Crystal Orientation Procedure

Cutting the Orientation Specimen

• Use the temporary boule holder to cut two orientation specimens from the boule. Cuts should be made at the extreme ends of the boule and waste should be minimized. The geometry of the orientation specimen should be made to distinguish front from back and retain the correct orientation to the boule as shown in Figure 1. See EDM and X-Ray procedures below.

• Have a keyed boule holder made by the AEM Shop for your boule. (The shop has the program on file.) While waiting for the key to be made, you can orient one of the original photos to speed up the orientation process - the orientation specimen you cut out of the boule after it is mounted in the keyed holder will be rotated by some angle, but you will already have the spots identified.

• After you have determined that you have a single crystal boule, secure the boule in a keyed boule holder shown in Figure 2.

• Using the EDM keyed mount shown in Figure 3, cut an additional orientation specimen for your first orientation of the boule. Follow the EDM, X-Ray, and Orientation procedures below.

EDM Procedure

See Dave Hultman in the AEM shop for training and scheduling on the EDM.

• Turn on Power and CRT (switches on right hand side).

• Check EPack to ensure flushing is on low. Turn FLUID ON and check water level by observing float stick - white region should be visible.

• Align part using square and dial indicator.

• Use EDGE POSI. to reset zeros. Press MANUAL DRIVE and use +X,-X,+Y and -Y buttons to move wire close to part (speed can be varied with HIGH SPEED, MEDIUM SPEED, x5 and x1 buttons). Press EDGE POSI., press direction to move (+X, or -X), when screen says position end press EDGE POSI. button again, Press X and C-B to clear block setting x to zero. Repeat for y direction.

• Turn water on with the FLUID ON button and adjust fluid level with WEAK valve to a steady stream. Minimize the amount of air entrained in the water. Adjust top and bottom water levels with the UP and DOWN dials. Move head down to 1-3 inches from part. Shut off water with the FLUID ON button.

• Select appropriate EPack and Feed Rate from the blue "C Series Skim Cut Data" notebook for the wire size and sample thickness. Usual EDM settings for orientation sample rough cuts in Copper alloys are an EPack of 691 and Feed Rate of 0.075.

• Use a program sent to the EDM by the computer or make an MDI program at the EDM. To make an MDI program, Press DISPLAY, select PROG. HANDLING, and MDI. Change commands to create your own program. The program used to make the cut shown in Figure 4 contains the following lines (comments shown in <> not to be entered):

```
F0.075; < desired feed rate >
M80; < standard line >
M82; < standard line >
M84; < standard line >
G90; < standard line >

G92 X0,Y0; < standard line >
G01 X0,Y-0.6; < cuts from 0,0 to 0,-.6 >
G01 X0.1,Y-0.7; < cuts to .1,-.7 >
G01 X0.1,Y0; < cuts to .1,0 >
M02; < standard line >
% < end program >
```

When the program is complete press INPUT.

• To display the program press: DISPLAY, 6 (for graphic), 1 (for program check), 4 (for program), M, D, I, INPUT, 5 (for viewpoint), 1 (on lower key pad), INPUT, 7 (for frame), *, INPUT, and 8.

• Press MANUAL DRIVE to position wire at desired start point. Then press RESET twice. Press AUTODRIVE, check to ensure MDI is highlighted if using a manual program, and enter EPack by pressing E, then the EPack number, and INPUT.

• Check EPack settings by pressing E SET. Make the following adjustments: LQ should be on 1 for low flushing unless the guards are up; IP should be at 3 or 4 at the start of the cut, then it can be increased; and WS should be reduced to 5 or 6 unless you are on the final skim cut.

• Enter the feed rate by pressing F, then the Feed Rate, and INPUT.

• Press START to make the cut.

• Press STOP to end the cut.

• To manually complete a program that was stopped for some reason, Press FLUID ON, WIRE ON, MACHINING, and START pausing between each.

• For rethreading and fine wire instructions see Wendy's lab notebook, b2p8-9.

• When done machining, press MANUAL DRIVE and x1, then turn off the CRT.

Backscatter Laue X-Ray Diffraction

You **must** contact Linda Sauer, 350 Shepherd Lab, 625-0776, for training on the x-ray system prior to use.

Schedule time on the Siemens Wide Angle in 354 by calling 626-7594 or using the World Wide Web scheduling system at http://charfacnu.cie.umn.edu/CF_SignUp.html. There is a one hour minimum charge. (You must get training and a user code from CIE before you can sign up.)

To obtain film, place an order with University Stores for Polaroid 57, PN 511. The film in the refrigerator of 354 Shepherd can be used; be sure to record the number of films used in the log book.

• Prior to performing x-ray diffraction on a sample, it must be chemically etched to remove the oxide layer (for Copper alloys, use 50% Nitric Acid (HNO_3) and 50% Water at room temperature for 3-5 minutes, rinse in Water). Avoid touching the specimen surface prior to x-ray to minimize contamination. If the sample is polycrystalline, often grains can be observed after the chemical etching process. For experiment samples, thickness measurement before and after etching is usually desirable.

• Get goniometer and film from 130C.

• The x-ray machine is in 354 Shepherd (door combination 13-5).

• Make log book entries.

- Put on dosimeter ring.

- Attach specimen to goniometer (usually with double sided tape) and load goniometer onto track in x-ray machine with sample facing x-ray tube.

- Mount camera on track in between x-ray tube and sample. Move the slider shown in Figure 5 to the left so that the open hole is in the beam path - not the lead. Don't push the camera too close or you won't get a beam.

- Space sample 3 cm away from film using the spacer bar shown in Figure 5.

- Prepare camera for film - lever in back to the right, expose lever down as shown in Figure 5. Film is available in the refrigerator in 354 Shepherd if you don't have any. If using Characterization Facility film, record how many photos used in the comments section of the log book.

- Put the film in - push it all the way down and pull the paper back up. The 57 should be just above the top of the camera as shown in Figure 6.

- Flip the expose lever up.

- Set power on system. Dials should be turned in increments of one. 20 kV, 30mA and 8 minute exposer works well for copper but you may have to try various settings and times. For wide angle diffraction you can run at 45 kV and 40 mA with a shorter exposure time. It is OK to go with high powers, but this makes a larger white spot in the center.

- Close the door of the x-ray machine, turn on wide angle switch, open the shutter (□) and start the stop watch.

- Time the photo (8 minute is a good starting point). Lower power requires a longer exposure.

- When done close the shutter, turn wide angle off, open the door, and flip the expose lever down.

- Push the film paper all the way down (even with the top of the camera) and flip the lever in the back (shown in Figure 5) to the left.

- Pull the film up and out of the camera with a quick, even speed. Let it develop for 15 seconds.

• Peel off the film paper and discard - do not touch the chemicals. Label photo with location of image and specimen orientation.

• If the center of the photo is too washed out to see spots near the center, then the power is too high and the voltage should be reduced. 20 kV is usually good. If only a smeared annulus is observed (no spots), then you are probably taking a backscatter from the goniometer sample holder and not your specimen. This most often occurs when the goniometer is used to angle the specimen. The slider on the goniometer sample holder will have to be used to make sure that the beam still hits the specimen.

• Cover the photo with fixer and wash hands. Fixer takes a while to dry - do not allow the photo surface to touch anything while the fixer is still wet or you will ruin your photo.

• To determine if your boule is a single crystal, take one image on the same side of both orientation samples. It is also useful to take multiple images across one side of an orientation sample to assure that there are not multiple grains in the boule. The photos should look very similar if it is a single crystal.

• When done remove camera and goniometer. Return film to refrigerator.

• Turn power down to 10 kV and 5 mA.

• Log out.

• Remove ring.

Determining Your Current Orientation

• Draw a cross onto photo connecting the four small black dots as shown in Figure 7. Label N, S, E, and W on photo note that the photo is a mirror image as described by Figure 7.

• Number all x-ray spots (either on photo or on traced image as in Figure 8). If you have more than 20 spots, choose all of the bright ones and then several spots along 3 intersecting arcs.

• Use Greninger Net (for 3 cm spacing) to measure spot locations. Note that each square is 2° by 2°. Record spot locations in chart as shown in Table 1. Convert all X measurements to -X in the next column of the Table as shown since the photo is a mirror image. These values are used for plotting on the Wulff Net.

• Using the lines drawn on the photo as a guide, measure the location of all spots in millimeters (to the nearest 10th of a millimeter) using a caliper. Take X and Y measurements from the reference lines. The standard +/- values are used for these measurements. These values are used for the LAUE Program.

• Tape a square piece of tracing paper in center and pierce it with the tack on Wulff Net (tape side down). Fix anchor paper to Wulff Net with tape and mark N, S, E, and W poles as in Figure 9.

• With the tracing paper secured to the Wulff Net, plot the Greninger Net mirror values onto the Wulff Net like the example shown in Figure 10. Note that each square is 1° by 1°.

• Remove tape anchors from tracing paper and rotate the trace to draw arcs between spots that lie on Great Circles (complete arcs to the poles of the Wulff Net). Great Circles connect E to W on the Wulff Net as shown in Figure 9. You may want to check the original photograph to determine if spots lie on an arc by using the arcs on the Greninger Net if questions arise.

• Label the Zone Axis of each arc. The Zone Axis is 90° from the arc through the center of the the Wulff Net as demonstrated by figure 11. Zone Axes are useful for correcting the location of subsequent arcs. Important Zone Axes will be intersected by more than one arc. Note that the Zone Axis labeled in Figure 10 does not exactly coincide with the arc - ideally this should be corrected to get a more accurate orientation.

• The LAUE Program is available on the MAC in 301 in the LAUE PROGRAM folder. Open the RAWDATA file. Type all spot values into the file with the spot number. Values must be in 10x(millimeter value), i.e. the measurement +10.2,-13.5 for spot number 2 becomes 2,102,-135. Save this file as your own data file (see Table 2).

• Consider Wulff Net plot and look for three arcs that intersect to form a triangle with spots (preferably bright spots) at the vertices as demonstrated by Figure 12. For best results, consider two triangles that share a side so that results can be cross checked. If this is not possible, you can try to use three arcs that intersect at unknown locations, however this data may be unreliable. If this is the case, you should ideally take another photo at a different orientation (e.g. a 10° change in the goniometer position).

• Copy the list of spots in your file below itself and reorder the spot entries such that the vertex spots are the first three given. Save this file, copy the reordered spots to the RAWDATA file. Set 'a=1.5' on the first line of the file and remove the spot entry numbers (i.e. above entry becomes 102,-135). The value of 'a' is the error radius allowed when running the program. You want to use the smallest 'a' possible which gives you results. A very large 'a' will give many possible solutions.

• Run the LAUE APP. After the program is complete, copy the solution, press return to exit the program, reopen your file and paste the solution below the order used. If more than two solutions are generated you will not be able to copy them, you will have to rerun the program and manually write down the values and type them into your file.

• Notice that you usually get multiple solutions related by symmetry.

• Note that the program renumbers your spots starting with 0. Only the spots on the three arcs forming the triangle will have solutions. Carefully renumber the solution generated by the computer in your file and save it.

• If you have more arcs forming triangles repeat the process.

• After all of the runs are complete, transfer the direction information to the table (see Table 2). The runs should agree on shared spots. If the runs do not agree, add more spot locations and choose additional triangles. Two triangles that share a side are ideal. If the photo is over exposed and center spots washed out, the photo should probably be retaken at a lower power.

• Label the Wulff Net plot with the directions determined and see that they make sense.

You can corroborate your results using standard tables of angles between planes. See "Crystal Orientation Manual" by Elizabeth Wood; section 21 gives angles between planes in crystals of cubic symmetry. (Copper and Copper alloys have cubic crystal structures. In cubic structures directions and planes are the same.) The angles given can be measured using the Wulff Net. The chart angles should match the number of degrees between spots along the arcs you have drawn. You can do the entire orientation process with these charts, but the computer program removes a lot of the frustration associated with this process!!

Determining How to Move to a Desired Orientation

• The easiest move is if the desired specimen normal is shown as a spot on the Wulff Net. In this case, you measure the angle of rotation required to move the spot from its current location to the EW-axis or NS-axis. This value is the angle you must rotate your boule in the EDM fixture (about the u-axis) as shown in Figure 13.

• Next measure the number of degrees between your spot and the center of the Wulff Net along the EW-axis as shown in Figure 14. This angle is the amount of rotation about the w-axis.

• The Boule will only rotate in 15° increments. You may have to use a taper cut if an angle other than a multiple of 15° is desired. For instance, if you wanted a 10° rotation you would rotate the boule by 15° and taper the EDM wire by 5° as shown in Figure 15. Use the u (or v) displacement to offset the upper (or lower) hear to give the desired taper angle. Measure the exact distance between the heads to calculate the proper value for u (or v). The head must also be maintained at this position when cutting.

• If the desired specimen normal is not a spot shown on the Wulff Net, its location can be determined by finding angles between the [100], [010] and [001] directions. For instance to find the normal [a,b,c], calculate the angle θ (in degrees from [100]) between [100] and [010] using,

$$\theta = \tan^{-1}(b/a) \ .$$

Measure θ along the arc connecting [100] and [010]. Repeat this process for ϕ and χ. The angle ϕ between [100] and [001] measured from [100] is calculated using,

$$\phi = \tan^{-1}(c/a) \ .$$

The angle χ between [001] and [010] measured from [001] is calculated using,

$$\chi = \tan^{-1}(b/c) \ .$$

Connect these new points with the axis perpendicular to it. The three lines with cross at a point which it the location of the desired specimen normal as demonstrated by Figure 16.

• After the desired angles are calculated, perform EDM cutting and recheck the orientation. Often multiple iterations are required.

Revised by Wendy Grebner, 2/12/96.

Created by Wendy Grebner and Shad Jeseritz, 12/28/95.

References

"Crystal Orientation Manual", Elizabeth Wood, Columbia University Press.

"Elements of X-Ray Diffraction", B.D. Cullity, Addison-Wesley Publishing Co.

"Crystallography and Crystal Defects", A. Kelly and G.W. Groves, Longman Group Limited, 1970.

"Laue Solving Program Users Guide", Cameron Davidson, available in Wendy Grebner's Orientation Notebook.

"Interpretation of Back-Reflection Laue", available from Linda Sauer, 340 Shepherd Lab, 5-0776 (an alternative method).

Table 1. Example of spot locations and corresponding directions (for C2-01, 0° Back, Orientation 0).

Point	Greninger	Greninger E to W for Wulff Net	x,y (mm) x10 for Laue Program	Direction
1	-20.5, 4	20.5, 4	-27, 4.7	211
2	-11.5, 1.5	11.5, 1.5	-12.6, 1.5	311
3	-6, 12	6, 12	-6.4, 13.1	713
4	-1, 19	1, 19	-1.3, 22.7	201
5	4, 12	-4, 12	4.6, 13.2	301
6	6, 8	-6, 8	7.3, 8.9	401
7	14, -4	-14, -4	16.8, -4.6	100
8	2, -12	-2, -12	2.3, -13.4	401
9	unclear			
10	-9, -18.5	9, -18.5	-11.8, -23.2	201
11	-12.5, -12.5	12.5, -12.5	-15.6, -15.7	814
12	-10, -10	10, -10	-11.6, -11.7	
13	-15, -7	15, -7	-18.6, -8.6	412

Table 2. Example of Laue Program Results (for C2-01, 0° Back, Orientation 0).

1 -270,47
2 -126,15
3 -64,131
4 -13,227
5 46,132
6 73,89
7 168,-46
8 23,-134
10 -118,-232
11 -156,-157
12 -116,-117
13 -186,-86

a1.3
1 -270,47
7 168,-46
10 -118,-232
2 -126,15
3 -64,131
4 -13,227
5 46,132
6 73,89
8 23,-134
11 -156,-157
12 -116,-117
13 -186,-86

**** SOLUTION ****
point h k l
1 2 3 3
7 0 1 3
10 0 2 2
2 4 7 9
8 0 5 8
11 1 6 6
13 1 3 3

**** SOLUTION ****

point h k l

1 2 1 1

7 1 0 0

10 2 0 1

2 3 1 1

8 4 0 1

11 8 1 4

13 4 1 2

a1.3

2 -126,15

4 -13,227

7 168,-46

1 -270,47

3 -64,131

5 46,132

6 73,89

8 23,-134

10 -118,-232

11 -156,-157

12 -116,-117

13 -186,-86

**** SOLUTION ****

point h k l

2 3 1 1

4 2 0 1

7 2 1 2

1 7 2 1

3 7 1 3

5 8 1 5

6 10 2 7

13 4 2 1

**** SOLUTION ****

point	h k l
2	3 1 1
4	2 0 1
7	1 0 0
1	2 1 1
3	7 1 3
5	3 0 1
6	4 0 1
13	4 2 1