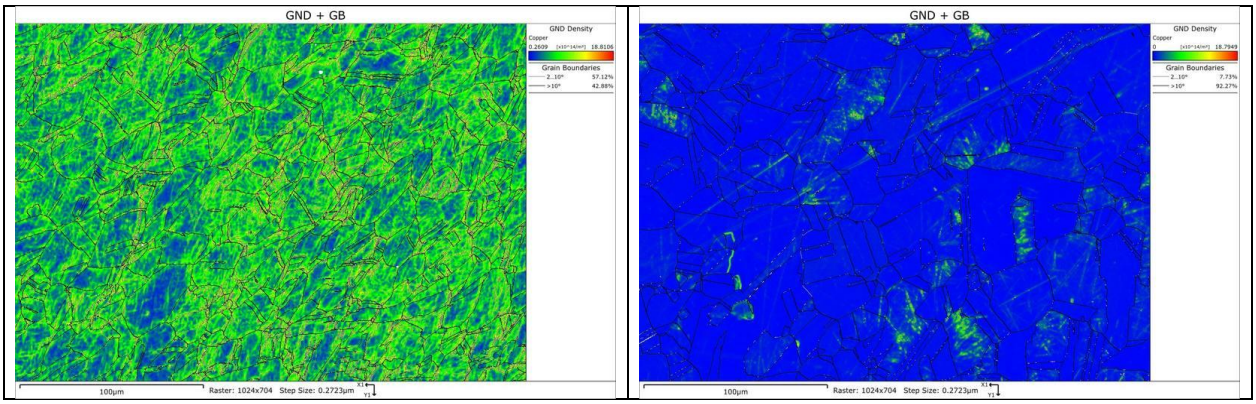

Figure 6. GND density after Extended OP-S. The annealed sample (right) is mostly clean blue, but look closely and you can still see faint green scratch lines cutting across grains. The wheel did not quite get everything.

Extended OP-S is where everything jumps. Indexing went from ~70% to 99.7% and 99.8%, band contrast nearly doubled to 176 and 191, and mean GND on the annealed sample dropped to 0.61 $\times 10^{14}/m^2$, about 17 times lower than Standard Prep. KAM on the annealed sample fell to 0.13° and grain count dropped to 396, right about where the real grain-plus-twin count should be. At this point the surface is good enough for the large majority of analytical EBSD work, including grain sizing and morphology, texture and orientation analysis, and phase mapping. Keep an eye on those faint scratches still visible in the GND map though, they matter for the next state.

Vibratory

Vibratory swaps the Extended OP-S step for five hours on the VibroMet with colloidal silica. It is a low-energy, long-duration version of the same chemo-mechanical action. Same idea as OP-S on a wheel, just spread out evenly over the surface, at the cost of a lot more hands-off time on the machine.

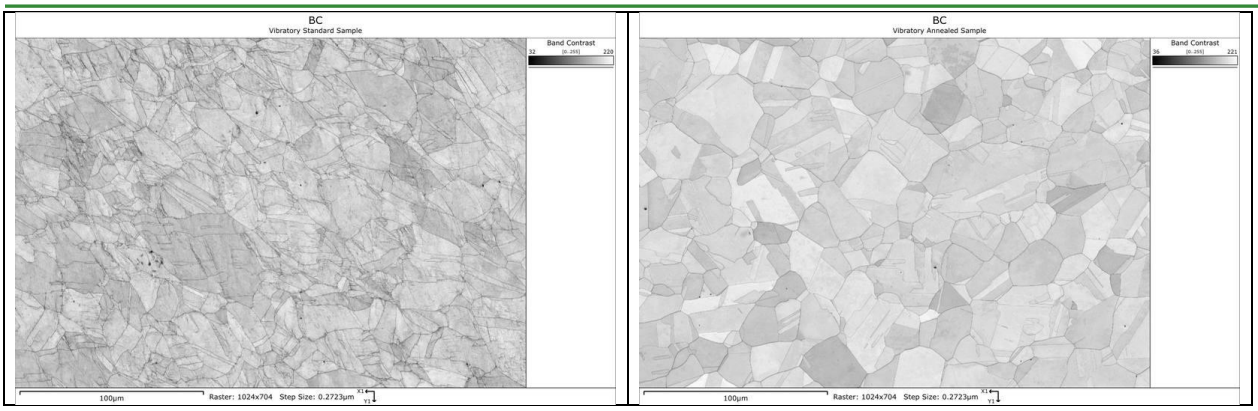

Figure 7. Band contrast after Vibratory (left: as-received, 99.7%; right: annealed, 99.8%). The annealed map shows cleaner grain interiors and sharper boundaries than Extended OP-S (Figure 5, right).

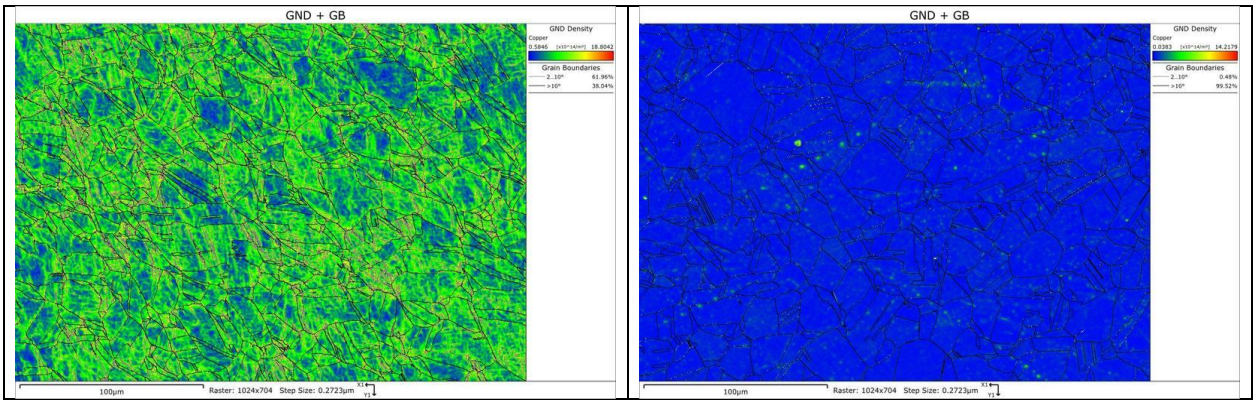

Figure 8. GND density after Vibratory. The annealed sample (right) is almost uniformly blue. The residual scratches from Extended OP-S (Figure 6, right) are gone.

Here is the interesting bit. By the numbers, vibratory is basically identical to Extended OP-S. Mean GND 0.63 $\times 10^{14}/m^2$ and KAM 0.12° on the annealed sample, versus 0.61 and 0.13. The averages say they are the same. But put the GND maps next to each other (Figure 6 vs Figure 8) and the residual scratches that were still there after Extended OP-S are gone after vibratory. The averages miss it, but the standard deviations catch it. GND standard deviation on the annealed vibratory sample is 0.32, versus 0.83 for Extended OP-S, about 2.5× lower, with KAM following the same pattern. Vibratory does not lower the average damage, because that is already on the floor. What it does is make the surface uniform.

So for grain sizing, texture, and orientation work, you do not need to go to vibratory. Extended OP-S already gives you statistically valid grain data and bulk metrics on the floor. Vibratory earns its five hours when you need pixel-level uniformity. Cases that benefit include high-resolution GND and residual strain measurements, intragranular misorientation and deformation studies, fine defect and slip-trace characterization, and EBSD maps intended for publication.

The Numbers

Table 2 pulls everything together. The bold rows are the two annealed states where the bulk metrics have bottomed out. Notice how close their means are, and how far apart their GND standard deviations are. That gap is the whole story of Extended OP-S versus vibratory.

State	Sample	Index %	BC	GND mean	GND std	KAM°	Grains
Mirror	As-Received	57.6	92	10.85	3.22	1.64	5222
Mirror	Annealed	51.7	88	10.58	3.58	1.64	5489
Standard	As-Received	70.6	110	8.24	3.17	1.43	2606
Standard	Annealed	68.1	105	6.62	3.11	1.17	2034
Ext. OP-S	As-Received	99.7	176	5.84	2.57	0.92	690
Ext. OP-S	**Annealed**	**99.8**	**191**	**0.61**	**0.83**	**0.13**	**396**
Vibratory	As-Received	99.7	176	6.33	2.67	1.00	749
Vibratory	**Annealed**	**99.8**	**189**	**0.63**	**0.32**	**0.12**	**660**

Table 2. EBSD metrics by prep state and sample. GND in ×10¹⁴/m². KAM from a 3×3 first-nearest-neighbor kernel. "Grains" is the detected grain count, inflated at the poor prep states by damage fragmentation. Index % is the fraction of points returning a valid solution.

Texture Check

The pole figures are a quick sanity check that the two samples really are in the states we think they are. The as-received sample carries a stronger texture from the original tube-drawing process. The annealed sample is more randomized after recrystallization. Exactly what you would expect if the anneal did its job.

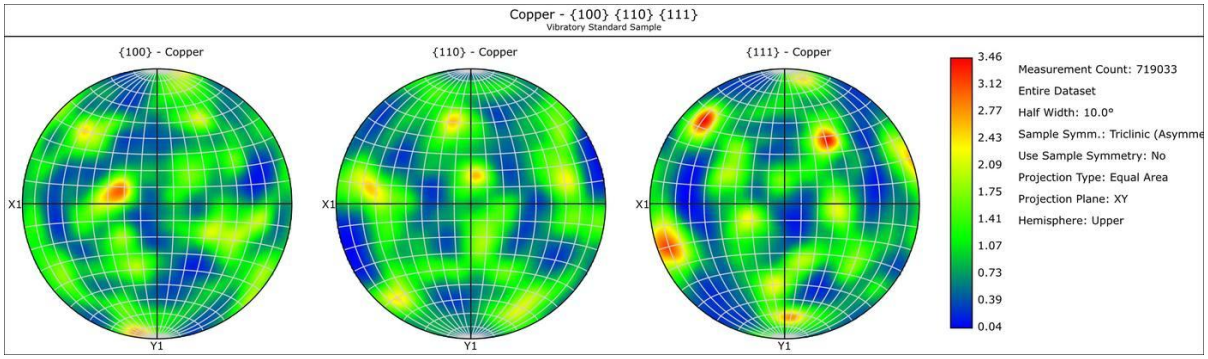

Figure 9a. {100}, {110}, {111} pole figures, as-received sample. Stronger texture from cold drawing.

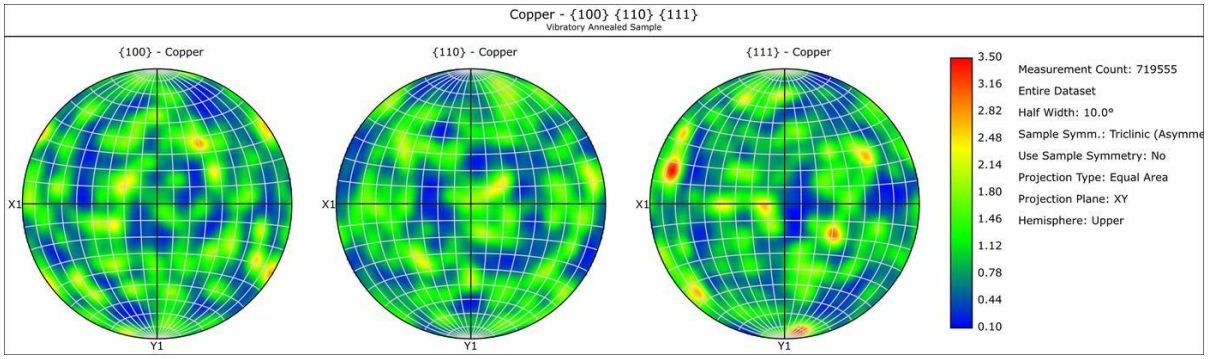

Figure 9b. {100}, {110}, {111} pole figures, annealed sample. More randomized after recrystallization.

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Recommendations

Pulling it all together, here is how we would approach EBSD prep on copper, depending on what you are after:

- Do not bother with Mirror Polish or Standard-Prep-only for serious analytical EBSD. Mirror Polish is not sufficient for EBSD. Standard Prep gets you to ~70% indexing with real residual damage.
- If all you need is grain sizing, texture, or orientation statistics, Standard Prep with a generic AZtec cleanup will give you statistically valid grain data. You can stop there if that is genuinely all you need.
- For just about everything else, go to Extended OP-S. It costs about 20 minutes more than Standard Prep and drops mean GND by roughly 17×, putting every bulk metric on the instrument floor. The quality jump per minute spent is enormous. It is the sweet spot for routine analytical work.
- Vibratory polishing buys spatial uniformity, not a better average. The bulk numbers match Extended OP-S, but the standard deviations drop ~2.5× and the residual scratches disappear. Spend the five hours when you need pixel-level uniformity, like high-resolution GND/strain work, defect characterization, or publication-quality maps.
- When you are characterizing deformation in a real copper part, run an annealed reference alongside it if you can. The annealed sample shows you where your noise floor is, so you can tell prep artifacts from the real cold-work deformation you are trying to measure.

Summary

We compared four mechanical preparation workflows for EBSD on annealed and as-received C12200 copper. The key findings are summarized below.

- A mirror finish under an optical scope tells you nothing about EBSD readiness on copper. Our mirror-polished samples looked perfect and gave sub-60% indexing dominated by prep damage.
- Textbook short-step prep with a quick OP-S finish is not enough for analytical EBSD on copper. Indexing tops out around 70% with real residual damage, though it is fine for basic grain sizing with an AZtec cleanup.
- Extending the OP-S polish to 25 minutes is the big win. Indexing above 99.7%, band contrast nearly doubled, mean GND down 17×. For about 20 extra minutes, it is the obvious move for routine analytical work.
- Five hours of vibratory does not beat Extended OP-S on the averages, but it makes the surface uniform. GND and KAM standard deviations drop ~2.5×, and the residual scratches vanish. Worth it when every pixel has to count.
- Running an annealed reference next to your real sample lets you separate prep artifacts from real deformation. The two only pull apart once the prep stops generating its own signal.
- Ion milling, both as a finishing step and as a direct prep route, gets its own note soon.

The JH team is always available for quality support when needed.